A STUDY ON THE USE OF NON-CONVENTIONAL ADDITIVES FOR STABILISATION OF EXPANSIVE SOILS

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ORIGINALITY STATEMENT

I hereby declare that this submission is my own work and to the best of my knowledge it contains no materials previously published or written by another person, or substantial proportions of material which have been accepted for the award of any other degree or diploma at University of Granada or any other educational institution, except where due acknowledgement is made in the thesis. Any contribution made to the research by others, with whom I have worked at University of Granada or elsewhere, is explicitly acknowledged in the thesis. I also declare that the intellectual content of this thesis is the product of my own work, except to the extent that assistance from others in the project's design and conception or in style, presentation and linguistic expression is acknowledged.

Signed ....

Date........................ 15th June 2014 ....

A boat, beneath a sunny sky
Lingering onward dreamily
In an evening of July

Children three that nestle near,
Eager eye and willing ear
Pleased a simple tale to hear

Long has paled that sunny sky:
Echoes fade and memories die:
Autumn frosts have slain July.

Still she haunts me, phantomwise
Alice moving under skies
Never seen by waking eyes.

Children yet, the tale to hear,
Eager eye and willing ear,
Lovingly shall nestle near.

In a Wonderland they lie,
Dreaming as the days go by,
Dreaming as the summers die:

Ever drifting down the stream
Lingering in the golden gleam
Life what is it but a dream?

Lewis Carroll

ABSTRACT

This dissertation describes an investigation on the use of non-conventional additives for the stabilisation of expansive soils. The main conclusions of this dissertation are based on the results of the original experimental program carried out. The physico-chemical characterisation and pozzolanic properties of industrial by-products such as dolomite ash and biomass ash (fly and bottom ash from different types of biomass) were studied and compared. Three sets of natural soils with poor engineering properties (including clays and marls) were obtained from the southeastern region of Spain to study the effects of the addition of non-conventional additives. The effects of magnesium hydroxide, seawater and olive mill wastewater on a pure bentonite soil were studied in terms of bearing capacity, swelling pressure, plasticity and mineral composition. A significant reduction in the plasticity and swelling potential was found to exist due to the treatment. The effects of the wetting-drying cycles in a lime-treated marly soil were also studied. The addition of biomass fly ash and dolomite ash to the clayey and marly soils led to coarser particle size distributions and reduced plasticity indices. A long study on the mineralogy of these treated soils by means of FESEM is presented to show the importance of the chemical composition of the natural soils (presence of carbonates and sulphates) and the mineral transformations caused by the treatments. The study on the mechanical behaviour and pH of the treated soils showed a significant correlation among strength parameters and an improvement in the properties of the soils when an alkaline environment is provided. Finally, a quantification of the importance of the stabilisation was carried out by modelling a real embankment and calculating the relationship between the strength parameters of the soil used as a fill and the overall stability of the embankment. Numerical models (finite element methods) were used for this purpose. The study showed that the factor of safety of the embankments is very sensitive to the improvements in the mechanical properties of the soils used. As a main conclusion of this dissertation, there is a wide range of non-conventional additives. Their use in this study promoted significant modifications in the physico-chemical properties and mechanical behaviour of the natural soils used.

RESUMEN

Esta tesis doctoral describe una investigación acerca del uso de agentes no convencionales para la estabilización de suelos expansivos. Las principales conclusiones de la tesis se basan en los resultados del programa experimental original realizado. Los sub-productos industriales utilizados, tales como la cal dolomítica y la ceniza de biomasa (procedente de distintas fuentes) han sido estudiados desde el punto de vista de la caracterización físico-química y las propiedades puzolánicas. Tres suelos naturales con pobres propiedades ingenieriles y principalmente formados por arcillas y margas fueron obtenidos de la región sureste de España para estudiar los efectos de la adición de agentes no convencionales. Los efectos del hidróxido de magnesio, el agua de mar o el alperujo sobre una bentonita pura fueron estudiados en términos de capacidad portante, presión de hinchamiento, plasticidad o composición mineralógica. Se comprobó la reducción significativa de la plasticidad y del potencial de hinchamiento en las muestras. Se estudiaron los efectos de los ciclos de curado de humectación-desecación sobre una muestra de marga estabilizada con cal. La adición de ceniza de biomasa y ceniza dolomítica a suelos arcillosos y margosos produjo una distribución más gruesa del tamaño de partícula y unos índices de plasticidad reducidos. Se presenta en esta tesis un estudio de la mineralogía de los suelos tratados llevado cabo por medio de microscopia de alta resolución FESEM. Esto muestra la importancia de la composición química del suelo natural (presencia de carbonatos y sulfatos) y las transformaciones mineralógicas producidas por los tratamientos. El estudio del comportamiento mecánico y del pH de los suelos tratados mostró la existencia de una correlación significativa entre los parámetros resistentes y la mejora de las propiedades de los suelos tratados cuando se proporciona a las mezclas un ambiente alcalino. Finalmente, la cuantificación de la importancia de la estabilización se llevó a cabo mediante la modelización de un terraplén y el cálculo de la relación existente entre los parámetros resistentes del suelo utilizado como relleno y la estabilidad global del terraplén. Para este propósito, se utilizaron modelos numéricos de elementos finitos. El estudio demostró que el factor de seguridad de los terraplenes estudiados es muy sensible a las mejoras de las propiedades mecánicas de los suelos utilizados. Como conclusión principal de esta tesis doctoral, hay un amplio abanico de agentes de estabilización no convencionales. Su uso en este estudio promovió importantes modificaciones en las propiedades físico-químicas y en el comportamiento mecánico de los suelos tratados.

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In my opinion, one of the most rewarding tasks of every project, and very especially of this PhD thesis, is the beautiful moment of relaxing for a short while and trying to remember the name of all those people who made this work possible with their modest and invaluable contributions.

Despite the fact that it may result a bit tedious for the reader, I wholeheartedly want to list the name of all those who encouraged me to go on working on this PhD. Dear reader, given the tight schedule we all face nowadays, please feel free to skip this section.

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This PhD is fully dedicated to the memory of my Father, the most special person I ever met.

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PREFACE

Over the last four years, I dedicated most of my time to study the effects of the addition of a wide range of materials on natural soils with low engineering properties. In the seek for a contribution to the scientific world, I found Science to be a significant contribution to my own world. The path I followed was long and windy and it made me travel around the globe and meet really interesting people. At some moments, it occupied all my thoughts and the course of my research did determine my mood. During this time, Science (and hence an analytical and sceptical way of thinking) was for me much more than an occupation: it was a lifestyle.

It is difficult to explain when and how a scientific research starts. The soil stabilisation problem is quite an old engineering issue to which many different solutions have been offered in the past. The use of lime, cement and coal fly ash developed in the 20th century was found to be an outstanding solution for the use of low-profile soils which were otherwise doomed to the undignified destiny of being disposed off in landfills. This solution, effective and valid as it is, had to face significant criticism at the turn of the 21st century. The lime, cement and coal fly ash were not always locally available, environmentally friendly and cost effective. A wide range of natural resources and industrial by-products forming the so-called non-conventional additives started to be tested in an attempt to find alternative construction solutions for a world whose needs change at an overwhelming speed.

From the very beginning of my involvement with the soil stabilisation research community, I tried to keep a holistic point of view, questioning not only the currently used additives, but also the procedures, the evaluation tests and the scientific theories behind. I could not say now that I was totally successful. But I really enjoyed the approach, which was a perfect excuse to use different types of soils, additives, tests and theories. I developed a particular interest in the relationship which may exist between the mineral modifications promoted by the addition of the stabilisation agents, the environmental chemistry and the mechanical behaviour of the soil. Therefore, I tried to study the relationships between the existence of carbonates and sulphates in the natural soil and the environmental conditions. The relationship between the environmental conditions and the formation of new minerals. The relationship between the formation of new minerals and the changes in the physical properties of the soil. And, finally, the existing relationship between the changes in the physical properties and the building up of an enhanced mechanical strength.

I tried to use the most sophisticated means to evaluate the results of my experiments. I compared the results of the traditional sieving or hydrometers with more modern laser diffraction methods to analyse the particle size distributions of fine-grained soils. I applied the FESEM microscopy and complemented it with EDS spectroscopy. I combined the geotechnical (compaction, bearing capacity, strength), physical (particle size, consistency, swelling potential), chemical (X-ray fluorescence) and mineral (X-ray diffraction) tests to characterise the soils.

As a professional civil engineer, I profoundly believe that all the previously described methods and all the derived findings would not be relevant without the application of a practical approach. I applied the most up-to-date earthworks design procedures (numerical modelling) to evaluate the feasibility of the newly proposed soil stabilisation additives and methods and their advantages for the real construction industry.

This dissertation is the culmination of that investigation I just described and an attempt to obtain a PhD degree. I dreamed of this moment many times and now that it is to come, I cannot help feeling a bit nostalgic. But, as someone told me once, “nostalgia is not what it is used to be”. I just can hope that this investigation will not represent the end of my academic career whatsoever, but the exit to a new road which is yet to be discovered. I am keen to follow the stars and feel again the excitement of the unpredictable adventure.

Finally, should the reader of this dissertation find any erratum and/or is keen to discuss any aspect with the author, please feel free to contact me at cgunieto@ugr.es.

Carlos G. Ureña
15th June 2014

INTRODUCTION

Chapter 1

Objectives of the Investigation

Abstract

In this chapter (Chapter 1), an academic background of this PhD dissertation is presented. The initial approach to the soil stabilisation problem and the objectives of the investigation are also described. A description of the structure of this dissertation is presented in this chapter. The next chapter (Chapter 2) is dedicated to the literature review and state of the art on soil stabilisation.

1.1 ACADEMIC BACKGROUND

The research work presented here is based on an original experimental program carried out during the last four years in the University of Granada (Spain), University of Jaen (Spain) and Imperial College London (UK), within the PhD programme on “Earth Sciences” of University of Granada, where the author developed his MSc studies on Engineering Geology.

The subject of this dissertation emerges from the identification of a real need in the construction industry of Southern Spain, where the existence of widespread outcrops of clayey and marly soils with poor engineering properties requested an effective solution, both from an economic and from an environmental point of view.

Despite the fact that this research is intimately related to the conditions of the construction industry and the geology in Southern Spain, the approach, the results and most of the interpretations carried out can be easily extrapolated to many other places, where the geological and industrial conditions are similar.

The stabilisation of unsuitable soils is a geotechnical solution which has been largely described in the existing literature and in international standards. However, that does not mean that everything is known, especially regarding the use of alternative additives. In the last few years, a significant increase in the amount of scientific contributions reporting experiments on soil stabilisation is clearly appreciated.

The modern society demands the construction of safe and reliable infrastructures. Hence, the quality required by the international standards for the earthworks included in highways, railways, etc. is increasing. In the case of the embankments, the quality will rely on the materials located at the lower layers. And the limited amount of fine-grained plastic and expansive soils in those layers will be crucial for the stability and durability of the embankment and to ensure a reduced maintenance cost when the embankment is subjected to variable environmental conditions.

To achieve this challenge, the current construction industry must face the reality of the limited availability of materials and financial resources. Hence, the use of the natural soils encountered in the excavations in lieu of spending valuable resources to replace them is an option which must be favoured. Even when the geotechnical experience might discourage the use of the natural soils encountered (for examples when they are clayey or marly soils with high plasticity or swelling potential), the soil stabilisation can offer an interesting solution. In the construction of linear infrastructures, the earthworks represent a major activity playing a significant role in the overall budget and having a dramatic influence on the environmental impact of the construction works. Hence, the importance of this line of investigation.

1.2 APPROACH

The unsatisfactory lime-stabilisation of soils reported in real construction sites in Southern Spain was one of the conditions which initially motivated the investigation presented here. On the other hand, even in the case of successful treatments, there are many reasons why the use of conventional additives (such as lime, cement and coal fly ash) might not be an adequate or desirable solution.

From the beginning of this investigation, the approach adopted by the author was to draw a detailed picture of the soil stabilisation problem, focusing on both the performance of new alternative stabilisation agents and on the reactions and process of the treatment itself.

On the other hand, attention has been paid to the mineral, mechanical, chemical and physical properties of the soils.

Due to the experimental approach followed, the idea of mixing natural soils with traditional and non-conventional additives was soon adopted. The natural soils used were obtained in the South of Spain. The geological setting of the soils used, which were mainly formed of marls and clays, is described in chapter 4 of this dissertation.

The first stage of this investigation was the study of the lime-stabilisation of marly soils. In real embankments formed of treated soils, the effects of the seasonal variations in the environmental conditions (temperature, moisture) is a key feature. To emulate those effects, the samples were subjected to wetting-drying cycles. This experiment allowed for a better explanation of the lime-stabilisation treatment, which is presented in the section of experimental results.

The next step of this research, was the use of economic and locally available resources to treat a very expansive soil. Magnesium hydroxide, seawater and olive mill wastewater were used to treat a very pure bentonite and the effects on the plasticity and swelling potential of the original soil were studied.

Once the lime-stabilisation treatment had been studied and cost-effective locally available materials had been used to modify the behaviour and physical properties of the soil, the search for suitable additives started. A review of some existing non-hazardous industrial by-products in the South of Spain and a characterisation of the physical and chemical properties of those products (including their pozzolanic activity) was carried out, leading to the selection of biomass fly ash and dolomite ash as the most suitable additives.

The effects of these additives on clayey and marly soils were studied and compared with the effects promoted by traditional additives (cement, lime, coal fly ash). The behaviour of the treated soils was studied from a mechanical point of view and the use of sophisticated means led to relevant findings on the mineralogy and chemistry of the stabilised soils.

Finally, an evaluation of the soil stabilisation treatment and the importance of the improvement was carried out according to the currently used methods for design of embankments (including numerical modelling with Finite Elements).

1.3 METHODOLOGY OF THE INVESTIGATION

The conclusions and interpretations of this investigation are based on original experimental tests. The methodology followed (tests, procedures, laboratory conditions, standards used, etc.) is presented in Chapter 5.

The investigation presented in this work has been developed in different stages, each of them aiming at providing answers for different scientific questions. Each of these stages of the investigation, are presented in different chapters in the section of Experimental Results. At each stage, the research was conceived to have a motivation, objective, laboratory experimental program, interpretation and conclusions.

Geotechnical characterisation of the soils has been extensively carried out, both for the natural and for the treated soils. For that characterisation, the following tests were undertaken: standard and modified Proctor compaction tests, California bearing ratio, swelling pressure, free swell in oedometer, particle size distribution (laser diffraction, hydrometer, sieving), Atterberg consistency limits test, etc.

For the mineral characterisation of the soils and additives, the X-ray diffraction (XRD) methods have been used: oriented aggregates and powder method. This has been combined with the use of X-ray fluorescence (XRF). A field emission scanning electron microscope (FESEM) has also been used to analyse the surface of the soil particles in the search for newly formed minerals due to the stabilisation treatment. The FESEM technique used included an energy dispersive X-ray spectroscopy (EDS).

The study of the pozzolanic properties of the test additives requested the use of the strength activity index (SAI) tests, the alkaline activity and the Frattini test, based on a thorough review of the existing literature.

Finally, the pH of the soil and the water has been consistently measured to account for the influence of this parameter on the treatments.

All of the experimental results presented in this dissertation, has been developed in the laboratories of the universities involved in the investigation and/or in those of the companies supporting some parts of it.

1.4 OBJECTIVES

The main objectives of this investigation are described as follows:

- A characterisation of the available additives, trying to highlight the most significant properties of them as soil stabilisation agents. Furthermore, a study of the suitability of additives according to the properties of the specific soil to be treated.

- A search for new stabilisation agents, with special attention to their pozzolanic properties.

- Studying the effects of the lime treatment focusing on the mineral composition of the marly soils and its influence on the overall performance of the treatment.

- The study of new additives (obtained from natural resources, industrial by-products, etc.) for soil stabilisation focusing on the effects of those additives on the plasticity and swelling potential of the most expansive soils.

- The soil stabilisation has been described as based on primary and secondary reactions. A further definition of the soil stabilisation reactions, regardless of the type of additive used, has been pursued.

- A study of the microstructure of the treated soils by means of high-resolution microscopy along with an evaluation of some of the existing control tests (such as traditional particle size distribution).

- A study of the influence of the environmental pH and the importance of the pH modifications promoted by the additives.

- Studying the mechanical behaviour and the evolution of the strength parameters in the soils treated with non-conventional additives such as biomass fly ash and dolomite ash.

- An evaluation of the soil stabilisation treatments by means of numerical modelling: quantification of the achieved improvements and its importance for the design of earthworks.

1.5 STRUCTURE OF THE DISSERTATION

This dissertation has been divided in chapters, each chapter providing information on a relevant part of the global content. The structure is divided into four major sections called: introduction, methodology, experimental results and conclusions. The section of experimental results has been divided into five sub-sections (Part I to Part V) according to the different stages of the experimental program or steps of the research.

The section of Introduction is formed of two chapters: Chapter 1 and Chapter 2. Chapter 1 presents the background, approach and objectives of the investigation. Chapter 2 presents the literature review and state of the art on soil stabilisation.

The section of Methodology is formed of chapters 3 and 4. In Chapter 3, the materials used in the research and the geological setting of the soils used is described. Chapter 4 presents a detailed description of the experimental procedures, tests carried out, standards followed and laboratory conditions.

The section of Experimental Results presents in five different subsections the results of the original research. The section is formed of ten chapters which have been grouped in pairs due to their close relationship.

Part I of Experimental Results focuses on the additives. Several (non-hazardous) industrial by-products obtained in different areas of Southern Spain are studied. Chapter 5 shows a physico-chemical characterisation of biomass ash from the olive oil industry and presents a comparison of its properties with those of lime and coal fly ash. In Chapter 6, six additives with potential to alter the physical properties of the soils and to develop pozzolanic reactions are studied and characterised.

The Part II of the Experimental Results is the preliminary study of this research. Chapter 7 shows the results, interpretation and conclusions of an experiment on the use of lime to treat natural marly soils subjected to wetting-drying cycles. Chapter 8 presents a study on the use of magnesium hydroxide, seawater and olive mill wastewater to modify the physical properties of a bentonite soil.

Part III of Experimental Results is about the stabilisation process. Chapter 9 evaluates the evolution of the particle size distribution of soils by means of laser diffraction. Chapter 10 is a study on the microstructure of the treated soils using FESEM microscopy.

Part IV is a study on the mechanical behaviour of clayey and marly soils subjected to addition of biomass fly ash and dolomite ash. The Chapter 11 presents the relationships among strength parameters whilst Chapter 12 shows the importance of the environmental pH.

Finally, Part V of the section of Experimental Results highlights the importance of the practical and realistic approach of the investigation. The results obtained in previous chapters are used here to carry out a sensitivity study on the importance of different parameters for the design of embankments. In Chapter 13, the improvements are evaluated using Plaxis2D (finite element method). In Chapter 14, a comparison of the factor of safety against the slope stability is presented for soils with different strength parameters. Slope/W is used for the numerical modelling.

The last section of the dissertation is the section of Conclusions, in which there is a list of concluding remarks and an outline of suggested further research (Chapter 15).

Chapter 2

State of the Art

Abstract

In this chapter (Chapter 2), a state of the art on soil stabilisation is presented to complete the section of Introduction. The literature review is always a rewarding but challenging task. In this chapter, the author tried to provide the reader with a comprehensive insight of the scientific problem approached in this dissertation. To do that, the most significant contributions to this field of investigation have been extracted and summarised. First, a definition of the term "expansive soils" is presented, along with a theoretical explanation of the mineralogy of marls and clays. Although the geological setting of the different marls and clays used along this study is presented in the following section, a geographic map of the main outcrops is provided in this chapter. In the second place, the stabilisation process is described, both from a theoretical point of view and from a practical point of view (history of the use of this technique). This chapter will also approach the sustainability issue and its importance and current relevance for the international construction industry. This will bring the reader to the results and conclusions of the recent studies on alternative (usually sustainable) stabilisation agents. The tests used to evaluate the improvements achieved with the stabilisation treatment will also be outlined. Finally, there is a section on soil mechanics and design of embankments using numerical modelling.

2.1 EXPANSIVE SOILS

One of the most common geotechnical problems in civil engineering is the failure of structures or pavements due to large swelling and shrinkage of the underlying soils. Sometimes, the effect of the expansive soils is related to the instability of slopes. When a slope formed of expansive soil is steeply dug for the construction of a linear infrastructure, an intense rainfall can promote landslides due to the greater weight of the saturated soil and the reduction of friction due to the presence of water.

Expansive soil is a term which is applied to the soils that expand in the presence of water and shrink when they dry out. The change in the soil volume causes differential settlements and cracking on buildings, structures, roads, etc. The economic damage caused by expansive soils in the United States every year is estimated to be higher than that caused by any other natural hazard and weather conditions. Hence the important of the identification of the expansive soils.

Tables 2.1, 2.2 and 2.3 show some of the proposed methods to identify and characterise expansive soils. Attempts to classify expansive soils have been developed by scientific researchers based on plasticity, shrinkage, swelling and activity.

Table 2.1

Soil expansivity prediction by liquid limit (Chen, 1975)

Degree of expansion	Liquid limit, w_L (%)
Low	< 30
Medium	30-40
High	40-60
Very high	> 60

Table 2.2

Soil expansivity prediction by plasticity index

Degree of expansion	Plasticity index, PI (%)	Plasticity index, PI (%)
	Holtz and Gibbs, 1956	Chen, 1975
Low	< 20	0-15
Medium	12-34	10-35
High	23-45	20-55
Very high	> 32	>35

According to **Chen (1975)**, there is no conclusive evidence of the correlation between swelling potential and shrinkage limit. **Sridharan and Prakash (1998)** also concluded that the shrinkage limit can not be satisfactorily used to predict the swelling potential of a soil given shrinkage and swelling are governed by entirely different mechanisms.

Table 2.3

Soil expansivity prediction by other measures

Degree of expansion	Colloid content, % minus 0.001 mm	Shrinkage limit, %	% expansion in oedometer	% expansion in oedometer
	Holtz and Gibbs, 1956	Holtz and Gibbs, 1956	Holtz and Gibbs, 1956	Seed et al., 1962
Low	<17	>13	<10	0-1.5
Medium	12-27	8-18	10-20	1.5-5.0
High	18-37	6-12	20-30	5-25
Very high	>27	< 10	>30	>25

To experience significant volume changes, apart from the expansive nature of the soil, there must be an important variation of the effective stress in the soil. Normally, the increase in the effective stress takes place when one of the following circumstances occur (**Pousada, 1984**):

- Change in the humidity, without significant changes in the total stress
- Change in the total stress, without significant changes in the humidity
- Variation of total stress and humidity

Another situation which can promote changes in the effective stress of the soil is the variation of the environmental salinity.

Therefore, the volume changes in the expansive soils are a consequence of the variation of the stress acting on the soil, both external (total stress) and internal (pore pressure) (**Pousada, 1984**).

The expansive soils are characterised by the presence of a large proportion of highly active clay minerals of the smectite group which are responsible for the pronounced volume change capability of the soils (**Sridharan and Prakash, 2000**). The swelling potential of expansive soils can be attributed to the swelling potential of clays. Without the presence of expansive clays, the behaviour of expansive soils cannot be understood. It must be said that the plasticity of the soil which is very relevant for the swelling potential does not only depend on the amount of clay but also in the type of clay mineral, hence the importance of the mineralogical study of the soil.

2.1.1 CLAYS

The definition of clays depends greatly on the discipline. Some of the most accepted classifications of soils in civil engineering are based on the particle size. According to this, clay is often define as a soil with a particle size smaller than 2µm, to it them from sands, silts and gravels **(Torrijo and Cortes, 2007)**. This classification of clays (particle size <2µm) is also common among Geologists.

The formation of the clays is due to the decomposition of the feldspars obtained after disintegration of rocks. In a clayey soil, clay particles form the solid phase of the soil. The clay particles are sheet-shaped and they have a great capability to retain water in their surface (adsorption). Clays also have a significant plasticity which means that they can have their shape when a force is applied to them.

According to **Guggenheim and Martin (1995)**, the term "clay" refers to a naturally occurring material composed primarily of fine-grained minerals, which is generally plastic at appropriate water contents and will harden with dried or fired. Although clay usually contains phyllosilicates, it may contain other materials that impart plasticity and harden when dried or fired. The term "clay mineral" refers to phyllosilicate minerals and to minerals which impart plasticity to clay and which harden upon drying or firing.

Clays are mainly formed of phyllosilicates minerals in octahedral and tetrahedral chains (Silicon ion is located in the centre of these regular structures). Clay mineral structures are usually linked by weak covalent bonds. This allows for the access of molecules of water between those chains which promotes significant volume increase (which vanishes when the water evaporates). This phenomenon of swelling and shrinkage has been defined as a volumetric instability and it characterises the behaviour of expansive soils. The capacity of clay to adsorb water and increase in volume is considerably high promoting very relevant damages to the infrastructures when the expansive clays are involved in their foundations.

All the clay minerals have tetrahedral layers of Silicon and octahedral layers of Aluminium, Iron or Magnesium (similar to gibbsite or brucite). Clay minerals are usually divided into 1:1 and 2:1 according to the relationship existing between the tetrahedral and octahedral layers, respectively. Kaolinite is an example of clay 1:1 clay mineral, whilst montmorillonite is a 2:1 (two sheets of SiO_4 tetrahedral per octahedral layer).

There are four main groups of clay minerals, according to their mineral structure:

- **Kaolinite group**: includes kaolinite, dickite, nacrite and halloysite. This group is formed by the decomposition of orthoclase feldspar (e.g. in granite).

- **Vermicullite**: It is formed by weathering or hydrothermal alteration of biotite or phlogopite.

- **Illite group**: formed by decomposition of some micas and feldspars it is predominant in marine clays and shales. This group of clays is not very expansive. It includes: hydrous micas, phengite, brammalite, celadonite and glauconite.

- **Smectite group**: this is the group of the most expansive clays. It includes montmorillonite, bentonite, beidellite, nontronite, hectorite, saponite and sauconite.

Kaolinite clays have long been used in the ceramic industry, especially in fin porcelains, because they can be easily molded, have a fine texture, and are white when fired. These clays are also used as a filler in making paper. The composition of kaolinite varies very little. This clay mineral does not absorb water and hence does not expand and shrink depending on the available moisture. Kaolinite is the preferred type of clay for the ceramic industry.

Vermiculite is a high-charge 2:1 phyllosilicate clay mineral. Its formation is generally attributed to the weathering of micas. Vermiculite is also expansive: swelling potential up to 20 times its original volume when subjected to heat (> 1000°C).

Illite is the most common clay mineral, often composing more than 50 percent of the clay mineral suite in the deep sea. Illite is usually formed by the weathering in temperate climates or in high altitudes in the tropics. Illite usually arrives in the ocean due to the transport in the rivers and wind. Formed by alteration of potassium and aluminium rich rocks, illite clays are the main components of shales. In illites, the cations of calcium and magnesium can be replaced by potassium due to the charge imbalance. The potassium, calcium and magnesium interlayer cations prevent the adsorption of water into the structure of this clay mineral. Hence the moderate expanding potential of illites.

Smectite is a family of 2:1 expansive phyllosilicate clays with permanent layer charge because of the isomorphous substitution in the octahedral sheet. The most common smectite is Montmorillonite, which is a main constituent of bentonite. Montmorillonite can expand by several times its original volume when water is available. The soils with a high proportion of smectites can be subjected to large volume changes (up to 30%) due to wetting and drying.

Smectite is the most hazardous clay mineral in civil engineering. In addition to the changes in its physical properties, it can also act as a lubricant gel. Its role in landslides is also well-known and it can have an important effect even encountered in very thin layers.

The activity of clays was first defined by Sir. Alex Skempton in 1953 (**Skempton, 1953**). Skempton defined the term as follows: Activity (Ac) = Plasticity Index / % particles finer than 2μm. Skempton suggested three classes of clays according to their activity:

- inactive: clays with activity smaller than 0.75
- normal: clays with activities between 0.75 and 1.25
- active: clays with activities greater than 1.25

The activity of clays, as defined by Skempton, can provide a valuable insight of the potential for expansion. The typical values of activities for the three principal clay minerals is shown below:

- Kaolinite: 11 < PI (%) < 21 –> Activity = 0.33 to 0.46
- Illite: 55 < PI (%) < 67 –> Activity = 0.9
- Montmorillonite: 429 < PI (%) < 656 –> Activity = 7.2

According to **Dumbleton and West (1966)**, the activity of the soil can also be defined as a function of the charge and exchange capacity of the clay minerals, and it is determined by the relative proportion of the different minerals in the clay. The activity would decrease in the following direction: montmorillonite > illite > kaolinite.

2.1.2 MARLS

Marls are carbonate-rich sedimentary rocks usually formed of clay minerals and carbonates in varying proportions between 35% to 65% **(Bellair and Pomerol, 1980)**. The predominant carbonate mineral in marls is calcite (calcium carbonate, $CaCO_3$). The geotechnical properties of marls depend on the carbonate content but also on the type and content of clay minerals **(El Amrani Paaza et al., 1998)**.

According to their consistency, marls have been usually classified following the same criteria used to classify clays and silts (fine-grained soils), without taking into account the presence of carbonates. **Lamas et al. (2002)** proposed a characterisation of the geotechnical properties of marls as a function of their carbonate content with the objective of using marls for civil engineering purposes. The results of the investigation carried out by **Lamas et al. (2002)** on natural marls obtained in Southeastern Spain showed the existence of a strong relationship between expansion, plasticity and activity and the carbonate and clay content **(Figures 2.1)**.

Datta et al. (1982) and **Khamehchiyan et al. (1994)** found low variability of the expansivity among marls with different carbonate contents. However, according to **Lamas et al. (2002)**, the carbonate content is a key feature to explain the behaviour of the marl, especially when the carbonate content is very high.

Khamehchiyan et al. (1994) reported that the effective cohesion if the marls diminished, at constant density and moisture, when the carbonate content increased. For concentrations higher than 60%, the decline in effective cohesion starts being regulated by other variables, especially the clay content **(Conrad, 1993)**.

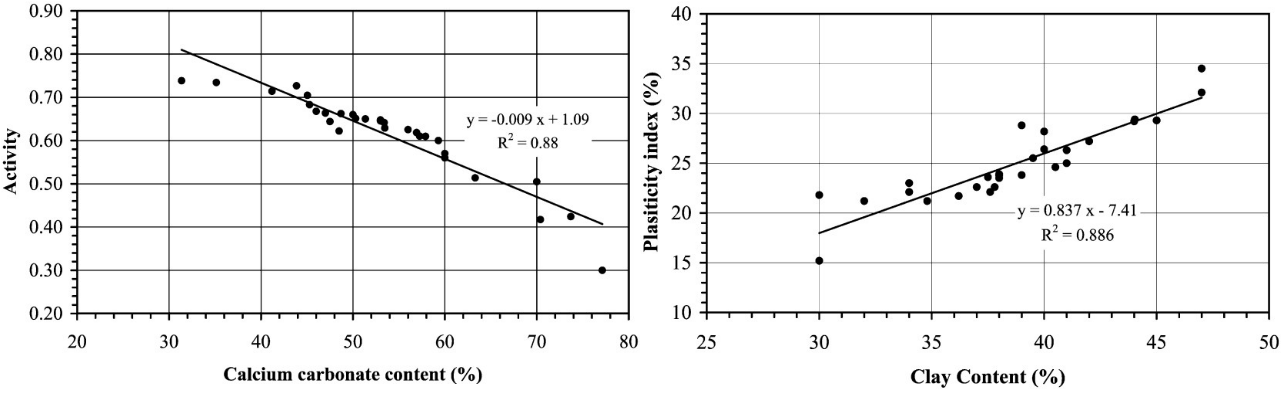


Figure 2.1. (a) Relationship between the activity and the carbonate content; (b) Relationship between the plasticity index and the clay content (Lamas et al., 2002)

2.1.3 OUTCROPS LOCATION

The expansive soils can be extensively encountered in many regions along the world. They are very common in the Southwestern states of USA (California, Nevada, Arizona, Colorado and others) and in tropical places (India, Southeastern Asia). They are also common in the Mediterranean countries, and specially, in the South of Spain.

Previous studies highlighted that consecutive wetting-drying cycles can affect the swelling-shrinkage behaviour of clay and clayey soils **(Day, 1994; Al-Homoud et al., 1995; Basma, 1996)** whilst **Guney et al. (2007)** described a reduction of the positive effect of lime in clays subjected to this type of curing. Due to the capability of interaction between the expansive clays and the water, the study of external factors such as lithology, climate, geomorphology, hydrogeology, tectonic activity or vegetation becomes important.

In Southeastern Spain, as in many other regions with a continental weather, the alternation of wet winters and dry summers promotes seasonal variations of moisture and temperature which affect the performance of the earthworks.

The study presented in this dissertation takes into consideration the existing industrial by-products in different locations of Southern Spain. The soils used can be defined as marls and clays and they all were obtained in the provinces of Granada, Jaen and Almeria (Southeastern Spain). Figure 2.2 shows the geographical location of this study.

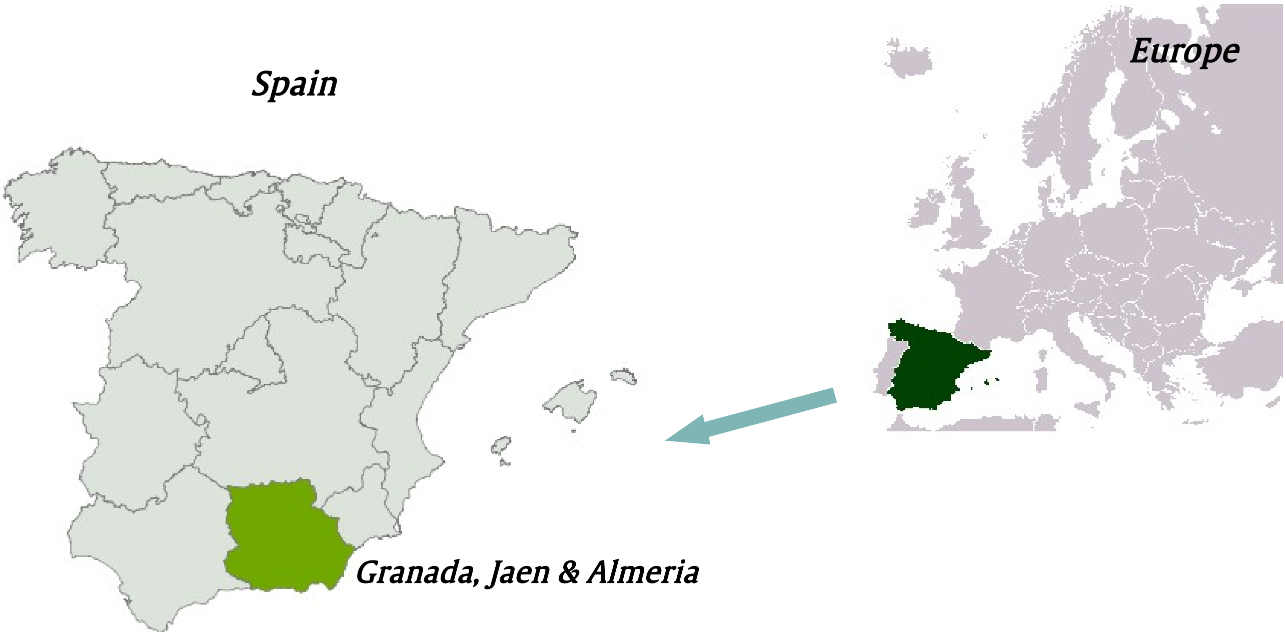


Figure 2.2. Geographical Location of the study

2.2 SOIL STABILISATION: PRACTICE

Petry and Little (2002) developed one of the most comprehensive reviews on the practical use of soil stabilisation treatments, focusing on additives and methods. According to this review, the behaviour of expansive clays has been thoroughly studied in the past. However, this has not promoted the establishment of one unique method to measure the swelling potential of expansive clays.

The types of foundation systems used in and on expansive soils have not changed significantly since the 1980s. The design of foundations is still determined by the initial costs giving less consideration to the long term performance. This is the reason why the stabilisation of expansive soils has not been given enough consideration.

According to **IECA (2008)**, the soil stabilisation technique started to be frequently applied in Spain in the decade of 1990 and not previously, due to the economic and technical difficulties experienced before.

The stabilisation of expansive soils (treatments carried out to reduce their swelling potential) can be classified in the following groups **(Rico et al., 1998)**:

- Mechanical stabilisation: compaction to increase the mechanical strength of the soil.
- Drainage Stabilisation: the reduction of the water table involves an immediate reduction of the expansive risk.
- Electrical stabilisation: electro-osmosis, electromechanical piles...
- Heat stabilisation: stabilisation by means of heat and calcination
- Chemical stabilisation: addition of stabilisation agents to promote chemical reactions.

There are more classifications in the existing literature. **Pintura i Llurba (2002)** proposed that there are three types of soil stabilisation: mechanical (compaction); physical (mixture of soil with different particle size distribution to obtain a more granular material); chemical (physico-chemical reaction between grains and additives).

The chemical stabilisation is the most effective and commonly used method for reduction of swelling potential of expansive soils. This dissertation is about the traditional and innovative methods for soil (chemical) stabilisation.

Most of the mitigation measures included in the chemical stabilisation of soils are related to the instability of smectite clays at low and high pHs. The acid treatment is not really desirable from a technical and environmental point of view. Hence different materials have been used to increase the pH of the soil (alkalinity) and reduce the swelling potential of expansive clays. The most commonly used soil stabilisation agents are: lime, cement and coal fly ash. The addition of cement, for example, modifies the pH and enable the formation of cementitious gels which increase the mechanical strength of the expansive soil.

The objectives of the soil stabilisation are **(Rico et al., 1998)**:

- Reduction of the potential for volume changes in the expansive soil
- Increase of the mechanical strength
- Reduction of the compressibility of the soil
- Enhancement of the durability of the earthworks

Usually the stabilisation treatment promotes an improvement in more than one property of the soil (strength, compressibility, expansivity...). However, sometimes the addition of a stabilisation agent to improve of one of those properties is accompanied by a secondary negative effect, which could be less important. An optimal solution must always be pursued. The properties of soils which are more susceptible to be improved by the stabilisation treatments are volume change and strength **(Rico et al., 1998)**.

The main objective of the soil stabilisation treatment could also be described as follows **(Petry and Little, 2002)**: (1) stabilisation of the volume changes of the expansive soil; (2) modification of the plasticity of the clayey soil to increase its workability; (3) increase of strength and bearing capacity.

The saturation of the soil in its natural state must also be taken into account to decide the soil stabilisation treatment. The saturated fine-grained soils are suitable for lime-stabilisation, however they are not suitable for treatment with asphaltic solutions or emulsions. Furthermore, the strength obtained by treating fine-grained saturated soils with cement would be lower than that obtained by using cement-stabilisation in fine-grained dry soils. In non-saturated fine-grained soils, the use of lime and cement is equally encouraged, however in these soils the homogeneous addition of the water in during the development of the treatment is as important as the election of the additive **(Rico et al., 1998)**.

Engineers, architects and contractors proved many different ways to reduce the damaging effects of expansive clays in the past. Their actions were usually based on a trial-error approach to the problem. Mechanical stabilisation was primarily used, but an alteration of the physico-chemical properties of the clayey soils was found to be necessary to permanently reduce their expansive behaviour. Geotechnical engineers learnt from soil mechanics and geology to find the best way to treat the clay and use it for civil engineering purposes. **Barshad (1950)**, a mineralogist, discussed the effects of the different type of cations located in the interlayers of the mineral structure of clays. Soil scientists **Alison, Kefaumer and Roller (1953)** studied the ammonium fixations in clayey soils. **Huang (1954)** provided one of the earliest descriptions of soil stablisation.

In the mid-1950s, engineers started to describe their success in the modification of the behaviour of clays. **Dubose (1955)** described the compaction as a method to control heavy clays. **Jones (1958)** studied the stabilisation of expansive clay using hydrated lime and Portland cement. **Taylor (1959)** explained the cation exchange process in clays. **McDowell (1959)** described the stabilisation of soils in Texas using a combination of lime and fly ash. Most of the ideas that are still currently in use regarding soil stabilisation and behaviour of clays are based on studies published around 1960 (**Petry and Little, 2002**).

Hit and Davidson (1960) studied the fixation of lime in clays as a start point for a decade of technological advances in lime and cement-stabilisation of soils. **Mitchell and Hooper (1961)** described the effects of the curing time in the lime-stabilised soils and noted that curing time would affect the workability and compactability of the soils. **Eades and Grim (1963)** developed their quick test to determine the required dosage of lime, based on the following concept: when a compound based on lime and caustic soda is added to the clayey soil, a reaction takes place in which the soil-silicon and soil-aluminium solubility is activated and this takes place at a high pH. Hence, the pH measurement rapidly became a quick test to identify the optimum dosage of lime. This test takes one hour due to the fact that the initial lime-soil reactions were estimated to take place in one hour. For further study of the earliest works on soil stabilisation, **Petry and Little (2002)** provided a detailed list of scientists and contributions.

The quality of the constructive method is crucial for the success of the earthworks constructed with layers of stabilised soil. The application of sheets to avoid loss of moisture have been very rarely used in the past. Two factors have a great influence on the quality of the chemical stabilisation process: the humidity and the degree of pulverisation. First, the layers of sub-base are rarely pre-saturated. They are usually worked in a dry state resulting in incomplete stabilisation reactions due to the lack of water. Secondly, the degree of pulverisation is often lower than recommended. Lime must react immediately with the soil particles to start the pozzolanic process. The greater the soil particles, the longest the duration of the stabilisation process (**Petry and Little, 2002**).

2.2.1 LIME

Petry and Little (2002) divided the existing stabilisation agents in three groups: traditional (lime, cement and fly ash); by-products: derived from lime and cement; non-conventional: sulphonated oils, potassium compounds, ammonium chloride, enzymes, polymers... Traditional stabilisation agents are characterised by the calcium exchange and pozzolanic reactions to produce the stabilisation. This mechanisms is also promoted by the so-called lime and cement by-products. The non-conventional stabilisation agents are characterised by the use of alternative chemical principals for soil stabilisation. For instance, the sulphonated oils produce a penetration of hydrogen ions in the mineral structure of clay altering its capability to adsorb water (**Petry and Little, 2002**).

The formation of interlaminar layers of aluminium hydroxides in acid environment could also promote the stabilisation process. Potassium ions can become included in the mineral structure of the smectite promoting its illitisation (reduction of its potential for volume change). This illitisation process could also be promoted by ammonium compounds.

Lime stabilisation has been widely studied and it is still an active research topic (**Al-Amoudi et al., 2010; Jauberthie et al., 2010; Cuisinier et al., 2011; Al-Mukhtar et al., 2012; Eisazadeh et al., 2012; Saride et al., 2013; Lemaire et al., 2013**). In the case of marly soils, lime treatment poses some problems which may reduce its efficiency (**Ouhadi and Yong, 2003; Eberhardt et al., 2005; Raymond and Ouhadi, 2006; Seco et al., 2011**). When working with marls, bearing in mind the presence of carbonates (among mineral components of soil) and the presence of bicarbonates (within the nearby water) becomes indispensable (**Skempton, 1969**). The chemical equilibrium of carbonates has an impact on the pH of the soil. A decrease in this pH value is critical to the effectiveness of the stabilisation reactions. Furthermore, a reaction between the carbonate ions and the lime added to the soil might reduce the actual amount of calcium oxide available for the stabilisation of the clay minerals.

Lime is the most widely used chemical stabilisation agent for clay soil subgrades. Lime-soil reactions are complex (**Diamond and Kinter, 1965; Little, 1995; Yusuf, 2001**); however, understanding of the chemistry involved and the results of field experience are sufficient to provide design guidelines for successful lime treatment of a range of soils. The sustained and slow pozzolanic reaction between lime and soil silica and soil alumina (released in the alkaline environment) is key to effective and durable stabilisation in lime-soil mixtures. Mixture design procedures that secure this reaction must be adopted.

According to **Rico et al. (1998)**, lime and cement are not equally suitable for all type of soils, but the design engineers have selected one additive depending on the specific index properties of the soil to be used. The lime is especially suitable for clayey soils whilst cement is more suitable for less plastic soils in which an increase in the strength is the final objective.

Design of lime-stabilised mixtures is usually based on laboratory analysis of desired engineering properties. Several approaches to mix design currently exist. In addition to engineering design criteria, users must consider whether the laboratory procedures adequately simulate field conditions and long-term performance.

In terms of roadway subgrades, aspects of these procedures are likely to be superseded as AASHTO shifts to a mechanistic-empirical approach. Laboratory testing procedures include determining optimum lime requirements and moisture content, preparing samples, and curing the samples under simulated field conditions. Curing is important for chemically stabilised soils and aggregates because lime-soil reactions are time and temperature dependent and continue for long periods of time. Pozzolanic reactions are slower than cement hydration reactions and can result in construction and performance benefits, such as extended mixing times in heavy clays and autogenous healing of moderately damaged layers, even after years of service. On the other hand, longer reactions may mean that traffic delays are associated with using the pavement. In addition, protocols for lime-soil mixture design must address the impact of moisture on performance (**Petry and Little, 2002**).

Lime to be used for soil stabilisation can be in the form of quicklime or slaked lime. **Males** states in **Little et al. (2000)** that lime-stabilisation construction is relatively straightforward. In-place mixing is usually employed to add the proper amount of lime to a soil, mixed to an appropriate depth. Pulverisation and mixing are used to combine the lime and soil thoroughly. For heavy clays, preliminary mixing may be followed by 24 to 48 h of moist curing prior to final mixing. This ability to mellow the soil for extended periods and then remix it is unique to lime. During this process, a more intimate mixing of the lime and the heavy clay occurs, resulting in more complete stabilisation. For maximum development of strength and durability, proper compaction is necessary; proper curing is also important.

Lime has very low effect in organic soils or in soils with low content of clays. In terms of strength, lime is especially effective in clayey gravels where the strength achieved with lime is even greater than that achieved with addition of cement. However, the most frequent use of lime treatment is on plastic clays which become more workable and compactable the addition of lime. Sometimes, the addition of limes is used as a pre-treatment before the addition of cement (**Rico et al., 1998**). On the other hand, a soil treated with 10% lime is more workable than a soil with 10% of cement.

The effect of lime on montmorillonite clays is quick: increase of strength, reduction of plasticity. In all the expansive clays, addition of lime has a great influence on the swelling potential. In the short term, the addition of lime also promotes a reduction of the moisture of the soil, that is why lime has been used as a drying agent for soils during the construction works (**IECA, 2008**).

Camacho et al. (2006), studied the effect of high dosages of lime on a plastic clayey soil. The addition of 10% of lime to the soil produced a reduction of 75% in the liquid limit and plastic limit of the soil from the very early stages of curing. Higher dosages of lime could not achieve significantly better results. In terms of comparison with fly ash and sulphonated oils, experimental results showed that lime is the most effective agent to treat expansive clays (bentonite) with low dosages of additive **(Camacho et al., 2006)**.

Guney et al. (2007) reported the effect of low dosages of lime (3-6%) on expansive soils subjected to wetting-drying cycles. As a result of the wetting-drying cycles, the swelling potential of the soil diminished from the very early stages. However, according to this study, the positive effects of the lime treatment on expansive and plastic clayey soils can be negatively affected by the ongoing of the wetting-drying cycles (which acquires a great relevance in regions with variable environmental and weather conditions).

According to **Rico et al. (1998)**, the gain of strength in clays due to the addition of lime takes a maximum for dosages of 8-10%. Higher dosages used in experimental tests did not promote further strength improvements. This statement is bolstered by **Rafalko et al. (2007). IECA (2008)** also recommended that the optimum dosage use of lime depends on the maximum amount of lime which can be consumed by the clays.

The performance of lime-stabilised subbases and bases has been somewhat difficult to assess in the current AASHTO design protocol because the measure of structural contribution—the structural layer coefficient—cannot be ascertained directly. Indirectly determined coefficients for lime-stabilised systems, however, have been found to be structurally significant. As AASHTO shifts to a mechanistic-empirical approach, measurable properties, such as resilient moduli, will be used to assess stress and strain distributions in pavement systems, including stabilised bases and subbases. These properties will be coupled with shear-strength properties in assessing resistance to accumulated deformation **(Petry and Little, 2002)**.

The lime industry **(Little, 1999a; Little, 1999b; Little, 2000)** has submitted a three to four-step design and testing protocol to be considered for inclusion in the AASHTO design protocol:

- Step 1: Determine optimum lime content using the Eades and Grim pH test (ASTM D-6274);

- Step 2: Simulate field conditions. Use AASHTO T-180 compaction and 7-day curing at 40°C to represent good-quality construction techniques. After curing, subject samples to 24 to 48 h of moisture conditioning via capillary rise (soak);

- Step 3: Verify compressive strength, stiffness, and moisture sensitivity, and measure unconfined compressive strengths using ASTM D-5102 methods. For most applications, the above three steps are sufficient because design parameters such as flexural strength, deformation potential, and stiffness (resilient modulus) can be approximated from unconfined compressive strength. For more detailed designs, a direct measure of resilient modulus may be required; and

- Step 4: Perform resilient modulus testing using AASHTO T-294-94 or expedited alternatives, such as the rapid triaxial test. This protocol and its mechanistic-empirical basis provide a sound foundation for future lime-stabilisation applications.

Mixture design was recently validated by **Yusuf et al. (2001)**. In this study, the performance of stabilised clay subgrade soils was evaluated via in situ testing: falling weight deflectometer (FWD) testing to determine in situ moduli; dynamic cone penetrometer (DCP) testing to determine in situ strengths of the stabilised and natural clay soils; and ground penetration radar (GPR) to assess in situ layer thicknesses and in situ dielectric properties of the lime-stabilised layers.

These pavements were between 15 and 20 years old at the time of testing. Samples of the native clay were collected and subjected to the four-step United States National Lime Association (NLA) mixture design protocol. Engineering properties (compressive strengths and resilient moduli) from the laboratory mixture design protocol were then compared to field results. Following this comparison, the pavement structures were modelled using layered elastic theory, GPR-determined layer thicknesses, and back-calculated resilient moduli to assess the impact of the stabilised layer (typically 200 mm thick) on the pavement performance life. The impact was deemed to be substantial as it typically extended pavement life by several hundred percent **(Petry and Little, 2002)**.

2.2.2 CEMENT

There are many different cements to be used in the construction industry. The most commonly used is Portland cement. Portland cement is the base and origin of the majority of cements. Its components are derived from the reciprocal action primary raw elements when they are subjected to the effects of a rotatory kiln at a temperature of 1450°C. In the manufacture of Portland cement, clinker is produced by sintering limestone and alumino-silicate (clay) in the cement kiln stage. A small dosage of gypsum is added in the last stage of fabrication **(Torrente and Sagues, 1974)**.

The components of cement are bicalcium and tricalcium silicate, tricalcium aluminate and tetracalcium aluminate ferrite. Those components are stable in a dry state. When water is added to cement, a progressive hydration takes place. Some of the particles get dissolved and precipitate in the form of gels or microscopic crystals and this phenomenon is accompanied by a progressive hardening **(Torrent and Sagues, 1974)**. The hardening of cement is divided in phases along the curing time. Gypsum is necessary to slow down the speed of the hardening process (otherwise cement would not be a workable material). The first stages of hardening are known as setting. In this period the cement paste acquires its consistency.

Prusinski (Little et al. 2000) stated that since 1915 more than 140,000 km of equivalent 7.5 m wide pavement bases have been constructed from cement-stabilised soils. Cement has been found effective in stabilising a wide variety of soils, including granular materials, silts, and clays.

Soil-cement bases have more stringent requirements than cement-modified soil subgrades. For soil-cement bases, two types of testing have typically been used—durability tests and strength tests. The Portland Cement Association has developed requirements for AASHTO soils A-1 to A-7 that make it possible to determine the durability of cement on the basis of maximum weight losses under wet-dry (ASTM D559) and freeze-thaw (ASTM D560) tests. Many state departments of transportation (DOTs) in USA currently require minimum unconfined compressive strength testing (ASTM D1633) in lieu of these durability tests **(Little et al., 2000; Petry and Little, 2002)**.

This requirement is often based on many years of experience with soil-cement. The advantage of using these strength tests is that they can be conducted more rapidly than the durability tests (7 days versus 1 month) and require less laboratory equipment and technician training. However, achievement of a specified strength does not always ensure durability **(Little et al. 2000)**.

Typical minimum strength varies from 1,400 to 5,250 kPa. For cement-modified soils, the engineer selects an objective and defines the cement requirements accordingly. Objectives may include one or more of the following: reducing the plasticity index (Atterberg limits, ASTM D4318); increasing the shrinkage limit; reducing the volume change of the soil (AASHTO T116); reducing clay/silt-sized particles (hydrometer analysis); meeting strength values/indexes such as the California bearing ratio (ASTM D1883) or triaxial test (ASTM D2850); and improving resilient modulus (ASTM D2434). Cement has been incorporated successfully into soils in the field with plasticity indexes ranging as high as 50 **(Little et al. 2000)**.

Construction of soil-cement and cement-modified soil is normally a fast, straightforward process. Cement can be incorporated into soil/aggregate in a number of ways. The most common method is to spread dry cement in measured amounts on a prepared soil/aggregate and blend it in with a transverse single-shaft mixer to a specified depth. Cement slurries—in which water and cement are combined in a 50/50 blend with a slurry-jet mixer or in a water truck with a recirculation pump—have been used successfully to reduce dusting and improve mixing with heavy clays (**Little et al. 2000**).

Compaction is normally a minimum of 95% of either standard or modified proctor density (ASTM D588 or ASTM D1557, respectively), with moisture content 12% of optimum. **Prusinski (Little et al. 2000)** explains that soil-cement shrinks as a result of hydration and moisture loss. Shrinkage cracks develop in the base and can reflect through narrow bituminous surfaces as thin (3 mm) cracks at a spacing of 2 to 12 m. If proper construction procedures are followed, shrinkage cracks may not reflect through, and if they do, they generally pose no performance problem.

However, cracks can compromise performance if they become wide and admit significant moisture. A number of techniques have been used to minimise this problem, including: compaction at a moisture content slightly drier than optimum; pre-cracking through inducement of weakened planes or early load applications; and delayed placement of surface hot mix, reduced cement content, and use of interlayers to absorb crack energy and prevent further propagation.

Prusinski (Little et al. 2000) explains that many state DOTs now use compressive strength testing as the sole criterion for determining cement content in soil-cement; additional research is needed to ensure that durability is also achieved at the specified strength for a variety of soil types. He believes that new durability tests may need to be developed for this purpose. A rapid and reliable test for assessing the impact of wet-dry and freeze-thaw cycles on durability remains a key need as well.

Prusinski further explains that as cracking appears to be the greatest performance concern, further research is needed to confirm the applicability of the techniques discussed above for various soil types. Particularly promising is the use of precracking techniques, which can be as simple as applying a vibratory roller 24 to 48 h after final compaction (**Petry and Little, 2002**).

Prusinski further explains (**Little et al. 2000**) that though no longer widely used, probably because of higher first costs, combination treatment with lime and portland cement of plastic clay soils offers an attractive alternative. The initial lime treatment offers the ability to reduce plasticity and improve mixing efficiency, and since the lime allows a mellowing period of up to three or four days, time is not a constraint. The addition of a relatively low amount of Portland cement (approximately 2 to 3%) upon final mixing provides the required strength even in clays that have moderate or low reactivity with lime.

Regarding the dosage of cement in the soil, **Rico et al. (1998)** reported a successful stabilisation of very plastic clays with a pre-treatment of 2-3% of cement or lime to improve the workability followed by a proper treatment of cement. The combined pre-treatment/treatment with lime/cement is the most adequate for very fine-grained soils with high plasticity **(IECA, 2008)**. The recommendations by IECA include the in situ testing of the soils before the treatment to determine the optimum dosage of additive to be used.

Al-Abdul and Asi (1997) studied the effects of Portland cement and lime on a sub-base stabilised with asphalt. They tried all the possible combinations of 0, 2 and 4% of dosages for each additive. Cement was considered to be more effective to accompany the asphalt emulsion as a main stabilisation agent. A similar study by **Hossain et al. (2007)** was carried out on the optimisation of dosages of additive for Papua New Guinea soils. As a main conclusion, the treatment with low dosage of cement achieved the same results than the treatment with high dosages of volcanic ash.

2.2.3 COAL FLY ASH

Coal fly ash are obtained from the combustion of coal in thermoelectric power plants. The composition of the fly ashes and the percentage of coal in that composition depend greatly on the industrial process of the power plant. The fly ash is a very fine-grained material mainly formed of silicates, aluminates, free lime and other oxides.

The stabilisation of soils with coal fly ash is an increasingly popular option. Fly ash stabilisation is used to modify the engineering properties of locally available materials and produce a structurally sound construction base. The coal fly ash has pozzolanic properties and its addition to an expansive soil can reduce it swelling potential **(Camacho et al., 2006)**. A dosage of 25% of fly ash in the soil can affect the particle size distribution and promote the flocculation of the clayey soil particles, reducing the liquid limit and the plastic limit of the soil. The pozzolanic reactions can increase the bearing capacity of a sub-base and hence improve the quality of the linear infrastructures. However, there are different types of coal fly ash according to their origin in the industrial process.

Class F fly ash is typically produced from anthracite and bituminous coal. This fly ash has pozzolanic properties and contains a limited amount of free lime (CaO). The active silica and alumina can react with cementing agents (such as lime or Portland cement) to produce cementitious compounds in the presence of water. The addition of chemical activators such as sodium silicate (water glass) can also lead to the formation of geopolymers from the class F ash.

Class C fly ash is produced from the burning of younger lignite or sub-bituminous coal. This class of fly ash has pozzolanic properties (like Class F fly ash) due to the presence of amorphous silica and alumina in its chemical composition. However, this fly ash is also a self-cementing fly ash due to the high content (>20%) of calcium oxide (CaO). Class C fly ash does not require an activator to produce cementation of soils. The treatment with class C fly ash can reduce the swelling potential of clays with high plasticity **(Nalbantoglu, 2004)**.

Both self-cementing and non-self-cementing coal ash can be used in stabilisation of expansive soils. However, the use of fly ash to stabilise plastic clay soils must usually be combined with the use of lime or cement. Class F fly ash is useless as a stabilisation agent without the addition of lime as a source of calcium.

Little et al. (2000) explained that combinations of lime and Class C fly ash were successfully used for stabilisation of clay soils. Successful application is normally achieved by adding lime first to modify soil properties and then adding fly ash to promote the pozzolanic reaction and increase the final strength. This approach is interesting when a specified strength level cannot be achieved with fly ash alone. Although a two-stabilisation agents, two-step process may be economically disadvantageous under, it also offers certain advantages, including the ability and time to modify the soil with lime before adding the fly ash for stabilisation. Lime allows an extended mellowing period, which is necessary with some expansive clays.

Little et al. (2000) explained that most of the CaO in Class C fly ash is complexly combined with pozzolans, and only a small percentage is "free" lime. This characteristic may impact the suitability of the material for stabilisation of plastic clayey soils. According to ASTM D-5239 **(ASTM, 2012)**, "Standard Practice for Characterizing Fly Ash for Use in Soil Stabilization," the use of self-cementing fly ash can result in improved soil properties, including increased stiffness, strength, and freeze-thaw durability; reduced permeability, plasticity, and swelling; and increased control of soil compressibility and moisture.

Although these ashes have properties similar to those of Portland cement, they also have unique characteristics that must be addressed by both the mix design and construction procedures. The primary design consideration is the rate at which the fly ash hydrates upon exposure to water. Recognising and properly addressing the hydration characteristics of the ash can result in a significant enhancement of the potential benefits of its use.

Even self-cementing ashes can be enhanced with activators such as Portland cement or hydrated lime. This is particularly true if the self-cementing ash does not have enough free lime to fully develop the pozzolanic reaction potential. In stabilisation of clayey soils, the activator (lime or cement) may play a dual role: modifying the clay and activating the fly ash. A key role for fly ash in lime-clay stabilisation is to provide the required pozzolanic-based strength gain for 'non-lime-reactive clays.

To achieve optimum results, a thorough understanding of the influence of the compaction delay time and moisture content of the stabilised materials is essential **Little et al. (2000)**. Ash hydration begins immediately upon exposure to water. Strict control of the time between incorporation of the fly ash and final compaction of the stabilised section is required. A maximum delay time of 2 hours can be employed if contractors are not experienced in ash stabilisation or if achieving maximum potential strength is not a primary consideration for the application. This limits the clay mellowing period; thus a two-step process is often required in lime-fly ash clay stabilisation. The first step is lime modification followed by fly ash addition. A maximum compaction delay of 1 hour is commonly specified for stabilisation of pavement base or sub-base sections when maximum potential strength is required. Achieving final compaction within the prescribed time frame generally requires working in small, discrete areas, an approach that differs from the methods used for lime stabilisation.

The second major design consideration according to **Little at al. (2000)** is that there is an optimum moisture content at which maximum strength will be achieved. This optimum moisture content is typically below that for maximum density—often by as much as 7 to 8%. As reported by **Petry and Little (2002)**, the strength of the stabilised material can be reduced by 50% or more if the moisture content exceeds the optimum for maximum strength by 4 to 6%. An understanding of both the influence of compaction delay and moisture control of the stabilised material is essential to achieving the optimum benefit from stabilisation applications that use self-cementing fly ashes.

Camacho et al. (2006) carried out a comparative study about the effects of fly ash, lime and sulphonated oil on an expansive soil. The conclusions of the study are that fly ash acted as an inhibitor of the expansive properties of the soil. However, unlike the lime, the fly ash treatment required large amounts of additive, at least for a very expansive clay (bentonite) which poses a constraint to the use of this stabilisation agent.

Nalbantoglu (2004) showed that the addition of 10% to 25% of coal fly ash on a clayey soil was effective in reducing the plasticity of the original soil. The soil treated with this dosage of fly ash can shift from a clayey to silty according to the Casagrande chart classification. The reduction in the swelling pressure also indicated a reduction of the swelling potential (hazardous for foundations). The treatment with coal fly ash can also be very effective in the modification of the mineralogy of the treated soils. The pozzolanic properties of the coal fly ash can promote the formation of cementitious compounds, making the soil more granular and reducing the potential to adsorb water. According to **Hossain et al. (2007)**, the use of coal fly ash is promising as it can be suitable for certain type of soils and it offers a more economic solution than the use of lime and cement.

2.2.4 OTHER ADDITIVES

Between the conventional and non-conventional additives for soil stabilisation, gypsum can be found.

Gypsum is a soft sulphate mineral composed of calcium sulphate dihydrate ($CaSO_4 \cdot 2H_2O$). It is also an industrial product obtained from the natural mineral. Sometimes, chemical substances are added to the gypsum in the industrial process to alter its properties: setting, strength, adherence, density, water adsorption, etc. The gypsum is mixed with water and used as a construction material. It is also used in the elaboration of prefabricated elements. Gypsum, as an industrial products, is a hemihydrate calcium sulphate with the following formula: $CaSO_4 \cdot ½H_2O$.

Gypsum can be added to the expansive soil to reduce the swelling potential. **Ameta et al. (2007)** studied the use of lime-gypsum combinations to treat expansive soils. They tried different dosages and studied the evolution of the index properties of the soil. Using locally available materials, **Ameta et al. (2007)** showed that not all the soils are improved by the use of lime and gypsum. Only the reactive soils answer the treatment favourably, forming chemical compounds and well developed crystalline structures. The addition of lime and gypsum to the bentonite soil promoted a significant decrease of plasticity and swell. **Ameta et al. (2007)** found the following combination to be the optimal for the soils tested in the study: 2% lime + 4% gypsum.

However, some authors claimed that the use of gypsum must be approached carefully (**Rico et al., 1998**). Gypsum has been traditionally used in mixtures of soil-cement to diminish the setting time. The direct use of gypsum as stabilisation agent for expansive soils poses advantages and disadvantages similar to those promoted by the use of sodium chloride. However, the effect of gypsum on the compaction and the permeability is much lower.

2.2.5 INTERNATIONAL STANDARDS

There are many different soil classification systems. The first classification systems which were proposed were based on the particle size distribution. However, the introduction of the Atterberg limits (consistency limits and plasticity) motivated some changes in the classification systems. The Casagrande unified soil classification system (USCS) and the American Association of State Highway and Transportation Officials (AASHTO) are currently the most commonly used classification systems in civil engineering.

It is difficult to find a characterisation and classification of expansive soils in national codes of practice, despite the fact that the expansive soils are widely distributed over almost all the geographical locations in the world. **Sridharan and Prakash (2000)** proposed a classification of expansive soils based on the free swell ratio, defined as the ratio of the sediment volume of soil in distilled water to that in carbon tetrachloride or kerosene.

The American Society for Testing Materials (ASTM) prepared a standard to evaluate the expansion potential of a soil: ASTM D-4829 **(ASTM, 1995)**. This standard is based on the soil expansion potential test developed in California (USA) in the mid-1960s and introduced in the previously existing UBC Test Standard 29-2. UBC stands for Uniform Building Code, an extinct building code primarily used in the western United States and first published in 1927. The UBC was replaced by the International Code Council (ICC) in 2000.

According to ASTM, the expansion index has been determined to have a greater range and better sensitivity of expansion potential than other indices, such as Atterberg limits. This index (EI) is based on the soil fraction (F) passing No.4 sieve (4.75mm) and percent swell (Δh) obtained in an oedometer (EI = 100 x Δh x F).

Table 2.4

Soil expansion index according to ASTM D-4829 (1995)

Degree of expansion	Expansion Index
Very low	<20
Low	21-50
Medium	51-90
High	91-130
Very high	>130

In terms of design and methods, some of the most important standards applied in the US for the stabilisation of soils are:

- AASHTO (2008): Recommended practice for stabilization of subgrade soils and base materials.

- ASTM D7762 (2011): Standard Practice for the Design of stabilization of soil and soil-like materials with self-cementing fly ash

- ASTM D558-560: Test methods for soil-cement mixtures.

- NLA (2006): Mixture design and testing procedures for lime stabilized soil. Technical brief.

The procedures outlined by the US National Lime Association (NLA) in order to optimise the dosage of lime required for long-term strength, durability and the other desired properties of the stabilised soil includes (**Celauro et al., 2012**):

evaluation of the soil suitability for lime stabilisation (gradation, plasticity, presence of organic or harmful chemical substances);

determination of the minimum amount of lime required, via the Eades and Grim test;

evaluation of the lime-soil performance for long term durability, paying attention to cyclic freezing and thawing and periods of extended soaking. For doing this OMC and MMD of the lime-treated Soil has to be determined, using ASTM D698 procedures (standard compaction effort). Then, UCS specimens are fabricated at OMC (± 1%) with the minimum lime content. After 7 days of curing in air at 40°C, in protected condition, specimens are subjected to a 24 hour capillary soaking soak prior to testing for UCS;

only for expansive soils, change in expansion characteristics is determined after capillary soaking, via G_v measurements.

In US, among the others, the National Lime Association recommends to make use of the UCS mechanical requirements evaluated on soil-lime specimens after 7 days of soaking in water (**Celauro et al., 2012**).

The European standard BS EN 459-1 (specifications on the chemical and physical properties of quicklime and hydrated lime for construction) was revised in 2010 to extend the scope and cover the use of lime in civil engineering and particularly in soil stabilisation.

Soil stabilisation using lime is widely established in the UK (**Sherwood, 1993; Chaddock and Atkinson, 1997; Aggregate Advisory Service, 1999**). In 2009, the revised standard BS 6031 (BSI, 2009) was released in the UK to provide a code of practice for earthworks. It included the use of lime for soil stabilisation.

In the Spanish standard for the design and construction of subbases, PG-3 (**Ministry of Development, 1976, revised form 2004**), the soil stabilisation with lime and cement is considered. There is also a definition of the construction material called soil-cement, classified in EST-1, EST-2 and EST-3 according to the minimum dosage of cement added to the soil: 1%, 2% and 3%, respectively. In the South of Spain, the Agency of Public Works (local Government) released the standard ICAFIR for the design of earthworks and pavements which also defined some treatments of soil stabilisation with lime and cement. The Spanish agency for the Administration of Railway Infrastructure (ADIF) was responsible for the construction of many high-speed railways between 2000 and 2010. Hence, ADIF released several prescriptions on the use of lime-stabilised soils for high-speed railways which were extensively used in the construction industry.

In France and Belgium, a method can be found for the design of soil stabilisation with lime and cement. The method adopted by these standards makes a distinction between mixtures to be used for embankments and mixtures to be used as subgrade. When designing soil-lime mixtures to be used in embankments, lime treatment of clays is aimed at allowing the maximum reuse of the existing expansive soil. Therefore the objective of the treatment is the improvement of the compactability and workability of the soil mixture. The parameter used to characterise the mixtures from a mechanical point of view is the Immediate Bearing Index (IBI). In the case of the lime or lime-cement for soils to be used in subgrade layers, the interest of the treatment is not only in reusing the fine-grained soils, but also in improving their strength and bearing capacity to ensure the correct performance of the overlying pavement. Again, the parameters used to characterise the quality of the treatment is the IBI (**Celauro et al., 2012**).

In Italy, two methodologies are proposed by the National Agency for Railways (RFI) and the National Agency for Roads (ANAS). The RFI applies an internal technical specification based on a complex design methodology. The procedure is really money and time consuming, since at least 6 weeks are necessary for the third phase alone, together with the availability of laboratory and field equipment. This kind of method may be justified only for major works, such as the railways, where large volume of homogeneous soil have to be treated. In the case of minor construction sites, this method is not applicable. (**Celauro et al., 2012**). On the other hand, ANAS makes use of an internal specification for soil-lime mixtures, based on a laboratory study that includes Proctor compaction studies, CBR bearing capacity and UCS tests. Nevertheless, this specification prescribes the modified effort for the compaction test (which corresponds to an energy level much higher – 2.69 MJ/m^3 – of that gained with the standard effort – 0.6 MJ/m^3). The CBR is determined on mixtures cured for 7 days in air, with protected conditions, and 4 final days of soaking in water at 20°C (7+4s). The linear swelling (LS) too is measured on specimens cured with such curing procedure (**Celauro et al., 2012**).

2.3 SOIL STABILISATION: THE PROCESS

Many authors have recently studied the properties of industrial by-products (**Attom, 2007; Rahmat and Ismail, 2011; Shao et al., 2013**), natural resources (**Basha et al., 2005; Mathur, 2006; Mohamed, 2013**) and chemical compounds (**Xeidakis, 1996a; Xeidakis, 1996b**) as an alternative to the use of lime, cement and coal fly ash for soil stabilisation. However, a better understanding of the global effect of those traditional binders on soils is still necessary, especially for those soils whose mineral composition could hinder the development of stabilisation reactions.

In this section, the process of soil stabilisation and the reactions promoted in the soil by the additive will be approached with a focus on the importance of the calcium-based stabilisation agents and the definition of the pozzolanic reactions.

In soil stabilisation, the aim of the additives, as described by **Solanki and Zaman (2012)**, is to promote a first phase of agglomeration of particles and a second phase of pozzolanic reaction. **Seco et al. (2012)** defined the pozzolanic reaction as an interaction between the additive and the silica and alumina present in the clay to form cementitious compounds in an alkaline environment. The formation of cementitious gels is more likely to take place when calcium-rich stabilisers are used, but the increase in the pH of the soil is always a desirable effect, since it also promotes the modification of smectite, the most expansive species of clay mineral (**Drief et al., 2002**).

The reactions between the additives and the soil can be divided in two different groups: a flocculation-agglomeration of the particles and a pozzolanic reaction in which the clay particles react with the binder to form cementitious gels (**Solanki and Zaman, 2012**). The process which rules these reactions is still a common object of study as it is the discussion of which materials can be considered to be pozzolans (**Seco et al., 2012; Pourkhorshidi, 2010; Wong, 2013**).

In the existing scientific literature, many studies can be found which focus on the reactions occurred in the soil during the stabilisation treatment. First, a distinction must be made according to the additive used. The soil stabilisation with lime is considerably different from the cement treatment (**Rico et al., 1998**). There are two chemical reactions between the lime and the clayey soil: (1) an immediate reaction takes place in which the soil particles capture calcium ions which promotes a flocculation-agglomeration of the solid particles; (2) the second reaction takes place in long periods of time and it is the proper cementitious reaction: pozzolanic reaction due to the interaction between calcium hydroxide (from the hydrated lime) and silica and alumina (present in the soil structure). The result of the pozzolanic reaction is the formation of calcium silicates and aluminates which increase the strength of the soil (and reduce dramatically the swelling potential).

2.3.1 MICROSTRUCTURE AND REACTIONS

Dafalla and Mutaz (2012) defined the reactions promoted in the soil by the addition of lime and cement:

Lime: lime is generally added to soils to generate long-term strength gain through a series of pozzolanic reactions in the presence of water. The mixture is generally compacted to a desired water content and dry density. The different types of lime available are: quick lime (CaO), hydrated lime ($Ca(OH)_2$) and lime slurry which is a suspension of hydrated lime dissolved in water. Quicklime is manufactured by chemically transforming calcium carbonate (limestone-$CaCO_3$) into calcium oxide. Hydrated lime is created when quicklime reacts with water. It is the hydrated lime that reacts with clay particles and permanently transforms them into a strong cementitious matrix **(National Lime Association, 2004)**. According to the National Lime Association Publication **(Lime-treated Soil Construction Manual, 2004)** when lime and water are added to a clayey soil, chemical reactions occur immediately. When the quick lime is added to a soil it will start drying and the moisture holding capacity is then reduced to a great extent. Calcium ions start migrating from the lime to the surface of the clay particles and displace water and weakly bonded anions. The soil becomes friable and starts to granulate and this makes it easily compacted. When lime is added to the soil, the concentration of OH^- ions increases and elevates the pH level. This will result in the release of alumina and silica into the clay fraction **(Ouhadi and Yong, 2003)**. The released alumina and silica phases react with calcium ions producing CSH ($3CaO-2SiO_2-3H_2O$) and CAH ($3CaO-Al_2O_3-Ca(OH)_2-12H_2O$), which renders cohesive soils workable and less plastic **(Raymond and Ouhadi, 2006)**. In general, the higher the clay contents in soils the higher the plasticity values. Expansive clays have high liquid limits. The addition of lime to highly plastic clay has a significant effect in reducing plasticity index, liquid limit and linear shrinkage and increases shrinkage limit and plastic limit. The compacted soil-lime mixtures have lower maximum density values compared to untreated soil and this reduction increases with the increase in the lime content. The optimum water content also increases proportionally with the increase in the percentage of the lime **(Mallela et al., 2004)**. Lime can modify almost all fine-grained soils, but the noticeable improvement occurs in the case of clayey soils containing minerals such as; smectite, kaolinite and illite. The soil mineralogy is an important issue for the effectiveness of lime treatment. Generally, the optimum amount of lime required for soil treatment is usually between 2 and 8 % measured by dry weight of the soil **(Basma and Tuncer, 1991)**.

Cement: cement is a comprehensive chemical stabiliser often used in ground improvement. The most common cement type used to treat clayey soils is the Portland cement which when combined with water, hydrates to form the cementing compounds of calcium-silicate-hydrate and calcium-aluminates-hydrate, as well as excess calcium hydroxide. Calcium hydroxide released during the process of hydration reacts with soil alumina and soil silica fine-grained clay soils. This pozzolanic reaction is an important aspect of the stabilisation of clayey soils. Portland cement essentially contains five main components. In their pure form, these phases are tricalcium silicate (Ca_3SiO_5 or C_3S) **(Taylor, 1997)**, dicalcium silicate (Ca_2SiO_4 or C_2S), tricalcium aluminate ($Ca_3Al_2O_6$ or C_3A), a tetracalcium alumino ferrite (often given as $Ca_4Al_2Fe_2O_{10}$ or C_4AF) and a sulphate phase (often gypsum, $CaSO_4AL_2H_2O$ or CSH_2) **(Wesselsky and Jensen, 2009)**. Cement plays an important role in altering and enhancing many physical properties of the soil such as; plasticity, compaction, durability, compressibility, swell potential and shear strength.

Pozzolanic reaction: it is a chemical reaction which occurs in cement or in a mixture of hydrated lime (calcium hydroxide) with amorphous siliceous materials in the presence of water. The most important reaction of the siliceous pozzolans occurs between reactive silica, lime and water to produce calcium silicate hydrate **(Helmuth, 1987)**. A simple acid-base reaction between Calcium Hydroxide, ($Ca(OH)_2$) and silicic acid, (H_4SiO_4, or $Si(OH)_4$), can be shown as an example of pozzolanic reactions. According to **Cook (1986)**, the reaction can be schematically represented as follows:

$$Ca(OH)_2 + H_4SiO_4 \rightarrow Ca_2H_2SiO_4 + 2H_2O \rightarrow CaH_2SiO_4 \cdot 2H_2O$$

The above reaction can be simplified into:

$$CH + SH \rightarrow CSH$$

The product of ($CaH_2SiO_4 \cdot 2H_2O$) formed is a calcium silicate hydrate, also abbreviated as CSH in cement chemist notation **(Cook, 1986)**. Hydration reaction is the main hardening reaction of calcium silicate binders due to the reaction of calcium silicates, calcium aluminates and calcium-silicatealuminate phases (i.e., $2CaO.Al_2O_3.SiO_2$ and $4CaO.Al_2O_3.Fe_2O_3$) with water **(Cizer et al., 2009)**. This leads to the formation of hydrated compounds such as calcium silicate hydrates (CSH), calcium aluminate hydrates (CAH) and AFm phases (AFm phase is an alumina and monosulphate phase) **(Taylor, 1997)**. Calcium silicate hydrates possess a remarkable level of structural complexity **(Chena et al., 2004)**. More than 30 crystalline calcium silicate hydrate phases are known, these phases range from semicrystalline to nearly amorphous, all of which are described by the generic term, “C-S-H” **(Taylor, 1997)**. The C-S-H formed by the hydration of tricalcium silicate (C_3S) or ß-dicalcium silicate (ß-C_2S), which together constitute about 75 % of a portland cement by weight, is particularly a disordered variety, usually called C-S-H gel to distinguish it from CS-H phases made by “synthetic” preparation routes such as the reaction of CaO and SiO_2 or the double decomposition of a Ca salt and an alkali silicate in aqueous solution **(Chena et al., 2004)**. CSH has many crystal structures, some of which are depicted by **Richardson (2008)** as; Foshagite

$Ca_4(Si_3O_9)(OH)_2$, Nekoite $Ca_3Si_6O_{15}{\cdot}7H_2O$, Clinotobermorite $Ca_5Si_6O_{17}$. $5H_2O$, Tobermorite $Ca_5Si_6O_216(OH)_2$ and Jennite $Ca_9Si_6O_{18}(OH)_2{\cdot}8H_2O$. Calcium aluminate hydrates are formed when $Ca_3{\cdot}Al_2O_6$, a compound present in Portland cement, reacts with water **(Spierings and Stein, 1978)**. The chemical formulation of Calcium Aluminate Hydrate (CAH) is ($3CaO{\cdot}Al_2O_3$ $\cdot Ca(OH)_2{\cdot}2H_2O$). CAH has many crystal structures. **Richardson (2008)** depicted some of the crystal structures of CAH as; Bicchulite Ca_2 $(Al_2SiO_2)(OH)_2$, Katoite Hydrogarnet $Ca_{1.46}AlSi_{0.55}O_6H_{3.78}$ and Strätlingite $Ca_2Al(AlSi)_{2.22}O_2(OH)_{122.25}H_2O$.

2.3.2 FACTORS AFFECTING THE CEMENTITIOUS STABILISATION

According to **Solanki et al. (2012)**, the effectiveness of the cementitious stabilisation depends on properties of both soil and additive **(AFJMAN, 1994, Al-Rawas et al., 2002, Parsons et al., 2004, Evangelos, 2006)**. Among the soil properties affecting the performance of stabilisation treatment, **Solanki et al. (2012)** defined the following:

Gradation and plasticity index: Several researchers (**Diamond and Kinter, 1964; Haston and Wohlgemuth, 1985; Prusinski and Bhattacharja, 1999; Little, 2000; Qubain et al., 2000; Kim and Siddiki, 2004; Mallela et al., 2004; Puppala et al., 2006; Consoli et al., 2009**) recommended use of lime with fine-grained soils. However, CFA (**McManis and Arman, 1989; Chang, 1995; Misra, 1998; Zia and Fox, 2000; Puppala et al., 2003; Bin-Shafique et al., 2004; Phanikumar and Sharma, 2004; Nalbantoglu, 2004; Camargo et al., 2009; Li et al., 2009**) and CKD **(e.g., McCoy and Kriner, 1971; Baghdadi and Rahman, 1990; Zaman et al., 1992; Sayah, 1993; Miller and Azad, 2000; Miller and Zaman, 2000; Parsons and Kneebone, 2004; Sreekrishnavilasam et al., 2007; Peethamparan et al., 2008; Gomez, 2009**) is used successfully with both fine- and coarse-grained soils. Lower effectiveness of lime with coarse-grained soil can be attributed to scarcity of pozzolans (silicious and aluminacious material) in coarse-grained soils which is required for pozzolanic (or cementitious) reactions. **Little (2000)** and **Mallela et al. (2004)** recommend a soil with a minimum clay content (< 0.002 mm) of 10% and a plasticity index of 10 for lime-stabilisation.

Cation exchange capacity: Cation Exchange Capacity (CEC) is the quantity of exchangeable cations required to balance the charge deficiency on the surface of the clay particles **(Mitchell, 1993)**. During ion exchange reaction of soil with cementitious additive, cation of soil (e.g., Na^+, K^+) is replaced by cation of additive (Ca^{2+}) and the thickness of double diffused layer is reduced. Hence, the replacement of cations results in an increase in workability and strength of soil-additive mixture. The rate of exchange depends on clay type, solution concentrations and temperature **(Gomez, 2009)**. In soil stabilisation studies, CEC values have been used to a limited extent to explain the effectiveness of soil stabilisation **(Nalbantoglu and Tuncer, 2001; Al-**

Rawas et al., 2002; Nalbantoglu, 2004; Gomez, 2009).

Sulphate content: Primary "sulphate-induced heaving" problems arise when natural sulphate rich soils are stabilised with calcium-based additives (**Puppala et al., 2004**), also known as "sulphate attack." This heave is known to severely affect the performance of pavements, and other geotechnical structures built on sulphate rich soils stabilised with calcium-based additive (**Hunter, 1988; Mitchell and Dermatas, 1990; Petry and Little, 1992; Rajendran and Lytton, 1997; Rollings et al., 1999; Puppala et al., 2004**).

Specific surface area: Surface phenomena have an important influence on the behaviour of fine-grained soils; they affect many physical and chemical properties (**Cerato and Lutenegger, 2002**). SSA may be a dominant factor in controlling the fundamental behavior of many fine-grained soils (**Gomez, 2009**). The mineralogy of fine-grained soils is the dominant factor in determining the effect of SSA.

Among the additive properties affecting the cementitious stabilisation, **Solanki et al. (2012)** defined:

Free-lime content: In calcium-based stabilisers most of the lime (CaO) is bound up in compounds such as tricalcium silicate and tricalcium aluminate. The unreacted lime that is not combined in any of these compounds is called free-lime, which is expected to play a major role in stabilisation (**Collins and Emery, 1983; Misra, 1998; Zaman et al., 1998; Ferguson and Levorson, 1999; Miller and Azad, 2000; Miller and Zaman, 2000; Sezer et al., 2006; Khoury and Zaman, 2007; Peethamparan and Olek, 2008**).

Specific surface area: The specific surface area (SSA) of additives can be measured using the ethylene glycol monoethyl ether (EGME) method (**Cerato and Lutenegger, 2002**). According to the existing studies, higher SSA values indicate more reactivity of additive (**Nalbantoglu and Tuncer, 2001; Sreekrishnavilasam et al., 2007**).

Loss on ignition: Some researchers reported that high LOI indicates low free-lime content for CKDs, making CKDs less reactive, and therefore lower improvements (**Bhatty et al., 1996; Miller and Azad, 2000**).

Particle size: Several researchers noticed increased reactivity of additive with increase in amount of additive passing No. 325 (45 µm) sieve (**NCHRP, 1976; Bhatty et al., 1996; Zaman et al., 1998; Zheng and Qin, 2003; Khoury, 2005**).

pH: Several researchers used pH values on soil-lime mixture as an indicator of reactivity of lime (**Haston and Wohlgemuth, 1985; Prusinski and Bhattacharja, 1999; IRC, 2000; Little, 2000; Qubain et al., 2000; Mallela et al., 2004**).

2.3.3 DEFINITON OF POZZOLANS AND POZZOLANIC ACTIVITY

A pozzolan is defined as **(ASTM C125)** “a siliceous and aluminous material which, in itself, possesses little or no cementitious value but which will, in finely divided form in the presence of moisture, react chemically with calcium hydroxide at ordinary temperature to form compounds possessing cementitious properties”.

According to **Takemoto and Uchikawa (1980)**, the pozzolanic activity is a parameter and it can be defined as the degree of reaction over time or the reaction rate between a pozzolan and Ca2+ or Ca(OH)2 in the presence of water. The rate of the pozzolanic reaction is dependent on the intrinsic characteristics of the pozzolan such as the specific surface area, the chemical composition and the active phase content. Physical surface adsorption is not considered as being part of the pozzolanic activity, because no irreversible molecular bonds are formed in the process.

The pozzolanic activity has a high importance in the stabilisation of expansive soils. A key role for fly ash in lime-clay stabilization is to provide the required pozzolanic-based strength gain for “non-~lime! reactive” clays. Actually, the improvement of physical properties experienced in the first stages of curing need to be combined with the pozzolanic reactions. The whole process occurred might be explained as follows: a) in a first stage, the addition of lime promotes aggregation and flocculation of particles, leading to an improvement in physical properties of the soil; b) the increase in the pH (alkaline environment) promotes the dissolution of the clay minerals, releasing silica and alumina to react with the existing $Ca(OH)_2$ cementitious compounds (pozzolanic reaction); c) the pozzolanic reaction (which can promoted a reduction of expansive clay minerals) contributes to the physical improvement in the strength properties of the soil.

The reaction which rules the reduction of clay minerals is as follows: Al2Si4O10(OH)2n H2O + 2(OH)- + 10H2O = 2({2Al(OH)4- + 4H4SiO4} + nH2O. For the necessary clay mineral dissolution, pH must be higher than 10.5. This reaction is also called pozzolanic, as the Al and Si released from the clay, can react with slaked lime to form cementitious gels, similar to those formed in blended cements (cements with pozzolans).

According to **Seco et al. (2012)**, the pozzolanic reaction is as follows: Ca(OH)2 = Ca2+ + 2OH-; Ca2+ + 2OH- + SiO2 = CSH; Ca2+ + 2OH- + Al2O3 = CAH. This cementitious compounds contribute to enhance the strength of the soils and make cohesive soils more workable and less plastic **(Raymond and Ouhadi, 2006; Jaritngam, 2012)**. CAH and CSH can be identified by the presence of Ca, Si and Al. EDX analysis might become a useful tool to determine if some of the new compounds found in stabilised samples belong to the group of CSH and CAH.

2.3.4 PREVIOUS WORKS ON POZZOLANS

Pozzolans have been used in construction for many years as part of bricks, pavements, concrete and a wide range of construction materials. As explained above, the combined use of pozzolans and cement or lime is widespread due to the activating effect that these binders have on the pozzolans. The assessment of the pozzolanic activity of cement replacement materials is increasingly important because of the need for more sustainable cementitious products. The study of the pozzolanic activity of industrial by-products have been recently studied by different authors.

Petry and Little (2002) did a through review on the most commonly used additives for soil stabilisation in real construction sites in USA. Those traditional additives can be divided in two groups: binders (lime and cement) and pozzolans (coal fly ash). There are different types of coal fly ash, depending on their origin and composition. The class C coal fly ash has a high content of calcium and hence it can incorporate a high content of free lime. This coal fly ash is not equally powerful as a binder as lime or cement are. However, due to the significant content of calcium oxide, it is also included in a sub group of coal fly ash, called self-cementing coal fly ash. There are other types.

Anday (1961, 1963) provided further evidence of the importance of properly curing lime-stabilized soils to fully develop the pozzolanic reaction, which is the key to strength gain and long-term stability. **Diamond and Kinter's (1965)** classic paper clarifies the need for the pozzolanic surface reaction to achieve strength gain as well as plasticity reduction in plastic clay soils, especially those that are calcium-dominated prior to stabilization.

Pozzolanic mixtures have been successfully used to stabilize soils in Australia, Europe, and the United States. Generally, such mixtures are not especially beneficial for clay soils since clay itself is a pozzolan, but since some clay soils are simply not reactive enough with lime to produce substantial strength gain, the use of a pozzolan additive provides a means of strength enhancement. Studies reported by **McCallister and Petry (1990)** indicated that, in order to provide durability, soils must be stabilized with sufficient lime to develop pozzolanic reactions

Little et al. (1995) recommended a process by which to assign structural significance to lime-stabilized subgrade layers. This protocol can be extended to cement and lime-fly ash or cement-fly ash stabilized clay soils as well; however, modifications in the curing protocol will have to be made as the pozzolanic reactions are faster with cement and fly ash than with lime (**Petry and Little, 2002**).

Stewart (Little et al. 2000) explains that lime-Class C fly ash combinations have been successfully added to clay soils for stabilization. Successful application is normally achieved by adding lime first to modify soil properties and then adding fly ash to react with the lime to achieve a specified strength gain. This approach is attractive when a specified strength level cannot be achieved with fly ash alone. Although a two-stabilizer, two step process may be economically disadvantageous under certain circumstances, it offers certain advantages, including the ability and time to modify the soil with lime before adding the fly ash for stabilization. Lime allows an extended mellowing period, which is necessary with some expansive clays.

Even self-cementing ashes can be enhanced with activators such as portland cement or hydrated lime. This is particularly true if the self-cementing ash does not have enough free lime to fully develop the pozzolanic reaction potential. In clay soil stabilization the activator (lime or cement) may play a dual role: modifying the clay and activating the fly ash. A key role for fly ash in lime-clay stabilization is to provide the required pozzolanic-based strength gain for "non-~lime! reactive" clays.

Yusuf (2001) demonstrates that soil matrix suction and surface energy play key roles in defining the engineering properties of lime-stabilized clay soils and that these effects may rival pozzolanic effects in determining properties and performance. This means that physical processes in addition to or even in lieu of traditionally considered pozzolanic reaction and cation exchange may well be needed to explain part of the modification/stabilization process, even in the traditional stabilizers. One very important and promising area is to re-look at the effect of cations on altering the thickness of the double-diffused water layers that surround the basic clay sheets. These layers define the volume stability and strength of the soil.

Much industrial wastes can be absorbed by civil engineering and building construction, reducing at the same time both the consumption of Portland Cement and the environmental problems provoked by the dumping of wastes. **Seco et al. (2012)** explained the types of waste for the production of pozzolanic materials, including coal fly ash, ground granulate blast furnace (GGBS), silica fume, rice husk ash (RHA), phosphogypsum (PG), ceramic wastes (CW) and sewage sludge (SS).

As a main conclusion, **Seco et al. (2012)** claimed that there are industrial wastes with pozzolanic characteristics which can at least partially substitute Portland Cement without loss of resistance, and even increase other properties like durability. All of them have a potential use as a sustainable, green, and low cost binders. Thus the potential for industrial waste utilization in the field of construction has a potential so far not fully exploited.

Solanki and Zaman (2012) studied the mineralogy of the stabilised clay treated with calcium-based stabilisers, highlighting the importance of both flocculation and pozzolanic reaction on the soil stabilisation process.

The pozzolanic activity of metakaolin, silica fume, coal fly ash, incinerated sewage sludge ash and sand was studied by **Donatello et al. (2010)** using the Frattini test, the saturated lime test and the strength activity index test. A significant correlation between the strength activity index test and the Frattini test results was found, but the results from these tests did not correlate with the saturated lime test results. According to **Donatello et al. (2010)**, the mass ratio of $Ca(OH)_2$ to test pozzolan is an important parameter. In the Frattini test and strength activity index test the ratio is approximately 1:1, whereas in the saturated lime test the ratio is 0.15:1. This explains why the saturated lime test shows higher removal of $Ca(OH)_2$ and why the results from this test do not correlate with the other test methods. The results obtained by **Donatello et al. (2010)** are shown in **Figures 2.3 and 2.4**.

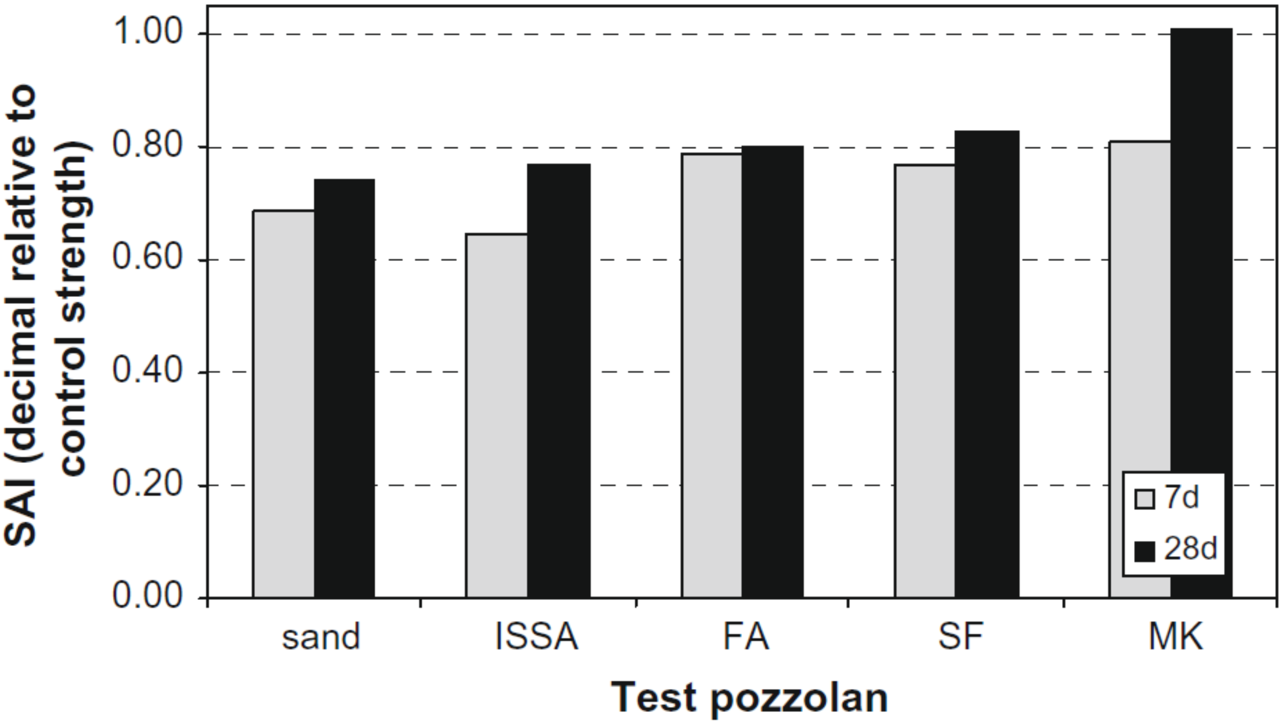


Figure 2.3. Strength activity index for different test materials after 7 and 28 days of curing (**Donatello et al., 2010**).

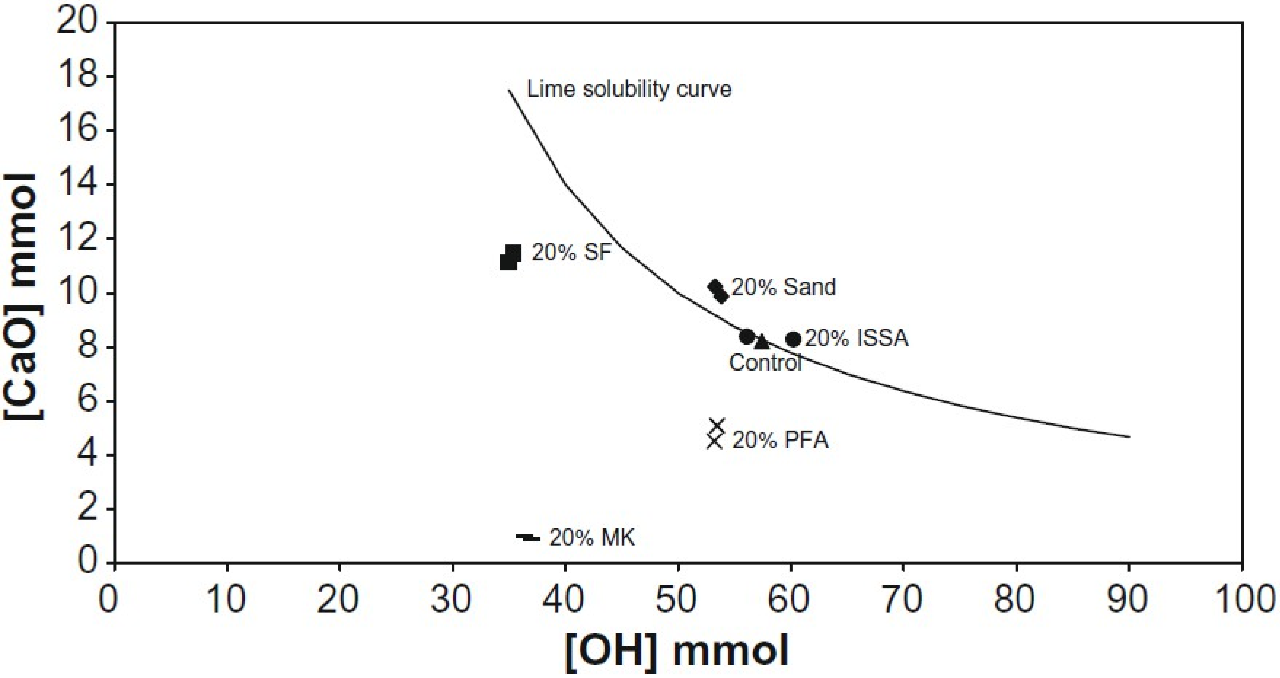


Figure 2.4. Strength activity index for different test materials after 7 and 28 days of curing (**Donatello et al., 2010**).

2.4 THE SUSTAINABILITY ISSUE

2.4.1 DEFINITION OF SUSTAINABILITY

'What is sustainability?' This is the question asked by **Hay et al. (2014)** in their recent review of the existing literature on sustainability. According to this review, sustainability is an issue of concern primarily because of the mounting evidence to suggest that human activity in the Earth system is following an unsustainable trajectory sustainability is an issue of concern primarily because of the mounting evidence to suggest that human activity in the Earth system is following an unsustainable trajectory.

According to **Hay et al. (2014)**, in spite of a considerable body of research on sustainability, recent reports suggest that we are barely any closer to a more sustainable society (**Eurostat, 2011a,b; United Nations Environment Programme, 2012**). There is an urgent need to improve the effectiveness of human efforts towards sustainability. A clearer and more unified understanding of sustainability among different people and sectors could help to facilitate this (**Hannon and Callaghan, 2011; Lindsey, 2011). Hay et al. (2014)**, pointed out that an issue that appears to be impeding progress is the lack of a clear and unified understanding of sustainability among different people and sectors (**Lindsey, 2011; Voinov, 2007**). For example, **Hannon and Callaghan (2011, p. 877)** argue that "the diffusion and popularity of the term sustainability with relatively little corresponding rigorous and grounded conceptualization may have created confusion over the basic concepts of sustainability". In turn, they suggest that the "lack of a unified and rigorous understanding of sustainability means that sustainability initiatives are often ineffectual", a point made in a business context but readily translatable to society as a whole.

2.4.2 SUSTAINABILITY IN THE CONSTRUCTION INDUSTRY

In the recent years, the concept of sustainability has gained an increasing importance within the field of building and construction. From a holistic approach, the sustainability can be understood as the feasibility of the future continuation of construction works and activities from a technical, economical and environment point of view.

According to this definition, it is obvious that the in situ stabilisation of a problematic soil contributes greatly to the sustainability of the construction activity. It can avoid the disposal of large volumes of soil in landfills, which is an environmental and economical issue. Furthermore, the use of local materials such as wastes and industrial by-products will contribute greatly to the sustainability if the materials are obtained from a sustainable industrial process. The moderate use of natural resources also will contribute to a more sustainable construction system.

2.4.3 WASTE MANAGEMENT

Beginning in the 1970s in the United States, concern about the environmental effects associated with increased waste generation and perceived decreasing landfill capacity led to increased public interest in recycling (**Greene and Tonjes, 2014**). In response to this concern, local municipalities implemented numerous recycling and waste reduction programs to reduce negative environmental effects associated with waste generation (**Sidique et al., 2010; Loughlin and Barlaz, 2006**). Awareness of the environmental impacts of waste has continued to grow and effective waste management is currently a key target in environmental policies worldwide (**Jenkins et al., 2009; Hazel, 2009**). It is now understood that growth in waste production unrestrained by proper reduction and management techniques can damage natural systems (**Mendes et al., 2012**). Therefore, how waste is managed directly affects local and global environmental quality (**Vergara et al., 2011; Bahor et al., 2009; Fischer, 2011**).

According to **Greene and Tonjes (2014)**, the main purpose of waste management technologies and policies are to protect human and environmental health by reducing the negative impacts of waste and finding beneficial reuses for it (**Melosi, 2000; USEPA, 2012a**). Specific foci of waste systems will differ depending on the level of system sophistication. Many developing countries still have unsophisticated, non-modernised waste systems (**Asase et al., 2009; Henry et al., 2006**), and this has led to a growing concern over the insufficiency of solid waste management in these countries (**Al-Khatib et al., 2007; Taboada-González et al., 2011**). Public health tends to be the motivating factor for waste policies in countries with unsophisticated waste management infrastructure (**Wilson, 2007; Vergara and Tchobanoglous, 2012**).

Green and Tonjes (2014) reported that in the United States, however, public health was a key driver of waste practices from the 19th century through the 1960s, but now drivers have shifted to environmental concerns (**Wilson, 2007**). Improved environmental protection through the optimisation of waste management practices is the typical focus of waste management policies and technologies in countries where strong legislation has been well established and immediate health concerns have been controlled (**Vergara and Tchobanoglous, 2012; Wilson, 2007**).

In terms of solution, recycling ranks highest on the waste hierarchy after reduction (**Snell and Hurst, 2009; Tojo and Fischer, 2011**). The beneficial re-use of industrial by products and general waste is an interesting solution for the waste management problem. For instance, the generalisation of use of coal fly ash in the construction industry (blended cements, soil stabilisation, etc.) promoted an environmental benefit and an improvement in the waste management of this residue, reducing the pollution of ground, air and water due to the disposal of fly ash **(Nalbantoglu, 2004)**.

2.4.4 INDUSTRY AND SUSTAINABILITY IN THE SOUTH OF SPAIN

The industry in the region of southern Spain had 290,000 employees in 2007 and a total revenue of 11.98 billion euros. The food industry is still the most important sector in the industry of this region with products such as olive oil or rice. The industrial processes carried out at the food factories produce a large amount of biomass. Nowadays, most of the biomass generated in the food industry is being re-used due to the recent installation of furnaces fuelled by biomass which generate energy for the industrial process itself.

The Junta de Andalucia (autonomous Government of Southern Spain) created in 2005 the Andalusian Agency of Energy to improve the energetic supply of the region. According to this agency, there is a great potential for the exploitation of renewable sources of energy in Andalusia, such as energy power plants in Andalusia, such as solar and wind power. Furthermore, in the area of southern Spain, several sustainable power plants have been installed in which the electric energy to be distributed to the national grid is produced from the incineration of the remaining biomass, which is an abundant waste. Due to the previously explained, the biomass and biomass ash are currently generated in large amounts in southern Spain posing a significant waste management issue but offering interesting by-products if alternative uses are found for them.

The industry of quarries in the area of Macael is also quite important within the industrial activity in the south of Spain. In Macael, limestone is extracted from huge natural outcrops. The layers of limestone have been encountered among layers of dolostone. From a commercial point of view, dolostone is not as much appreciated as limestone, because it is weaker and darker. Therefore, large amounts of dolostone are disposed off in the area of the quarries. The sludge generated during the extraction works of limestone must be considered as a waste. Since this sludge is rich in limestone and dolostone, it could add significant value to the construction industry, if a suitable use is found.

On the basis of the research carried out, there is a certain potential for the sustainable use of natural resources, waste and by-products in the area of southern Spain. The proposal of alternative and innovative production systems can also motivate the economical activity in the region.

2.5 ALTERNATIVE STABILISATION AGENTS

Searching a more sustainable construction process, traditional binders (like lime and cement) are being progressively replaced by non-conventional additives (wastes, industrial by-products, different kind of fly ashes, etc). These additives, if proved to have binding properties, are usually selected by a wide range of criteria, one the most important being the local availability and the specific conditions of different regions, areas and countries.

2.5.1 CRITIQUES TO TRADITIONAL ADDITIVES

Petry and Little (2002) presented a thorough review on the use of calcium-based additives for soil stabilisation: lime, cement and coal fly ash. Nowadays It is known that the most significant of these traditional binders, lime, promotes a quick effect called flocculation agglomeration of particles and a slow pozzolanic reaction which is responsible for the strength of lime-treated soils **(Solanki and Zaman, 2012)**.

However, some authors found lime to pose serious disadvantages for soil stabilisation in regions with great seasonal variations, marly soils, etc. **(Raymond and Ouhadi, 2006; Guney et al., 2007; Seco et al., 2011b)**. Hence the importance of testing new additives.

It is evident that the knowledge on the soil stabilisation treatment can still be broadened. For example, the performance of the treatment in the long term, the prevalence and durability of the flocculation of particle and the pozzolanic reactions, and the influence of the mineralogy of the soil on the result of the treatment can be further studied. However, beyond the technical doubts, there are increasing constraints to the use of products such as lime and cement. Lime and cement are expensive and they are not always locally available. On the other hand, the production process of lime and cement requires the use of huge amounts of energy and it releases carbon dioxide, hence contributing to a negative environmental impact of the construction industry. All this supports and motivates the current active search for alternative additives which is taking place in many regions.

2.5.2 STUDIES ON THE USE OF ALTERNATIVE ADDITIVES FOR SOIL STABILISATION

But which specific properties are required for a potential stabilisation agent? Based on the information provided by **Solanki and Zaman (2012)**, we would search for materials with a high amount of free lime and/or pozzolanic potential, i.e., alkalinity and high percentages of silica and alumina **(Seco et al., 2012)**. Such an additive would improve the mechanical behaviour of a cohesive soil. But according to **White (1949)** and **Drief et al. (2002)**, the swelling potential and plasticity of the soil depends strongly on the presence of expansive clay minerals within the soil.

The stabilisation of the expansive soils is an interesting option with the obvious economic advantage of using the natural soils instead of removing and disposing them off in landfills. The geotechnical engineer must choose between traditional and innovative soil stabilisation treatments. It is important that the geotechnical engineers keep on investigating new forms of soil stabilisation and that they keep up-to-date with the most recent studies so they can take them into account in their designs.

The use of traditional binders such as lime, cement and coal fly ash, has been thoroughly studied in the past (**Jones, 1958; McDowell, 1959; Petry, 2002**). However, these traditional agents are often expensive and not environmentally friendly. Thus, in recent years, a great effort has been done to study the potential benefits of the use of non-conventional additives for a really sustainable soil stabilisation process.

There is a wide variety of non-conventional additives with very different origins: agricultural wastes like rice husk ash (**Basha, 2005**) or olive cake residue (**Attom, 1998; Nalbantoglu, 2006**); industrial by-products such as recycled phosphogypsum (**Degirmenci, 2008**), waste plastic trays (**Ahmed, 2011**) or cement kiln dust (**Peethamparan, 2009**); and even natural resources like seawater (**Singh, 1978; Singh, 1999**).

The use of natural resources and industrial by-products in civil works can lead to a more sustainable construction process. Especially, the use of non-conventional additives for soil stabilisation can offer an interesting alternative to reduce the damaging effects of expansive clays. In recent years, many authors have tested the effectiveness of a wide range of alternative stabilisation agents, from biomass to recycled materials (**Clifton et al., 1980; Mathur, 2006; Attom, 2007; Hossain et al., 2007; Degirmenci, 2008; Eren and Filiz, 2009; Peethamparan et al., 2009; Hossain and Mol, 2011; Rahmat and Ismail, 2011; Seco et al., 2011a; Seco et al., 2011b**). Some studies focus on the mechanical behaviour of the treated soils whilst some other focus on the evolution of the microstructure, studying the electrical conductivity, mineralogy or formation of cementitious gels.

Xeidakis (1996; 1996b) studied the effects of a solution of magnesium hydroxide on a suspension of swelling clays, finding that the swelling potential of clays was reduced. The effects of the magnesium hydroxide in a powder form when it is added to a soil following conventional mixing techniques deserves to be further studied.

The same can be said on the effects of seawater and olive mill wastewater. In the existing literature there are studies reporting the effects of the sodium chloride (**Singh and Das, 1999; Lees et al., 1982; El-Sekelly, 1987; Osula, 1993; Singh and Ali, 1978; Davoudi and Kabir, 2011; Wu et al., 2007**) and olive cake residue (**Attom and Al-Sharif, 1998; Nalbantoglu and Tawqif, 2006**) on the physical properties of expansive soils. The chemical compositions of both seawater and olive mill wastewater involve elements such as calcium, potassium and magnesium which could promote a cation exchange when added to clay minerals. Hence the interest of a further research on the effect of this agents on the expansive soils.

The effect of the addition of different stabilisation agents to soils with poor engineering properties has been widely studied in the last few years. The use of waste materials with the suitable properties to interact with the soil can lead to a more efficient and sustainable construction process. Many authors claimed the importance of searching for new stabilisation agents with the potential to solve local, environmental and economic problems (**Clifton, 1980; Mathur, 2006; Attom, 2007; Hossain, 2007; Degirmenci, 2008; Eren, 2009; Peethamparan, 2009; Hossain 2011; Rahmat, 2011; Seco, 2011**).

Other researchers claim the importance of establishing a better understanding of the effects of traditional binders such as lime and cement, or study the combined effects of lime, cement and non-conventional additives admixtures (**Kaniraj, 1999; Prabakar, 2004; Cai, 2006; Ghosh, 2009; Sariosseiri, 2009**).

The potential to improve the soil behaviour throughout the use of magnesium was studied by **Xeidakis (1996a)**. The dolomite ash has a potential to introduce a combined effect of magnesium and calcium oxide in the soil. However, the presence of certain compounds such as carbonates or sulphates in the mineral composition of the soil can affect the performance of the additive used to enhance the soil behaviour (**McCarthy, 2012**). Several studies proved the importance of the alkalinity of the additive to produce mineral transformations in the soil towards more stable structures.

The term biomass fly ash involves many different materials whose properties and chemical composition strongly depend on the source of the biomass, the incineration process (time, temperature and methodology), collection points, etc. The potential of biomass fly ash from olive oil industry to improve the engineering properties of the expansive soils comes from the capacity of this fly ash to introduce an alkaline environment in the soil due to its high concentration of potassium.

There are some experimental treatments used when the origin of the problem is not smectite, e.g. when sliding between illite layers take place. In these cases, the exchange cation has been replaced. Sodium (Na) is easier to hydrate than potassium (K). Therefore, the injection of potassium solutions can reduce the expansivity.

2.6 EVALUATION AND CONTROL

According to **Sridharan and Prakash (2000)**, the index properties such as liquid limit, plasticity index and related parameters cannot satisfactorily predict the soil expansivity without taking into account the clay mineralogy.

However, and probably due to the difficulty of quantification of the mineral composition of fine-grained soils, not much is yet established about the relationship between changes in the mineral composition of the soil and the evolution of its physical properties, especially when non-calcium based additives are used.

The clarification of this aspect is crucial when testing new additives, since the swelling potential and plasticity of the soil depend on the minerals present in the soil, the montmorillonite being the most expansive **(White, 1949)**. According to **Drief et al. (2002)**, the smectite to illite conversion is controlled by temperature, pH and concentration of potassium. According to **Elert et al. (2007)**, the hydroxides such as KOH and NaOH are more efficient than $Ca(OH)_2$ when it comes to destroying clay minerals.

Many of the studies on lime-stabilised soils are based on changes in physico-mechanical properties **(Locat et al., 1990; Bell, 1996; Castro-Fresno et al., 2011)**. However, **Dafalla and Mutaz (2012)** and **Yong and Ouhadi (2007)** claimed the importance of analysing the chemical and mineral composition of soils and the use of currently available sophisticated means to improve our knowledge of the real process. In some soils, the mineral modifications can be crucial to achieve a long-term stabilisation. In fact, the results of the investigation carried out by **Cristelo (2012)** showed that the application of an alkaline activation, responsible for the mineral modifications, in conjunction with the addition of a low calcium fly ash could promote a better long-term stabilisation on marls than the use of a high calcium fly ash solely.

Mitchell (2005) highlights the type and amount of clay minerals present in the soil have enormous influence on its swelling potential. Previously, **White (1949)** showed that the plasticity of a soil depends on the type of clay minerals present. Therefore, the changes in the mineralogy of the treated soils along with the evolution of mechanical properties are important.

The transformation of smectite to illite (or interstratified smectite-illite) is known as illitisation **(Moore and Reynolds, 1997)**. This process is controlled by the temperature, the pH and the presence of potassium **(Drief et al., 2002; Elert et al., 2007)**.

2.7 DESIGN OF EMBANKMENTS

As it has been previously explained, in the last decades, and specially in the recent years, the soil stabilisation as a solution to enhance the low engineering properties of residual soils, such as marls, clays, etc. has become a very common process in the construction of earthworks and embankments all over the world. A really important feature which must be taken into account is the specific origin and properties of the soil, given the enormous influence which the presence of sulphates (gypsum), carbonates (marls) or extremely expansive phases (montmorillonite) can exert on the final outcome of the soil stabilisation. Many studies on soil stabilisation carried out in the lab only cover the first stage of the soil stabilisation, while earthworks and embankments are meant to last several decades.

Soil stabilisation lets geotechnical engineers face not only the engineering problems of soils (such as swelling potential or high plasticity) but also those related to the high economical and environmental costs of the infrastructure.

The most common use of soil stabilisation is in the construction of embankments for linear infrastructures such as railways and highways. The global design of these infrastructures depend greatly on many factor. The selection of the main route is aimed at providing a solution not only for the technical and engineering issues, but also for the geographical and social needs. The design of the route will determine the height of the embankments to be built. The geology of the area and the requirements of the infrastructure under service conditions will determine the minimum parameters to be achieved in the construction of the embankments.

In the design of embankments, the foundation and initial layers are very important, since the overall performance of the whole embankment will depend greatly on those, and the cost of maintenance of the highway or railway will be much higher when repairs will be needed in the base of the embankments.

The embankments are usually formed of a material with acceptable properties in terms of bearing capacity, plasticity, swelling potential, etc. The presence of organic matter will usually be limited to negligible values. The amount of fine-grained soils in the fill will also be limited. It is usual to encounter a capping layer on top of the fill. This capping layer is formed of a more competent material, with coarser particle size distribution, lower plasticity and higher bearing capacity. The capping layer is considered to be the top layer of the subbase and it is aimed at providing an efficient support for the overlying pavement (or rails).

In terms of standardisation, the detailed design of high-speed railway projects prepared for the Spanish Administration of Railways (ADIF) usually included specifications for the minimum acceptable characteristics of the soils to be used in each part of the embankment, which were usually more restrictive than the general Spanish standard for the construction of subbase for roads and highways (**PG-3, Spanish Ministry of Development, 1976**). The standard ICAFIR, released by Andalusian Agency of Public Works in Southern Spain (**2006**) highlighted the importance of the bearing capacity of the layers of natural soils located underneath the embankment and proposed a method of calculation of the minimum bearing capacity to be required in the foundation of embankments for roads and highways according to the design service conditions.

Therefore, the geometry of the embankments will be conditioned by the layout of the main route of the linear infrastructure. The availability of local soils to construct the embankment will depend on the geological conditions of the area. The bearing capacity of the existing ground will determine the need for excavation and replacement of the shallowest layers. Finally, the design of the embankment will be aimed at determining the minimum acceptable parameters to be requested in the material to be used for the core, capping and pavement.

When the natural soils have poor engineering properties and do not meet with the minimum requirements established, the stabilisation treatment can improve the physical properties of the natural soils and hence allow for their use.

In terms of performance, the main features of the embankment are: durability, settlement, stability. As it has been previously explained, the durability of the embankment is ensured throughout the specification of minimum physical properties. The settlement (deformation) and stability of the embankment (sliding, slope instability, etc.) will determine the usability of the embankment.

In an embankment with several metres of height, formed of layers of different soils and constructed along hundreds of metres of length, there are many parameters which can affect the settlement and stability. All these parameters can be taken into account throughout the use of numerical methods.

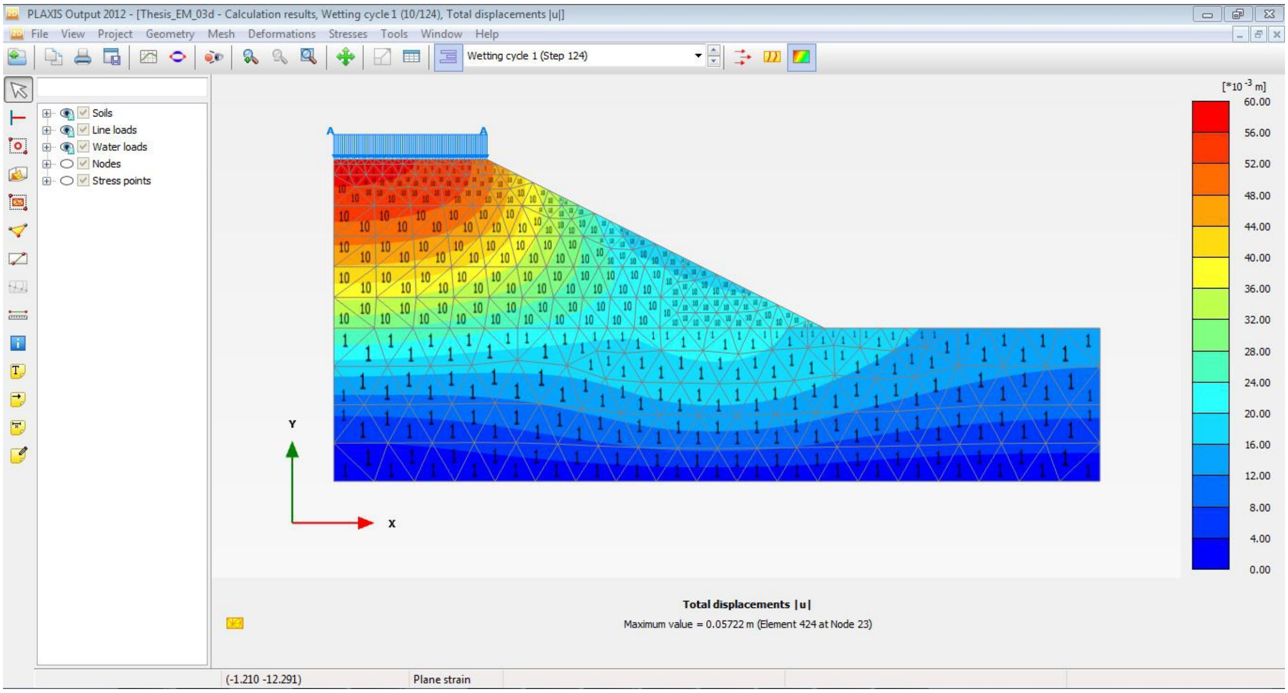
The numerical methods are mathematical tools aimed at provide approximate solutions to problems with a very complex analytical solution. As an example, the finite element methods and boundary element methods are some of the most widespread numerical model used in civil engineering. These numerical methods are extensively used in civil, structural and geotechnical engineering.

Nowadays, the numerical methods are usually applied in user-friendly pieces of software which allow for the preparation and solution of numerical models. These models can be very useful in the calculation of the expected stability of an embankment formed of treated soil according to the improvement of the mechanical properties of the soil. Therefore, the combination of these models and the laboratory experimental results can be very useful in the evaluation of the efficiency of the soil stabilisation treatments.

2.7.1 FINITE ELEMENT METHODS

Numerical analysis allows to find approximate solutions for problems defined by differential equations. One of the most significant example of this powerful analysis is the finite element method (FEM) which uses the calculus of variations to minimise an error function and produce a stable solution. Analogous to the idea that connecting many tiny straight lines can approximate a larger circle, FEM encompasses all the methods for connecting many simple element equations over many small subdomains, named finite elements, to approximate a more complex equation over a larger domain.

Finite element method (as many other numerical analysis methods) has been largely applied to physics, theory of structures and continuum mechanics and of course, to Geotechnics. In the day-to-day work of design engineers, FEM-based software has become an indispensable tool to evaluate and solve real geotechnical problems.


PLAXIS Output 2012 - [Thesis_EM_03d - Calculation results, Wetting cycle 1 (10/124), Total displacements |u|]

Wetting cycle 1 (Step 124)

Total displacements |u|

Maximum value = 0.05722 m (Element 424 at Node 23)

(-1.210 -12.291)

Plane strain

Figure 2.5. Example of output results in Plaxis 2D

2.7.2 MOHR-COULOMB IN PLAXIS 2D

Plasticity is associated with the development of irreversible strains. In order to evaluate whether or not plasticity occurs in a calculation, a yield function, f, is introduced as a function of stress and strain. Plastic yielding is related with the condition f = 0. This condition can often be presented as a surface in principal stress space. A perfectly-plastic model is a constitutive model with a fixed yield surface, i.e. a yield surface that is fully defined by model parameters and not affected by (plastic) straining. For stress states represented by points within the yield surface, the behaviour is purely elastic and all strains are reversible.

The basic principle of elastoplasticity is that strains and strains rates are decomposed into an elastic part and a plastic part:

$$\varepsilon = \varepsilon^e + \varepsilon^p$$

Hooke's law ($\sigma = E \cdot \varepsilon$) is used to relate the stress rates to the elastic strain rates leading to:

$$\sigma' = D^e \cdot \varepsilon^e = D^e (\varepsilon - \varepsilon^p)$$

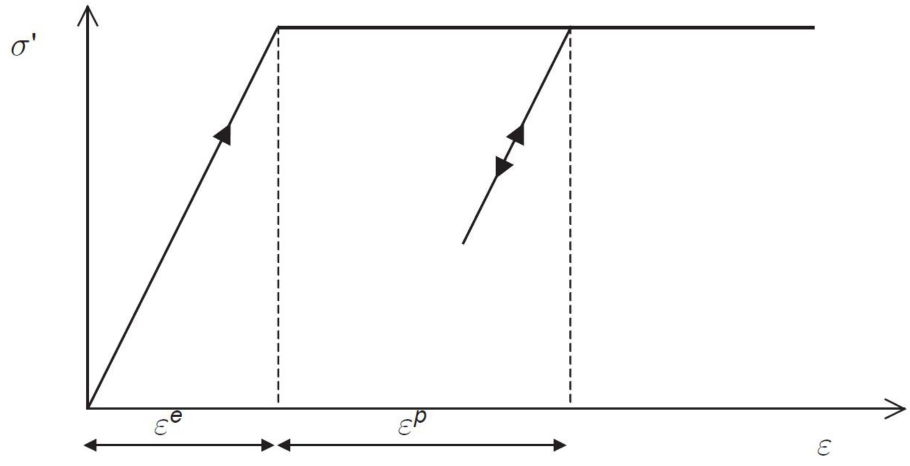


Figure 2.6. Basic idea of an elastic perfectly plastic model

The linear elastic perfectly-plastic Mohr-Coulomb model in PLAXIS 2D requires a total of five parameters, which are generally familiar to most geotechnical engineers and which can be obtained from basic tests on soil samples. These parameters with their standard units are listed below:

Young's modulus	kN/m^2
Poisson's ratio	-

Cohesion	kN/m^2
Friction angle	°
Dilatancy angle	°

The dilatancy angle is meant to model the capacity of soils to continue to dilate as long as shear deformation occurs (under drained conditions). The graphical presentation of the Mohr-Coulomb criterion is depicted in **Figure 2.6**.

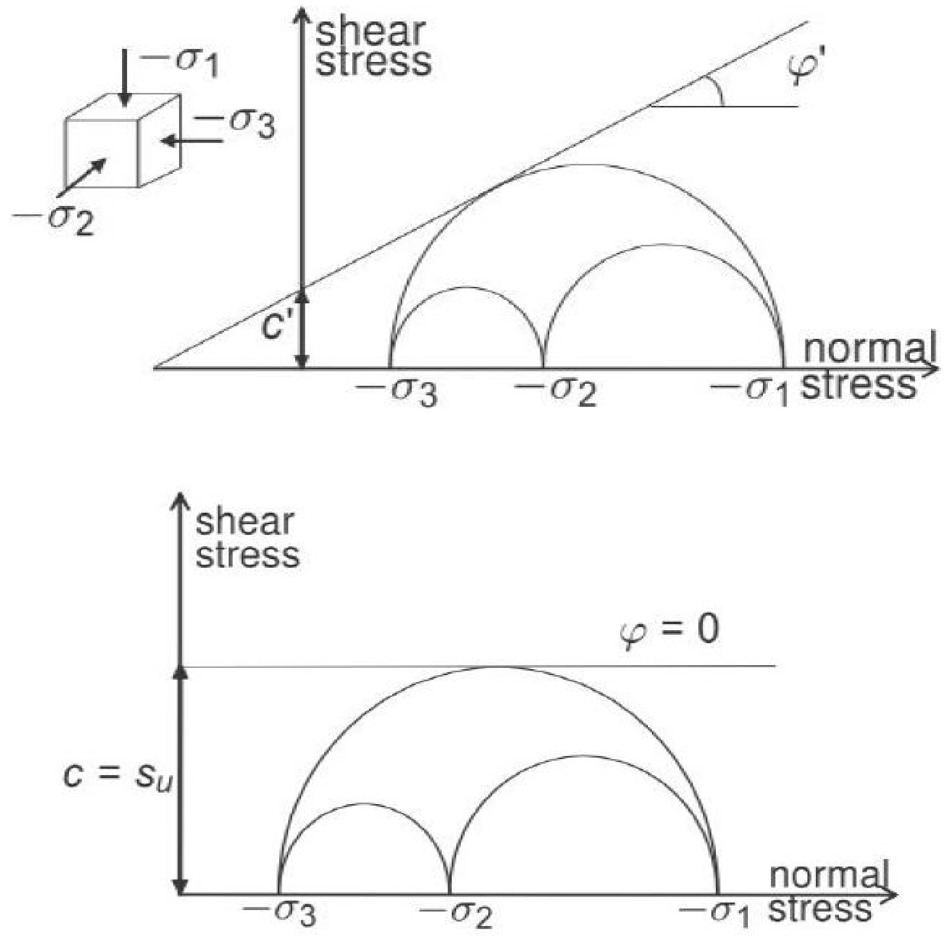


Figure 2.7 Mohr-Coulomb failure criterion:
a) using effective parameters; b) using undrained strength parameters

2.7.3 FACTOR OF SAFETY AGAINST FAILURE IN SLOPES

Slope instability remains a major preoccupation for geotechnical engineers, and routine quantitative stability analyses have long been based on limit equilibrium methods, in which a factor of safety, defined as the ratio of the actual strength of the soil to that required to maintain limiting equilibrium, is calculated. **Petterson (1955)** reviewed the history of the early work at Gothenburg leading to the introduction, in 1916, of the use of circular arc sliding surfaces to analyse the stability of clay slopes.

The March and June 1955 issues of Geotechnique formed the Proceedings of the European Regional Conference on the Stability of Earth Slopes, which had been held at Stockholm in September 1954. The proceedings included the paper by **Bishop (1955)** on effective stress slip circle analysis.

This type of limit equilibrium analysis is carried out by dividing the sliding mass into a number of vertical slices, and Bishop examined the error introduced by simplifying assumptions concerning the inter-slice forces. **Little and Price (1958)** programmed Bishop's method of analysis for the computer, which at the time was a major technical advance. The ability to carry out a large number of calculations very rapidly meant that it was possible to identify reliably the critical circular slip surface for which the factor of safety was a minimum. Probably from a notion that computers would never become widely available, several authors produced charts and stability coefficients from which the factor of safety of a slope could be obtained without laborious calculation **(Bishop and Morgenstern, 1960; Spencer, 1967)**.

Over the years more complex methods of analysis appeared in Geotechnique. In many field situations, the critical slip surface deviates significantly from a circle or a plane, and a method of limit equilibrium analysis for surfaces of any shape was developed by **Morgenstern and Price (1965)**.

Some of the most commonly used programs using general limit equilibrium and Morgenstern Price method are:

- Slide
- Slope/w
- Stabl WV
- SVSlope
- GALENA
- GSLOPE
- CLARA-W
- TSLOPE3

METHODOLOGY

Chapter 3

Research Stages and Geological Setting

Abstract

The section of Methodology is formed of two chapters. This chapter (Chapter 3) presents a chronological description of the different stages of this research, focusing on the motivations and scope of each stage. The steps followed in this research from the beginning of the investigation till the last stages will be described and outlined to provide the reader with a comprehensive insight of the study before the presentation of the experimental results and interpretations. Finally, a geological setting of the area of study is provided, given the fact that expansive soils were obtained from different locations in this area along the time of investigation.

3.1 RESEARCH STAGES

This dissertation presents a research on additives and methods for soil stabilisation. The process of the investigation was not immediate, but it was developed along time. Furthermore, the results of the initial investigation had an enormous influence on the subsequent steps to be followed. This section is an attempt to outline the story of this research and the composition of the experimental tests which will be presented in the section of results.

3.1.1. STAGE 2: THE STUDY OF THE POTENTIAL STABILISATION AGENTS

This dissertation presents the result of an experimental research. The approach of it has been the trial of soil-additives combinations to study different aspects of the soil stabilisation process.

In Southern Spain the use of biomass to fuel sustainable power plants started being widespread from the year 2000 and it is currently a well established power source. Both for the production and selling of energy to the electrical network and for the self-consumption of factories, the natural resource of biomass started being incinerated and the installation of biomass-fuelled furnaces became usual. This activity generates a large amount of residue in Spain derived from the biomass. This residue, like the biomass itself, is not unique but have variable properties. Among the different types of biomass, we can highlight: rice husk, olive mill wastewater (and its by-products), pruning residue, timber... Hence there is a variety of biomass ash generated as a residue of the previously explained industrial activity. Furthermore, biomass can be bottom ash or fly ash depending on its generation within the industrial process. Regardless of the type of ash and the origin of the biomass, the biomass ash is currently one of the most important industrial by-products in Spain and its waste management is susceptible to significant improvements.

Therefore, the beneficial re-use of biomass ash in the construction industry could have a very positive effect on the economy and the environment.

In this stage of the investigation, a characterisation of biomass fly ash and biomass bottom ash from the olive oil industry (a very important industry in Southern Spain) was carried out focusing on the comparative properties of these product and those of lime and coal fly ash.

In addition to it, a comparative study on several promising industrial by-products available in the same region (in this case, Southern Spain), was carried out. Rice husk ash, biomass ash from the olive oil industry, biomass ash from the paper industry and dolomitic ash from the waste of quarries were studied to characterise their physico-chemical properties and their pozzolanic activity, as a preliminary assessment of their suitability for use in soil stabilisation. All the mentioned agents were obtained in the form of a fine-grained fly ash.

3.1.2. STAGE 2: APPROACH TO THE CURRENT ISSUES ON THE SOIL STABILISATION

The second stage of the research was the development of two experiments with two clearly defined objectives: (1) study the effects of the wetting-drying cycles on a lime-stabilised marl; (2) study the effects of low cost locally available agents in Southern Spain on the index properties of a very expansive and plastic clay: bentonite. The agents used were magnesium hydroxide, seawater and olive mill wastewater from the olive oil industry.

In a holistic approach to the problem, the geotechnical and mineral tests were already used in this first stage and their combined results were taken into account in the interpretations.

The results of this stage suggested a further study, identification and classification of the available resources and a definition of the main properties which must have a potential stabilisation agent.

3.1.3. STAGE 3: CONTROL TESTS AND MICROSTRUCTURE OF STABILISED SOILS

The stages 1 and 2 were very illustrative in the knowledge and characterisation of the potential stabilisation agents. In the stage 3, traditional additives such as lime and coal fly ash, and alternative additives such as dolomitic ash and biomass fly ash from olive oil industry, were mixed with natural clayey and marly soils to observe the modification of the physico-chemical properties and mineralogy of the soils.

The experimental program carried out at this stage covered the following objectives:

- studying new methods to evaluate the performance of treated soils, such as the evolution of the particle size distribution measured by means of laser diffraction.

- studying the evolution of the index properties of the soils when treated with conventional and non-conventional additives.

- studying the comparative microstructure of the natural and treated soils by means of microscopy (field emission scanning electron microscopy was used).

3.1.4. STAGE 4: MECHANICAL PROPERTIES

The objectives and scope of this stage is motivated by the results and observations from previous research stages. Two natural soils (clayey and marly soil) were treated with dolomitic ash and biomass fly ash from olive oil industry. The study was focused on the mechanical behaviour of the treated soils. The evolution of the index properties and mineralogy of marly and clayey soils were also studied. The pH value of the soils was measured to analyse the effects of the variations of pH on the mechanical behaviour of the soil. Relationships were established among strength parameters.

3.1.5. STAGE 5: APPLICATION TO PRACTICAL DESIGN OF EARTHWORKS

The literature review provided the author with an accurate insight of the soil stabilisation problem and its current situation. Stages 1 to 4 of this research provided the required experimental results to identify further needs, characterise additives, draw evolution of index properties, infer explanations for the mechanical behaviour, propose the use of tests to evaluate the performance of the soil stabilisation treatments, etc.

The last stage of the research presented in this dissertation is motivated by all these experimental results previously obtained. Both in the case of alternative and traditional additives, what is the degree of improvement achieved by the treatment? What is the real impact of this improvement in the long-term performance of an embankment or foundation? Which is the criterion to accept or reject the use of stabilisation agents? This last stage of the investigation focused on the practical approach to the design of embankments.

In the real construction industry, there are standards and recommendations to be followed. But nowadays, the design of embankments and foundations usually rely on the predicted values of numerical modelling. In this last stage of the investigation, the range of improvement in the strength parameters and index properties of the soil are tested in the design of a typical embankment for linear infrastructure using two different methods of numerical modelling. The settlement, slope stability and factor of safety of an embankment is calculated for the case of natural soils and treated soils and the results obtained are compared and interpreted.

3.2. EXPERIMENTAL PROGRAM

Stage I

1) Several additives were studied and characterised: biomass fly ash and biomass bottom ash from the olive oil industry, coal fly ash, quicklime. The chemical composition, content of free lime, particle size and strength activity index (pozzolanic activity) were studied and compared. Chapter 5 summarises the findings of this stage of the research.

2) A thorough study and characterisation of available industrial by-products in the south of Spain is carried out. The agents studied were: biomass fly ash from the olive oil industry, biomass ash from the pulp and paper industry, rice husk ash and dolomitic ash. The results were compared with those of cement and sand. The main objective was the evaluation of the potential of these additives to be used for soil stabilisation. The study comprises particle size, chemical composition (by means of X-ray fluorescence (XRF)), pozzolanic activity (strength activity index, Frattini tests, NaOH solubility) and study of microstructure (SEM, XRD). The results and interpretations are presented in Chapter 6.

Stage II

1) A natural marly soil from the province of Jaen (Spain) was treated with 2.5 and 4.0% of lime and subjected to wetting-drying cycles. The results and interpretation of this experiment are presented in Chapter 7 of this dissertation.

2) An expansive and plastic bentonite was treated with 5%, 10% and 15% of alternative additives: magnesium hydroxide, seawater and olive mill wastewater. The results and interpretation of this experiment are presented in Chapter 8.

Stage III

1) Two natural soils from the area of study (marly soil and clayey soil) were treated with 5% and 10% of lime, coal fly ash, biomass fly ash and dolomitic ash. The evolution of the geotechnical and mineral properties of the treated soils along curing time was studied: plasticity, particle size distribution, carbonate and sulphate contents, mineral composition, etc. A further description of this experiment and a discussion of the results is presented in Chapter 9. A mineralogical study of the samples based on the FESEM (microscopy) results can be found in Chapter 10.

Stage IV

1) Chapter 11 presents a study on the mechanical behaviour of a marly soil treated with 5% and 10% of biomass fly ash and dolomitic ash. Treatment with cement was also used as a control. The shear strength, consolidation and unconfined compressive strength were studied along with the consistency, compaction and particle size distribution. Relationships between strength parameters were studied.

2) The mechanical behaviour, mineral composition and geotechnical index properties (consistency, compaction) were studied on a clayey soil treated with 5% and 10% of cement, biomass fly ash and dolomitic ash. The effects of the additives on the environmental pH and the influence of pH on the mineral composition of the soils was studied. The discussion of results and main findings are summarised in Chapter 12.

Stage V

1) The results of the previous stages were applied to the design of embankments by means of finite element method. The geotechnical software Plaxis2D^{TM} was used to analyse the relationship between the strength parameters of the soil and the performance of an 11-metre tall embankment: settlement on the top layer, factor of safety. Results and discussion are presented in Chapter 13.

2) The results of the finite element method for design of embankments were compared with those obtained using SLOPE/W (Geostudio^{TM}). A thorough study of the factor of safety of the embankment against slope instability was carried out. Further explanation of the calculations and results can be found in Chapter 14.

3.3 GEOLOGICAL SETTING

In this section, a geological setting of the natural soils used in this dissertation is presented. Along the research, marly and clayey soils were obtained from the provinces of Jaen, Granada and Almeria (Southern Spain) to test the effects of traditional and non-conventional additives on the soil.

3.3.1 MARLY SOILS FROM JAEN

A definition of the marl and its most significant features was provided in the Introduction. Marly soils are formed of clay and carbonates, with calcium carbonate being the most commonly encountered.

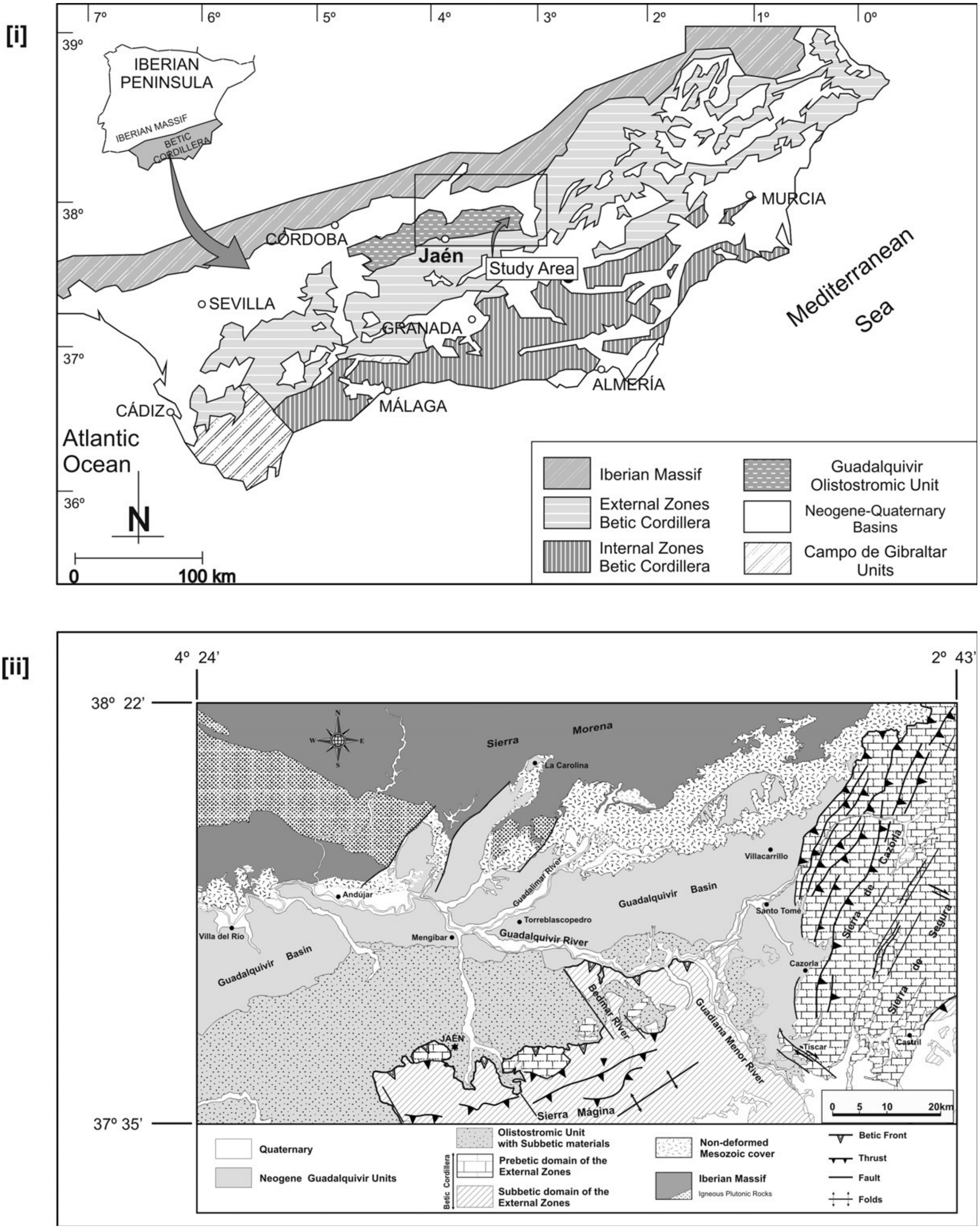


Figure 3.1. Geological framework of the study area: (i) regional geology map; and (ii) local geology map showing the main lithological units, from Lorite-Herrera et al. (2008)

Marly soils from the province of Jaen (Spain) are characterised for their poor engineering properties, high plasticity and expansivity. Marly soils from the province of Jaen (Spain) were used in the experimental programs described in chapters 5, 9 and 11.

From a geological point of view, the area of Linares-Baeza (province of Jaen, Spain) is located within the Guadalquivir basin, located between the front of the Betic Cordillera (active margin) and the Iberian Massif (passive margin), as shown in **Figure 3.1**. According to **Lorite et al. (2008)**, several lithological units, ranging from middle Miocene-Pliocene to Quaternary in age, crop out in the Guadalquivir River basin. The depression is filled with unconsolidated Quaternary and Tertiary sediments. The alluvial sequence overlies basement rocks that crop out on both sides of the alluvial terraces. The alluvial Quaternary deposits consist of conglomerates, gravels, sands and silts, whereas the basement rocks consist of Miocene marls and clays **(Vera, 2004)**. Also worthy of note are the presence of detrital rocks (quartzites, shales and red sandstones) deposited in the basin from the Iberian Massif Paleozoic sequence to the north. Tertiary sedimentary rocks rich in evaporites such as halite and gypsum were also deposited during the peak orogenic activity from the External Zones of the Betic Cordillera to the south. As a result, autochthonous materials crop out within the northern half of the basin, whereas the southern half of the basin comprises important allochthonous deposits (Olistostromic Unit). These olistoliths derive from gravitational slides from the Betic Cordillera and consist of Mesozoic and Cenozoic materials (marls, clays, sandstones dolomite and gypsum) with a chaotic structure **(Vera, 2004)**. In addition, celestite deposits in Triassic (Keuper) and Lower Liassic materials crop out in the southern half of the Guadalquivir basin in the Province of Jaén. . According to **Rey et al. (2013)**, the depression of Guadalquivir is filled with olistostromic units on which the autochthonous filling sediments of the basin from the late Tortonian-Messinian age are deposited **(Roldan Garcia et al., 1991)**. This second group of sediments is mainly formed of four units: conglomerate sediments, calcarenite sediments, marlstone and sandy sediments.

3.3.2 CLAYEY SOIL FROM GRANADA

A natural clayey soil obtained in Alicun, Granada (Southern Spain), was used along this study to test the effect of traditional and alternative stabilisation agents. This soil is involved in the experiments described in chapters 9 and 12. Figure 3.2 shows the location of the natural outcrops, close to river Fardes.

Alicún de las Torres is located in the southeast of the Iberian Peninsula, at the contact zone between the two main domains of the central sector of the Betic Cordillera: the External and Internal Zones. This contact is fossilised by the filling materials of the Guadix-Baza Basin that is the main intramontane Neogene basin in the Betic Cordillera.

Therefore, the basement of this geological system includes Triassic metamorphic materials, which outcrop to the south (Internal Zones), and Mesozoic carbonate materials, outcropping to the north (External Zones). In the Internal Zones, materials from the Alpujárride complex are well represented. This complex is the largest of the Betic Cordillera that, in the studied area, particularly in the Sierra de Baza, overlies the Nevado-Filábride Complex. The Alpujárride materials were affected by a moderate to severe Alpine metamorphism, and lithologically are characterised, from the bottom to the top, by: i) a metapelitic formation, mainly formed by Paleozoic shales; and ii) a second Permo-Triassic unit of the same nature, with interlayer limestone, dolomite and quartzite levels. Finally, towards the top of the Alpujárride Complex, a middle-upper Triassic formation appears, constituted of both calcite and dolomite marbles, with frequent interbedded gypsum levels that have been frequently identified in the field **(Prado Perez and Perez del Villar, 2011)**. Materials that outcrop to the north belong to the External Zones and are characterised by sediments aged between the upper Triassic and the Eocene, and mainly formed by evaporitic-bearing marls from Keuper; dolomitic limestones from the Jurassic Milanos formation; and the interbedded marls belonging to the Fardes and Lechos Rojos formations, aged between the Cretaceous and Paleogene **(Viseras et al., 2004)**. The Tertiary materials, consisting of the infill of the Guadix-Baza Basin, are mainly characterised by sandstones and conglomerates, as well as marls, clays and gypsum **(Prado Perez and Perez del Villar, 2011)**.

Figure 3.2 Location of the extraction points of clayey soil from Alicun, Granada (SE Spain)

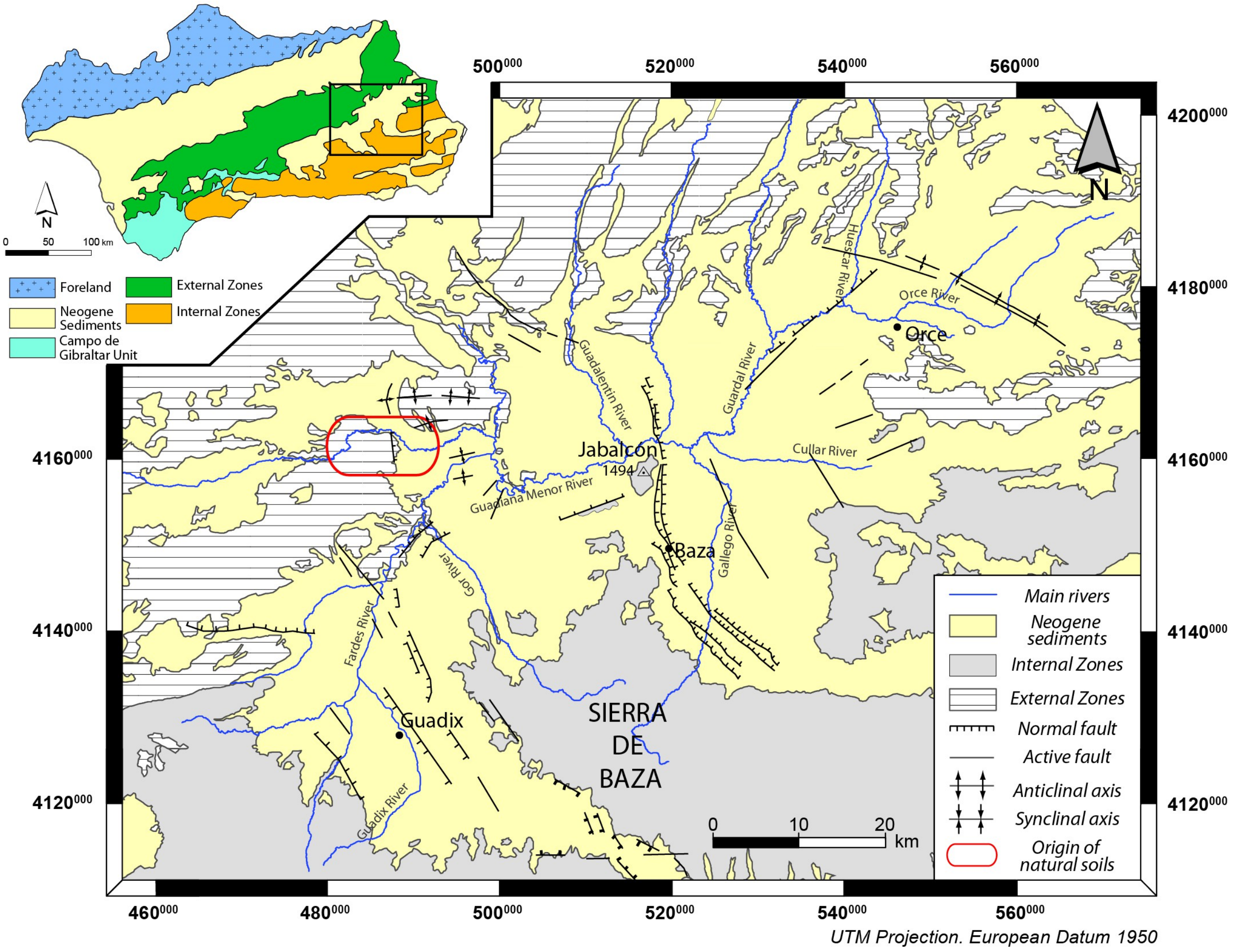


Figure 3.3. General geologic map of the Neogene Guadix-Baza basin with an insert showing the general tectonic setting (modified from Perez-Peña et al., 2009)

Perez-Peña et al. (2009) developed a geological study on the Guadix–Baza basin, where the area of Alicun is located. According to this work, the Guadix-Baza basin is a Neogene intramontane depression of the Betic Cordillera. The basin blankets the contact between the Internal and the External Zones of the Cordillera. Its basement comprises Mesozoic carbonate rocks (External Zones) in its northern sector and Triassic metamorphic rocks (Internal Zones) in the southern sector **(Fig. 3.3)**. The sediment fill is markedly continental and extends from the Late Tortonian to theUpper Pleistocene **(Vera, 1970; Viseras,1991; Fernández et al., 1996)**. On the southern and eastern borders of the Guadix sub-basin, the continental fill is formed by an alternation of poorly cemented conglomerates and sands of the Guadix Formation **(Vera,1970)**; towards the central part of this sub-basin, these deposits gradually change to marls and clays **(Viseras, 1991; Fernández et al., 1996)**. The Neogene fill of the Baza sub-basin is composed of an alternation of lacustrine and palustrine deposits together with alluvial deposits in the distal parts. The upper levels of these deposits have been dated by means of amino acid racemisation ratios from ostracods, yielding ages around 280 ka (**Ortiz et al., 2004**).

The sedimentation in the Guadix–Baza basin ends in the Upper Pleistocene with the formation of a thick calcrete (0.5–1 m) that constitutes a residual surface or glacis (**Perez-Peña et al., 2009**). This glacis, where preserved, is visible across the whole Guadix–Baza Basin and has been used as a reference level to characterise active tectonics in the basin (**García-Tortosa et al., 2008**). Subsequent to the development of this glacis, sedimentation in the basin is restricted to alluvial and colluvial facies, slope deposits, and fluvial terraces. The Pliocene–Pleistocene history of the Guadix–Baza basin seems to be dominated by sedimentary processes, with a lack of significant tectonic events. The Quaternary deformation in this basin is constrained to a few important normal faults as well as some open folds (interlimb angle 70°–120°; **Ramsay and Huber, 1987**) that slightly deform the glacis surface (**García-Tortosa et al., 2008**), some related with diapirism. The most prominent tectonic structure of this basin is the Baza fault.

3.3.3 BENTONITE FROM ALMERIA

Bentonite is a very pure clay which is mainly formed of smectite. The bentonite of Cabo de Gata, Almeria (Southern Spain) is usually extracted and sold for commercial uses, such as the preparation of bentonite sludge which is used as a retaining method for excavations in civil engineering. Provided in the form of powder, this bentonite was used in one of the first experiments carried out in this investigation: the addition of locally available natural resources and industrial by-products on an expansive bentonite soil. This experiment is described in chapter 6.

According to **Leone et al. (1983)**, the bentonites from Cabo de Gata, Almeria, is mainly formed of montmorillonite, hence its enormous swelling potential. Accessory minerals are plagioclase, quartz, hornblende, biotite, low-temperature tridymite, zeolites, sanidine and kaolinite.

In terms of genesis, the experimental works carried out suggested that the smectites formed mainly as a result of the alteration of plagioclase by Na-C1-SO_4 hydrothermal solutions (**Reyes et al., 1980**). The absence of paragonite sets an upper limit of ~325°C (**Hemley et al., 1961**) for the bentonitisation process; the presence of zeolites of the clinoptilolite-mordenite type indicates temperatures ranging from ~25°C to a maximum of 100°C (**Iijima and Utada, 1971**) for the less altered areas (**Leone et al., 1983**).

The deposits of bentonite are associated with volcanic rocks of Cabo de Gata, Southern Spain (**Figure 3.4**). Volcanic activity in this area lasted from Upper Oligocene to Tortonian times, different cycles being separated by transgressive fossiliferous limestones and calcarenites (**Lopez-Ruiz and Rodriguez-Badiola, 1980**). The volcanic rocks give K:Ar ages ranging from 8 to 17 m.y. (**Bellon and Brouse, 1977; Bellon and Letouzey, 1977**). Each of the cycles started with strong explosive activity, which produced tuffa, ignimbrites and conglomerates, followed by lava flows, tuff lavas and subvolcanic intrusions. The intrusions vary in composition from micaceous-amphibolic andesite, pyroxenic andesite and micaceous dacite to massive glassy dacite, depending on the time of emplacement (**Leone et el., 1983**).

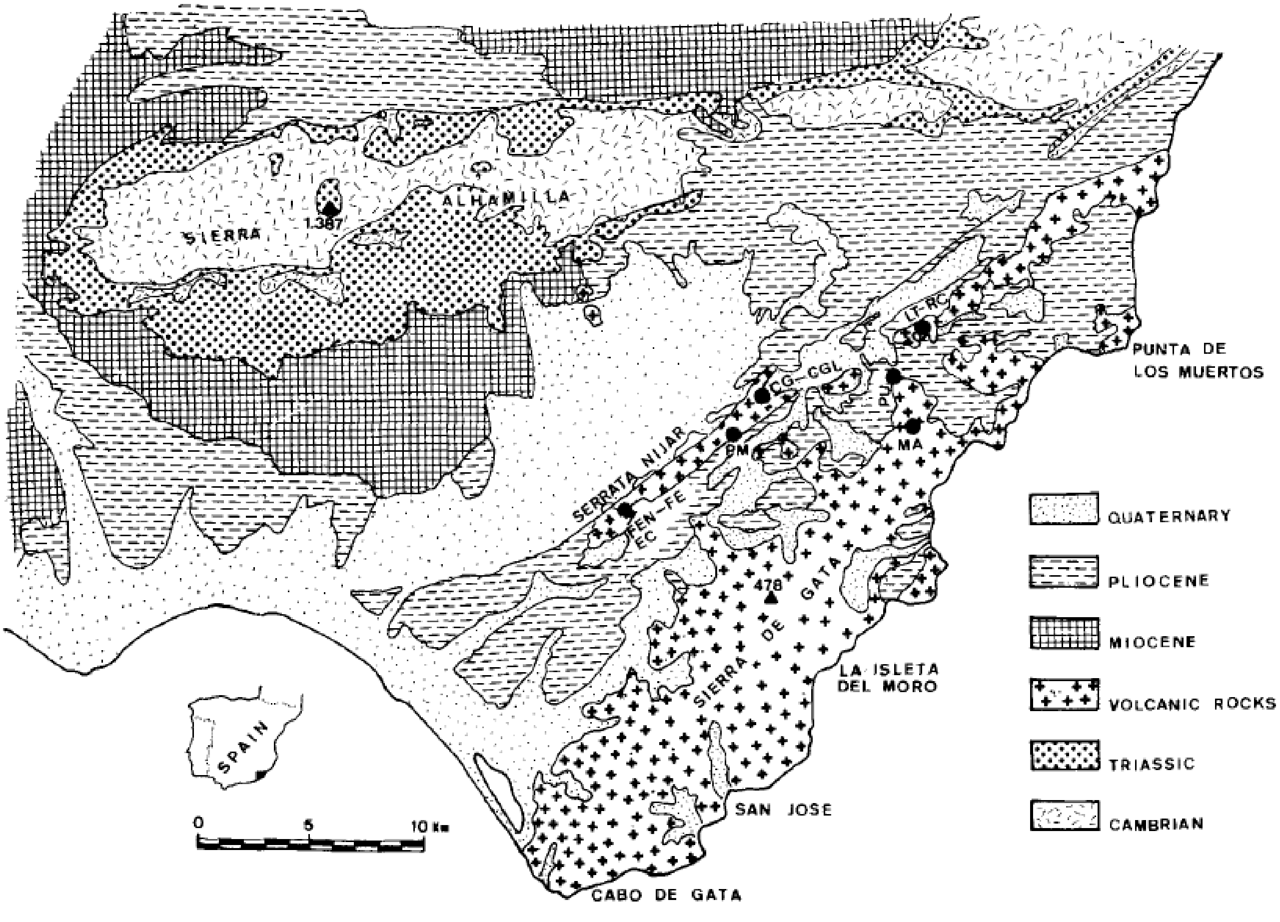


Figure 3.4. Geological map of the Cabo de Gata, Southeastern Spain, as presented in Leone et al. (1983)

The volcanic events were followed by acidic and near-neutral hydrothermal activity. The former mostly affected the ignimbrites, altering them to alunite, jarosite and kaolinite. It has been suggested **(Reyes, 1977)** that the bentonites formed as a result of the action of near-neutral hydrothermal fluids on tuffa and tuff lavas.

An initial investigation on the bentonites from Cabo de Gata was carried out by **Gonzalez-Garcia and Martin-Vivaldi (1949)**. Several reviews of the mineralogical and geochemical studies carried out on these bentonites of Cabo de Gata are available in the existing literature **(Reyes, 1977; Linares et al., 1978; Reyes et al., 1980)**. These data, in conjunction with geological evidence (fracture systems, relics of geothermal activity), suggest a hydrothermal origin for regarding the environment of formation or the nature and temperature of the altering fluids **(Leone et al., 1983)**.

Some of the most important outcrops of bentonite in Cabo de Gata, Almeria, were listed by **Leone at el. (1983)**:

Pozo Usero (PU). This deposit is related to a N50E fracture partially fossilized by Miocene conglomerates and limestones. The most altered materials are dacitic and rhyofitic tuffa, dacitic ignimbrites and agglomerates. The colour of the bentonites ranges from white to dark ochre **(Linares et aL, 1972)**.

Los Traneos (LT). This is the most important deposit of the Cabo de Gata region, and is related to two large fractures trending N 10E and N60E. The altered materials are dacitic tuffa and agglomerates. In some places there is a gradual transition from the less-altered parent rock to a pure white bentonite.

Rincon de las Caleras (RC). The most altered zone follows an approximate trend of N30W, close to that of the regional fractures. Because of the presence of much superficial cover, it is not possible to determine whether or not the deposit is located in a fracture zone. The bentonites contain much sand-size material and are yellow-white to grey in colour.

La Valentina (MA). This deposit is related to N-S and N30W fractures. The most highly altered materials are dacitie and amphibolic agglomerates, and dacitic and rhyolitic tuffa. The overburden is calcareous and about 40 m thick. The predominant colours of the bentonites are grey, yellow and green.

Collado del Aire (EC, FE, PM, FEN). These bentonites are localized in a zone of intense fracturing with E-W, N-S and N40E trends. Colours are red, green, blue, brown and yellow. The enclosing rocks are massive and agglomeratic andesites.

Cerro Gordo (CG and CGL). The bentonites occur below a volcanic series consisting of dacites, dacitic agglomerates and cinerites. The latter, occurring at the contact with the bentonites, are only slightly altered. The bentonites are green or olive green.

Chapter 4

Test Procedures

Abstract

This chapter (Chapter 4) presents an outline of the experimental program and an insight of the different materials used along this research, focusing on the natural soils used to study the performance of the soil stabilisation treatments. The experimental procedures followed during the development of the different stages of this research are also presented in this chapter. This includes the laboratory conditions, mixing techniques, curing storage, testing procedures, etc. Along the research presented in this dissertation, many different tests have been carried out. The standards and references observed for the development of those tests are also presented in this chapter to provide the reader with a comprehensive description of the methodology used.

4.1 INTRODUCTION AND STRUCTURE

As explained in the previous chapter, along this research several natural soils were mixed with traditional and non-conventional additives to study the soil stabilisation process. The soils were obtained from natural outcrops in Southern Spain (see Geological Setting in Chapter 3). All the soils used had poor engineering properties: high plasticity, high swelling potential, low bearing capacity, etc.

In section 4.2, the mixing procedures and laboratory conditions followed during the experimental program are outlined. Section 4.3 describes the standard and references followed for the development of the geotechnical tests. Section 4.4 explains the tests carried out to study the pozzolanic activity of additives. Finally, an explanation of the tests carried out to study the mineralogy of the soils can be found in section 4.5.

4.2 MIXING PROCEDURES AND LABORATORY CONDITIONS

All mixes were prepared under the same conditions in a laboratory with a controlled temperature of 20 ± 2°C. The soil-additive mixtures were initially prepared dry to create an even mixture. After weighing the exact quantities of dry soil and dry additive, they were placed in a mixing tray and thoroughly mixed for at least 10 min. Water was then added to the mix in order to reach the optimum moisture content calculated for the original untreated soil. The soil, additive and water were mixed in an industrial mixer for at least ten more minutes. Afterwards, the mixes were placed in a curing room for the required length of time. The curing room had the following conditions: temperature of 20 ± 2°C, humidity of 95 ± 5%.

When the samples were subjected to wetting-drying cycles, the samples were prepared in a laboratory tray and their weight was greater than 3 kgs each to ensure there was enough amount of treated soil for all the different tests to be conducted. The trays were then saturated for 7 days in a curing room with stable conditions of moisture and temperature (95-100%; 20ºC). The samples were finally subjected to 1, 3 and 5 wetting-drying cycles. One wetting-drying cycle consisted on 7 days of water immersion followed by 7 days of heat (50ºC). After the cycles, the samples were stored for 7 days under water saturation conditions to complete the curing process. The final curing time was 77 days.

4.3 GEOTECHNICAL TESTS

4.3.1 STANDARD AND MODIFIED PROCTOR COMPACTION TEST

The standard Proctor tests were carried out according to the British standard BS 1377-4 **(BSI, 1900)** and its equivalent Spanish standard UNE 103500 **(AENOR, 1994)**. Following this standards, several samples of soil are to be prepared at different moisture contents. The sample is compacted in a mould using a hammer of 2.5 kgs. The test must be repeated for samples with different moisture contents which promotes a variation of the compaction degree achieved in the mould. A curve can be plotted which shows the relationship between moisture of the samples (%) and density of the samples (Mg/m^3). The maximum value of density is called maximum dry density (MDD). The moisture at which MDD is achieved is the optimum moisture content (OMC).

In the case of the modified Proctor compaction test, the sample is then compacted by 27 blows of a 4.5 kg hammer. A second and third layer of soil is compacted in the mould following the same procedure. The test is repeated for different moisture contents resulting in different values of the density. The density and corresponding moisture content are plotted. The maximum value is called maximum dry density (MDD). The moisture at which the MDD was attained is called the optimum moisture content (OMC).

4.3.2 ATTERBERG LIMITS TEST

The Atterberg consistency limits were determined in accordance with the Spanish standards UNE 103103 **(AENOR, 1994)** and UNE 103104 **(AENOR, 1993)** which present a methodology to obtain the liquid limit (LL) and plastic limit (PL) respectively. Plasticity index (PI) is obtained according to the following formula: PI = LL–PL. These standards are equivalent to the ASTM standard D4318 **(ASTM, 2010)** and British standard BS 1337-2 **(BSI, 1990)**.

4.3.3 CALIFORNIA BEARING RATIO TEST

California Bearing Ratio (CBR) test on stabilised soil specimens was conducted as per Spanish standard UNE 103502 **(AENOR, 1995)**, equivalent to ASTM standard D1883 **(ASTM, 2010)**. To carry out the CBR test, the specimens were first assessed under a Modified Proctor Test in accordance to the standard UNE 103501. The specimens were then cast into the CBR-mold with the same compactive energy per volume as in the Modified Proctor Test.

4.3.4 PARTICLE SIZE: SIEVING AND LASER DIFFRACTION

Sieving tests were conducted following the ASTM standard D6913 (**ASTM, 2009**).

Laser diffraction measures particle size distributions by measuring the angular variation in intensity of light scattered as a laser beam passes through a dispersed particulate sample. Large particles scatter light at small angles relative to the laser beam and small particles scatter light at large angles, as illustrated below. The angular scattering intensity data is then analysed to calculate the size of the particles responsible for creating the scattering pattern, using the Mie theory of light scattering. The particle size is reported as a volume equivalent sphere diameter. Mie theory of light scattering is used to calculate the particle size distribution, assuming a volume equivalent sphere model. Mie theory requires knowledge of the optical properties (refractive index and imaginary component) of both the sample being measured, along with the refractive index of the dispersant. Usually the optical properties of the dispersant are relatively easy to find from published data, and many modern instruments will have in-built databases that include common dispersants. For samples where the optical properties are not known, the user can either measure them or estimate them using an iterative approach based upon the goodness of fit between the modelled data and the actual data collected for the sample. A simplified approach is to use the Fraunhofer approximation, which does not require knowledge of the optical properties of the sample. This can provide accurate results for large particles. However it should be used with caution whenever working with samples which might have particles below 50µm or where the particles are relatively transparent.

4.3.5 SWELLING PRESSURE

The British Standard BS 1377-6 (**BSI, 1990**) (equivalent to Spanish Standard UNE 103602 (**AENOR, 1996**)) was followed to determine the swelling pressure tests. All the samples for this test were prepared at the optimum moisture content calculated by the Standard Proctor compaction test. The swelling pressure test is described in section 3.5.2.2 of the BS 1377-6.

To carry out the swelling pressure test, the same equipment used for consolidation tests is required. The objective of this test is measuring the pressure exerted by the soil on the upper layers due to a swelling phenomenon. For that, a sample of soil is placed in the consolidation cell between two porous stones and confined laterally using several collars. Afterwards, the sample is subjected to saturation. The vertical strain of the specimen must be prevented during the whole test. If the initial saturation promotes an upward movement, the seating pressure applied must be increased to hold the reading of the gauge at the initial value. Finally, the swelling pressure value

(Ph) is obtained by the following formula: Ph = (Q/S) x 103 (kPa), where Q is the equilibrium loading and S is the section area of the sample. For low permeability soils, the equilibrium loading can be reached after at least 24 h of testing. **Figure 4.1** shows a diagram of the swelling pressure test apparatus according to **Akcanca and Aytekin (2012)**. The procedure is described in the standard as follows:

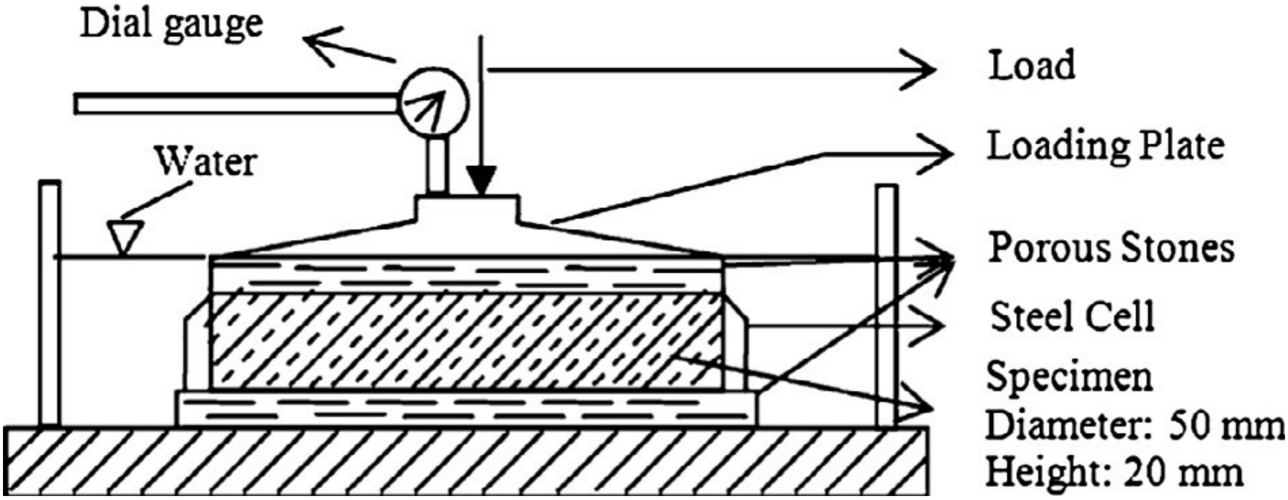


Fig. 4.1. Swelling pressure test apparatus, according to Akcanca and Aytekin (2012).

A soil that is susceptible to swelling shall not be allowed free access to water without provision for applying a vertical confining stress to prevent swell. Initial saturation is effected by allowing de-aerated water to enter at the base and to percolate upwards, while observing the compression gauge. If this indicates an upward movement increase the seating pressure applied to the diaphragm to hold the reading at the initial value. When conditions become steady, record the diaphragm pressure.

NOTE 1 Considerably longer than 24 h might be needed to reach steady conditions in large specimens of low permeability soils. The difference between the corresponding vertical stress applied to the surface of the specimen and the pressure of the water applied to the base is reported as the "swelling pressure". Pockets of air remaining between the diaphragm and the cell wall may be removed by the application of a vacuum through the rim drain valve, but this should be done with great care. During the subsequent saturation stage the effective vertical stress applied to the specimen shall not at any time be less than the swelling pressure.

NOTE 2 The pressure differential between the vertical applied pressure and the base water pressure should be not less than the swelling pressure, nor large enough to cause premature consolidation of soft soils. A differential pressure of 10 kPa has been found to be suitable for many soils that are not susceptible to swelling.

4.3.6 FREE SWELL

The standard used for this test was ASTM D2435 **(ASTM, 2004)**.

4.3.7 CONSOLIDATION TEST IN OEDOMETER

The oedometer test was performed according with BS 1377-6 **(BSI, 1990)**. This test was carried out on the natural soil and the samples treated with 10% of BFA and 10% of DA, after 28 days of curing. The samples were tested at a moisture content 1.5 times their liquid limit. The samples were loaded up to 910 kg.

The procedure followed and the parameters calculated in accordance with BS 1377-5: 1990. The consolidation process uses the initial cap weight loading of 1.86kPa followed by 7.44kPa, 12.99kPa, 29.62kPa, 64.9kPa, 127.58kPa, 253.23kPa, 504.82kPa, 1008.78kPa and 2015.47kPa, to calculate the compression curve (CC). After consolidation a series of unloading was under taken from 2015.47kPa followed by 1008.78kPa, 504.82kPa, 253.45kPa, 127.50kPa, 52.51kPa, 27.25kPa, 12.99kPa, 1.86kPa to develop the swelling curve (CS). In accordance with BS 1377-5: 1990 the following parameters are calculated:

- Total pressure on sample (P) / Vertical Effective Stress ($\sigma V'$),
- Height of sample at start of loading (H1),
- Height of sample at end of loading (H2),
- Mass of sample (mO),
- Bulk density (ρ),
- Moisture content (WO),
- Dry density (ρd),
- Void ratio at start of loading (eO),
- Void ratio at end of loading (e),
- Coefficient of volume compressibility (mV),

The Coefficient of Compressibility (CV) for each loading stage is determined using the Log Time curve fitting method in accordance with BS 1377-5: 1990. In BS 1377-5: 1990: clause 3.6.3.2.4, the Coefficient of Compressibility (CV) = (0.026 * H2) / t50. H is the average specimen thickness for the load increment (mm).

Hydraulic Conductivity / Permeability (k) (m / year) = Coefficient of Compressibility (CV) (m2 / year) * Coefficient of Volume Compressibility (mV) (m2 / kN) * Bulk Unit Weight of Water (γW) (kN / m^3) (O'Sullivan, 2011).

All applicable parameters are calculated three times:

- Initial Calculation: as the test is being conducted using the initial sample mass measurement, (m_0 = 53.2g).
- Back Calculation: when the test is finished and the sample mass measurement is repeated, (m_0 = 32.49g).
- Final Calculation: when the dry mass of the removed sample is measured and the initial sample's mass measurement is corrected for error using the sample's initial moisture content, (m_0 = 50.20g).

4.3.8 SHEAR STRENGTH

The small shear box apparatus was used for this experiment in accordance with BS 1377-7 **(BSI, 1990)**. The sample was initially consolidated under a normal stress of 70kg and then sheared using the appropriate rate to avoid the development of excess pore water pressures. The test was performed on the natural soil, and the samples treated with 10% of BFA and 10% of DA, after 28 days of curing.

The small shear box test apparatus is prepared in accordance with BS 1377-7: 1990. The initial consolidation to avoid loss of material is carried out by consolidating under cap weight (equivalent 1.26kPa), hangar weight (equivalent 13.79kPa), followed by 26.05kPa, 50.58kPa, 90.65kPa and 197.76kPa, each for a minimum 24hr period. The confining stress ($\sigma v'$) on the sample is equal to 197.76kPa. This is a low confining stress. The sample height measurement is measured using a two dial gauge system. The first dial gauge is used during initial cap weight loading. The second dial gauge is used during the hangar weight loading and subsequent loadings. This induces a slight error in the calculation of the height of the sample at the start of hangar weight loading. The calculated height of the sample at the end of cap weight loading = 26.15mm from first dial gauge. The calculated height of the sample at the start of hangar weight loading from dummy height measurement = 26.11mm. Dial gauge reading error = 26.15mm - 26.11mm = 0.04mm. This error could not be avoided. It is difficult to control due to the reality of placing hanger weight onto the sample and swinging of dial gauge into position. The sample has consolidated before the first dial gauge reading is read. The 26.15mm figure is used for the further calculations, as it continues from the cap weight loading.

The BS 1377-7: 1990 standard is used to calculate the shearing rate. The shearing rate used curing testing is 0.003mm / min. This shearing rate is used to simulate drained conditions during shearing and to prevent the build-up of excess pore pressures. The shearing rate is calculated for all the five consolidation stages.

In accordance with BS 1377-5: 1990 the following parameters are calculated:

- Total pressure on sample (P) / Vertical Effective Stress (σV'),
- Height of sample at start of loading (H1),
- Height of sample at end of loading (H2),
- Shearing Rate,
- Horizontal displacement (δh),
- Vertical displacement (δv),
- Initial Area of Shear Plane (AInitial),
- Current Area of Shear Plane (ACurrent),
- Normal Effective Stress (σV'), Normalised by ACurrent,
- Shear stress (τ), Normalised by ACurrent,
- Axial Strain (γ),
- Volumetric Strain (εv)
- Stress Ratio: Shear Stress / Normal Effective Stress (τ / σ'v),

4.3.9 UNCONFINED COMPRESSIVE STRENGTH

Soil is prepared by mixing the sample as close as possible to the optimum moisture content from the corresponding heavy laboratory compaction curve calculated in the previous section. The sample is compacted in the mould used for the standard Proctor compaction test.

The standard Proctor compaction test is carried out on the soil using a hammer weight of 4.5kg ± 50g with a fall height of 450 ± 4mm. The soil is placed in the mould in three layers with each layer receiving 27 hammer blows, in accordance with BS 1377-4: 1990. The compacted sample is trimmed in the mould. The moisture content is measured from the trimmings. The sample dimensions measured and the sample wrapped in cling film. The UCS test is carried out with the testing machine and the strength recorded.

4.3.10 PH

The determination of pH values of the samples was carried out following the standard ASTM D4972 **(ASTM, 2007)**.

4.4 POZZOLANIC ACTIVITY TESTS

4.4.1 FREE LIME

Free lime contents of all fly ashes were determined by a titration method. Initially, 1 gram of fly ash was dissolved in 50 cm3 of ethylene glycol at a temperature of 60–70° C for 30 min. The solution was then filtered by #1 filter paper. In this process, another 30 cm3 of ethylene glycol at the temperature of 60–70 °C was added to make sure that all particles were washed out. The filtrate was titrated with 0.1 N hydrochloric acid using Bromocresol green as an indicator while the residues on filter paper were collected and dried in an oven. These residues were later sampled and used for the second test of free lime determination. According to **Kaewmanee et al. (2013)**, the free lime content can be computed from:

$$\text{Free lime content (\%)} = (M_{fl} / M_{fa}) \times 100$$
$$M_{fl} = s \times n \;\; 56.07 \times 0.5 \times 10^{-3}$$

where M_{fl} is weight of free lime (g), s is the amount of HCl acid used in the titration (cm^3), n is normality of HCl acid and M_{fa} is weight of the fly ash sample (g). Blaine fineness, free lime content of the fly ashes in the first and the second free lime content determinations are tabulated in Table 1. Free lime content obtained from the first titration of fly ashes tends to increase with an increase of fineness. However, the increase is quite small and is insignificant, and does not affect properties of fly ash concrete. Second free lime content tests of all fly ashes yield the same results, which can be explained from the characteristics of the test method. Unlike ethylene glycol, the filtrate is not a pure chemical solution and may consist of a very small amount of lime. When Bromocresol is added to filtrate, the colour of solution does not immediately turn into an emerald green as in the case of pure ethylene glycol. A few drops of HCl acid is required in order to turn the colour of solution to be the same as that of the comparative solution. This results in the same non-zero value of free lime content of the second test of all fly ashes. From the results of free lime contents in Table 1, it can be concluded that a very small amount of free lime was encapsulated inside the fly ash particles.

4.4.2 STRENGTH ACTIVITY INDEX

The assessment is based on the relative strength development of cement mortars with partial cement replacement by the test material to that of a control mortar. Standard procedures exist for this test which was originally designed for coal fly ash (FA) or silica fume (SF) but have also been reported in the literature for assessing the pozzolanic activity of other materials (Paya et al. 2003; Tseng et al. 2005; Wild et al. 1997).

The SAI procedure used was based on BS 3892 (1997). Control mortar blocks were prepared by mixing 1350g sand, 450g Portland cement and 225ml water in a planetary orbital mixer. The mixing procedure used was adapted from BS EN 196-1 (2005). Cement and water were added first and mixed at low speed for 30 seconds. The mixer was stopped and sand added before mixing at low speed for 30 seconds then another 30 seconds at high speed. The mixer was then stopped and the mixture homogenised by hand, scraping mortar from the sides and bottom of bowl. Finally the mixer is set to high speed for 60 seconds. Flow tests were carried out on mortars according to EN 1015-3. Mortar pastes were then remixed for 30 seconds at low speed and cast into six 50mm cube moulds on a vibrating table.

BS 3892 (1997) requires that 30% of the cement be replaced by coal fly ash (FA). ASTM C618 (2008) requires a cement substitution of 20% by FA and ASTM C1240 (2005) a 10% substitution of cement by silica fume. Therefore test mortars were prepared in the same manner as the control mortar, except that 10%, 20% or 30% of the Portland cement was replaced with the test pozzolan. When flow test results did not agree within +/- 5mm of the control mortar, a new mixture was prepared with different water to binder ratios until test mortar flow agreed with the control mortar. After a satisfactory flow test, mortar pastes were then remixed for 30 seconds at low speed and cast into six 50mm cube moulds with the aid of a vibrating table.

Moulds were covered with glass plates to prevent water evaporation from the cube surface. All blocks were de-moulded after 24 hours and placed in a water bath at 23°C for 6 or 27 days. They were then removed from the bath, surface dried with a chamois leather cloth and tested for 7 or 28 day compressive strength (Contest Instruments Ltd model GD10-A compression machine, loading rate 300kPa/s). The strength activity index (SAI) was calculated by comparing the strength of test mortar with the average strength of control mortar blocks after 7 or 28 days.

ASTM C618 (2008) requires a SAI value above 0.75 after both 7 and 28 days for mortars with 20% cement replaced by FA. BS 3892 (1997) requires a SAI value greater than 0.80 for test mortars with 30% of the cement replaced by FA.

4.4.3 FRATTINI TEST

EN 196-5 describes the Frattini test as a standardised procedure for measuring pozzolanic activity of cement admixtures and blended cements. The test requires the use of 20g samples, containing between 9 and 18.8g of CEM-I and the remainder being the test pozzolan. Test pozzolans were sieved to 150µm, and larger material was manually ground until sufficiently fine to pass the sieve. Samples were mixed with 100ml of distilled water at 40oC. After preparation, samples were left for 8 days in a sealed plastic bottle in an oven at 40oC during which cement hydration saturated the solution with Ca(OH)2. After 8 days, samples were removed from the oven, quickly vacuum filtered through a double layer of 3μm nominal pore size filter paper (Whatman no. 542) and the flask sealed. After cooling to room temperature a 50ml aliquot of filtrate was then measured for [OH-] and [Ca2+] by chemical titration and compared to a standard lime solubility curve to determine the extent of Ca2+ removal from solution. Any removal of dissolved CaO was assumed to be due to pozzolanic reaction.

To allow a wide range of possible pozzolanic cement formulations to be tested, the % mass of test pozzolan and cement is not defined in BS 196-5 (2005). According to BS EN 197-1 (2000) between 6 and 35% of the cement mass can be replaced by pozzolanic materials in CEM II Portland pozzolana cement. For CEM IV pozzolanic cements, the cement replacement range is 11-55%. To account for this range of possible compositions, the Frattini test was carried out with cement substitutions of 10, 20 and 40% by mass for each of the test pozzolans.

The following reagents were prepared for chemical titrations to measure filtrate [OH-] and [Ca2+]. The 0.1mol.l-1 HCl and 0.03mol.l-1 EDTA solutions were standardised according to BS EN 196-5 (2005) by titrating with solutions containing 0.200g of Na2CO3 and 1.00g of CaCO3, respectively. A 50ml aliquot of filtrate from Figure 3.2 was analysed for [OH-]. Addition of 15-20 drops of methyl orange indicator turned the solution yellow and titration with the standardised 0.1mol.l-1 HCl was carried out until the colour changed to orange. [Ca2+] was measured by pH adjustment of the remaining orange titrated solution to 12.3-12.5 by addition of around 20 drops of 10% NaOH solution, followed by addition of approximately 0.1g of Patton and Reeders reagent, causing the solution to turn purple. The solution was titrated with standardised 0.03mol.l-1 EDTA solution until an intense blue colour formed.

Frattini test results simply give a positive or negative answer as to whether a blended cement or cement admixture can be considered to be pozzolanic. To allow correlation of results with other methods, it is necessary to quantify Frattini test results.

Quantification of results was achieved by considering the distance of data points from the lime solubility curve and from the zero point on the vertical axis of a given data point [OH-]. Between 35 and 90mmol/l [OH-], the theoretical maximum [CaO] concentration at 40°C can be calculated using the formula below, given in EN 196-5, to plot the lime solubility curve:

4.4.4 SODIUM HYDROXIDE SOLUBILITY

To measure the reactive Al and Si content of a material, a dissolution technique has been proposed (Arjunan et al. 1997; Fernández-Jimenez et al. 2006; Ruiz-Santaquiteria et al. 2011). By dissolving reactive material in either 1% HF or 8 M NaOH, filtering and analysing the residue, the amount of reactive Al can be determined. In this study, 1g MK was mixed with 100 ml 8 M NaOH, stirred for 20 hours at ambient temperature and filtered. Preliminary work has shown that 20 hours are enough to dissolve the reactive MK. The ash free filter paper was washed until pH neutral using deionised water and calcined at 1000 °C for 1 hour prior to cooling and measurement of the residual mass.

4.5 MINERALOGY AND MICROSTRUCTURE

4.5.1 X-RAY DIFFRACTION (XRD)

In this study, X-ray diffraction (XRD) techniques were used to assess the evolution of clay minerals after the treatments focusing on the intensity and position of the first peak of montmorillonite. XRD patterns were obtained using the powder method (total fraction) and preparing oriented aggregates (fine fraction). The oriented aggregates are usually divided in two subsamples, one of which can be subjected to ethylene–glycol (EG) vapor exposure which allows to characterise more accurately the first peak of montmorillonite. All the samples prepared were analysed with a Philips X'Pert–MPD diffractometer using an anticathode Cu Ka at 45 kV and 50 mA. A scan rate of 2°(2θ)/min was used for the powder samples over the range 2–70°(2θ) while the scan rate for oriented aggregates was 1°(2θ)/min. In this study, the identification of the mineral phases in the soils prepared was made by using the specific software Xpowder 2004 **(Martin-Ramos, 2004)**.

For the X-ray diffraction (XRD) analysis, two subsamples were prepared from each of the mixtures. The first subsample was homogenised and milled for powder analysis of total fraction. In this subsample all the soil mineral phases were recognised. The second subsample was an oriented aggregate prepared by milling the sample, dissolving it in water and inducing decarbonation and decantation of the clay fraction (fraction finer than 2 µm). The oriented aggregate was introduced in the diffractometer (XRD, PAN analytical X-Pert Pro MPD, Philips, Figure 2a) after a quartz tablet to calibrate the equipment. The oriented aggregate was subjected to 2°/minute scanning at a wavelength of 1.5405 Å between an angle (2θ) 2° to 60°.

4.5.2 FIELD EMISSION SCANNING ELECTRON MICROSCOPY (FESEM)

High-resolution field emission scanning electron microscopy (FESEM) is achieved through use of a field emission gun (FEG) in place of the more conventional tungsten hairpin or LaB6 filaments. A resolution of 0.5 nm has been achieved under ideal conditions and 5 nm is regularly achieved at 30 kV with conducting materials (Fig. 1). However, with insulating materials such as rocks this figure is closer to 50 nm. The amount of clay data which can be acquired using back-scatter or conventional secondary electron imaging techniques is limited by the resolution of such instruments. For clay mineral studies the optimal resolution is 0.5 p.m for back-scattered electron microscopy (BSEM) or 2.5 nm for conventional secondary electron microscopy (CSEM) compared with 0.5 nm for FESEM (Table 1). Good quality photomicrographs can be obtained at a magnification of x 2,000 for BSEM, x 5,000 for CSEM and x 30,000 for FESEM. In favourable circumstances (i.e. observations of electrically conducting materials rather than rock samples), photomicrographs of acceptable quality can be obtained at x 15,000 magnification for CSEM and • 50,000 for FESEM.

A field emission gun produces an electron beam with an extremely high current density, obtained by applying an intense electric field to a tungsten single crystal with a needle-shaped tip. Unlike the conventional thermal emission gun this requires ultra high vacuum (10 -1~ Torr). The FEG is often referred to as the high brightness gun because the emission source for electrons is extremely small and because the number of electrons emitted per unit area is much greater than with other types of electron guns. This, together with the much narrower range of emitted electron energies than is obtained with thermal emission guns, permits high resolution at low kV (Fig. 1). The ability to work at low kV is particularly valuable for ultra-thin clay mineral particles which would appear to be 'electron transparent' at high kV. For FESEM examination it is not always necessary to apply a conductive coating, particularly if working at very low kV, but for most clay work 10 kV is about the optimum and a conductive coating is preferred. The specimens should have either gold-palladium or platinum coatings of 30-40 nm thickness as the gold coating used in conventional SEM is too coarse and can be observed as a mosaic-like pattern on the sample surface thus obscuring morphological features. This is demonstrated by comparing the micrographs in Figs. 2 and 3. A Polaron 5000 series coater with a two minute coating time and normal resistance at 20 mA, was used to provide the gold-palladium and platinum coatings.

4.5.3 ENERGY-DISPERSIVE X-RAY SPECTROSCOPY (EDS)

EDS makes use of the X-ray spectrum emitted by a solid sample bombarded with a focused beam of electrons to obtain a localized chemical analysis. All elements from atomic number 4 (Be) to 92 (U) can be detected in principle, though not all instruments are equipped for 'light' elements (Z < 10). Qualitative analysis involves the identification of the lines in the spectrum and is fairly straightforward owing to the simplicity of X-ray spectra. Quantitative analysis (determination of the concentrations of the elements present) entails measuring line intensities for each element in the sample and for the same elements in calibration Standards of known composition.

By scanning the beam in a television-like raster and displaying the intensity of a selected X-ray line, element distribution images or 'maps' can be produced. Also, images produced by electrons collected from the sample reveal surface topography or mean atomic number differences according to the mode selected. The scanning electron microscope (SEM), which is closely related to the electron probe, is designed primarily for producing electron images, but can also be used for element mapping, and even point analysis, if an X-ray spectrometer is added. There is thus a considerable overlap in the functions of these instruments.

EXPERIMENTAL RESULTS PART I

Chapter 5

Physico-chemical characterisation of additives for soil stabilisation

Abstract

This first part of the experimental study has the characterisation of the potential additives as a main objective. As a main conclusion, there are many different materials which can be considered for potential use in soil stabilisation, including industrial by-products, natural resources and materials suitable for combinations with the so-called traditional additives. In the section of Introduction (Chapters 1 to 4), the two main reactions of the stabilisation process have been outlined: a) flocculation-agglomeration of particles; b) pozzolanic reactions. The wide range of possibilities of the soil stabilisation technique, which includes different dosages, combinations of additives, etc. depending on the specific purpose of each case, allows the design engineer to come out with the most adequate solution, whilst at the same time make the preliminary study of the additives prior to the treatment (and their predicted relationship with the soil to be treated) indispensable. At this stage of the investigation, a deep analysis of the properties of the additives becomes necessary. This chapter will evaluate the physical and chemical properties of lime, coal fly ash (class A) and two different types of biomass fly ash. The chemical composition, particle size and pozzolanic activity will be studied, trying to establish a relationship between those properties and the suitability of the additive to treat a specific soil. As a main conclusion, it is observed that additives with a very similar origin can have significantly different properties and those differences can be crucial in the performance of those materials for soil stabilisation.

5.1 INTRODUCTION

In the last decades, and specially in the recent years, the soil stabilisation as a solution to enhance the low engineering properties of residual soils, such as marls, clays, etc. has become a very common process in the construction of earthworks and embankments all over the world. Soil stabilisation let geotechnical engineers face not only the engineering problems of soils (such as swelling potential or high plasticity) but also those related with the high economical and environmental costs of the infrastructure.

Searching a more sustainable construction process, traditional binders (like lime and cement) are being progressively replaced by non-conventional additives (wastes, industrial by-products, different kind of fly ashes, etc). These additives, if proved to have binding properties, are usually selected by a wide range of criteria, one the most important being the local availability and the specific conditions of different regions, areas and countries.

Another really important feature which must be taken into account is the specific origin and properties of the soil, given the enormous influence which the presence of sulphates (gypsum), carbonates (marls) or extremely expansive phases (montmorillonite) can exert on the final outcome of the soil stabilisation. Many studies on soil stabilisation carried out in the lab only cover the first stage of the soil stabilisation, while earthworks and embankments are meant to last several decades.

In the soils with high content of calcium carbonates (like marls), slaked lime and carbonates can interact diminishing the long-term effect of the hardening primary reaction. On the other hand, some additives can contribute to formation of cementitious compounds within the soil through secondary pozzolanic reactions. Thus, a proper study of the inherent characteristics of a potential stabilisation agent is highly required, even as a complement to consistent trial-error processes for soil-additive systems in the field.

In this study, four different additives which might be locally available in a certain area are thoroughly studied as potential stabilisation agents. Two of those additives are considered to be traditional (lime and coal fly ash) and two of them belong to the so-called non-conventional additives (biomass fly ash and biomass bottom ash from olive tree residues). Chemical composition, pH, free lime, particle size and pozzolanic activity tests were carried out in this research. This work proposes the study of the properties of the additive and the analysis of their accuracy for soil stabilisation, regarding the kind of soil to be treated and the specific objectives pursued. Therefore, the present work is applied to commercial and non-commercial stabilisation agents alike.

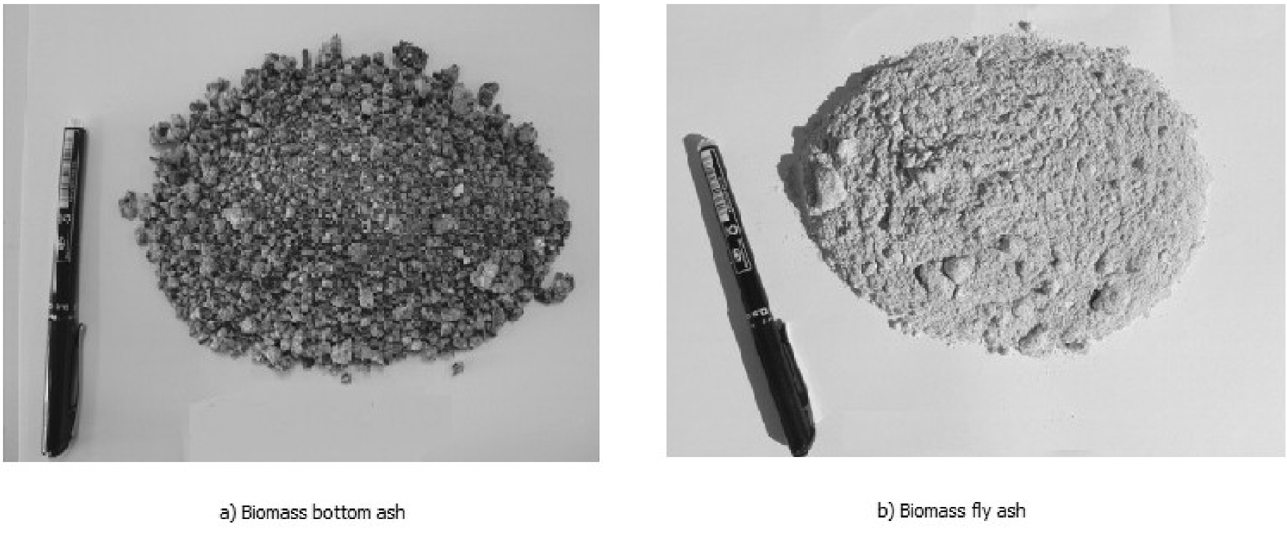
5.2 EXPERIMENTAL PROGRAM

Materials

In this study, several additives were tested: lime, coal fly ash, biomass fly ash and biomass bottom ash. The lime used in this work was commercial quicklime called CL-90-Q in Spanish Standard UNE EN 459-1. The coal fly ash used in this study came from the thermal power plant of Carbon eras (Almeria, Spain) fuelled by coal. This fly ash is commonly used as a component of some blended cements and concretes.

The biomass ash is formed by the non-flammable residues remaining after incineration. Depending on the origin of the biomass (rice husk, wood, hay, etc.) the composition of the ashes may substantially varies. In this case, the biomass ash was collected from the a power station in Linares (Spain) where olive cake residue is burnt to generate electrical energy.

Two different kind of biomass ash were collected and studied: fly ash and bottom ash (figure 1). Their available amount and economic cost is quite different. And so it is its properties and composition. The biomass fly ash is transported as a suspension in the airways of the power station and collected in silos. The biomass bottom ash is formed in a secondary process of the power generation by disposal on the ground. Hence the impurities and heterogeneity of this kind of ash which may be in contact with sand, dust and other wastes.


a) Biomass bottom ash

b) Biomass fly ash

Figure 5.1. Image of biomass bottom ash (BBA) and biomass fly ash (BFA)

Methodology

The additives studied in this research were undergone to physical and chemical characterisation. X-ray fluorescence (XRF) tests were conducted to accurately know the chemical composition of additives: percentage of oxides and elements. Carbonate content, pH and free lime tests were also carried out.

Regarding the physical characterisation of additives, sieving was carried out to study the particle size distribution of the tested samples. The pozzolanic activity of the samples were studied by the strength activity index (SAI). Finally, some pictures of scanning electron microscopy (SEM) were taken to study the form of the compounds present in the additives.

5.3 RESULTS

The results of the XRF tests are tabulated in **Table 5.1**. One of the most relevant oxides for soil stabilisation is CaO (calcium oxide). There is a high content of CaO within the lime. However, CaO content is negligible for fly ash. BFA and BBA proved to have CaO contents higher than those present in fly ash, but much lower than the lime. The free lime tests will show the relevance of the CaO content present in the tested additive.

Table 5.1

Chemical composition of tested additives

Oxides (%)	*LIME*	*BFA*	*BBA*	*COAL FLY ASH*
SiO_2	1.5	30.1	52.5	48.7
Al_2O_3	≤ 0.1	6.5	6.9	29.7
Fe_2O_3	≤ 0.1	2.8	4.9	4.0
MnO	≤ 0.1	0.5	0.1	0.0
MgO	4.0	3.4	2.6	1.7
CaO	86.0	25.8	15.0	6.2
Na_2O	≤ 0.1	0.5	1.2	0.2
K_2O	≤ 0.1	20.9	2.0	0.6
TiO_2	≤ 0.1	1.5	2.6	1.6
P_2O_5	≤ 0.1	4.2	0.6	1.0
Sum (%)	91.5	96.2	88.4	93.7
Loss on ignition (%)	4.0	18.0	16.7	4.0

Table 5.2 shows the values of free lime obtained in the test for the additives studied and its relationship with the original CaO content in the samples. The differences between different kind of biomass ashes can be appreciated. **Table 5.3** shows the results of the pH and carbonate content tests undertaken.

Table 5.2

CaO (%) and Free-Lime (%) in chemical composition of additives

Chemical composition	*LIME*	*BFA*	*BBA*	*COAL FLY ASH*
CaO (%)	86.00	25.80	15.00	6.20
Free lime (%)	42.80	2.90	0.22	0.03
Free lime (%) / CaO (%)	0.498	0.112	0.015	0.005

Regarding the physical properties of the tested additives, the result of the strength activity index (SAI) tests are shown in **Table 5.4**. The fly ash proved to have a really high activity showing high capacity enabling the pozzolanic reactions. SAI is usually applied to cement fillers. Since strength activity index value is related to the fineness of the sample, two batches of BBA were prepared: one with the original particle size distribution and one with a milled sample with particle size below 0.32mm.

Table 5.3

CaO (%) and Free-Lime (%) in chemical composition of additives

Chemical composition	*LIME*	*BFA*	*BBA*	*COAL FLY ASH*
pH	11.8	12.5	11.8	12.5
% Carbonates	-	17.20	16.80	-

Table 5.4

Activity index

	LIME	*BFA*	*BBA(*)*	*BBA(**)*	*COAL FLY ASH*
Control mortar strength (28 days)	60.55	60.55	60.55	60.55	60.55
Test pozzolan mortar strength (28 days)	46.18	23.75	33.10	37.08	50.17
Control mortar strength (90 days)	63.17	63.17	63.17	63.17	63.17
Test pozzolan mortar strength (90 days)	48.02	28.75	36.77	40.57	62.73
Activity index (%)	76.00	46.00	58.00	64.00	99.00

(*) BBA with original particle size;
(**) BBA milled to maximum particle size of 0.32mm;

The results of particle size tests by sieving showed that BFA particle size distribution is very similar to that of fly ash. However, BBA mean particle size proved to be very coarse. BBA was the only additive with a fineness higher than 30% (85.1%). Results of particle size tests are tabulated in **Table 5.5**.

Table 5.5

Particle size distribution of additives

Sieve size (mm)	*LIME*	*BFA*	*BBA*	*COAL FLY ASH*
6.3	100.0	100.0	99.0	100.0
5	99.4	100.0	98.5	100.0
4	98.4	100.0	98.2	100.0
2	96.3	100.0	80.3	100.0
1	92.4	100.0	56.6	100.0
0.800	91.4	100.0	52.0	100.0
0.630	89.5	100.0	45.1	99.9
0.500	87.8	99.9	40.3	99.8
0.400	86.1	99.3	34.5	99.7
0.250	82.0	96.1	26.9	99.5
0.125	79.9	85.4	18.9	95.5
0.080	73.1	77.8	16.2	88.7
0.063	71.9	75.7	15.8	83.3
0.050	69.9	73.1	15.0	80.3
0.040	69.7	73.0	14.9	80.1
Fineness (% > 0.045 mm)	30.2	27.0	85.1	19.8
Mean particle size (mm)	0.355	0.085	1.472	0.060

5.4 DISCUSSION

Commercial quicklime is formed by CaO, which is traditionally considered to be the most important compound to produce alteration of physical properties of expansive soils. However, there are some studies (**Eberhardt et al., 2005; Mathys et al., 2005; Yong and Ouhadi, 2007**) claiming the problems which lime can present in certain soils, like those containing carbonates. Lime adds Ca^{2+} ions to the soil, but in marls, where calcium carbonate can represent up to 65% of its mineral composition, the water is saturated in HCO_3^-, which will react with this Ca^{2+} ions and producing $Ca(HCO_3)_2$. Thus Ca^{2+} may be consumed by HCO_3^- reducing the efficiency of lime treatment before it is completed and reducing the alkaline pH needed to form stable mineral structures.

In this conditions, other oxides different from CaO can improve the reaction soil-additive. XRF tests showed that BFA and BBA contain certain amounts of Na_2O and K_2O. These oxides, which are slightly present in fly ash, as we can see in **Table 5.1**, can introduce $(OH)^-$ in the soil when mixed with water. Hence the increase in the pH value.

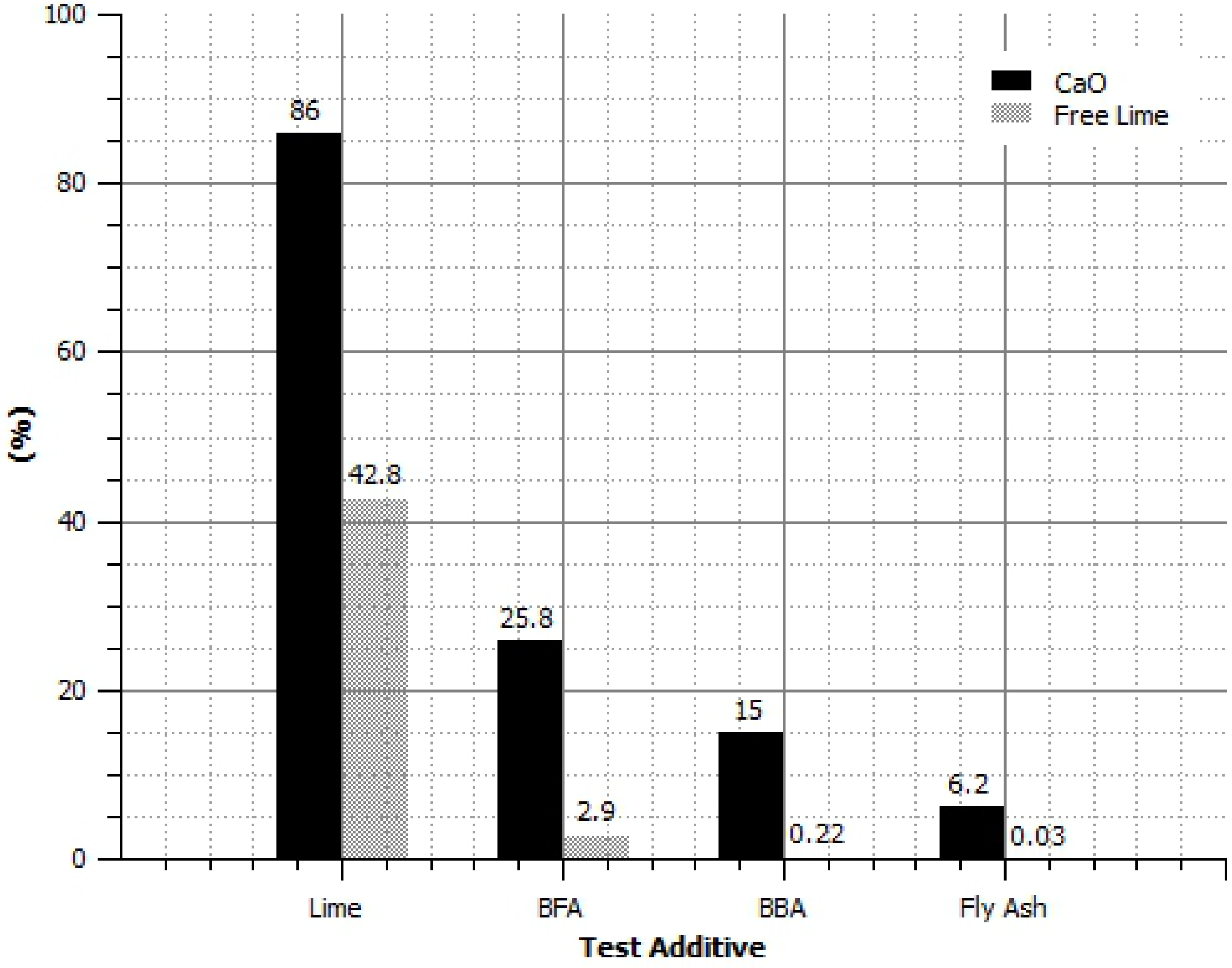


Figure 5.2. CaO in chemical composition of additives

In lime-stabilised soils the long term pozzolanic reaction can be attributed to the presence of SiO_2 and Al_2O_3 in the structure of clay minerals. XRF tests showed that BFA, BBA and fly ash contain significant amounts of silica and alumina which lead to formation of cementitious materials (CSH and CAH) when proper conditions (alkaline pH, presence of active silica and alumina) are provided.

Free lime becomes a significant feature in soil stabilisation, as lime promotes the first hardening reaction and agglomeration of particles by flocculation. In the case of development of pozzolanic reactions, the presence of an activator like cement or lime might become indispensable. Free lime in the sample can play the role of activator of the reaction. **Figure 5.2** shows the values of CaO(%) and Free lime (%) for the tested additives. Free lime in the additive is related to the amount of CaO in the chemical composition of that additive. This relationship is not a constant, but a linear relationship meaning that the higher the absolute amount of CaO in the sample, the higher the relative availability of free lime. **Figure 5.3** shows this relationship which is 99% fixed in the samples studied in this research.

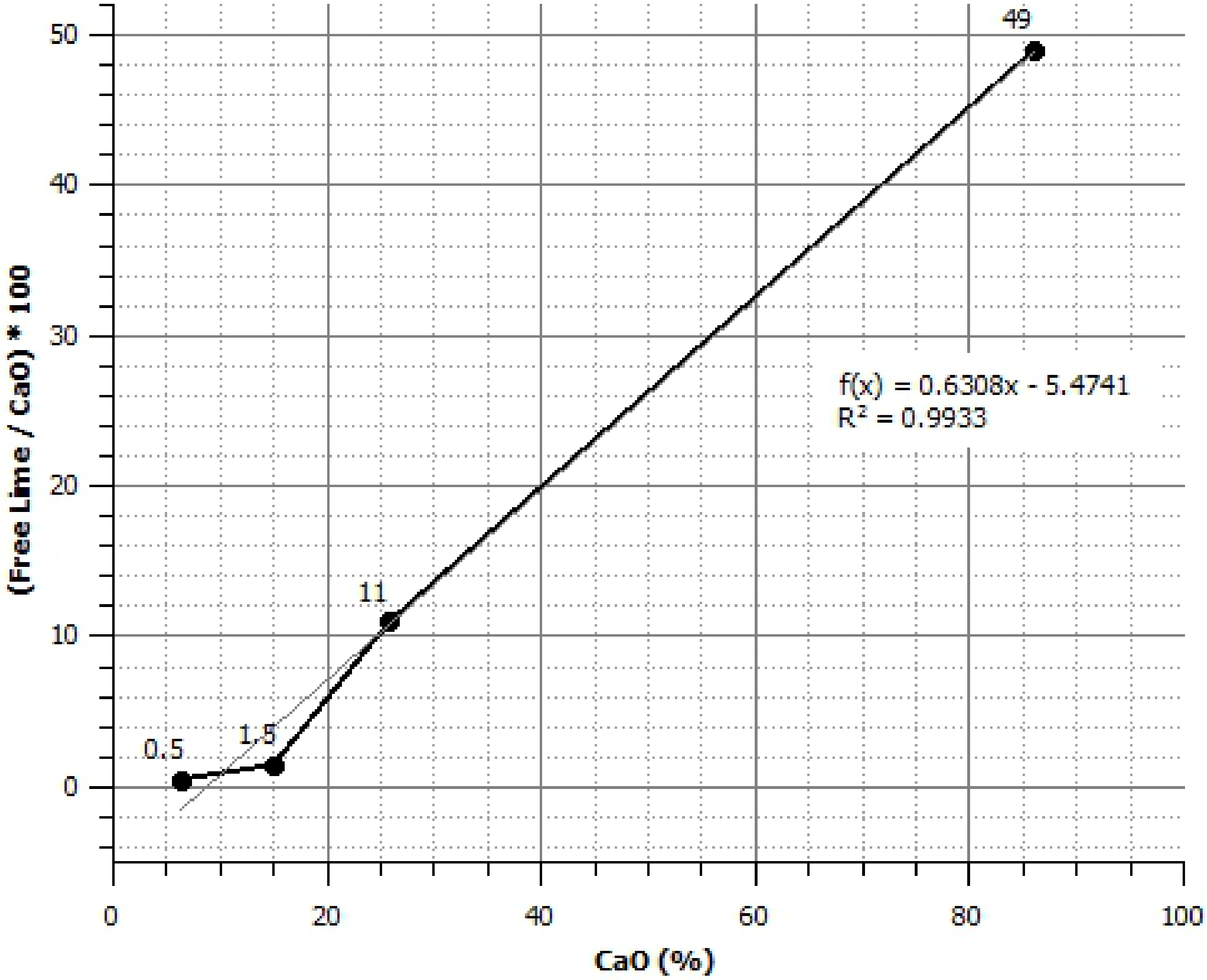


Figure 5.3. Relationship CaO Vs. Free lime

Figure 5.4 plots the particle size distribution of the sample studied in this research. The particle size of BBA is considerably coarse, probably because this ash is formed in last stages of the power generation and it is collected from the ground. As seen in **Figure 5.4** and **Table 5.5**, the particle size distribution of BFA is very similar to that of fly ash. This small particle size in the sample lets the additive fill the pores present in the poor soil reducing the void ratio. Fly ash is usually used as a filler in cements. The particle size of BBA is not suitable for the soil-additive mixture due to the fact that finer grain size distributions lead to better conditions of reaction. Lime has a fine particle size, coarser than fly ashes grain size.

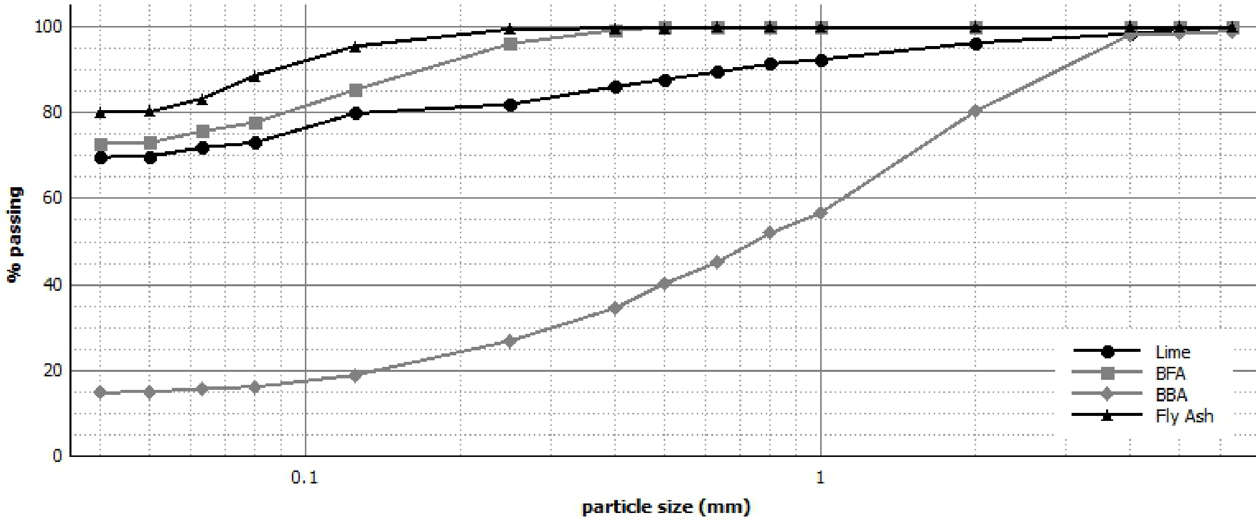


Figure 5.4. Particle size distribution of additives

In this research, the strength activity index (SAI) was studied to characterise the pozzolanic activity of different additives. The SAI test is commonly used to study the properties of fillers and pozzolans for blended cements. One of this pozzolans, fly ash, proved to have a very high pozzolanic activity, since its activity index was 99%, which means that if up to 20% of cement is replaced by this pozzolan in the preparation of a sample with the same w/s ratio, the strength loss of that sample at 90 days of curing is as low as 1%. Lime proved to have high values in SAI tests. This can be attributed to the hardening effect of the lime-water reaction more than the real consumption of portlandite in a secondary reaction. As explained above, BFA has rather the same particle size distribution than fly ash. However, the SAI was much lower for BFA. This can be attributed to the slow pozzolanic reaction of consumption of portlandite (Ca(OH)2) and formation of CSH and CAH. High performances in SAI test are usually related to the presence of important amounts of silica and alumina in the pozzolan, which explains why BBA performs better at SAI test than BFA. Comparative values of SAI tests are depicted in **Figure 5.5**. SAI value of lime is considerably high.

The pH is a very relevant feature in soil stabilisation. Alkaline environments allow the destruction of expansive phases of clay mineral such as montmorillonite, and its transformation in other more stable mineral structures, like illite or interstratified minerals. Alkaline pH values are also favourable to the development of secondary pozzolanic reactions which, as explained above, may result in cementitious compounds like CSH and CAH which enhance the strength and mechanical behaviour of the soil. All the samples tested in this study proved to have pH values above 11, leading to alkaline environments in the soil. The maintenance of that alkaline environment is crucial for the ongoing of the stabilisation process and the long-term improvement of soil properties.

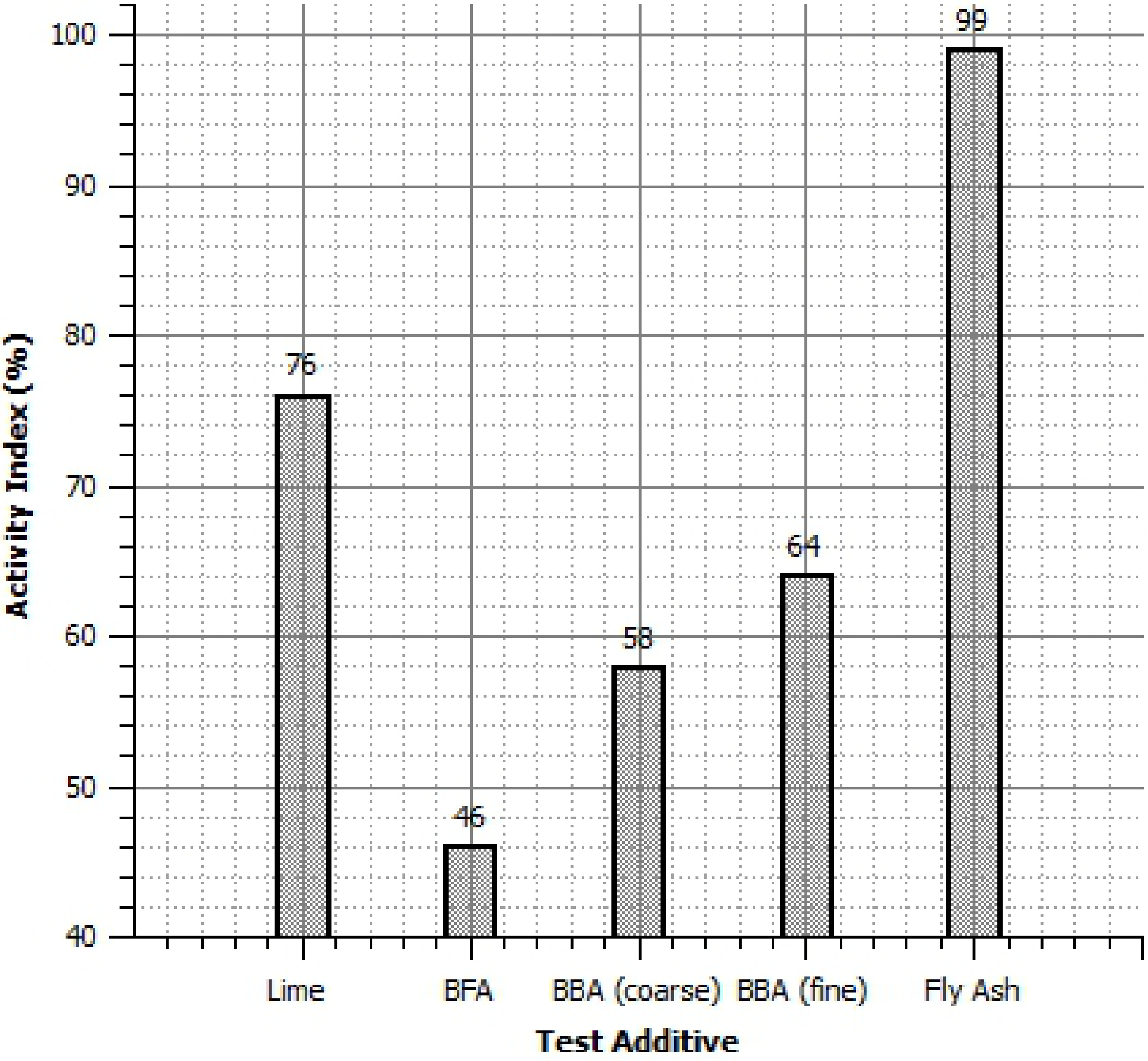


Figure 5.5. Strength Activity Index results

The carbonate content test carried out for the BFA and BBA proved that those ashes, after a short period of time from their formation, reached a carbonate content between 17.2% (BFA) and 16.80% (BBA). This non-negligible formation of carbonates might be due to the reactivity of the ashes when the are in contact with CO2 and available moisture. So it is very important to properly store the ashes derived from industrial processes. Till the moment of soil mixture, those ashes must be isolated from environmental conditions to reach their maximum efficiency in soil stabilisation.

As mentioned above, BFA and BBA proved to have significant amounts of silica (SiO_2) in their compositions. The amount of alumina (Al_2O_3) found out in the XRF tests is higher than that of the lime, but it is much lower than the alumina present in the fly ash. The silica in the chemical composition of BBA was higher than silica in BFA. This may be due to the presence of traces of sand and dust in the composition of BBA. The amorphous or active silica is partially responsible for the pozzolanic properties of the additives. **Figure 5.6** shows pictures taken through the scanning electron microscopy (SEM). In those figures the shape of the active silica (SiO_2) found in the biomass fly ash can be appreciated.

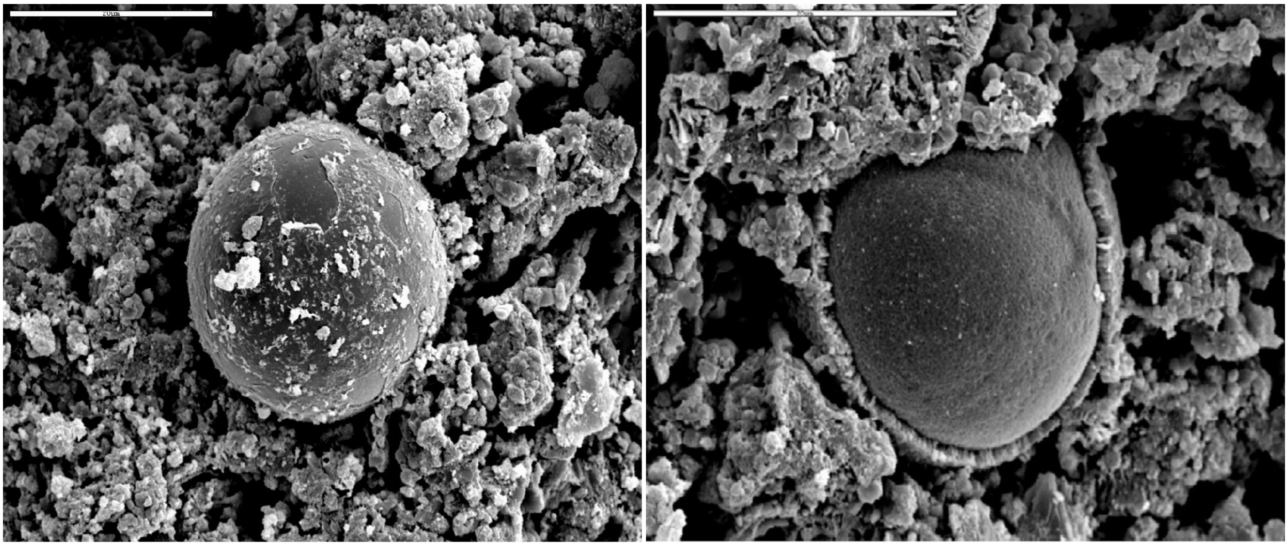


Figure 5.6. Active silica formations in BFA composition

Finally, as shown in this study, the different kind of biomass ash can have different physical properties and chemical compositions. This may be due to the process in which those ashes are formed and collected. On the other hand, biomass ashes come from the incineration of biomass which involves a wide range of materials: hay, rice husk, olive residues, wood, etc. Thus it is very important to characterise the sample of the additive which is meant to be used in soil stabilisation. To some extent, the accurate election of the additive depending on the soil to be treated becomes indispensable.

5.5 CONCLUSIONS

From the study of the different properties of the tested additives, the following concluding remarks can be drawn:

- The study of the chemical and physical properties of the additives available is indispensable to properly select the most suitable one, always bearing in mind the specific kind of soil to be treated.

- Additives with similar origin and manufacturing process can have significantly different characteristics which will definitely affect the final performance of the additive in soil stabilisation. This is the case of biomass ash, where the difference between biomass fly ash and biomass bottom ash are really appreciable. It is also important to study the kind of biomass to be incinerated since it will influence the chemical composition of ashes.

- Strength activity index test is useful to better understand the capacity of the additives to enhance long-term pozzolanic reactions. However, the oxides present in the chemical composition can have a greater influence in the performance of additives for soil stabilisation.

- The particle size distribution can present noticeable differences among different kind of additives. Since the particle size distribution has an enormous influence on the reactivity of the additive in the soil-agent mixture, it is very important to properly characterise the grain size of the samples and determine in which cases the sample of additive should be modified.

- The biomass fly ash (BFA) proved to have interesting properties for soil stabilisation. There are significant amounts of CaO and free lime within its chemical composition. This free lime will produce the first hardening reaction in the soil. The presence of active silica and alumina in BFA let this additive solve the problems of traditional lime treatments in soil with carbonates. Finally, BFA introduces Na+ and K+ for the cation exchange of clay minerals and (OH)- which can maintain the alkaline environment needed.

Chapter 6

Pozzolanic activity of additives and its influence on the soil stabilisation process

Abstract

In the literature review carried out in this study, the combined effect of flocculation and pozzolanic reactions has been highlighted. Furthermore, in the previous chapter, the importance of the study of an additive prior to its use as a soil stabilisation agent has been stated and a feel for the importance of the pozzolanic activity of each additive has been outlined. At this stage of the investigation, an experimental program is developed which is intended to broaden the range of additives to be used, with a focus on the pozzolanic activity and the presence of free lime (since both parameters together are responsible for the most significant effects of the soil stabilisation). In this chapter, a definition of pozzolans and pozzolanic activity is offered. The literature review let us select the most recognised methods to measure the pozzolanic activity. Then, a wide range of additives which can be found in the south of Spain will be selected. The origin and source of those additives will be explained prior to investigate their chemical properties and pozzolanic activity. Particle size, strength activity index, Frattini, NaOH solubility, SEM and XRD tests will determine the properties of those additives. Some well-known pozzolanic materials, such as clay brick waste, will be studied as a control. The results obtained for each additive will be compared and the main conclusions extracted from those results will be included in the last section of this chapter.

6.1 INTRODUCTION

The following materials were selected to study their chemical composition, content of lime and pozzolanic activity, and hence their potential use in soil stabilisation:

- DA: dolomite ash (also known as dolomitic lime), it was obtained from a marble quarry in Macael, Almería (Southern Spain).

- BFA: Biomass fly ash from the olive oil industry, obtained from the filters of the thermal power station of Valoriza, S.A., located in Linares, Jaén (Southern Spain).

- Paval: it is an aluminium oxide obtained from recycled salt slags, obtained from the company Befesa, S.A. Valladolid (Central Spain).

- RHA: Rice husk ash produced in the factory of Herba Ricemills, S.A., Sevilla (Southern Spain).

- PSA: biomass ash obtained from the calcination of prune waste for the production of paper (kraft process), ENCE, S.A. in Huelva (Southern Spain).
- CBW: clay brick wastes, obtained during the production of bricks for the construction industry in the factory of Ladrillos Bailen, S.L., located in Bailén, Jaén (Southern Spain).

As it can be seen, among the tested additives there were different types of biomass ash, such as the biomass fly ash obtained from the thermal power station, the rice husk ash or the paper ash. Although all of them belong to the group of biomass ash and are obtained from a material considered to be biomass, for simplification purposes, in this study only the biomass obtained from the thermal power station will be called biomass fly ash (BFA), whilst the other two materials will be called rice husk ash (RHA) and paper ash (PSA), respectively.

The characteristics and origin of the test additives are described below.

6.1.1 DOLOMITE ASH (DA)

Marble is around 90% formed of calcite ($CaCO_3$). Dolomite ash or dolomitic lime was obtained from a marble quarry in Macael (Southern Spain). In this area, there are outcrops of marble ($CaCO_3$) and dolomite ($CaMgCO_3$) in alternative layers. The layers of dolomite are removed to continue the extraction of marble. However, the large amounts of dolomite are disposed off in a landfill. In the seek for a new use of this dolomite, a sample of dolomite was treated to obtain a dust mainly formed of calcium oxide and magnesium oxide.

The sample of dolomite was first ground to a particle size of 1-2 cm. In samples of 1 kg, the dolomite was milled and sieved to a particle size of 0.100mm. The dust was then calcinated with a temperature raise rate of 5°C/min up to 950°C. This temperature was maintained during 1 hour. The sample was then allowed to cool down. The final product is a white very fine-grained material, mainly formed of magnesium and calcium oxides. The process described above includes a certain amount of carbon emissions and the waste cannot be used without being processed (calcination). However, the resulting product has an adequate chemical composition and the original raw material is very cost-effective which make the use of this DA worth being studied.

6.1.2 BIOMASS FLY ASH FROM THE OLIVE OIL INDUSTRY (BFA)

Biomass fly ash was obtained from the thermal power station of Valoriza, S.A. in Linares (Southern Spain) **(Figure 6.1)**. In this power station, olive mill wastewater, a residue of the olive oil industry (which is a major industry in the area), is burnt in to produce electrical energy. The residue is first dried up and the remaining oil is removed. The biomass is then stored along with forest and pruning wastes. Both lots of biomass are introduced in the 20-metre high boiler with a variable temperature ranging from 500°C at the bottom to 750°C at the top of the boiler.

Figure 6.1. Pictures of the thermal power station in Linares (Southern Spain)

The additive used in this study was biomass fly ash which is trapped by the filters and collected in silos. There is another type of biomass ash produced in this process which is the bottom ash, collected at the bottom of the boiler after the incineration. As explained in the previous chapter, this bottom ash contains more impurities and it has slightly different physical and chemical properties.

6.1.3 ALUMINIUM RESIDUE (PAVAL)

Paval is an aluminium oxide obtained from recycled salt slags. This residue is a coarse dust, with a mean particle size of 0.250mm. Its colour is grey due to the carbon content. Its melting point is quite high. The paval used in this study was obtained from the company Befesa, S.A. Valladolid (Central Spain).

In this factory, commercial aluminium is made using bauxite as a raw material. Bauxite is formed of different minerals such as gibbsite ($Al(OH)_3$), bohemite ($Al(OH)$) and diaspore ($HAlO_2$). Throughout the Bayer process **(Figure 6.2)**, principal industrial means of refining bauxite, bauxite is transformed into pure alumina (Al_2O_3). Once the alumina is obtained, the aluminium metal is produced via electrolysis. In this process, a slag is produced due to the handling of the liquid metal. The aluminium content of this slag can be greater than 30%.

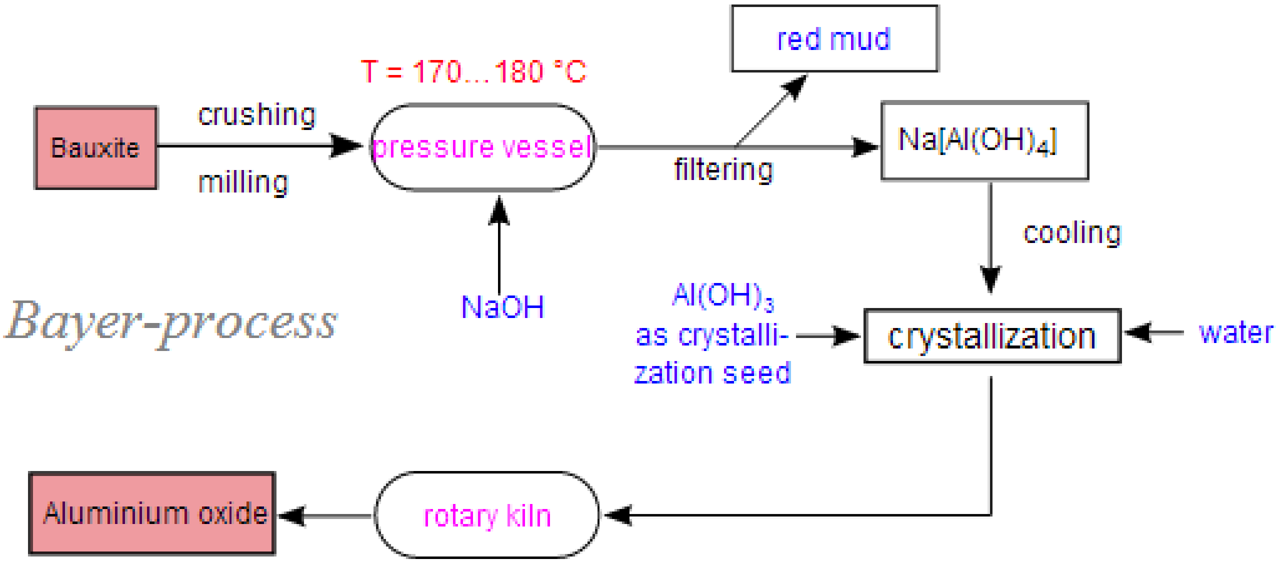


Figure 6.2. Industrial process of aluminium metal production

6.1.4 RICE HUSK ASH (RHA)

The rice husk ash used in this study was obtained in the factory of Herba Ricemills, S.A., in Sevilla, Southern Spain. In this factory, food products are produced, specially those made of rice. To run the factory, a thermal power plant was installed to burn waste of the industrial activity, such as rice husk. The boiler has a fixed bottom and it receives a continuous input of biomass which is burnt at a temperature ranging from 500°C and 750°C.

The rice husk ash **(Figure 6.3)** is a fly ash which is collected in the airways of the described system.

Figure 6.3. Rice husk ash

6.1.5 BIOMASS FLY ASH FROM PULP AND PAPER INDUSTRY (PSA)

In this study, a paper ash from the paper factory of Ence, S.A. (Huelva, Southern Spain) was used. It was obtained from the biomass used to obtain the paper (eucalyptus, pruning and forestal waste). The waste of the biomass remaining after the kraft process is incinerated and the energy generated with this incineration is used in the industrial process of the paper making. was not obtained from the paper sludge ash but from the biomass used to obtain the paper kraft (eucalyptus, pruning and forestal waste). The boiler in the factory of Hence, S.A. in Huelva has a fluidised bed to produce the necessary heat to burn the biomass. The fluidised bed is made of sand at a temperature of 850°C. When this system is used, there is a jet of air travelling from the bottom of the boiler to the top of the boiler. The particles of biomass introduced in the boiler are instantly burnt and the remaining material is caught in the air stream towards the exit and the filtering system. As a result, a very fine-grained fly ash is collected in the filters in the form of a dust **(Figure 6.4)**.

Figure 6.4. Renewable energy power station and paper factory, Ence, S.A. (Huelva)

6.1.6 CLAY BRICKS WASTE (CBW)

The clay brick waste or clay brick dust is a well-known pozzolan obtained after milling ceramic bricks produced for the construction industry. It was used in this study as a control. The comparison of the results obtained for the rest of additives tested and the results obtained for the CBW in terms of pozzolanic activity (measured throughout direct and indirect methods) will give information about the accuracy of the methods and the potential of the tested additives.

The CBW used in this study was obtained in the factory of Ladrillos Bailen, S.L. in Bailen, Southern Spain (**Figure 6.5**).

Figure 6.5. Production of clay bricks at Ladrillos Bailen, S.L. (Bailén, Spain)

6.2 METHODOLOGY AND EXPERIMENTAL PROGRAM

In this study, the pozzolanic activity of the additives described below was investigated. A wide range of test methods for assessing pozzolanic activity have been reported in the literature. These can be categorised as either direct or indirect methods. Direct methods monitor the presence of $Ca(OH)_2$ and its subsequent reduction in abundance with time as the pozzolanic reaction proceeds, using analytical methods such as X-ray diffraction (XRD), thermo-gravimetric analysis (TGA) or classical chemical titration (**Donatello et al., 2010**).

The Frattini test is a commonly used direct method that involves chemical titration to determine the dissolved Ca^{2+} and OH^- concentrations in a solution containing CEM-I and the test pozzolan. This method has been used to measure the pozzolanic activity of metakaolin (**Talero, 2005**), catalytic cracking residues (**Paya et al., 2001**), crushed bricks **(Wild et al., 1997)** and fly ash **(Rahhal and Talero, 2004)**.

Indirect test methods measure a physical property of a test sample that indicates the extent of pozzolanic activity. This may involve measurement of properties such as compressive strength, electrical conductivity **(Frias et al., 2005; McCarter and Tran, 1996; Paya et al., 2001)** or heat evolution by conduction calorimetry **(Mostafa and Brown, 2005)**. Compressive strength test methods have been used to assess the pozzolanic activity of catalytic cracking residues **(Tseng et al., 2005)**, coal bottom ash [16], glass powders **(Shi et al., 2005)**, crushed bricks [5], silica fume **(Mostafa et al., 2001; Agarwal, 2006)** and sewage sludge ash **(Monzo et al., 1999; Pan et al., 2003)**. Results from an indirect pozzolanic activity test are often corroborated using direct tests to confirm that pozzolanic reactions are occurring **(Tseng et al., 2005; Shi et al., 1999)**.

For this study, the Frattini test was carried out on samples of the test additives (BFA, DA, CBW, RHA, PSA, PAVAL). Sand was used as a control. The Frattini tests is described in the standard **BS EN 196-5 (2005)**. To assess the reactivity of the additives, another direct method was used: NaOH solubility test. This dissolution technique was proposed by several authors **(Arjunan et al., 1997; Fernández-Jimenez et al., 2006; Ruiz-Santaquiteria et al., 2011)** to measure the reactive Al and Si content of a material,. Hence, the combined results of Frattini and NaOH solubility tests show a qualitative potential and intensity of the pozzolanic activity. The results of these tests obtained for different additives were compared.

In addition to Frattini and solubility tests, samples of mortar were created with the test additives and cement in similar proportions to those of Frattini test, to ensure that the results of both techniques were comparable **(Donatello et al., 2010)**.

The first batch of mortars was prepared following the standard **BS 3892 (1997)** for the strength activity index test (SAI). This means that there was a control sample of cement (phase solid: 100% cement). An additional control sample of sand was prepared (phase solid in the mortar: 80% cement + 20% sand). The rest of the samples were prepared with a solid phase formed of cement (80%) and test pozzolan (20%). For each additive to be tested (BFA, DA, CBW, RHA, PSA, PAVAL), at least three specimens were prepared, so the value of the SAI was obtained as an average of three results. The sand used was a common sand mainly formed of crystalline silica (SiO_2). The cements used was a sample of Portland cement classified as CEM-I.

The flowability and setting time of the samples of mortar were recorded to ensure all of the samples were prepared with the same conditions and similar water/solid ratio. These tests were carried out following the standard **BS EN 1015-3 (1999)**.

Prior to the pozzolanic activity tests, the test pozzolans were subjected to a physico-chemical characterisation: X-ray fluorescence (XRF) for the chemical composition, particle size (using particle size laser diffraction), density by picnometer and initial moisture content.

The procedures of the tests used in this experimental program (Frattini, SAI, solubility, setting, flow table, XRD, SEM, particle size, moisture content, density) are described in Chapter 4.

Figure 6.6 shows a picture of the tested additives stored in sealed bottles in the lab during the development of this investigation.

Figure 6.6 Lab picture of the test pozzolans stored in a sealed plastic bottles

6.3 RESULTS AND DISCUSSION (I): CHARACTERISATION OF ADDITIVES

6.3.1 MOISTURE CONTENT

In this section, the physico-chemical properties of the selected additives will be studied. **Table 6.1** shows the initial moisture content of the additives. The moisture content depends on several parameters such as the production process of the pozzolans and the storage conditions. The initial moisture content affects the results of the subsequent tests, specially those in which the sample of additive is to be mixed with cement to create a mortar. Hence, all the samples were dried in an oven at 110°C during 24 hours before preparing the mortar mixtures for SAI or the Frattini samples. As it can be seen, the initial moisture content of paval is quite high, probably due to its origin as a liquid slag obtained from a liquid metal.

Table 6.1

Moisture content of the additives

Sample	*Moisture content (%)*
Dolomite ash (DA)	0.00
Biomass fly ash (BFA)	4.85
Clay brick wastes (CBW)	0.90
Paper ash (PSA)	2.30
Rice husk ash (RHA)	2.50
Paval	16.00

6.3.2 DENSITY

The density of the additives was studied by means of picnometry. The results are shown in **Table 6.2**. The test pozzolans had very different apparent densities. However,

Table 6.2

Density of additives measured by picnometry

sample	*mass*	*ref*	*cell*	*volume (cm^3)*	*density (gr/cm^3)*	*Mean density (gr/cm^3)*
SAND	1.1244	17.179	5.388	0.385	2.923	2.854
	1.1244	17.141	5.382	0.404	2.785	
BFA	1.0052	16.985	5.328	0.387	2.595	2.533
	1.0052	17.224	5.409	0.407	2.471	
DA	1.0648	16.894	5.294	0.369	2.882	2.798
	1.0648	16.894	5.301	0.392	2.713	
CBW	1.0362	17.078	5.341	0.335	3.096	2.964
	1.0362	17.207	5.391	0.366	2.831	
RHA	0.9791	16.943	5.315	0.388	2.524	2.379
	0.9791	17.053	5.365	0.438	2.235	
PSA	0.9176	16.883	5.286	0.354	2.589	2.623
	0.9176	16.911	5.292	0.345	2.657	
PAVAL	1.1047	17.270	5.436	0.447	2.472	2.381
	1.1047	17.260	5.444	0.482	2.290	

as it can be observed in the previous table, the density of the solid particles was quite similar for all the pozzolans. Rice husk ash (RHA) and Paval had the same density, despite the fact that RHA was presented the form of pulverised ash whilst paval was quite coarse. DA and CBW had values of density similar to those of silica and the biomass from paper (PSA) and the biomass from olive oil industry (BFA) had slightly lower density.

6.3.3 CHEMICAL COMPOSITION

The chemical composition of the additives was studied by X-ray fluorescence (XRF). This technique allows to obtain the chemical composition of the materials studied, including a quantification of the different oxides and elements present. The loss on ignition (%) was also obtained. The chemical composition of the samples is one of the most important parameters of the test pozzolan, especially if those pozzolans are being studied for their use in soil stabilisation. The amount of silica (SiO_2) and alumina (Al_2O_3) is a very significant feature, because those compounds react with the portlandite ($Ca(OH)_2$) formed after hydration of calcium oxide (CaO) to produce cementitious gels hence increasing the strength of the mortar and/or the soil. The results obtained for the tested pozzolans are shown in **Table 6.3**.

Table 6.3

Chemical composition of the tested additives by means of XRF (X-ray fluorescence)

Oxides (%)	*SAND*	*DA*	*BFA*	*PSA*	*CBW*	*PAVAL*	*RHA*
SiO_2	98.014	0.840	7.415	2.821	50.868	9.120	*> 90*
Al_2O_3	0.624	0.358	1.781	1.096	15.048	60.012	-
Fe_2O_3	0.413	0.541	1.734	0.67	5.283	1.001	-
MnO	0.000	0.050	0.046	2.3	0.045	0.000	-
MgO	0.082	25.262	1.899	11.731	2.723	7.153	-
CaO	0.046	54.212	12.982	63.983	14.888	2.462	-
Na_2O	0.037	0.000	0.306	7.136	0.559	0.000	-
K_2O	0.040	0.047	44.394	0.870	3.630	0.000	-
TiO_2	0.154	0.020	0.159	0.048	0.784	0.315	-
P_2O_5	0.013	0.000	2.568	1.350	0.155	0.000	-
Loss on ignition (%)							
LOI	0.45	18.6	15.9	4.39	3.62	7.00	4.00

The results of the XRF tests show that the chemical composition of paval and cbw includes great amounts of silica and alumina, which is indicative of a potential pozzolanic activity. The chemical composition of sand and rice husk ash is very similar. However, this materials have very different sources and they behave in completely different ways. This is due to the difference in the mineral structure of both materials. The silicon oxide in the sand has a very organised crystalline structure. However, the silica present in the rice husk ash is amorphous, which gives this material a high potential for pozzolanic reactivity.

PSA and BFA are different types of biomass fly ash with a considerable content of calcium oxide. Apparently, the amount of calcium oxide in their composition is quite similar. However, it can be stated that the calcium oxide present in PSA is not in the form of free lime, because several experiments showed that PSA was not reactive with water and mortars of PSA did not harden at all unless cement was incorporated to the mixture. To the contrary, BFA was very reactive with water and air, and a failure of the storage conditions (exposure to open air) promoted a quick hardening of the pozzolan, which can be attributed to the carbonation of the calcium oxide present in the form of free lime. The presence of free lime, as explained in previous chapters, is very important for the soil stabilisation because the cementation of the soil requires the presence of lime. However, the addition of non-reactive fly ash could also affect the behaviour of the treated soil due to alterations of the alkalinity of the environment or modifications of the particle size.

6.3.4 PARTICLE SIZE

The particle size of the tested materials was studied using laser diffraction. This test is very accurate and it is especially indicated for particle size distributions below 1000μm. To avoid the alteration of results due to the agglomeration of particles (which is usual in this type of pulverised pozzolans and also in fine-grained soils), a sonication was activated in the moment of the test, applying a soft ultrasound wave to break the weakest bonds between particles (those due to weak agglomeration). The same sonication was applied to all the pozzolans tested to make the results comparable. The results of this particle size test are displayed in **Figure 6.7**. Mean values are shown in **Figure 6.8**.

As it can be observed, all the pozzolans had a very fine particle size distribution. The fly ashes, such as PSA, BFA and RHA were obtained in the filters of the ventilation systems, hence the fine particle size. In the case of CBW, the residue obtained from clay bricks was milled to a very fine particle size. Paval is a slag from the aluminium industry. It was provided in a particle size which was considerably higher than that of the rest of materials studied. The solid particles of paval were significantly hard, making it difficult to be milled. The particle size of the additives has a strong influence on the overall performance of a ground improvement treatment, as stated in the studies by

Ahmed et al. (2011) and **Ahmed and Ugai (2011)**. On the other hand, the test pozzolans were used to prepare blended cements in this study. According to the results shown in this section, the particle size distribution of the test pozzolans were within a similar range and hence the results of strength activity index or reactivity are comparable. To explain the performance of the test pozzolans, the particle size effects of the particle size distribution will be taken into account.

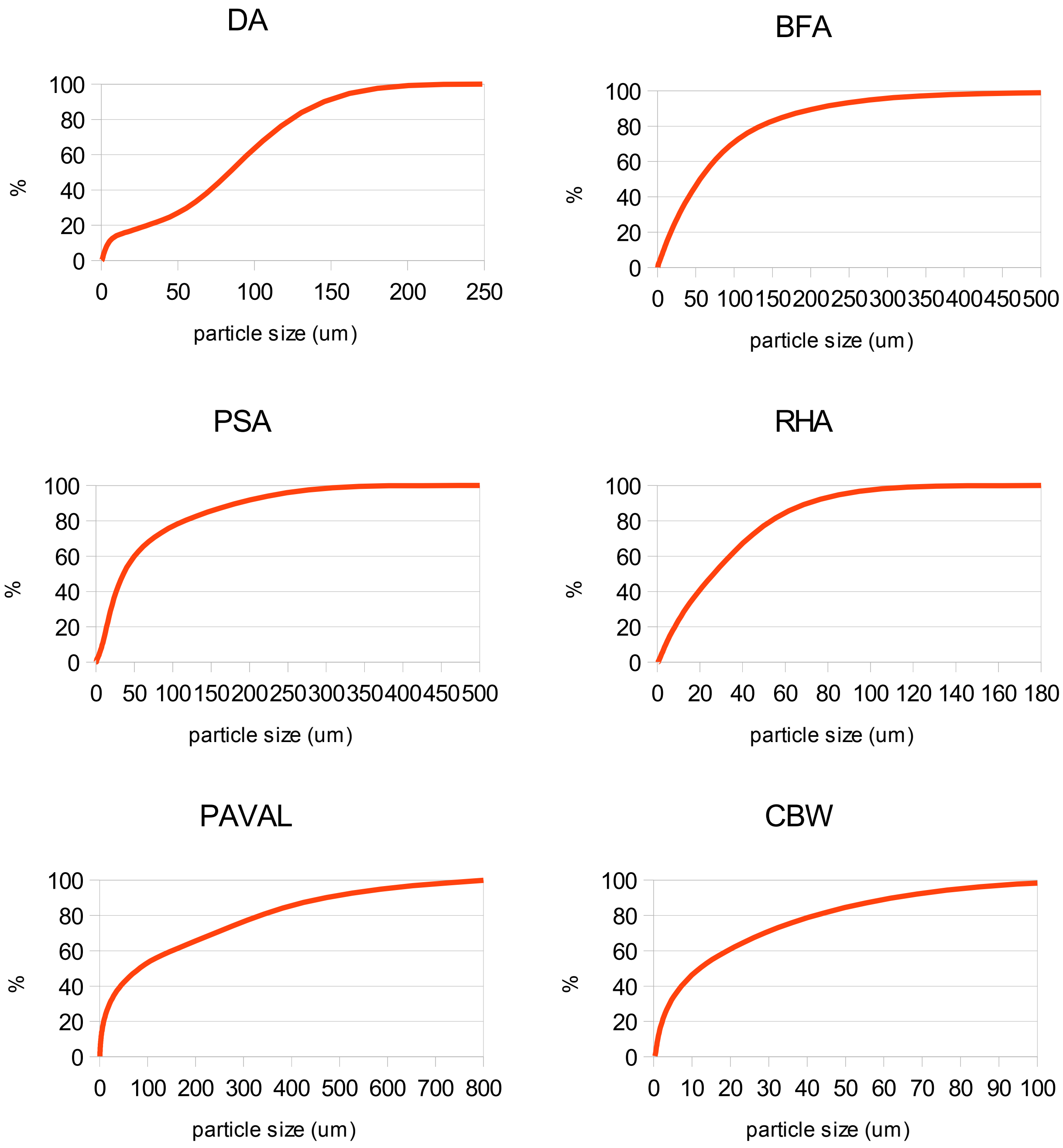


Figure 6.7 Particle size distribution of DA, BFA, PSA, RHA, CBW and Paval

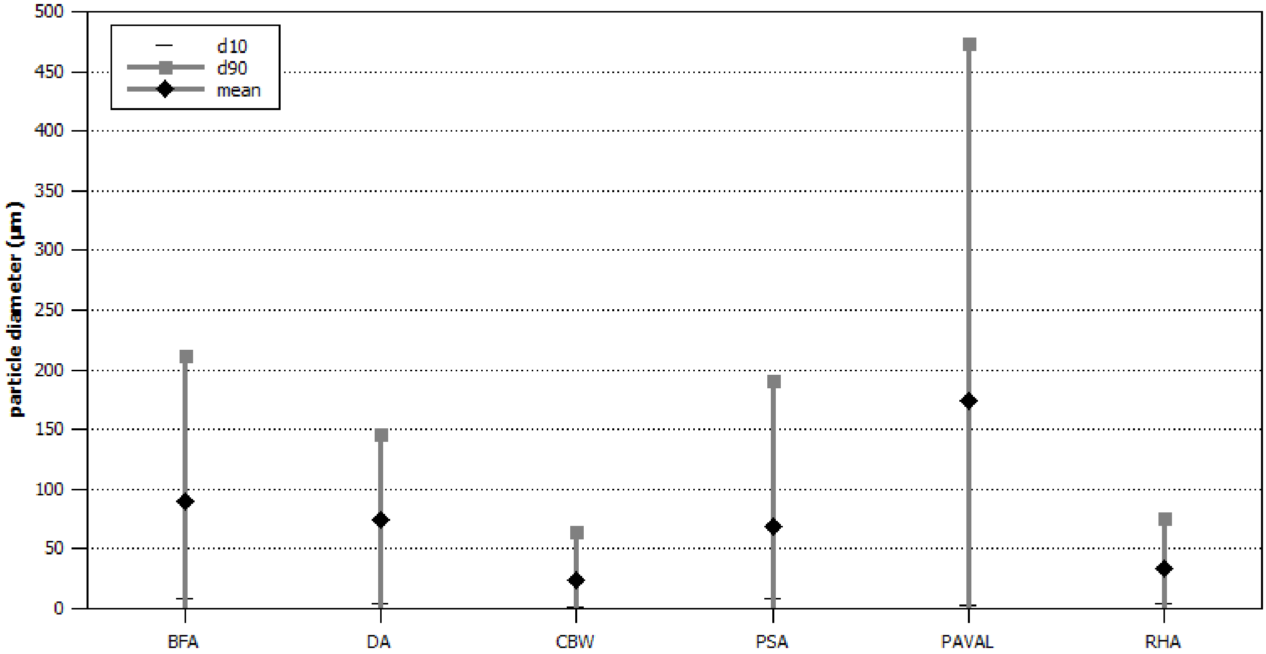


Figure 6.8 Particle size mean values of DA, BFA, PSA, RHA, CBW and Paval

6.3.5 SCANNING ELECTRON MICROSCOPE (SEM)

Scanning electron microscope (SEM) pictures of the additives were obtained. They are included below (**Figures 6.9a, 6.9b, 6.9c**).

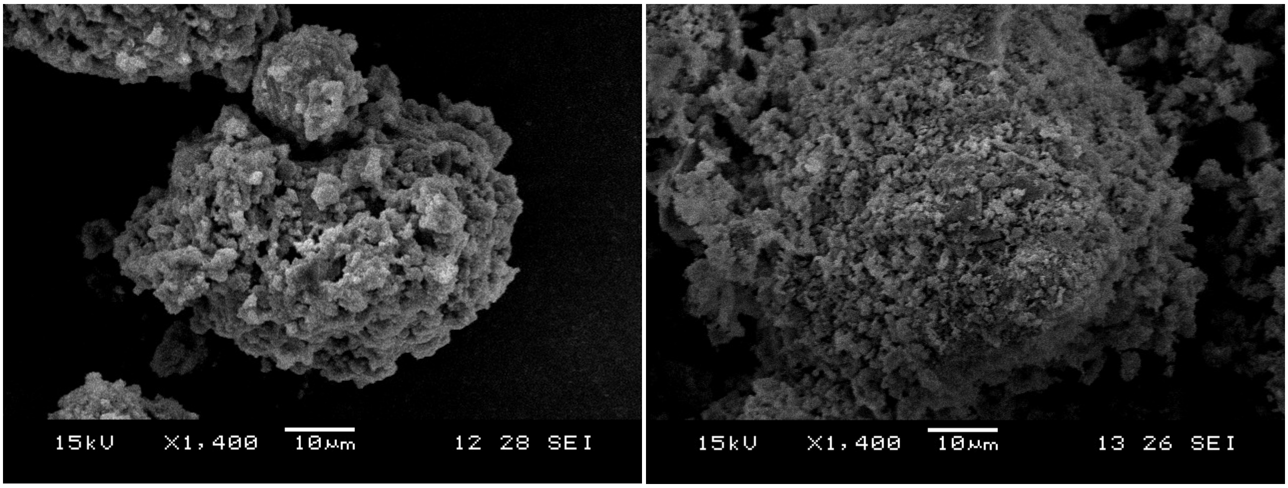


Figure 6.9a. SEM pictures of BFA and CBW

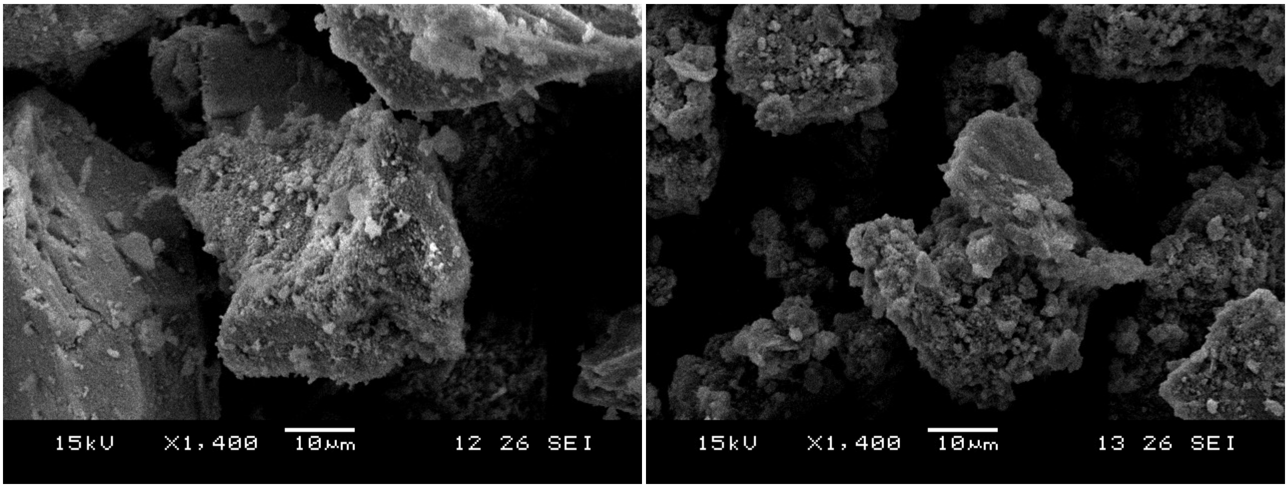


Figure 6.9b. SEM pictures of DA and Paval

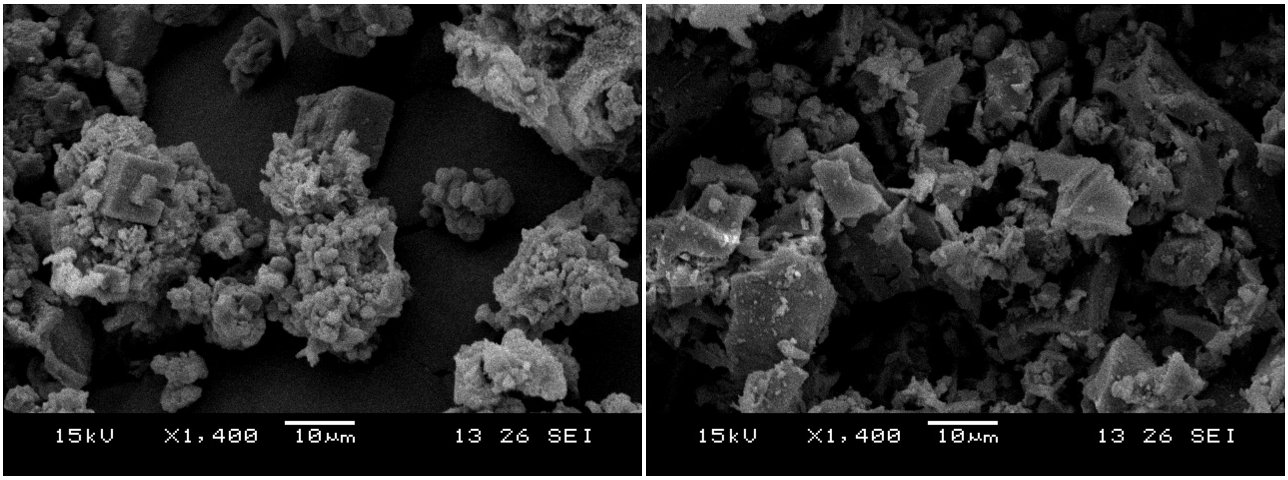


Figure 6.9c. SEM pictures of PSA and RHA

The chemical composition of the rice husk ash is very similar to the composition of the silica sand: around 90% of silica (silicon dioxide, SiO_2). However, potential for pozzolanic activity is usually attributed to rice husk ash (Viet-Thien-An Van et al., 2011), despite the fact that silica sand is inert, in terms of reactivity with water and cement. The different behaviour of RHA and sand is based on the amorphous form of the mineral structure in RHA. The SEM pictures presented in **Figure 6.9c** seem to show a certain crystalline structure of the RHA which must be attributed to the incineration process in the factories and which can affect its final performance as a pozzolan. The resolution of the SEM pictures used in this study was not high enough as to observe the presence of active silica in the composition of the test pozzolans obtained from biomass (BFA, PSA). PSA also presented evidence of crystalline formations.

6.4 RESULTS AND DISCUSSION (II): POZZOLANIC ACTIVITY BY DIRECT METHODS

The pozzolanic activity can be measured using a wide range of test methods. In the literature, there is an accepted categorisation of these methods in either direct or indirect **(Donatello et al., 2010)**. Among the most commonly used direct methods, we can find Frattini test and saturated lime method. The most widespread indirect method to evaluate the pozzolanic activity is the strength activity index (SAI).

6.4.1 FRATTINI TEST

The Frattini test is a commonly used direct method that involved chemical titration to determine the dissolved Ca^{2+} and OH^- concentrations in a solution of cement (CEM-I) and test pozzolan **(Donatello et al., 2010)**. In the existing literature, there are examples on the use of Frattini test for the evaluation of the pozzolanic activity of ISSA **(Donatello et al., 2010)**, metakaolin **(Talero, 2005; Donatello et al., 2010)**, catalytic cracking residues **(Paya et al., 2001)**, crushed bricks **(Wild et al., 1997)** and fly ash **(Rahhal and Talero, 2004)**.

In this study, the Frattini test (15 days of curing time) was used to evaluate the pozzolanic activity of the test additives: BFA, DA, RHA, PAVAL, PSA and CBW. The results of the titration before and after the curing time are plotted in the solubility curve provided by the BS EN 196-5 **(BSI, 2005)**. The comparison between the calculated amount of calcium oxide (CaO) ($mmol.l^{-1}$) and the theoretical maximum amount of CaO ($mmol.l^{-1}$) allows for the calculation of the percent reduction.

The Frattini test results (reduction of calcium oxide) obtained in this study are presented in **Figure 6.10**. The results are the average of two measurement.

Sand is used as a control, given the null potential for pozzolanic activity. It must be notices that the slight deviation in the results obtained for sand (<10%) can be attributed to slight errors in the measurement process (titration, plot of solubility curve...). The solution of 100% cement (CEM-I) is also used as a control. The values of the titration (OH^- and Ca^{2+}) for the solution of cement are located just over the solubility curve in the plot, and hence the reduction is zero (this is a control feature). According to **Figure 6.10**, the pozzolanic activity of paval and PSA was zero (values in located between those of cement and sand).

Dolomite ash showed a reduction of the calcium oxide in the solution. Therefore it promoted the ongoing of some pozzolanic reactions in the solution (consumption of portlandite ($Ca(OH)_2$). Clay brick waste promoted a significant reduction of the CaO and hence it proved to have a high potential for pozzolanic activity.

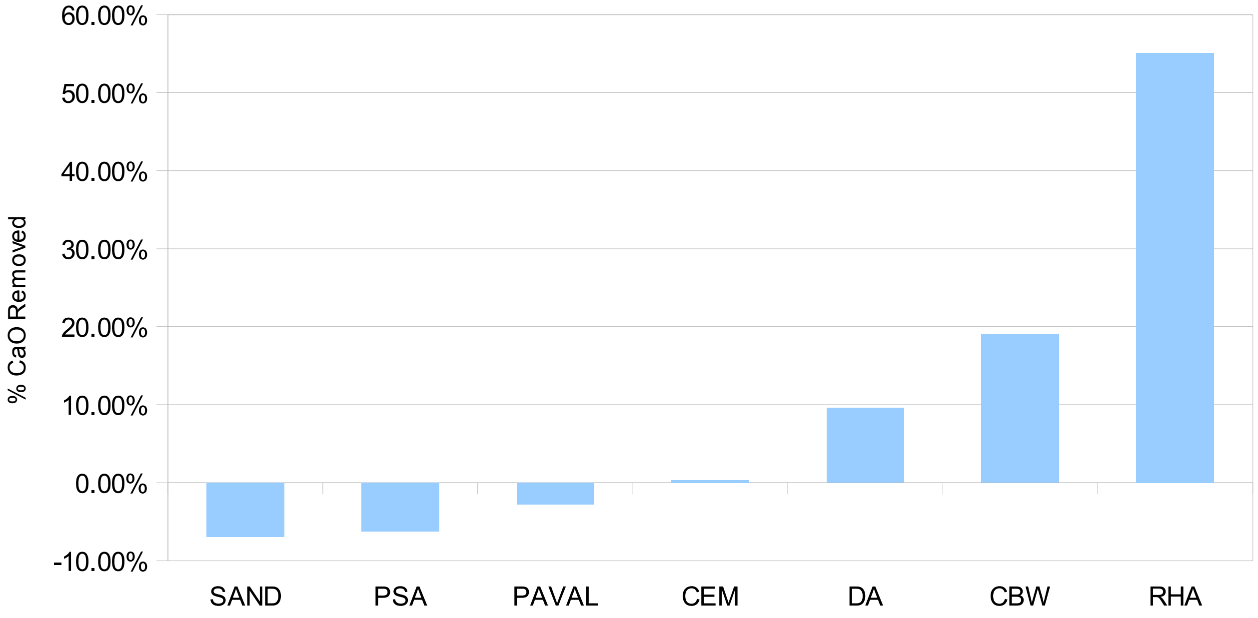


Figure 6.10. Results of Frattini Test after 15 days of curing (BS 196-05)

The rice husk ash presented an enormous potential for pozzolanic activity, according to the Frattini test results **(Figure 6.10)**. The reduction of CaO (consumption of Ca(OH)2) were higher than 50%. Those results are comparable with reported pozzolanic activity of coal fly ash and they are much better than results obtained for silica fume **(Donatello et al., 2010)**.

The Frattini test was not able to provide a value of pozzolanic activity for the biomass fly ash from olive oil industry (BFA). The solubility curve proposed in BS EN 196-1 **(BSI, 2005)** does not consider concentrations of hydroxyl (OH^-) higher than 90 mmol.l^{-1}. However, the chemical titration showed concentrations of hydroxyl ions higher than 200 mmol.l^{-1} in the Cement-BFA solutions, after 15 days of curing.

BFA was obtained from the incineration of a biomass mainly formed of residues from olive trees and olive oil. This olive-related residue is very rich in potassium and the biomass fly ash is therefore very rich in KOH. The pH of the BFA is very alkaline. Therefore, it must be noticed that the Frattini tests is not suitable for the study of pozzolans (or test pozzolans) with a very high pH.

The results of the Frattini test must be compared with other measurements to support the derived findings. The development of other methods for the measurement of the pozzolanic activity will give information on the pozzolanic properties of BFA and additional information for the rest of pozzolans.

6.4.2 REACTIVITY OF POZZOLANS (NaOH SOLUBILITY)

The NaOH solubility is a dissolution technique to measure the reactive Al and Si content of a material, which can be considered a direct method to measure the pozzolanic activity of that material.

Different procedures can be used, mainly referred to the concentration of NaOH in the dissolution used. Typical values range from 1 to 8 mol of NaOH. In this study, both values were used and the measurements were carried out twice on each test pozzolan. The results can be compared and the difference between the results from 1-mol dissolutions and 8-mol dissolutions can be used to explain the different behaviour of the material according to the alkalinity of the environment.

The results of the solubility tests are presented in **Figures 6.11 and 6.12** for the dissolutions of 8mol and 1mol, respectively. Columns represent the test pozzolans (axis x), whilst the values of axis y represent the percentage of undissolved residue weighed after 24 hours of dissolution (rotary shaking methods used for the entire curing time). The higher the value of the undissolved residue, the lower content of reactive material.

In the dissolutions prepared with 8 mol of NaOH (**Figure 6.11**), the most reactive materials were the biomass fly ashes (BFA and PSA). Paval performed similarly to CBW. Surprisingly, the undissolved residue of RHA was quite high, which means that it did not react with the NaOH.

The dissolutions prepared with 1 mol of NaOH (**Figure 6.11**) showed similar results, although the weight of undissolved residues were generally higher. For this dissolution, with lower alkalinity, the undissolved residue of CBW, PSA and RHA were higher than 80%, which shows a very low reactivity. Paval was almost equally reactive for both dissolutions. And the BFA and DA were the most reactive materials when the dissolution of 1 mol was used.

The NaOH solubility test showed interesting results, such as the reactivity of BFA which could not be calculated using the Frattini test. The results obtained for CBW were surprisingly low, given the fact that CBW is usually a material with high pozzolanic properties. According to the experimental results obtained, the reactivity of RHA was also lower than expected. The dissolution of Paval was mild in both concentrations of NaOH.

The results of the NaOH solubility method must be compared with those obtained from other direct and indirect methods to obtain a wider picture of the expected behaviour of the additives when they are used in soil stabilisation or fabrication of blended cements.

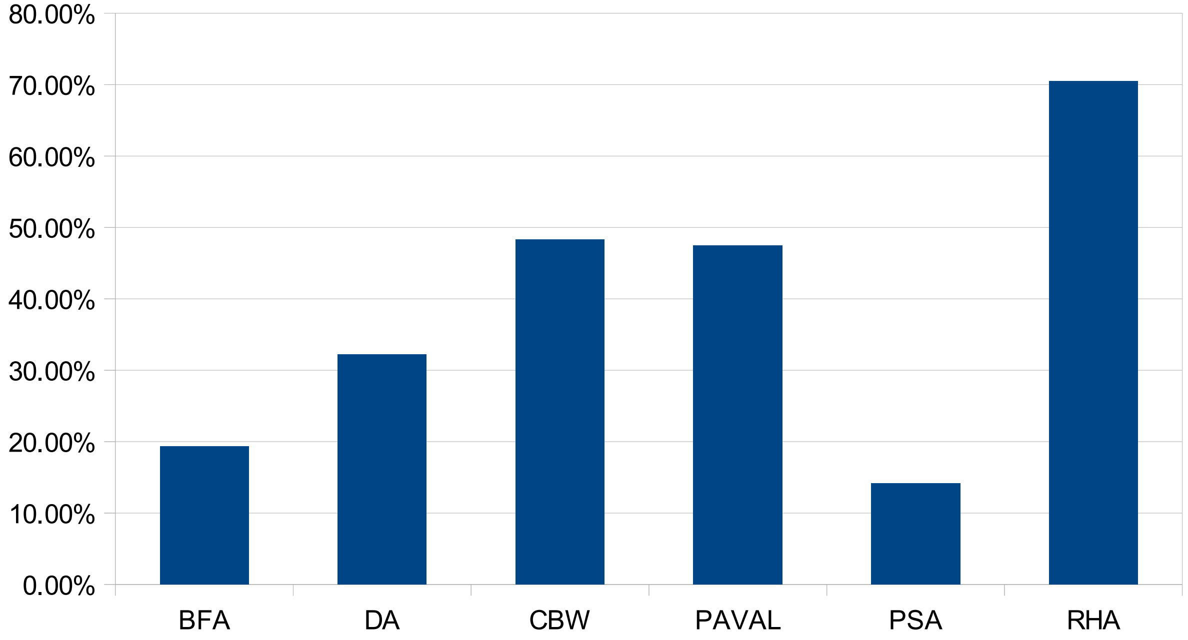


Figure 6.11. NaOH solubility test results (8mol)

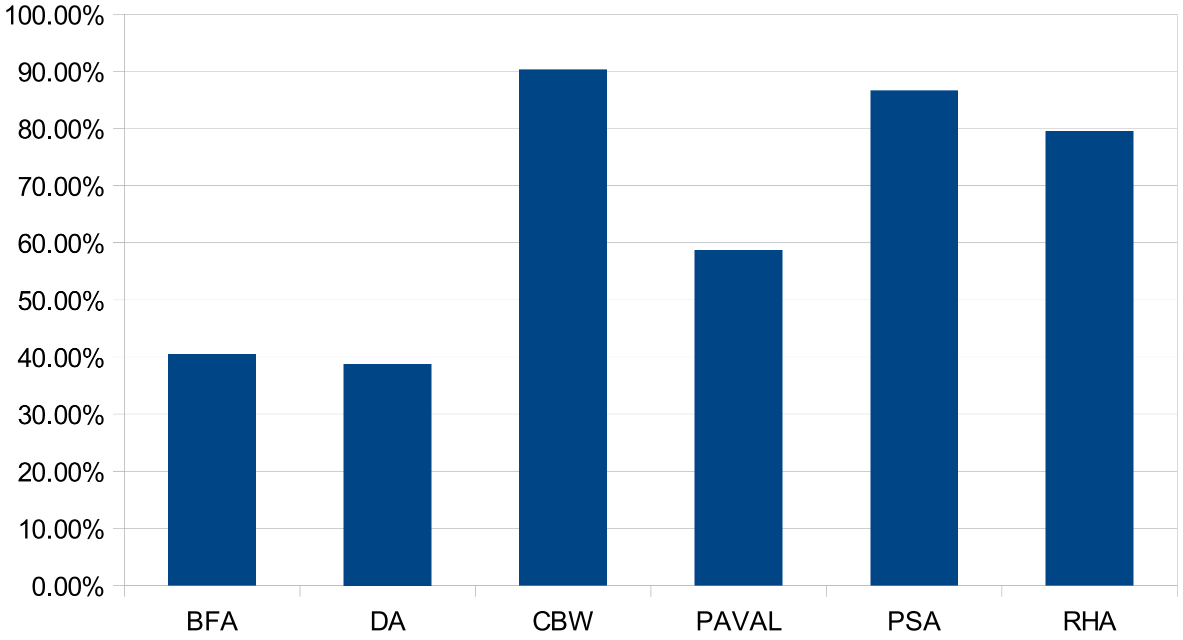


Figure 6.12. NaOH solubility test results (1mol)

6.5 RESULTS AND DISCUSSION (III): POZZOLANIC ACTIVITY BY INDIRECT METHODS

The most commonly used indirect method to evaluate the pozzolanic activity is the strength activity index (SAI). Indirect methods do not measure the chemical consumption of portlandite ($Ca(OH)_2$) but they evaluate the effects of the addition of pozzolans to the final performance of the blended cement prepared.

In the case of SAI, 20% of the weight of cement is replaced by the test pozzolan and the unconfined compressive strength of the resulting blended cement is tested in several curing times (usually between 28 and 90 days).

6.5.1 PREPARATION OF MIXTURES: SETTING AND FLOWABILITY

The preparation of the mixtures was carried out under controlled laboratory conditions (temperature of 20±2°C). Samples of blended cement (80% CEM-I + 20% Test pozzolan) were prepared with an approximate total weight of 150gr per sample. The blended cements were then mixed with water in the minimum necessary dosage of water to produce a workable mixture. The test pozzolans were oven-dried before the preparation of the blended cements. **Table 6.4** shows the required water/cement ratio for each of the samples prepared.

Table 6.4

Water/cement ratio

Test Pozzolan	*Water/Cement ratio*
Cement	0.285
Sand	0.280
Dolomite ash (DA)	0.325
Biomass fly ash (BFA)	0.280
Clay brick wastes (CBW)	0.285
Paper ash (PSA)	0.415
Rice husk ash (RHA)	0.370
PAVAL	0.275

The flow table test allowed for the comparison of the flowability of the blended cements prepared, before the start of the curing and subsequent hardening. The standard BS EN 1015-3 **(BSI, 1999)** was followed. The flowability of the samples was not modified after the measurement of the flow table test. The results obtained are shown in **Figure 6.13**.

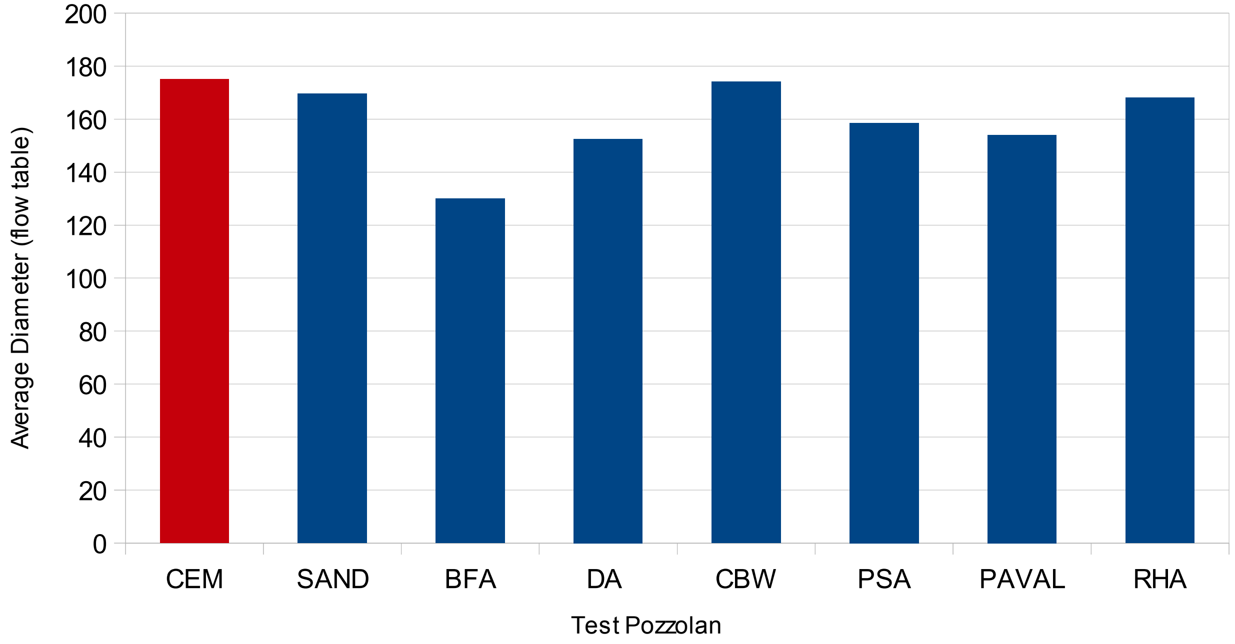


Figure 6.13. Flowability (flow table test, BS 1015-3) of the mixtures prepared for SAI test

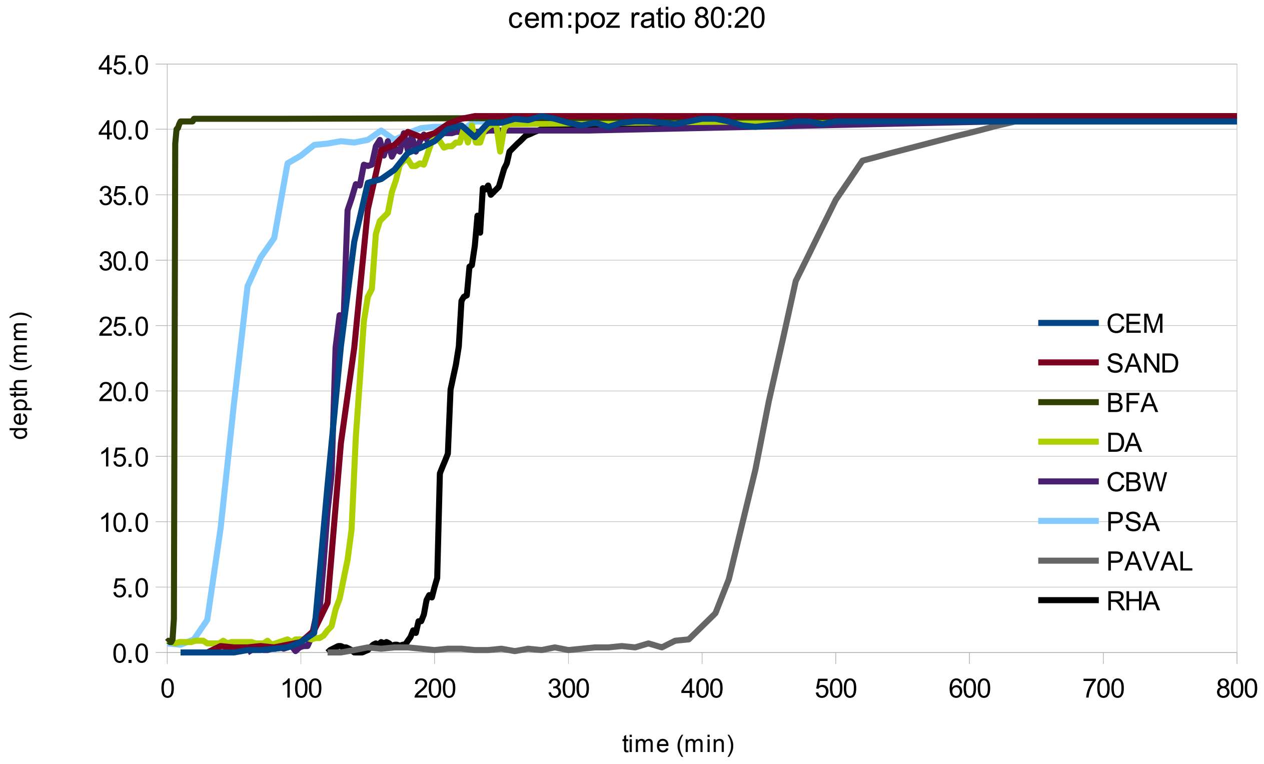


Figure 6.14. Result of the setting test for the mortars prepared for SAI test

In terms of water/cement ratio, all the samples requested a similar dosage of water to produce a workable mixture. However, the RHA and PSA which were very fie grained (almost pulverised) requested the higher dosage of water. The average water/cement ratio was 0.31.

In general, the flowability of the different samples prepared was not very variable. The use of BFA requested a low water/cement ratio and the flowability of the sample was lower than that of cement or sand. DA, PSA and Paval had similar flowability (values similar to those of cement). The blended cements prepared with CBW and RHA were found to be more flowable.

A standard Vicat apparatus was used for the determination of the setting time in the laboratory. A blended cement was prepared with a weight proportion of 80% CEM-I and 20% test pozzolan. The previously stated water/cement ratio were used for the preparation of these samples. The setting tests was initiated always two minutes after the addition of water to the mixture of blended cement. The same procedure was followed for all the samples prepared. The setting apparatus is provided with a needle located 40mm above the top level of the sample. The needle is vertically inserted in the sample of cement every one minute (the time of insertion can be corrected). As the blended cement hardens, the insertion of the needle in the sample becomes shorter and shorter. The results of the test can be plotted: depth of insertion versus time **(Figure 6.14)**.

Significant variations were found to exist regarding the setting time of the different blended cements prepared. Most of the samples prepared (including the control samples of 100% cement and 80% cement-20% sand) presented a setting time around 100-120 minutes. This is the case of DA and CBW.

A relevant phenomenon was found during the development of this experiment: the setting time of the blended cements prepared with CEM-I and BFA was dramatically short. Several samples of this blended cement were prepared and measured. The setting time obtained was always shorter than 10 minutes, with a quick hardening found to occur after 7 minutes of curing. This setting times were dramatically shorter than those obtained for all the other samples prepared (using test pozzolans other than BFA). This effect can be attributed to the alkalinity of the BFA and its high content of potassium which could accelerate the hardening process.

The samples prepared with the biomass fly ash from the pulp and paper industry (PSA) was also found to produce a similar effect of reduction of the setting time. The setting of the samples prepared with CEM-I and PSA started at the 30 minutes of curing and was almost completely finished before 100 minutes of curing.

The opposite effect was found to exist in the case of blended cements prepared with paval and rice husk ash (RHA). The setting time for the sample prepared with RHA was delayed to 200-250 minutes. In the case of the sample prepared with paval, the setting was found to take place at 400-600 minutes of curing.

After the preparation of the blended cements, they were cast into moulds to prepare prismatic samples. These samples were stored at a curing room with controlled humidity (>90%) and temperature (20±2°C).

6.5.2 STRENGTH ACTIVITY INDEX (SAI)

The unconfined compressive strength (UCS) of the blended cements prepared with 20% (in weight) of the test pozzolans was tested at 2, 7, 28 and 67 days of curing. The UCS of the sample prepared without pozzolans (UCS_{CEM}) is taken as a control. The SAI of each test pozzolan is defined as the percentage of UCS related to the original UCS_{CEM}. The results obtained after 28 and 67 days of curing are presented **Table 6.5**.

Table 6.5

SAI Results

Composition of blended cement	*UCS after 28 days*		UCS after 67 days (MPa)
	UCS (MPa)	SAI (%)	
100% CEM-I	77.0	-	94.7
80% CEM-I + 20% Sand	74.2	96.4	100.5
80% CEM-I + 20% BFA	66.5	86.4	75.2
80% CEM-I + 20% DA	67.9	88.2	89.1
80% CEM-I + 20% RHA	67.4	87.6	87.5
80% CEM-I + 20% CBW	66.4	86.2	81.7
80% CEM-I + 20% PSA	52.6	68.3	51.4
80% CEM-I + 20% PAVAL	48.5	63.0	55.8

According to BS 3892, SAI results greater than 0.80 after 28 days are indicative of a positive pozzolanic activity for FA for a cement replacement of 30%. ASTM C618 requires a SAI greater than 0.75 after 7 and 28 days for FA and natural pozzolans at a cement replacement of 20% **(Donatello et al., 2010)**.

The results presented in **Table 6.5** show that after 28 days, SAI was greater than 75% for BFA, DA, RHA and CBW. According to the SAI test results, PSA and Paval did not show pozzolanic activity, since the strength of the blended cement was not increased by the addition of these agents.

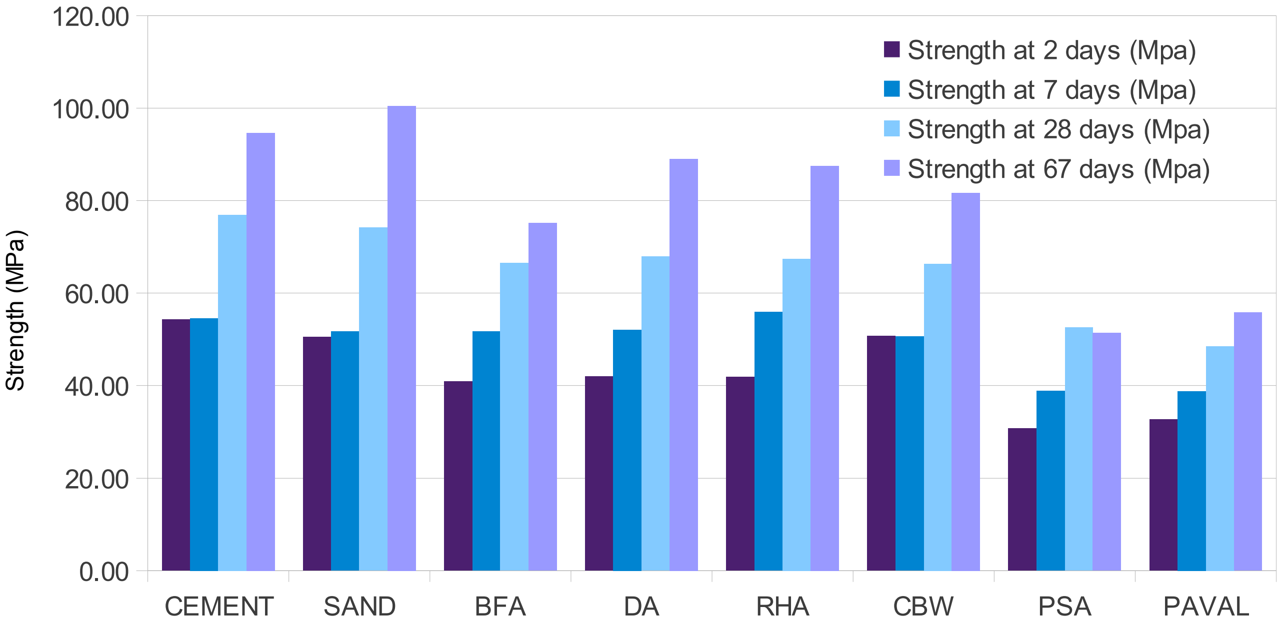


Figure 6.15. Results of Strength Activity Index along curing time

Figure 6.16. Comparison of the results of strength obtained after 67 days of curing

The evolution of the compressive strength along curing time is presented in **Figure 6.15**. The comparative results of UCS after 67 days could not be considered as a strength activity index according to the previously mentioned international standards. However, the study of the percentage of strength after 67 days of curing can provide interesting results (**Figure 6.16**). In terms of comparison, the percentage of compressive strength of the blended cements prepared with pozzolans provided similar results after 28 and 67 days. After 67 days, the samples prepared with BFA, DA, RHA and CBW obtained a compressive strength greater than 75% of that of the cement sample. The PSA and Paval did not promoted an increase of the strength of the samples, which bolsters the hypothesis of low pozzolanic activity of PSA and Paval according to the result of the indirect method.

6.6 CONCLUSIONS

The chemical composition and physical properties of six industrial by-products obtained in the south of Spain were tested in this experiment to evaluate their pozzolanic activity and their potential use in the construction industry as additives for blended cements and soil stabilisation treatments. Along with the characterisation of the additives, direct and indirect methods have been used to evaluate the pozzolanic activity of the selected by-products.

According to the results of the experimental program, the following concluding remarks can be drawn:

- The industrial by-products used in this study were not considered to be hazardous materials. Their chemical composition showed the presence of calcium and magnesium oxides, silica, alumina, etc. They were obtained in the form of very fine-grained materials, and in the case of PSA an RHA, they were in the form of pulverised materials. The SEM pictures indicated a certain crystalline structure of PSA and RHA which could decrease their pozzolanic properties, given the fact that the reactivity is based on the amorphous structures of the fly ash.

- The chemical composition of BFA and DA showed highest values of calcium oxide. PSA and CBW presented high concentrations of calcium oxide in their chemical composition, however laboratory tests showed that PSA and CBW (unlike DA and BFA) were not reactive in the presence of water and setting and hardening of those pozzolans did not take unless cement was added to the mixture.

- The Frattini test is a direct method to measure the pozzolanic activity of a test pozzolan (chemical titration to evaluate the consumption of calcium hydroxide). The solubility curve provided in the standard BS EN 196-1 is not prepared for concentrations of hydroxyl greater than 90 mmol.l^{-1}. The Frattini test could not evaluate the pozzolanic activity of BFA due to its great concentration of hydroxyl ions. This must be due to the concentration of potassium and high alkalinity of BFA.

- The results of the Frattini test showed the pozzolanic activity of DA, CBW and RHA. The latter presented the most remarkable potential for pozzolanic activity, according to Frattini. PSA and Paval would be considered to be non-pozzolanic according to this test.

- The preparation of blended cements for the development of the SAI test showed significant variations in the setting times of the samples. In the samples prepared with BFA and PSA, the setting took place in a really short period of time (shorter than 10 and 50 minutes, respectively) whilst for the rest of the samples the setting did not start until 100 minutes of curing. In the case of BFA, this might be due to the presence of potassium and the alkalinity of the additive.

- The flowability of the samples prepared for the SAI test was not very variable. The samples prepared with BFA requested a low water/cement ratio to produce a workable mixture. However, the mixtures prepared with PSA and RHA requested a high dosage of water to become workable. This might be due to the fine-grained nature of these additives.

- The SAI test showed the pozzolanic properties of BFA, DA, RHA and CBW both for short and long curing periods. The results of SAI suggested that PSA and Paval did not show pozzolanic properties, since the strength of the blended cement paste prepared with these additives was not increase due to the addition of PSA and Paval.

- PSA and Paval did not show pozzolanic activity in both direct and indirect methods. The Frattini test showed that these additives did not contribute to the further consumption of portlandite in a cement solution. Strength activity index test showed results of 68.3 and 63% for PSA and Paval, respectively. The values lower than 75% are indicative of a null pozzolanic activity.

- The NaOH solubility test was reported to evaluate the presence of active silica and alumina in the existing literature. However, the values of solubility were surprisingly low for CBW and RHA, additives which are expected to have remarkable pozzolanic properties. Furthermore, the results of this test were not in accordance with those of traditional tests such as Frattini and SAI. The results of this test were comparable for the use of 1mol and 8mol dissolutions.

- In terms of comparison, BFA, DA and RHA presented promising properties for their use in the construction industry. Paval and PSA showed the poorest properties. CBW and RHA proved to be a non-self-cementing additive with pozzolanic properties.

However, DA was among the best additives in terms of direct and indirect methods for evaluation of pozzolanic activity. BFA promoted increase of the strength of the blended cement according to the SAI results. Both BFA and DA provided positive results in terms of pozzolanic activity and they both had large amounts of calcium oxide in their chemical composition which make them self-cementing pozzolans.

- From a sustainable point of view, the use of the additives tested in this study as stabilisation and/or pozzolans for blended cements could improve the environmental and economic costs associated with their waste management and could lead to a more efficient construction system. BFA and DA are both present in the south of Spain as residues with large amounts to be disposed off. Both can be obtained in a cost-effective manner.

EXPERIMENTAL RESULTS
PART II

Chapter 7

Effect of lime-treatment on marls subjected to wetting-drying cycles

Abstract

The effect of the addition of lime on the physical properties and mineral composition of a marly soils is studied in this chapter. One of the main conclusions which can be extracted from the introduction of this book (and especially from the literature review carried out) is the need for a further study of the effects that the so-called traditional binders (such as lime, cement or coal fly ash) promote on different type of soils. The amount of clay, the presence of carbonates, the environmental conditions, etc. can significantly affect the performance of the soil-additive system. In this stage of the investigation, a natural marly soil with a high swelling potential was treated by addition of different dosages of lime (2.5% and 4.0% by weight of dry soil). The samples prepared are subjected to 1, 3 and 5 wetting-drying cycles (total curing time of 77 days) to emulate real conditions of earthworks in regions with seasonal variations of moisture. The performance of the treatments was assessed by physico-chemical tests: particle size, consistency limits, bearing capacity, pH and free swell. Mineral changes of soils were studied by XRD and SEM. The results showed the quick effect of lime: flocculation of particles (tending to coarser particle size distributions of the soil) and reduction of plasticity. The physical properties of the marly soil were significantly improved due to the treatment. However, a funny tendency was found to exist in the evolution of the soil properties, specially bearing capacity and swelling potential, with those parameters suffering a set-back around the third wetting-drying cycle. The improvements continued afterwards and a significant enhancement was recorded in the soils after 77 days of curing and 5 wetting-drying cycles.

7.1 MATERIALS USED

The physical properties of the natural marl from Southeastern Spain used in this study are shown in **Table 7.1**. This marly soil **(Figure 7.1)** is rich in clay and calcium carbonate. This soil has a swelling potential based on the expansive clay phases present within its mineral composition.

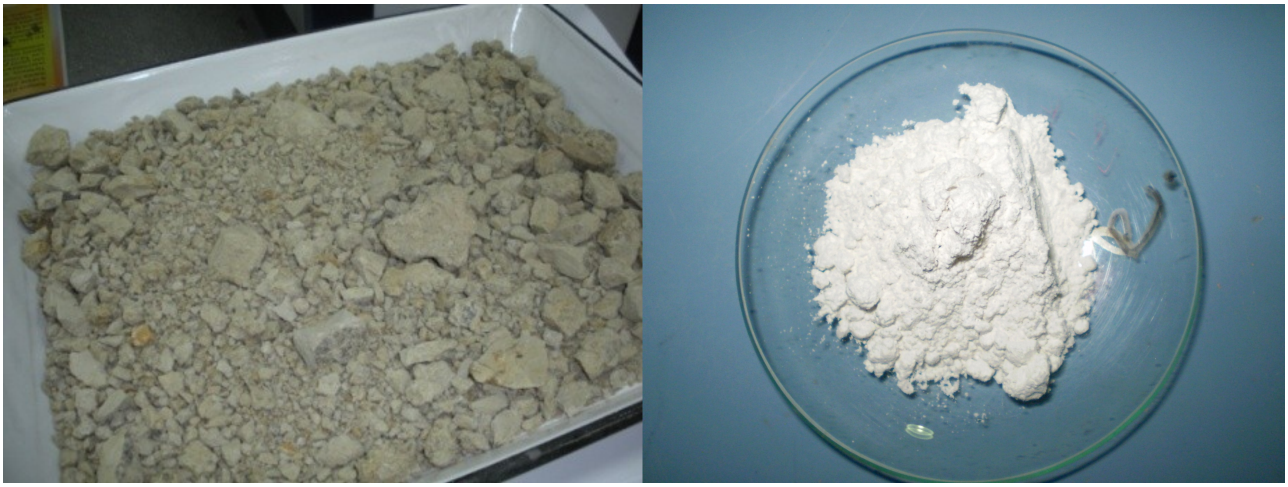


Fig. 7.1. Pictures of natural marl and commercial lime used in this study

The soil used belongs to the calcareous silt and clay (marl) cropping out in the Neogene basins of Granada (Betic Cordillera, Southeastern Spain). The geology and engineering geology of these marls have been studied by **El Amrani and Chacon (1996)** and **El Amrani et al. (1998)**. According to those works, the amount of carbonates in the natural soil studied typically ranged from 20 to 35%. The stabilisation agent used in this study was a commercial quicklime with more than 90% concentration of CaO **(Figure 7.1)**.

7.2 METHODOLOGY

Several mixtures of marly soil and lime were prepared with different dosages of additive: 2.5% and 4.0% (weight of dry sample of soil). Those percentages of lime were added to the dry sample of soil before adding water to the mixture. The samples were prepared in a laboratory tray and their weight was greater than 3 kgs each to ensure there was enough amount of treated soil for all the different tests to be conducted. The trays were then saturated for 7 days in a curing room with stable conditions of moisture and temperature (95-100%; 20ºC). The samples were finally subjected to 1, 3 and 5 wetting-drying cycles to study the effects produced on the earthworks by the seasonal variations.

Table 7.1

Characterisation of natural marly soil

Consistency	
Liquid limit (%)	57.4
Plastic limit (%)	26.8
Plasticity index (%)	30.6
Compaction	
Modified Proctor MDD (kN/m^3)	17.6
Modified Proctor OMC (%)	13.9
Bearing Capacity	
C.B.R. (%)	2.5
Swelling	
Free Swell (%)	6.19
CBR Swell (%)	2.29
pH	
pH	8
Sulfates	
Soluble sulfates (%)	0.24

In this study, one wetting-drying cycle consisted on 7 days of water immersion followed by 7 days of heat (50ºC). After the cycles, the samples were stored for 7 days under water saturation conditions to complete the curing process. The final curing time was 77 days, The preparation of the samples and the wetting-drying cycles carried out in this study are summarised in **Table 7.2**.

Table 7.2

Characteristics of lime-soil mixtures prepared

Samples	Additive	Dosage (%)	Wetting-drying cycles	Curing time (days)
Sample 1	Lime	2.5	0	7
Sample 2	Lime	2.5	1	21
Sample 3	Lime	2.5	3	49
Sample 4	Lime	2.5	5	77
Sample 5	Lime	4.0	0	7
Sample 6	Lime	4.0	1	21
Sample 7	Lime	4.0	3	49
Sample 8	Lime	4.0	5	77

To study the physical properties of the soils samples, the following tests were carried out: particle size using sieve analysis, Atterberg limits, California bearing ratio (CBR), free swell in oedometer, pH and X-ray diffraction and SEM (**Figure 7.2**).

Fig. 7.2. Lab equipment used in this study (University of Granada, Spain): a) Field Emission Scanning Electron Microscopy; b) Phillips X'Pert diffractometer

7.3. RESULTS

As mentioned above, the consistency of the treated samples was studied by Atterberg limits tests. Based on these tests, the main remark which can be drawn is the noticeable reduction of plasticity of soils after the addition of lime. For samples 1 and 2, the plasticity index found was 14.6% and 13.1%, respectively, reducing the plasticity index of the original untreated soil (30.6%). The rest of samples studied were found to be non-plastic from the earlier stages of curing. No additional effect of the wetting-drying cycles on the plasticity of the treated soils could be noticed.

Table 7.3

Results of free swell, CBR and pH tests on lime-stabilised samples

Samples	Dosage of Additive (%)	Cycles	Curing Time (days)	Free Swell (%)	pH	CBR (%)
Sample 1	2.5	0	7	0.2	10.84	90.0
Sample 2	2.5	1	21	0.7	11.21	134.5
Sample 3	2.5	3	49	1.4	10.10	69.5
Sample 4	2.5	5	77	0.3	11.12	98.3
Sample 5	4.0	0	7	0.0	11.86	112.9
Sample 6	4.0	1	21	0.2	11.60	247.4
Sample 7	4.0	3	49	0.4	10.23	191.5
Sample 8	4.0	5	77	0.2	10.85	265.8

The samples of lime-stabilised soil were subjected to CBR and free swell tests to analyse the evolution of their bearing capacity and swelling potential. The results of the CBR tests are tabulated in **Table 7.3** together with the free swell index and the pH values of the samples. Those results are plotted in **Figures 7.3, 7.4 and 7.5**, respectively.

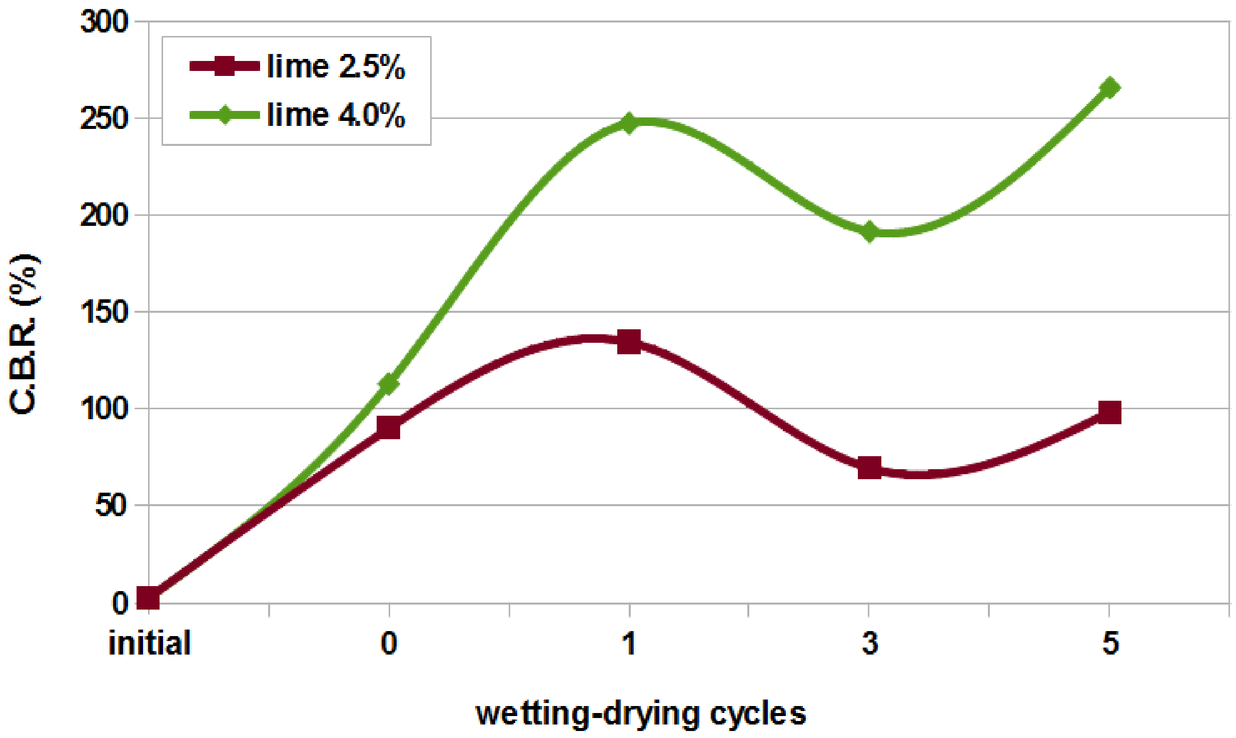


Fig. 7.3: Evolution of C.B.R. in samples treated with 2.5% and 4.0% lime

The particle size distribution changed dramatically after the treatment, although it can be noticed that for the mixtures with 4.0% lime the remarkable change occurs immediately after the addition of lime while for the 2.5% lime mixtures this effect takes place later. **Figures 7.6 and 7.7** show the results of the particle size tests undertaken for the natural soil and the mixtures with 2.5% lime and 4.0% lime, respectively, after 0, 1, 3 and 5 wetting-drying cycles.

Once all the samples had been scanned by XRD, a characterisation was made by using the reference pattern sheets databases and XPowder software, version 2004.3.1 **(Martin-Ramos, 2004)**. The following minerals were found in all the mixtures: calcite, dolomite, quartz and clay minerals (kaolinite, illite and smectite (montmorillonite-beidellite)). The XRD pattern of sample 1 (marl + 2.5% wt. lime) is depicted in **Figure 7.8**.

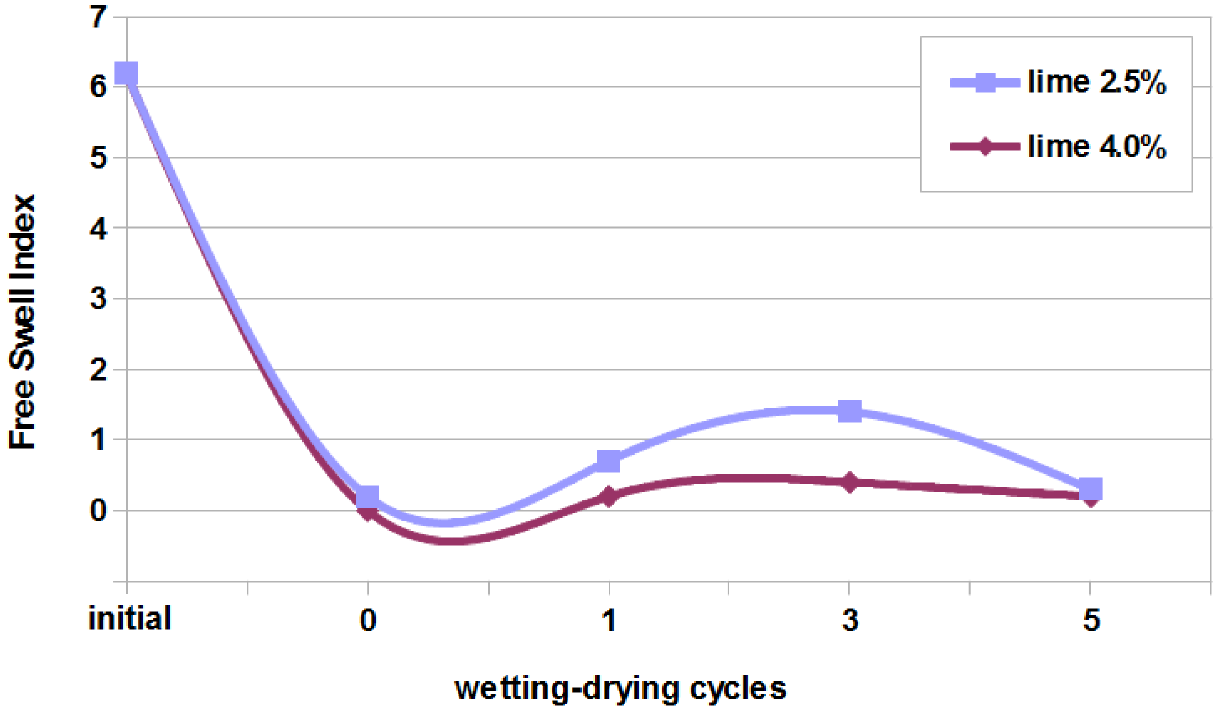


Fig. 7.4: Evolution of free swell in oedometer in treated samples

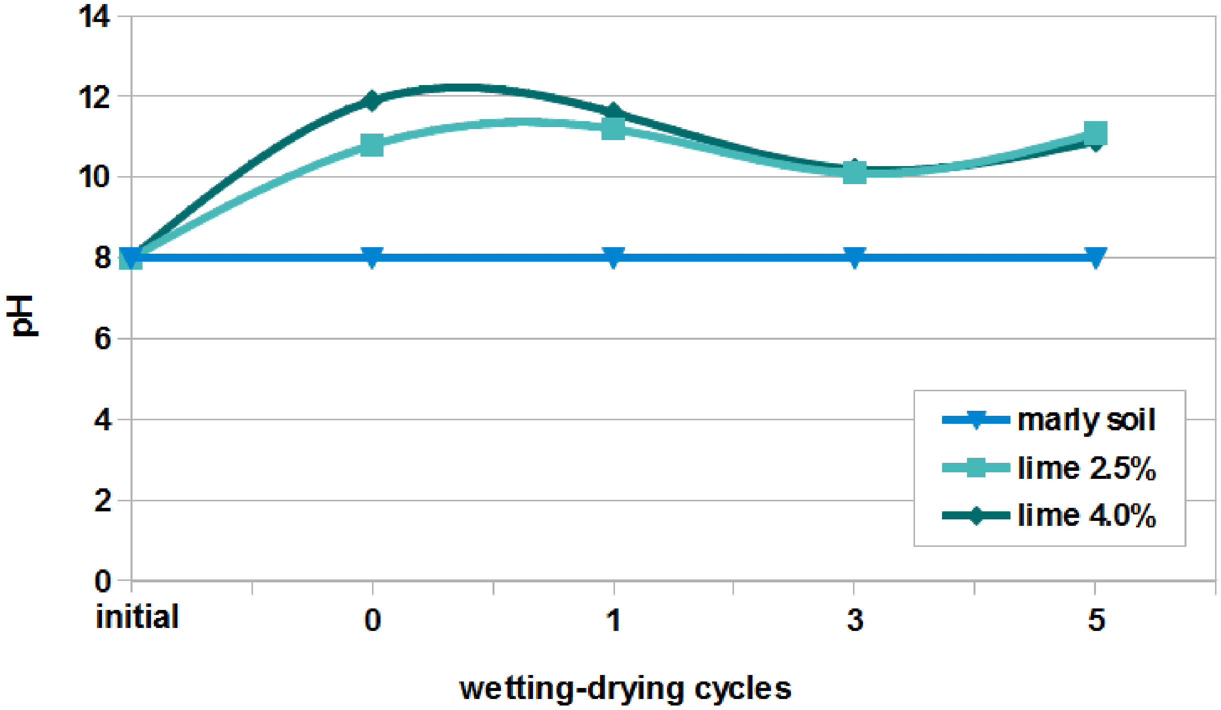


Fig. 7.5. Evolution of pH in stabilised samples

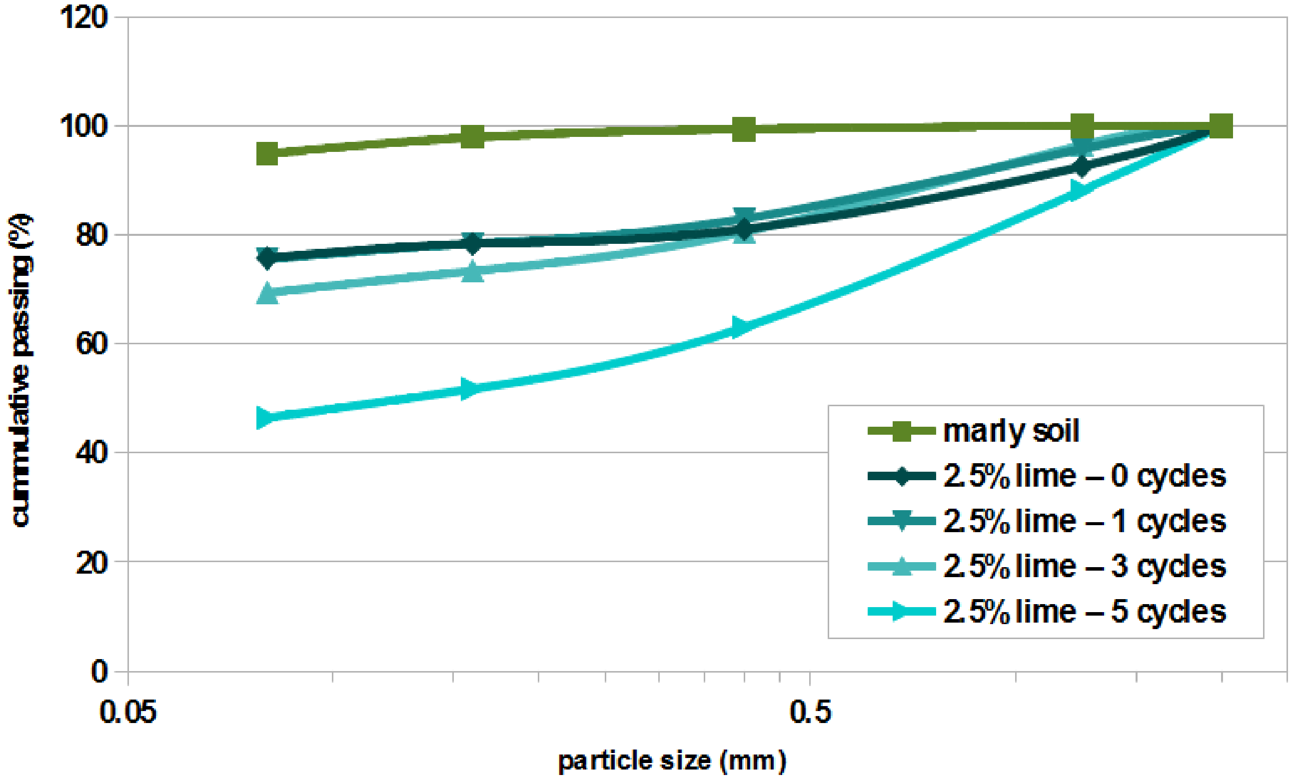


Fig. 7.6. Particle size distribution of samples treated with 2.5% lime after wetting-drying cycles

The fine fraction XRD pattern of the sample 1 is plotted in **Figure 7.9**. There are some peaks in the total fraction pattern of sample 1 which might belong to chlorite, although the study of the clay minerals on oriented aggregates shows that those peaks belongs to smectite and kaolinite respectively. The peak located at 14.3 Å belongs to smectite (**Figure 7.9**), since this peak shifted to 17 Å d-spacing when glycolated (**Cuadros and Linares, 1996**). On the other hand, other clay mineral peaks disappear after 550ºC heating. It can be inferred that those peaks belong to kaolinite and therefore there was not chlorite in the sample (**Nieto et al., 2008**). Thus, in the fine fraction, the following clay minerals were present: illite, smectite and kaolinite. A comparative representation of oriented aggregates patterns of all samples is depicted in **Figure 7.10**.

7.4 DISCUSSION OF RESULTS

Lime addition to the expansive soil can produce the aggregation of clay particles (**Cuisinier et al., 2009**). This phenomenon of flocculation and aggregation of clay particles has been stated to produce a rapid improvement in the mechanical properties of soils (**Seco et al., 2011; Locat et al., 1990; Dafalla and Mutaz, 2012**). Additionally, lime-soil reaction is exothermic, which can explain the celerity and effectiveness of the process. The results of the consistency limits tests obtained in this study (sharp decrease of plasticity index) might be due to these physical modifications induced by lime addition, which take place very fast and are appreciated from the very early stages of curing. The quick reduction of plasticity was not affected in later stages by the wetting-drying cycles.

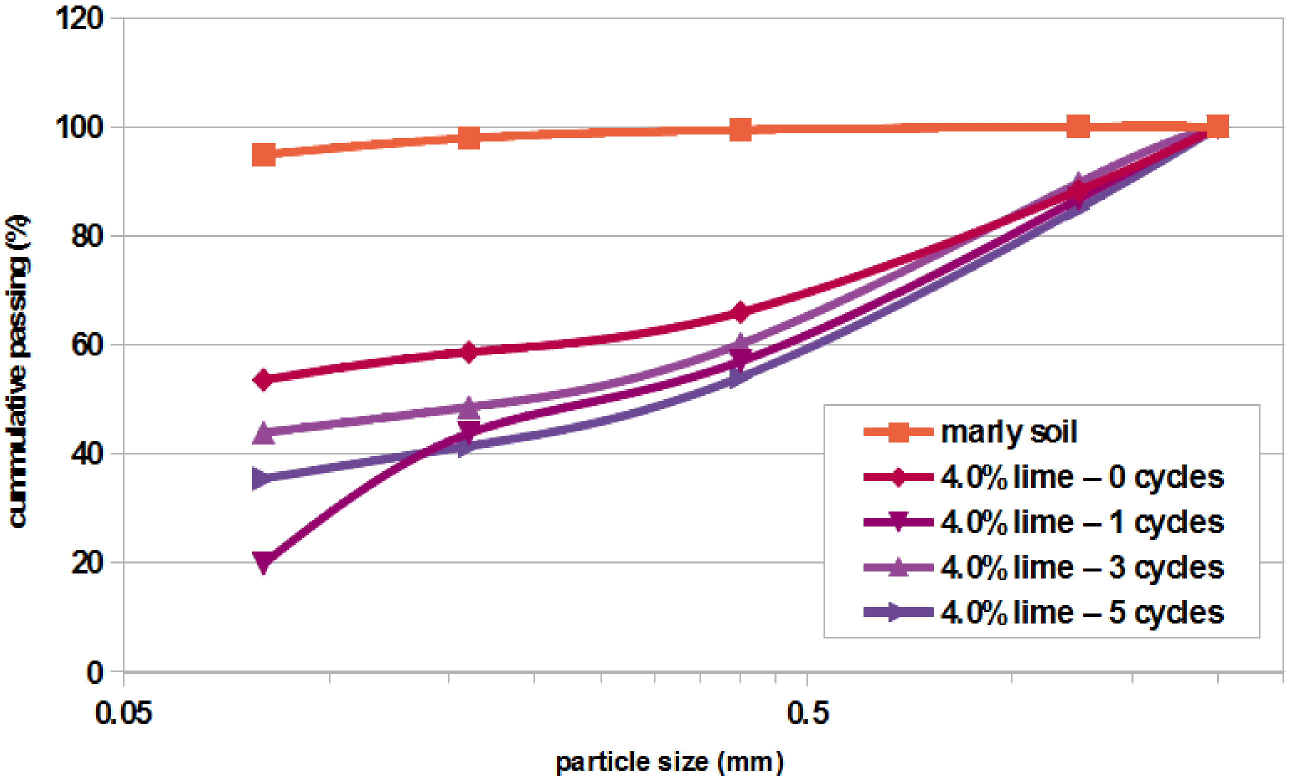


Fig. 7.7. Particle size distribution of samples treated with 4.0% lime after wetting-drying cycles

Burland et al. (1996) explained the role of particle size in mechanical behaviour of soils and how the presence of coarse particles can directly affect the performance of the soil making it more resistant to changes in environmental conditions. The changes in particle size obtained in this work are plotted in **Figures 7.6 and 7.7**. Grain size of the stabilised soils after treatment with lime became coarser and coarser compared with the original marly soil. After the wetting-drying cycles, the particle size continued becoming coarser. As expected, the changes were more remarkable for the samples treated with 4.0% lime than for those treated with 2.5% lime.

The values of CBR of the treated samples increased with the curing time, the improvement being greater for those samples treated with 4.0% lime than those treated with 2.5% lime. The same could be observed for the swelling potential of the treated samples. The initial value of 6.19% was reduced to values under 1.4% for the samples treated with 2.5% lime and under 0.4 for the samples treated with 4.0% lime. Regarding the effect of the wetting-drying cycles, the samples seem to suffer a reduction in the bearing capacity and a slight increase in the value of the free swell after the third cycle. However, the values of CBR and free swell reached at this stage are still much better than the initial values and the values obtained after 5 cycles were found to show a further improvement. The modification of the physical properties of the soils such as the consistency and the particle size is undoubtedly related to the improvement in the bearing capacity and swelling potential. The effect of the wetting-drying cycles is not remarkable and the treatment with lime is, in terms of physical properties, successful after 77 days of curing.

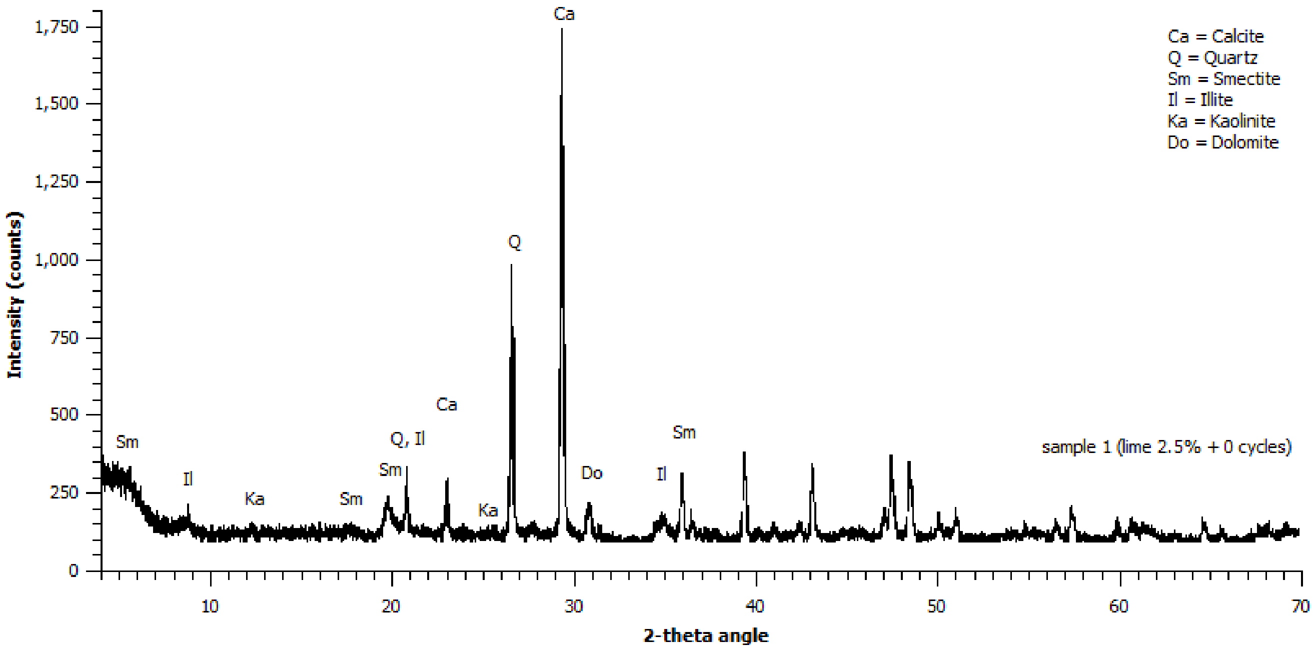


Fig. 7.8. XRD pattern of total fraction of sample 1 (2.5% lime)

A relationship between the values of pH and free swell was found for all the samples tested. The pH reached values ranging from 11-12 confirming the alkaline environment provided by lime addition. However, this pH value could have not been high enough to enable the dissolution of the clay minerals (pH > 12.3), according to **Eades and Grim (1966)**. This could be behind the fact that the physical modifications observed were accompanied only by minor modifications of the mineral composition of the soil. The comparison of the XRD tests carried out **(Figure 7.10)** let us infer that, although there was a tendency to changes in the intensity of the smectite peaks, the amount of smectite present in the treated samples seem to remain very significant. At this point, it is necessary to mention the limitations of the XRD technique to evaluate quantitatively the mineral composition of soils.

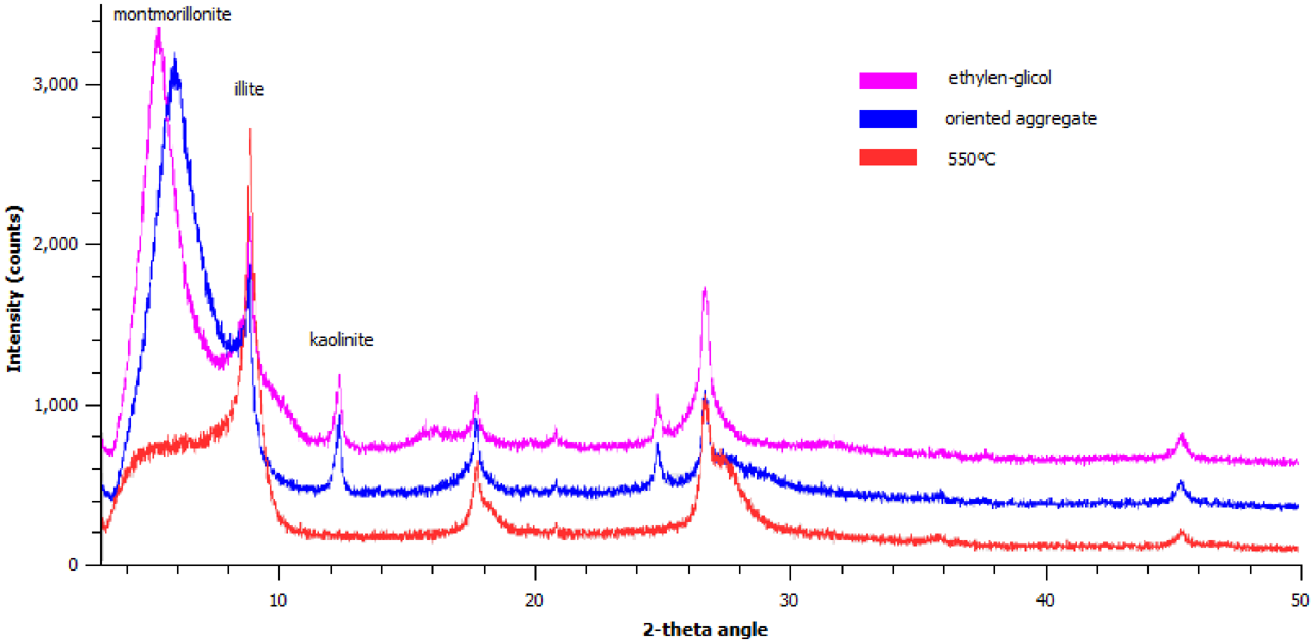


Fig. 7.9. XRD pattern of oriented aggregates of sample 1 (2.5% lime)

On the base of these results, it can be stated that the dissolution of clays was not intense enough for the samples prepared in this research. This can be due to the fact that the development of this reaction takes longer than the high-impact quick effect of agglomeration of particles. But this phenomenon can also be attributed to the specific properties of marly soils. When lime is added to marls, the flocculation starts very quickly. However, as a result of the reaction, new calcium carbonates could precipitate, promoting a faster consumption of the calcium oxide in this type of soils. If lime is consumed quicker in marls than it is in pure clays, an immediate consequence is the reduction of pH, and hence the lack of the alkaline environment needed for the removal of expansive mineral phases. This is consistent with previous studies, such as those carried out by **Seco et al. (2011)** which found improvements in lime-stabilised marls due to aggregation of particles, but no formation of cementitious gels was detected.

The comparison of the SEM pictures of the original marly soil **(Figure 7.11a)** and the samples treated with 2.5% and 4.0% lime (**Figures 7.11b and 7.11c**, respectively) shows little difference among them at reference distances of 1 μm. In the original marl, structures of clay layers can be seen, along with plenty of fossils which represent the $CaCO_3$ of the natural soil. The formation of cementitious compounds in the samples treated in this study could not be proved.

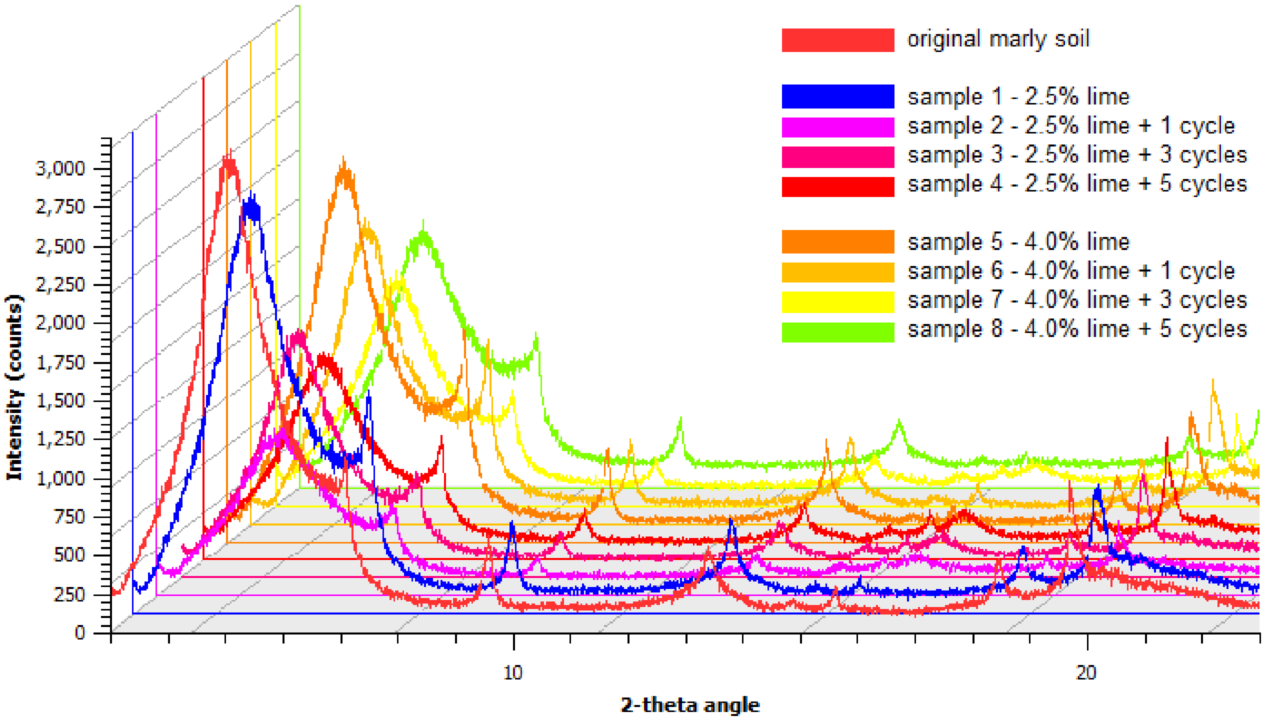


Fig. 7.10. Comparative XRD patterns of oriented aggregates

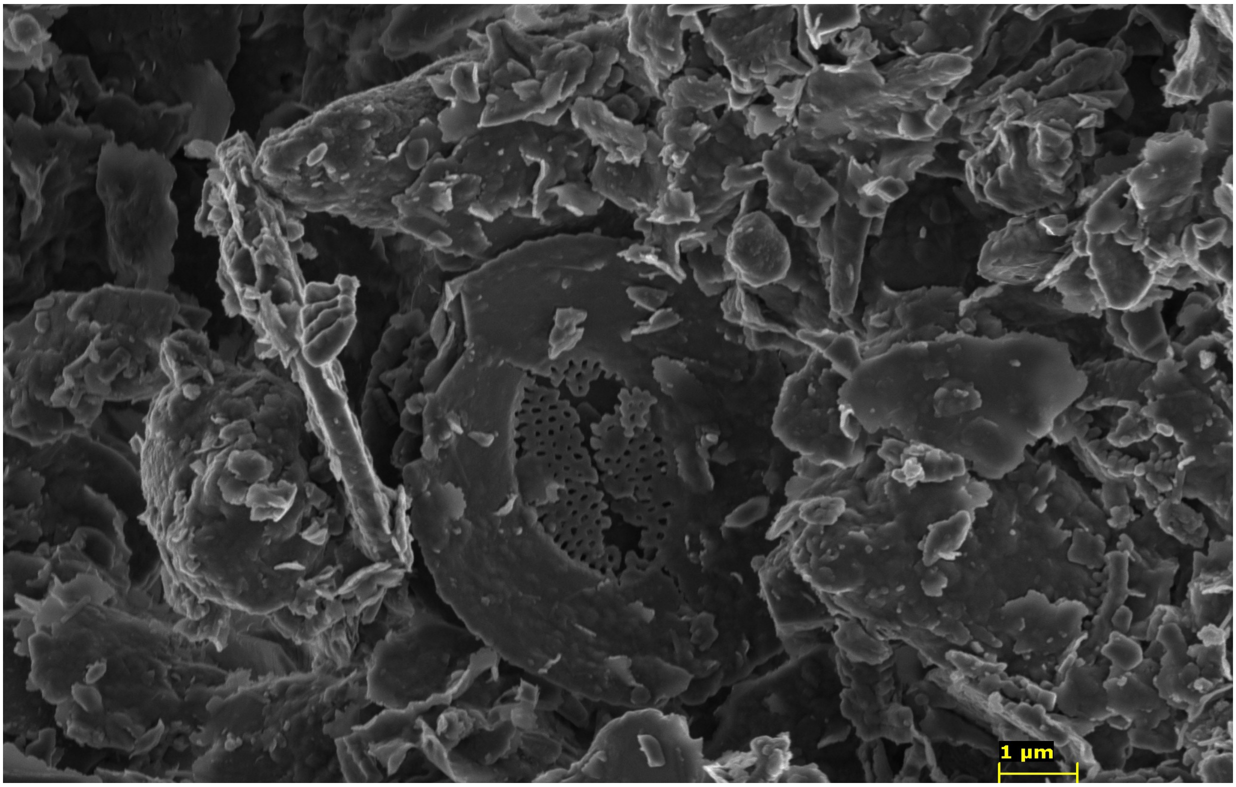


Fig. 7.11a. Clay particles and fossils carbonates in natural marly soil (SEM picture)

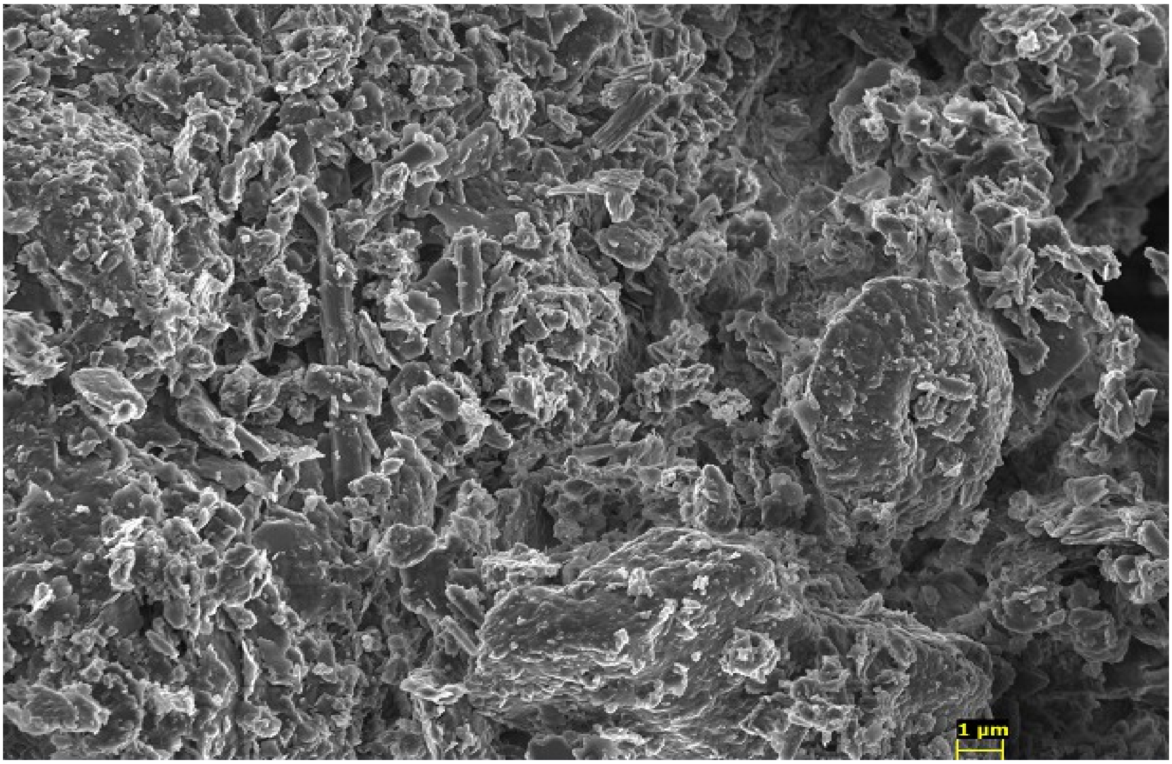


Fig. 7.11b: SEM picture of 2.5% lime treated soil

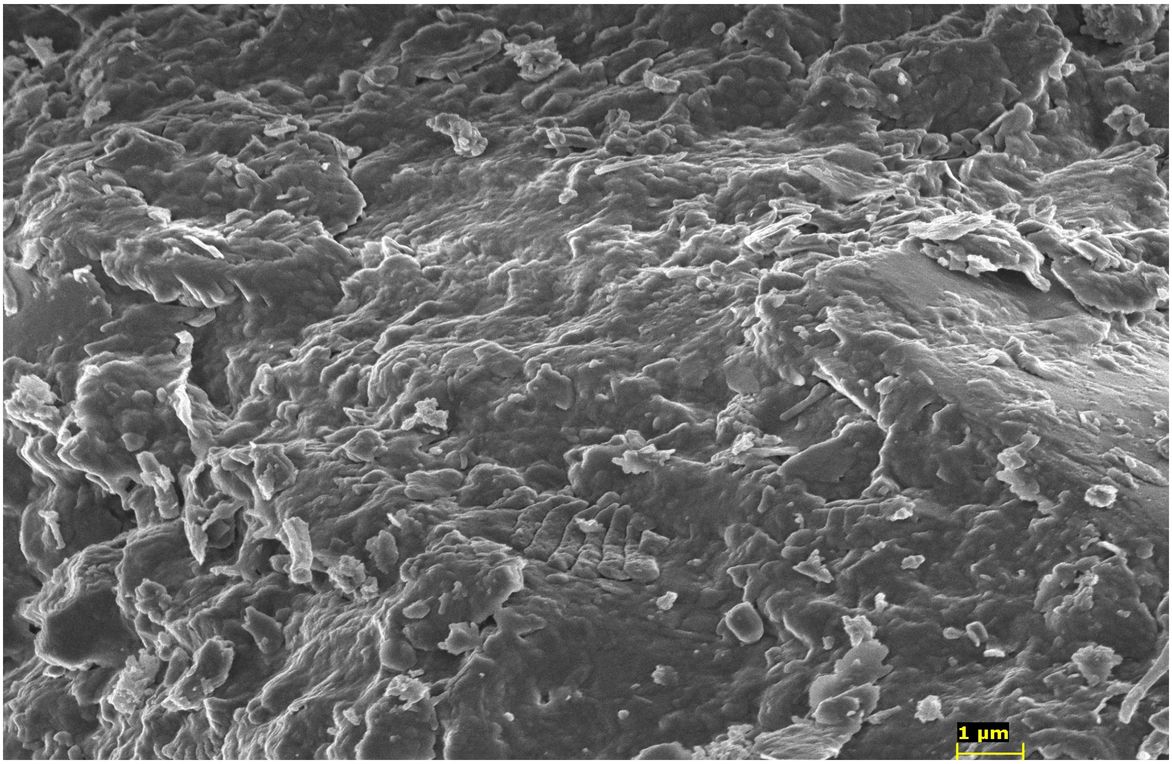


Fig. 7.11c: SEM picture of 4.0% lime treated soil

7.5 CONCLUSIONS

Given the significant amount of clay in the composition of marly soils, these soils can suffer important volume changes when the environmental conditions of moisture vary. The objective of the use of a wetting-drying type of curing in this study was to evaluate the effect of those moisture changes in the treated soils. Based on the results of this research, the following remarks can be drawn:

- Lime addition has a high-impact effect on marls, promoting quick changes in the consistency and grain size of the soils, from the first stages of curing. This modification of particle size of the soil (tending to coarser distributions) is responsible for improvements in the physical properties of soil immediately after treatment. The evolution of this increase in particle size is not linear along time which could be partially due to the wetting-drying cycles. However, the improvement was remarkable for all the different stages of curing.

- The bearing capacity of the marly soils treated in this study was dramatically increased by the addition of small amounts of lime whilst the free swell diminished from an initial value of 6.19% to values under 1.4% and 0.4% for dosages of 2.5% and 4.0%, respectively. Those improvements in the bearing capacity and swelling potential of the soil remained after 77 days of curing and 5 wetting-drying cycles.

- The small dosages of additive and the use of wetting-drying cycles were used to emulate the real conditions of curing. The treatment with lime was effective to reduce the sensitivity of the marly soil to changes in the available moisture. After 5 wetting-drying cycles, the samples treated with lime had a higher bearing capacity, coarser particle size and lower plasticity and swelling potential than that of the original soil.

- In the analysis of the performance of a soil treatment, both XRD and SEM techniques must be approached carefully and the conclusions made must always be supported by the results of complementary tests. In this study, the results of the XRD tests suggested that smectite was the most important mineral in the composition of the marly soil both before and after the treatment with lime.

- Marly soils have to be clearly distinguished from pure clays since the presence of carbonates can affect their behaviour. Hence the presence of carbonate ions in the interstitial water could affect the stabilisation treatment by accelerating the lime consumption. The maintenance of an alkaline environment is crucial to enable the dissolution of clay minerals and the modifications of the mineral composition of the marly soil.

Chapter 8

Magnesium hydroxide, seawater and olive mill wastewater

Abstract

According to the previous sections of this study, concerns arose with the use of lime and other traditional binders for soil stabilisation. Those so-called traditional additives were not always available, cost effective or environmentally friendly. It became necessary to evaluate the performance of alternative additives for soil stabilisation. As a start point, three non-conventional stabilisation agents were selected. The main criteria followed to select these additives were the following: a) an agent which had been slightly studied and which presented promising properties in previous studies; b) a cost-effective agent which could be found easily in many different regions; c) a non-hazardous industrial by-product which could add significant value to the soil and whose use in soil stabilisation could solve a significant waste management issue. Hence, magnesium hydroxide, seawater and olive mill wastewater were selected as candidates. Mixtures of soil and additives were prepared in different proportions. The performance of the additives was then studied from a physical and mineral point of view. In this chapter, the characterisation of the materials used is presented and the results obtained in the experimental tests are discussed. A set of conclusions about the use of these additives is drawn at the end of this chapter based on the results obtained in the experimental study. This chapter investigates the effects of non-conventional additives such as magnesium hydroxide, seawater and olive mill wastewater on a sample of bentonite, a clay of high plasticity and high swelling potential. The study is focused on the mineral changes promoted by the additives and their influence on the swelling potential and plasticity of bentonite. The main physical properties of soil, such as compaction, consistency, bearing capacity and swelling pressure are investigated. X-ray diffraction tests were carried out to analyse the evolution of the mineral composition of bentonite.

8.1 MATERIALS USED

The soil used in this study was a sample of bentonite. This soil was chosen for being a well known pure clay. It is a highly expansive material which undergoes significant volume changes when the environmental moisture varies.

The Bentonite was analysed in the laboratory, and found to have poor engineering properties. It was characterised for its low-load bearing capacity, high swelling potential and plasticity. X-ray diffraction (XRD) tests were conducted to analyse the mineral composition of the expansive soil. The main mineral constituent of the bentonite used in this study was Montmorillonite with small amounts of other clay minerals and quartz. Montmorillonite is a clay mineral within the group of smectites. The most expansive clay minerals belong to this group.

Xeidakis (1996a, 1996b) studied the effects of a solution of magnesium hydroxide on a suspension of swelling clays, finding that the swelling potential of clays was reduced. The effects of the magnesium hydroxide in a powder form when it is added to a soil following conventional mixing techniques deserves to be further studied.

The same can be said on the effects of seawater and olive mill wastewater. In the existing literature there are studies reporting the effects of the sodium chloride (**Singh and Das, 1999; Lees et al., 1982; El-Sekelly, 1987; Osula, 1993; Singh and Ali, 1978; Davoudi and Kabir, 2011; Wu et al., 2007**) and olive cake residue (**Attom and Sharif, 1998; Nalbantoglu and Tawqif, 2006**) on the physical properties of expansive soils. The chemical compositions of both seawater and olive mill wastewater involve elements such as calcium, potassium and magnesium which could promote a cation exchange when added to clay minerals. Hence the interest of a further research on the effect of this agents on the expansive soils.

Table 8.1 presents the engineering properties and chemical composition of the expansive soil, while **Figure 8.1** shows the X-ray diffraction pattern of the untreated soil. Two types of montmorillonite were found to be present in the bentonite, with their first peak located at 2-theta angles of 5.895° and 7.076°, respectively.

Magnesium hydroxide ($Mg(OH)_2$), which can be found in nature in the form of brucite, is also obtained nowadays from certain industrial by-products (**Seco et al., 2011a; Seco et al., 2011b; Xeidakis, 1996a; Xeidakis, 1996b; Garcia et al., 2004**). The magnesium hydroxide used in this study was a very pure commercial type supplied in a powder form. Previous works showed the reduction of swelling potential of clays, when introducing magnesium hydroxide solutions in clay suspensions (**Xeidakis, 1996a; Xeidakis, 1996b**). In this study, a powder of magnesium hydroxide was added to the soil following conventional mixing techniques to evaluate the effects of this agent on the expansive soil in the presence of water and emulate the real conditions of stabilisation process in construction sites.

Table 8.1

Properties of Bentonite

Chemical Composition (%)		*Physical Properties*	
SiO_2 (%)	50.23	Standard Proctor Maximum Dry Density (kN/m^3)	0.91
Al_2O_3 (%)	7.21	Standard Proctor Optimum Moisture Content (%)	66.10
Fe_2O_3 (%)	2.17	California Bearing Ratio (C.B.R.)	1.3
K_2O (%)	1.54	Liquid Limit (%)	294.9
MgO (%)	17.77	Plasticity Index (%)	250.9
Na_2O (%)	2.36	Swelling Pressure (kPa)	220
TiO_2 (%)	0.42	pH	9.5
MnO (%)	0.04	Particle size (μm)	75
P_2O_5 (%)	0.06		

The widespread availability of seawater makes it very interesting for its beneficial use in construction activities, always searching for a more sustainable construction process. To carry out this study, natural seawater from the Mediterranean sea was collected in Malaga, Southern Spain coast.

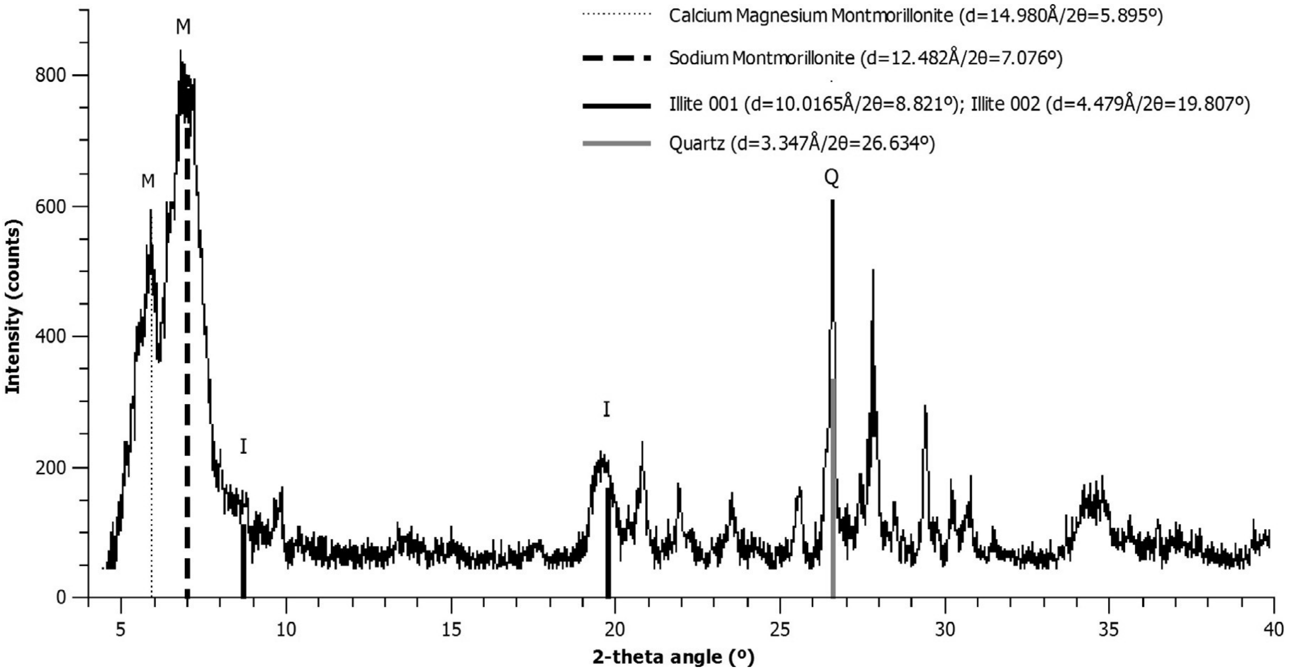


Fig. 8.1. XRD pattern of bentonite

Bruland (1983) carried out a thorough review on the chemical composition of seawater. More recently, **Hodge and Stetzenbach (1998)** published the average values of the elements contained in this natural resource. According to these previous works, the main elements which can be found within seawater are sodium (Na+) and chlorine (Cl^-), but the presence of other ions such as calcium, magnesium and potassium is not negligible. **Table 8.2** shows the main values of the chemical composition of seawater according to **Hodge and Stetzenbach (1998)**.

Table 8.2

Chemical composition of seawater according to Hodge (1998)

Element	Concentration (mol/kg)	Element	Concentration (mol/kg)
Cl	0.546	N	$3.0\cdot10^{-5}$
Na	0.468	Li	$2.5\cdot10^{-5}$
Mg	0.0532	Rb	$1.4\cdot10^{-6}$
S	0.0282	Mo	$1.1\cdot10^{-7}$
Ca	0.0103	Ba	$1.0\cdot10^{-7}$
K	0.0102	V	$3.0\cdot10^{-8}$
Br	$8.4\cdot10^{-4}$	As	$2.3\cdot10^{-8}$
Sr	$9.0\cdot10^{-5}$	Al	$2.0\cdot10^{-8}$
F	$6.8\cdot10^{-5}$	U	$1.4\cdot10^{-8}$

* Compounds with concentration > $1\cdot10^{-8}$

The olive mill wastewater (OMW) is a by-product of the olive oil industry. It is generated in centrifuges during the production of olive oil in the so-called two-phase extraction system. OMW used in this study, which had a humidity of 62%, was produced in Jaen (SE Spain) where the olive grove covers a total extension of 5,900 km^2. Previous studies on the characterisation of OMW concluded that it has a high content of potassium **(Alburquerque et al, 2004; De la Casa et al, 2012; Tortosa et al., 2012)**. **Table 8.3** shows the results of the study carried out by **Tortosa et al. (2012)** which established the chemical composition of the solid phase of the same OMW used in this study.

8.2 METHODOLOGY

The untreated bentonite soil was firstly characterised. Afterwards, several non-conventional stabilisation agents were added to the original untreated bentonite soil: magnesium hydroxide, seawater and olive mill wastewater (OMW). The mixtures were prepared with three different dosages of additive (5–10–15%).

Table 8.3

Chemical composition of OMW according to Garcia Tortosa et al. (2012)

pH = 5.38			
P (g·kg^{-1})	0.8	Cu (g·kg^{-1})	0.022
K (g·kg^{-1})	10.4	Mn (g·kg^{-1})	0.056
Ca (g·kg^{-1})	8.0	Zn (g·kg^{-1})	0.017
Mg (g·kg^{-1})	3.1	Pb (g·kg^{-1})	0.004
Na (g·kg^{-1})	0.3	Cr (g·kg^{-1})	0.019
S (g·kg^{-1})	1.1	Ni (g·kg^{-1})	0.055
Fe (g·kg^{-1})	2.4	Cd (g·kg^{-1})	nd

All mixes were prepared under the same conditions in a laboratory with a controlled temperature of 21 ± 1°C. After weighing the exact quantities of dry soil and dry additive, they were placed in a mixing tray and thoroughly mixed for at least 10 min. Water was then added to the mix in order to reach the optimum moisture content calculated for the original untreated soil (66%). The soil, additive and water were mixed in an industrial mixer for at least ten more minutes. Afterwards, the mixes were placed in a curing room for the required length of time. The curing room had the following conditions: temperature of 21 ± 1°C, humidity of 95 ± 3%. Two experiments were conducted. On one hand, all the specimens made were tested to determine their engineering properties. On the other hand, X-ray diffraction (XRD) tests were developed, obtaining the XRD pattern of the samples. All the tests were carried out at two curing times: 15 days and 30 days.

To determine the engineering properties of the original soil and the soils prepared in the laboratory, the following tests were carried out: standard Proctor compaction test, Atterberg limits test, California bearing ratio test and swelling pressure. **Figure 8.2** shows pictures of the equipment used: (a) soil and cell for swelling pressure test; (b) Proctor compaction moulds; (c) oedometers. The specific methodology of each test is included in **chapter 4**.

8.3 EXPERIMENTAL RESULTS

The values of maximum dry density (MDD) and optimum moisture content (OMC) in the treated samples are plotted in **Figures 8.3 and 8.4**, respectively.

In this study, a relationship was found to exist between the MDD of the sample and the percentage of additive present (linear correlation factor R^2 = 0.88). The values of MDD obtained at oldest samples with the highest dosage of additive were 1.09 Mg/m^3 (15%

$Mg(OH)_2$, 1.09 Mg/m^3 (15% seawater) and 1.03 Mg/m^3 (15% olive mill wastewater). Those values represent an increase over the original value (0.91 Mg/m^3). This increase in the MDD can be attributed to changes in the grain size since the additives used in this study had not high unit weights but they could promote changes in the particle size distribution.

Fig. 6.2. Pictures of lab equipment: (a) preparation of swelling pressure cell; (b) molds for standard Proctor test; (c) oedometers

Figure 8.4 shows that addition of magnesium hydroxide, seawater or olive mill wastewater yields to decreases in the OMC. There are studies in which the addition of fly ash (**Kolias et al., 2005**) or rice husk ash (**Basha et al., 2005**) promoted an increase of the optimum moisture content, probably due to the porous properties of ashes and the increasing amount of water required to achieve MDD when soil is mixed with fine grained materials (**Ahmed et al., 2011; Ahmed and Ugai, 2011**).

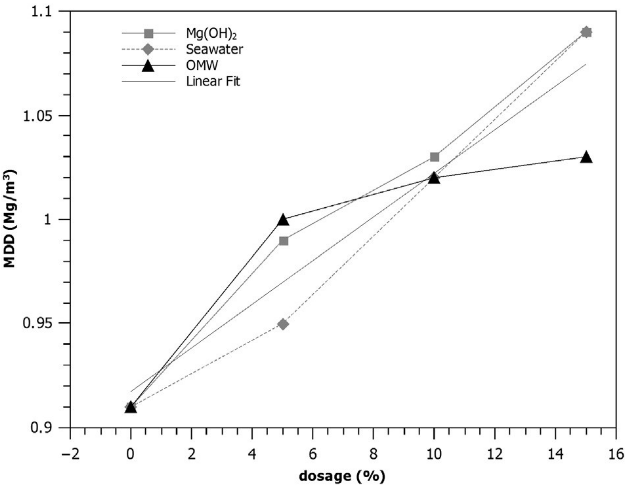


Fig. 8.3. Maximum dry density of treated samples after 30 days of curing

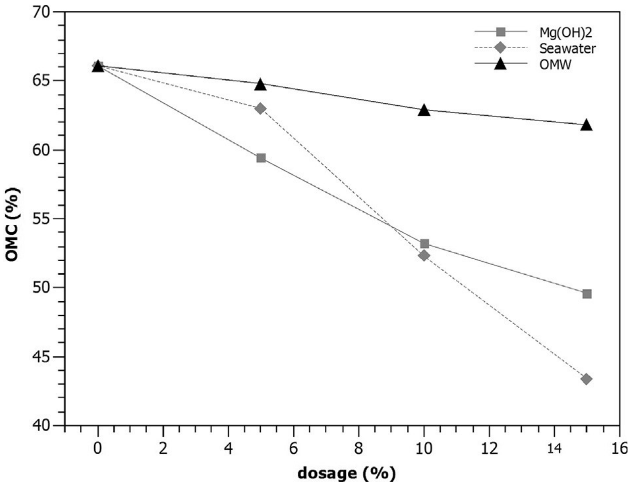


Fig. 8.4. Optimum moisture content of treated samples after 30 days of curing

Table 8.4

Results of Atterberg Limits tests

Combination	1st stage of curing			2nd stage of curing		
	5%	10%	15%	5%	10%	15%
Liquid limit (%)						
Mg-hydroxide	216.2	193	169.1	283.5	199.4	177.6
Seawater	253.7	234.7	206.6	238.6	215.6	193.9
OMW	211.5	199	178.9	205.5	197.6	163.5
Plastic limit (%)						
Mg-hydroxide	46.9	48.9	42	44	58.9	53.1
Seawater	35.6	34.4	33	39	30.9	32.6
OMW	41.5	42.7	43.7	41	46.8	34.6
Plasticity index (%)						
Mg-hydroxide	169.3	144.1	127.1	239.5	140.5	124.5
Seawater	218.1	200.3	173.6	199.6	184.7	161.3
OMW	170	156.3	135.2	164.5	150.8	128.9

The results of the Atterberg limits tests carried out for soil treated with magnesium hydroxide, seawater and olive mill wastewater are summarised in **Table 8.4**. The addition of magnesium hydroxide to bentonite clay rapidly produced a sharp decrease in PI from 250.9 to values under 170, as displayed in **Figure 8.5**. The PI values were proved to maintain a decrease tendency during the second stage of curing, decreasing slowlier than in the first stage. The addition of seawater to the sample of bentonite also yielded a decrease in PI of treated soils, although the final value obtained were not as low as the values achieved by adding magnesium hydroxide or olive mill wastewater **(Figure 8.5)**. This reduction of plasticity of the soil is consistent with the results of the study carried out by **Horpibulsuk (2012)** which show a reduction of plasticity in clays when the percentage of sodium chloride increases. According to **Horpibulsuk (2012)**, the reduction of plasticity is due to the compression of the diffuse double layer of kaolinite particles.

As seen in **Table 8.4** and **Figure 8.5**, the addition of olive mill wastewater (OMW) to the soil reduced the plasticity of the soil. Regarding the initial value of PI of bentonite (250.9), dosages of 5%, 10% and 15% of OMW produced reductions of 34%, 40% and 48% over the total value of the plasticity index, respectively.

Given the different plasticity of soils depending on their mineral composition **(White, 1949)**, the reduction of plasticity promoted by magnesium hydroxide, seawater and

OMW can be attributed to the alteration of the pH and the alteration in the concentration of calcium, magnesium and potassium, etc. which leads to transformations from expansive phases (smectite) to non-expansive clay minerals (such as illite). In addition, as explained by **Xeidakis (1996a)**, the adsorption of magnesium hydroxide by clays is similar to the adsorption of calcium hydroxide. This could have promoted a coarser particle size distribution and hence a reduction in plasticity in the samples treated with magnesium hydroxide.

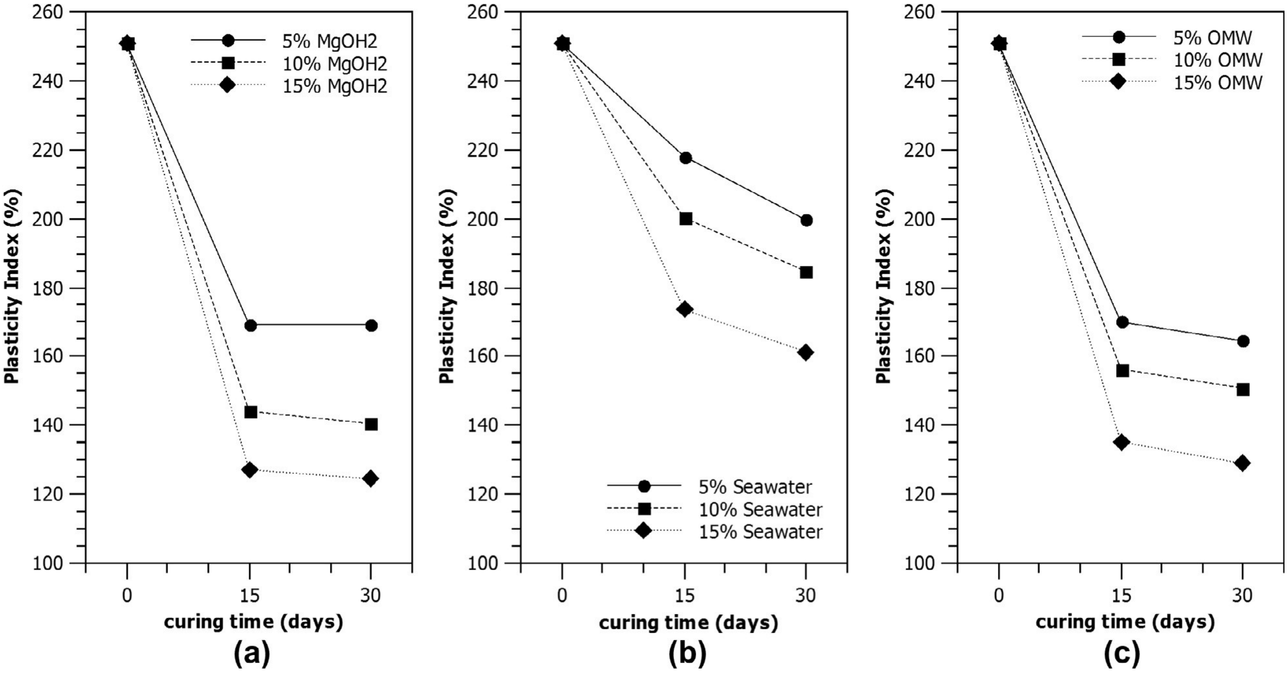


Fig. 8.5. Evolution of plasticity index: (a) magnesium hydroxide; (b) seawater; (c) OMW

In general, the treatments carried out in this work produced an increase in the CBR values of the samples (**Figure 8.6**). This is because the addition of magnesium hydroxide, seawater and olive mill wastewater produces a flocculation of particles, due to the variations of pH and the cation exchange induced in clays. As a result, the physical properties of bearing capacity and compaction are modified. Indeed a relationship was found to exist between the CBR and the MDD of samples in this study, with a linear correlation factor $R^2 = 0.72$. This means that, to some extent, the changes in CBR were related with the compaction properties of the soil. In the first stages of curing, low dosages (5%) of seawater and OMW produced a decrease in the CBR of the bentonite, which can be partially attributed to the formation of voids in the soil as a consequence of the flocculation of particles, without achieving a well graduated particle size distribution. For the rest of dosages, the CBR increased from the first stages of curing. Within curing time, the continuation of the flocculation can produce the readjustment of particles leading to a more compacted sample and hence a sample with higher bearing capacity.

The results obtained with OMW were slightly lower than those obtained with magnesium hydroxide and seawater. Given magnesium hydroxide and seawater provide a more alkaline environment, this could be behind this difference.

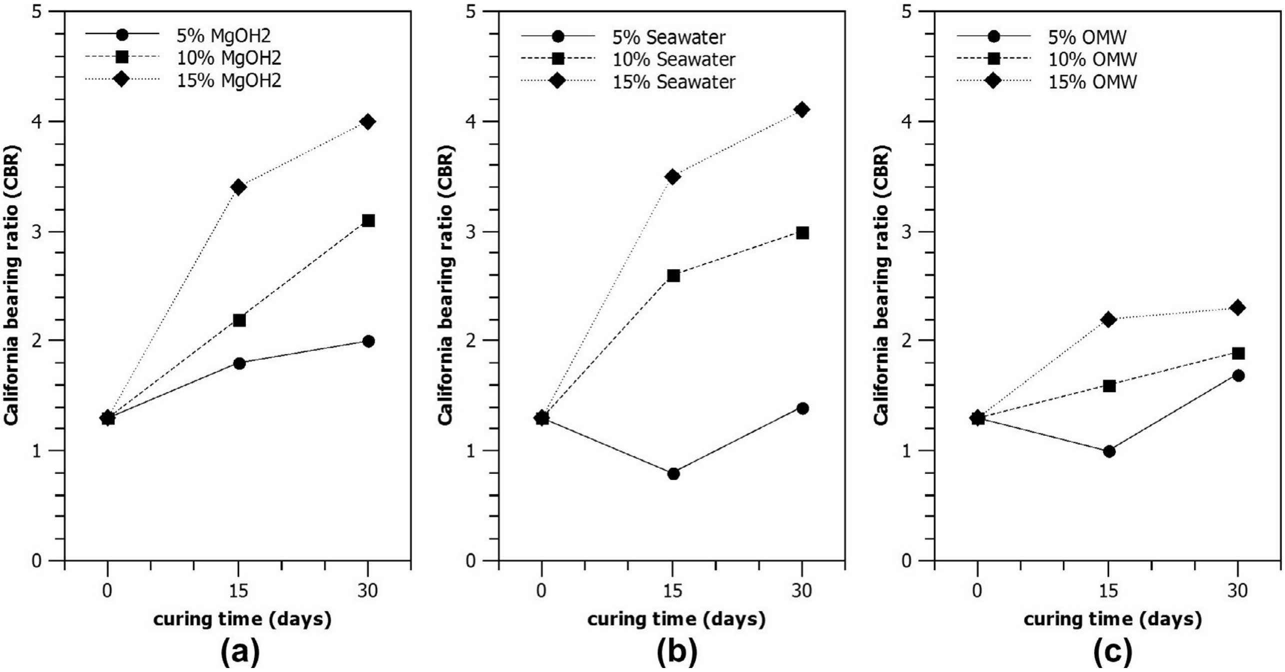


Fig. 8.6. Evolution of CBR in treated samples: (a) magnesium hydroxide; (b) seawater; (c) OMW

In Spanish standard PG-3 (**1976**), a minimum CBR of 3% is required to classify the soil as tolerable for its use in core of embankments. The initial California bearing ratio (CBR) of bentonite clay was 1.3%. After 30 days, the CBR values of all the samples prepared were higher than the initial one. The increase of CBR due to the addition of 10% and 15% of magnesium hydroxide and seawater was significant enough as to change the classification of the soil to tolerable.

Given the undoubted necessity of reducing the swelling potential of expansive soils, these results are the most significant of this study. As a first statement, it can be claimed that magnesium hydroxide, seawater and olive mill wastewater produced, when added to the expansive soil, a dramatic decrease in the swelling pressure of the bentonite. The evolution of swelling pressure is presented in **Figure 8.7**. The initial swelling pressure of the original bentonite was 220 kPa. After the addition of magnesium hydroxide, the swelling pressure obtained was 90 kPa (for 5% additive), 75 kPa (for 10% additive) and 40 kPa (for 15% additive). This means reductions of 59–81% in the swelling pressure of the soil. As it can be seen in **Figure 8.7**, the addition of different percentages of seawater and OMW produced reductions of the swelling pressure of bentonite ranging from 68% to 75% in the case of seawater and from 73% to 89% in the case of OMW. The final values of swelling pressure achieved for the OMW-treated samples were between 25 and 60 kPa, depending on the dosage of additive, and between 55 and 70 kPa in the case of samples treated with seawater.

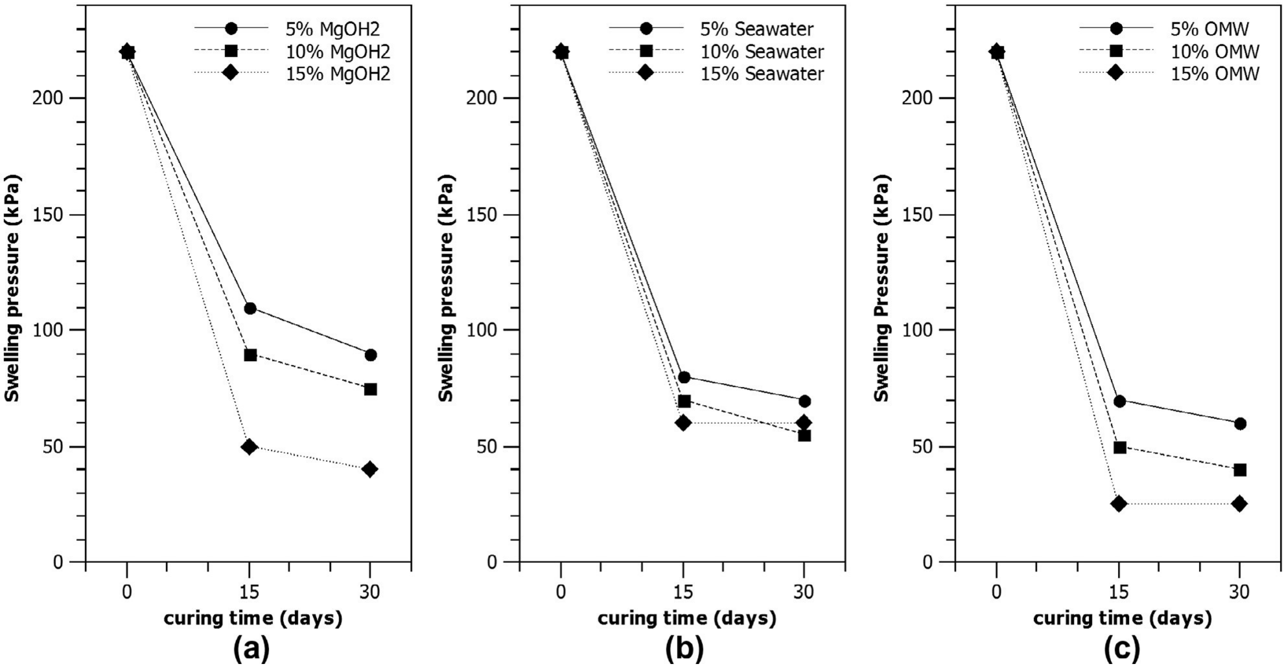


Fig. 8.7. Evolution of swelling pressure: (a) magnesium hydroxide; (b) seawater; (c) OMW

Using up to 3% of lime, **Akcanca and Aytekin (2012)** achieved reductions of 78% in swelling pressure of an expansive soil made of sand and bentonite, leading to final values of swelling pressure as low as 30 kPa. Despite the fact that the dosages of lime used in the mentioned work were low, these results are comparable to the results presented in this paper, especially taking into account that the expansive soil tested in the mentioned study had 50–80% content of sand which do not contribute to the swelling potential.

The reasons for such reductions in the swelling pressure of the treated samples are related to the weak stability of montmorillonite. **Elert et al. (2008)** studied the destruction of both expandable and non-expandable clay minerals in alkaline environments. Furthermore, **Drief et al. (2002)** pointed out that montmorillonite to illite conversion is easily enabled. Both magnesium hydroxide and seawater alter the pH of the soil, whilst OMW increases the concentration of potassium, both of them being main parameters to destroy montmorillonite, whose presence is responsible for the swelling potential of expansive soils. Therefore, all the additives tested had accurate properties to produce changes in the mineral composition of bentonite, and those changes are behind the reduction of swelling pressure of treated samples.

The effects of the additives tested in this study on the mineral composition of bentonite have been observed by X-ray diffraction (XRD) tests. **Figure 8.8** shows the pattern of treated samples with 15% of each additive in comparison with the XRD pattern of bentonite. In the pattern of bentonite, two different types of montmorillonite with their first peaks located at 2h angle of 5.895° and 7.076° were identified as calcium montmorillonite and sodium montmorillonite, respectively.

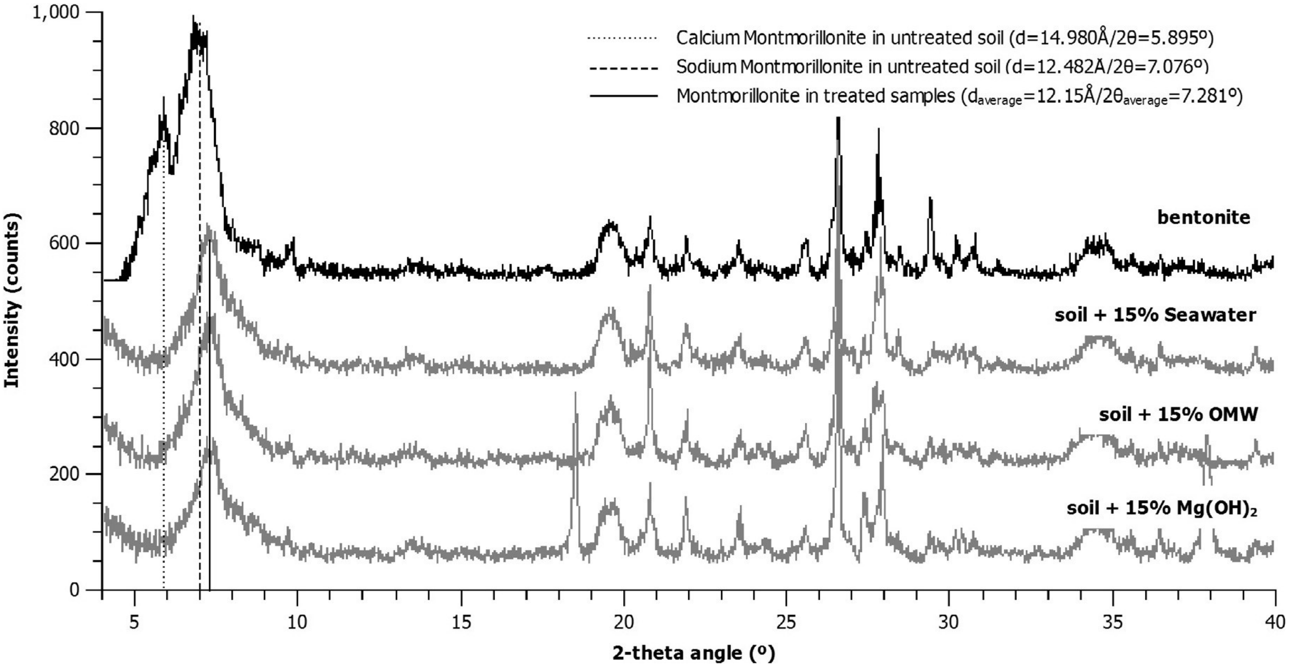


Fig. 8.8. XRD pattern of untreated bentonite and samples treated with 15% dosage of additives. Evolution of first peak of montmorillonite

According to **Moore and Reynolds (1997)**, the name of sodium, calcium or magnesium montmorillonite refers to the main interlamellar cation. The cation exchange capacities (CECs) of montmorillonite is high, and expansion takes place as water or some polar inorganic compound enters the interlayer space (**Moore and Reynolds, 1997**). It can be seen that in the treated samples, regardless of the additive used, these two peaks have turned into just one located at 2h angle of 7.281°. This peak, identified as the first peak of a montmorillonite, was not only shifted to the right in the pattern but it also had lower intensity of reflection. According to **Moore and Reynolds (1997)**, this entails a reduction in the amount of montmorillonite present in the sample along with a reduction in the d-spacing from 12.482 Å and 14.980 Å to just 12.15 Å.

The presence of imbalance electrical charge, sodium based clay minerals and CEC constitutes the swelling nature of bentonite (**Yilmaz, 2009**). Magnesium hydroxide, seawater and OMW changed the pH of the medium. Magnesium hydroxide and seawater provided magnesium and calcium. OMW altered the concentration of potassium.

Therefore, this shift of the first peak of montmorillonite can be attributed to the exchanges taken place among different cations, namely Ca^{2+}, Mg^{2+}, Na^{+} or K^{+}. This exchange can produce a reduction in the interlamellar spacing leading to more stable structures. **Table 8.4** shows the exact position of the first peak of montmorillonite in all the samples treated with magnesium hydroxide, seawater and OMW. The same effects previously described (lower intensity of reflection and right-shift of first peak of montmorillonite) can be observed for all the treated samples.

Therefore, on the basis of these results, it can be stated that the treated samples proved to have a lower amount of montmorillonite, and the montmorillonite present proved to have lower expansion potential. The additives used produced a noticeable change in the mineral composition of bentonite, tending to a reduction of montmorillonite present in the sample. Since montmorillonite is responsible for the swelling potential but also for the high plasticity of the soil, whilst illite is a non-expansive mineral, these changes let us understand the reductions in plasticity and swelling pressure observed in the treated samples and addressed in previous sections.

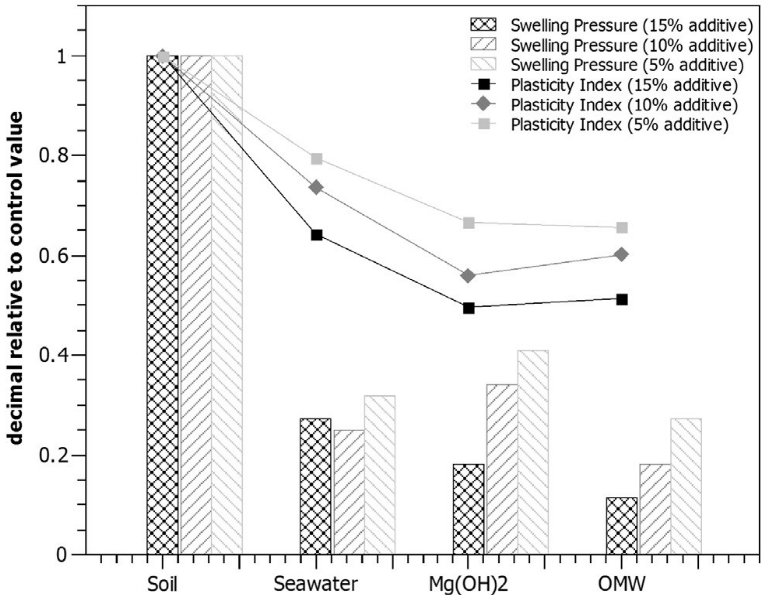


Fig. 8.9. Comparison of additives in terms of plasticity and swelling pressure of the treated soils

The non-conventional additives tested in this study (magnesium hydroxide, seawater and OMW) produced similar effects on the bentonite. First, a flocculation of particles responsible for the changes in the compaction and bearing capacity of the soil. Second, a change in the mineral composition of bentonite, tending to decrease the amount of montmorillonite present in the sample.

The alteration of the original conditions of formation of montmorillonite can easily enable the smectite to illite conversion. As stated by **Drief et al. (2002)**, this conversion is mainly controlled by the pH, temperature, curing time and access to potassium.

Magnesium hydroxide and seawater were able to alter the pH of the medium and provide cations for the cation exchange. Furthermore, OMW contained a high concentration of potassium. All these conditions altogether are behind the changes occurred in the mineral composition of bentonite. And the influence of the mineral composition on the physical properties of the soil was remarkable, since the reduction of montmorillonite was accompanied by a sharp decrease in the plasticity and swelling pressure of the treated samples.

In terms of comparison, the results obtained by addition of magnesium hydroxide, seawater and OMW were very similar, as seen in **Figure 8.9**. The final values of plasticity index achieved by treatment with magnesium hydroxide were slightly better than those obtained with the other additives. In terms of swelling pressure, OMW led to the greatest reductions. The addition of seawater also produced significant improvements in the plasticity and swelling potential of the soil. Hence, all the additives tested proved to have promising properties to reduce the plasticity and swelling potential of the additives while improving other physical properties such as compaction and bearing capacity. The effects of different combinations of these additives on the expansive soil deserve a further investigation.

8.4 CONCLUSIONS

In this study, different dosages of magnesium hydroxide, seawater and olive mill wastewater were added to a sample of bentonite. On the basis of the results of the geotechnical and mineral tests, the following conclusions can be drawn:

– The results of this study showed that the high swelling pressure and plasticity index of bentonite depend on its mineral composition, i.e., the amount of expansive phases such as montmorillonite present in the sample. Changes in the intensity and position of the first peak of montmorillonite and illite in the samples correspond with noticeable changes in plasticity and swelling pressure.

– The addition of magnesium hydroxide, seawater or olive mill wastewater to the bentonite promotes a reduction in the amount of montmorillonite present in the soil, partially due to the cation exchange capability enabled by introduction of cations (magnesium, potassium, calcium, sodium, etc.).

– In addition, the variation of the original conditions in which montmorillonite was formed can easily enable the montmorillonite to illite conversion. After addition of magnesium hydroxide, seawater or olive mill wastewater to the bentonite, this soil suffers sharp decreases in swelling pressure and plasticity index, due to the alterations promoted by the additives on conditions such as pH and concentration of different cations (magnesium, calcium, potassium, etc.)

– Along with the improvement on plasticity and swelling potential, the non-conventional additives tested in this study produced increases of maximum dry density and California bearing ratio (CBR) of the bentonite, whilst the optimum moisture content of the treated samples was always inferior to that of the original bentonite. The samples treated with at least 10% of additive reached CBR index above 3%, which is the value required for Spanish standard PG-3 to use a soil in core of embankments.

EXPERIMENTAL RESULTS
PART III

Chapter 9

Performance of the treatment: resources to assess the evolution of properties

Abstract

A review and state-of-the art of the current problem of soil stabilisation has been described in the introduction of this work. The experimental program is aimed at evaluating the current additives, methods and general approach of the soil stabilisation process. In part I of the experimental results, the investigation focused on the additives: properties, source, chemical composition... The content of free lime, the presence of oxides and elements which can be beneficial for the soil treatment or the potential for pozzolanic activity was studied. In Part II of the experimental results, some of the basics of the soil stabilisation theory have been tested, such as the benefits of using lime to treat all kind of soils under variable environmental conditions, or the performance of soil samples mixed with alternative additives. It has been stated that there are differences between the expected pozzolanic activity of an additive depending on the final purpose; e.g., the formation of ettringite in a cement mortar can promote the required strength gain with no or little negative effects, whilst the formation of ettringite in a soil to be used in earthworks and embankments can pose a significant problem due to the swelling potential of this mineral.

In this Part III of the experimental results, natural soils (clayey and marly soils) will be treated with both conventional (lime) and non-conventional additives: BFA and DA (those non-conventional additives have been previously studied in this work). A new insight on the methodology used to evaluate the performance of the additives used for soil stabilisation will be proposed: scanning electron microscopy and X-ray diffraction to assess the mineral modifications and the formation of new compounds, evolution of particle size as a feature in the performance of the treatment, effect of the presence of carbonates and sulphates in the soil... Furthermore, traditional techniques used to analyse those parameters will also be compared with new more sophisticated means which can become very useful in the assessment of the stabilisation of soils.

9.1 MATERIALS AND METHODS

For the study presented in this chapter, two different natural soils were used: a natural clayey soil from Alicun, Granada (Southern Spain) and a natural marly soil from the province of Jaen (Southern Spain). The main geotechnical properties of both soils are presented in **Tables 9.1a and 9.1b**. The geological setting of those soils was presented in chapter 3.

Table 9.1a

Properties of Clayey soil	
Optimum moisture OMC (%)	45
Max Dry Density (g/cm^3)	1.65
Liquid limit (%)	70
Plastic Limit (%)	28
Plasticity Index (%)	42

Table 9.1b

Properties of Marly soil	
Optimum moisture OMC (%)	19
Max Dry Density (g/cm^3)	1.6
Liquid limit (%)	58
Plastic Limit (%)	30
Plasticity Index (%)	28

The samples were treated with some of the additives which have been characterised in previous sections. The additives used were lime, dolomite ash (DA) and biomass fly ash (BFA). The lime used was a commercial quicklime classified as CL-90-Q according to Spanish standard UNE EN 459-1 **(UNE, 2002)**. The used BFA comes from the olive oil industry (BFA from thermal power plants in Linares, Spain). The origin of BFA and DA is explained in Chapter 9.

Three batches of treated samples were prepared.

- Batch 01: The marly soil is mixed with 5% and 10% of BFA, DA and quicklime (by weight of total sample). The samples are prepared in a laboratory and stored in a curing room with controlled conditions of humidity and temperature. Chemical characterisation and physical tests are carried out on samples of these soils after 7, 14, 21 and 42 days of curing.

- Batch 02: The clayey soil is mixed with 5% and 10% of BFA, DA and quicklime (by weight of total sample). The samples are prepared in a laboratory and stored in a curing room with controlled conditions of humidity and temperature. Chemical and physical tests are carried out on samples of these soils after 7, 14, 21 and 42 days of curing.

The samples prepared in each of the batches are presented in **Table 9.2**. The chemical composition of the additives used is presented in **Table 9.3**.

Table 9.2

Soils prepared and methodology used

<u>*BATCH 01*</u>		
Original Soil	*Additives*	*Concentration of additive (%)*
Marls	Lime	5, 10
Marls	Biomass fly ash (BFA)	5, 10
Marls	Dolomite ash (DA)	5, 10
<u>*BATCH 02*</u>		
Original Soil	*Additives*	*Concentration of additive (%)*
Clay	Lime	5, 10
Clay	Biomass fly ash (BFA)	5, 10
Clay	Dolomite ash (DA)	5, 10

Table 9.3

Chemical composition of the additives used

	Lime	*Biomass Fly Ash*	*Dolomite Ash*
Oxides			
SiO_2	1.5	7.42	0.84
Al_2O_3	≤ 0.1	1.78	0.36
Fe_2O_3	≤ 0.1	1.73	0.54
MnO	≤ 0.1	0.05	0.05
MgO	4.0	1.90	25.26
CaO	86.0	12.98	54.21
Na_2O	≤ 0.1	0.31	0.00
K_2O	≤ 0.1	44.39	0.05
TiO_2	≤ 0.1	0.16	0.02
P_2O_5	≤ 0.1	2.57	0.00

9.2 AVAILABLE TECHNIQUES FOR PARTICLE SIZE ANALYSIS

9.2.1 LASER DIFFRACTION FOR PARTICLE SIZE ANALYSIS

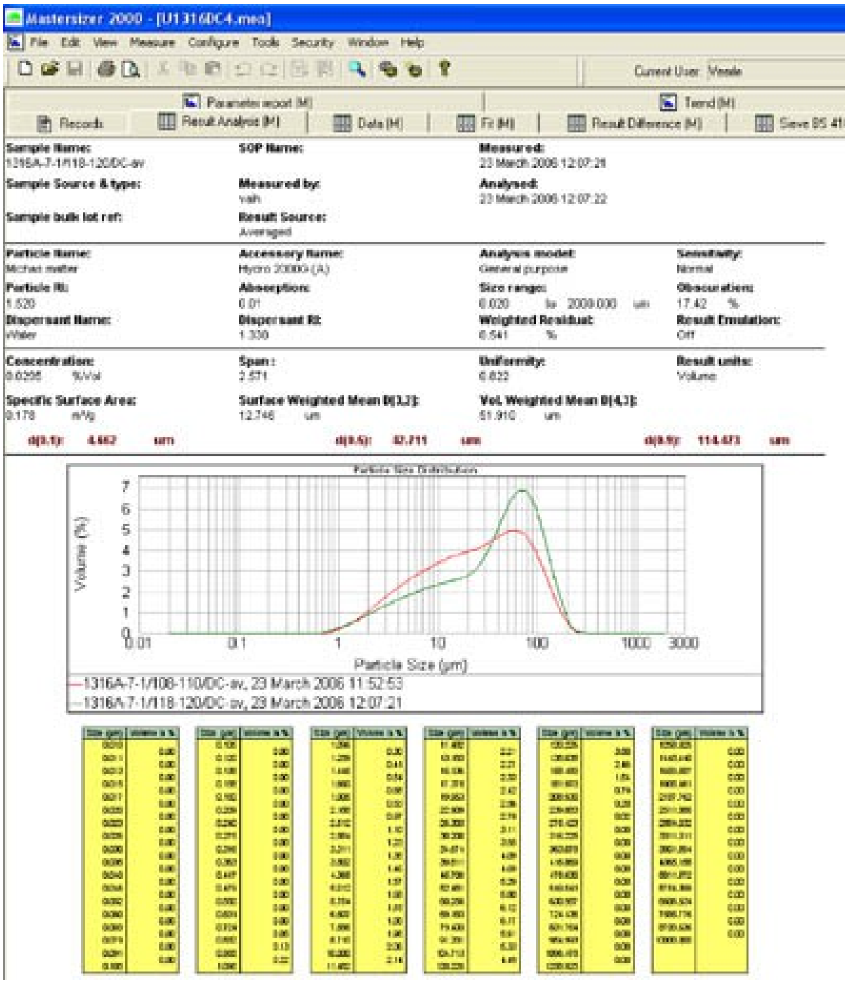
As explained in the methodology section (Chapter 4), Laser diffraction is a widely used particle sizing technique for materials ranging from hundreds of nanometers to several millimetres in size **(Figure 9.1**).

Figure 9.1 Particle size analyser (Malvern Mastersizer 2000LF). University of Granada

Laser diffraction measures particle size distributions by measuring the angular variation in intensity of light scattered as a laser beam passes through a dispersed particulate sample. Large particles scatter light at small angles relative to the laser beam and small particles scatter light at large angles, as illustrated below. The angular scattering intensity data is then analysed to calculate the size of the particles responsible for creating the scattering pattern, using the Mie theory of light scattering. The particle size is reported as a volume equivalent sphere diameter. Mie theory of light scattering is used to calculate the particle size distribution, assuming a volume equivalent sphere model.

Mie theory requires knowledge of the optical properties (refractive index and imaginary component) of both the sample being measured, along with the refractive index of the dispersant. Usually the optical properties of the dispersant are relatively easy to find from published data, and many modern instruments will have in-built databases that include common dispersants. For samples where the optical properties are not known, the user can either measure them or estimate them using an iterative approach based upon the goodness of fit between the modelled data and the actual data collected for the sample.

A simplified approach is to use the Fraunhofer approximation, which does not require knowledge of the optical properties of the sample. This can provide accurate results for large particles. However it should be used with caution whenever working with samples which might have particles below 50µm or where the particles are relatively transparent. **Figure 9.2** shows a report produced by the software Malvern™ Mastersizer2000 (by Malvern).



Sample Name: 1316A-7-1/118-120/DC-av
SOP Name:
Measured: 23 March 2006 12:07:21
Sample Source & type:
Measured by: vah
Analysed: 23 March 2006 12:07:22
Sample bulk lot ref:
Result Source: Averaged

Particle Name: Michael matter
Accessory Name: Hydro 2000G (A)
Analysis model: General purpose
Sensitivity: Normal
Particle RI: 1.520
Absorption: 0.01
Size range: 0.020 to 2000.000 um
Obscuration: 17.42 %
Dispersant Name: Water
Dispersant RI: 1.330
Weighted Residual: 0.541 %
Result Emulation: Off

Concentration: 0.0295 %Vol
Span : 2.571
Uniformity: 0.822
Result units: Volume
Specific Surface Area: 0.178 m²/g
Surface Weighted Mean D[3,2]: 12.746 um
Vol. Weighted Mean D[4,3]: 51.910 um

d(0.1): 4.662 um
d(0.5): 42.711 um
d(0.9): 114.673 um

—1316A-7-1/108-110/DC-av, 23 March 2006 11:52:53
—1316A-7-1/118-120/DC-av, 23 March 2006 12:07:21

Figure 9.2 Report of particle size analysis (Malvern Mastersizer 2000)

9.2.2 PARTICLE ANALYSIS OF FINE-GRAINED SOILS

Particle size distribution is a very relevant feature in the geotechnical characterisation of a soil. One of the main classifications of soils, adopted in many standards to evaluate their potential use in earthworks and embankments, is the soil classification by particle size, defining gravels, sands silts and clays. The so called fine-grained soils or soils with a very fine particle size distribution (clay) are commonly found to have a plastic behaviour. Furthermore, one of the most appreciated effects of the addition of traditional stabilisation agents (lime, cement, coal fly ash) to the soil, is the modification of the grain size tending to promote coarser distributions through flocculation and agglomeration of particles.

The most traditional method for measuring the particle size distribution of a soil (and develop a primary classification of that soil) is the sieving method. This method is usually included in all the geotechnical classifications carried out both in research works and actual site investigations.

The clay is characterised by its very fine grain size (< 2μm = 0.002mm). Due to the plastic nature of this soil, the sieving method is not able to provide an accurate result for clays (and marls, and other fine-grained soils). The particles physically stuck together can lead to a fake image of coarser particle size. Therefore, when it comes to clay, the most commonly used technique of particle size analysis is the hydrometer test method.

The hydrometer method is based on Stoke´s law governing the rate of sedimentation of particles suspended in water. The sample is treated with sodium hexametaphosphate to complex Ca^{2+}, Al^{3+}, Fe^{3+}, and other cations that bind clay and silt particles into aggregates. Organic matter is suspended in this solution. The density of the soil suspension is determined with hydrometer calibrated to read in grams of solids per litre after the sand settles out and again after the silts settles. Corrections are made for the density and temperature of the dispersing solution. The principal source of error in this procedure is the incomplete dispersion of soil clays. These clays are cemented by various chemical agents and organic matter into aggregates of larger size. Failure to effect complete dispersion results in low values for clay and high values for silt and sand. The rate of sedimentation is also affected by temperature and the density of the dispersing solution (**Bouyoucos, 1962**).

The use of laser diffraction methods to analyse the particle size of cements, pozzolans, etc. in materials engineering is wide-spread. The application of this technique in natural and treated soils could lead to a faster and more accurate system to evaluate the original particle size of fine-grained soils and the evolution of this relevant property in treated soils.

One of the main objectives of the study presented in this section is to provide a comparison between the techniques explained above. Therefore, the particle size distribution of a clayey soil (natural clayey soil from SE Spain) is studied through laser diffraction (using Malvern^{TM} Mastersizer2000, available at Centre of scientific instrumentation, University of Granada) and traditional hydrometer test. The compared results obtained are presented in **Figure 9.3**.

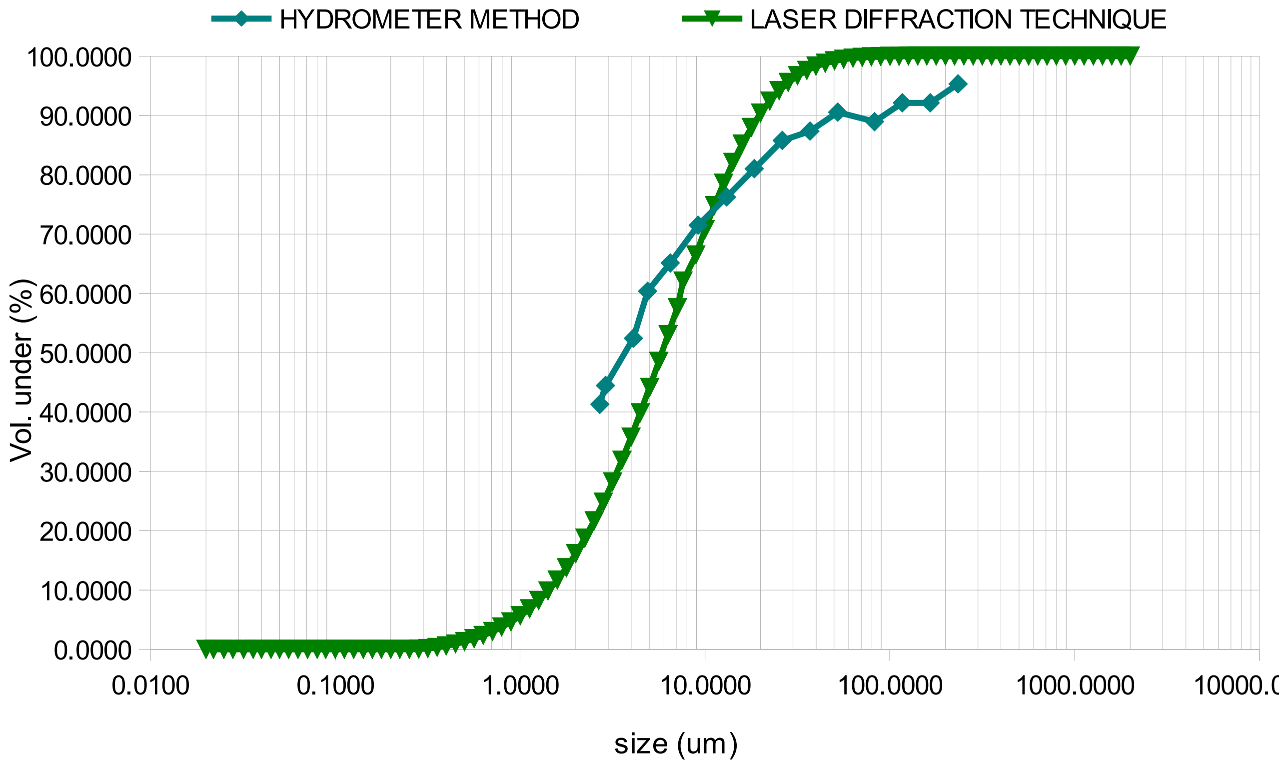


Figure 9.3 Comparison of particle size distribution obtained by laser diffraction and hydrometer method

As it can be observed in **Figure 9.3**, the results of particle size distribution obtained for the natural untreated clay soil studied are very similar using hydrometer test and laser diffraction. If the results of cumulative volume percentage obtained with both methods for each specific particle size are plotted in the same chart, a linear relationship with a $R^2 = 0.98$ (98% accuracy) is found **(Figure 9.4)**. However, the laser diffraction technique produced a more extensive, more accurate and faster result of the calculation.

After this comparison, the particle size distribution of the natural and treated samples of batches 01 and 02 were studied using laser diffraction. The accurate results allowed the comparison of the effects of different additives and dosages and the evolution of these results with time of curing.

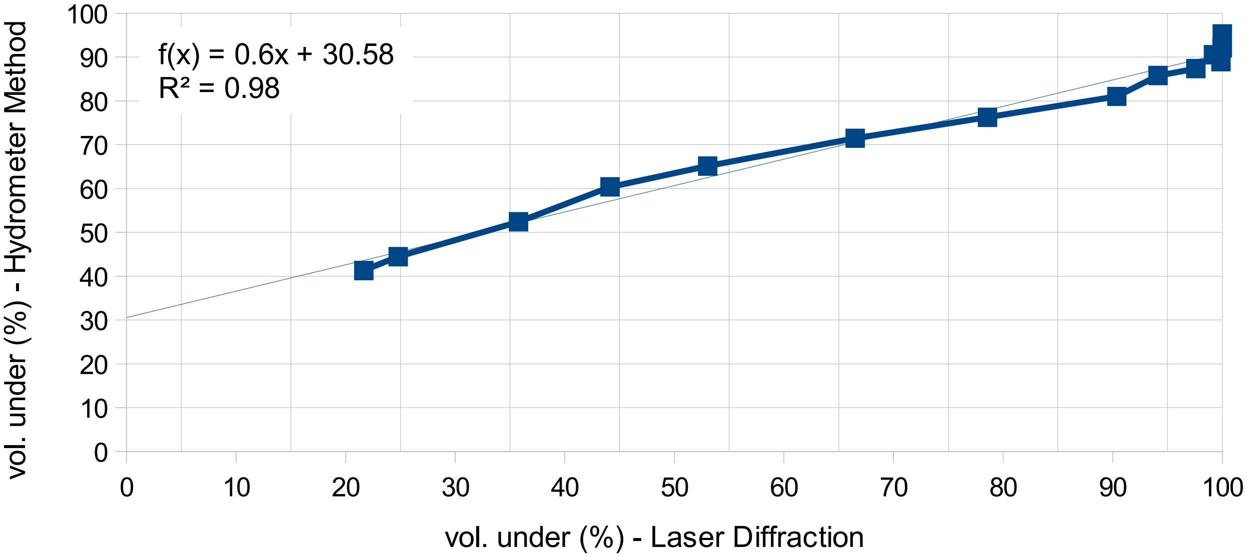


Figure 9.4 Linear relationship between hydrometer and laser diffraction results (obtained for natural clayey soil)

9.2.3 STUDY OF PARTICLE SIZE OF THE TREATED SAMPLES

The marly and clayey soils were mixed with 5% and 10% of lime, BFA and DA. The samples were cured during 42 days (6 weeks). The results of the particle size analysis by means of laser diffraction is presented in figures below. **Figure 9.5** shows the particle size distribution of the natural marly soil treated with lime. As it can be observed, the original marly soil showed a finer particle size distribution than the treated samples. The treatment with 10% of lime was more effective than the treatment with 5% of lime for the same time of curing.

Figure 9.6 and 9.7 show the particle size distribution of marly soil treated with biomass fly ash and dolomite ash, respectively. The use of BFA in dosages of 5% and 10% did not promote any significant change in the particle size of the soil treated. However, **Figure 9.10** showed a significant increase in the particle size when BFA was added to the bentonite soil (clayey soil). The treatment with dolomite ash promoted a shift to the right in the particle size distribution of the marly soil, showing a significant agglomeration of the soil particles.

A comparison of the performance of the three additives in terms of modification of particle size of the marly soil is presented in **Figure 9.8**. The effect of biomass fly ash, as mentioned above, was not remarkable. The modification of the particle size of the soil due to the addition of dolomite ash was comparable to that produced by the addition of lime.

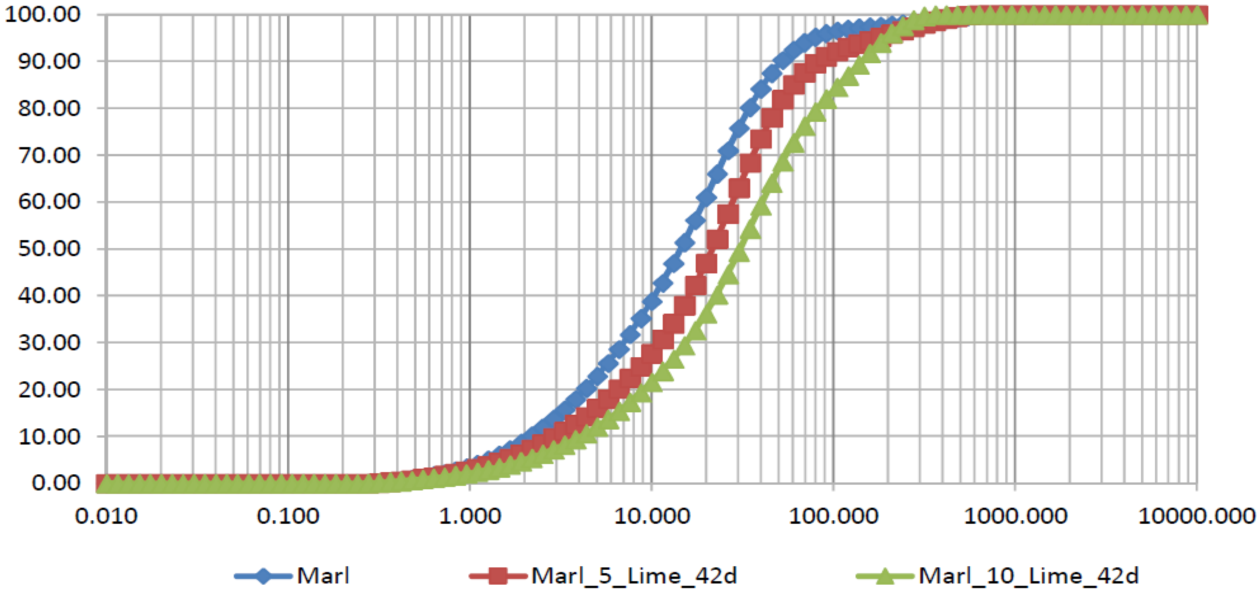


Figure 9.5. Particle size distribution of marly soils treated with 5%-10% of lime (42 days of curing)

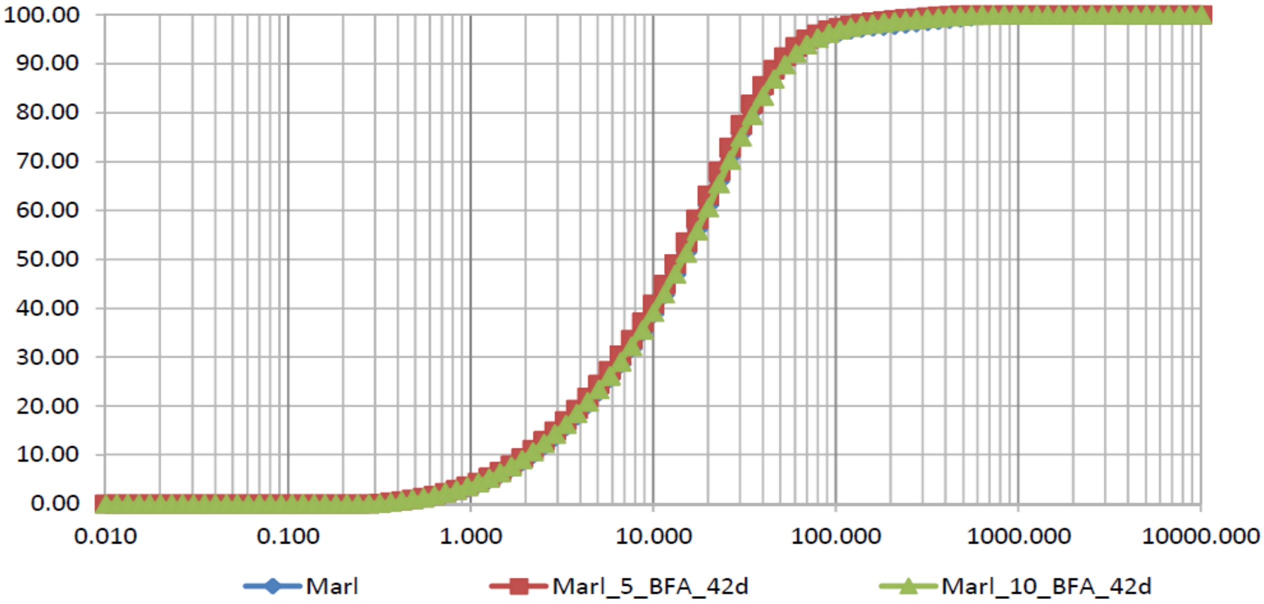


Figure 9.6. Particle size distribution of marly soil treated with 5%, 10% of BFA, after 42 days of curing

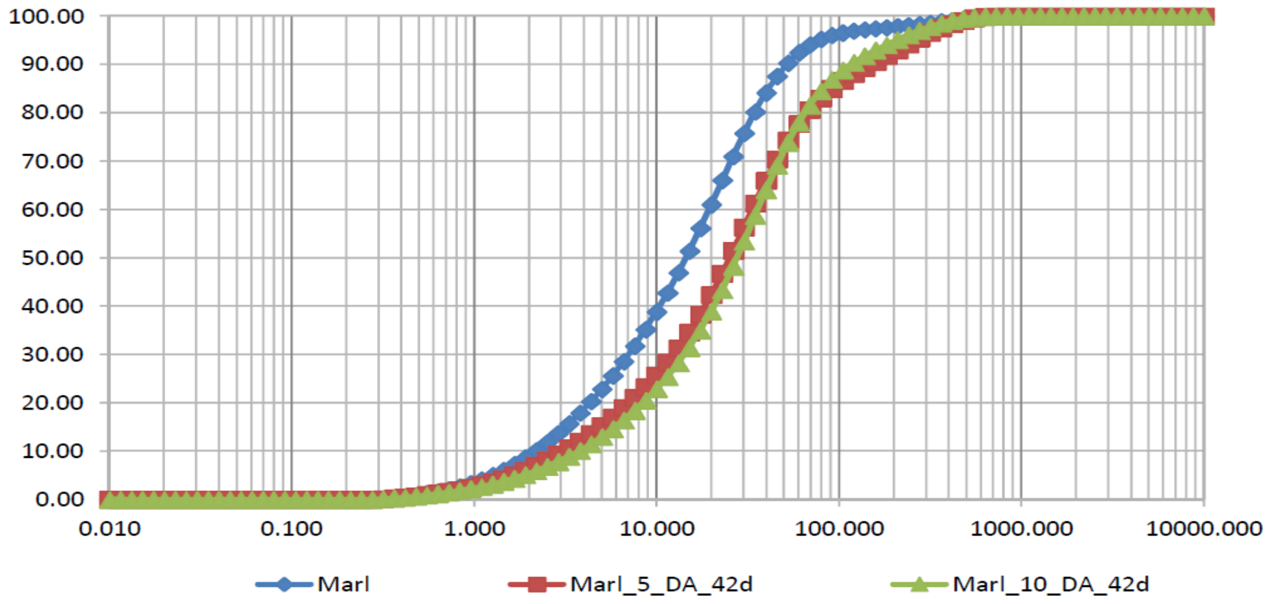


Figure 9.7. Particle size distribution of marly soil treated with 5%, 10% of DA, after 42 days of curing

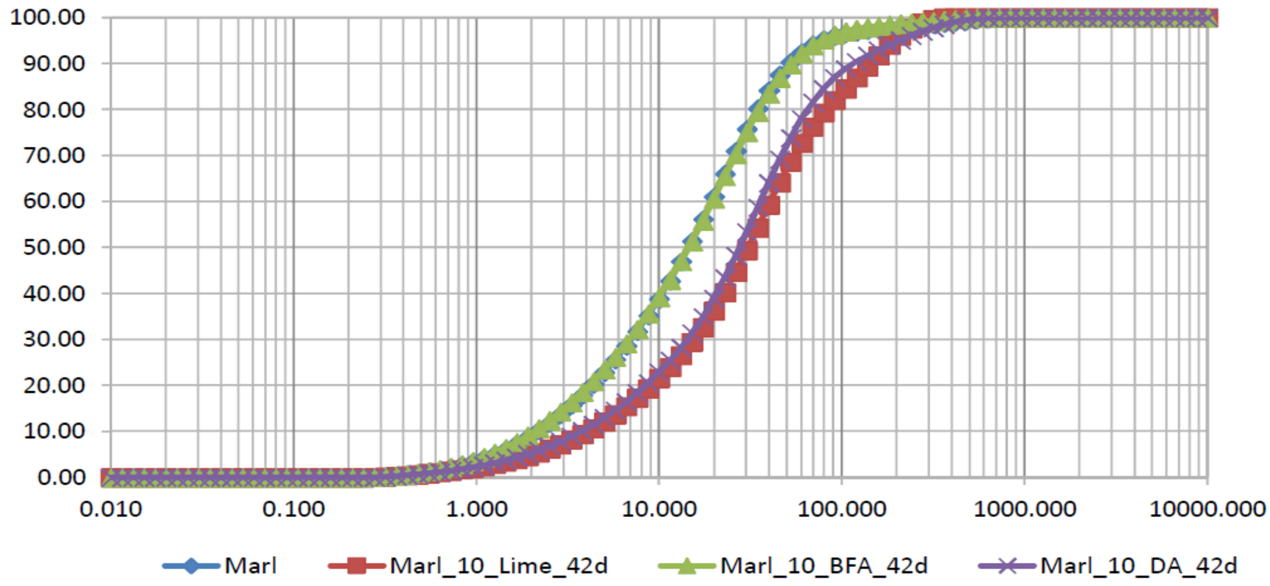


Figure 9.8. Particle size distribution of marly soil treated with 10% of lime, BFA and DA

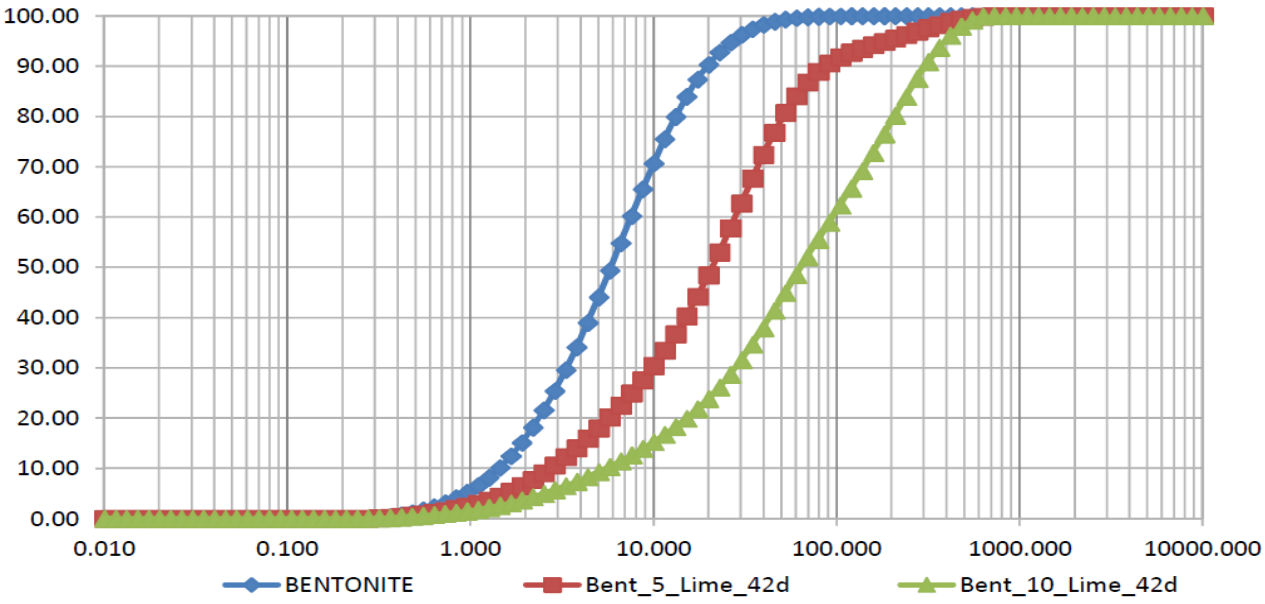


Figure 9.9. Particle size distribution of clayey soil treated with 5%, 10% of lime, after 42 days of curing

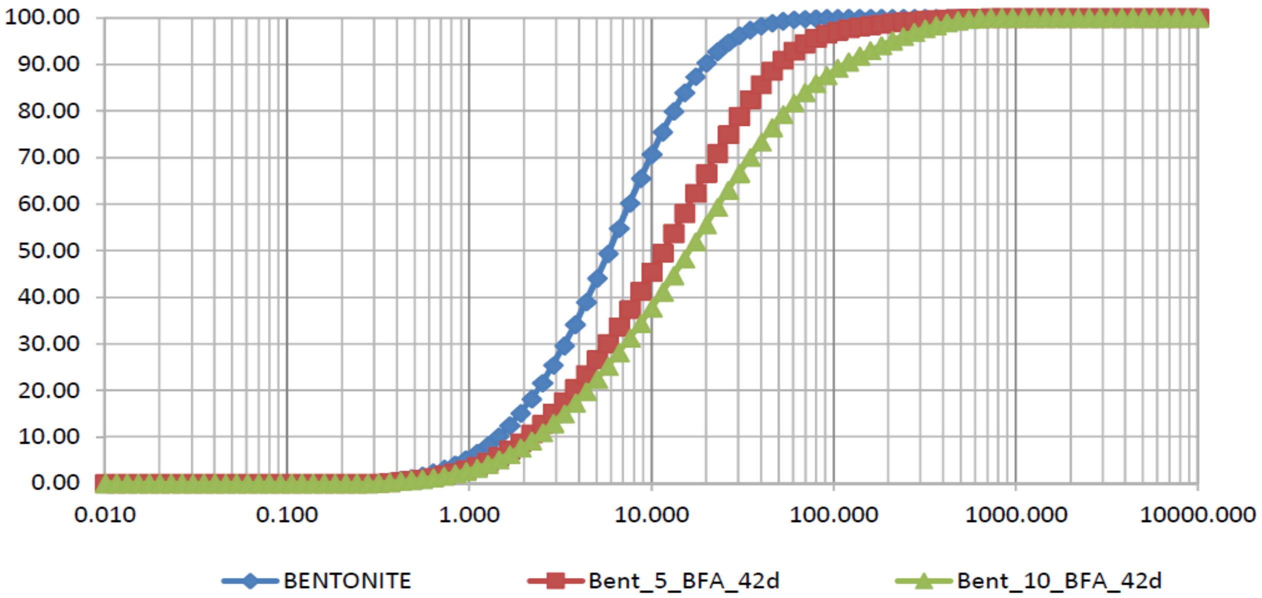


Figure 9.10. Particle size distribution of clayey soil treated with 5%, 10% of BFA, after 42 days of curing

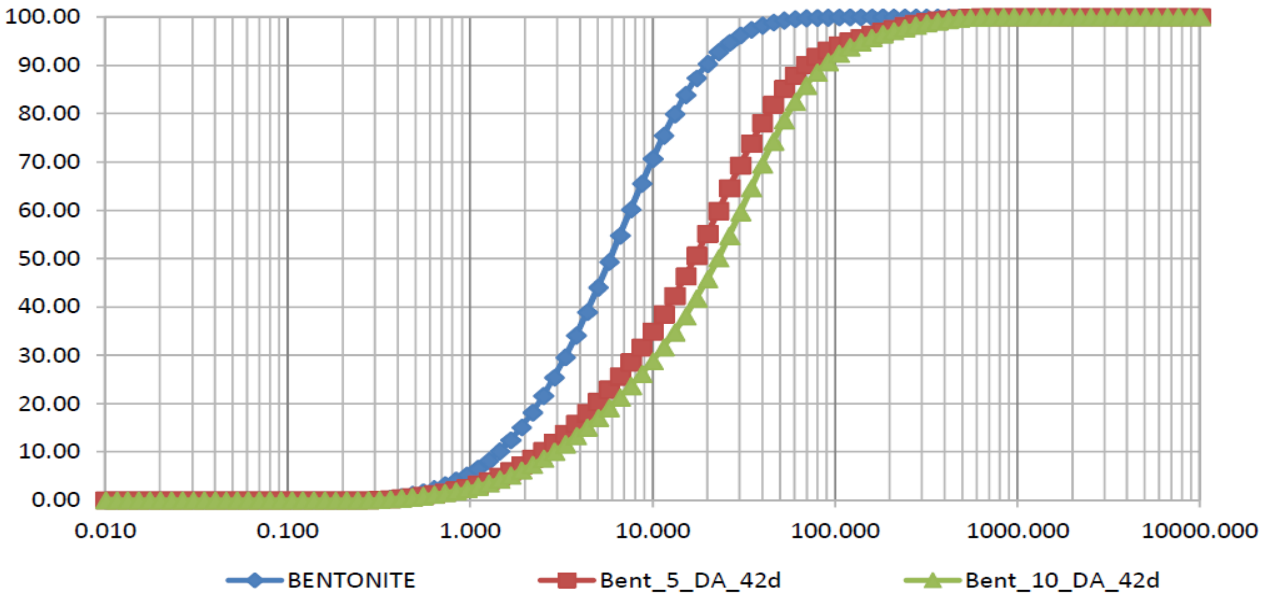


Figure 9.11. Particle size distribution of clayey soil treated with 5%, 10% of DA, after 42 days of curing

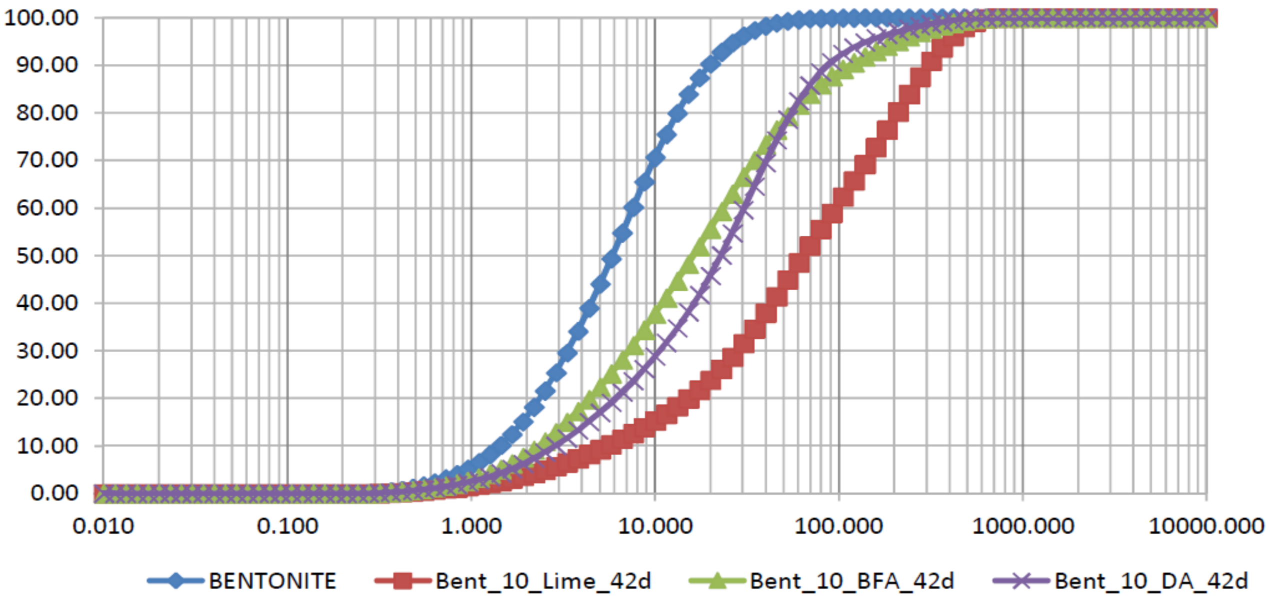


Figure 9.12. Particle size distribution of clayey soil treated with 10% of lime, BFA and DA

In general, the effect of the additives on the particle size of the soil samples were greater in the case of a bentonite soil (clayey soil). The effect of BFA in the bentonite soil is especially remarkable.

The original particle size distribution of the untreated clay revealed a very fine-grained soil (**Figure 9.9**). 90% of the soil sample was found to be under the 20µm size (0.02mm). The treatment with 5% and 10% of lime promoted a dramatic change in the particle size distribution. After 42 days of curing, only 50% of the 5%-lime treated soil sample was under 20µm size, whilst for the 10%-lime samples, the amount decreased to only 25%. For the mentioned treated samples, the percentile D90 (90% of the particles showing a size under D90) increased from 20µm in the untreated soil, to 100µm for the 5%-lime treated soil and 300µm for the 10%-lime treated soil.

The addition of 5% and 10% of BFA to the clayey soil promoted a dramatic modification of the particle size distribution after 42 days of curing. The changes promoted were more remarkable when the dosage of the additive was higher (**Figure 9.10**).

In the case of clayey soil, the addition of dolomite ash led to a coarser size distribution (**Figure 9.11**). As observed in all the samples in this study, the dosage was an important feature in promoting those modifications. In terms of comparison, the effect of lime was more remarkable than that promoted by addition of BFA and DA (**Figure 9.12**). The performance of BFA and DA was found to be very similar after 42 days of curing. The percentile D90 was found to be between 80 and 100µm when BFA or DA were added to the soil. However, the percentile D90 was 300µm for the 10%-lime treated soil.

The total range of particle size shown by the untreated clayey and marly soils was not significantly increased by the treatments with lime, biomass fly ash or dolomite ash. As mentioned above, the percentile D90 was found to shift to the right in the curve for almost all the treated samples studied. However, the percentile D10 (10% of the soil presenting particle size under D10) was also increased in significant proportions. This is a positive effect since it showed an efficient modification of the particle size distribution, affecting both the coarsest and the finest particles of the soil. This effect took place for both the treatment of clayey and marly soils.

In addition to it, the treatment with BFA was much more efficient in the treatment of bentonite soil than in the treatment of marly soil.

9.3 MEASURING THE CARBONATE AND SULPHATE CONTENT

9.3.1 AVAILABLE TECHNIQUES FOR MEASURING CARBONATE AND SULPHATE CONTENT

a) Chemical methods

There are traditional techniques to measure the carbonate and sulphate content of materials, based on chemical interactions. Some of those widespread methods used also to analyse the carbonate and sulphate content in soils are explained below.

The carbonate content can be measured using a Bernard calcimeter. This method is explained in the Spanish Standard **103200 (1993)**. The analysis of sulphate content in a sample of soils by precipitation of sulphates present in a barium chloride solution is described in Spanish standard **UNE 103201 (1996)**.

The Bernard calcimeter method for determination of carbonate content is based on the decomposition of carbonates by action of hydrochloric acid releasing carbon dioxide. The reaction is as follows:

$$CO_3M + 2ClH = Cl_2M + CO_2 + H_20$$

In this method, the apparatus is calibrated using pure calcium carbonate, taking pressure and temperature into consideration. The sample to be analysed is formed of 10 grams and must be oven-dried and crushed to particle size under 250µm prior to analysis. A hydrochloric acid solution is allowed to react with both the pure calcium carbonate and the sample to be analysed. The carbonate content is calculated from the comparison of the CO_2 released in the pure calcium carbonate and the test sample, as follows:

$$carbonate\,content\,(\%)=\frac{0.2*v}{W*V}*100$$

where,

0.2 represent the original weight of calcium carbonate used for calibration
v is the carbon dioxide released by test sample (in cm^3)
W is the weight of the studied sample
V is the carbon dioxide released in calibration (in cm^3)

The analysis of sulphate content by precipitation of sulphates present in a barium chloride solution is described in Spanish standard UNE 103201 (1996). The concentration of soluble sulphates is measured by means of a barium chloride solution following the formula:

$$MSO_4 + BaCl_2 = SO_4 + MCl_2$$

a) b) c)

Figure 9.13. a) Bernard calcimeter method; b),c) Sulphate precipitation method in laboratory

In this method, also a 10-gram-weight oven dried sample is used. The content of sulphate is obtained by comparison between the precipitated mass of barium sulphate promoted by the barium chloride solution used as a control and the precipitated mass of the test sample. The formula is as follows:

$$sulphate\ SO_3\ content(\%) = \frac{M_p * 0.34299}{M_m}$$

where,

M_p is the precipitated mass of $BaSO_4$
M_m is the precipitated mass of the test sample

b) Combustion analysis, infrared measurement

The chemical methods explained above present a major disadvantage: they are time consuming and their accuracy depends greatly on the technician developing the test. New sophisticated method to study the presence of carbon and sulphur in materials are commonly used in materials engineering.

The most significant method to analyse the carbon and sulphur content of a sample is the analysis by combustion and infrared measurements. With this technique, carbon and sulphur contained in inorganic substances, fuels and high-purity metals and electronics materials can be accurately measured. The use of an electrical furnace allows temperature raising functions and automated opening and closing function of the sample-insertion providing efficient combustion and highly precise analyses.

The principle of Carbon and Sulphur analysis is to oxidise the sample by heating in an Oxygen atmosphere to form CO_2, and SO_2, respectively, which are then measured by infrared detector. The heating is generally done in a high frequency induction furnace as this provides both speed and accuracy. The sample is placed in a ceramic crucible and an accelerator is often added to ignite the sample. The accelerator can also act as a flux to dissolve any oxide skins to improve melt fluidity, which allows the carbon and sulphur in the sample to be oxidised in a short time. It is imperative that complete combustion is achieved in order to gain accurate results. A wide range of accelerators are used and the choice depends on the base material being analysed. One feature of any accelerator is a low Carbon content and low Sulphur content.

A key issue in Carbon determination by this method is the purity of the Oxygen gas used. Typical impurities include CO_2 and CH_4. CH_4 can be oxidised at the temperatures involved in the test to $CO_2 + H_2O$, so for accurate results the effects of these impurities have to be resolved in the test method. This can be done in a number of ways, for example by using high purity Oxygen or by employing an in line gas purifier.

In the test a signal will be obtained that is not attributable to the sample. The cause is a combination of impurities in the gas, crucible and accelerator. The average contributions to the carbon and sulphur signal from these sources are determined to allow the sample contribution to be calculated. The inconsistency of impurity levels from these sources cannot be completely eliminated and this affects the ability to determine accurately to very low levels. As the method is relatively quick, repeat determinations are often requested to improve the accuracy of the determination and the average of several results taken.

Sulphur results can be affected if water is present in the sample. However, dealing with this issue is not straightforward. Residual water can be dealt with by heating the sample to 110°C for one hour. Crystalline water is less easy to deal with as the high temperatures required to dissociate it also mean combustion of the Sulphur.

A coefficient based on the molecular weight of carbonate and sulphate allows the calculation of the percentage of these compounds present in the samples from the values of carbon and sulphur encountered. The objectives of this study is to evaluate the suitability of these techniques to be applied for soils (both treated and untreated) comparing the results obtained with traditional chemical methods and those obtained by means of combustion analysers. The evolution of the carbonate and sulphate content with curing time is also studied.

9.3.2 Evolution of carbonate and sulphate content in the treated samples

As previously explained, in this study, the samples of marly soil were treated with 5% and 10% of lime, dolomite ash and biomass fly ash. The carbonate and sulphate content of the samples were studied at 7, 28 and 42 days of curing. The carbonate content was studied by two different methods: Bernard calcimeter and combustion analysis. For the latter analysis, an equipment of EMIA-8100 series (by Horiba™) equipped with electrical resistance furnace was used. This equipment was available at the Andalusian Institute of Earth Sciences (Granada, Spain).

The results obtained by the calcimeter method are expressed in percentage of carbonate. However, the results of the combustion analysis are originally expressed in percentage of carbon. The following assumption is considered: the carbon is present in the soil sample just in the form of carbonates. Then, the comparison between the molecular weight of carbonate (CO_3) and carbon (C) allows the adjustment of this calculation.

Table 9.4 shows the molecular weight of different compounds and the coefficient used to adjust the original results.

Table 9.4

Molecular weight of Carbon/Sulphur and Carbonate/Sulphate and Correlation Factors

Molecular weights	
Carbon	12.011
Sulphur	32.066
Carbonate CO_3	60.0092
Sulphate SO_4	96.0636
Calcite ($CaCO_3$)	100.0872
Gypsum ($CaSO_4 \cdot 5H_2O$)	226.218
Correlation Factors	
C-CO_3	4.996186829
S-SO_4	2.995808645
C-Calcite	8.332961452
S-Gypsum	7.054762053

The **Table 9.5** shows the results of carbon/sulphur content obtained for marly soils using combustion analysis. The evolution of the carbonate content in the treated samples of marly soil is presented in **Table 9.6a** (5% of additives) and **Table 9.6b** (10% of additives).

Table 9.5

Results of the combustion analysis of Carbon/Sulphur content in treated samples of marly soil

Sample	*Curing time (day)*	*C%*	*S%*	*CO_3%*	*SO_4%*
MARLY SOIL	0	4.681019	0.548931	23.38724547	1.644492235
Lime 5%	7	5.150959	0.455758	25.73515351	1.365363756
Lime 5%	28	4.642450	0.515921	23.19554678	1.545600592
Lime 5%	42	4.659121	0.279727	23.27783897	0.838008565
DA 5%	7	3.813431	0.180184	19.05261373	0.539796785
DA 5%	28	4.969640	0.372947	24.82924991	1.117277847
DA 5%	42	5.178586	0.250356	25.87318316	0.750018669
BFA 5%	7	4.873003	0.416609	24.34643341	1.248080844
BFA 5%	28	4.759707	0.498249	23.78038542	1.492658661
BFA 5%	42	4.627693	0.335774	23.12081881	1.005914652
Lime 10%	7	5.117363	0.297766	25.56730162	0.892049957
Lime 10%	28	4.630363	0.392454	23.13415863	1.175717086
Lime 10%	42	5.038650	0.292857	25.17403676	0.877343532
DA 10%	7	4.904232	0.280567	24.50245932	0.840525044
DA 10%	28	4.664218	0.312531	23.30330454	0.936283072
DA 10%	42	4.860822	0.305996	24.28557485	0.916705462
BFA10%	7	4.888121	0.370903	24.42196576	1.111154414
BFA 10%	28	4.876304	0.588245	24.36292582	1.762269456
BFA 10%	42	3.439939	0.085219	17.18657792	0.255299817

Table 9.6a

Evolution of carbonate content for samples of marly soil treated with 5% additive

Curing time (days)	*5% Lime Concentration of CO_3 (%)*	*5% Dolomite Ash Concentration of CO_3 (%)*	*5% Biomass Fly Ash Concentration of CO_3 (%)*
0	23.3872455	23.3872455	23.3872455
7	25.7351535	19.0526137	24.3464334
28	23.1955468	24.8292499	23.7803854
42	23.277839	25.8731832	23.1208188

Table 9.6b

Evolution of carbonate content for samples of marly soil treated with 10% additive

Curing time (days)	*10% Lime Concentration of CO_3 (%)*	*10% Dolomite Ash Concentration of CO_3 (%)*	*10% Biomass Fly Ash Concentration of CO_3 (%)*
0	23.3872455	23.3872455	23.3872455
7	25.5673016	24.5024593	24.4219658
28	23.1341586	23.3033045	24.3629258
42	25.1740368	24.2855749	17.1865779

The evolution of the sulphate content in the treated samples of marly soil is presented in **Table 9.7a** (5% of additives) and **Table 9.7b** (10% of additives).

Table 9.7a

Evolution of sulphate content for samples of marly soil treated with 5% additive

Curing time (days)	*5% Lime Concentration of SO_4(%)*	*5% Dolomite Ash Concentration of SO_4(%)*	*5% Biomass Fly Ash Concentration of SO_4(%)*
0	1.64449224	1.64449224	1.64449224
7	1.36536376	0.53979678	1.24808084
28	1.54560059	1.11727785	1.49265866
42	0.83800856	0.75001867	1.00591465

Table 9.7b

Evolution of sulphate content for samples of marly soil treated with 10% additive

Curing time (days)	*10% Lime Concentration of SO_4(%)*	*10% Dolomite Ash Concentration of SO_4(%)*	*10% Biomass Fly Ash Concentration of SO_4(%)*
0	1.64449224	1.64449224	1.64449224
7	0.89204996	0.84052504	1.11115441
28	1.17571709	0.93628307	1.76226946
42	0.87734353	0.91670546	0.25529982

The evolution of carbonate and sulphate content in the treated samples of marly soil with 5 and 10% of additives is presented in **Figures 9.14 to 9.17**.

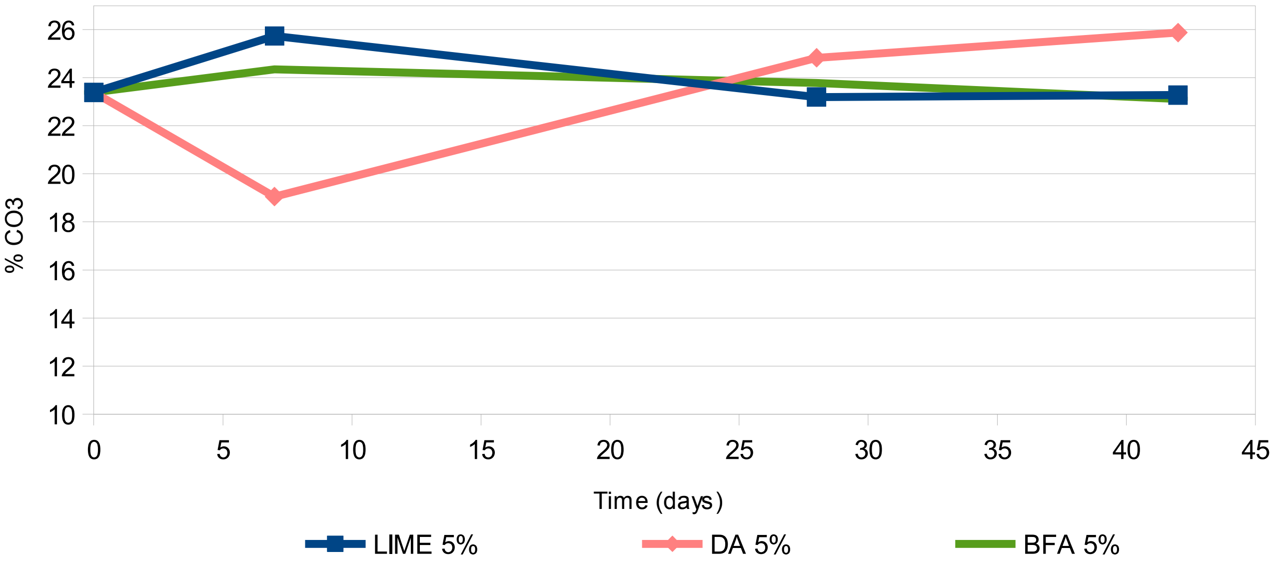


Figure 9.14. Evolution of carbonate content in samples of marly soil treated with 5% additives

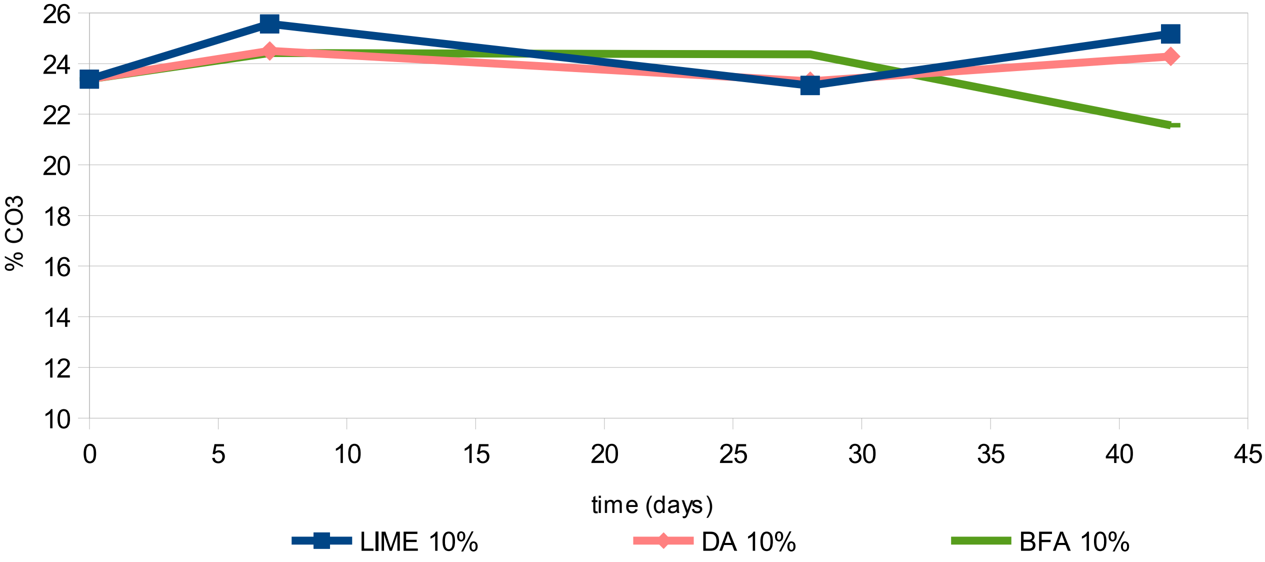


Figure 9.15. Evolution of carbonate content in samples of marly soil treated with 10% additives

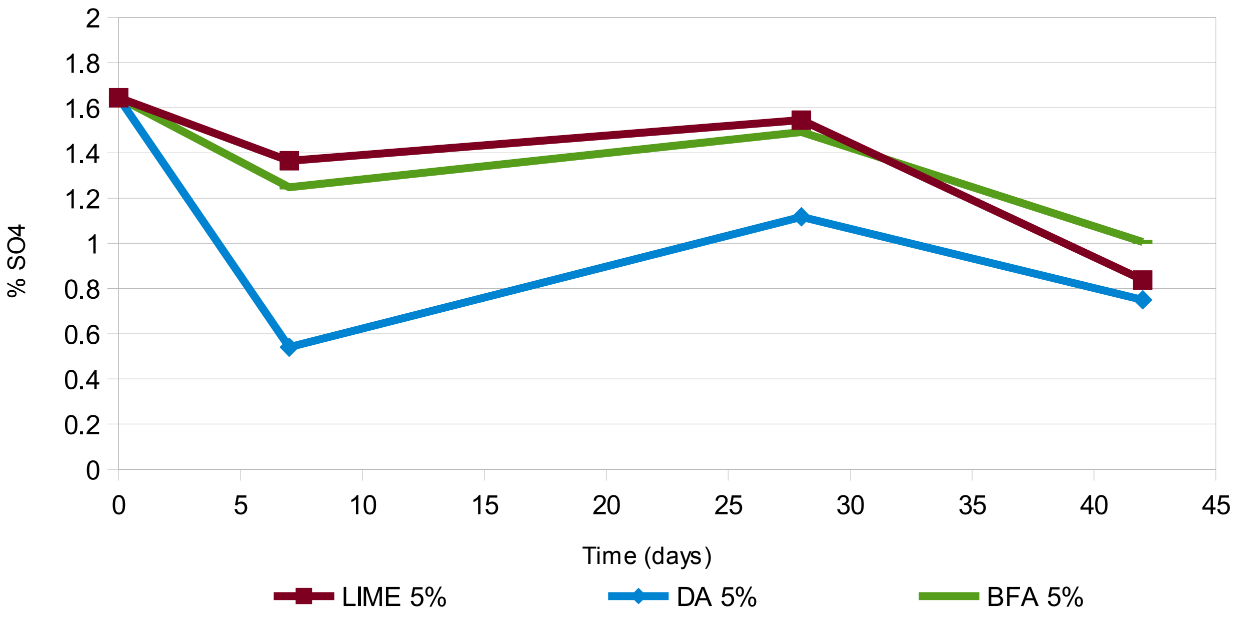


Figure 9.16. Evolution of sulphate content in samples of marly soil treated with 5% additives

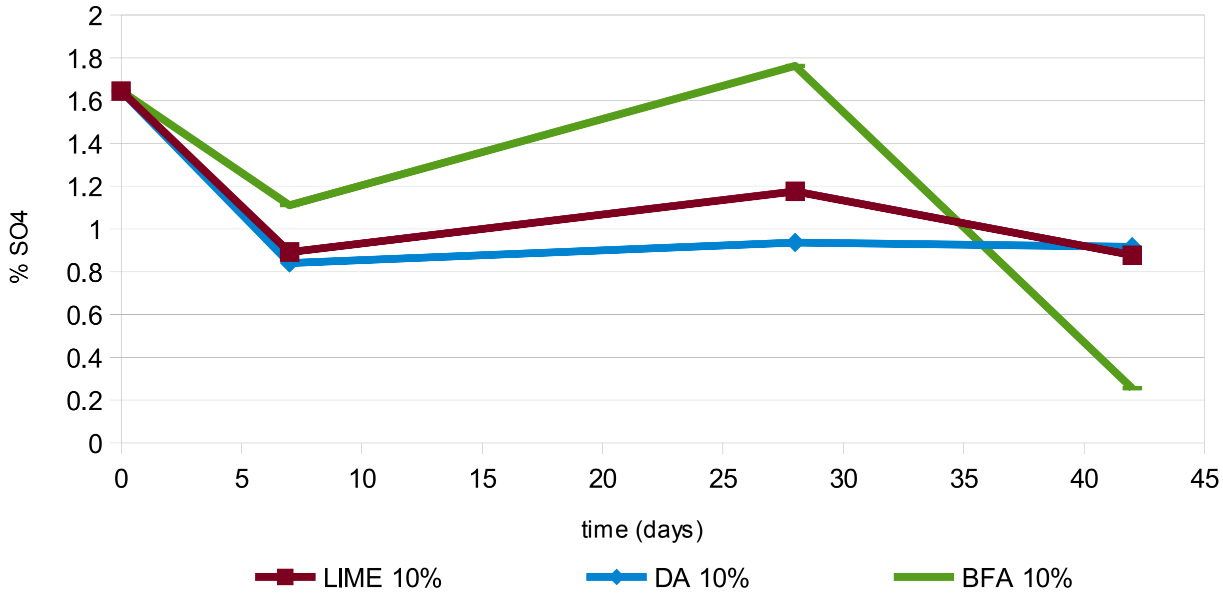


Figure 9.17. Evolution of sulphate content in samples of marly soil treated with 10% additives

The carbonate content of the treated samples of marly soil and its evolution with curing time was also studied with the Bernard calcimeter. The results obtained are shown in **Figure 9.18 and 9.19**.

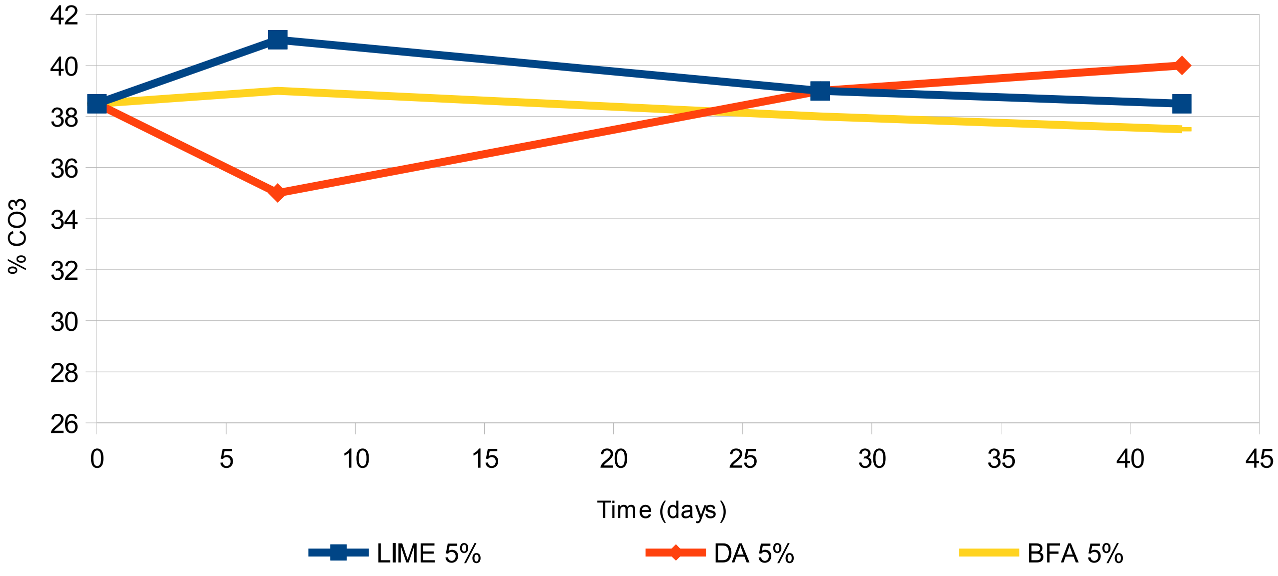


Figure 9.18. Evolution of carbonate content in marly soils measured by Bernard Calcimeter

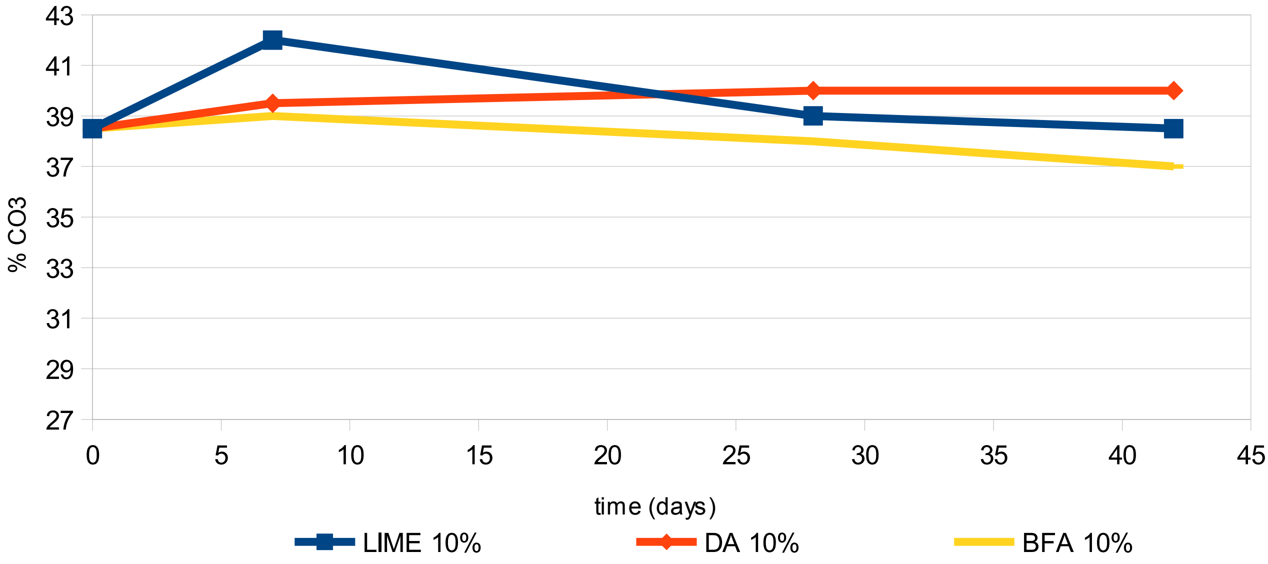


Figure 9.19. Evolution of carbonate content in marly soils measured by Bernard Calcimeter

The comparison between the **Figures 9.14 and 9.18** and between **Figures 9.15 and 9.19** shows that both method obtained similar results for the evolution of carbonate content. The trend lines of the carbonate content for different dosages and additives encountered with both methods were the same.

The first method, the combustion analysis and infrared measurement, present clear advantages for analysis of carbonate and sulphate content. The relationship between the measurements obtained by means of combustion analysis and traditional Bernard calcimeter were found to be highly comparable. However, the results obtained with the more sophisticated method of combustion analysis were obtained in a much shorter time. The accuracy of the method is beyond doubt and it does not depend on the technician as much as the traditional chemical methods do. For all the explained above, the use of combustion analysis to study the carbonate content and sulphate content of soil samples (treated and untreated) is highly recommended.

In general, the final amount of carbonate in the samples remained constant after 42 days of curing, whilst the amount of sulphates within the treated soils was found to decrease after 42 days of curing.

Despite the fact that carbonate content was similar at the beginning and end of the treatment, significant modifications of this amount were found along the curing. When samples of soil were treated with 5% of lime, the carbonate content increased in the first 7 days of curing. This increase was more remarkable when 10% of lime was added to the soil. Nevertheless, the carbonate content in the marly soil decreased significantly when 5% of DA was added, although this decrease was not recorded for higher dosages of this additive. In terms of evolution, the BFA showed a poorer performance, not affecting the carbonate content significantly along the curing time. However, the carbonates seem to decrease slightly when BFA is added in both 5% and 10% dosages.

The sulphate content of the original marly soil was not very high. However, the presence of sulphates in the form or gypsum and other mineral formations can affect significantly the performance of the treatment. The sulphate content in the tested soils seemed to decrease clearly with time. The original amount of sulphate present in the untreated marly soil was reduced to 51%, 46% and 61% by addition of 5% of lime, DA and BFA, respectively. The addition of higher amounts of these additives also promoted reductions in the final sulphate content of the treated samples, with this amount being reduced to 53%, 56% and 15%, respectively, by addition of lime, DA and BFA. The reduction in the sulphate content promoted by the addition of BFA is particularly remarkable. This reduction took long time to be revealed for the 5%-BFA treated samples.

9.4 PLASTICITY OF THE SAMPLES

The samples of the batch 01 (marly soil) and batch 02 (clayey soil) were subjected to Atterberg limits tests to study their consistency. The plasticity of the soils is a key feature in order to classify the soils and to determine whether they could be used for engineering purposes. The consistency is also related to other remarkable properties of the soil, such as the swelling potential and particle size. A plastic behaviour is commonly attributed to fine-grained soils. A high plasticity index (PI=LL-PL) shows a significant capacity of the soil to adsorb water without changing its state. Therefore, when a treatment is carried out to stabilise the soil, a reduction in plasticity is usually accompanied by a reduction in the swelling potential and an increase in the strength parameters of the soil.

The evolution of the plasticity index in the marly and clayey samples prepared in this study is shown in **Figures 9.20 to 9.23**. The results showed a significant reduction of the plasticity index (PI) due to the addition of the selected additives (lime, biomass fly ash and dolomite ash).

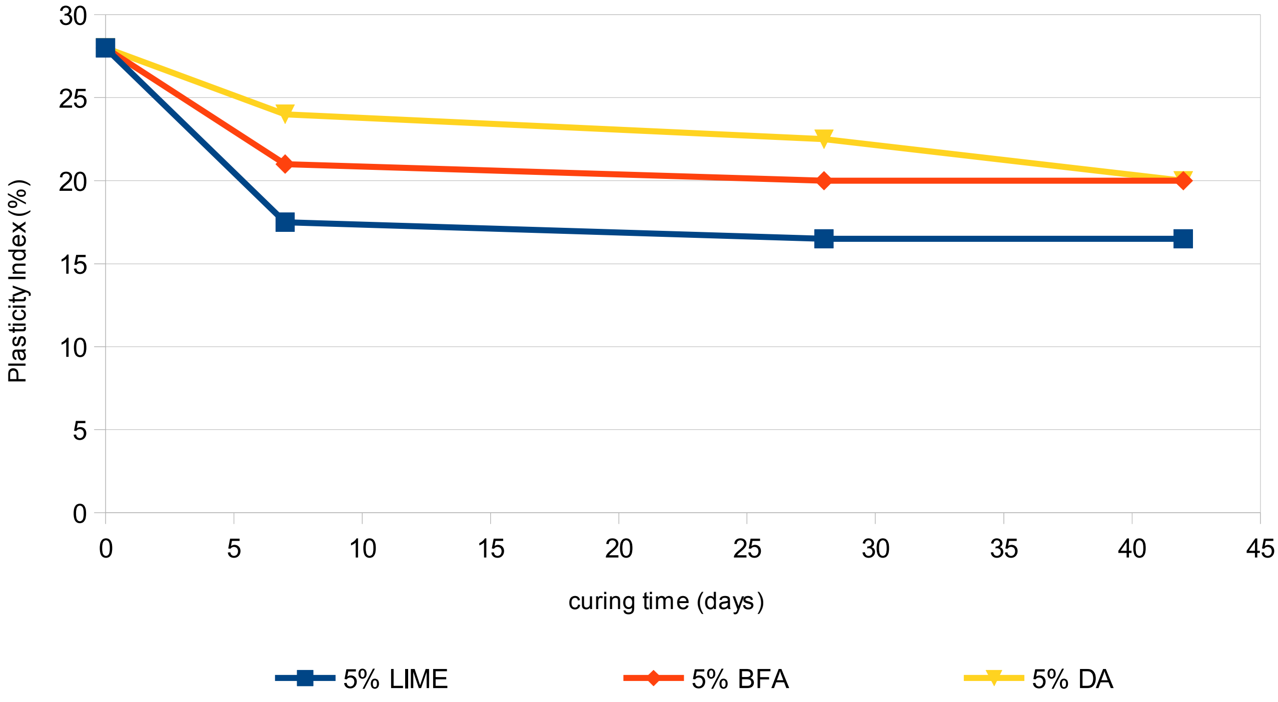


Figure 9.20. Evolution of Plasticity Index (PI) in marly soils treated with 5% additives

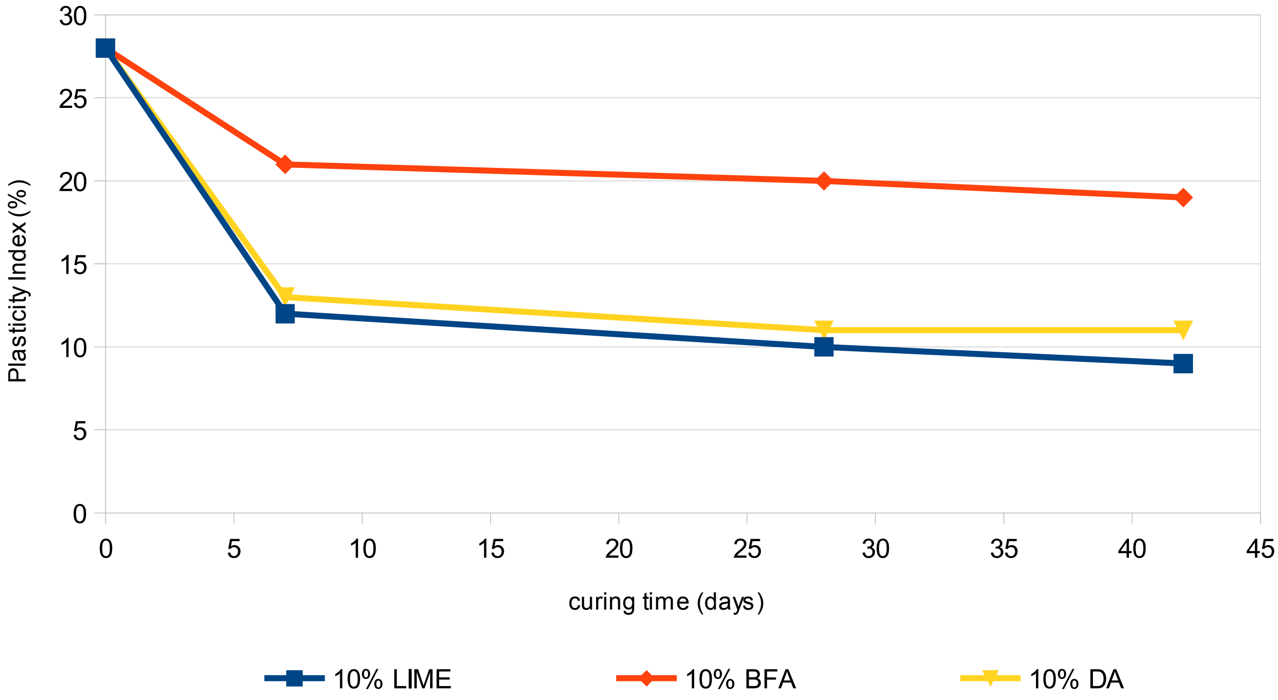


Figure 9.21. Evolution of Plasticity Index (PI) in marly soils treated with 10% additives

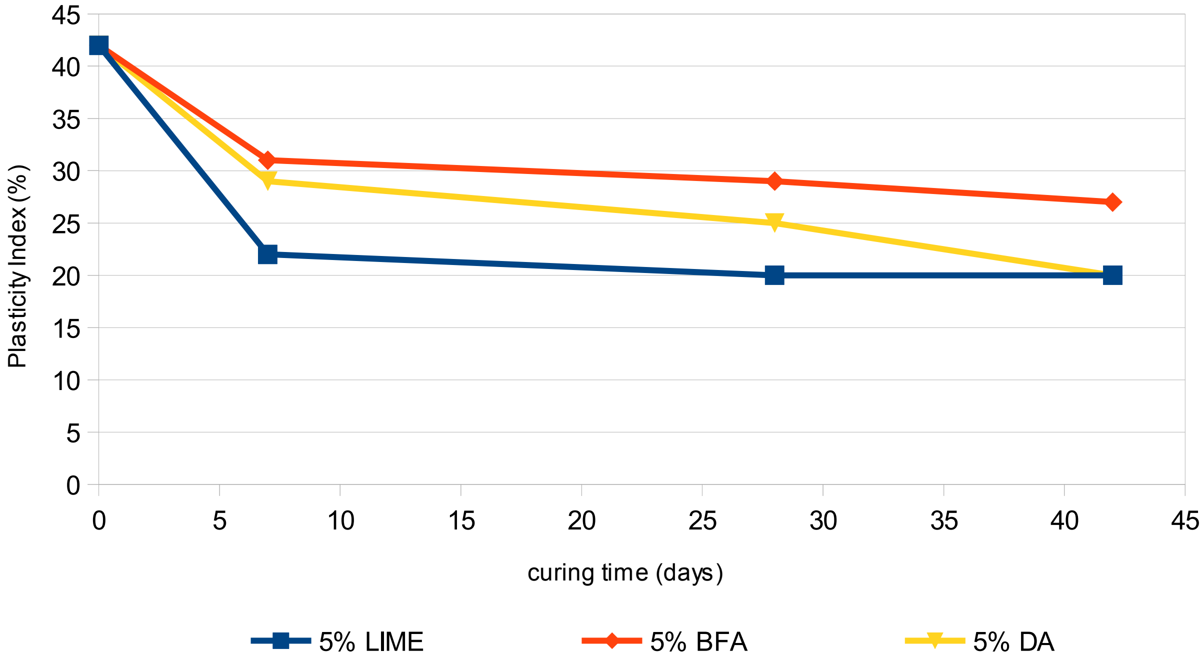


Figure 9.22. Evolution of Plasticity Index (PI) in clayey soils treated with 5% additives

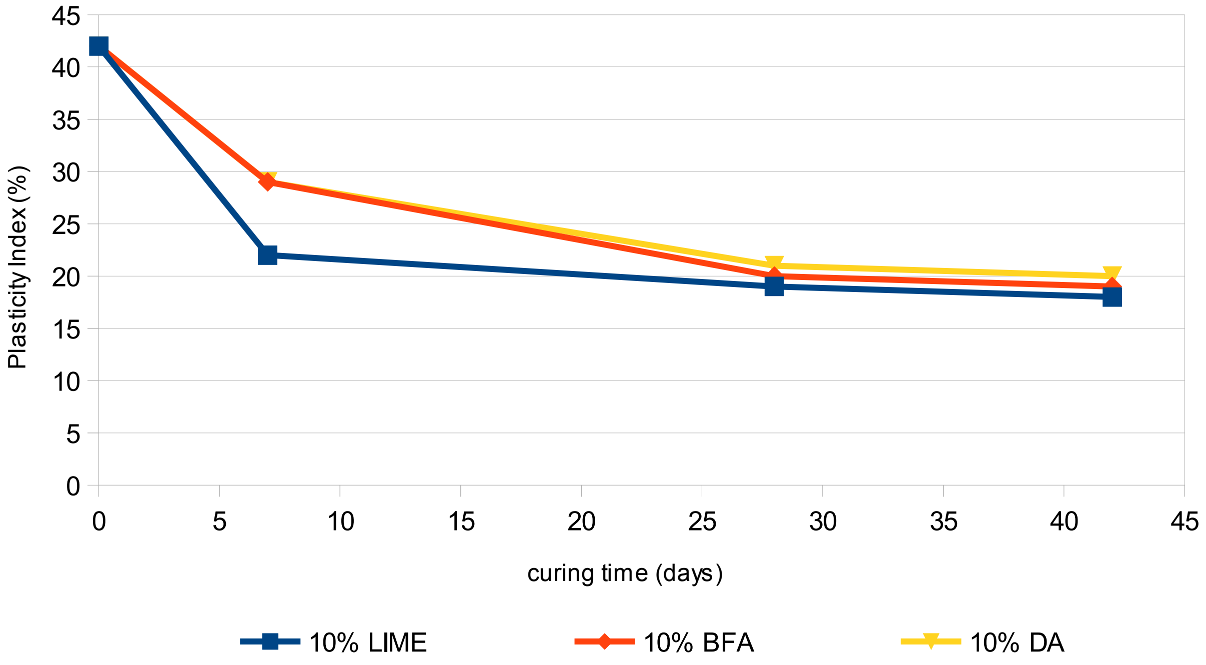


Figure 9.23. Evolution of Plasticity Index (PI) in clayey soils treated with 10% additives

As mentioned above, a relationship between the effects on the particle size distribution and the effects on the plasticity of the soil was found to exist. The additives used in this study promoted a shift to the right in the particle size distribution of both marly and clayey soils studied. This took place for all the samples, except those treated with 5% of BFA. The reduction in the plasticity of the soil also took place for all the samples studied, including those treated with 5% of BFA. The most significant changes (reduction of plasticity index) were observed for the samples treated with lime, which is consistent with the results obtained for particle size distribution which showed a more dramatic alteration of the particle size (towards coarser distributions) when lime was used. However, the modification of important physical properties of the soil such as plasticity and particle size by addition of BFA and DA allows to attribute promising properties to these additives in the soil stabilisation. A further mineral analysis must be carried out.

9.5 X-RAY DIFFRACTION

The X-ray diffraction (XRD) technique has been used in previous sections to explain the mineralogy of the soils and the evolution of the mineral modifications promoted by the undertaken treatments.

The samples of this study (samples from Batch 01 and Batch 02) were also subjected to X-ray diffraction to analyse the evolution of smectite in the samples. Smectite is a group of clay minerals comprising the most expansive and plastic phases of clay. X-ray diffraction is not a reliable technique to carry out a quantification of the minerals present in a soil. However, it provides a valuable approach in terms of qualitative mineral changes.

The XRD patterns have been studied with XPowder 2004. The comparative XRD patterns of the samples studied are plotted in **Figures 9.24 to 9.27.**

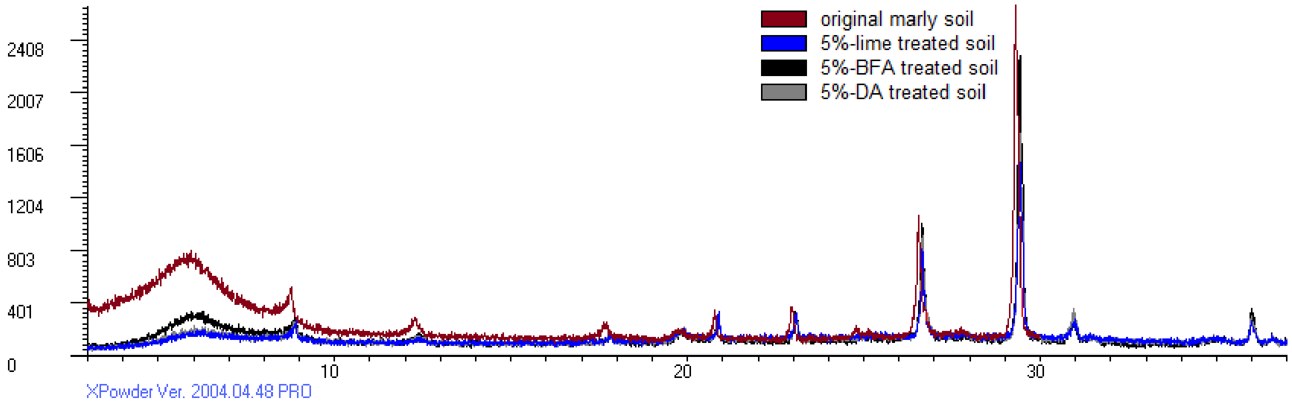


Figure 9.24. XRD pattern of marly soils treated with 5% additives (42 days of curing)

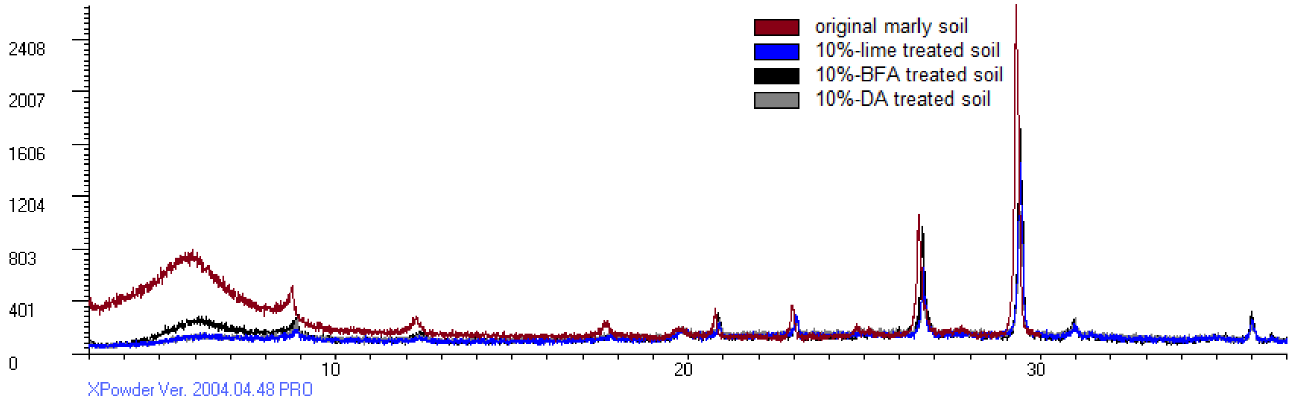


Figure 9.25. XRD pattern of marly soils treated with 10% additives (42 days of curing)

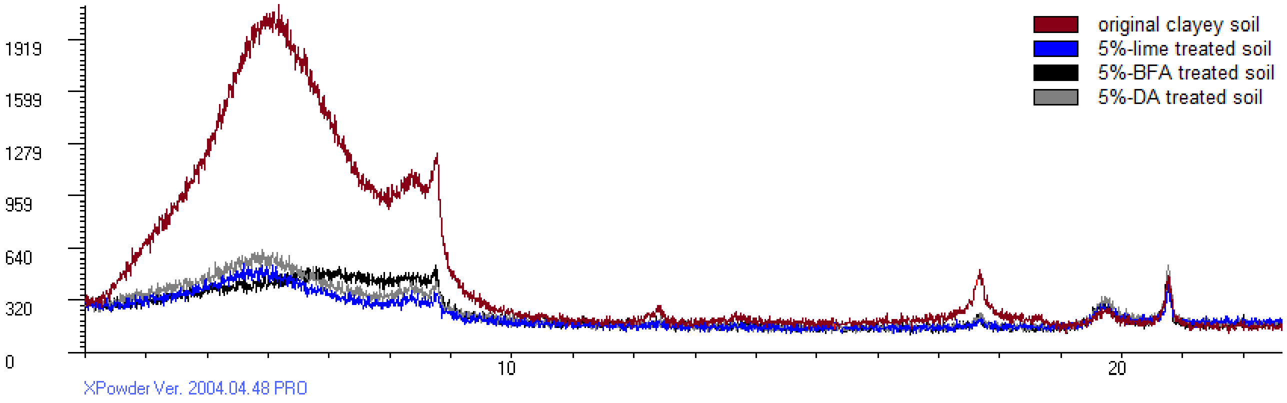


Figure 9.26. XRD pattern of clayey soils treated with 5% additives (42 days of curing)

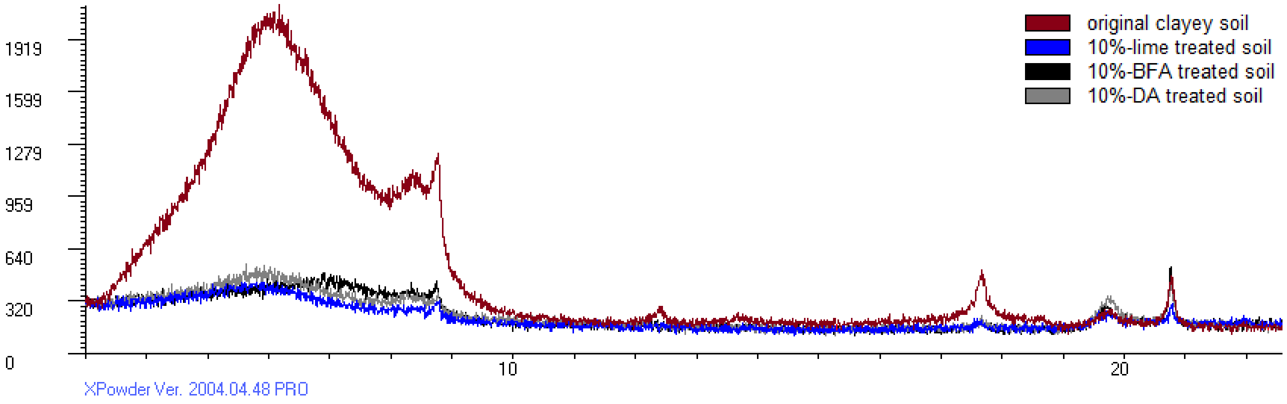


Figure 9.27. XRD pattern of clayey soils treated with 10% additives (42 days of curing)

Figure 9.24 and Figure 9.25 show the XRD pattern of the marly soils when they are treated with 5% and 10% additives, respectively. All the XRD pattern shown in this chapter were obtained at 42 days of curing.

As it can be observed, the untreated marl and clay had an important peak of smectite located around 2-theta angle of 6°. **Figure 9.24** shows how this peak was reduced after 42 days of curing due to the addition of 5% of lime, BFA and DA. In terms of comparison, DA and lime promoted similar effects, slightly greater than that promoted by addition of BFA. **Figure 9.25** shows the same trend, with results being slightly more remarkable when higher dosage (10%) was used. The same tendency is observed for the clayey soil in **Figures 9.26 and 9.27**. Again, lime promoted the most remarkable results, although the reduction of the smectite peak promoted by addition of BFA and DA was very similar. The use of higher dosages promoted a further reduction.

9.6 CONCLUSIONS

- The use of biomass fly ash (BFA) and dolomite ash (DA) as alternative stabilisation agents for natural marly and clayey soils with high swelling potential and plasticity was proved to have a significant effect on the most important physical properties of the soils.

- The particle size distribution of the samples of soils was studied by means of hydrometer (commonly used for fine-grained soils) and laser diffraction. The results obtained were quantitatively comparable. The calculation was obtained in an easier and quicker way using laser diffraction. Laser diffraction is a powerful tool to measure the evolution of the particle size distributions in treated soils.

- The addition of 5% and 10% of BFA did not produce relevant modifications in the particle size of the marly soil but it had a greater and positive effect on the clayey soil: a coarser distribution was found to exist after the treatment. The addition of 5% and 10% of dolomite ash produced a coarser particle size distribution in both the marly and clayey soil. Therefore, the flocculation of particles promoted by the addition of lime was also found in the treatments with BFA and DA due to the presence of free lime in their chemical composition. In terms of particle size, the results obtained with the addition of dolomite ash were comparable to those obtained after addition of lime.

- The plasticity of the soils were reduced with the addition of BFA, DA and lime from the very early stages of curing. In the case of the marly soil, the results of the BFA were comparable to those promoted by lime and DA when a dosage of 5% of additive was used. However, the reduction of the plasticity for dosages of 10% of additive was more remarkable in the case of lime and DA than in the case of BFA. The reduction of plasticity also took place in the clayey soil (which was originally more plastic than the marly soil). The quantitative results of plasticity index obtained after the treatment with DA, BFA and lime were comparable. The results showed that most of the reduction in the plasticity of both soils due to the treatment took place in the first periods of curing: greater reduction obtained in the first week.

- The carbonate and sulphate content in a soil can affect the performance of the stabilisation treatment and must be taken into account for the selection of the most suitable additive. The comparison of the existing chemical methods for the measurement of sulphate and carbonate content and the combustion analysis showed that there is a correlation between the results obtained using both methods. The advantage of the use of the combustion analysis and infrared measurement is the easy development of the test, the quick calculation of the results and the accuracy of the measurement.

- According to the experimental results, the carbonate contents in the marly soil before and after the treatments with lime, DA and BFA were quite similar. There is a consumption of carbonate when dolomite ash is added to the soil observed in the first stages of curing. However, this consumption is not reflected in the measurements carried out for longer curing times. The sulphate content in the marly soil, which was originally low, decreased significantly with the addition of 5% and 10% of lime, BFA and DA. The results obtained with both dosages were very similar. The sulphate content was found to decrease around 50% due to the treatments.

Chapter 10

Mineralogy and microstructure of treated soils: FESEM

Abstract

In this section, a study on the microstructure of the treated soils by means of electron microscopy is presented. A Field Emission Scanning Electron Microscope (FESEM) is used to visualise the surface of the treated and non-treated samples. The use of this technique allows for the identification of changes in the particle size, particle bonding, new mineral formations, etc. Furthermore, the Electron Dispersive Spectroscopy (EDS) was used to identify the new minerals formed by their chemical composition. This sophisticated technique allows for the chemical characterisation of limited areas in the sample. The development of these chemical studies in different spots along the microscopic surface of the treated sample and the comparison between samples treated with different additives (and cured for different times) led to significant findings, such as the formation of amorphous cementitous gels. The study presented in this chapter, also confirmed the importance of the chemical composition of the original soil to be treated, showing how small amounts of sulphates in the soil can promote the formation of minerals such as ettringite or thaumasite whose effects on the stabilisation process will be analysed and discussed.

10.1 MATERIALS AND METHODS

In this chapter, a microstructure analysis of the samples treated in the previous chapter (see Chapter 9 for characterisation of natural soils and chemical composition of additives used) is presented. The samples of natural clayey and marly soils treated with 5 and 10% of lime and biomass fly ash (BFA) are studied. To carry out the microstructure analysis of the natural and treated samples, a Field Emission Scanning Electron Microscope (FESEM) was used. This microscope was located in the Centre of Scientific Instrumentation – University of Granada (Spain). Images of the microstructure of the samples were taken at a reference distance down to 1μm. The study was carried out on samples cured for 7 and 42 days to show the effect of the curing time on the mineralogy. The samples were coated with carbon prior to the scan.

As previously explained, FESEM allows for the development of energy dispersive x-ray spectroscopy (EDS). Throughout this technique, immediate chemical analysis can be carried out on some areas of the surface of the samples. This allowed for the identification of the main chemical elements present in the newly formed mineral encountered during the scanning. This chemical analysis is useful to carry out assumptions on the new mineral being formed in the treated samples.

10.2 NATURAL CLAYEY SOIL

The clayey soil used in the study was a natural expansive soil with a high plasticity (PI > 40) and high swelling potential. This soil was obtained in the area of Alicun (Granada, Southern Spain). The geological setting of this area was presented in chapter 3 of this dissertation.

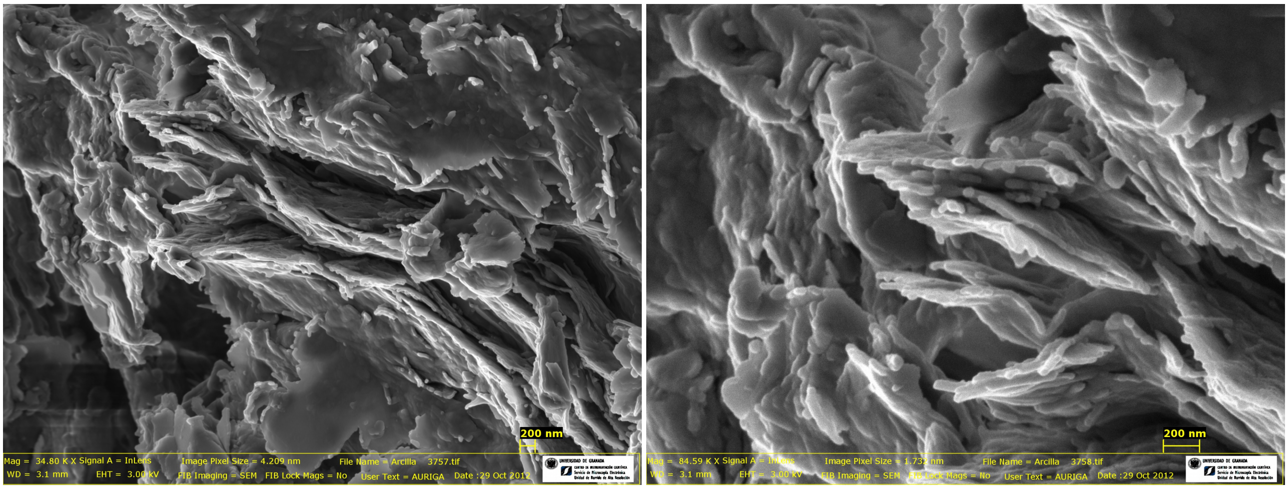


Figure 10.1 FESEM pictures of natural clayey soil

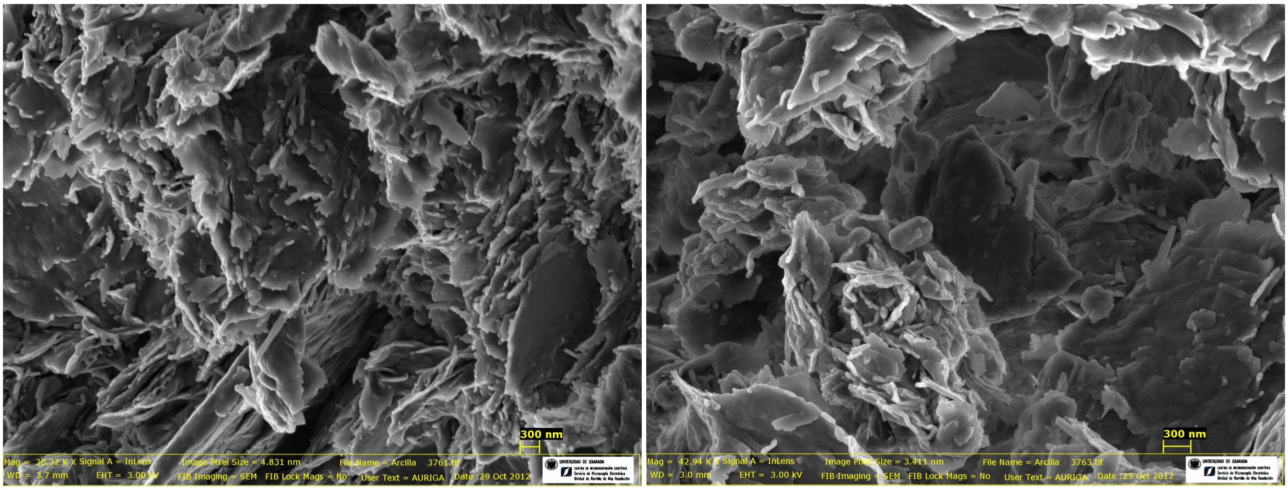


Figure 10.2 FESEM pictures of natural clayey soil

Small amounts of carbonates and sulphates were encountered in the composition of this soil. The FESEM pictures of the natural clayey soil showed the typical structure of clay minerals, with flat sheets of clay. The surface of the studied samples was very homogeneous. Layers of clay mineral were predominantly encountered, as shown in **Figures 10.1 and 10.2**.

10.3 NATURAL MARLY SOIL

The natural marly soil was obtained in the area of Baeza. The geological setting of this area was also presented in chapter 3. This soil is mainly formed of clay and calcium carbonate. Three mineral formations were mainly encountered in the analysis of this samples: 1) layers of clay minerals; 2) crystals of quartz; 3) fossils. The latter were formed of calcium carbonate, and most of the calcite encountered in the composition of the soil was in the form of fossils, probably due to the marine origin of this soil as a sedimentary marly rock.

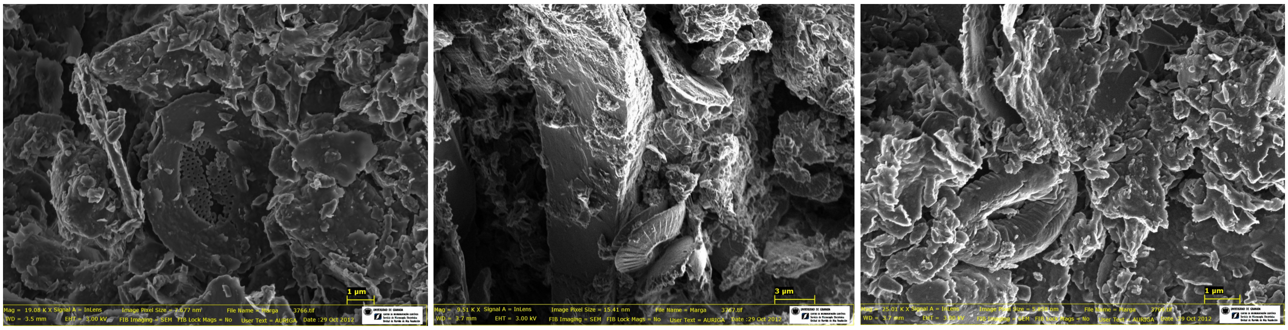


Figure 10.3 FESEM pictures of the natural marly soil showing clay, quartz and fossils

10.4 TREATED CLAYEY SOIL

The FESEM analysis of the treated samples of clayey soil showed a clear difference between the treated and the untreated samples, both in the case of lime and in the case of BFA as a stabilisation agent. Given the FESEM is a technique used for a qualitative analysis, no significant difference was found between the samples treated with the same additive and different dosage (5%-10%). However, in the case of the curing time, the effects (agglomeration of particles, formation of portlandite, etc.) was found to be more remarkable for further stages of curing.

Figure 10.4 shows the FESEM pictures of clayey samples treated with lime. The first evident effect which can be observed is the agglomeration of particles, especially visible in picture shown in **Figure 10.4 b) and d)**. The formation of acicular (needle-like) crystals is also observed in the pictures and they are widespread in the samples.

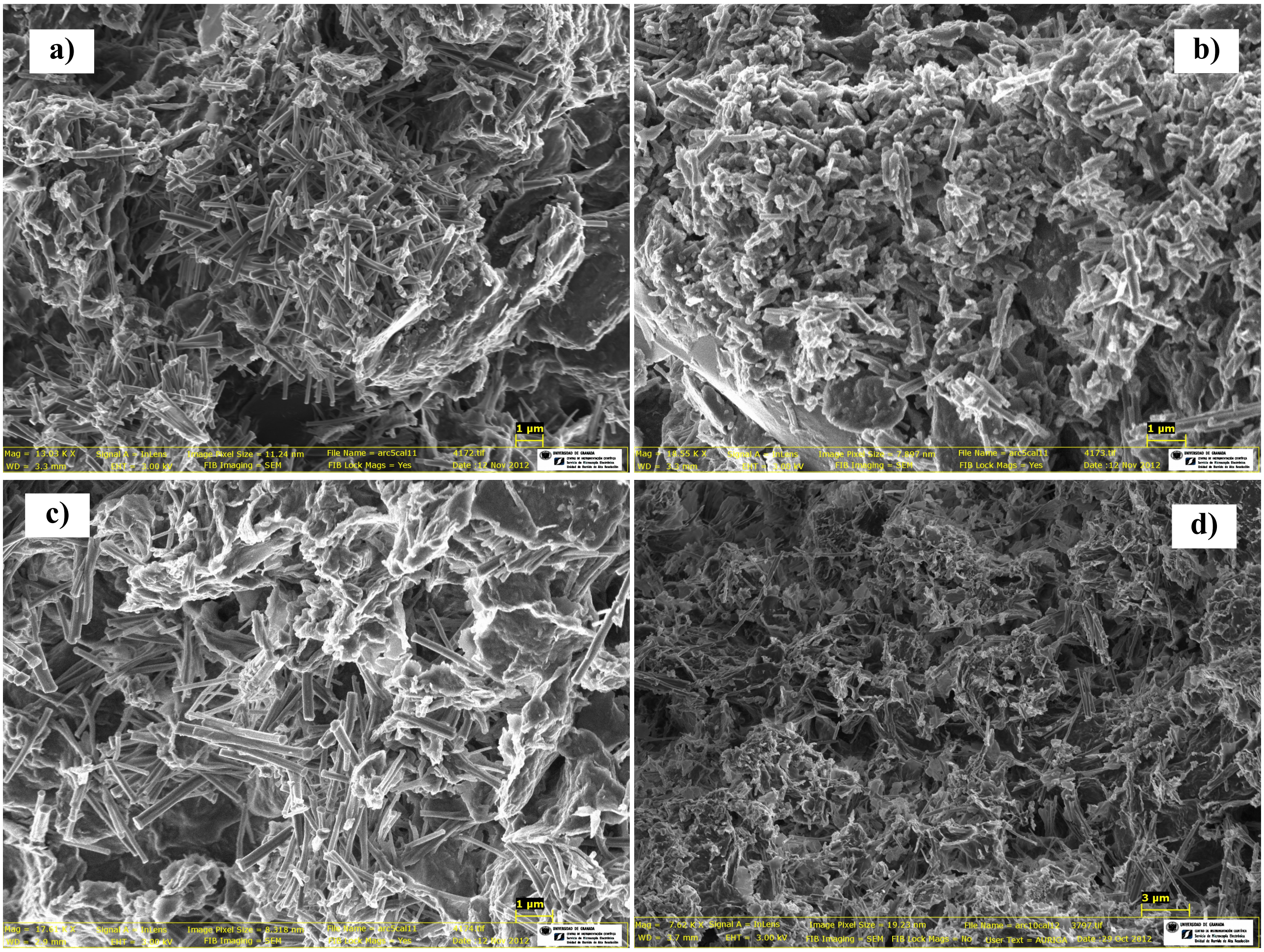


Figure 10.4 FESEM pictures of the samples of clayey soil treated with lime a), b) and c): 5% lime; d) 10% of lime. Curing time in all samples: 90 days.

Figure 10.5 shows the FESEM pictures of the samples of clayey treated with biomass fly ash (BFA). Apparently, a thin layer of a cementitious compound could have been formed, since the layers of clay mineral encountered in the original samples of natural untreated clay seem to be covered by a homogeneous layer. This effect must be attributed to the pozzolanic properties of the BFA and its self-cementing properties due to the presence of free lime in its chemical composition.

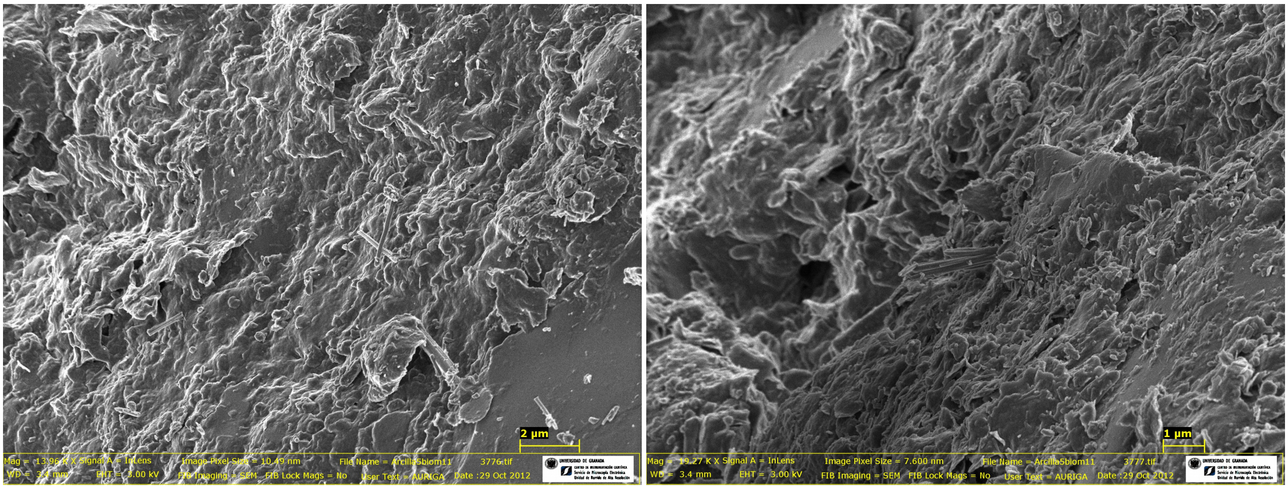


Figure 10.5. FESEM pictures of the samples of clayey soil treated with 5% BFA (after 90 days of curing)

10.5 TREATED MARLY SOIL

In the samples treated with BFA, the FESEM pictures revealed the predominant presence of spherical particles located among the layers of clay mineral of the soil. The agglomeration of particles and the formation of new minerals (crystals of portlandite, acicular minerals, etc.) was clearly identified. The **Figure 10.6** shows the pictures of BFA-treated marly soil after 90 days of curing.

Figure 10.7 shows the FESEM pictures of the sample treated with 10% lime, after 90 days of curing. The picture on the left shows the formation of acicular minerals whose chemical composition was studied by means of EDS. The picture on the right shows the agglomeration of particles due to the flocculation effect of the lime on the soil and the formation of hexagonal crystals. These crystals can be attributed to the formation of portlandite ($Ca(OH)_2$) from the reaction between calcium oxide and water. As explained in previous chapters, the pozzolanic reaction between portlandite and silica and alumina to promote the formation of cementitious compounds is a desirable effect. This effect could not completely proved to occur just on the base of the FESEM analysis.

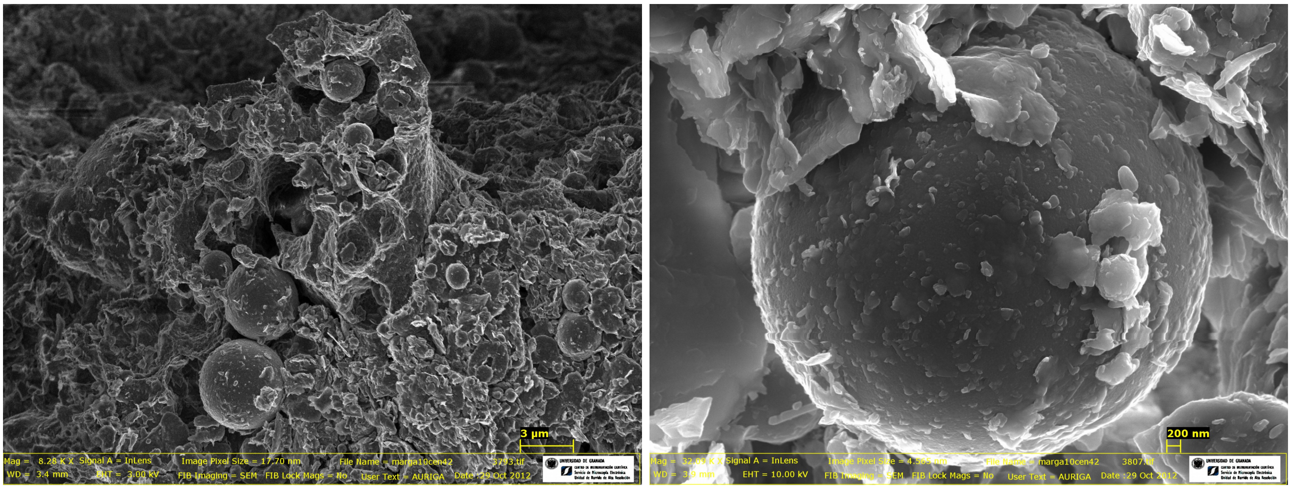


Figure 10.6 FESEM pictures of the samples of marly soil treated with 5% BFA (90 days of curing)

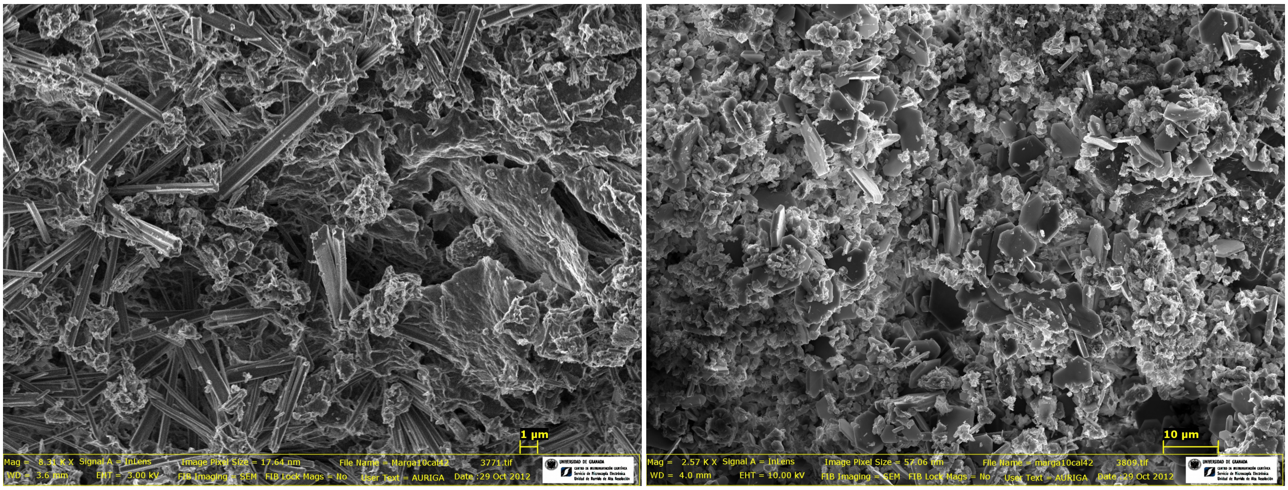


Figure 10.7 FESEM pictures of the marly soil treated with 10% Lime (45 days of curing)

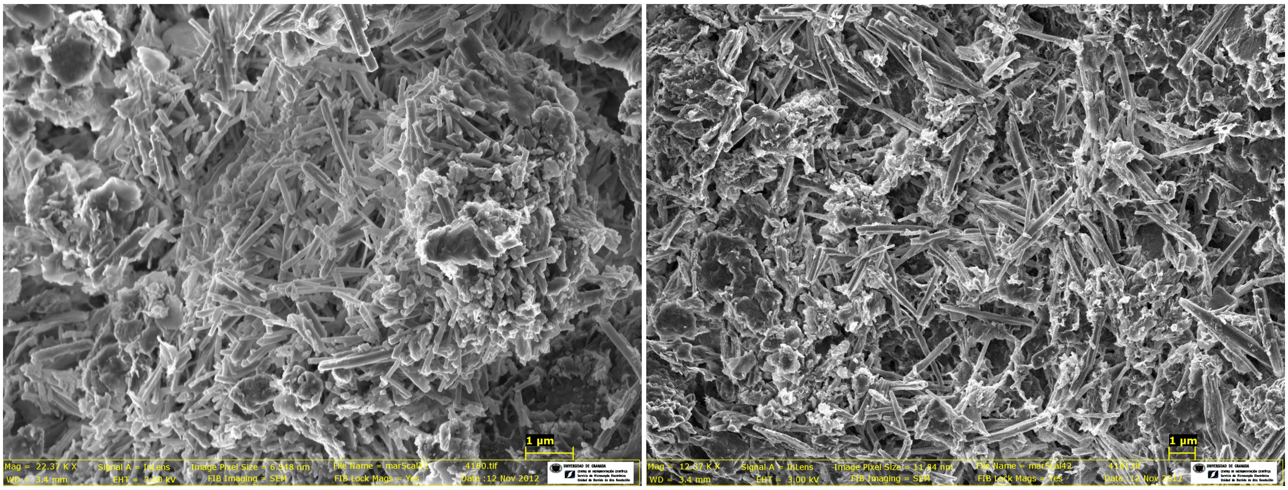


Figure 10.8 FESEM pictures of the marly soil treated with 5% Lime (45 days of curing)

Similar effects promoted by the addition of 5% lime to the samples of marly soil were observed: agglomeration of particles and formation of acicular minerals (**Figure 10.8**).

10.6 EDS RESULTS

The FESEM pictures were accompanied by the use of energy dispersive x-ray spectroscopy (EDS) which allows for the rapid evaluation of the chemical composition of the materials encountered. EDS provides in which the intensity of the peaks reflects the qualitative intensity of elements encountered.

An EDS pattern is shown in **Figure 10.9**. In the case of this figure, the EDS was carried on a sample of natural untreated marly soil, on a zone were a fossil was found. The most likely composition of those fossils encountered in the marine marls was the calcium carbonate ($CaCO_3$). The EDS showed the presence of carbon, calcium and oxide, which confirms this hypothesis.

The acicular mineral observed in the FESEM pictures could be encountered in many of the treated samples, regardless of the original untreated soil and additive. The geometry and layout of these minerals suggest that they could be formed of ettringite.

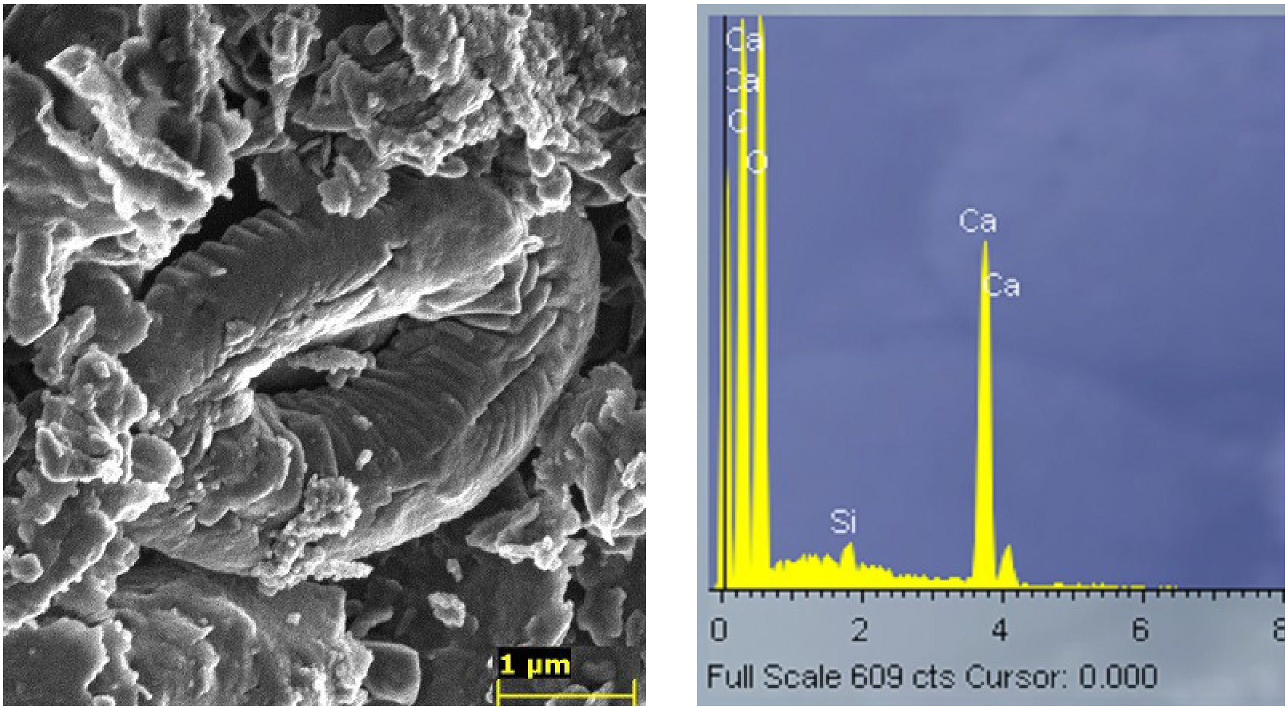


Figure 10.9 FESEM picture of fossil inside the sample of marly soil and EDS pattern obtained

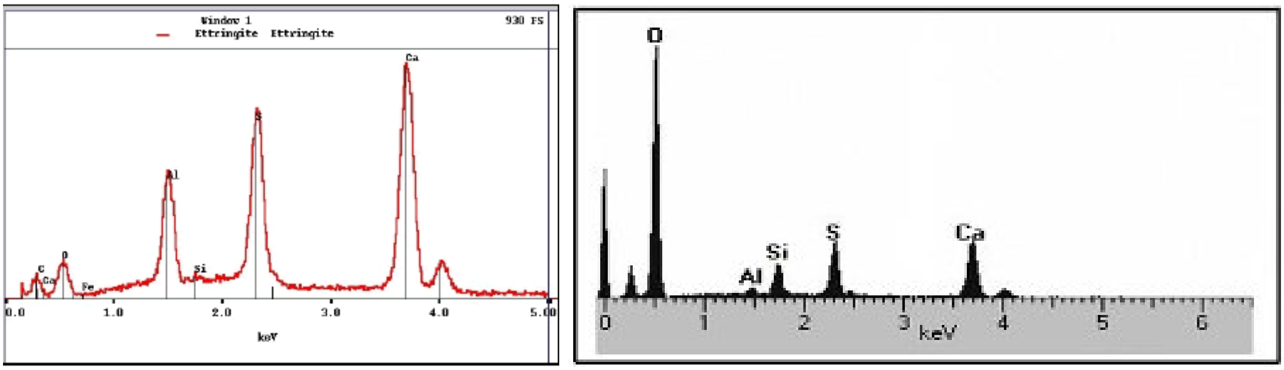


Figure 10.10 a) EDS pattern of ettringite, according to FHWA, 2006; b) EDS pattern of thaumasite according to Bellatreccia and Fiori (2003)

As previously explained, EDS is an adequate technique for the identification of minerals based on their chemical composition. It must be noticed that the results of EDS are only qualitative. **Figure 10.10** shows the EDS patterns of two minerals which could be formed due to the soil stabilisation treatment with calcium oxide: ettringite and thaumasite. The pattern of ettringite was included in the report on petrography of hardened concrete carried out by U.S. Federal Highway Administration (FHWA, 2006).

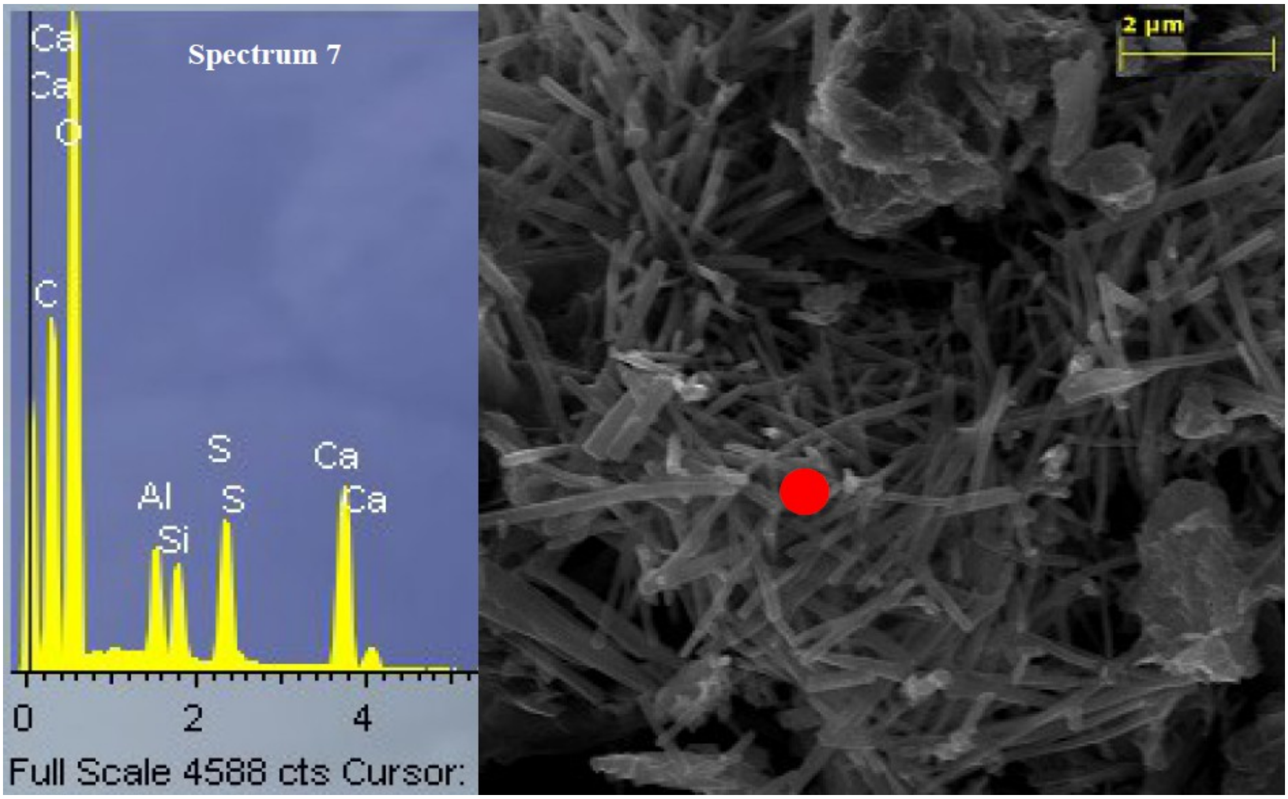


Figure 10.11 FESEM picture and EDS pattern on marl treated with 10% lime (45 days of curing)

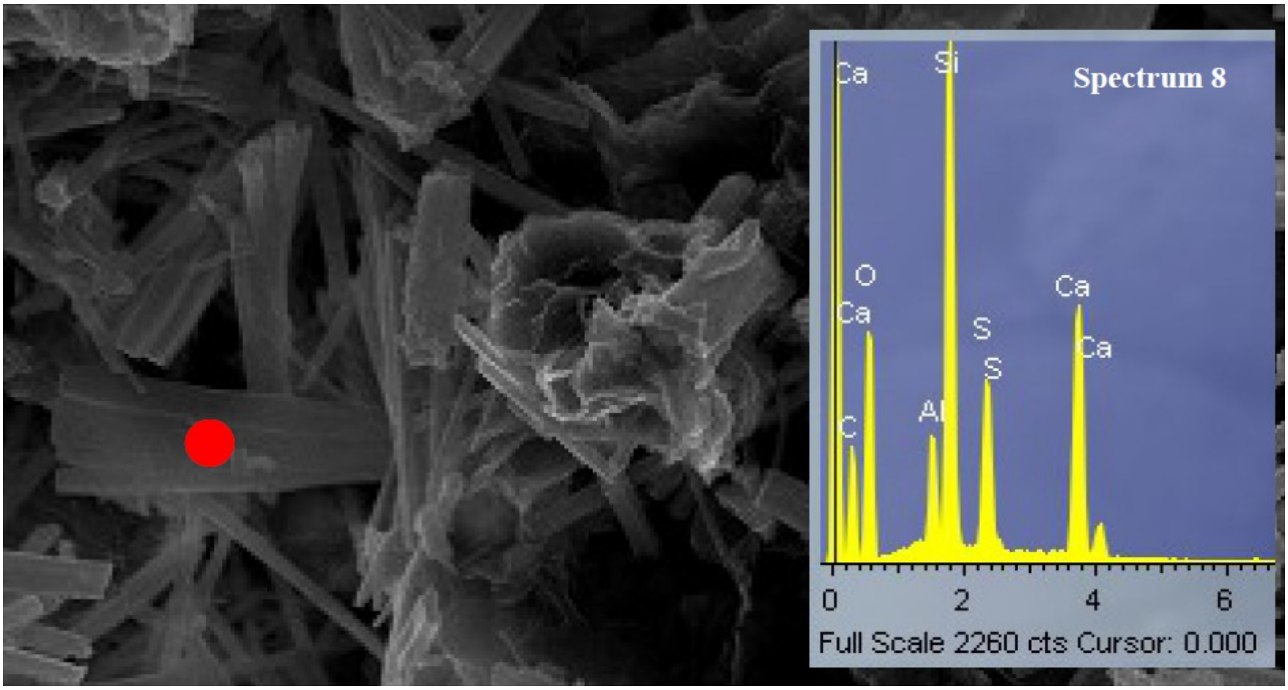


Figure 10.12 FESEM picture and EDS pattern on marl treated with 10% lime (45 days of curing)

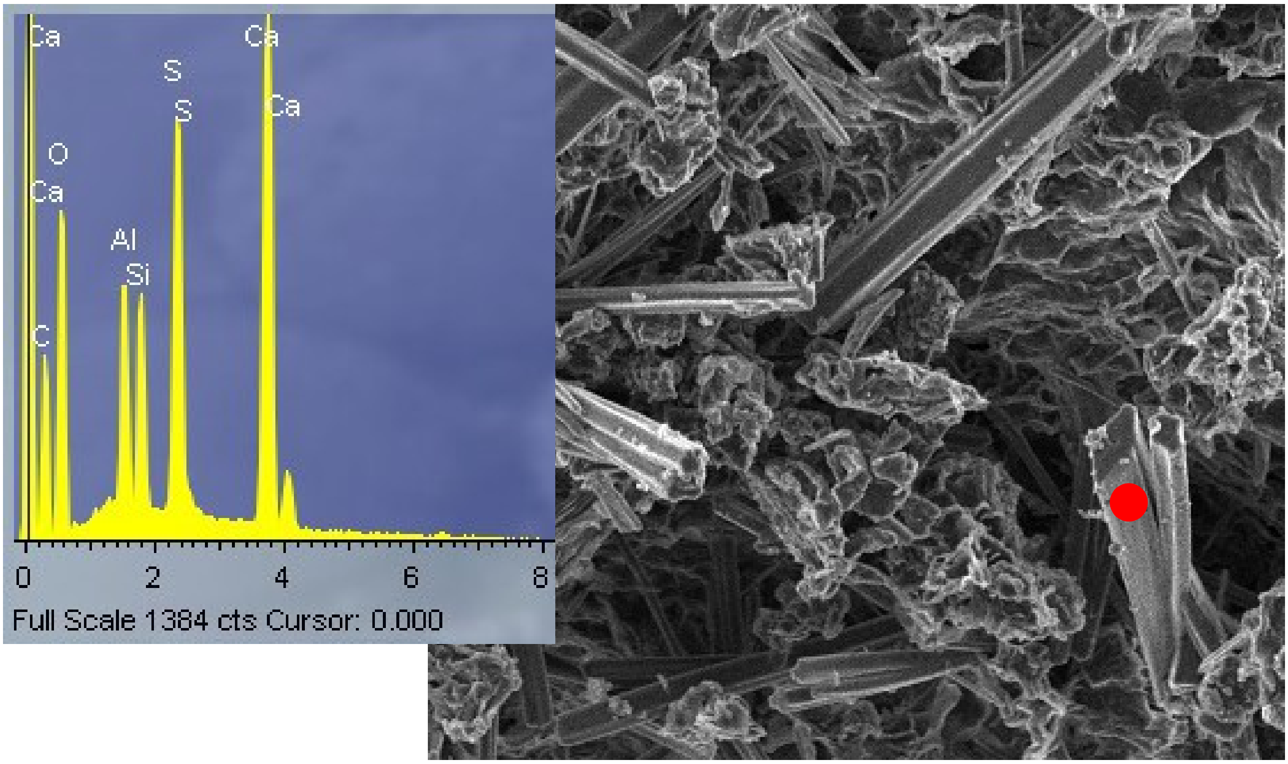


Figure 10.13 FESEM picture and EDS pattern on marl treated with 10% lime (45 days of curing)

As it can be observed, the EDS of ettringite is characterised by the presence of calcium, sulphur and alumina, whilst the EDS pattern of thaumasite is characterised by the presence of calcium, sulphur, alumina and silicon. The silicon and alumina requested for the formation of ettringite and thaumasite can be obtained from the clay present in the mineral composition of both the clayey and marly soils. The chemical formula of these minerals is provided below:

Ettringite: $Ca_6Al_2(SO_4)_3(OH)_{12}\cdot 26H_2O$

Thaumasite: $Ca_3Si(OH)_6(CO_3)(SO_4)\cdot 12H_2O$

Both ettringite and thaumasite are expansive minerals which can increase their volume in the presence of water. The potential for water adsorption is obvious from the chemical formulae presented above. The expansivity of ettringite is considered to be a positive effect in the hardening process of cements and concretes, given the fact that ettringite is usually formed from the sulphates of the cement and it occupies the voids present in the cement paste, promoting an increase in the strength of the paste.

However, in the case of soil stabilisation, the presence of ettringite (or thaumasite) is not a positive effect whatsoever since it means that part of the additive is consumed to produce this mineral, which reduces the efficiency of the treatment, and the ettringite itself is an expansive mineral which does not contribute to the stabilisation of the expansive soils.

Figures 10.11 and 10.12 show the FESEM pictures and EDS pattern obtained on the acicular minerals formed in a sample of marly soil treated with 10% of lime. The FESEM pictures show the agglomeration of particles due to the addition of lime and the formation of a dense grid of needle-like minerals. The EDS confirmed the presence of silicon, alumina, calcium and sulphur. The comparison of the EDS obtained and the EDS reported in the existing literature suggests that the mineral formed might be thaumasite. One of the differences in the composition of ettringite and thaumasite is the presence of carbonates, which can explain the formation of thaumasite in the marls.

The rhomboidal section of some of the acicular minerals **(Figure 10.13)** seem to corroborate that they belong to thaumasite. On the other hand, the hexagonal minerals formed in the soils due to the treatments (they were never identified by means of FESEM and EDS in the natural soils) were found to be mainly formed of calcium and oxide which bolster the hypothesis of the formation of portlandite ($Ca(OH)_2$).

The formation of thaumasite requires the presence of carbonate (which is present in the soil in the form of calcium carbonate in fossils), calcium (added to the soil in the form of calcium oxide), silicon (present in the composition of clay minerals) and sulphur. It must be noticed that the content of sulphur in the soils used for this study, which was studied in the previous chapter, showed that the sulphur content in both the clayey and marly soils was quite low. However, the formation of thaumasite in the marly soil treated with lime was quite widespread. The presence of newly formed acicular minerals in the samples treated with biomass fly ash (BFA) was very residual and the presence of thaumasite or ettringite in those samples could not be confirmed by the FESEM and EDS techniques used.

This confirms that the addition of lime in the presence of small amounts of sulphates and carbonates in the original soil can lead to the formation of expansive minerals which were not previously encountered in the composition of the soil, hence the importance of studying the specific properties of the soil before choosing the suitable additive for soil stabilisation.

10.7 CONCLUSIONS

According to the results obtained in this study, the following conclusion can be drawn:

- The addition of lime to the samples of clayey soil promoted an immediate agglomeration and flocculation of particles which could be observed from the very early stages of curing along with the formation of hexagonal crystals (attributed to the formation of portlandite) and acicular minerals. The difference between the treatments with 5% and 10% of the additive was not easy to identify in this analysis. However, the modifications were found to be more remarkable for further stages of curing. The addition of lime promoted similar and comparable effects on the samples of marly soil.

- The addition of BFA to the clayey and marly soils used in this study promoted a significant modification of the soil observed in the FESEM pictures. In some areas, the layers of clay mineral seemed to be covered by a thin layer which could be formed of cementitious compounds.
- Along with the agglomeration of particles, the addition of lime to the soils also promoted the widespread formation of acicular minerals. The results of the FESEM and EDS scanning suggested that this minerals could be thaumasite. The formation of thaumasite, which is an expansive mineral, is considered to be detrimental for the soil stabilisation treatment. The formation of thaumasite in the soils took place despite the small amount of sulphates and carbonates present in the original soil.

EXPERIMENTAL RESULTS
PART IV

Chapter 11

The engineering properties of soils treated with BFA and DA

Abstract

The effect of alternative additives on engineering properties of marly soil is studied. A marly soil with a well-known mineral composition (60% bentonite, 30% calcite, 10% kaolinite) is stabilised by addition of different dosages (5% and 10%) of biomass fly ash and dolomite ash. In this study, cement was used as a control. The samples prepared are subjected to physical and mechanical tests to evaluate the effects of the treatments. Compaction, consistency, particle size, pH, shear strength, unconfined compressive strength and oedometer tests are carried out to characterise the engineering properties of the treated samples. The performance of the alternative additives used in this study was significant from the very early stages of curing. The results showed noticeable improvements in the physical and mechanical properties of the treated soil. The flocculation of particles due to the increase in the pH produced a coarser particle size distribution after the treatment with dolomite ash and biomass fly ash. The increase in the environmental pH could be responsible for changes in the mineral composition of the soil. A significant linear relationship was found to exist between the results of the unconfined compressive strength and the shear strength.

11.1 MATERIALS USED AND METHODOLOGY

The soils used is marly (clay and calcium carbonate) soil with more than 60% content of bentonite.

The term biomass fly ash involves many different materials whose properties and chemical composition strongly depend on the source of the biomass, the incineration process (time, temperature and methodology), collection points, etc. The potential of biomass fly ash from olive oil industry to improve the engineering properties of the expansive soils comes from the capacity of this fly ash to introduce an alkaline environment in the soil due to its high concentration of potassium.

Table 11.1 shows the chemical composition of the Biomass Fly Ash used for the experiments. The composition of this biomass fly ash is similar to pozzolanic materials with the substantial percentages of Calcium Oxide (CaO) and Silicon Dioxide (SiO_2). The Biomass Fly Ash has a high percentage of Potassium Oxide (K_2O) which may not allow for the same level bonding as the Dolomite Ash. Potassium Oxide (K_2O) will react on hydration but does not produce the cementitous bonding as produced by Calcium Oxide (CaO) or Silicon Dioxide (SiO_2).

Table 11.1

Physical properties of the original untreated soil

Physical properties	
Liquid limit (%)	90
Plastic limit (%)	35
Plasticity index (%)	55
Maximum dry density (kN/m^3)	14.8
Optimum moisture content (%)	25.5
pH	8.5
Unconfined compressive strength (MPa)	0.294
Shear strength (MPa)	0.147
Compression index (Cc)	0.61
Mineral Composition	
Bentonite (%)	60
Calcite (%)	30
Kaolinite (%)	10

Table 11.2 shows the chemical composition of the Dolomite Ash used for the experiments. The composition is similar to Portland Cement with the high percentage of Calcium Oxide (CaO). The Dolomite Ash has higher values of Magnesium Oxide (MgO) and lower levels of Silicon Dioxide (SiO_2). In Dolomite Ash the Calcium Oxide (CaO) is the important parameter and reacts on hydration with water forming bonds. The desired effect of the addition of Dolomite Ash to the Original Unsuitable Soil is to create bonding of particles and therefore improve the engineering properties.

Table 11.2

Chemical composition of additives

Oxides (%)	DA	BFA	*Elements (pp)*	DA	BFA
SiO2	0.84	7.42	S	94	28939
Al2O3	0.36	1.78	Cl	192	44086
Fe2O3	0.54	1.73	Cr	48	104
MgO	25.26	1.90	Ni	11	65
CaO	54.21	12.98	Cu	49	355
Na2O	0	0.31	Zn	42	362
K2O	0.05	44.39	Sr	169	481
P2O5	0	2.57	Zr	8	76
Loss on ignition					
LOI (%)	18.6	15.9			

Figure 11.1 shows pictures of the lab procedures. **Figure 11.2** shows the SEM pictures of the additives used in this study.

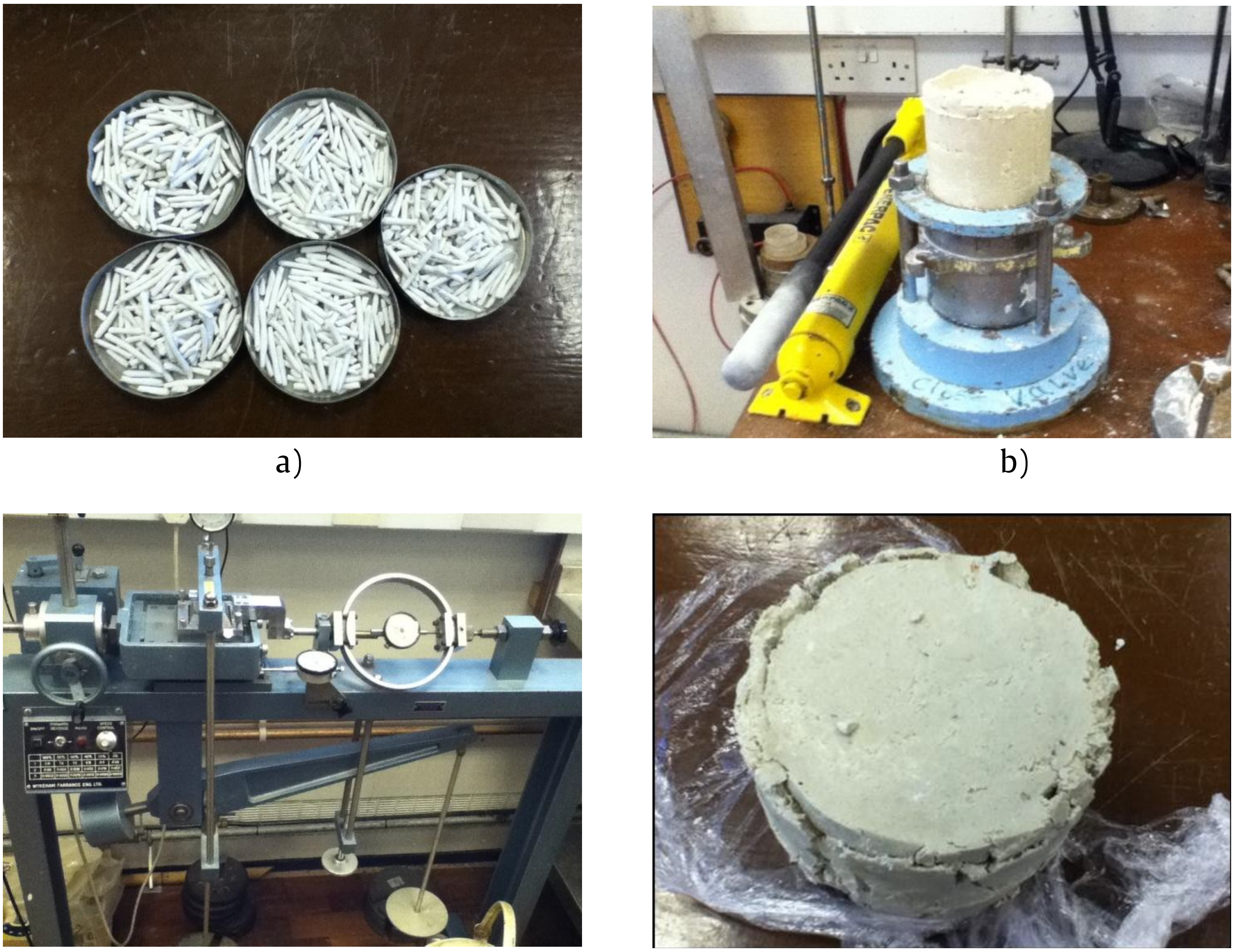
The marly soil is mixed with biomass fly ash and dolomite ash in different dosages (5-10%). Cement is used as a control.

The following tests were carried out in the samples after 1, 7, 14, 28 and/or 42 days of curing: standard Proctor compaction test, Atterberg limits, pH, particle size, shear strength, consolidation in oedometer, unconfined compressive strength **(Figure 11.2)**.

11.2 COMPACTION AND CONSISTENCY OF SOIL

The results of the standard Proctor compaction tests are given in **Table 11.3** in terms of maximum dry density (MDD) and optimum moisture content (OMC) of the treated soils. The MDD of the original untreated soil was 14.8 kN/m^3 and the OMC was 25.5%.

The treatment with biomass fly ash did not produce any significant change in the values of MDD and OMC after 28 days. The addition of dolomite ash produced a slight decrease in the MDD and increase of OMC. The increase in MDD was proportional to the dosage of dolomite ash added to the sample. Similar results were obtained in previous research studies (**Kolias et al., 2005; Rahmat and Ismail, 2011**).



a) b)

c) d)

Figure 11.1: Laboratory test equipment and samples: a) Dried plastic limit samples; b) Compacted sample being extruded for trimming and measurement; c) Shear box apparatus; d) State of original unsuitable soil sample after being tested for UCS

In the case of dolomite ash, the optimum moisture content increased with increase of percentage of additive. According to **Hossain and Mol (2011)**, this increase in the optimum moisture content is attributed to the extra water required for higher fineness and subsequent enhanced hydration.

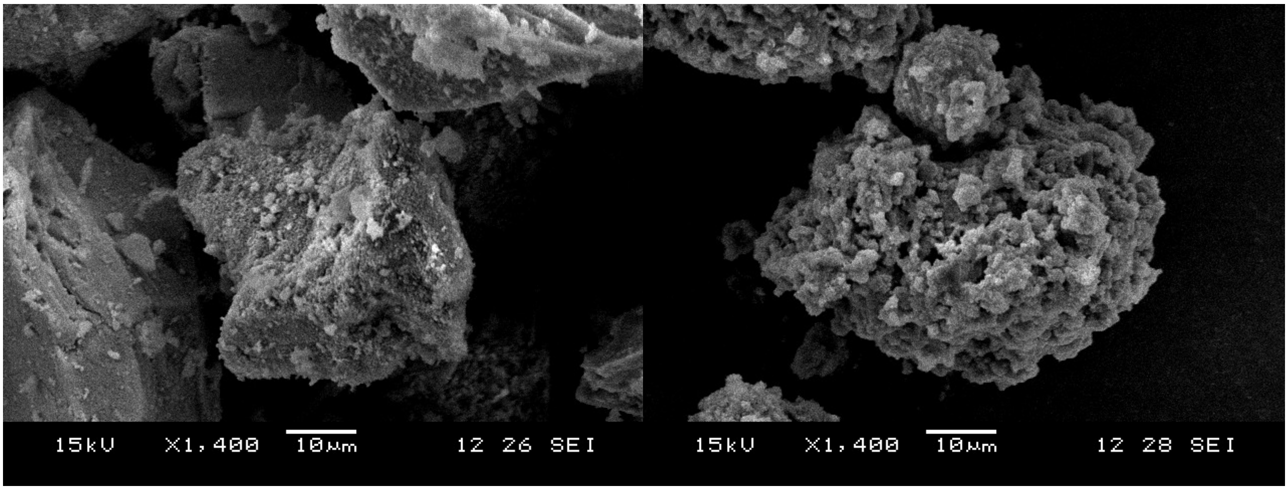


Figure 11.2: SEM pictures of dolomite ash and biomass fly ash

One of the most noticeable effects of the treatments carried out in this study was the reduction of the plasticity index (PI) of the soil after addition of biomass fly ash and dolomite ash in different dosages. As a control, Atterberg limits tests were carried out for cement-treated soil samples. **Figure 11.3** shows the evolution of PI with curing time for all the samples prepared. The original value of PI = 54% of the untreated soil is dramatically reduced from the very early stages of curing. The addition of 5% and 10% of dolomite ash produced PI values of 23 and 25.5, respectively. The addition of 5% and 10% of biomass fly ash also produced a great reduction of the PI to reach values of 32 and 25, respectively.

The discussed reduction in plasticity of the soil can be due to the effect of flocculation-agglomeration of particles after addition of tested binders. Furthermore, the reduction of the plasticity in the dolomite ash-treated samples took place in the first days. However, the reduction promoted by biomass fly ash was slowlier and not equally sharp. This can be attributed to the different properties of dolomite ash (cementing) and biomass fly ash (pozzolanic).

Table 11.3

Results of compaction tests

<u>Compaction results after 28 days of curing</u>

	Original soil	BFA-5%	BFA-10%	DA-5%	DA-10%
Maximum dry density (kN/m3)	14.8	14.8	14.8	13.3	13.1
Optimum moisture content (%)	25.5	25.5	25.5	27	28

The activity of the clay is an indicator of the volatility of the clay and the potential of the clay to swell or contract. According to **Head (1992)**, it is obtained by the following formula: A = PI/%C, where A is the activity of the clay, PI is the plasticity index and %C is the percentage of clay fraction in the soil. **Table 11.4** shows the evolution of the activity of clay in the treated samples. As it can be seen, the activity is reduced by all the treatments carried out which let us infer a more stable behaviour of the clay **(Skempton, 1953)**.

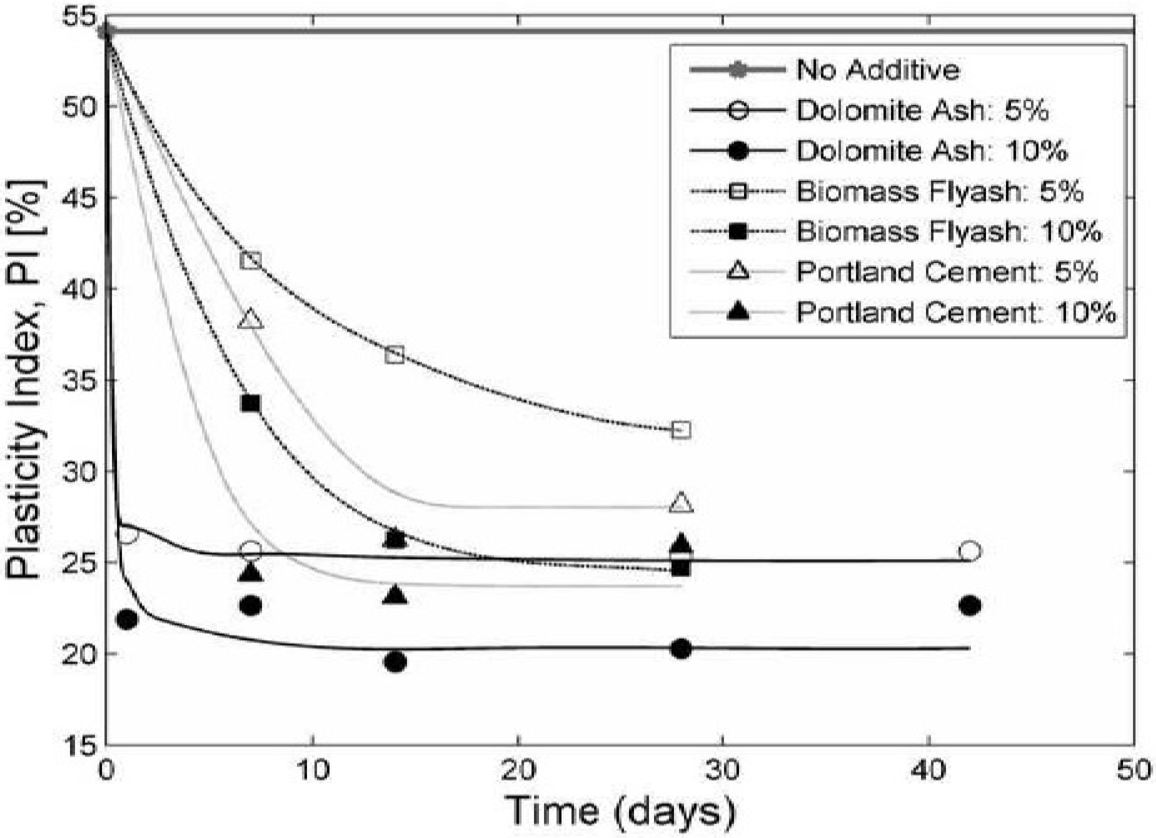


Figure 11.3: Evolution of Plasticity Index in the treated samples

11.3 EFFECTS ON PH AND PARTICLE SIZE

To evaluate the influence of the addition of different quantities of BFA and DA, the pH of the samples was recorded along with the values obtained for cement-soil mixtures. **Figure 11.4** shows the results of the pH tests.

The natural soil had a pH of 8.6. The addition of 5%, 10% of dolomite ash or 10% of biomass fly ash promoted an increase in the pH value of the soil equal to that produced by the addition of the same dosages of cement. However, the samples treated with 5% of biomass fly ash were found to have a pH value (9.1) only slightly higher than that of the original soil (8.6).

Table 11.4

Activity Index

Mix No.	Soil	Curing time (days)	Composition (grams)				% Clay Fraction	Plasticity Index	Activity
			Untreated Marly Soil	Dolomite	Biomass	Cement			
	Original Soil		2500	0	0	0	70.0	54	0.77
1	5% DA	1	2500	125	0	0	66.67	27	0.40
1		7	2500	125	0	0	66.67	26	0.38
1		14	2500	125	0	0	66.67	20	0.29
1		28	2500	125	0	0	66.67	25	0.38
1		42	2500	125	0	0	66.67	26	0.38
2	10% DA	1	2500	250	0	0	63.64	22	0.34
2		7	2500	250	0	0	63.64	23	0.36
2		14	2500	250	0	0	63.64	20	0.31
2		28	2500	250	0	0	63.64	20	0.32
2		42	2500	250	0	0	63.64	23	0.36
3	5% BFA	7	2500	0	125	0	66.67	42	0.62
3		14	2500	0	125	0	66.67	36	0.55
3		28	2500	0	125	0	66.67	32	0.48
4	10% BFA	7	2500	0	250	0	63.64	34	0.53
4		14	2500	0	250	0	63.64	26	0.41
4		28	2500	0	250	0	63.64	25	0.39
5	5% Cement	7	2000	0	0	100	66.67	38	0.57
5		14	2000	0	0	100	66.67	26	0.39
5		28	2000	0	0	100	66.67	28	0.42
6	10% Cement	7	2000	0	0	200	63.64	24	0.38
6		14	2000	0	0	200	63.64	23	0.36
6		28	2000	0	0	200	63.64	26	0.41

Previous studies (**Drief et al., 2002; Elert et al., 2008**) claimed that expandable clay minerals could be destroyed in alkaline environments. According to **Seco et al. (2012)**, the pH must be higher than 10.5 to promote a significant pozzolanic reaction (dissolution of clay minerals). As it can be seen in Figure 11.4, this value is reached and remains after 28 days in the samples treated with 5% and 10% of dolomite ash and 10% of biomass fly ash.

The study of the particle size of the samples after 28 days confirms the flocculation-agglomeration effects on the treated soils after addition of dolomite ash and biomass fly ash. A laser diffraction analyser was used to obtain the particle size distribution of the samples. As shown in **Figure 11.5**, both the original soil and treated samples had a very fine distribution. However, after the addition of dolomite ash and biomass fly ash, a displacement of the particle size curve towards a coarser distribution can be observed. No significant differences are appreciated for different dosages and additives, but the samples treated with the so-called non-conventional additives presented a similar particle size distribution of those treated with the same dosage of cement.

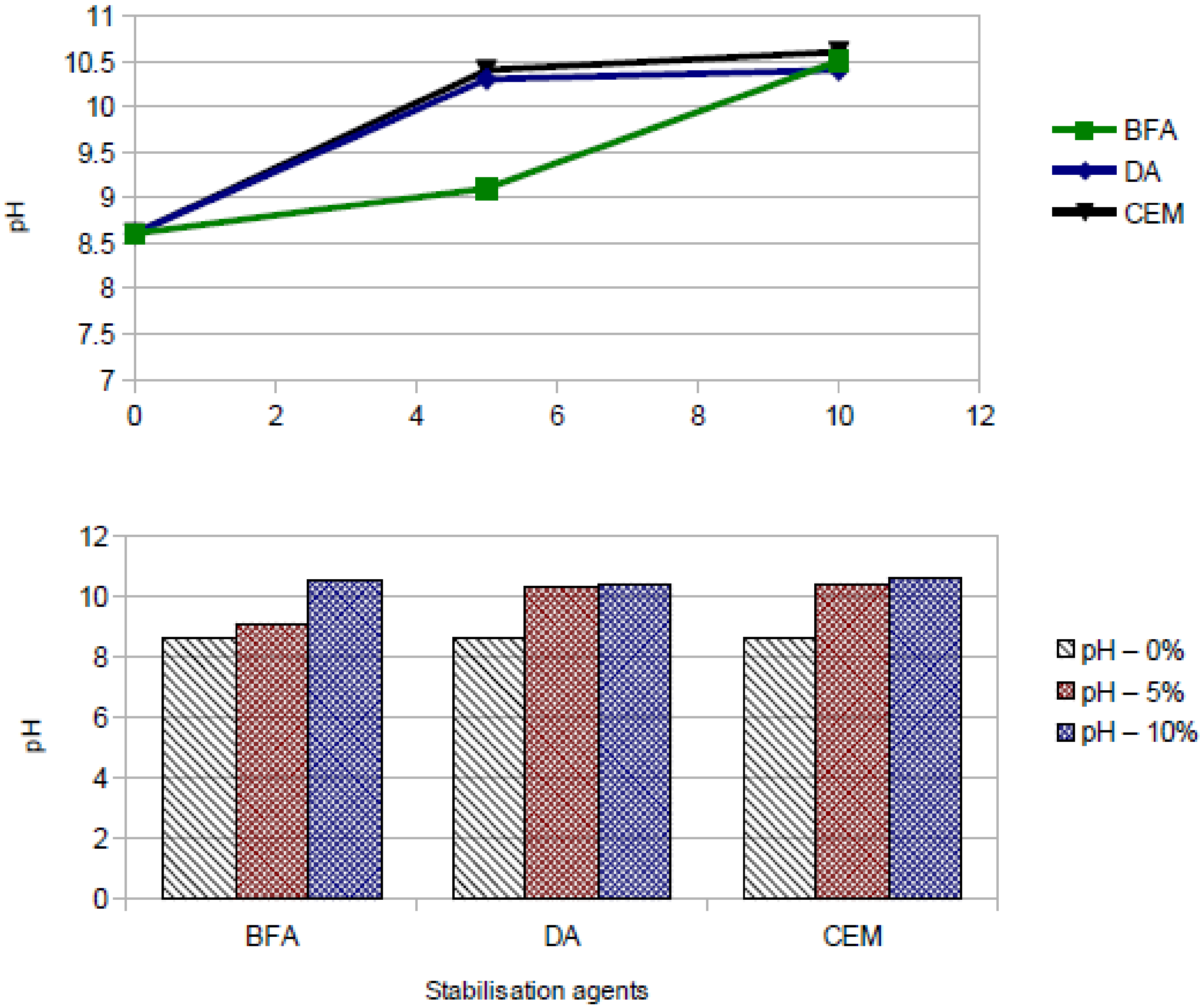


Figure 11.4: Evolution of pH with curing time and dosage of additives

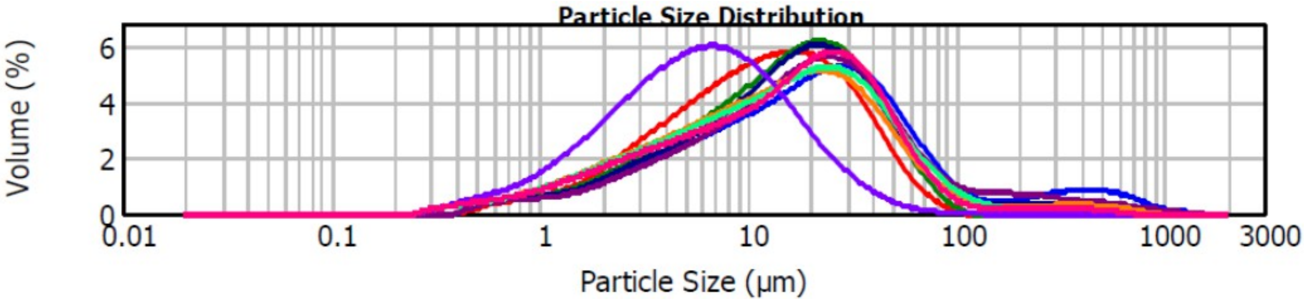


Figure 11.5: Particle size distribution of untreated and treated samples

This effect of flocculation-agglomeration of particles in the treated soils is crucial, since it affects the compaction, strength and plasticity of the soil (**Ureña et al., 2013**).

11.4 STRENGTH PARAMETERS

The shear strength is one of the most important parameters of a soil with a strong influence on the suitability for its use in embankments or foundations. **Figure 11.6** shows the evolution of the shear strength of the soil for various dosages of additives. A significant increase in the shear strength for the first 28 days of curing can be observed. The pace of the increase is similar for both additives tested in this study (dolomite ash and biomass fly ash), although the final value was slightly higher when biomass fly ash was used. The percentage of additive did not have a significant influence on the reached value. In the case of the dolomite ash, the use of 5% and 10% promoted similar results along different curing times.

The original value of 147 kPa obtained for the untreated marly soil is increased after 28 days of curing to an average value 325 kPa after addition of dolomite ash and 408 kPa after addition of biomass fly ash. These results are similar to those found by **Cai et al. (2006)** after treatment of clayey soil with polypropylene fibre and lime admixture and 28 days of curing.

In the case of the dolomite ash, the improvement in the shear strength seems to take place in the first 15 days of curing, whilst the biomass fly ash-treated samples continued improving the shear strength until further stages. This may be due to the different effect that these additives promote on the soil. The presence of potassium in the biomass fly ash makes it more alkaline and the presence of silica and alumina makes it more pozzolanic. The presence of calcium oxide and magnesium oxide in the dolomite ash produced a quicker flocculation and agglomeration effect than can conclude if these oxides become exhausted.

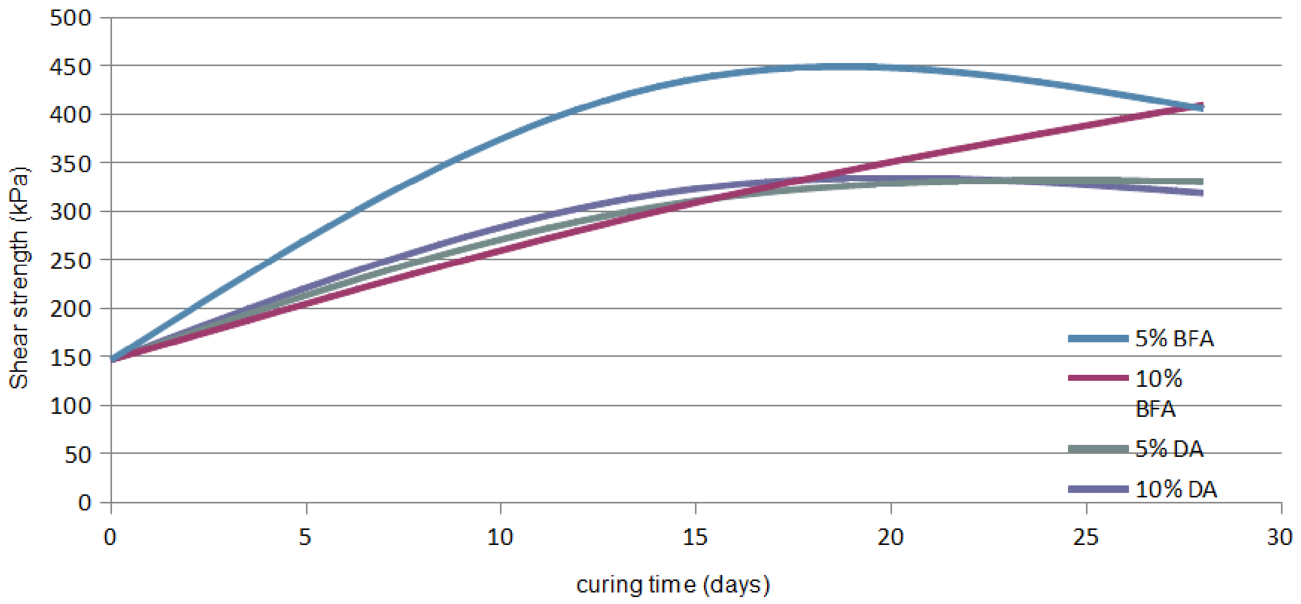


Figure 11.6: Shear strength

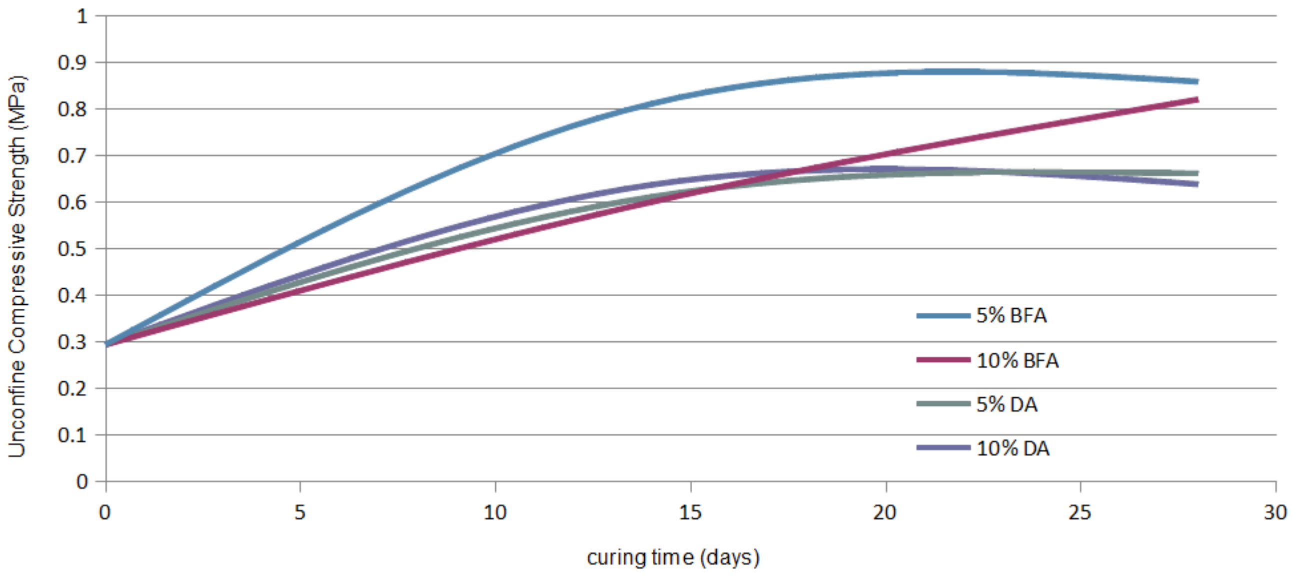


Figure 11.7: Unconfined compressive strength (UCS)

The evolution of unconfined compressive strength (UCS) in the treated samples followed a similar pattern to that shown for shear strength. The untreated soil had a UCS value of 294 kPa. This value was increased after the treatments with dolomite ash and biomass fly ash alike. As seen in **Figure 11.7**, the addition of 5% and 10% of biomass fly ash produced an increase in the UCS value of the treated soil of 291% and 279% respectively, reaching an average value of 839 kPa. The treatment with dolomite ash increased the UCS of the soil to reach values of 661 and 638 kPa for 5% and 10% dolomite ash, respectively.

Again, the increase in the UCS was slowlier but longer in the samples treated with biomass fly ash whilst in the samples treated with dolomite ash the higher percentage of increase took place in previous curing stages.

The increase in the strength of the soil must be attributed to the flocculation-agglomeration of particles and the mineral dissolution of expandable phases of clay.

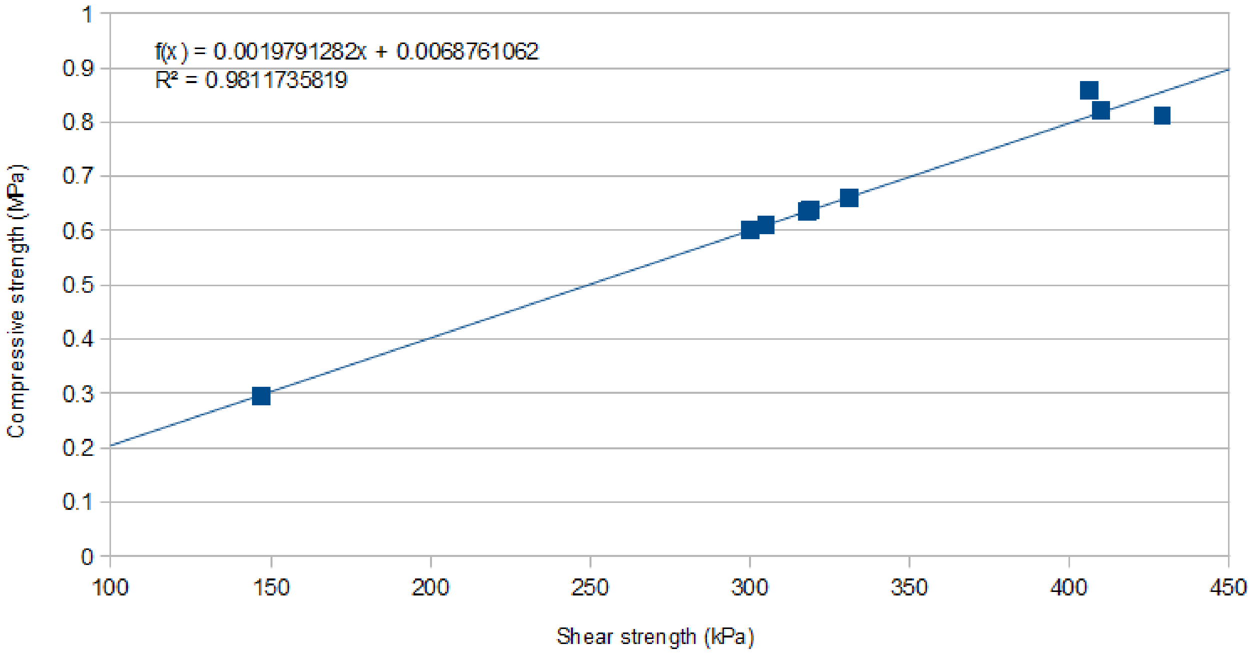


Figure 11.8: Correlation between shear strength and compressive strength

11.5 CORRELATION BETWEEN PROPERTIES

The relationship between shear strength and unconfined compressive strength is shown in **Figure 11.8**. A linear relationship between these two parameters exists. **Sherwood (1993)** and **Hossain and Mol (2011)** found similar relationships between different mechanical properties, such as CBR, Young's modulus, compressive strength and tensile strength, when a soil was treated with conventional (lime and cement) and non-conventional (pozzolans, waste materials) additives respectively. This linear relationship was found to exist throughout measures made in different curing times.

As discussed in previous sections, the treatments carried out in this study promoted flocculation of the particles along with an increase in the environmental pH and a reduction of the plasticity index. The formation of new bonds among particles is behind the correlated increase of shear and compressive strength in the soil. The increase in the strength parameters and the bearing capacity due to the addition of dolomite ash and biomass fly ash represents a significant soil improvement.

11.6 CONCLUSIONS

The following conclusions are drawn from the results:

- The treatment with dolomite ash and biomass fly ash shows a significant improvement in the physical and mechanical properties of the soil such as reduced plasticity, coarser particle size distribution, lower compressibility and enhanced strength parameters. The potential benefit of the treatment with these non-conventional additives seems to be related with the dosage of additive in the mixture and the curing time.

- The plasticity index was reduced due to the addition of BFA and DA. This reduction took place earlier in the DA-treated soils than in BFA-treated soils, obtaining similar final results. The results of the tests carried out in this study showed that the reduction in plasticity index occurred from the very early stages of curing. The pH of the samples increased significantly after addition of DA and BFA. The values of pH obtained after 28 days of curing for soils treated with these non-conventional additives were similar to those obtained with cement-soil mixtures when the dosage of additive was 10%.

- The study of the particle size of the samples by means of laser particle size analyser proved that the tested additives promoted the formation of bonds among particles, hence producing a coarser soil.

- The mechanical properties of the treated soil were improved by the addition of dolomite ash and biomass fly ash. The compressibility of the soil was reduced and the strength parameters were significantly enhanced. A linear relationship has been found to exist between UCS and shear strength.

- The use of non-conventional additives such as dolomite ash and biomass fly ash as an alternative to the traditional soil stabilisation agents (lime, cement, coal fly ash) could improve the engineering properties of the available soil whilst leading to a more sustainable construction process.

Chapter 12

Improvement of the mechanical behaviour of soil by alteration of pH and mineral composition

Abstract

In this part of the investigation, the effect of the non-conventional stabilisation agents on the mechanical behaviour of the soil is studied. Based on the results obtained in previous stages, biomass fly ash (BFA) and dolomite ash (DA) have been selected to treat the soil. In the previous chapter, a bentonite with calcium carbonates has been treated. In this chapter, a natural marly soil will be mixed with BFA and DA in different proportions (5% and 10%) and the performance of those additives will be assessed. The samples were left to cure for 7, 14 and 28 days. To study the effects of the additives on the soil, the following tests were carried out: Atterberg limits tests, standard Proctor compaction tests, consolidation tests in oedometer and shear box tests. The pH of the samples were recorded to evaluate the effect of the additives on the alkalinity of the soil. The evolution of the mineral composition of the treated soils was studied by X-ray diffraction (XRD) tests. The additives tested (biomass fly ash and dolomite ash) performed very well to alter the plasticity index of the soil. The PI was reduced from the original value of 43, to values 17 and 14 in the samples treated with 10% of biomass fly ash and dolomite ash, respectively. The peak shear strength of the soil treated with 10% biomass fly ash was 140 kPa after 28 days, whilst for the sample treated with 10% of dolomite ash this value was 127 kPa. Both values were higher than the peak shear strength of the original untreated soil, 83 kPa. In terms of compressibility and swelling, the values obtained in consolidation tests proved that dolomite ash had been more effective than biomass fly ash. The pH of the samples increased significantly after the treatments and the amount of smectite in the soil diminished dramatically. A relationship was found to exist between the increase of pH, the modification of the mineral composition and the improvement of physical and mechanical parameters of the soil.

12..1 MATERIALS USED

In this study, a sample of soil was collected from a natural outcrop in Alicun (Granada, Spain) located near the river Fardes. The area of the Granada basin is rich in bentonite and marine marls. The soil collected is a clay with a short amount of carbonates (<8%). The physical and engineering properties of the soil are presented in **Table 12.1**. The X-ray diffraction (XRD) shows the soil to contain beidellite and nontronite (smectite group), palygorskite, illite, quartz and calcite **(Figure 12.1)**.

Table 12.1

Laboratory tests on engineering properties of natural soil

Atterberg Limits (%)			Compaction			Chemistry			Mechanical Behaviour			
LL	PL	PI	MDD (gr/cm^3)	OMC (%)	Density of solid phase (gr/cm^3)	pH	%CO_3	%SO_4	Shear strength (kPa)	Angle of shear resistance (º)	Cc	Cs
72	29	43	1.63	23	2.93	8.7	7.79	0.43	83	20	0.99	1.1

Table 12.2

Chemical composition of the additives tested

Oxides (%)	*DA*	*BFA*	*Elements (ppm)*	*DA*	*BFA*
SiO_2	0.84	7.42	S	94	28939
Al_2O_3	0.36	1.78	Cl	192	44086
Fe_2O_3	0.54	1.73	Cr	48	104
MgO	25.26	1.90	Ni	11	65
CaO	54.21	12.98	Cu	49	355
Na_2O	0	0.31	Zn	42	362
K_2O	0.05	44.39	Sr	169	481
P_2O_5	0	2.57	Zr	8	76
Loss on Ignition					
LOI (%)	18.6	15.9			

In the recent years, new power plants have been established in the Mediterranean countries to use the biomass from the olive oil industry as a fuel for energy generation. This produces biomass fly ash (BFA) as a waste product.

The BFA used in this study was collected from the flue gas filters in a power plant located in Linares (Jaen, Spain). One of the main characteristics of this BFA is the high content of potassium. Other compounds which are relevant for soil stabilisation, such as silica, alumina and calcium oxide, are also present **(Table 12.2)**.

The dolomite ash (DA) is an industrial by-product derived from the incineration of dolomite minerals from a calcite quarry in Macael (Almeria, Spain). Dolomite is considered as a waste product and stored as spoil. The dolomite ash is obtained after calcination of the dry residue above 900ºC, which transforms the carbonates into separate calcium oxide and magnesium oxide **(Table 12.2)**.

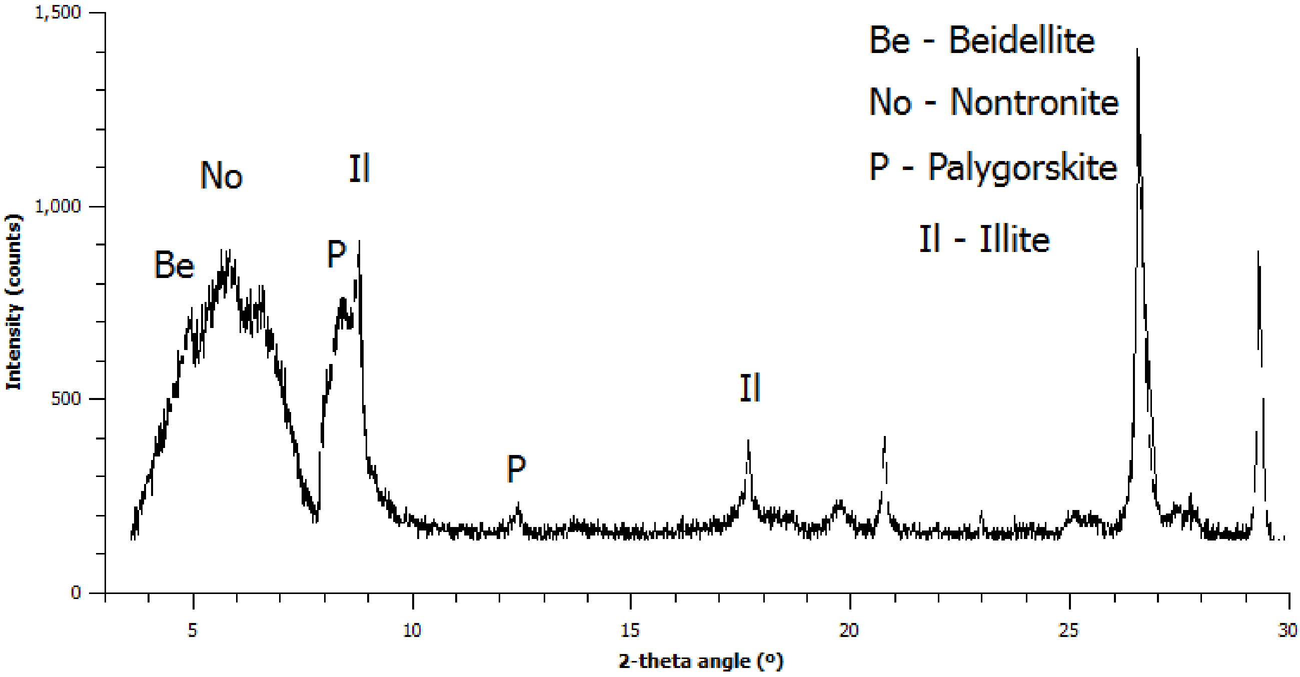


Figure 12.1. XRD pattern of natural clayey soil

12..2 METHODOLOGY

The soil test samples were prepared by mixing the natural clayey soil with the tested additives in the presence of water. The BFA and DA were added in dosages of 5% and 10% by mass of soil. The samples were cured for 7, 14 and 28 days before testing. As a control, a series of Portland cement-treated samples were also prepared. Several geotechnical tests, such as standard Proctor, Atterberg limits test, consolidation in oedometer and shear box test were carried out. The pH of the prepared specimens were also measured. A second set of samples treated with BFA and DA were prepared in an identical manner for X-ray diffraction analysis to investigate the effects of the additives on the mineralogical composition of the soil.

The soil-additive mixtures were initially prepared dry to create an even mixture (**Figure 12.2**). Water was then added in an amount enough to reach the liquid limit of the natural soil. The water, soil and additive were then mixed for at least ten more minutes to ensure an even mixture of soil and additive.

Fig. 12.2. Preparation of samples in a mixing tray

The prepared soils were stored in a curing room at 22±1ºC and humidity of 95±3%. The floor of the curing room was flooded in order to maintain a constant humidity level.

The following tests have been carried out: Atterberg limits, determination of pH, standard Proctor compaction test, consolidation tests in oedometer, shear box test, X-ray diffraction.

12.3 EXPERIMENTAL RESULTS

The effects of the additives on the compaction properties of the soil are presented in **Table 12.3**. In general, the addition of BFA and DA promoted a decrease in the MDD of the soil whilst the OMC increased. Those changes occurred regardless of the additive or the dosage used. However, the increase in the MDD and the decrease in the OMC increased with increasing amount of additive (**Figure 12.3**). The samples treated with DA also showed greater change in MDD and OMC than those treated with BFA.

Hossain and Mol (2011) states that the continuation of the pozzolanic reaction between the soil and the stabilisation agent promotes an increase in the water required and hence an increase in the optimum moisture content.

Table 12.3

Summary of results: engineering properties of treated samples

Sample	*Plasticity index (%)*	*MDD (gr/cm3)*	*OMC (%)*	*pH*	*Shear box*		*Oedometer*	
Original soil								
	43	1.63	23	8.7	83	20	1.10	0.99
5% Biomass fly ash								
7 days	33	1.54	26	9.4				
14 days	21	1.51	28					
28 days	22	1.46	28					
10% Biomass fly ash								
7 days	17	1.44	31	11.5				
14 days	16	1.60	23					
28 days	17	1.36	40		140	32	0.73	0.77
5% Dolomite ash								
7 days	21	1.40	33	11.6				
14 days	21	1.44	31					
28 days	21	1.25	40					
10% Dolomite ash								
7 days	14	1.20	45	12.4				
14 days	14	1.15	46					
28 days	14	1.14	48		127	29	0.38	0.61

Atterberg limit tests were carried out to evaluate the influence of the addition of BFA and DA on the consistency of the soil, as well as the evolution of plasticity index (PI) with curing time. The values of LL, PL and PI obtained for all the samples are summarised in **Table 12.3**.

The addition of BFA promoted a significant decrease in the plasticity index. The PI of the original soil (43%) was reduced to 21% after 15 days of curing for the specimen treated with 5% of BFA. In the case of 10% of BFA, the plasticity index was reduced to 16% after 15 days of curing. Further curing of the BFA showed little change in PI (**Figure 12.4**).

The addition of DA also caused a sharp decrease in PI (**Figure 12.4b**). The reduction of the PI occurred earlier in the BFA-treated samples, reaching a minimum at 2-days curing and then remaining unchanged with further curing. The final PI values were very similar for both the samples treated with BFA and DA.

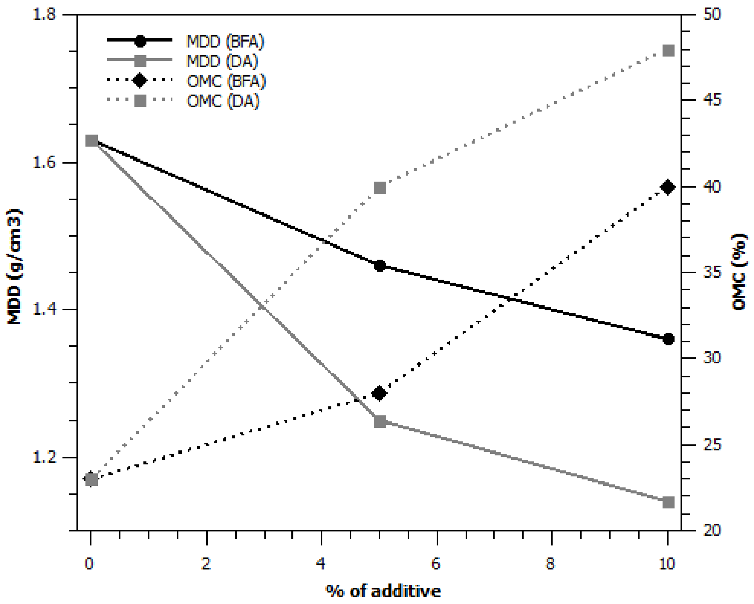


Fig. 12.3. Result of compaction tests: maximum dry density and optimum moisture content

As a control, Atterberg limit tests were conducted on samples treated with 5% and 10% Portland cement. The results showed that, in terms of consistency, the performance of the BFA and DA additives were very similar to that obtained using conventional soil treatment (**Figure 12.4**).

Although it is observed that the reduction in the PI increases with the percentage of the additive, this is least pronounced with Portland cement (**Figure 12.4c**). The PI of the cement-stabilised soil drops by only 1-3% with the addition of more additive. The greatest change in PI for cement-stabilised samples was noted during the first 7 days of curing where the PI decreased from 21 to 18%. The difference on further curing was negligible. On the other hand, BFA and DA treated samples show greater differences between the 5% and the 10% additive (**Figure 12.4a, 12.4b**).

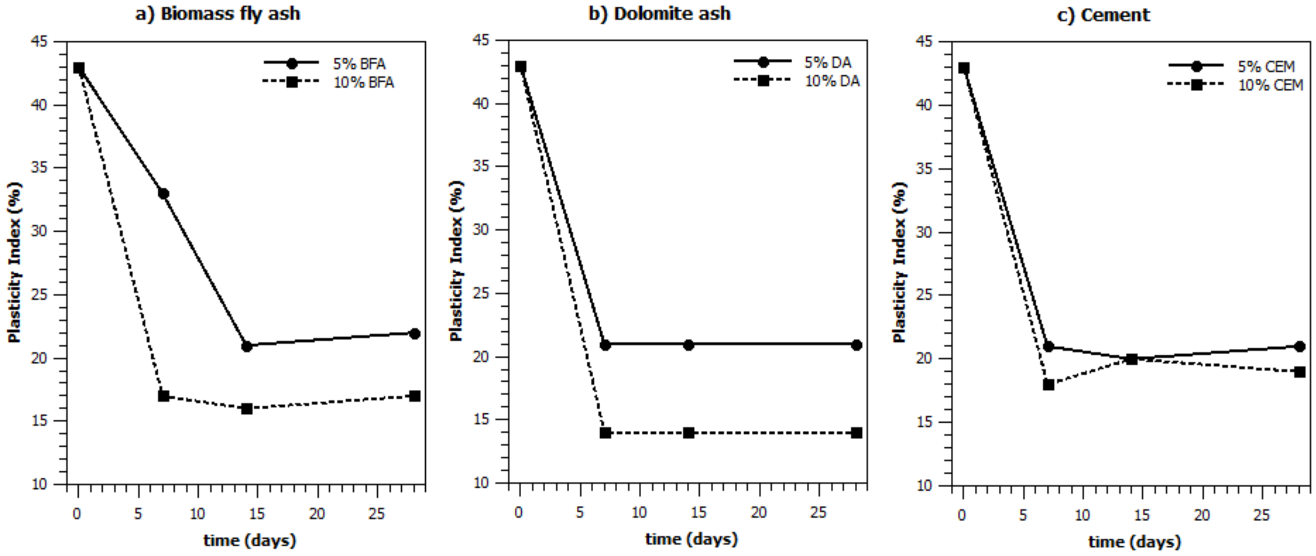


Fig. 12.4. Evolution of plasticity index: a) biomass fly ash; b) dolomite ash; c) cement

According to the definition of activity of clays (**Skempton, 1953**), the reduction of plasticity index indicates that the soil will experience lower volume change during wetting-drying cycles, which is a noticeable improvement in their engineering performance. The observed reduction of PI can be due to the promotion of particle flocculation-agglomeration on the addition of each additive. This effect is least pronounced with Portland cement (**Figure 12.4c**). All the additives used in this study ultimately change the natural soil from a high plasticity inorganic clay to an inorganic silt as the additives promote inter-particle bonding resulting in increasing grain size.

White (1949) showed the mineral composition of the soil influences the plasticity of the soil. In particular, the presence of mixed-layer of smectites is responsible for high plasticity. The reduction of PI of the samples treated with BFA and DA as well as those treated with Portland cement could also be the result of a reduction in the smectite present in the soil.

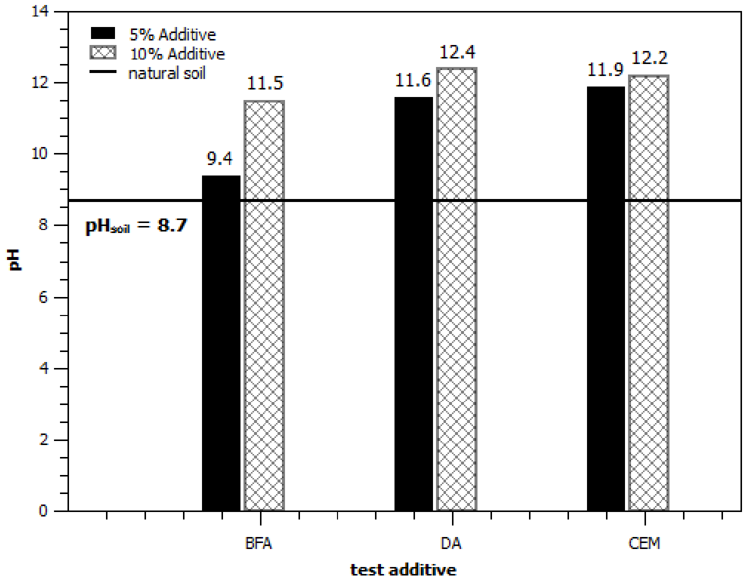


Fig. 12.5. pH values of the soils prepared (measurements according to BS ISO 10390:2005)

The pH of the samples was recorded in order to evaluate the influence of the addition of different quantities of BFA and DA. **Table 12.3** shows the results obtained in the samples after 21 days of curing. The pH values of the samples treated with Portland cement were also measured.

The natural soil had a pH of 8.7. The addition of 5% of BFA promoted an increase in pH. Increasing the amount of BFA caused a greater increase in pH. Similar changes are noted on the addition of DA. The addition of additives increases the soil alkalinity with a slightly greater increase measured on the DA (**Figure 12.5**). **Solanki and Zaman (2012)** shows that when using traditional stabilisation agents (calcium based binders), an increase in the pH of the soil is necessary for the pozzolanic reactions to occur. The pozzolanic reaction involves the interaction between silica (SiO_2) and alumina (Al_2O_3) present in the clay and the additive to form cementitious compounds (**Seco et al., 2012**). Furthermore, in clay-rich soils, such as the natural soil used in this study, the reduction in the amount of plastic and expansive clay, such as the smectite, becomes a priority for improving their engineering behaviour.

Oedometer (consolidation) and shear box testing was used to study the mechanical behaviour of the additive-treated soils. The consolidation test allows the calculation of the coefficient of compressibility (Cc) and the coefficient of swelling (Cs). The shear box test provides the peak strength and the angle of shearing resistance.

Consolidation tests were performed on the natural soil and the samples treated with 10% BFA and 10% DA after 28 days of curing **(Figure 12.6)**.

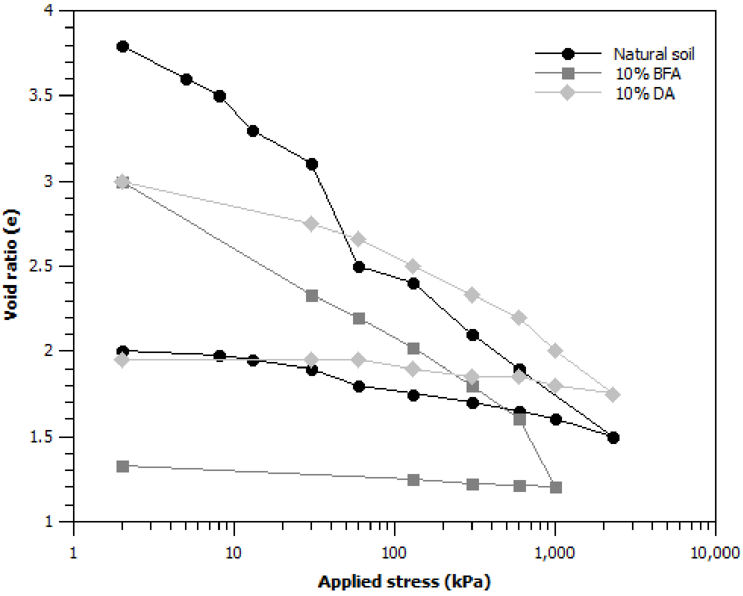


Fig. 12.6. Result of consolidation tests in oedometer: original soil, soil + 10% BFA and soil + 10% DA

The Cs of the untreated soil was 0.99. The addition of 10% of BFA reduced this value to 0.77. The addition of 10% of DA was even more effective reducing Cs to 0.61. The reduction in Cc was also observed to be greater for the samples treated with DA compared to BFA **(Table 12.3)**.

The shear box test results **(Figure 12.7a and Figure 12.7b)** show an increase of 70% in the peak value for the 10% BFA treated soil compared to the original soil. The 10% DA sample reached a peak of 127 kPa which is the same as the peak value of the BFA sample. Even though the two treated samples reached a very similar peak strength, the BFA-treated soil showed a brittle behaviour whereas the DA-treated soil behaved plastically.

The natural soil had an angle of shearing resistance (φ’) of 20º. The 10%-BFA-treated sample had the highest φ’ of 32°, whereas the 10%-DA-treated sample reached φ’ of 29° **(Figure 12.7b)**.

The shear box and the consolidation test results indicate an improvement of the mechanical properties of the original soil with the addition of BFA and DA. Firstly, there is the reduction of both Cc and Cs. Secondly, the shear strength of the soil significantly increases achieving values almost twice the initial strength. The angle of shearing resistance of the soil also significantly increased. The increase in both the strength and φ’ is due to the bonding of particles as a result of the pozzolanic action of the additives.

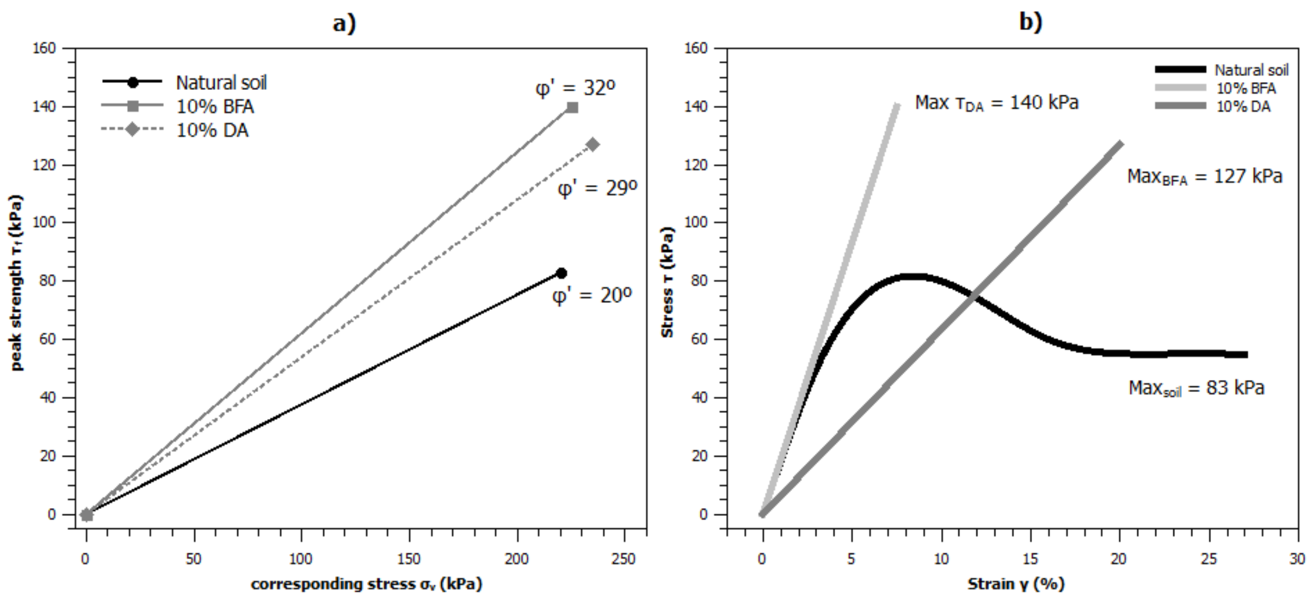


Fig. 12.7. Mechanical behaviour of natural and treated soils:
a) stress-strain relationship; b) angle of shearing resistance (φ')

The improvement in the mechanical properties of the soil increases their strength and ultimately their behaviour as foundation materials.

The original soil is predominantly formed of clay minerals, although small amounts of quartz (SiO_2) and calcite ($CaCO_3$) are also present. XRD analysis indicates that the clay fraction predominantly comprises smectite **(Figure 12.8)** in the form of beidellite, nontronite, etc. Non-expansive clay minerals, palygorskite and illite, are also detected **(Figure 12.8)**.

XRD analysis of the BFA and DA treated soils shows a reduction in the intensity of the peaks for all clay minerals. The smectite peak diminished dramatically after addition of 5% of additive and almost vanishes with the addition of 10% of additive (BFA or DA separately). The peaks for palygorskite and illite also reduce with a greater reduction observed in the samples treated with DA and the reduction increasing on increasing the amount of additive.

The sharp reduction of the smectite peaks after 28 days of curing is of vital importance. Smectites are responsible for the swelling potential and plasticity of the expansive soils **(Moore and Reynolds, 1997)**. The cation exchange capacity of the smectite can be activated by the presence of cations such as calcium, magnesium and potassium present in the BFA and DA **(Table 12.2)**.

Drief et al. (2002) claimed that the process of transformation of smectite into non-expansive phases is controlled by access to potassium, temperature and pH. **Elert et al. (2007)** confirmed that the destruction of smectite is likely to take place in an alkaline environment. Therefore, the reduction of the smectite content in the treated soils indicated by XRD analysis can be attributed to the cation exchange between the soil and the additives and to the increasingly alkaline environment created. It should be noted that the sample treated with 5% of BFA had the greatest clay minerals content of all the treated soils as a result of this soil having the lowest pH, i.e. least alkaline conditions.

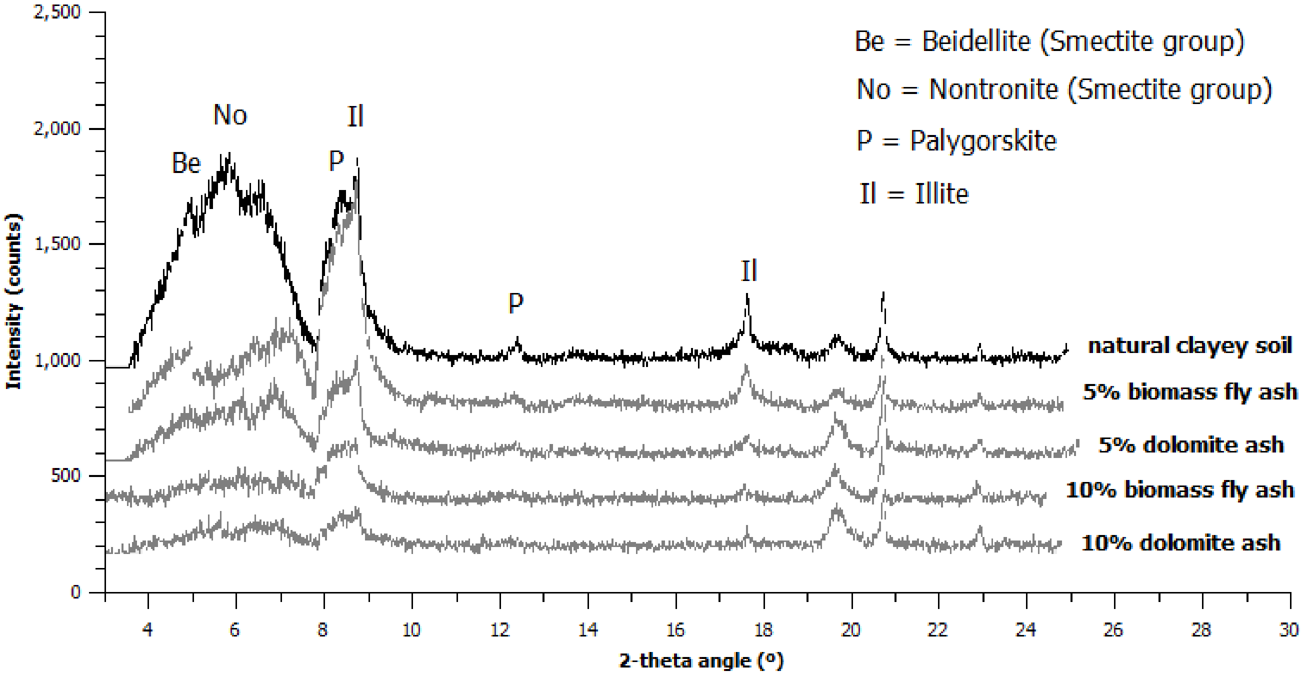


Fig. 12.8. XRD pattern of treated samples; comparison with the natural untreated soil

12.4 SUMMARY OF RESULTS AND CONCLUDING REMARKS

Both BFA and DA promoted improvement in the engineering properties of the original soil. The PI was reduced in similar proportions by the two additives. The shear strength of the BFA-treated samples was slightly higher, whilst the samples treated with DA proved to have better consolidation properties.

Increasing pH led to a greater reduction in smectite content resulting in improved plasticity and mechanical behaviour.

On the basis of the results obtained in this investigation, the following conclusions can be drawn:

- The non-conventional additives tested in this study (biomass fly ash and dolomite ash) presented promising properties for their use in soil stabilisation. In terms of comparison, both additives promoted similar results on the properties of the treated soil.

- The engineering properties of the natural clayey soil studied were significantly improved after addition of biomass fly ash and dolomite ash. The shear strength of the treated samples increased after treatment, whilst the coefficient of compressibility and coefficient of swelling were reduced for both BFA and DA-treated samples.

- In terms of plasticity, the addition of biomass fly ash and dolomite ash promoted a significant decrease in the plasticity index of the natural clayey soil. The results obtained were comparable with the results obtained using cement as a binder. The natural soil was originally classified as an inorganic clay of high plasticity, according to Casagrande chart of plasticity. However, all the specimens prepared in this study, regardless of the dosage or additive used (BFA, DA or cement) were found to be classified as inorganic silts after only 7 days of curing, except for the 5%-BFA-treated samples which reached the same classification after 15 days of curing.

- The addition of BFA and DA to the soil promoted an increase in the pH of the soil similar to the increase promoted by addition of cement. The alkaline environment provided by these non-conventional additives enabled the continuation of the pozzolanic reactions responsible for the formation of cementitious compounds and decomposition of clay minerals.

- The natural clayey soil had significant amounts of nontronite and beidellite, both of them members of the smectite group of clay minerals. The X-ray diffraction patterns of the treated samples showed that, after 28 days of curing, a significant reduction in the amount of smectite present in the soil had occurred. The presence of smectite, which can be reduced by the presence of potassium and alkaline environment, is responsible for the plasticity and swelling potential of the soils. A relationship between the reduction of smectite and the improvement of engineering properties of treated samples can be found.

EXPERIMENTAL RESULTS
PART V

Chapter 13

Quantification of the improvements achieved: numerical models

Abstract

The main objective of the last part (Part IV) of the experimental results is to provide the reader with a realistic and numerical idea of the degree of improvement achieved with the soil treatment, in terms of design of earthworks and embankments. To do this, two chapters have been prepared: Chapter 13 shows a sensitivity study of the most important features of the embankment (such as stress and deformations) to changes in the strength parameters of the soil. PLAXIS^{TM} 2D, a powerful piece of software based on finite element methods, is used. In Chapter 14, a numerical limit equilibrium method is used to evaluate the factor of safety of an embankment slope, depending on the properties of the soil used to construct that embankment. GeoStudio's SLOPE/W (version 2012) will be used to obtain the numerical results. In Chapter 13, first, the principles of design of earthworks and foundations using PLAXIS^{TM} 2D, are explained. Different soil models are available in PLAXIS^{TM} 2D: Mohr-Coulomb, Soft soils, Hardening Soils, Modified CLAM Clay, etc. Those materials can be studied in both drained and undrained states. In this study, the Mohr-Coulomb material (linear-elastic perfectly plastic) is used to model the materials forming the embankment. The modelled embankment has a simple geometry which could be encountered in linear infrastructures such as highways and railways. The physical properties and strength parameters such as particle size, Young´s modulus, cohesion, friction angle, etc. of the original soil (soil with low engineering properties) are modified in successive models and the effects of these modifications are studied.

13.1 METHODOLOGY OF THE STUDY. PARAMETERS USED

13.1.1 GEOMETRY OF THE MODEL

In this study, several models were prepared in Plaxis2D to evaluate the performance of an embankment with the use of finite element methods. A typical embankment of a linear infrastructure such as highways or railways was used in the study. For simplification purposes, only one half of the embankment was modelled. The geometry of the embankment, ground model and modelling conditions are described below:

- The height of the embankment was considered to be 11.5m. The width of the platform at the top level was 10m. The slope of the embankment was 2H:1V.

- The core of the embankment is made of a semi-granular fill. The fill is considered to be placed in layers of 1m maximum thickness to a height of 10m. There is a capping layer of 1.0m with an enhanced material on top of the core. The embankment is capped with a layer of pavement (thickness of 0.50m).

- The ground model is considered to be formed of a cohesive stiff clay. A depth of 10m is included in the model. All the soils were modelled as Mohr-Coulomb materials with drained behaviour. The water table was considered to be at the existing ground level. - Usual boundary conditions were applied to the models.

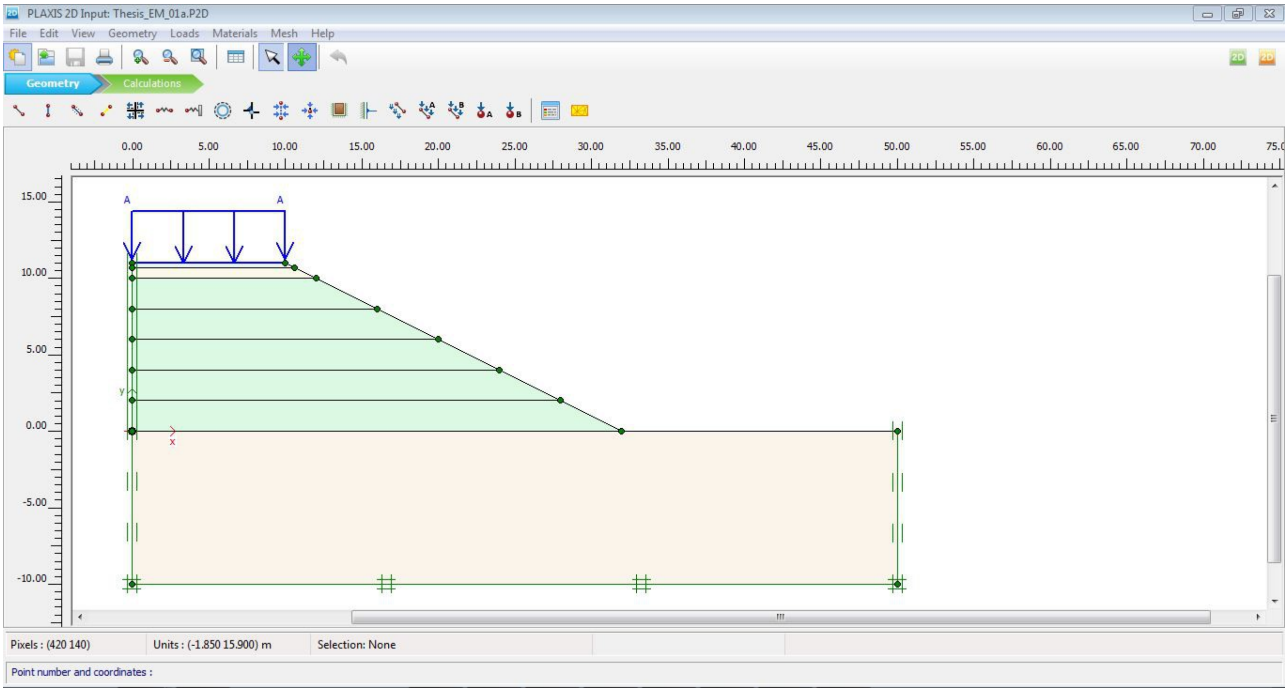


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Figure 13.1. Definition of geometry of embankments in Plaxis 2D

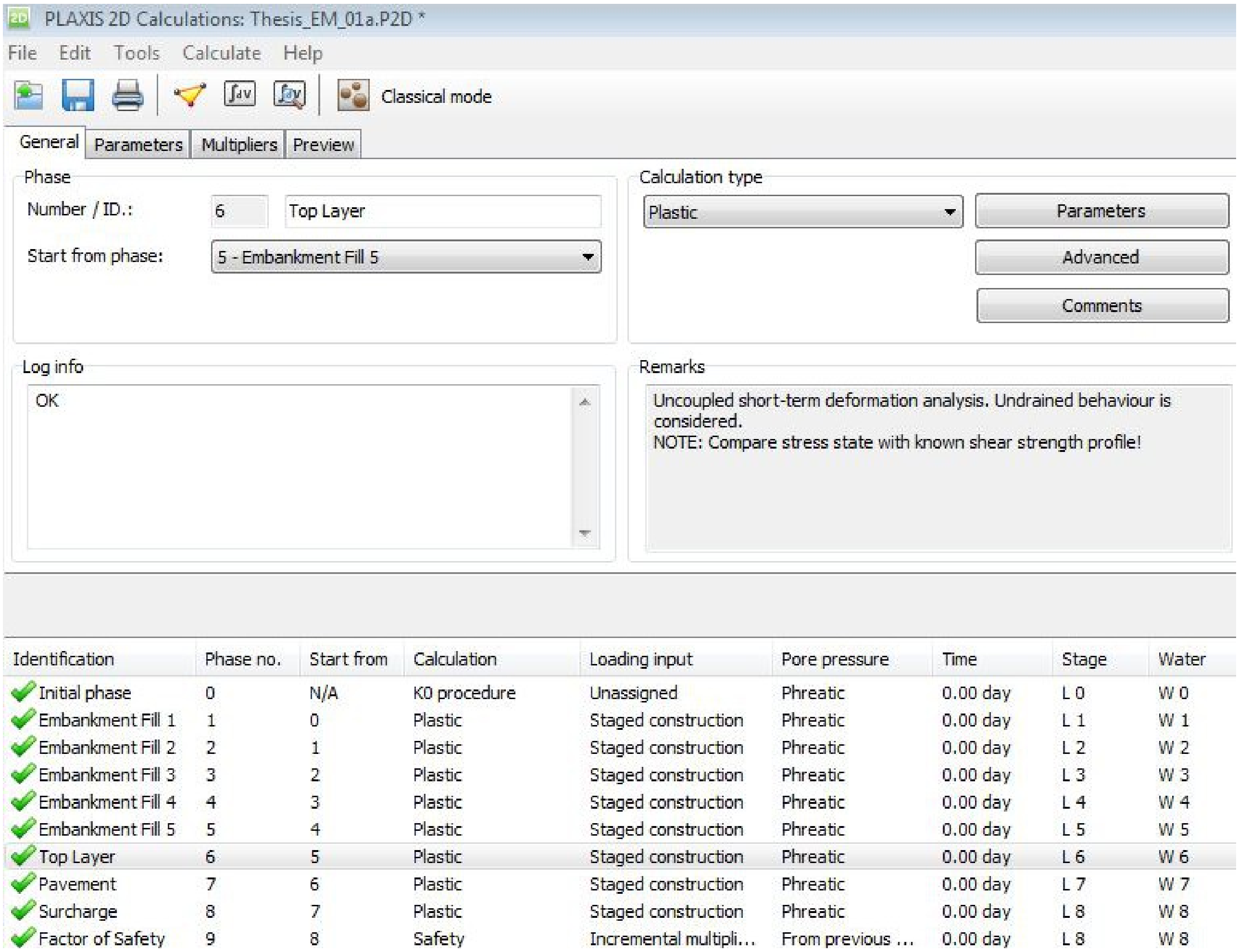
- A surcharge of 10kPa was considered to exist on top f the embankment under service conditions. This surcharge is model as a distributed load.

All the models prepared had the same geometry. The geometry is shown in **Figure 13.1**. A medium resolution of the grid was used to obtain the mesh of the FEM models.

13.1.2 STAGED CONSTRUCTION

A staged construction was considered in the FEM models. The following construction stages are shown in **Figure 13.2** and described below.

- Initial Phase: this is an equilibrium phase used to establish the initial stress of the soils in the model. At this construction stage only the existing ground formed of stiff clay was considered. Water table is located at the existing ground level.



Phase

Number / ID.: 6 Top Layer

Start from phase: 5 - Embankment Fill 5

Calculation type: Plastic

Log info

OK

Remarks

Uncoupled short-term deformation analysis. Undrained behaviour is considered.
NOTE: Compare stress state with known shear strength profile!

Identification	Phase no.	Start from	Calculation	Loading input	Pore pressure	Time	Stage	Water
✓ Initial phase	0	N/A	K0 procedure	Unassigned	Phreatic	0.00 day	L 0	W 0
✓ Embankment Fill 1	1	0	Plastic	Staged construction	Phreatic	0.00 day	L 1	W 1
✓ Embankment Fill 2	2	1	Plastic	Staged construction	Phreatic	0.00 day	L 2	W 2
✓ Embankment Fill 3	3	2	Plastic	Staged construction	Phreatic	0.00 day	L 3	W 3
✓ Embankment Fill 4	4	3	Plastic	Staged construction	Phreatic	0.00 day	L 4	W 4
✓ Embankment Fill 5	5	4	Plastic	Staged construction	Phreatic	0.00 day	L 5	W 5
✓ Top Layer	6	5	Plastic	Staged construction	Phreatic	0.00 day	L 6	W 6
✓ Pavement	7	6	Plastic	Staged construction	Phreatic	0.00 day	L 7	W 7
✓ Surcharge	8	7	Plastic	Staged construction	Phreatic	0.00 day	L 8	W 8
✓ Factor of Safety	9	8	Safety	Incremental multipli...	From previous ...	0.00 day	L 8	W 8

Figure 13.2. Plaxis 2D Calculation screen – construction stages

- Embankment Fill 1: This is the first construction stage. In this phase, the first 2m of fill are activated.

- Embankment Fill 2: This is the second construction stage. In this phase, the height of the embankment is increased up to 4m.

- Embankment Fill 3: This is the third construction stage. Six metres of embankment fill are activated.

- Embankment Fill 4: This is the fourth construction stage. Eight metres of embankment fill are activated.

- Embankment Fill 5: This is the fifth construction stage. The whole core of the embankment (fill) is activated.

- Top Layer: In this construction stage, the capping layer is activated (height = 11.0m).

- Pavement: This is the final construction stage. The embankment with the core, capping layer and pavement is activated in this phase (height = 11.5m).

- Surcharge: To model the service surcharge of 10kPa on top of the embankment, a distributed vertical load -10kPa is activated.

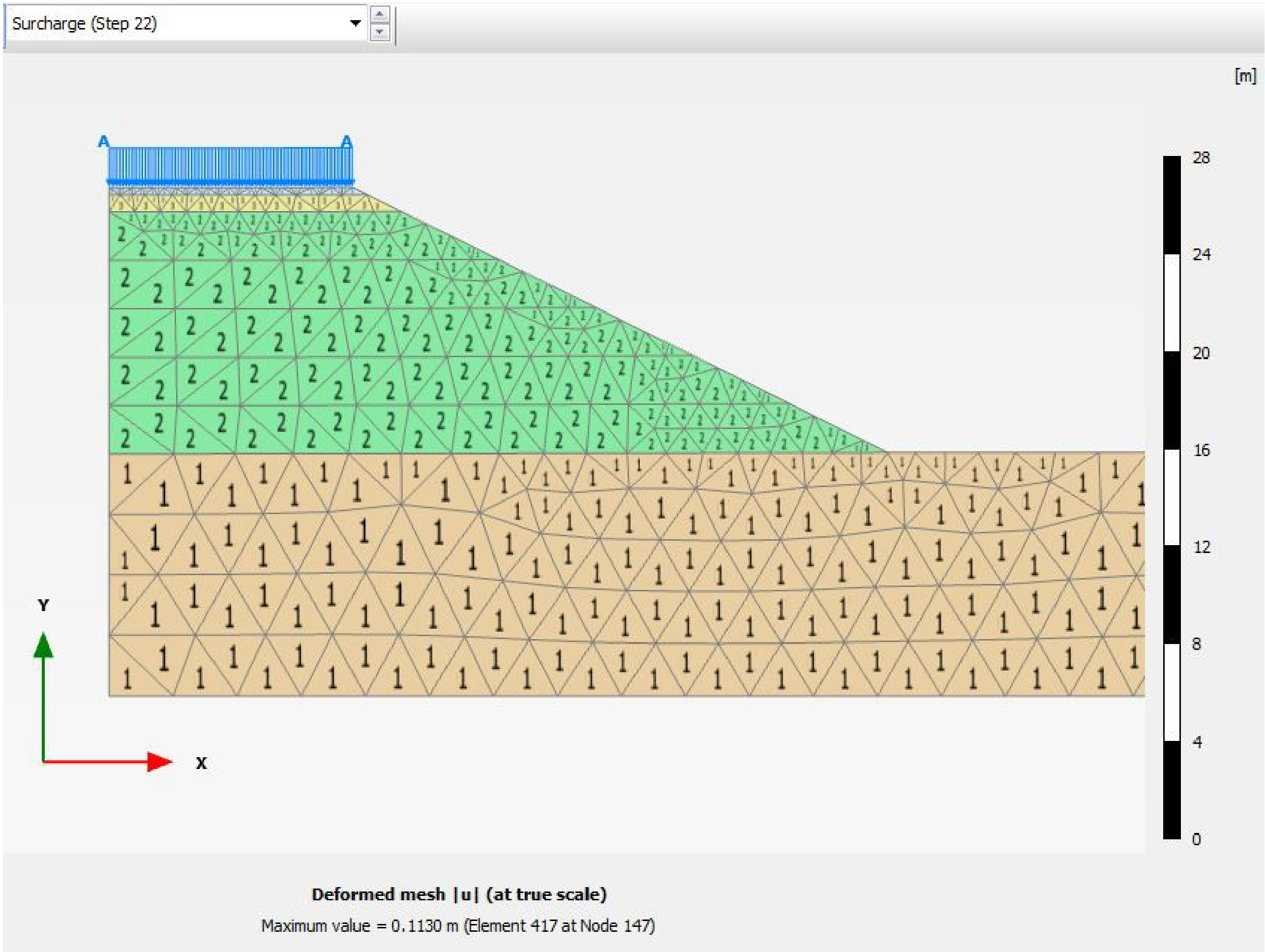
- Factor of safety: This stage is not a "Plastic" calculation but a Safety Analysis. In this phase, the material properties of all the soils are progressively reduced to produce a failure of the structure (embankment). The material properties of the soils at the point of the failure determine the factor of safety for comparison with the initial material properties. This is the last stage which was calculated in the models.

- All the previous phases were run for all the models prepared.

13.1.3 VISUALISATION OF OUTPUT RESULTS

The results of the analysis carried out was visualised with Plaxis 2D Output. The typical view of the deformed mesh is presented in **Figure 13.3**. The scale of the deformed mesh can be exaggerated to visualise the direction of the displacements occurred.

The output results included graphical presentation of plastic points, displacements (horizontal, vertical and total), total and effective stress and plot of curves to show the comparative displacements of points in different models.



Deformed mesh |u| (at true scale)

Maximum value = 0.1130 m (Element 417 at Node 147)

Figure 13.3. Plaxis 2D: Deformed mesh of Model a) after 10 kPa surcharge

13.1.4 MATERIAL PROPERTIES

Two different sets of models were prepared:

Models 1: In these models, the properties of the fill used for the core of the embankment were modified with a progressive increase of the friction angle (φ). The range of friction angle used was: 20° to 32°. This range was obtained from the experimental results of the previous chapters of this dissertation. The stiffness of the fill was defined by a Young's modulus of $15x10^3$ kPa. The material properties of the rest of the soils remained invariable.

Models 2: In these models, the increase of the friction angle in the fill was accompanied by an increase in the stiffness: Young's modulus from $9x10^3$ kPa to $30x10^3$ kPa. The material properties of the rest of the soils remained invariable.

A comprehensive explanation of the FEM models prepared can be found in **Table 13.1 and Table 13.2**.

The initial material properties of the soils used are presented in **Figure 13.4**. As explained, the strength parameters of the soil called “embankment fill” were modified in the different models. The properties of the rest of the soils remained invariable (**Figure 13.5**).

Figure 13.6 shows the results of a simulation of uniaxial and triaxial tests on the embankment fill of model 1a. The figure shows the envelope of the failure according to the Mohr-Coulomb criterion.

Table 13.1

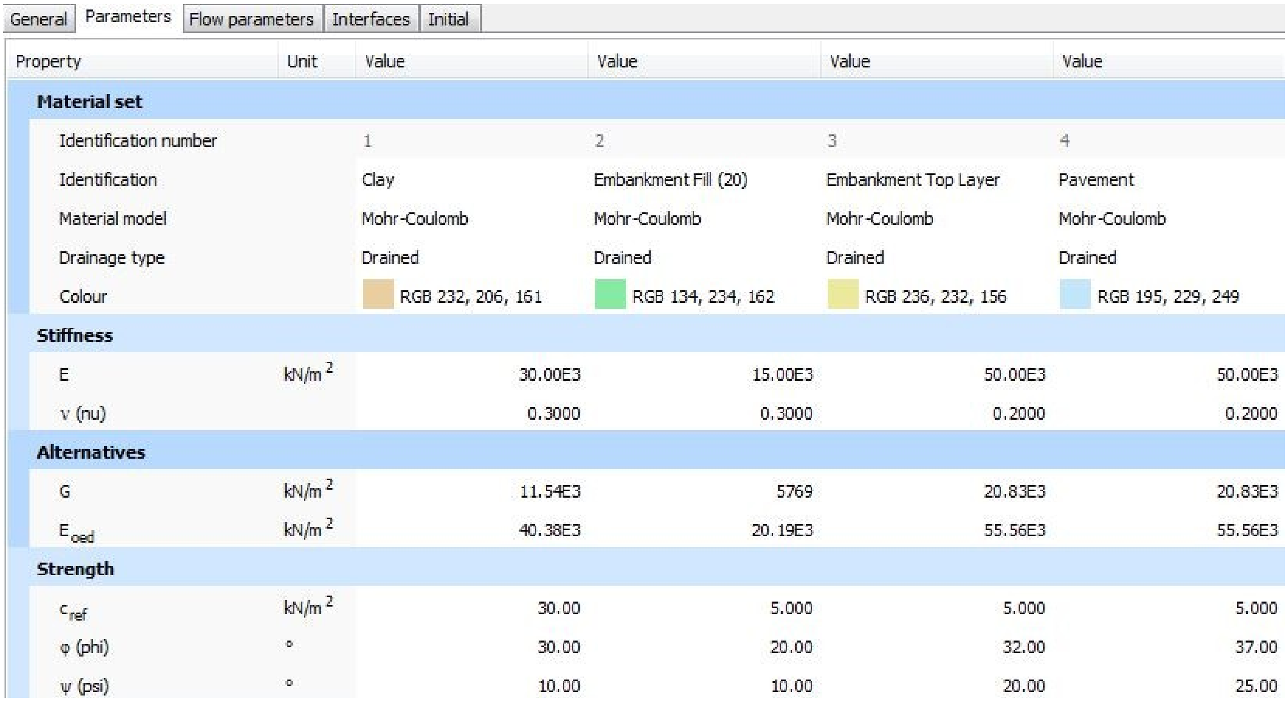
FEM Models prepared

Models 1		*Models 2*	
Clay (existing ground)	Fixed properties	Clay (existing ground)	Fixed properties
Embankment Top Layer	Fixed properties	Embankment Top Layer	Fixed properties
Pavement	Fixed properties	Pavement	Fixed properties
Embankment Fill	Variable friction angle. Fixed stiffness	Embankment Fill	Variable friction angle. Variable stiffness

Table 13.2

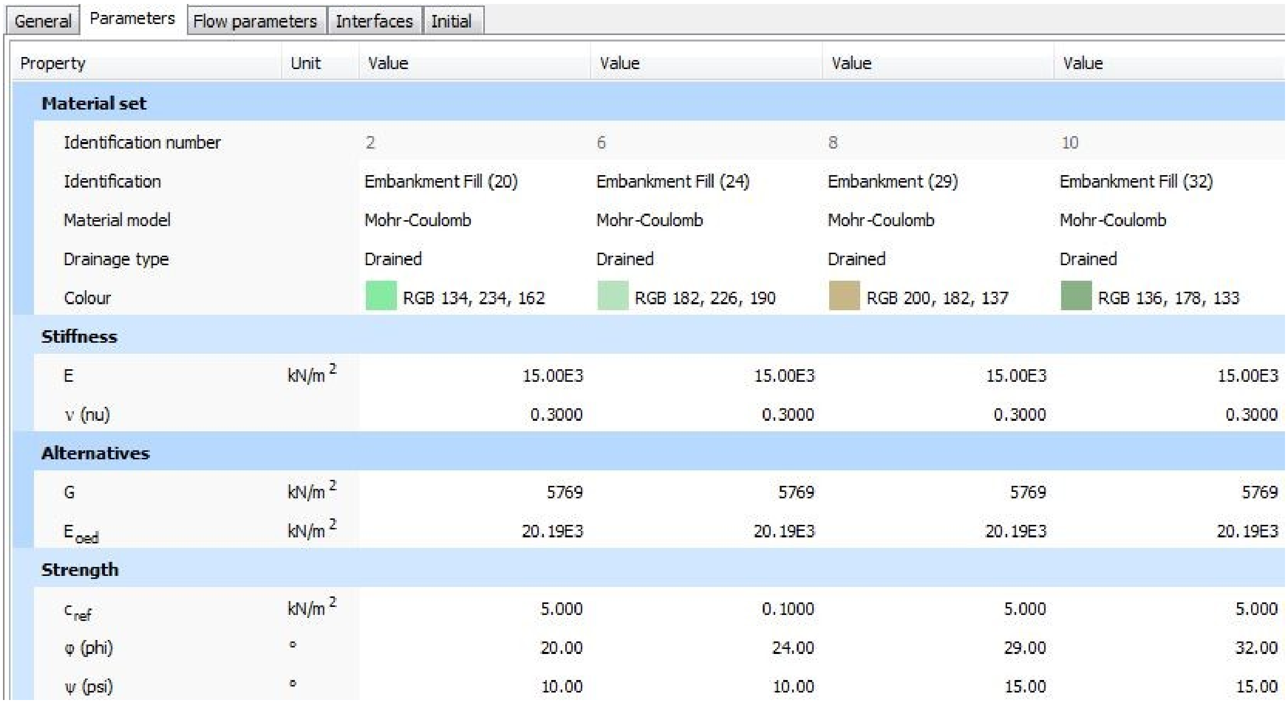
Modification of material properties of the fill in the FEM models prepared

Models 1	Unit weight, γ (kN/m^3)	Cohesion, c (kN/m^2)	*Friction angle, φ (°)*	Young's modulus (kN/m^2)
1a	17.0 – 20.3	5.0	20	15x10^3
1b	17.0 – 20.3	5.0	24	15x10^3
1c	17.0 – 20.3	5.0	29	15x10^3
1d	17.0 – 20.3	5.0	32	15x10^3
Models 2				
2a	17.0 – 20.3	5.0	20	9x10^3
2b	17.0 – 20.3	5.0	24	15x10^3
2c	17.0 – 20.3	5.0	29	20x10^3
2d	17.0 – 20.3	5.0	32	30x10^3



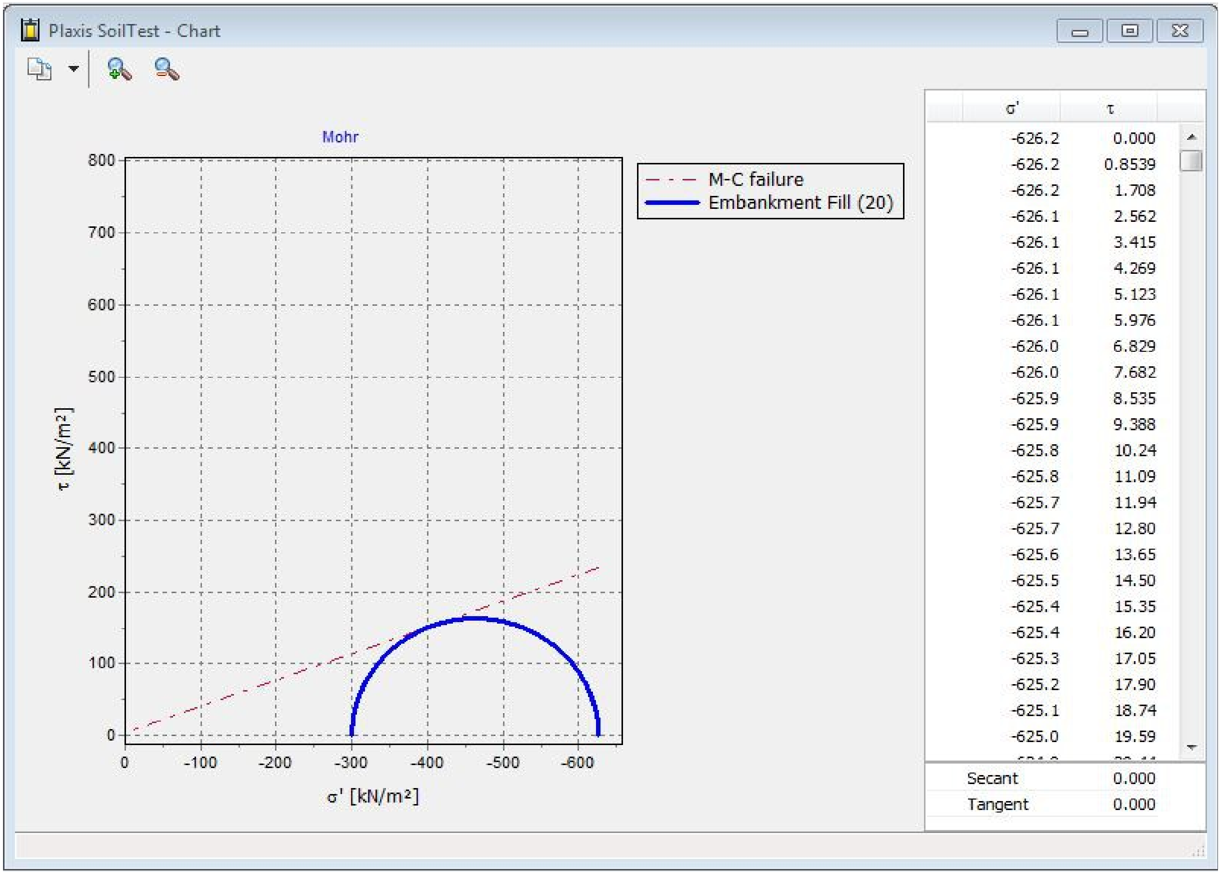
Property	Unit	Value	Value	Value	Value
Material set					
Identification number		1	2	3	4
Identification		Clay	Embankment Fill (20)	Embankment Top Layer	Pavement
Material model		Mohr-Coulomb	Mohr-Coulomb	Mohr-Coulomb	Mohr-Coulomb
Drainage type		Drained	Drained	Drained	Drained
Colour		🟫 RGB 232, 206, 161	🟩 RGB 134, 234, 162	🟨 RGB 236, 232, 156	🟦 RGB 195, 229, 249
Stiffness					
E	kN/m^2	30.00E3	15.00E3	50.00E3	50.00E3
ν (nu)		0.3000	0.3000	0.2000	0.2000
Alternatives					
G	kN/m^2	11.54E3	5769	20.83E3	20.83E3
E_{oed}	kN/m^2	40.38E3	20.19E3	55.56E3	55.56E3
Strength					
c_{ref}	kN/m^2	30.00	5.000	5.000	5.000
φ (phi)	°	30.00	20.00	32.00	37.00
ψ (psi)	°	10.00	10.00	20.00	25.00

Figure 13.4. Material properties of original soils in the Plaxis 2D model (example model 1a)

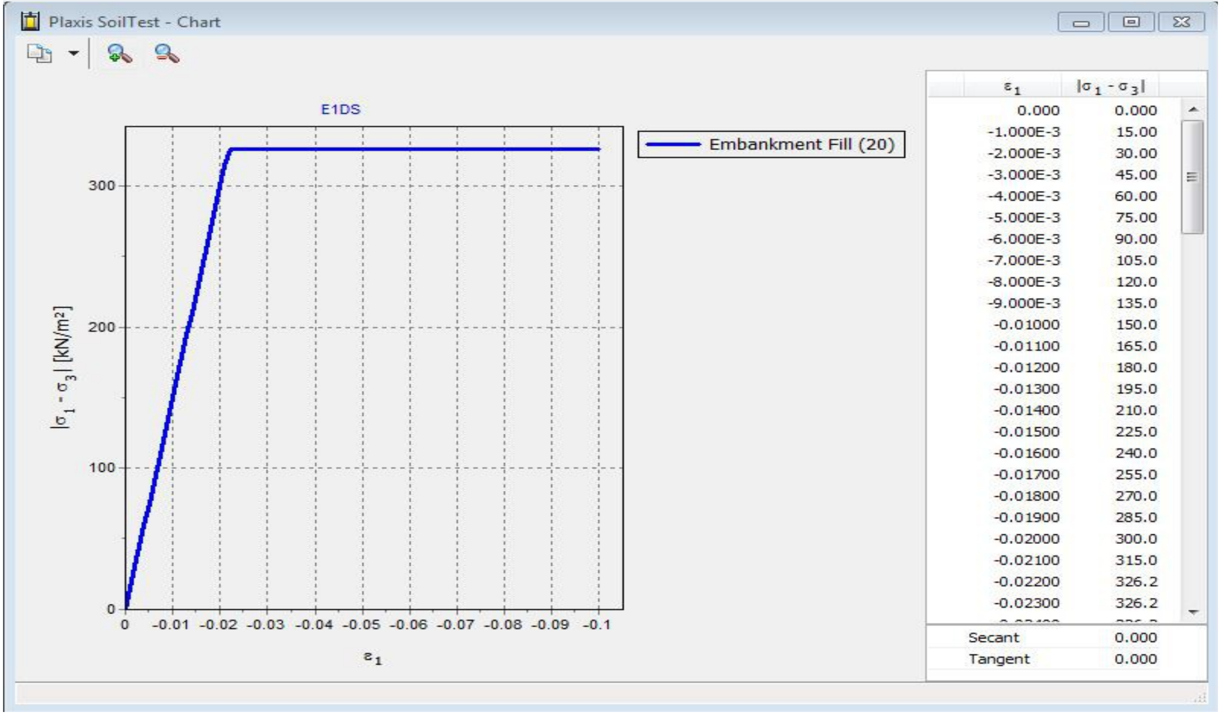


Property	Unit	Value	Value	Value	Value
Material set					
Identification number		2	6	8	10
Identification		Embankment Fill (20)	Embankment Fill (24)	Embankment (29)	Embankment Fill (32)
Material model		Mohr-Coulomb	Mohr-Coulomb	Mohr-Coulomb	Mohr-Coulomb
Drainage type		Drained	Drained	Drained	Drained
Colour		🟩 RGB 134, 234, 162	🟩 RGB 182, 226, 190	🟫 RGB 200, 182, 137	🟩 RGB 136, 178, 133
Stiffness					
E	kN/m^2	15.00E3	15.00E3	15.00E3	15.00E3
ν (nu)		0.3000	0.3000	0.3000	0.3000
Alternatives					
G	kN/m^2	5769	5769	5769	5769
E_{oed}	kN/m^2	20.19E3	20.19E3	20.19E3	20.19E3
Strength					
c_{ref}	kN/m^2	5.000	0.1000	5.000	5.000
φ (phi)	°	20.00	24.00	29.00	32.00
ψ (psi)	°	10.00	10.00	15.00	15.00

Figure 13.5. Material properties of fill in models 1a, 1b, 1c and 1d.



σ'	τ
-626.2	0.000
-626.2	0.8539
-626.2	1.708
-626.1	2.562
-626.1	3.415
-626.1	4.269
-626.1	5.123
-626.1	5.976
-626.0	6.829
-626.0	7.682
-625.9	8.535
-625.9	9.388
-625.8	10.24
-625.8	11.09
-625.7	11.94
-625.7	12.80
-625.6	13.65
-625.5	14.50
-625.4	15.35
-625.4	16.20
-625.3	17.05
-625.2	17.90
-625.1	18.74
-625.0	19.59
Secant	0.000
Tangent	0.000



ε_1	$\|\sigma_1 - \sigma_3\|$
0.000	0.000
-1.000E-3	15.00
-2.000E-3	30.00
-3.000E-3	45.00
-4.000E-3	60.00
-5.000E-3	75.00
-6.000E-3	90.00
-7.000E-3	105.0
-8.000E-3	120.0
-9.000E-3	135.0
-0.01000	150.0
-0.01100	165.0
-0.01200	180.0
-0.01300	195.0
-0.01400	210.0
-0.01500	225.0
-0.01600	240.0
-0.01700	255.0
-0.01800	270.0
-0.01900	285.0
-0.02000	300.0
-0.02100	315.0
-0.02200	326.2
-0.02300	326.2
Secant	0.000
Tangent	0.000

Figure 13.6. Simulation of triaxial test and uniaxial compressive test in fill of embankment

13.2 ANGLE OF INTERNAL FRICTION. SENSITIVITY STUDY (MODELS 1)

As previously explained, the models 1 were prepared to carry out a sensitivity study on the value of the friction angle. The friction angle (φ) in the models 1a, 1b, 1c and 1d was 20°, 24°, 29° and 32°, respectively. The models were run and the results are presented in this section. **Figure 13.7** and **Figure 13.8** shows the effective stress (plastic points) and the displacements the models in the stage of safety calculation.

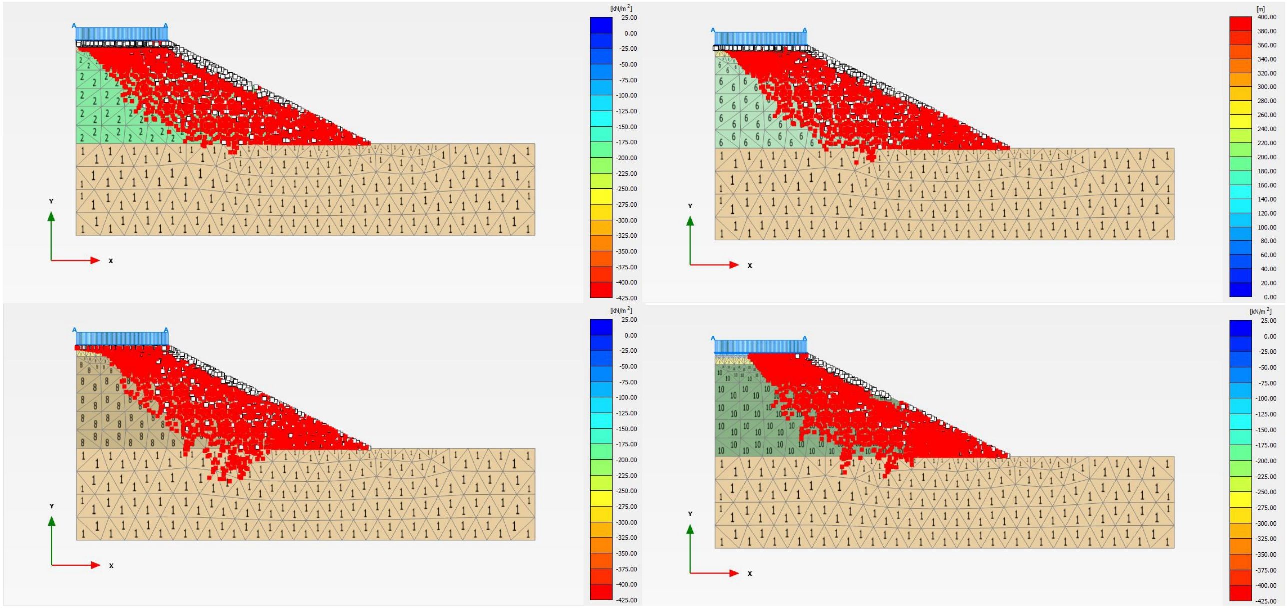


Figure 13.7. Plaxis 2D: Stress points in models 1a, 1b, 1c and 1d

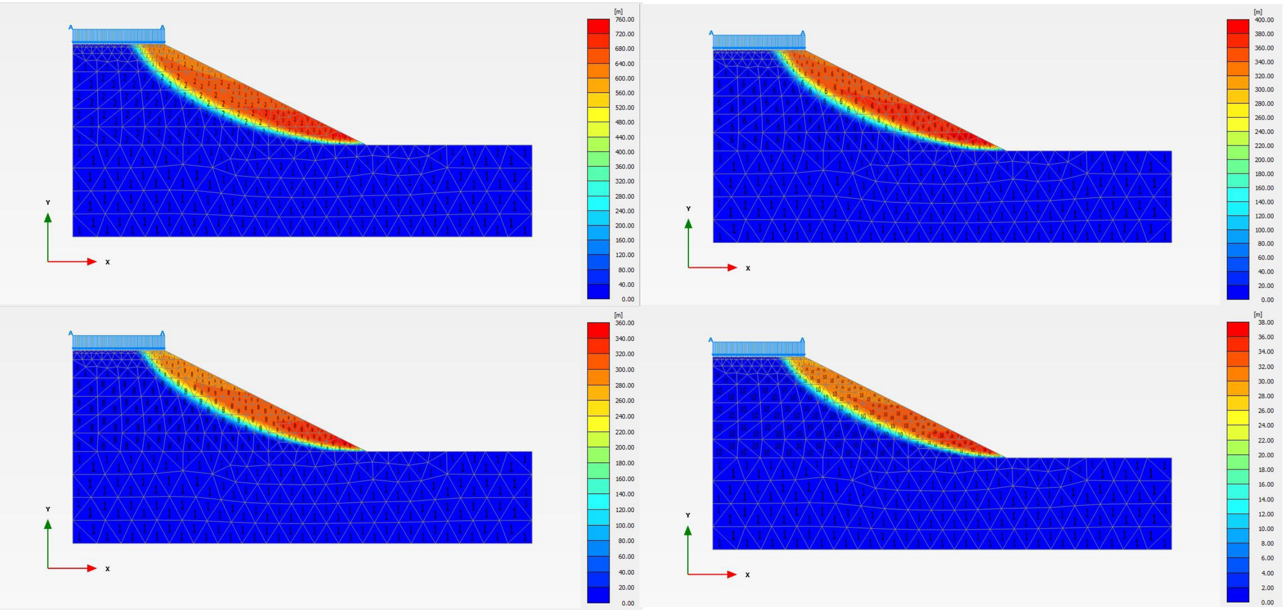


Figure 13.8. Plaxis 2D: Effective stress in models 1a, 1b, 1c and 1d

The graphical presentation of the effective stress results and the displacements in the models allows for the visualisation of the potential slope instabilities in the embankment. As the friction angle increases, the area of plastic points is reduced and the slice of potential instability becomes thinner.

The quantification of these improvements is carried out throughout the safety analysis. As explained, the factor of safety ($\sum M_{sf}$)is obtained due to an imposed reduction of the soil properties until the failure of the embankment takes place. The values of factor of safety and the material properties of the fill in the models 1 is presented in **Table 13.3**.

Table 13.3

Evolution of Factors of Safety in Models 1

Models	*Fill of embankment*	Friction angle, φ (°)	*Cohesion (kN/m^2)*	*Young's modulus (kN/m^2)*	**Factor of safety, ΣM_{sf}**
Model 1A	Fill 2	20	5	$15x10^3$	**1.107**
Model 1B	Fill 6	24	5	$15x10^3$	**1.291**
Model 1C	Fill 8	29	5	$15x10^3$	**1.540**
Model 1D	Fill 10	32	5	$15x10^3$	**1.693**

The soil used as a core of embankment in the model 1A had a friction angle of 20°. The associated factor of safety of this model after the construction stages and the application of the surcharge (10 kPa) was 1.107 which means that the model was stable (factor of safety > 1).

The increase of the friction angle (models 1B, 1C and 1D), even without the improvement of any other feature of the soil, promoted a significant increase in the factor of safety obtained after running the models and hence an increase in the overall stability of the embankment.

The factors of safety for the models 1B, 1C and 1D (friction angle of 24°, 29° and 32°) were 1.291, 1.540 and 1.693, respectively.

An analysis of the displacements was carried out. The results showed that the settlement of the embankment during the construction stages and the application of the surcharge (service conditions) did not vary in models 1A, 1B, 1C and 1D.

Therefore, despite the strong relationship obtained between friction angle of the fill and factor of safety in the embankment, the displacements in the embankment were not found to increase when the fill used as a core of embankment was improved only by increases of its friction angle.

13.3 YOUNG'S MODULUS. SENSITIVITY STUDY (MODELS 2)

A study was carried out on the relationship between the stiffness of the fill and the performance of the embankment. In models 2 (A, B, C and D), the parameters of the soils were remained invariable except for those of the fill. The friction angle was increased in the same values used for models 1. However, in models 2, the Young's modulus was also increased to obtain a higher stiffness of the fill. **Figure 13.9** shows the effective stress in model 2A in the stage of safety calculation. As it can be observed, the slice of slope instability is very similar to that observed in models 1.

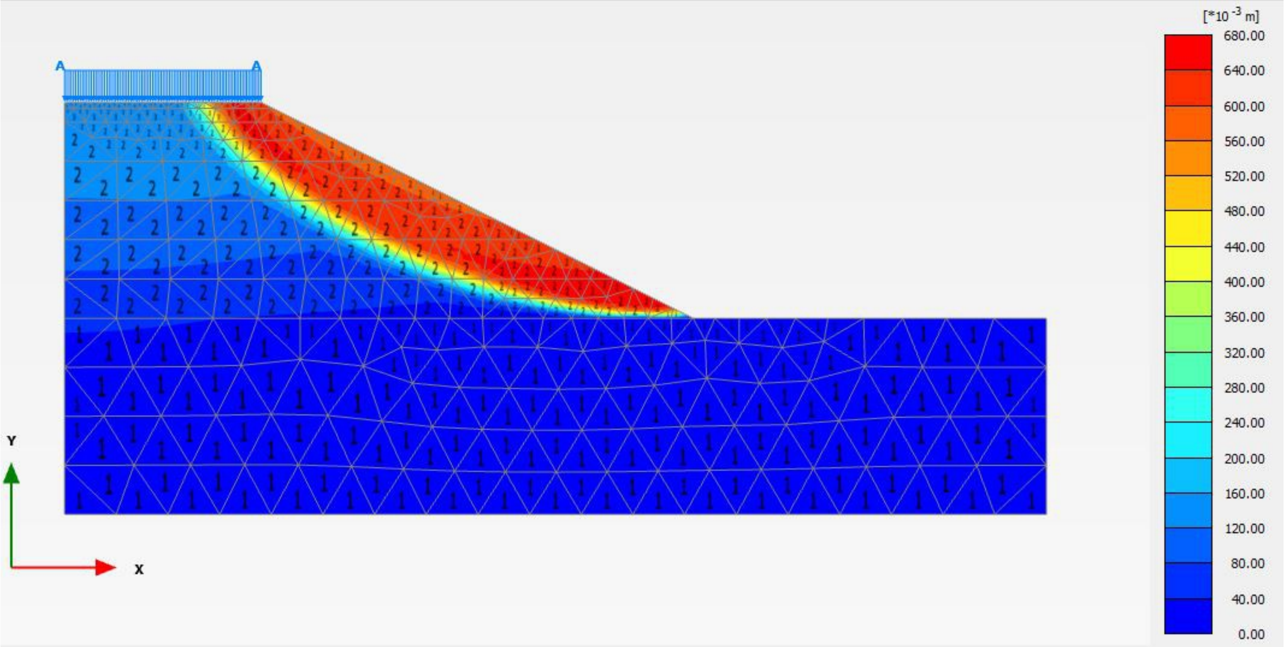


Figure 13.9. Effective stress in model 2A

The maximum value of settlements (vertical displacements, u_y) in the model takes place in the centre of the platform at the top level of the embankment **(Figure 13.10)**. In this location, at the level of the pavement, a point A was considered to be located. This point (marked on this node of the grid) was used to study the evolution of settlements.

Figure 13.11 plots the settlement of Point A (u_y) along the construction stages (steps of the FEM calculation in axis x). As it can be observed, most of the settlement of the point A takes place immediately after the construction, with a low influence of the application of surcharge, which does not promote further settlements. The comparison of the settlement among the models 1 (modification of friction angle, fixed value of stiffness, E = $15x10^3$ kPa), showed that the settlement did not vary from model 1A to model 1B, which shows a low dependency of the settlement on the friction angle.

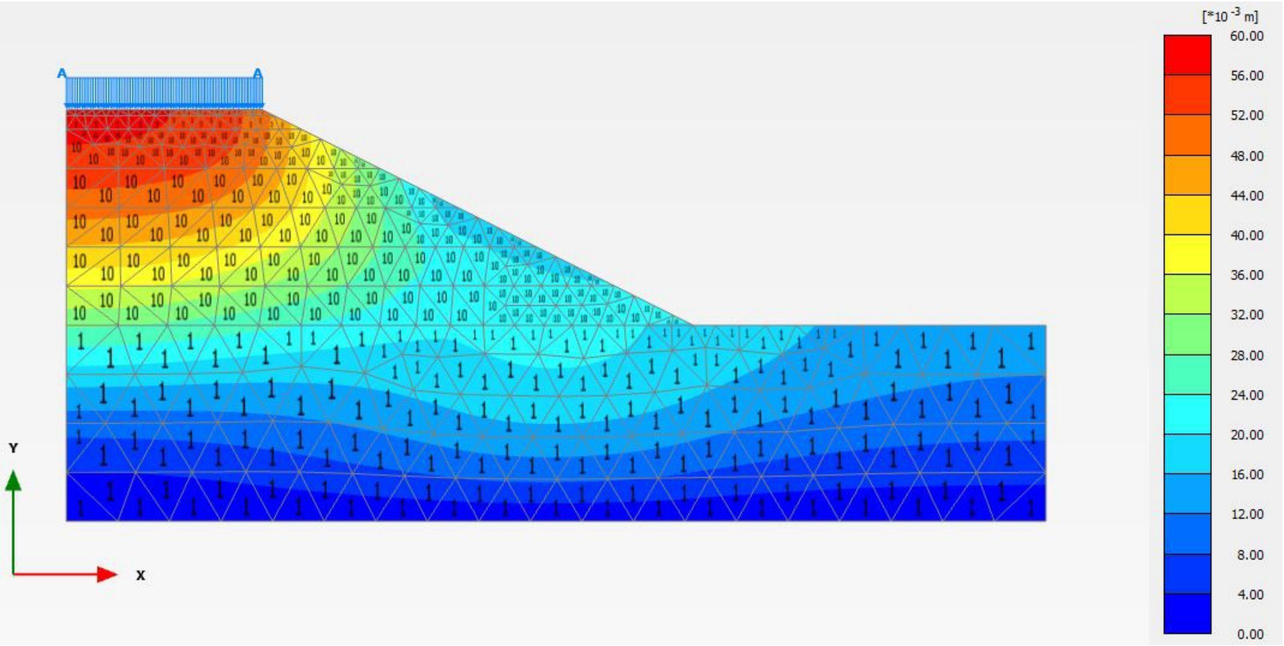


Figure 13.10. Settlement in model 2D (vertical displacement, u_y)

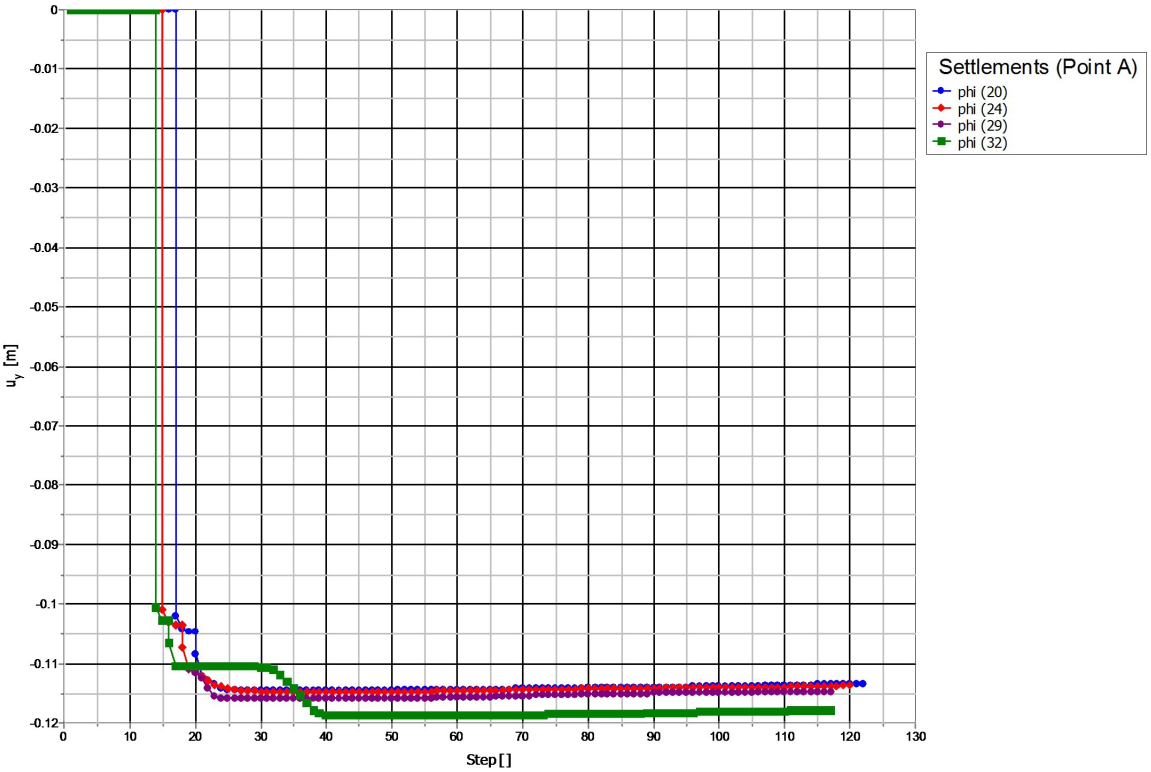


Figure 13.11. Comparison of settlement in models 1

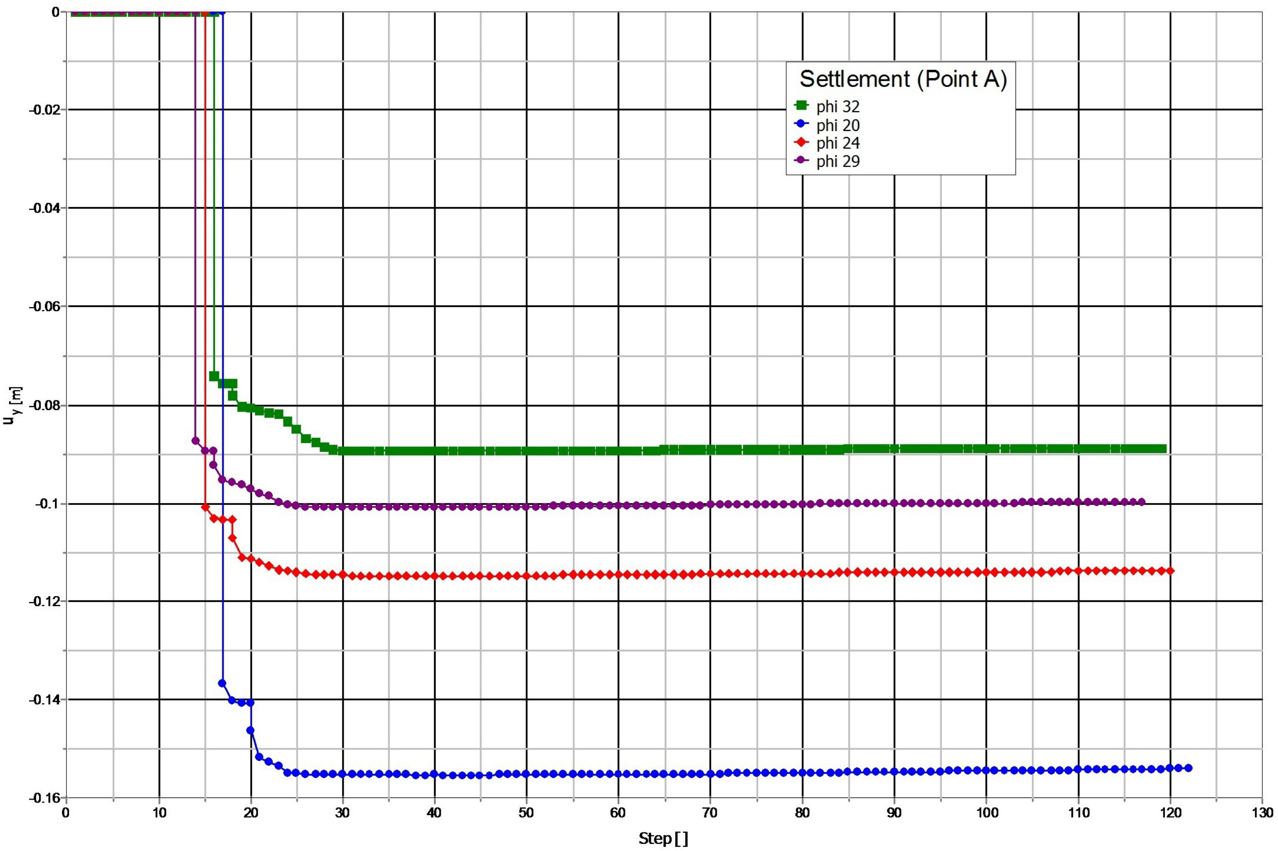


Figure 13.12 Comparison of settlement in Point A (top of embankment) in models 2

Figure 13.12 plots the settlement of point A in models 2A, 2B, 2C and 2D. From model 2A to model 2D, the friction angle increased from 20° to 32° and the Young's modulus increased from $9x10^3$ kPa to $30x10^3$ kPa. Model 2B and 1B are equal, hence the value of settlement in point A is the same. However, in the models 2, a very remarkable reduction of the settlement is obtained according to the increase of the stiffness.

The original value of settlement of 160mm in model 2A is reduced to 115mm in model 2B, 100mm in model 2C and 90mm in model 2D.

The results showed that the value of settlement, which is one of the main feature in the performance of the embankment was not influenced by the friction angle of the fill, which affected the factor of safety and effective stress. The settlement of the top of embankment was directly related with the stiffness of the fill used as core. The combined increase of the Young's modulus and the friction angle promoted a remarkable reduction of the settlement and hence a better performance of the embankment.

13.4 CONCLUSIONS

In this chapter, a sensitivity study on the variation of strength parameters (friction angle, Young's modulus) and its influence on the performance of an embankment was carried out with the use of numerical modelling: Plaxis2D FEM models. A typical embankment of linear infrastructure was modelled with a foundation of clays and a granular fill. The height of the embankment was 11.5 metres and the width of the platform at the top level was 20 metres. For simplification purposes, only one half of the embankment was included in the model.

The following concluding remarks can be drawn from the analysis:

- The FEM models prepared in this study showed that the overall performance of the embankment was very sensitive to variations in the strength parameters of the fill used: the factor of safety was significantly increased with slight increases of the friction angle and the settlement of the top layer decreased when Young's modulus was improved.

- An increase of 60% in the friction angle of the fill used to construct the embankment promoted an increase of 52% in the factor of safety of the embankment, without including any further modifications in the material properties.

- The settlement of the embankment was found to depend greatly on the stiffness of the soil used (fill). The settlement (Vertical displacement) of a point located at the top layer of the embankment did not diminish when the friction angle of the fill was improved. However, the increase of the Young's modulus from $9x10^3$ kPa to $30x10^3$ kPa promoted a significant reduction of the settlement at the top level, from 160mm to 90mm.

- Finite element methods are a powerful tool which can be used for the design of earthworks, embankments, foundations, etc. The use of numerical modelling and finite element methods led to a comprehensive analysis of the situation of the embankment and an evaluation of the effects of the changing material properties of the fill to be used in the construction.

Chapter 14

Slope stability in embankments formed of treated soils

Abstract

As explained in the previous chapter, a limit equilibrium analysis is carried out to assess the evolution of the slope stability factor of safety in an embankment formed of treated soils. As it can be observed, the factor of safety against failure of the slope is highly influenced by the material properties of the soil used to construct the embankment. To carry out this analysis, SLOPE/W is used. This piece of software belongs to a wider package called GeoStudio (version 2012 was used in this study) and it is developed and distributed by Geo-Slope™. The cross section of an embankment slope is modelled. The model is run in steps, each step using a soil with improved engineering properties than that used in previous one. SLOPE/W allows to assess the factor of safety of the whole system, for drained and undrained conditions, and it allows to identify the section of sliding failure most likely to occur. The use of numerical modelling was found to be very useful to evaluate the improvements achieved with the treatment of the soil. Those improvements were found to be quantitatively very important. Finally, the results obtained in this chapter are compared with those obtained using Plaxis 2D. Both using finite element methods and limit equilibrium analysis, the study proved that an original soil with poor strength parameters and fine-grained particle size distribution will promote greater displacements and lower factors of safety (against failure by bearing, sliding, etc.) and its behaviour could be insufficient to maintain the stability of the embankment after wetting-drying cycles.

14.1 METHODOLOGY OF THE STUDY

In this chapter, a calculation of the factor of safety against slope instabilities is carried out for an embankment with realistic geometry and soil properties. The geotechnical software Slope/W is used to develop this calculation.

Four models were prepared with the same geometry and soil properties (models A, B, C and D). The friction angle of the fill used as a core of the embankment was varied in the models to observe the influence of this value on the overall safety conditions of the embankments.

The initial geometry and soil properties used in the Slope/W models are the same of the Plaxis2D models explained in the previous chapter. The geometry of the embankment and main features of the model are described below:

- Embankment formed of 10m of fill ($E = 15x10^3$ kPa, cohesion $c = 5$ kPa, friction angle $\varphi = 20°$). There is a capping layer of an enhanced material on top of the fill with a thickness of 1metre. The top layer of the embankment is formed of a pavement with a thickness of 0.50m.

- The width of the platform at the top level is 10m. The embankment has a slope of 2H:1V. Only half of the embankment is included in the model, for simplification purposes.

- The existing ground is formed of a stiff clay. A depth of 8 metres of the existing soil is considered in the model.

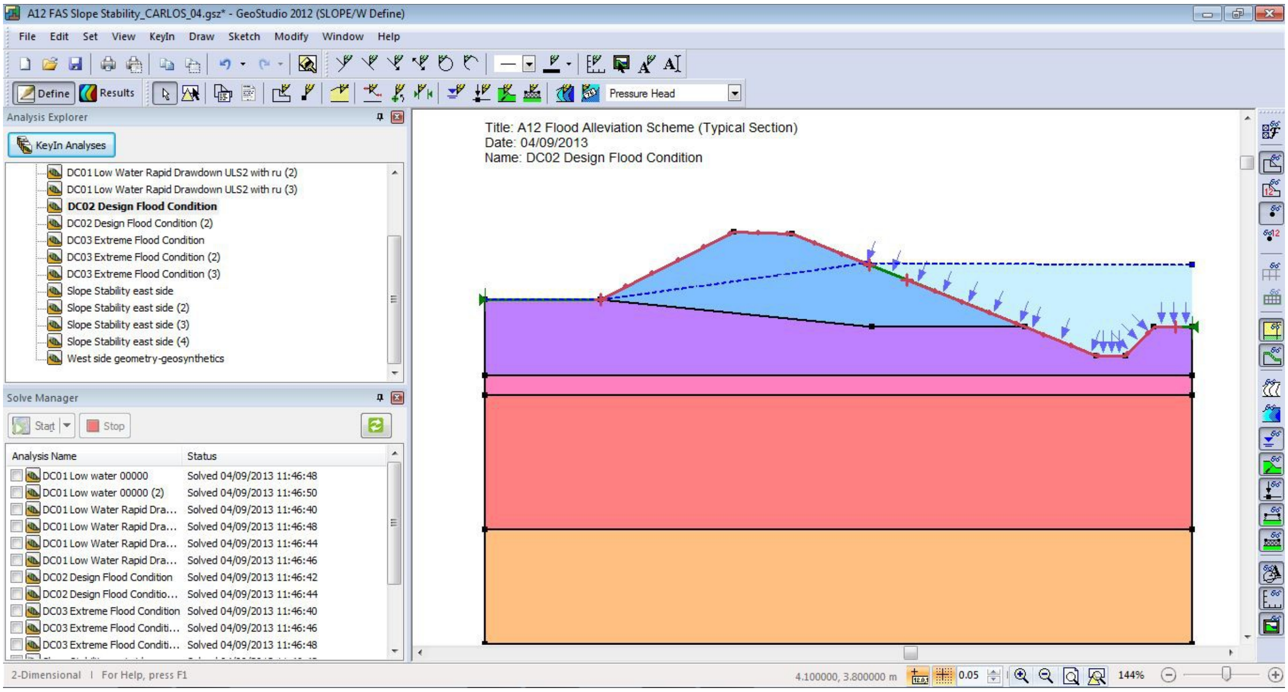
- All the soils are modelled as Mohr-Coulomb materials. The water table is considered to be located at the existing ground level.

14.2 SLOPE/W (GEOSTUDIO 2012)

SLOPE/W is formulated in terms of moment and force equilibrium factor of safety equations. For example, the Morgenstern-Price method satisfies both force and moment equilibrium. This general formulation makes it easy to compute the factor of safety for a variety of methods and to readily understand the relationships and differences among all the methods.

SLOPE/W can use finite element computed stresses from SIGMA/W or QUAKE/W to calculate a stability factor by computing both total shear resistance and mobilized shear stress along the entire slip surface. SLOPE/W then computes a local stability factor for each slice.

Probabilistic analysis can be performed by using normal distribution functions to vary soil properties and loading conditions. Using a Monte Carlo approach, SLOPE/W computes the probability of failure in addition to the conventional factor of safety.



DC01 Low Water Rapid Drawdown ULS2 with ru (2)
DC01 Low Water Rapid Drawdown ULS2 with ru (3)
DC02 Design Flood Condition
DC02 Design Flood Condition (2)
DC03 Extreme Flood Condition
DC03 Extreme Flood Condition (2)
DC03 Extreme Flood Condition (3)
Slope Stability east side
Slope Stability east side (2)
Slope Stability east side (3)
Slope Stability east side (4)
West side geometry-geosynthetics

Analysis Name	Status
☐ DC01 Low water 00000	Solved 04/09/2013 11:46:48
☐ DC01 Low water 00000 (2)	Solved 04/09/2013 11:46:50
☐ DC01 Low Water Rapid Dra...	Solved 04/09/2013 11:46:40
☐ DC01 Low Water Rapid Dra...	Solved 04/09/2013 11:46:48
☐ DC01 Low Water Rapid Dra...	Solved 04/09/2013 11:46:44
☐ DC01 Low Water Rapid Dra...	Solved 04/09/2013 11:46:46
☐ DC02 Design Flood Condition	Solved 04/09/2013 11:46:42
☐ DC02 Design Flood Conditio...	Solved 04/09/2013 11:46:44
☐ DC03 Extreme Flood Condition	Solved 04/09/2013 11:46:40
☐ DC03 Extreme Flood Conditi...	Solved 04/09/2013 11:46:46
☐ DC03 Extreme Flood Conditi...	Solved 04/09/2013 11:46:48

Title: A12 Flood Alleviation Scheme (Typical Section)
Date: 04/09/2013
Name: DC02 Design Flood Condition

Figure 14.1 Slope/W Screenshot

The main features of this program are listed below:

- Limit equilibrium methods include Morgenstern-Price, GLE, Spencer, Bishop, Ordinary, Janbu and more. Soil strength models include Mohr-Coulomb, Spatial Mohr-Coulomb, Bilinear, Undrained (Phi=0), anisotropic strength, shear/normal function, and many types of strength functions.

- Specify many types of interslice shear-normal force functions. Pore-water pressure options include Ru coefficients, piezometric lines, pressure contours, a grid of values, spatial functions, or finite-element computed heads or pressures.

- Define potential slip surfaces by a grid of centres and radius lines, blocks of slip surface points, entry and exit ranges, fully specified shapes, or automatic. Use probabilistic soil properties, line loads and piezometric lines. Transient stability analyses.

14.3 MODELS PREPARED

The initial material properties of the soils included in the model are presented in **Table 14.1**. All the soils were modelled according to the Mohr-Coulomb criterion.

The friction angle of the fill of embankment in model A was φ = 20°. However, this value of friction angle was increased in models B, C and D to study the effect of this parameter on the overall performance of the embankment throughout the observation of modifications in the factor of safety. **Table 14.2** presents the variations applied to the soil properties of the fill in models A, B, C and D.

Table 14.1

SLOPE/W Model. Material Properties

Soil	*Model*	*Unit weight (kN/m^3)*	*Cohesion (kN/m^2)*	Friction angle, φ (°)
Clay	Mohr-Coulomb	24	30	30
Fill of Embankment	Mohr-Coulomb	20	5	20
Top Layer	Mohr-Coulomb	21	5	32
Pavement	Mohr-Coulomb	21	5	37

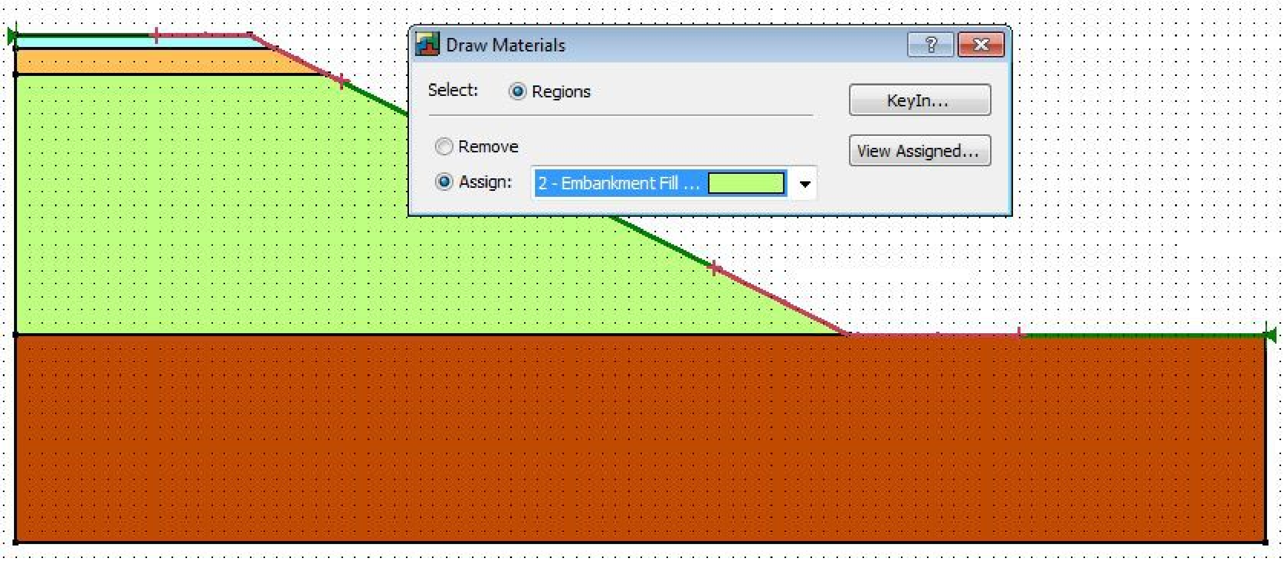
Table 14.2

SLOPE/W Model. Material Properties of Fill

Model	*Fill of embankment*	*Model*	*Unit weight (kN/m^3)*	*Cohesion (kN/m^2)*	Friction angle (°)
Model A	Fill 1	Mohr-Coulomb	20	5	20
Model B	Fill 2	Mohr-Coulomb	20	5	24
Model C	Fill 3	Mohr-Coulomb	20	5	29
Model C	Fill 4	Mohr-Coulomb	20	5	32

Figure 14.2 and **Figure 14.3** show the definition of the geometry of the model and the input of material properties in Slope/W, respectively. **Figure 14.4** shows the definition of the surcharge in the Slope/W models. A surcharge of 10 kN/m^2 due to the service conditions of the embankment was applied as a distributed vertical load.

Prior to the development of the calculations in Slope/W, the potential slip surface must be defined. The slip surface is defined by the range of locations of the upper and lower points of the slip surface. The definition of those points has a certain influence on the overall results of the numerical calculation. In the models prepared in this study, the potential slip surface of the slope was conservatively defined by the wide selection of points located at the top of the embankment and at the toe level (**Figure 14.5**).



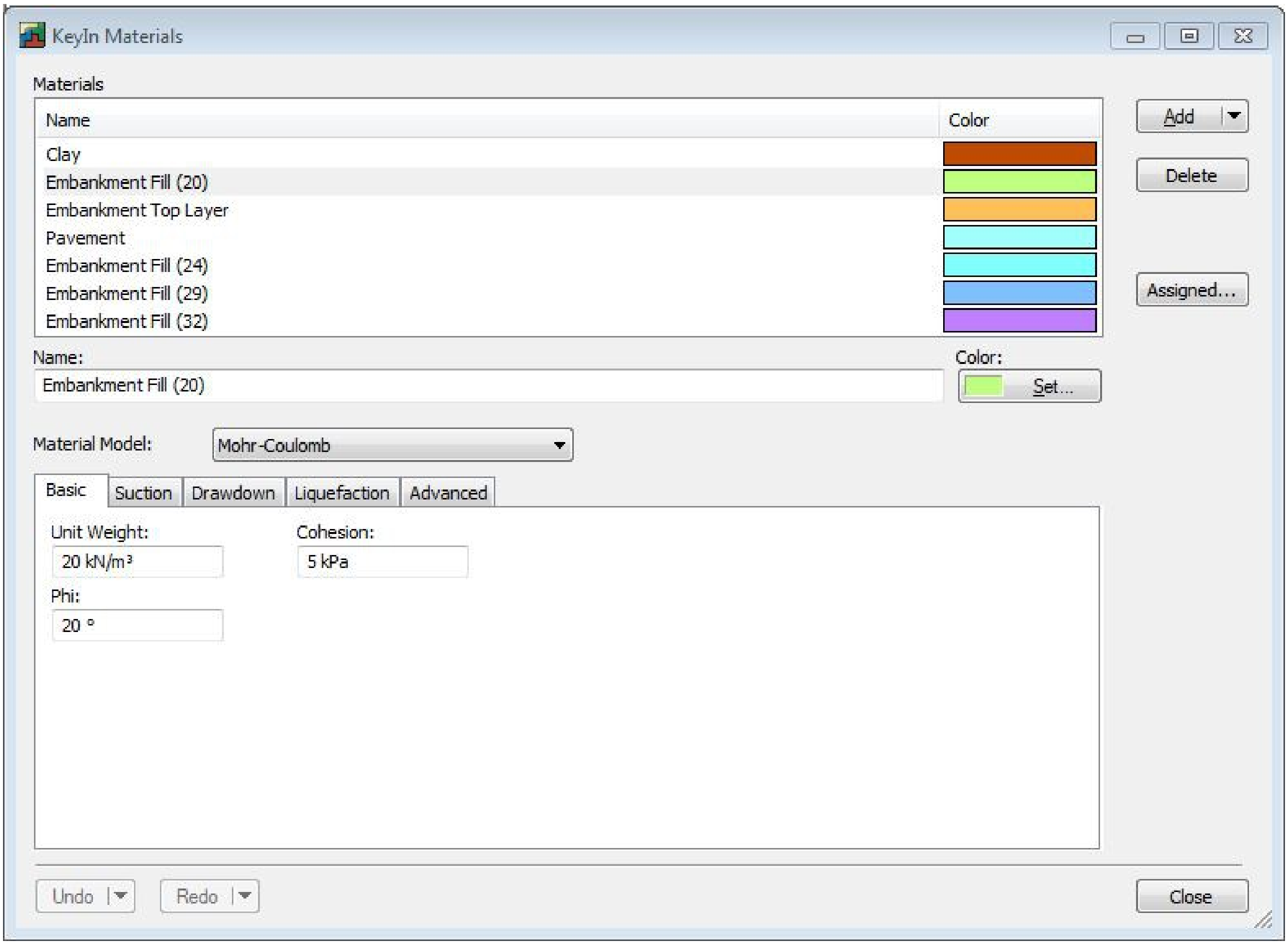
Draw Materials

Select: ● Regions

○ Remove

● Assign: 2 - Embankment Fill ... 🟩

Figure 14.2. Geometry of the SLOPE/W model



KeyIn Materials

Materials

Name	Color
Clay	🟫
Embankment Fill (20)	🟩
Embankment Top Layer	🟧
Pavement	🟦
Embankment Fill (24)	🟦
Embankment Fill (29)	🟦
Embankment Fill (32)	🟪

Name: Embankment Fill (20)

Color: 🟩

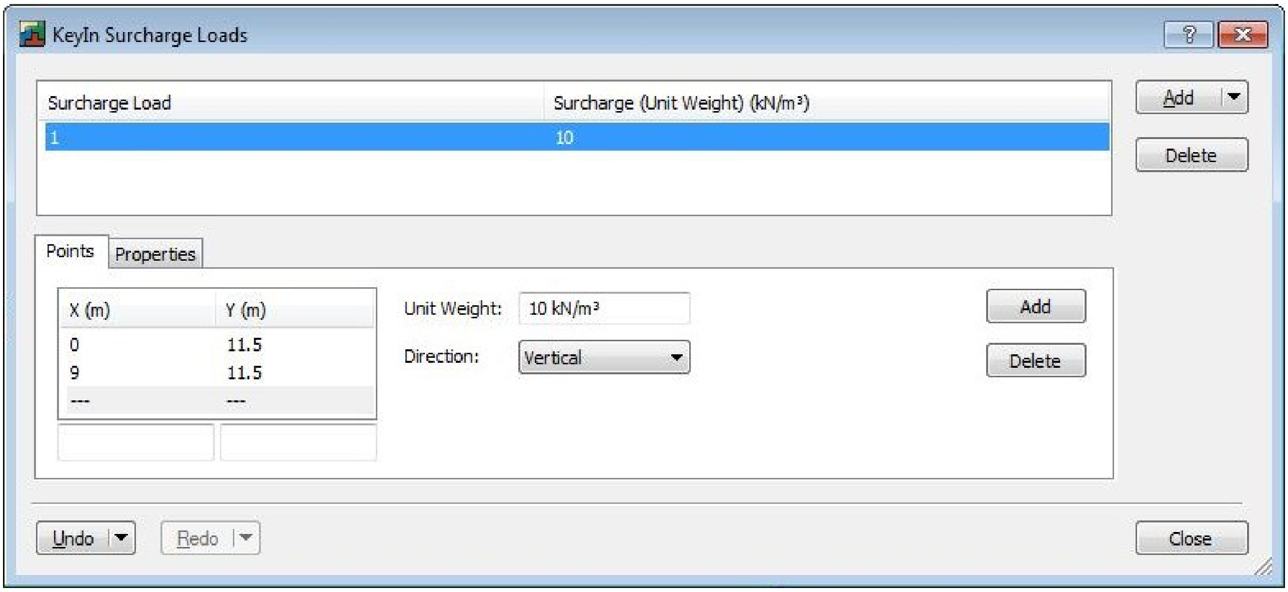
Material Model: Mohr-Coulomb

Unit Weight: 20 kN/m³

Cohesion: 5 kPa

Phi: 20 °

Figure 14.3. Definition of material properties in SLOPE/W



KeyIn Surcharge Loads

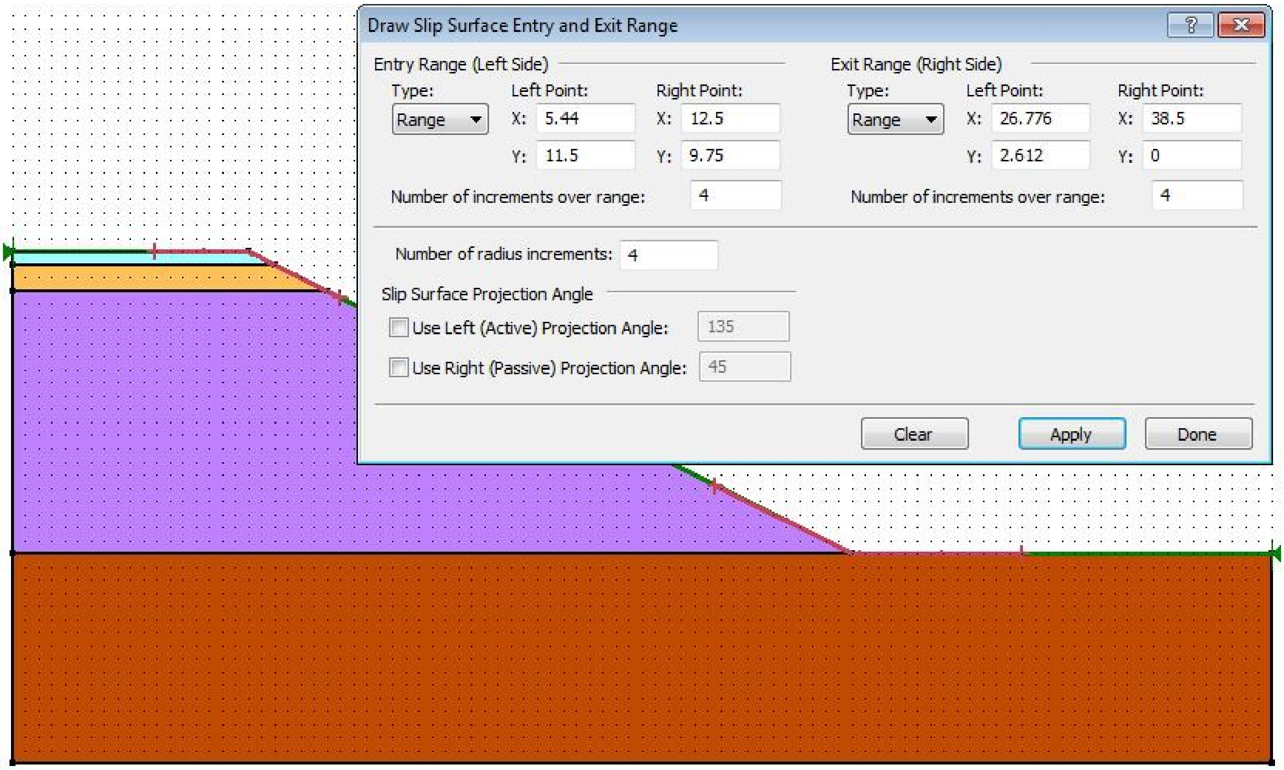
Surcharge Load	Surcharge (Unit Weight) (kN/m^3)
1	10

X (m)	Y (m)
0	11.5
9	11.5
---	---

Unit Weight: 10 kN/m^3

Direction: Vertical

Figure 14.4. Definition of service surcharge on top of embankment (q = 10 kN/m^2)



Draw Slip Surface Entry and Exit Range

Entry Range (Left Side)

Type: Range

Left Point: X: 5.44 Y: 11.5

Right Point: X: 12.5 Y: 9.75

Number of increments over range: 4

Exit Range (Right Side)

Type: Range

Left Point: X: 26.776 Y: 2.612

Right Point: X: 38.5 Y: 0

Number of increments over range: 4

Number of radius increments: 4

Slip Surface Projection Angle

☐ Use Left (Active) Projection Angle: 135

☐ Use Right (Passive) Projection Angle: 45

Figure 14.5. Definition of slip surface in SLOPE/W

14.4 RESULTS AND DISCUSSION

The models prepared were run and the factor of safety for each of the models was obtained.

The initial model (model A) represent an embankment formed of a fill with a friction angle of 20°. The slope of the embankment in this model is stable, with a factor of safety of FOS = 1.105, according to the results of the Slope/W calculation (**Figure 14.6**).

The model B represent the same conditions of model A, with an increase in the friction angle of the fill, from 20° to 24°. As a result, the factor of safety of the slope increased to a value of FOS = 1.287 (**Figure 14.7**).

In model C, the material properties of the soils and the geometry of the embankment remained invariable, except for a further increase in the friction angle of the fill, from 24° to 29°. According to the results of the numerical model, this further increase in the friction angle had a positive influence on the stability of the slope which increased to a factor of safety FOS = 1.521.

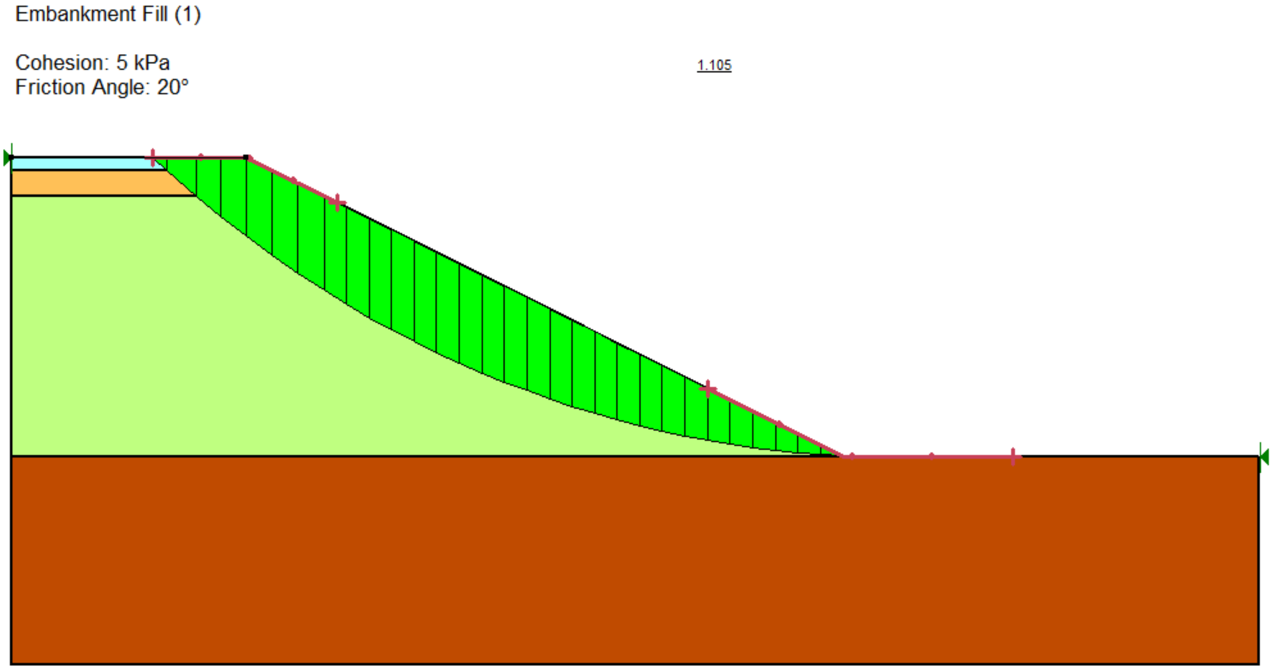


Figure 14.6. Results of Slope/W Model using Embankment Fill type 1 (φ = 20°) , model A

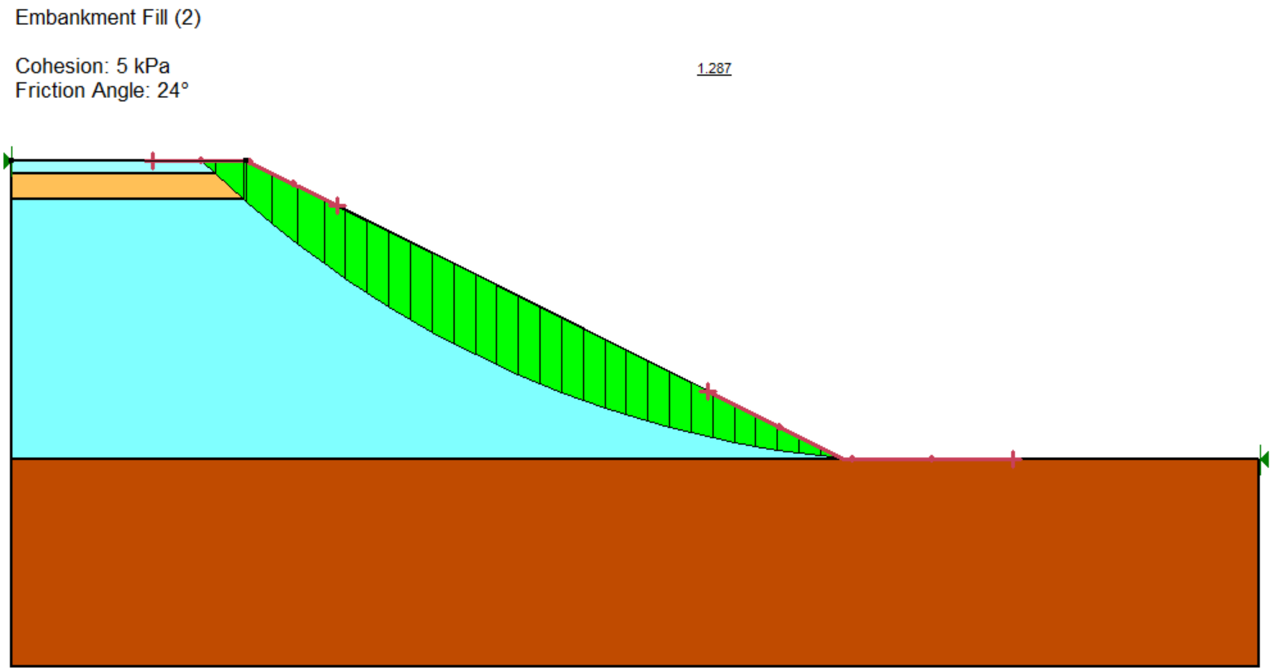


Figure 14.7. Results of Slope/W Model using Embankment Fill type 2 (φ = 24°) , model B

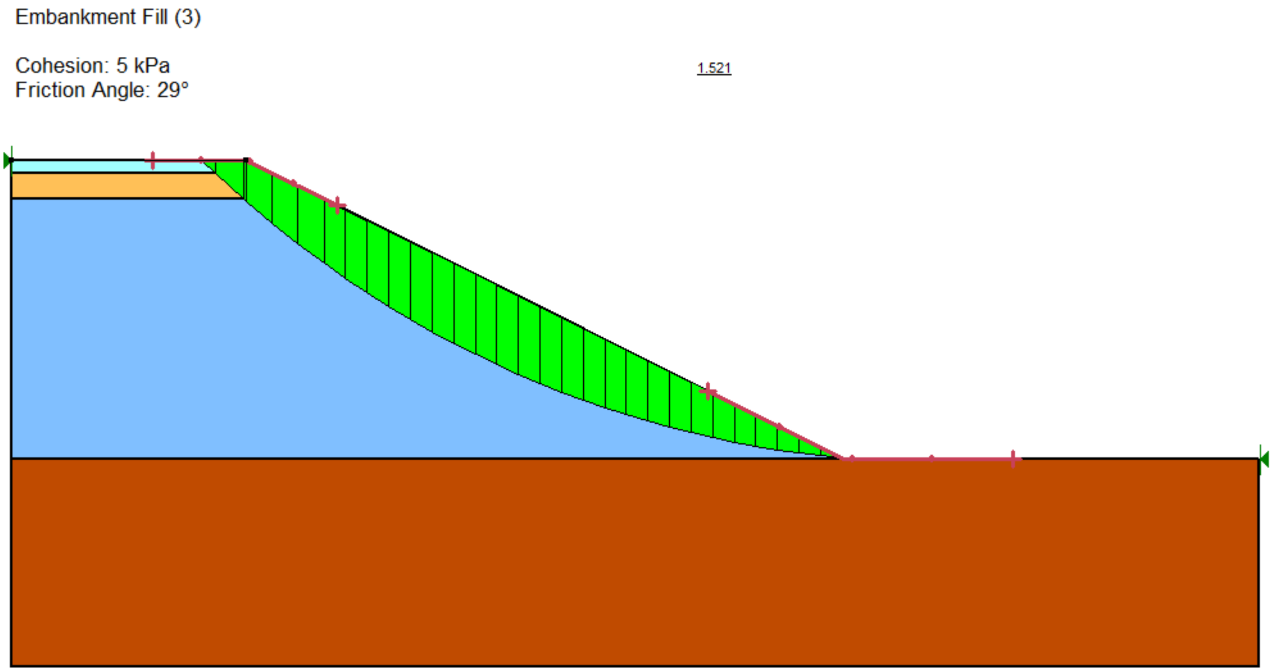


Figure 14.8. Results of Slope/W Model using Embankment Fill type 3 (φ = 29°), model C

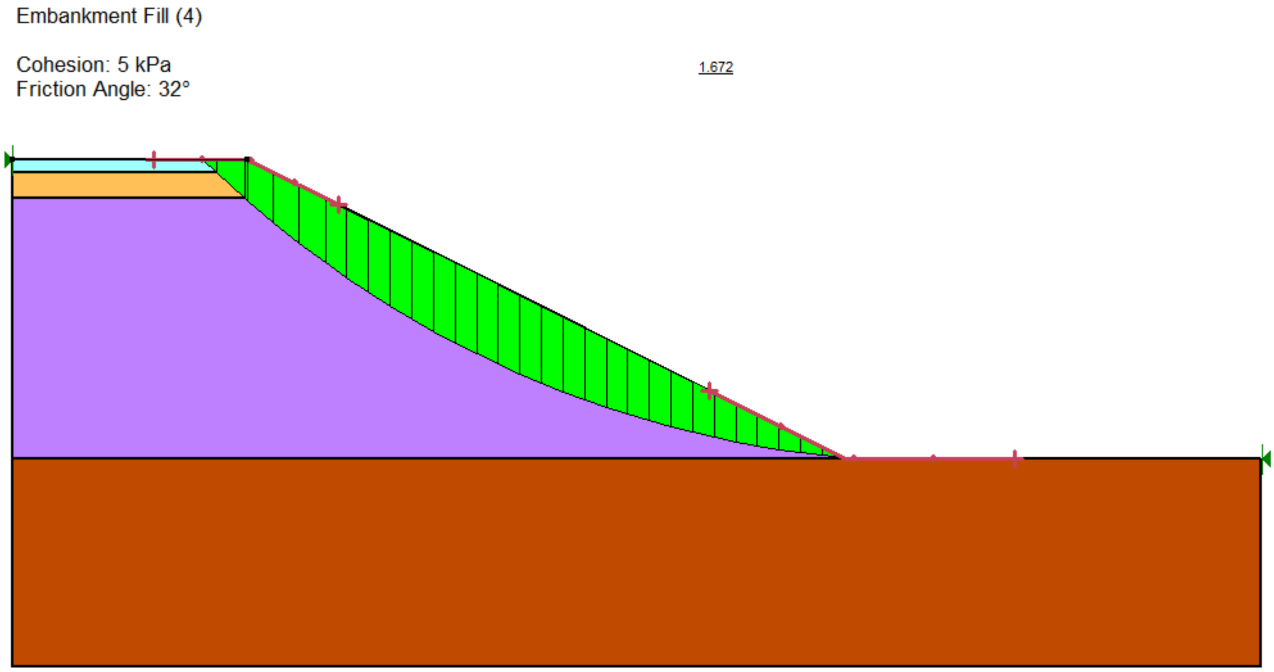
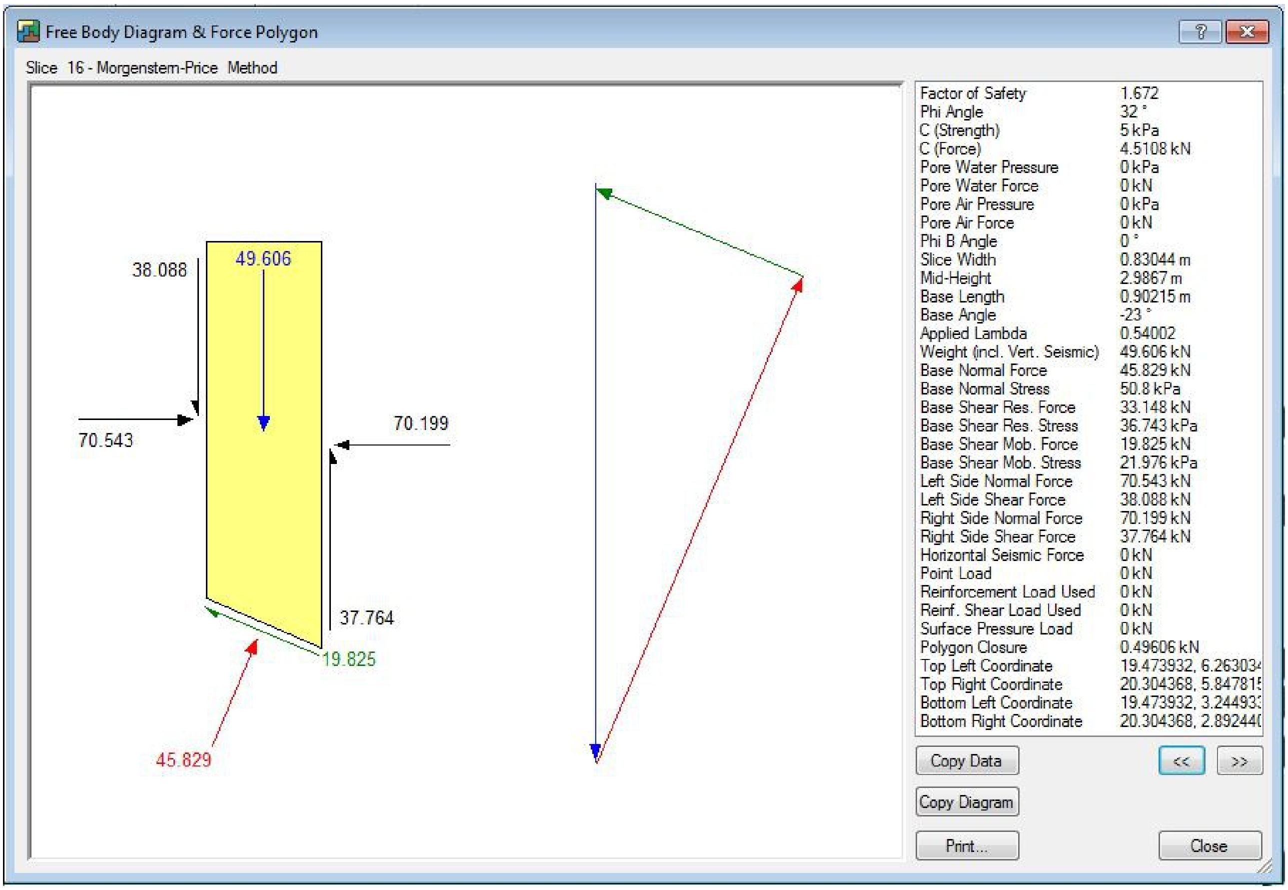


Figure 14.9 Results of Slope/W Model using Embankment Fill type 4 (φ = 32°) , model D

Finally, the results of the last model (model D), showed that a further increase in the friction angle of the fill up to φ = 32° promoted an increase in the stability of the slope. A factor of safety FOS = 1.672 was obtained for this model.



Factor of Safety	1.672
Phi Angle	32 °
C (Strength)	5 kPa
C (Force)	4.5108 kN
Pore Water Pressure	0 kPa
Pore Water Force	0 kN
Pore Air Pressure	0 kPa
Pore Air Force	0 kN
Phi B Angle	0 °
Slice Width	0.83044 m
Mid-Height	2.9867 m
Base Length	0.90215 m
Base Angle	-23 °
Applied Lambda	0.54002
Weight (incl. Vert. Seismic)	49.606 kN
Base Normal Force	45.829 kN
Base Normal Stress	50.8 kPa
Base Shear Res. Force	33.148 kN
Base Shear Res. Stress	36.743 kPa
Base Shear Mob. Force	19.825 kN
Base Shear Mob. Stress	21.976 kPa
Left Side Normal Force	70.543 kN
Left Side Shear Force	38.088 kN
Right Side Normal Force	70.199 kN
Right Side Shear Force	37.764 kN
Horizontal Seismic Force	0 kN
Point Load	0 kN
Reinforcement Load Used	0 kN
Reinf. Shear Load Used	0 kN
Surface Pressure Load	0 kN
Polygon Closure	0.49606 kN
Top Left Coordinate	19.473932, 6.26303
Top Right Coordinate	20.304368, 5.84781
Bottom Left Coordinate	19.473932, 3.24493
Bottom Right Coordinate	20.304368, 2.89244

Figure 14.10. Results of Slope/W Model using Embankment Fill type 4 (φ = 32°), model D

Slope/W allows for the visualisation of the results of the analysis in each particular slice, carried out throughout the Morgenstern Price method. Unlike the classical Spencer method, Morgenstern Price method does not consider the overall moment equilibrium of the slope, but only the moment equations of individual slices. As it can be observed in the previous figures, the area of slope instability used for the calculation is divided in slices. **Figure 14.10** shows the results of the equilibrium analysis for one of the slices in model D, where the forces acting on the slice can be seen.

14.5 COMPARISON BETWEEN PLAXIS 2D AND SLOPE/W

In this chapter, a study has been carried out on the slope stability of an embankment formed of regular fill on top of an existing ground formed of stiff clay. The models prepared in Slope/W (models A, B, C and D) have the same geometry and material properties of the set of models 1, prepared in Plaxis2D and explained in the previous chapter (Chapter 13).

Both geotechnical pieces of software used allows for the calculation of the factor of safety of the embankment against slope instabilities. Plaxis2D develops the calculation throughout the progressive reduction of the material properties and the application of finite element methods. The calculations on Slope/W are based on the Morgenstern Price method for limit equilibrium.

In models A, B, C and D, the material properties of the existing ground, the capping layer, the pavement and the surcharge applied on top of the embankment remained invariable. However, from model A to model D, the friction angle of the fill used as a core of the embankment was progressively increased from 20° to 32°.

Table 14.3 presents the results of the safety calculations for each models prepared in both Slope/W and Plaxis2D.

Table 14.3

Factors of Safety. Comparison between Plaxis 2D and SLOPE/W

Model	*Fill of embankment*	Friction angle, φ (°)	*Cohesion (kN/m²)*	*Young's modulus (kN/m²)*	**Factor of safety (ΣM_{sf}) Plaxis2D**	**Factor of Safety SLOPE/W**
Model A	Fill 2	20	5	15×10^3	**1.107**	**1.105**
Model B	Fill 6	24	5	15×10^3	**1.291**	**1.287**
Model C	Fill 8	29	5	15×10^3	**1.540**	**1.521**
Model C	Fill 10	32	5	15×10^3	**1.693**	**1.672**

As stated in the previous chapter for the case of Plaxis2D, the increase in the friction angle of the fill had an immediate effect on the overall performance and safety of the embankment, even without further modifications of the properties of the rest of the soils involved in the model.

As a main conclusion, the results obtained with both geotechnical programs were really comparable. The quantitative results varied less than 1.5% for all the models prepared.

14.6 CONCLUSIONS

The concluding remarks of the study are described below:

- The use of Slope/W (limit equilibrium analysis) was an effective and fast way to analyse the factor of safety against slope instabilities of a typical embankment (the geometry and material properties of the modelled embankment were extracted from realistic examples of linear infrastructures).

- The increase in the friction angle of the fill used as a core of embankment promoted an immediate increase in the stability of the embankment: the factor of safety, calculated in slices according to Morgenstern Price method, was increased from values of 1.105 for friction angle of 20° to 1.672 for friction angle of 32°.

- The increase of the friction angle in the models prepared in Slope/W was not accompanied by an improvement in the rest of material properties of the fill (fixed stiffness, Young's modulus $E = 15x10^3$ kPa).

- The results obtained with Slope/W were comparable with those obtained in Plaxis2D. Models with the same geometry and material properties were prepared for both geotechnical programs and the quantitative results obtained did not differ more than 1.5%.

CONCLUSIONS

Chapter 15

Conclusions

Abstract

The last part of this dissertation is aimed at providing the reader with a list of the most important conclusions which can be drawn from the experimental results and an outline of the further research which the author considers to be necessary within the line of investigation on soil stabilisation. At the beginning of this dissertation, the main objectives of the scientific investigation were presented. This chapter reviews the most remarkable and concluding results found along the different steps of the research, once the results have been presented.

15.1 CONCLUSIONS

In this dissertation, a section of conclusions has been included in all the chapters reporting the results of the experimental program (Chapters 5 to 14). In addition to it, the main general conclusions of this research are presented below:

- The chemical composition, particle size and pozzolanic activity are some of the most important features for the soil stabilisation agents. Biomass fly ash (BFA) was found to have a variable chemical composition which depends greatly on the type of biomass incinerated. The fly ash and the bottom ash obtained after the incineration of the biomass have very different properties, due to the different process of their formation. The presence of free lime in the biomass fly ash provides it with self-cementing properties which are crucial for the soil stabilisation process.

- CBW, RHA, DA, BFA, PSA and Paval were subjected to direct and indirect methods to study their pozzolanic activity. The results of the strength activity index showed that BFA, DA, RHA and CBW had pozzolanic properties. The results of Frattini tests showed the same for RHA, CBW and DA. The Frattini test could not provide results for very alkaline samples, such as BFA. The results of the NaOH solubility test could not be correlated with the results of the rest of methods. In addition to it, the chemical composition of DA and BFA showed a significant amount of calcium oxide in the form of free lime which can provide these additives with self-cementing properties.

- In the second stage of the research, a marly soil was treated with lime and the effects of the wetting-drying type of curing were studied. In general, the plasticity, swelling and bearing capacity of the soil were improved due to the addition of 2.5% and 4.0% of lime. However, the evolution of these values was proved to be non-linear along curing time. The values in the plot went up and down in a similar manner for different parameters. This tendency was also observed for the pH and the plasticity, although the final values obtained after 77 days in the treated soil showed that the initial properties of the marly soil had been significantly improved. The mineral composition of the soils also seemed to be modified, with important reductions in the intensity of reflection of the smectite peak reported in the XRD patterns.

- The use of magnesium hydroxide, seawater and olive mill wastewater showed that the high swelling potential and plasticity of a very expansive soils such as pure bentonite is very susceptible to change. The addition of 5, 10 and 15% of the mentioned additives, which were used partially due to their local availability, promoted dramatic reductions on the plasticity index and the swelling pressure of the original soil. As a main conclusion, the expansive soil proved to be influenced by a wide range of additives with different chemical properties and the addition of those stabilisation agents was really efficient in reducing the plasticity and swelling potential of the soil.

- The particle size distribution of a fine-grained soil can be studied with laser diffraction which provides similar results to the traditional hydrometer method. The plasticity of the natural marly and clayey soils were found to decrease when BFA and DA were added. The study of the mineral composition of natural and treated soils by means of microscopy also showed that ettringite and thaumasite can be formed in soils with very low amount of sulphates. The FESEM and EDS techniques proved that the formation of new minerals takes place in the treated soils from the earliest stages of curing.

- A relationship was found to exist between the shear strength and compressive strength of the marly soils treated with 5% and 10% of BFA and DA. The addition of BFA and DA promoted similar reductions of the plasticity index than those obtained with addition of cement (PI decreased from 55 to <25). The ph of the soil was found to increase from 8.5 to 10.5 with addition of these additives, although the increase of the pH was quicker in the case of DA treatments. The XRD results showed a variation in the mineral composition of the soils treated with BFA and DA after 28 days of curing. The presence of smectite in the samples seemed to be reduced. This could be due to the alkalinity provided by both alternative additives. This reduction would be very beneficial for the engineering properties of the soil.

- The numerical modelling techniques currently used for the design of earthworks were proved to be useful in the evaluation of the improvements which can be achieved by the use of treated soils for the construction of embankments for linear infrastructures. The numerical methods used to evaluate the factor of safety in an embankment formed of treated soils proved that the increase in the friction angle of the soil used as a fill promoted an increase in the factor of safety of the embankment.

15.2 FURTHER RESEARCH

The following lines of investigation could contribute to a better understanding of the soil stabilisation process, construction system and applications:

- The study of non-conventional additives as an alternative to cement, lime and coal fly ash (traditionally used) seem to be a never ending field of investigation. Nowadays, the casuistic of industrial by-products with pozzolanic and self-cementing properties is almost infinite. The construction industry needs a further and continuous study of all the resources available in each region of the globe to find more sustainable process and adapt them to the local conditions of each country.

- On the other hand, the durability of the construction works is becoming longer and longer whilst the laboratory studies carried out on stabilised soils are still focused on the primary stages of the process. Despite the fact that the initial conditions are very relevant for the overall performance of the treatment, there is a need for a study on the long term conditions of the treated embankments, with a focus on the impact of the seasonal variations of environmental conditions such as temperature and moisture.

- The treatments of soil stabilisation are very often studied with a focus on the physico-chemical properties of additives and soils and the mechanical improvements promoted in the soil. A further study on the applications of the stabilised soils, such as their use in the foundation of structures, is required.

- The studies carried out on soil stabilisation frequently uses natural clayey soils with a high swelling potential and plasticity. Under some circumstances, these soils have been reported to be responsible or triggering agents in landslides. The use of stabilised soil and the soil stabilisation treatment in the mitigation measures for landslides must be further studied.

- The soil stabilisation treatment can promote the enhancement of some mechanical and physical properties. The relationships among different mechanical and physical parameters (such as compressive strength, shear strength, compaction, bearing capacity, etc.) have been reported to exist in the past. The study of the optimisation could help define the most efficient dosages of additives in some treatments of soil stabilisation. The use of genetic algorithms is currently widespread for optimisation purposes.

- In this dissertation, the effects of soil properties of a treated fill on the slope stability and settlement of an embankment have been studied by means of numerical modelling. The further use of numerical modelling for the design of earthworks formed of treated soils is yet to provide interesting results.

Capítulo 15

Conclusiones

Resumen

La última parte de esta tesis doctoral tiene como objetivo ofrecer al lector una lista de las conclusiones mas importantes que se derivan del estudio, así como un esbozo de las líneas de investigación futuras que el autor considera necesarias e interesantes. Al comienzo de la tesis, se presentaron los principales objetivos de la investigación. Este último capítulo revisa la consecución de esos objetivos, una vez que los resultados experimentales han sido presentados.

15.1 CONCLUSIONES

En esta tesis doctoral, se ha incluido una sección de conclusiones al final de cada uno de los capítulos experimentales (Capítulos 5 a 14). Además, las principales conclusiones de la investigación se presentan a continuación:

- La composición química, el tamaño de partícula y la actividad puzolánica son algunos de los parámetros más importantes de los agentes de estabilización de suelos. En este estudio, se encontró que la ceniza de biomasa (BFA) tiene una composición química muy variable que depende en gran medida del tipo concreto de biomasa incinerada. La presencia de cal libre en la ceniza de biomasa le proporciona a este agente unas propiedades auto-cementantes que son de vital importancia para el proceso de estabilización.

- CBW, RHA, DA, BFA, PSA y Paval se sometieron al estudio de la actividad puzolánica por medio de métodos directos e indirectos. Los resultados del test de índice de actividad demostraron que la ceniza de biomasa (BFA), la cal dolomítica (DA), la ceniza de cáscara de arroz (RHA) y la chamota (CBW) tienen propiedades puzolánicas. Los resultados del ensayo de Frattini mostraron lo mismo para RHA, CBW y DA. El ensayo de Frattini no pudo proporcionar resultados fiabes para las muestras muy alcalinas, tales como la ceniza de biomasa. Los resultados del ensayo de solubilidad en hidróxido de sodio (NaOH) no pudieron correlacionarse con los resultados de los dos métodos anteriores. Además, a composición química de la cal dolomítica y la ceniza de biomasa mostraron una importante cantidad de óxido de calcio lo que puede promover las propiedades auto-cementantes de estos aditivos.

- En la segunda fase de esta investigación, un suelo margoso fue tratado con cal y se estudiaron los efectos de los ciclos de curado de humectación-desecación. En general, la plasticidad, el hinchamient y la capacidad portante del suelo mejoraron con la adición de un 2.5% y un 4.0% de cal. Sin embargo, la evolución de estos valores en el tiempo mostró una tendencia no lineal. Los valores representados en las gráficas de evolución subieron y bajaron siguiendo patrones parecidos en varios parámetros. La tendencia se observó para la plasticidad y el pH, aunque los valores finales obtenidos después de 77 días mostraron que las propiedades iniciales del suelo margoso mejoraron significativamente. Aparentemente, la composición mineral del suelo también se modificó, con importantes reducciones en la intensidad de reflexión del pico de esmectita, según se observó en los difractogramas (difracción de rayos X).

- El uso de hidróxido de magnesio, agua de mar y alperujo mostró que el alto potencial de hinchamiento y la elevada plasticidad de un suelo muy expansivo, como a bentonita muy pura, son muy susceptibles de sufrir cambios. La adición de 5, 10 y 15% de los aditivos mencionados, que fueron testados en parte debido a su disponibilidad local, promovió fuertes reducciones del índice de plasticidad y la presión de hinchamiento del suelo original. Como conclusión principal, el suelo expansivo se mostró muy influenciado por un amplio abanico de aditivos con diferentes propiedades químicas. La adición de estos agentes alternativos fue muy eficiente en la reducción de la plasticidad y el potencial de hinchamiento del suelo.

- La distribución del tamaño de partícula de un suelo muy fino puede estudiarse por medio de la difracción laser que proporciona resultados similares a los del método tradicional de granulometría por sedimentación. La plasticidad de los suelos naturales arcillosos y margosos disminuyó cuando se añadió al suelo ceniza de biomasa o cal dolomítica. El estudio de la composición mineralógica de los suelos utilizados en el estudio por medio de microscopía de alta resolución demostró que minerales expansivos y perjudiciales para el tratamiento de estabilización como la etringita y la taumasita se pueden formar incluso en suelos con muy bajo porcentage de sulfatos. Las técnicas de FESEM y espectroscopía EDS demostraron también que la formación de nuevos minerales ocurre en los suelos tratados desde las primeras etapas del curado.

- Se encontró una relación entre la resistencia al esfuerzo cortante y la resistencia uniaxial de compresión, en un suelo margoso tratado con 5% y 10% de ceniza de biomasa y cal dolomítica. La adición de ceniza de biomasa y cal dolomítica produjo una reducción de la plasticidad en las muestras similar a la producida con la adición de cemento (índice de plasicidad, PI, decreció de 55 a 25). El pH del suelo aumentó de 8.5 a 10.5 con la adición de estos aditivos, aunque el aumento del pH fue más rápido en el caso de tratamientos con cal dolomítica. Los resultados de la difracción de rayos X probaron una modificación de la composición mineralógica del suelo después de ser tratado con ceniza de biomasa y cal dolomítica y curado durante 28 días. La presencia de esmectita en las muestras pareció disminuir. Esto podría deberse a la alcalinidad producida por la ceniza de biomasa y la cal dolomítica. Esta modificación puede tener efectos beneficiosos en las propiedades ingenieriles del suelo.

- En esta investigación, las técnicas de cálculo numérico utilizadas hoy en día para el diseño de terraplenes y obras de tierra se mostraron muy útiles para la evaluación de las mejoras alcanzadas gracias al uso de suelos estabilizados en la construcción de terraplenes para obras lineales. Los métodos numéricos probaron que el factor de seguridad del terraplen formado por suelos tratados aumentó significativamente cuando se utilizó un suelo tratado y con un mayor ángulo de rozamiento interno.

15.2 FUTURAS LÍNEAS DE INVESTIGACIÓN

Las siguientes líneas de investigación puede contribuir a una mejor comprensión del proceso de estabilización de suelos y sus aplicaciones:

- El estudio de aditivos no convencionales como alternativa al cemento, la cal y la ceniza volante (tradicionalmente usados) parece tener un largo camino por recorrer. Actualmente, el abanico de sub-productos industriales con propiedades puzolánicas y/o auto-cementantes es muy amplio. La industria de la construcción necesita una estudio más profundo de los recursos disponibles en cada región del planeta para encontrar una proceso más sostenible y adaptar esos recursos a las condiciones locales de cada país.

- Por otro lado, la durabilidad de las obras construidas es cada vez mayor, mientras que los estudios de laboratorio llevados a cabo en suelos estabilizados, aún están centrados en la obtención de resultados en las primeras etapas de curado. A pesar de que las condiciones iniciales son relevantes para el resultado final del tratamiento, se necesita un estudio de las condiciones a largo plazo de los terraplenes construidos con suelo estabilizado, con especial atención al impacto de las variaciones estacionales de humedad y temperatura.

- Los tratamientos de estabilización de suelos son a menudo estudiados desde el punto de vista de las propiedades físico-químicas de los aditivos y los suelos y las mejoras mecánicas producidas. Se requiere un estudio más detallado de las posibles aplicaciones de los suelos estabilizados, como por ejemplo su uso para cimentaciones de estructuras.

- Con frecuencia, los estudios realizados en materia de estabilización de suelos usan suelos naturales arcillosos con un alto potencial de hinchamiento y una elevada plasticidad. Bajo algunas circunstancias, estos suelos se han definido como desencadenantes o condicionantes en procesos de deslizamientos de ladera. Debe estudiarse en profundidad el uso de los suelos estabilizados en el tratamiento de laderas susceptibles de inestabilidad.

- En esta tesis doctoral, se han estudiado los efectos del uso de suelos estabilizados en la estabilidad y deformación de terraplenes. El estudio de llevó a cabo por medio de modelos numéricos. Sin embargo, el campo de estudio es amplio y debe estudiarse en mayor profundidad el uso de modelos numéricos para evaluar las mejoras debidas a la estabilización e incorporarlas en el proceso de diseño.

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