



**UNIVERSIDAD
DE GRANADA**

**Analysis of the influence of design parameters
of asphalt mixtures on their susceptibility to
ageing phenomenon**

Doctoral Thesis

A Thesis Submitted in Partial Fulfilment of the Requirement
for the degree of

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My sister and brother who have always supported me morally. I love you enormously.

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Abstract

Bituminous mixtures are used as layers in a pavement structure to distribute stresses caused by loading, to protect the underlying unbound layers from the effects of water and to provide safe rolling surface for the users. To adequately perform these functions over the pavement design life, mixtures must be durable. One of the factors that affects the ability of bituminous mixtures to meet this requirement is ageing. This phenomenon has a significant impact on the performance and durability of asphalt mixtures, and it is vulnerable to a variety of factors that can be changed throughout both the design and manufacturing processes. The current thesis investigates the phenomenon of ageing in asphalt mixtures, taking into account both external and internal factors that influence ageing.

The first section of the study used laboratory procedures to analyse the impact of temperature, time, and pressure on the ageing caused by bituminous mixtures. The ageing of two mixtures, manufactured with a high void content and two different modified binders (SBS polymer and crumb rubber), was examined as a function of several conditions (different temperatures, time and pressure conditionings) and compared with cores extracted from road sections that have used similar mixtures in the surface layer. For this purpose, stiffness and fatigue cracking tests were used. Based on the results obtained in the first part, the second part of thesis examined the impact of the main design factors (type and quantity of bitumen, filler dosage, and the impact of mineral skeleton) on asphalt mixture ageing rates.

The results revealed that long ageing periods at a high temperature (at least 9 days and 135°C based on the results obtained in this research) were required to determine significant ageing influence while also allowing for the differentiation of ageing susceptibility of different materials. Moreover, the findings revealed that in spite of all factors had an impact on the range of changes in material stiffness, deformation susceptibility, and crack resistance due to the ageing process, the most relevant factors were the type of bitumen and filler content.

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1. Introduction

1.1 Thesis motivation

For the mobility of people and goods, road infrastructure that assures secure, comfortable, and efficient traffic is critical, where the surface layer plays a significant role, offering a variety of functional features that require adequate protection. In this sense, bituminous pavements are commonly used since the viscoelastic, cohesive, and adhesive capabilities of bitumen allow it to bind the aggregates together, giving the pavement appropriate strength and bearing capacity while providing good surface conditions to satisfy the expectation for safe and comfortable circulation. Nevertheless, the hydrocarbon composition of this binder, combined with climatic actions over the road surface layers (temperature changes, UV rays, rain, and so on), leads to the degradation of this material (Hunter et al., 2015; Sol-Sánchez et al., 2015), making it difficult to characterize and prevent in order to program a good asphalt pavement conservation program.

One of the most important aspects over time in degradation of asphalt mixtures is bitumen ageing (Tauste et al., 2018). On the one hand, previous research (Dickinson, 1980; Lesueur, 2009; Abu Qtaish et al., 2018) indicated that physical and chemical ageing are the two types of ageing processes that cause bitumen to harden and stiffen as a result of processes such as volatilization and oxidation. The ageing process begins during the procedures involved in constructing the bituminous layer (short-term ageing due to mixing, transportation, and placing) and continues during the pavement's service life (long-term ageing owing to oxygen and UV radiation) (Branthaver et al., 1993 ; Read et al., 2003 ; Porot et al., 2008 ; Hofko et al., 2017). Combining short- and long-term

ageing is a complex process in which a number of elements, including manufacturing and in-service conditions, as well as design considerations for the asphalt mixture, have been seen to influence the impacts and rate of material hardening. Previous researchers (Yin et al., 2015; López-Montero et al., 2016) proposed up to 15 different parameters that influence the three primary ageing mechanisms of oxidation, evaporation of the lightest components (volatiles such as saturates and aromatics), and physical hardening, among others. Furthermore, some studies have demonstrated (Harrigan, 2007; Khan et al., 2013; Aliha et al., 2020) that other factors such as the type of bitumen, or even its origin (for the same type of bitumen), and even the pressure or exposure time used during laboratory ageing simulations, have a significant impact on bitumen ageing. As a result, its cohesive and viscoelastic qualities affect the asphalt mixture's durability and mechanical performance. Previous research (Harrigan, 2007; Valdés et al., 2014) examined the impact of key factors like aggregate types and air void content, and discovered that mixtures with high air void content and aggregates with low capacity to absorb polar fractions (like quartzite and granite) are more prone to oxidation. Furthermore, because these types of mixtures (high air void content and resistant aggregates) are commonly used in surface layers in road infrastructure to provide a safe, comfortable, and durable pavement, they are more susceptible to the ageing phenomenon, which could reduce their service life, necessitating proper design in accordance with this issue.

On the other hand, factors related to production and in-service conditions have also been investigated for short and long-term effects, with findings indicating that mixing temperature, as well as thermal and solar radiation, had a significant impact on changing the susceptibility

of mixture ageing (Epps Martin et al., 2014; Mogawer et al., 2012) implying that the higher the in-service or mixing temperatures, the faster the ageing rate. Furthermore, recent research (Harrigan, 2007; Houston et al., 2005) has shown that elements associated to pavement construction or mixture design, such as final air void content achieved after layer compaction, are important contributors in material ageing, with reduced voids being preferred to decrease ageing.

However, several researchers (Morian et al., 2013; Rolt, 2000) found that other parameters such as asphalt content or aggregate gradation had contradicting effects on ageing rate. This feature, together with the paucity of studies examining the impact of designing mixture components on its long-term ageing, necessitate further research to properly understand the impact of these characteristics. It is also interesting to learn about the impact of possible variations in mixture composition that can occur during industrial manufacturing due to dairy dosage adjustments and possible minor changes in aggregate and bitumen properties due to high consumption, as well as the impact of these variations on the durability and ageing resistance of the road mixtures.

As a result, it is proven that there is still disparity in the understanding of the ageing mechanism, particularly at mixture level to evaluate the impact of the main factors influencing this process, from variables associated with material design and manufacturing. In this sense, the current Doctoral Thesis assesses, through a number of different laboratory procedures to simulate mixture ageing, the influence of the main variable that can be adjusted during the design and manufacturing

of asphalt mixtures, aiming to optimize the design of these materials from a durability point of view.

1.2 Thesis content

The current thesis is divided into eight chapters. This first chapter provides an introduction and thesis motivation for the study, which focuses on the impact of design parameters on the ageing susceptibility of asphalt mixtures. This first chapter also includes the content of each section of the paper.

Chapter 2 describes a state of the art carried out with the goal of reviewing the understanding about ageing of bituminous mixtures and their performance. This second chapter presents a description of ageing phenomenon of bituminous mixtures caused by different factors (external & internal) influencing this phenomenon. Moreover, this chapter also focuses on the laboratory tools for ageing simulation at bitumen level as an important parameter affecting ageing and then at bituminous mixture level in order to analyse the influence of ageing on bituminous mixtures. Finally, this section of the thesis gives conclusions of the state of the art.

Chapter 3 describes and lists the main general objective as well as the secondary objectives of this doctoral thesis.

The methodology used during this thesis is presented in Chapter 4. It describes each of the work phases that was performed in order to achieve the aforementioned objectives. This chapter contains the description of the material employed to manufacture the bituminous mixtures, the testing plan and the testing methods followed during the thesis. The testing plan includes a study of laboratory procedures for mixtures ageing which divides into two parts: assessment of impact of

ageing factors (temperature, time and pressure) and field experience to compare laboratory results; and an evaluation of the weight of designing parameters. The testing methods contain the stiffness and fatigue cracking tests in order to evaluate the impact of each design parameters on the ageing of all mixtures studied in this thesis.

Chapter 5 analyses the results obtained in each of the research phases, based on the schedule established in the methodology.

Chapter 6 presents the conclusions obtained from the analysis of results, responding to the objectives marked in this thesis. This is followed by Chapter 7 that states future research possibilities. The final section of the Thesis, Chapter 8, includes a list of the references cited.

2. State of the Art

2.1 Ageing phenomenon

Bituminous mixtures are composed of a combination of aggregates and hydrocarbon binders that, when mixed at high temperatures, form a continuous film that envelops the aggregates. Aggregates are elastoplastic material and viscoelastic bitumen, therefore bituminous mixtures are considered a visco-elastoplastic material. These mixtures are manufactured in fixed or mobile plants and are subsequently transported to the site for spreading and compaction. Bituminous paving mixtures are used as surface or base layers in a pavement structure to distribute stresses caused by loading and to protect the underlying unbound layers from the effects of water. However, degradation over time of any new or repaired pavement is one of the problems that affects the durability of the pavement. Multiple mechanisms in an asphalt binder/mixture can be defined as ageing. Changes in the chemical composition of asphalt binders/mixtures during construction and its service life cause rheological characteristics of asphalt binders/mixtures to change in pavement.

Asphalt binders degrade during the manufacturing of asphalt mixtures and in service when exposed to the environment. When asphalt mixture is manufactured at a very high temperature, the initial stage of ageing occurs at a very fast rate. Short-term ageing is a term used to describe this stage. A very thin film of asphalt is contacted to air at high temperatures during this step, resulting in a significant change in the rheological properties of the asphalt binders. High viscosity and stiffness are two symptoms of such changes (Roberts, et al, 1996). The second stage of ageing begins when asphalt is exposed to the

environment as in-service pavement for an extended period of time at a relatively lower temperature. The degree of hardening is determined by the amount of air voids in place and the surrounding environment.

Some researchers (Rostler and White, 1962) divide asphalt into two large chemical groups: asphaltenes and low molecular weight maltenes. Maltenes are viscous liquids formed of resins and oils that have a high viscosity (Asphalt Institute, 1980). Chemical and physical reactions between these fractions result in a complex mixing system of asphalt (Hveem, et al, 1959; Pan, et al, 2012). According to studies on the chemical components of asphalt as it aged, the amount of asphaltenes increases while the amount of resins and aromatics decreases. As the amount of asphaltenes increases, the asphalt becomes harder (i.e., stiffer), which can be seen as a decrease in penetration and an increase in softening point and viscosity (Heneash, 2013). Researchers also discovered that as asphaltenes/maltenes ratios vary with age, bitumen viscosity increases, making it harder and brittle (Lesueur, 2009).

Since asphalts are exposed to a variety of environmental conditions in the field during their service life, their physical and chemical qualities vary over time. The mechanisms that contribute to short- and long-term ageing have continued to be studied in depth (Welborn, 1979). Oxidation, volatilization, thixotropy (or steric hardness), polymerization due to actinic light, and condensation polymerization due to heat are some of the mechanisms that cause binder ageing (Bell, 1989; Traxler, 1961; Petersen, 1984; Bell, et al, 1994). The primary mechanisms involved with the ageing process of asphalt mixtures are oxidation, volatilization, and steric hardness (Petersen, 1984; Bell, et al, 1994; Apeageyi, 2011). The asphalt mixture is exposed to higher

temperatures during manufacture, laying, and compaction, which causes ageing owing to oxidation and the loss of volatile components. Long-term ageing during service periods, on the other hand, occurs at lower temperatures due to the oxidation mechanism (Anderson and Bonaquist, 2012).

2.1.1 Factors influencing ageing

Asphalt ageing is affected by a combination of factors. Plant type, mixing temperature, and silo duration during short-term ageing, as well as in-field circumstances (temperature, ultraviolet (UV) ray, and rainfall) and time during long-term ageing, are all external factors. Ageing speed and extent are further influenced by mixture characteristics such as asphalt source and type, aggregate gradation and absorption, void content/permeability, and the thickness of the asphalt binder film over the aggregate. According to a study conducted by (Morian et al. 2013), the effective binder content of asphalt mixtures has offered the strongest marker on the ageing properties of asphalt mixtures, regardless of the type of granular aggregate used. Degradation over time of any new or repaired pavement is one of the problems that affect the durability of the pavement.

In this context, factors can be qualified into two main families that influence the deterioration process. First, the internal factors caused by the pavement design and manufacturing, such as the materials used, its thicknesses, the dosages and the method of execution. In addition, other external environmental and traffic factors are taken into account, affecting the durability of a pavement, among others: temperature variations (seasonal), the gel-to-gel effect, oxidation, flow of oils.

Internal factors

Aggregates

Aggregates are sets of different particles of inert nature; they have contact with different materials in the environment including water and binders. Two main classes of aggregates often found: Natural aggregates (extracted from rocks), are used after several processes including: modification of its characters (their sizes and their distribution) so that they are well adapted to the manufacturing requirements. Artificial aggregates, resulting either from an industrial treatment of natural aggregates, or from the recycling of old pavements.

Some studies have shown the importance of analyzing some factors such as compaction of the mixture and its influence on the void percentage, the importance of taking into account the void percentage, because it determines the rate at which the ageing phenomenon is controlled while controlling the degree to which oxygen can access the bitumen. In other words, a high void content will cause a rate of ageing higher during the life of the material. Others have analyzed the porosity of the mixture, the permeability to air is related to the porosity of the mixture, the higher the porosity, the more the mixture is permeable to air, which automatically contributes to the ageing of the binder in most of the layer.

According to several studies, the ageing process can be accelerated or retarded due to the influence of aggregates following three mechanisms: First, oxidation of the bitumen can be catalyzed from the minerals found on the surface of aggregates. Second, the creation of a viscous layer around the functional groups of binders, absorbed by the surface of the aggregates, causes the retard in increasing the viscosity of the bitumen. Finally, creation of an internal imbalance following the

absorption of the bitumen fractions, which leads to the acceleration of the ageing.

Bitumen

Bitumen consists of hydrocarbon (solid, binder, liquid). Given its property of thermoplastics, the consistency of bitumen changes with temperature. Bitumen, which is a homogeneous mixture of many molecules, can be divided into two classes: polar and non-polar (Mouillet, et al. 2008). The non-polar molecules serve as a matrix or solvent for the polar molecules, which form weak “networks” of polar-polar associations that give elastic properties to bitumen. The polar materials are uniformly distributed throughout the bitumen, and upon heating the weak interactions are broken to yield a Newtonian fluid. When perturbed, in response to temperature changes and physical stresses, these interactions break and reform up to produce a new combination of interactions (Jones and Kennedy, 1992). The polar molecules interact and primarily give bitumen its elastic characteristics. The non-polar molecules mainly contribute to the viscous behavior of the bitumen and control the low temperature properties of the bitumen (Youtcheff and Jones, 1994).

Bitumen ageing is a subject that has been addressed from different perspectives. One of them, consisted in proving that the bitumen is a mixed of several components, the nature of which can vary from one binder to another, making it difficult to isolate and identify shared characteristics, all of this undermines the role of bitumen chemical components (which in turn depend on the nature of its crude oil) in the ageing process. In other words, bitumen faces an ageing process that starts during the operations involved in the construction of the

bituminous layer [short-term ageing: this includes all the changes that occur in the bitumen during mixing, at a temperature between about 150 and 190°C), transport and laying, and cooling (from 130°C to ambient temperature)], and continues during the service lifetime of the pavement (long-term ageing: this comprises all the changes that occur on site in the bitumen over the years of service of the pavement layer in which it has been used).

According to (Masson, Collins, & Polomark, 2005; Traxler, 1961), Bitumen's behavior changes over time as a result of a mix of chemical and physical processes triggered by temperature. In pavement engineering, bitumen and bituminous bound pavements age in two ways: short-term ageing (STA) occurs during the production and compaction of hot mix asphalt (HMA) in a few hours, and long-term ageing (LTA) occurs over the period of a pavement's in-service life. The general ageing effects of oxidation, volatilisation, steric hardness, and polymerisation are incorporated in both ageing phases. According to (Baek, Underwood, & Kim, 2012; Hofko et al., 2017; Petersen, 1994; Steiner, Maschauer, & Hofko, 2018), STA is initiated by quick oxidation caused by high temperatures and a high specific surface area, as well as a physical action in which residual volatile components in the bitumen may evolve (non-oxidative, thermal effects). According to (Menapace & Masad, 2018; Morian, Hajj, Glover, & Sebaaly, 2011), UV radiation and slow oxidation, particularly of the upper pavement layers, by oxidant gases in the atmosphere drive LTA.

Some researchers have found that in-service ageing may be influenced by a great number of factors, one of them is, the binder's intrinsic susceptibility to ageing, which laboratories are trying to assess from the

commonly practiced accelerated tests (at temperatures above 100°C). Since this assessment is essentially relative, the greatest caution should be exercised in extrapolating to ambient temperatures. Additionally, the use of a kinetic approach to binder ageing is evident in estimating reaction constants of bituminous concrete at service temperature. Other researchers have observed that the mixing temperature and the exposure conditions such as the diffusion rate and the length or thickness of the diffusion path cause the evaporation of the volatile components (saturated and aromatic). This mechanism plays a role in the ageing process, but its influence is limited face to other factors. Macroscopic changes during bitumen ageing include a decrease in penetration and ductility and an increase in the softening point. This is accompanied by a reduction in other properties of bituminous pavements, such as anti-rutting, water damage resistance, and crack resistance.

Several studies (Li et al., 2016; Wu et al., 2017) have investigated the ageing process and mechanism of bitumen for many years. Using the film furnace test, PAV, and ultraviolet (UV) radiation, Zhang et al. (2017) investigated the effects of various ageing procedures on the physical characteristics and chemical composition of SBS-modified bitumen. Wang et al. evaluated at the ageing of polymer-bitumen composite systems in terms of chemical functional groups, molecular size, and rheological properties. Lana et al. used gel permeation chromatography (GPC) and an atomic force microscope to investigate the impact of ageing on the micromechanical and chemical properties of foam warm mix bitumen (AFM). Other study stated that the main cause of bitumen ageing is oxygen absorption. Bitumen's viscoelastic components changed as it aged, and it became harder. Bitumen's

macromolecule could be broken down into smaller molecules, resulting in a significant molecular weight. Even though bitumen ageing has a significant impact on pavement durability and recyclability, it is important to evaluate the ageing behavior and resistance to ageing of binders and mixes during the mix design optimization process in order to achieve cost- and energy-efficient pavements with low maintenance requirements, a long service life, and high recycling potential.

External factors

Oxidation

The oxidation is the principal mechanism responsible for the long-term ageing of asphalt pavements. This oxidation is an irreversible chemical reaction between asphalt species and oxygen molecules. It leads to the formation of carbonyl chemical functional group and asphaltene components and further increases the polarity and stiffness of asphalt binder (Gao, et al. 2019). The impact of binder oxidation on pavement performance is complicated. The complex organic components of asphalt react with air oxygen and ultraviolet (UV) radiation, hardening the pavement surface and causing fractures. The contribution of ultraviolet to ageing can be neglected even the absorption of binders is high in the ultraviolet. Because the influence of these rays is limited on a small layer of the surface of the bitumen (one or two micrometers) so it is negligible with respect to the total amount of binder in the layer. Moreover, the influence of infrared (IR) radiation must not be neglected since its absorption leads to a rather considerable increase in mean temperature in the layer.

Some previous studies (Branthaver, et al. 1993; Davis and Petersen, 1967; Petersen, 1998) have demonstrated that the production of oxygen-containing polar chemical properties on asphalt molecules is

thought to promote oxidative ageing, which can lead to agglomeration between molecules due to higher chemophysical interactions such as hydrogen bonding. The Mechanistic Empirical Pavement Design Guide (AASHTO, 2004) considers that binders oxidize only in the top inch. Others studies (Wright, 1965; Corbett and Merz, 1975) have described the chemical composition of asphalt changes as a result of oxidation. Due to their low chemical reactivity, saturates remain remarkably stable, but the other three fractions show significant variation. As a result, physicochemical properties (such as carbonyl and sulfoxide groups) occur in asphalt molecules, resulting in lower aromatic fractions and higher asphaltene fractions. Moreover, binder oxidation in pavements can have a considerable serious effect on pavement fatigue life, according to previous researches (Walubita et al., 2005; Walubita, 2006).

Steric hardening

When asphalt cements are exposed to low temperatures physical hardening (steric hardening) happens over time. According to previous research (Masson, 2005), the molecular structure of asphalt is changed during this process, influencing the asphaltene fractions. Increased viscosity, significant volume contraction, and, consequently, asphalt hardening are all effects of steric hardening (Traxler, 1961; Pechenyi and Kuznetsov, 1990). Some studies (Swiertz, 2010; Petersen, 1984) have demonstrated that this hardness is caused by the molecule's structural reconfiguration at low temperatures, it can be reversed with temperature or mechanical effort.

Volatilization

A mechanism that happens during hot mixing and construction of asphalt cement is volatilization. Some researchers (Lesueur, 2009; Traxler, 1961) confirmed that smaller molecular weight particles can evaporate and migrate into the environment at high temperatures. A study by Farcas, 1996; has approved that the aromatic fractions evaporate quickly and asphaltene fractions increase when thin film asphalt due to the contact between aggregates a thin asphalt film at temperature of 150° C and over. Other researchers (Christensen and Anderson, 1992; Bell, 1989) discovered that viscosity rises by 150 to 400%. According to (Shalaby, 2002; Fernandez-Gomez, et al., 2013), asphalt flow characteristics are reduced as a result of weight loss, i.e., viscosity is influenced by volatilization, particularly considering the speed with which volatilization occurs. Volatilization caused a considerable rise in modulus and a decrease in phase angle (Cui, 2014).

2.1.2 Impact of ageing on mixture performance

Ageing produces a number of changes in asphalt mix characteristics, which are reflected in asphalt pavement performance. As a result of ageing, the ductility and penetration of asphalt binder are reduced, while the softening point and ignition temperature increase, according to experimental research (Siddiqui and Ali, 1999). The viscosity of the asphalt is gradually increased, resulting in a stiffer asphalt mixture (Sirin, et al, 2017).

According to Yoder and Witczak (1975), deterioration is usually attached with the constraints of the pavement which is manifested by several failures: Fatigue cracking, Potholes and peelings, Permanent

deformations, Gravel sinking, Detachment of gravels or polishing of aggregates. It is important to make a distinction between two kinds of failure (Yoder and Witczak, 1975) that can occur on a road surface. The first type, called "structural failure", includes a collapse of the structure of the pavement or one or more of its components, making them unable to withstand the loads imposed on its surface by the traffic. The second, called "functional failure", may or may not be accompanied by structural failure, but it causes great efforts on vehicles travelling on the pavement, and great discomfort for its passengers due to its great roughness. Based on their origin, a road's fatigue and damage mechanisms can be divided into five categories according to (Saarenketo, et al., 2012): 1) Under repetitive loading, the pavement and unbound structural layers get fatigued. This generally necessitates millions of heavy axle load repetitions on a new road. 2) Permanent deformations in the road's structural layers. Even after a few large truck passes, these permanent deformations may occur. The majority of these irreversible deformation damages occur during the frost thaw phase in the spring. 3) Damages caused by frost and a lack of drainage. These issues are frequently the source of the prior category's issues. In winter, when the road structure is frozen, frost fractures can emerge. 4) Geotechnical issues: Settlement is the most common example of geotechnical issues. 5) Errors in design and construction, such as culvert bump issues, transition wedge damage, and reflection cracks on enlarged highways.

Stiffness

The stiffness of the asphalt mixtures applied in the wearing course is a significant mechanical property. It is one of the influencing factors in

tyre/road noise generation. The physical and mechanical characteristics of the components that comprise the upper layer of the road surface, as well as traffic load, determine its dynamic stiffness.

The first case was made through the UNE-EN 12697-26. This determined the effect of ageing on the elastic response of the material, and its ability to support loads without deforming. The stiffness of a mixture is an important mechanical feature used in the wearing course of asphalt pavements. This test was carried out at 20°C on cylindrical specimens (height 35 mm, and diameter close to 100 mm) by applying 10 charge pulses to adjust the amplitude and duration of the load, and subsequently another 5 pulses to determine the rigidity of the material. This same procedure was repeated on another diameter of the test piece located at $90 \pm 10^\circ$ in relation to the previous case, thus being able to obtain a mean value of the rigidity module from the measurements in both diameters.

Fatigue cracking

Fatigue cracking begins as a series of microcracks in the wheel tracks then develops into a network of interconnected cracks, generally results in potholes (Anonymous, 2010). Crack initiation and crack propagation are two phases of fatigue cracking in flexible pavements, which are induced by tensile strains generated in the pavement due to traffic loading and temperature changes (Kim & Park, 2015). Different experiments can be used to determine the initiation of cracks. Despite multiple efforts to test and measure fracture initiation and propagation, due to their stochastic character, these efforts continue to fail. According to the same study (Kim & Park, 2015), fatigue cracking is divided into two categories: alligator or bottom-up fatigue cracking and

longitudinal or top-down fatigue cracking, which can appear to be the same on the pavement surface. Alligator exhaustion due to mechanical failure induced by the highest tensile stress, cracking begins at the bottom of the asphalt layer and develops gradually upwards to the pavement's surface (Moreno-Navarro & Rubio-Gómez 2016). Longitudinal cracking, which is similar to alligator cracking in concept, begins at the surface where there is high localized tensile stress and strain due to tire-pavement interaction and progresses down to the asphalt layer's bottom. It should be noted that according to (Guercio, et al. 2015; Ambassa, et al. 2012), bottom-up fatigue cracking is particularly common in thin pavement layers, which makes it an issue that is accentuated by cold temperatures and top-bottom fatigue cracking is most probable to appear in thick pavement layers.

Potholes and peelings

Peeling and delamination of road pavement, commonly known as potholes, are of critical interest. Infrastructure failures that peel off will generate serious problems, such as tunnel concrete colliding with vehicles and car accidents caused by potholes. Peeling occurs as a result of delamination.

According to Tedeschi and Benedetto (2017), Potholes and cracks are the two most common types of road surface degradation, and they have a major impact on vehicle performance. Others researches (Zhao et al., 2021) have stated that potholes are paved mostly for maintenance or repair, and the asphalt mixture must be compacted. However, due to a problem with construction quality, the compacting degree of the asphalt mixture may not be sufficient, and the void ratio of the asphalt mixture may not satisfy the standards, causing potholes to deteriorate

prematurely after repair. Another study was done by Pan et al., 2018 has detected the pavement potholes and cracks based on the Unmanned Aerial Vehicle Multispectral Imagery method. This method were used to distinguish between the normal pavement and pavement damages (e.g., cracks and potholes) using machine learning algorithms which demonstrates that a UAV remote sensing system presents a potential tool for monitoring asphalt road pavement condition, which can be utilized to aid in road maintenance decisions.

2.2 Tools for ageing simulation at laboratory level

2.2.1 Simulation at bitumen level

Over the last three decades, researchers have made numerous tries to link accelerated bitumen ageing in the lab with field performance. The majority of this study has used thin film ovens to accelerate the ageing of bitumen, with the majority of thin film oven ageing methods relying on extended heating (oven volatilisation) procedures. To imitate short-term bitumen ageing (hardening) associated with asphalt mixture preparation activities, extended heating processes are commonly used. The thin film oven test (TFOT), the rolling thin film oven test (RTFOT), and the rotating flask test are the most frequently used standardised tests to control the short-term ageing of conventional, modified bitumen (RFT). Thin film oven tests can effectively evaluate the relative hardening characteristics of bitumens during the mixing process, however they can't predict long-term field ageing. Coupling thin film oven ageing with oxidative ageing has been attempted to overcome this problem.

According to previous studies, one of the most important elements impacting the service life of bituminous pavement structures is bitumen ageing. Some experts suggest that laboratory ageing circumstances are

insufficient for simulating the exact ageing mechanism of in-service pavements, and they have proposed an ageing test of bitumen under a combination of conditions. Asphalt degradation can be classified into two categories: Thermal ageing and UV radiation ageing. The methods for simulating thermal ageing in the laboratory are established. To complete the thermal ageing process, researchers may follow standards such as the rolling thin film oven test (RTFOT), thin film oven test (TFOT), and Pressure Ageing Vessel (PAV) (Lu & Isacsson, 2002; Lu & Isacsson, 1998; Abbas et al., 2002). With growing global warming and related increases in Ultra Violet radiation emissions, important to pay more attention to the influence of UV radiations on asphalt ageing (Durrieu, 2007). Naskar exposed the asphalt film in the UV chamber for 30 minutes at ambient temperature (Naskar et al., 2013). Virginie Mouillet et al. employed binder film in a UV chamber with a working temperature of 60 degrees Celsius (Mouillet, 2008). Henglong Zhang et al. used a draft oven with a 500 W UV light and an 80 °C working temperature to imitate the UV ageing process (Zhang, 2011). Asphalt was stored in Ultra Violet radiation chambers for 20 days at 50 degrees Celsius by Pan et al. (2014). Both asphalt binders and asphalt mixtures were subjected to UV ageing in a UV ageing box at 80 °C by Feipeng Xiao et al. (2014).

To simulate short-term bitumen ageing (hardening) associated with asphalt mixture preparation activities, extended heating processes are commonly used. The TFOT, RTFOT, and rotating flask tests are the most widely used standardised tests for controlling the short-term ageing of conventional, unmodified bitumen (RTFT). Lewis and Welborn (1940) were the first to use the TFOT to simulate short-term ageing by applying a temperature of 163°C to asphalt with a film

thickness of 3.2 mm for 5 hours. The TFOT at 163 °C for 5 h was used to simulate the STA of rubberized bitumen during bitumen mixture mixing, transportation, and paving progress. Many researchers tried to enhance or establish testing methods for ageing asphalt with more representative film thickness. Edler et al. (1985) used the Modified thin film oven test with a film thickness of 100 m and an additional exposure time of 24 hours. The Rolling Thin Film Oven Test (RTFOT), which simulates short-term ageing, is most frequent, developed by the California Division of Highway (Hveem et al., 1973). The method assesses the combined effects of heat and air on a thin film of bitumen or bituminous binder that is continuously renewed. It simulates the hardening of a binder in an asphalt factory during mixing. The effects of heat and air are determined by measuring the change in mass of the sample (reported as a percentage) or the evolution of binder properties such as penetrability (EN 1426), ball softening point-ring (EN 1427), or dynamic viscosity (EN 12596) before and after passing through the oven. According to (Whiteoak, 1990), heat and oxidation on thin films of 1.25 mm are used to age eight glass bottles each containing 35 gram of asphalt. This method permits homogeneous asphalt ageing with no skin formation and closely replicates asphalt hardening observed in the hot-mixing process.

The High-pressure ageing test (Hayton et al., 1999), Accelerated ageing test device (Verhasselt & Choquet, 1991), Pressure oxidation bomb (Edler et al., 1985), Iowa durability test (Lee, 1973), and PAV (Petersen et al., 1994; Christensen & Anderson, 1992) have all aimed to estimate long-term ageing by combining thin film oven testing with oxidative ageing. The Pressure Ageing Vessel (PAV) test, which compensates for long-term ageing and is usually produced on the

RTFOT residue, is one of the most frequent. To simulate field ageing effects, RTFOT aged asphalt is heated to 100°C for 20 hours at 2.07 MPa to simulate field ageing effects, RTFOT aged asphalt is heated to 100°C for 20 hours at 2.07 MPa pressure, according to USA standards (Fernandez et al., 2013) it simulates ageing of 8 to 10 years of pavements service life. A method for simulating long-term oxidative ageing of bitumen in the field using the PAV created by the SHRP-A-002A research team (Christensen and Anderson, 1992; Petersen et al., 1994). The standard components hardening bitumen in an RTFOT or TFOT, then oxidation the residue in a pressurized ageing vessel. The PAV process involves ageing 50 g of bitumen in a 140 mm diameter pan (3.2 mm binder layer) within a heated container for 20 hours at temperatures ranging from 90 to 110°C (AASHTO PP1, 1993).

Thin Film Oven Test

Lewis and Welborn (1940) were the first to use the TFOT to distinguish between bitumens with varying volatility and hardening properties. A 50 mL sample of bitumen is deposited in a flat 140 mm diameter container in the TFOT, resulting in a 3.2 mm film thickness. After that, two or more of these containers are placed in the oven on a rotating shelf (5 to 6 rpm) for 5 hours at 163°C. As a method of evaluating bitumen hardening during plant mixing, the TFOT was adopted by AASHTO in 1959 and ASTM in 1969 (ASTM D1754). Nonetheless, the thick binder layer, which results in a huge volume to exposed surface area for the aged binder, is a key complaint of the TFOT. Because the bitumen is not disturbed or rotated during the test, there is a risk that ageing (mainly volatile loss) will be limited to the sample's skin.

Rolling Thin Film Oven Test

The RTFOT (figure 1), developed by the California Division of Highways (Hveem, et al., 1963), was the most significant modification of the TFOT, in which eight glass bottles each carrying 35 gram of asphalt were aged by applying heat and oxidation to thin films of 1.25 mm. This equipment (AASHTO T240, 2013) consists of: 1. Oven— Inside dimensions are as follows: 381 mm (15 in.), 483 mm (19 in.), and 445 ± 13 mm ($17 \frac{1}{2} \pm \frac{1}{2}$ in.) for a double-walled electrically heated convection type. A symmetrically situated window with dimensions of 305 to 330 mm (12 to 13 in.) width by 203 to 229 mm (8 to 9 in.) high shall be included in the door. Two panes of heat-resistant glass must be separated by an air space in the window. The window should provide a clear view of the oven's inside. The heating element must be beneath the oven floor and sufficient to maintain the desired temperature. The top and bottom of the oven must be ventilated. 2. Flowmeter— The flowmeter can be of any type that can measure airflow at a rate of 4000 mL/min or less. The flowmeter should be placed upstream of the copper coil and downstream of all regulating devices. The flowmeter should be placed in a way that is kept at around room temperature. A wet-test meter or equivalent method must be used to regulate the flowmeter at least once every 12 months. This calibration will be done with the oven turned off and at room temperature, and will be based on airflow exiting the air jet. 3. Thermometer—An ASTM 13C (13F) thermometer with a precision of 0.2°C (0.5°F) as specified in ASTM E 1. The thermometer must be calibrated in accordance with the R 18 criteria. All temperature measurements required by this approach must be taken with this

thermometer. The thermometer may be entirely or partially enclosed in an optically clear polymer sheath with a maximum thickness of 0.25 mm to limit the danger of thermometer breaking (0.01 in.). If a sheath is utilized, it must be fitted in such a way that the thermometer is in substantial mechanical contact with it. After the sheath has been installed, the thermometer must be standardized.

4. Container— Heat-resistant glass with a smooth interior is required for the container in which the asphalt binder will be tested.

5. Balance— A Class B balance adhering to the standards of M 231 is required if the loss on heating is desired. A Class G 2 balance according to M 231 can be utilized if only the residue is required. The balance must be harmonized in accordance with the standards of R 18.

6. Cooling Rack—A stainless steel or aluminium wire or sheet metal rack that permits sample containers to cool in a horizontal position. The rack must be built in such a way that air may freely flow around each container, with a minimum of 25 mm (1 in.) separation between containers and a minimum of 25 mm (1 in.) separation between containers and any solid surface. This approach assures homogeneous asphalt ageing with no skin formation and links asphalt hardening to that seen in the hot-mixing process (Whiteoak, 1990).

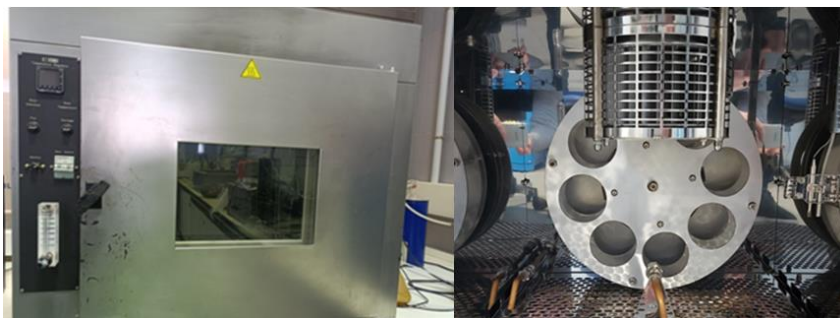


Figure 1 Rolling Thin Film Oven Test

Shell Microfilm Test

Another variation of the principle utilized with the TFOT is the Shell microfilm test. In this test, a very thin bitumen layer of 5 microns is aged on a glass plate at 107°C for 2 hours (Griffin et al., 1955). To simulate the film thicknesses found in asphalt mixtures, a thinner film thickness was chosen. The viscosity of the bitumen is measured before and after testing to provide a 'ageing index'. Besides the study done on the ZacaWigmore test roads, there has been little reported association between field performance and laboratory ageing using the Shell microfilm test (Wellborn, 1979; Zube and Skog, 1969). Simpson et al. (1959) compared the viscosity data for bitumen collected from the two test roads to the Shell microfilm test and discovered a clear relationship between field and laboratory results. Hveem et al. (1963) and Skog (1967) considerably modified the Shell microfilm test by raising the film thickness to 20 microns and the exposure time to 24 hours with a small reduction in temperature to 99°C. These changes did show that field and laboratory hardening have an indirect link. Traxler (1963) and Halstead and Zenewitz (1961) made minor changes to the binder film thickness, increasing it from 5 to 15 microns.

Rotating Flask Test

The RFT method involves ageing a 100 g bitumen sample for 150 minutes at 165°C in the flask of a rotary evaporator. The substance comprising the specimen's surface is constantly replaced as the flask rotates at 20 rpm, preventing the creation of a skin on the bitumen's surface.

Tilt-Oven Durability Test

The California tilt-oven durability test (TODT), where the oven is inclined 1.06 higher in the front to avoid bitumen migrating from the bottles, is another variation to the RTFOT (Kemp and Predoehl, 1981). Furthermore, when compared to the RTFOT, the TODT uses a lower temperature and a longer duration for ageing, namely 168 hours at 113°C. Kemp and Predoehl (1981) aged laboratory specimens in four different climates in the field and concluded that the TODT may be used to predict bitumen hardening in hot climates. Moreover, according to (Petersen, 1989), this rate of ageing is similar to that observed in hot desert regions after two years for pavement mixtures.

Modified Rolling Thin Film Oven Test

One of the most major disadvantages of employing the RTFOT for modified bitumens is that, due to their high viscosity, these binders will not roll inside the glass bottles during the test. Furthermore, some binders have a tendency for rolling out of the bottles. Bahia et al. (1998) devised the Modified Rolling Thin Film Oven Test to address these issues (RTFOTM). The test is identical to the normal RTFOT except that during oven ageing, a set of 127 mm length by 6.4 mm diameter steel rods are placed into the glass bottles. The idea is that the steel rods provide shearing forces that spread the binder into thin films, solving the problem of high viscosity binders ageing. Furthermore, recent study at the Turner-Fairbanks Research Center suggests that utilizing metal rods in the RTFOTM does not reduce the problem of modified binder roll-out, and that more validation work is needed before the technology can be approved.

Pressure Oxidation Bomb

Edler et al. (1985) employed a method in which residue from an eight-hour ERTFOT was followed by pressure oxidation utilizing a pressure oxidation bomb (POB). A cylindrical pressure vessel with a screw-on cover holding a safety blow-off cap, pressure gauge, and stopcock makes up the POB. Twelve 40 mm by 40 mm glass plates coated with 30 micron bitumen films are positioned horizontally in the boat's metal support. The bitumen residue is aged for 96 hours at a pressure of 2.07 MPa and a temperature of 65°C.

Pressure Ageing Vessel

The SHRP-A-002A investigators invented a system for simulating long-term, in-service oxidative ageing of bitumen in the field using a pressure ageing vessel (PAV) (Christensen and Anderson, 1992) (figure 2). The standard components hardening bitumen in an RTFOT or TFOT, then oxidizing the residue in a pressurized ageing vessel. The PAV technique involves ageing 50 g of bitumen in a 140 mm diameter pan (3.2 mm binder film) within a heated vessel for 20 hours at temperatures between 90 and 110°C, pressurized with air to 2.07 MPa (AASHTO PP1). Verhasselt and Vanelstraete (2000) determined that 20 hours of PAV ageing is nearly similar to 178 hours of RCAT ageing. Migliori and Corte (1999) studied if PAV testing for unmodified penetration grade bitumens might be used to simulate RTFOT (short-term ageing) and RTFOT + PAV (long-term ageing). They discovered that 5 hours of PAV ageing at 100 degrees Celsius and 2.07 MPa was equivalent to standard RTFOT ageing, and 25 hours of

PAV ageing at 100 degrees Celsius and 2.07 MPa was equivalent to conventional RTFOT + PAV ageing.



Figure 2 Pressure Ageing Vessel

High Pressure Ageing Test

The High-Pressure Ageing Test (HiPAT) is a variation of the PAV process that uses a lower temperature of 85 degrees Celsius and a 65-hour duration (Hayton et al., 1999). These changes were made in response to claims that the PAV procedure's temperatures were excessively high in comparison to projected pavement temperatures. Furthermore, it was considered that the technique, particularly for modified binders, had the potential to profoundly alter the binders in a way that was not typical of what was seen in the field. For a solid asphalt combination with a 10-year service life, preliminary investigations suggest that the HiPAT process may be more severe than normal ageing (Hayton et al., 1999). The technique, on the other hand, has potential as a means for identifying binders that have aged significantly in service. The extended recovery test, which is an

elaboration of the RRT used to age emulsions or cuts containing extremely volatile oil, is an alternative to the HiPAT approach. The process is keeping emulsion or cutback bitumen samples at 85°C in the RTFOT for two hours with nitrogen gas flow, then another 22 hours with air flow.

Iowa Durability Test

The Iowa Durability Test (IDT), which combines thin film ageing with oxidative ageing (Lee, 1973). The test involves ageing binder residue from a normal TFOT for up to 1000 hours in a pressure vessel at 2.07 MPa with pure oxygen at 65°C. The film thickness during the pressure-oxidation process remains 3.2 mm since the TFOT residual binder is not moved from its container. According to (Lee, 1973), the IDT utilises to age bitumen created a hyperbolic relationship comparable to that observed in binders aged in the field for five years. Moreover, he had founded that 60 months field ageing is similar to 46 hours of ageing with the IDT test.

Microwave Ageing

Bishara et al. (2000) proposed a microwave method of ageing neat, unmodified bitumen to generate a product that is comparable to that obtained by combining RTFOT and PAV ageing. The one-step method involves exposing bitumen to microwave radiation for 4.5 hours at a temperature of 147°C and an air pressure of 3.08 MPa, with an output power of around 1000 W. The results from the microwave approach were found to be comparable to those obtained through RTFOT + PAV ageing based on both physical and chemical examination.

Ultraviolet and Infrared Light Treatments

Vallerga et al. (1957), who aged bitumen films in TFOT containers, reported on the utilization of UV and IR radiation to age bitumen. When compared to infrared light, UV treatment was found to be more successful in modifying the physical properties of the bitumen. Actinic light was employed by Traxler (1963) to simulate bituminous photochemical deterioration. His findings reveal that the photochemical reaction has a considerable influence on thin (3 micron) bitumen films, but that the effect diminishes as the film thickness increases. Montepara et al. (1996) constructed an ultraviolet ageing chamber to age ordinary paving grade bitumen over time. A mercury gas lamp with a frequency range of 180 to 315 nm is used in the chamber (UVC and UVB). To obtain a binder film thickness of roughly 1.5 mm, bitumen is heated to 140°C and placed on glass plates (25 cm x 20 cm). The plates are then aged for 450 days on an ageing table at a specified distance below the lamp (equivalent to approximately 2000 solar days). Bitumen samples from the glass plates are tested for penetration, softening point, and viscosity at 20-day intervals, as well as Nuclear Magnetic Resonance (NMR) and Fourier Transform Infrared Spectroscopy (FTIR). Initial tests with a variety of binders utilizing the UV ageing approach demonstrate that normal long heating and oxidative procedures (RTFOT followed by PAV) create distinct ageing effects than the photochemical process. This means that, in most cases, the ageing outcomes acquired by photochemical treatments cannot be replicated by thermal-oxidative treatments, especially for UV-sensitive binders.

2.2.2 Simulation at mixture level

The main approach (Airey, 2003) is to age the mixture experimentally and then evaluate the influence of ageing on key material properties (eg stiffness, viscosity, strength etc). Before suitable testing (e.g. compressive testing, tests on recovered binder, etc.), prolonged heating techniques commonly expose the mixture to high temperatures for a specific period(s). To laboratory age specimens, oxidation tests typically use a mixture of high temperature and pressure oxidation. Specimens are exposed to UV or infrared radiation during ultraviolet/infrared treatment.

Thermal ageing

The American Association of State Highway and Transportation Officials (AASHTO) guidelines suggest drying asphalt mixtures for a few hours and days, respectively, for short- and long-term ageing. Some researchers have used three forms of conditioning described in the AASHTO R30 standard procedure, the mixture is conditioned in a forced-draft oven for various periods of time and at various temperatures in this common method.: Mixture conditioning for volume mixture design (2 hours, temperature varies), short-term conditioning to replicate the ageing that occurs during combination mixing and placement (4 hours, 135°C), long-term conditioning to imitate ageing after construction and over the lifetime of the pavement (5 days, 85°C).

Plancher et al. (1976) aged 25 mm thick by 40 mm diameter specimens at 150°C for 5 hours using oven ageing process. The materials were then cooled to 25°C for 72 hours before being subjected to resilient modulus tests. Kemp and Predoehl (1981) aged Ottawa sand mixes for

up to 1200 hours in a 60°C oven. After that, the bitumen was recovered and examined. However, they chose to age bitumen with the TODT since it produced significantly more bitumen than the Ottawa sand combinations. Von Quintas et al. (1988) used a forced draft oven to evaluate long-term ageing of compacted asphalt mixture specimens, which were aged for 2 days at 60°C followed by 3 days at 107°C. Furthermore, Bell (1989) states that the high temperature utilized in the test may cause specimen instability, especially for asphalt mixtures with significant void content and/or high penetration grade. The SHRP-A-003A research subsequently led to the development of short and long-term ageing methods. Von Quintas et al (1988) are the foundation for the SHRP short-term oven ageing (STOA) process. Due to compaction, loose mixtures must be aged for four hours at 135 degrees Celsius in a forced draft oven (AASHTO PP2). Bell et al., 1994; Monismith et al., 1994) discovered that the process represents ageing that occurs during mixing and placement as well as pavements that are less than two years.

SATS

The saturation ageing tensile stiffness (SATS) (Y.C. Choi, 2005) described by the European specification EN 12697-45, is the first of its type, combining in a laboratory test the ageing and water damage mechanisms to which an asphalt pavement is exposed in service. The test was found to successfully reproduce the loss in stiffness observed with high modulus asphalt material in the field (Collop et al. 2004a), as well as to distinguish between poor performing material and alternative mixtures incorporating aggregate with good durability track records (Choi et al, 2002, Airey et al. 2003, Collop et al. 2004b and Choi,

2005). The standard SATS process includes conditioning five pre-saturated specimens in a pressure vessel for 65 hours at a temperature of 85°C at 2.1 MPa air pressure. After that, the air pressure (Grenfell et al., 2012) is released and the vessel is opened to remove the specimens for stiffness testing after a 24-hour cooling time. According to (MCHW, 2004), in a design specimen tray, the pressure vessel can accommodate five nominally similar specimens (100 mm in diameter and 60 mm in thickness). As described by Airey et al., (2005) the conditions employed with the SATS process were selected to replicate in the laboratory the field observed moisture damage as evidenced by a decrease in stiffness modulus for specific asphalt mixtures. The following are the main characteristics of the conditioning procedure:

- A heated, well-insulated pressure vessel that can hold five compacted asphalt specimens (100 mm diameter 60 mm height).
- Pressure and temperature control can be controlled at the same time with this conditioning setup.
- On a specially constructed tray, asphalt specimens that have been pre-saturated with water (under vacuum).
- During the conditioning phase, a predetermined amount of water is introduced in the vessel such that the bottom specimen is totally immersed.

- Simultaneous conditioning of five specimens at 85°C for 24 hours under 0.5 MPa air pressure, followed by a 24-hour cooling phase before the pressure is released and the vessel opened to extract the specimens for stiffness testing.

Ultraviolet and Infrared Light Treatments

Kemp and Predoehl (1981) used an actinic light ageing test with 1000 MW/cm² Angstrom actinic radiation at a temperature of 35°C for 18 hours. The ageing test, according to the authors, only assesses hardening inside the top 5 microns of the bitumen layer, regardless of bitumen film thickness. For varying durations of time, Tia et al. (1988) used a range of ageing processes, including convection oven ageing at 60°C, force draft oven ageing at 60°C, and ultraviolet light ageing at 60°C. They suggested a better ageing method that included both ultraviolet light and forced draft oven heating. Furthermore, UVlight was recognized as a primary driver of mixture ageing, however the resulting effect is only visible on the surface, or at least not at any considerable depth inside the mixture.

TEAGE

The purpose of TEAGE conditioning is to imitate the ageing of asphalt mixtures in the laboratory. The TEAGE simulation focuses on two major factors that influence asphalt pavement ageing: sun radiation and moisture damage. TEAGE uses a mix of UV radiation and watering/drying cycles to replicate those processes.

CAI

The National Cooperative Highway Research Program (NCHRP) recently proposed a climatic ageing index (CAI), which is dependent on pavement location, age, and depth and can be calculated using historic field data. The loose asphalt mixture is conditioned at 95°C to simulate ageing in the laboratory in this case. The conditioning period is adjusted to match the desired CAI, which has been determined previously for the desired location (within US states), age, and pavement depth. The European specification CEN/TS 12697-52 defines the Viennese Ageing Procedure (VAPro), which recommends ageing of compacted asphalt mixture specimens by employing a flow of oxidant gas through to the volume of the specimen.

2.3 Conclusions of the state of the art

- Short-term and long-term ageing of asphalt mixtures are the two main types of ageing. Long-term ageing is mainly caused by oxidation and some steric hardness in the field, while short-term ageing is associated with bitumen volatilization inside the asphalt mixture during mixing and construction. Nonetheless, previous authors have stated that the type of bitumen play a key role on ageing of mixtures.
- As a result of exposure to temperature and climatic conditions, the complex molecular structure of asphalt and its chemical components are progressively altered, leading to changes in asphalt properties. Due to the importance of this process and the lack of knowledge into ageing factors, parametric analysis into this phenomenon is demanded.

- There has been relatively less research on the ageing of asphalt mixtures compared to research on asphalt binder. Many of the initial research on asphalt ageing focused entirely on binders, with little consideration of mixtures.
- Thus, there is still a gap in the understanding of the ageing mechanism, particularly for mixtures with high air void content (more susceptible to ageing phenomenon).
- Curing asphalt mixtures for 4 hours at 135°C for short-term ageing and 5 days at 85°C for long-term ageing is the typical protocol for simulating ageing. These typical ageing processes have limits and cannot be applied to a variety of environmental situations. As a result, it is desirable to create and validate a new ageing modelling approach that accounts for various climatic circumstances (time, temperature, pressure, etc.) as well as mixture properties such as (air void content, type/dosage of aggregates, cement, binder, etc.).
- Various modified binders have been employed to improve the asphalt pavement, resulting in improved flexible pavement performance and significant cost savings over time. Nonetheless, more study into ageing of modified bitumen mixtures is needed in order to develop a more effective and long-lasting asphalt mixture that can work effectively at both hot and low temperatures.
- Even though several studies were established for unmodified binders and need to be re-evaluated for polymer changed binders, laboratory simulation approaches of ageing actions on mixtures with modified binders are still a significant

outstanding challenge, evaluating also other designing and manufacturing variable that can affect mixture durability due to ageing phenomenon.

3. Objectives

3.1 Main objective

As the State of the Art conclusions illustrate, there is still a knowledge gap in terms of ageing factors and susceptibility, particularly for high air void content mixtures with polymer modified bitumen. As a consequence, the main objective of this thesis is to evaluate the impact of design factors on the resistance to ageing of bituminous mixtures for surface course in road pavements in order to improve their durability and resistance to ageing.

3.2 Specific objectives

- Studying different laboratory ageing conditions (climatic issues, external factors) in order to define those more representative to simulate ageing at laboratory level for asphalt mixtures.
- Studying the impact of design parameters (dosage/type of different components) on ageing phenomenon in asphalt mixtures.
- Defining the weight of each design parameter on the resistance to ageing of bituminous mixtures in order to improve their durability.

4. Methodology

4.1 Materials

This investigation used asphalt mixtures of type BBTM 11B (UNE-EN 13108-2), which is a gap-graded mixture with a maximum aggregate size of 11 mm. It is a 0/11 discontinuous granulometry mixture made from modified bitumen with polymers or rubber. This was used since it is commonly used for thin surface courses, which are exposed to climate changes, and it has an air void content of 12-18%.

Two modified binders commonly used in the manufacturing of this type of mixture were used to assess the impact of ageing factors and the influence of design parameters on mixtures manufactured with different binders (which has been demonstrated to play a key role in the ageing mechanisms): one with SBS polymer (PMB 45/80-65), which is widely used for asphalt mixtures in wearing courses, and another with SBS plus crumb rubber (PMBC 45/80-65), whose application is increasing due to its proper mechanical performance and its more sustainable material profile. These two binders have a penetration value between 45 and 80 mm⁻¹ and had a softening point of more than 65°C (UNE-EN 1427:2007).

The black diagram (Figure 3) shows a graph of the complex shear modulus (G) versus phase angle, with frequency and temperature removed. The complex shear modulus represents the effect of bitumen's mechanical reaction at various temperatures.

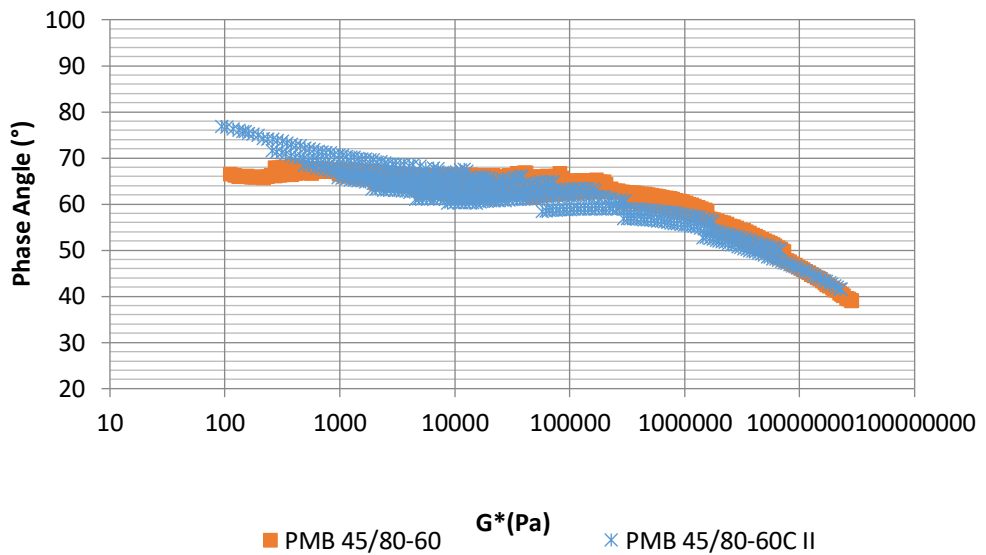


Figure 3 Black diagram

Table 1 summarizes the key physical and mechanical characteristics of the aggregates employed in this study, all of which meet the standards for use in the manufacturing of asphalt mixtures for surface layers, as defined by Spanish Standard PG-3. According to EN 1097-3, the cement filler was type CEMII/B-L 32.5 N, with more than 96% passing 0.063 mm and a bulk density of 0.6 Mg/m³.

Table 1 Aggregates properties.

Properties	Standard	Results		Standard limits
		6/12	0/4	
Coarse aggregate shape. Flakiness index	EN 933-3	15	-	<25
Percentage of fractured face	EN 933-5	98	-	>90
Resistance to fragmentation (Los Angeles coefficient)	EN 1097-2	11	-	<25
Cleaning (organic impurity content)	EN 13043	0.4	-	<0.5
Sand equivalent	EN 933-8	-	77	>55
Apparent density (Mg/m ³)	EN 1097-6	3.21	2.77	-
Saturated Surface Dry Density (Mg/m ³)		3.12	2.70	-
Density after drying (Mg/m ³)		3.15	2.73	-
Water absorption after immersion (%)		0.84	0.88	-

For the first part of study which focuses on assessing the influence of temperature, time and pressure, both mixtures (BBTM 11B-PMB and BBTM 11B-PMBC) had an identical mineral skeleton, with limestone aggregates for the fine fraction (0/6 mm) and ophitic aggregates for the coarse fraction (6/12 mm), and cement as the filler. Figure 4 illustrates the gradient of the BBTM 11B asphalt mixture.

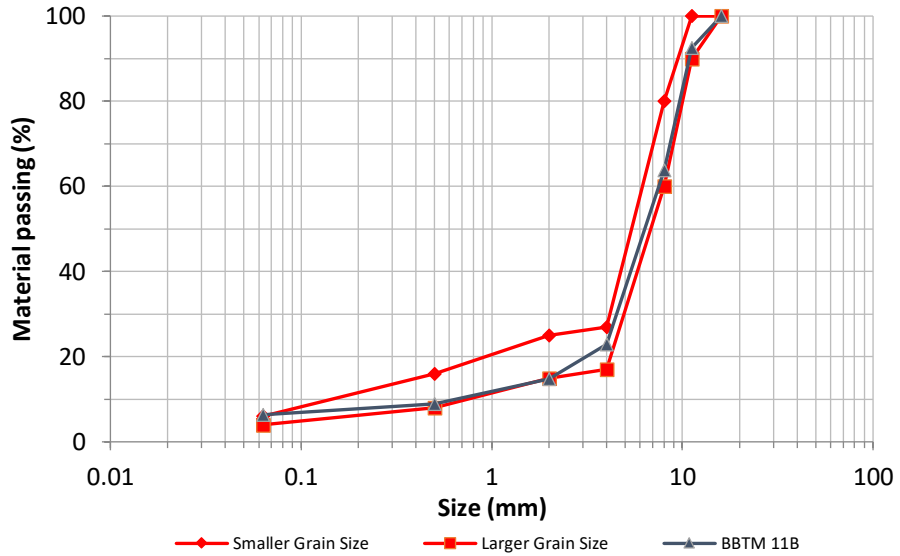


Figure 4 Aggregate gradation for BBTM 11B mixtures used in this study.

To prevent contributing other relevant variables except than the type of binder to the three primary parameters impacting mixture ageing (temperature, time and pressure), the binder content was maintained at 4.75% of the total mixture weight for both asphalt mixtures). The basic properties of the bituminous mixture BBTM 11B adopted for this research are summarized in Table 2. The response of the gap-graded mixture was characterized using the Marshall technique (UNE-EN 12697-34).

Table 2 Properties of BBTM 11B mixtures.

Mixture	BBTM11B PMB 45/80-65	BBTM11B PMBC 45/80-65
Apparent density (kg / m ³), EN 12697-6	2130	2110
Air voids (%), EN 12697-8	17.7	17.6
Indirect dry tensile strength, ITSd (kPa), EN 12697-23	1079.0	1008.2
Retained resistance to water, ITRSR (%), EN 12697-12	94.0	91.8
Deformation slope under load, WTS (mm / 10 ³ cycles), EN 12697-22	0.06	0.07

For the second part of experiment which concentrates on evaluating the impact on mixture ageing rate of the designing factors that can vary during the industrial manufacturing of the mixtures, the same materials as those used in the first part were examined, but adjusting the composition of different elements of mixtures, which could modify the ageing resistance.

4.2 Testing plan

This section describes the testing plan of the Thesis, the methodology was divided into 2 phases: laboratory procedures and factors that influence ageing.

4.2.1 Study of laboratory procedures for mixture ageing

The technique was subdivided into two parts: The first part focused on laboratory assessment of impact of ageing factors studies in this research which are temperature, time and pressure. The second part examined the field comparison with the laboratory techniques.

4.2.1.1 Assessment of impact of ageing factors: temperature, time and pressure

The testing plan was divided into three phases in response to the three primary factors that influence the ageing process studied in this research, which were simulated in the lab: (1) the temperature of the ageing process; (2) the exposure period; and (3) the pressure level during the ageing process. Table 4 shows a description of the testing program.

Table 3 Testing programme summary.

Ageing variable of analysis	Material-type of bitumen	Ageing parameter			Tests
		T ^a (°C)	Time	Pressure process	
(reference)		-	-	-	
Temperature	BBTM 11B-PMB	80 vs 135	9 days	-	-Stiffness
Time		135	4h vs 9 days	-	
Pressure	BBTM 11B - PMBC	80	9days	+ 65h at 85°C under 2.1 MPa	-Fatigue cracking
		135	4 hours	+ 65h at 85°C under 2.1 MPa	

Eighteen specimens were manufactured and compacted using a gyratory compactor (UNE-EN 12697-31) for each type of mixture (BBTM 11B with bitumen type PMB and PMBC, respectively), turning until specimens with a height of 35 mm were obtained while obtaining an appropriate density (greater than 98 percent of the design density). Each sample had a thickness of 35 mm, which is a good value for this type of mixture when employed as a thin surface course layer in pavements. Different conditioning processes were carried out on these specimens to simulate ageing and assess the three selected factors (temperature, time, and pressure), with the goal of determining the

change in material stiffness and fatigue cracking behavior in comparison to specimens that had not been conditioned.

- Ageing temperature. According to previous studies (Harrigan, 2007; Valdés et al., 2014), 6 specimens of each mixture were stored at 80°C in an oven and 6 specimens of each mixture were stored at 135°C in an oven for the assessment of the first factor (ageing temperature). This represents the extreme temperature range of values commonly used in the simulation of ageing in the laboratory. Two different temperatures (one low at 80°C and the other high at 135°C) were utilized to simulate an unfavorable and accelerated state (extreme condition in the case of 135°C) that reflects the influence of ageing. According to other experts (Lee et al., 1997; Sol-Sánchez et al., 2015), 9 days is an adequate time for obtaining a long-term mixture ageing corresponding to a service life longer than 10 years. To examine the influence of temperature independently of other parameters, no pressure changes were created. To avoid deformation in the specimens, all specimens that were stored in an oven were encircled by a metallic grill (Figure 5).



Figure 5 Bituminous mixture specimen surrounded with a metallic grill.

- Ageing time. The second factor was used to investigate the effect of time by placing 6 specimens of each type of mixture in an oven for four hours and 6 more specimens for nine days. The storing temperature was kept constant at 135°C during both durations, which was considered an unfavorable situation, and no pressure was applied.
- Ageing pressure. To examine the third factor (ageing pressure), 6 specimens obtained from the processes of conditioning at 135°C for 4 h and at 80°C for 9 days (selected as intermediate cases from the conditions previously studied, and thus susceptible to requiring additional ageing) were subjected to an additional process consisting of applying pressure at under 2.1 MPa at a temperature of 85°C for 65 hours. This method was used in the SATS equipment (Saturation Ageing Tensile Stiffness), where the conditions from the standard test UNE-EN 12697-45 (2013) were replicated, but without the humidity factor to avoid material degradation due to the presence of water, since the goal was to evaluate the pressure action separately from other factors (Figure 6).



Figure 6 The Saturation Ageing Tensile Stiffness (SATS) test.

3.2.1.2 Field experience laboratory – field comparison

An approach was used to analyze the performance of asphalt cores collected from a road where both types of asphalt mixtures were utilized as a surface layer 60 months before, with the goal of comparing the laboratory ageing techniques to the real phenomenon in road pavements.

In aspects of the field phase, the laboratory results were compared to those obtained from cores extracted from a pavement in which these combinations were utilized, with both binders PMB and PMBC, 60 months after the road was built. This experiment was conducted on the A-316 highway in southern Spain, in the province of Jaén.

4.2.2 Evaluation of the weight of designing parameters

Six specimens were manufactured for each mixture (designing factor) and compacted using a gyratory (UNE-EN 12697-31) compactor with 60 gyros, defined to achieve identical density in the control mixture to that designed using the Marshall technique. Each sample had a thickness of 35 mm, which is a good value for this type of mixture when used as a thin surface course layer in pavements. Three

specimens were maintained in an oven for 9 days at 135° C for each mixture. This ageing parameter (which requires extended periods of time) was developed based on a prior laboratory study that increased the parameter's ability to duplicate a visible ageing effect while also identifying the sensitivity of different materials to ageing.

The testing plan was divided into four phases in response to four design factors that are thought to have a strong impact on bituminous mixture performance and, as a result, were used in this research to determine their impact on ageing resistance, with the goal of improving the durability and long-term behavior of asphalt mixtures that were manufactured and simulated in the lab. The following were the study steps, which are also listed in Table 4:

- The effect of bitumen type: Polymer modified bitumen (PMB) and rubberized bitumen (PMBC) were used to compare two asphalt mixtures. These binders were chosen because they are commonly used in this sort of mixture for wear course in road pavements, and therefore evaluating the impact of utilizing two different, but commonly used, bitumens on the mixtures' resistance to ageing.
- Bitumen content impact. This parameter was investigated even though it is normal for it to change based on the mixture design. The dosages used in this study were 4.75% and 5.5% over total mixture weight, which can be common designing values for different mixtures while also replying to possible variations that may occur during the industrial manufacturing process, and thus being critical to understanding the impact on mixture long-term performance.

- Dosage of fillers effect. As with bitumen, possible changes of this parameter are expected throughout the mixture design or manufacturing process, necessitating the examination of common values such as those chosen in this study: 4% and 6% of total mixture weight.
- The effect of the mixture's mineral skeleton. Two dosages of sand were tested to understand the impact of modifying the granulometric curve of the mixture on its ageing resistance: 20% and 35%, which indicate an adequate range of sand for this type of combination, according to standards like the Spanish one (PG-3). To create a correct design and cohesive mastic for the combination with a larger proportion of sand, the mixes used to analyze the influence of sand had a bitumen dosage of 5.5 percent and 6 percent of filler in this study stage.

Table 4 Testing programme summary.

Study step / Designing factors	Mixtures composition				Ageing process	Tests
	Bitumen	% b	% f	% s		
Type of bitumen	PMBC vs PMB	4.75	6	20	None (reference) Aged during 9 days at 135°C in oven	Indirect Tensile Stiffness
Bitumen dosage	PMB	4.75 vs 5.5	6	20		
Filler percentage	PMB	4.75	4 vs 6	20		Fatigue cracking resistance (UGR-FACT)
Sand quantity	PMB	5.5	6	20 vs 35		

4.3 Testing methods

To evaluate the impact of each design parameter on the ageing of all the mixtures studied in this thesis, stiffness and fatigue cracking were explored. To determine the effect of ageing, researchers examined the material's bearing capacity and its durability under repetitive pressures.

The material stiffness was assessed using the UNE-EN 12697-26 standard. This examined at how the material's elastic response and ability to support weights without deforming changed as it aged. The stiffness of a mixture is a significant mechanical property in the wearing course of an asphalt surface. This test was carried out at 20°C on cylindrical specimens (height 35 mm, diameter close to 100 mm) by applying 10 charge pulses to control the amplitude and duration of the load, followed by another 5 pulses to determine the stiffness of the material. The same approach was used on a second diameter of the test piece, but this time at a $90 \pm 10^\circ$ angle to the previous case, allowing a mean stiffness module value to be derived using the readings from both diameters (Figure 7).



Figure 7 Stiffness test

The UGR-FACT model (Moreno-Navarro, Rubio-Gómez, 2014) was used to evaluate the fatigue cracking life of bituminous layers in road pavements (Figure 8), which duplicates similar efforts made in bituminous layers during their service life (i.e., bending, shear and tensile stresses). Vertical loads are applied to the specimen's upper surface, which is supported by two flexible pads that allow for flexion movements. Furthermore, the load is passed to the bottom, where there are two carriages, causing tensile strains as well as shear forces due to the obstruction of one slide. This test was performed at a frequency of 5 Hz with a vertical cyclic stress amplitude of 0.2 MPa. For each test condition, three specimens measuring 100 x 60 x 35 mm were used at a temperature of 15 °C.

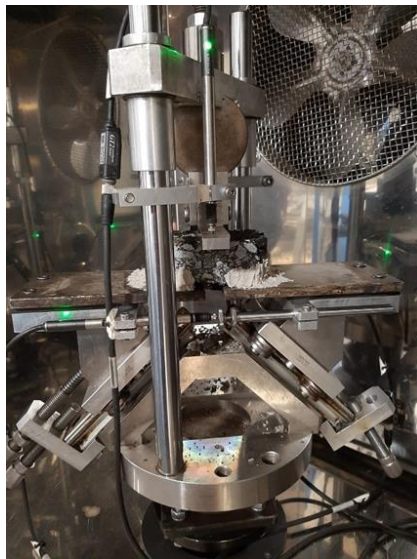


Figure 8 UGR-Fact test.

Four LVDTs (one vertical and one horizontal on each side of the specimen) are used to assess the vertical and horizontal displacements induced in the material during each load cycle. Two types of displacements can be seen in each direction (horizontal and vertical),

from which the displacement of the specimen can be calculated based on the measurements taken, depending on the load cycle: a "permanent" displacement (h_i, v_i), which persists after the load cycle and is related to the material's non-recoverable deformations or damage; and a "relative" displacement (H_i, V_i), which is related to the material's consistency (stiffness) or damage state.

The testing was performed until the material failed (i.e., after the complete breakage of the specimen due to bottom-up cracking). The total number of cycles up to failure, the initial (at cycle number 100) amplitude of horizontal deformation at the bottom of the layer, which indicates the material's flexibility or stiffness, and the parameter of susceptibility ($|\delta|$) to plastic deformation (ductility), calculated from the slope of permanent deformations (in vertical and horizontal directions) during cycles 500 and 1,500, were the main parameters calculated from the following equation (1):

$$\delta = \sqrt{\left(\frac{d_{v1,500} - d_{v500}}{1500 - 500}\right)^2 + \left(\frac{d_{h1,500} - d_{h500}}{1500 - 500}\right)^2} \quad (\text{Eq.1})$$

5. Analysis of the results

This section analyses the results obtained during the Thesis, showing firstly the results of the step focused on analysing different laboratory procedures for mixtures ageing which influence by temperature, time and pressure as the most relevant factors. Secondly, the results of the step devoted to evaluating the weight of design parameters of mixtures are presented.

5.1 Analysis of study of laboratory procedures for mixture ageing

5.1.1 Influence of temperature on the ageing laboratory process

Figure 9 shows the changes in mixture stiffness after 9 days of conditioning when exposed to different temperatures (80°C versus 135°C, compared to the reference mixture without ageing), as well as the relationship between the variation in deformation susceptibility and temperature (measured through the fatigue test). The results for the BBTM 11B with polymer modified bitumen (PMB) and the case with rubberized bitumen (PMBC) are shown in Figure 9, with the goal of determining the impact of ageing on mixtures with various binders.

The tendency is identical for both mixtures, according to the findings. The higher the ageing temperature, the larger the change in stiffness of the materials (i.e., the more rigid the material), which effects the material's deformation capacity, which decreases at deformation level (reducing the ductility of the mixture). This is consistent with prior research (Airey, 2003; Naskar et al., 2012) aimed at determining the effect of temperature on bitumen ageing and, as a result, demonstrating repeatability at the mixture level.

Regardless of the type of bitumen used, ageing at 80°C resulted in identical stiffening (approximately 40% increased stiffness) for both combinations. However, depending on the bitumen type, a higher ageing temperature (135°C) resulted in a variable response from the combinations. There was a 212% increase in stiffness (for the mixture with PMBC binder) and a 137% increase in stiffness (for the mixture without PMBC binder) (for the mixture with PMB binder). Additionally, the susceptibility of the material to permanent deformation is reduced more in the PMBC binder mixture (about 82%) than in the PMB binder mixture (where the decrease in the capacity to deform is less) (about 25%).

As a result, the significance of the temperature of ageing may be demonstrated. When comparing both types of combinations with a different binder, it was discovered that the mixture with tire crumb rubber binder content (PMBC) had more alterations owing to temperature-related ageing, resulting in a stiffer and more deformable material. Other authors (Gallego et al., 2016; Sol-Sánchez et al., 2020) have found similar results.

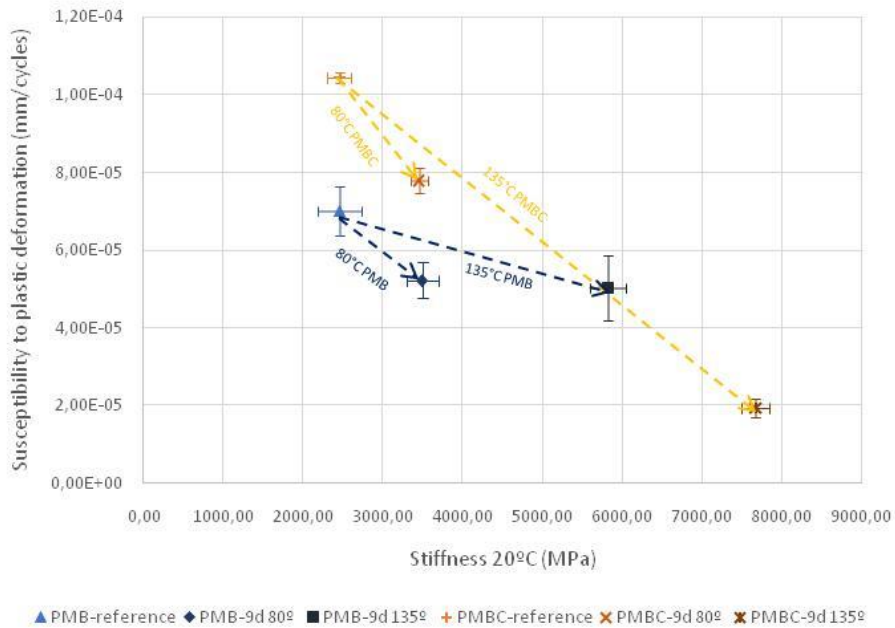


Figure 9 Influence of temperature in the ageing laboratory process.

Figure 10 shows the average values of cycles to failure obtained from both mixtures, as well as the relationship with the values of initial horizontal amplitude (measured in cycle 100 during the fatigue process in the UGR-FACT test), depending on the ageing process, in order to assess the impact of temperature ageing on the material's durability. The stiffening causes smaller initial horizontal deflections at the layer's bottom, which is consistent with previous research (Bahia, 1999; Sol-Sánchez et al., 2018). The effect of the conditioning temperature was validated in specifically, as both mixtures reduced inflexibility by approximately 30% at 80°C and doubled at 135°C. Despite having varying stiffnesses, the effect on horizontal deflections was found to be similar for both combinations (mainly because of bitumen hardening). Such stiffening due to the ageing process leads to a higher number of cycles up to failure, but has various effects depending on bitumen type, as a result of the reduction in horizontal deflections at the bottom of the

specimen (more accentuated while increasing temperature process). The mixture with rubberized bitumen had a higher durability, with the changes being more pronounced when the ageing temperature was increased, as observed in earlier results where the mixture with crumb rubber was more vulnerable to ageing.

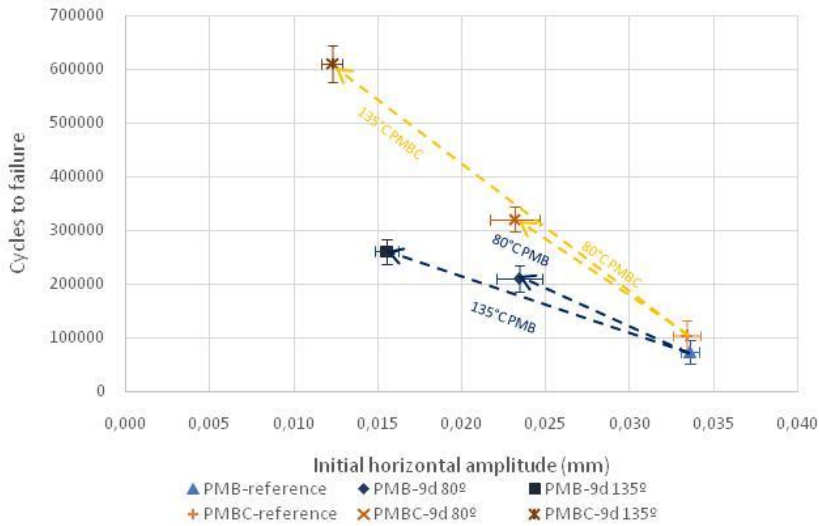


Figure 10 Results measured in the UGR-FACT for the mixtures after different temperatures of ageing.

Figure 11 shows the mean values of final horizontal deflection for each case at the end of the test (assessing the material's deformability before cracking) in relation to the number of cycles for a better understanding of the effect of temperature on ageing and mixture durability. Although it was demonstrated that ageing resulted in a higher number of cycles (due to lower initial horizontal deflections as seen in previous results), it must also be considered that this phenomenon resulted in a lower capacity of the material to deform, resulting in a more brittle material with a lower capacity to deform before cracking, which is consistent with previous studies (Moreno-Navarro et al., 2016). The increase in ageing temperature, in particular, has a significant impact on material

behavior, resulting in materials with up to half of their deformation capacity (depending again on the type of bitumen, which confirmed to be a key parameter to be considered in this type of analysis). As a result, its use on supports with a tendency to settle (which necessitates materials with high ductility) could affect the durability of these aged mixtures in comparison to other materials with a greater capacity to adapt to deformation, with the ageing process, as well as the simulation temperature, playing a key role in this material property.

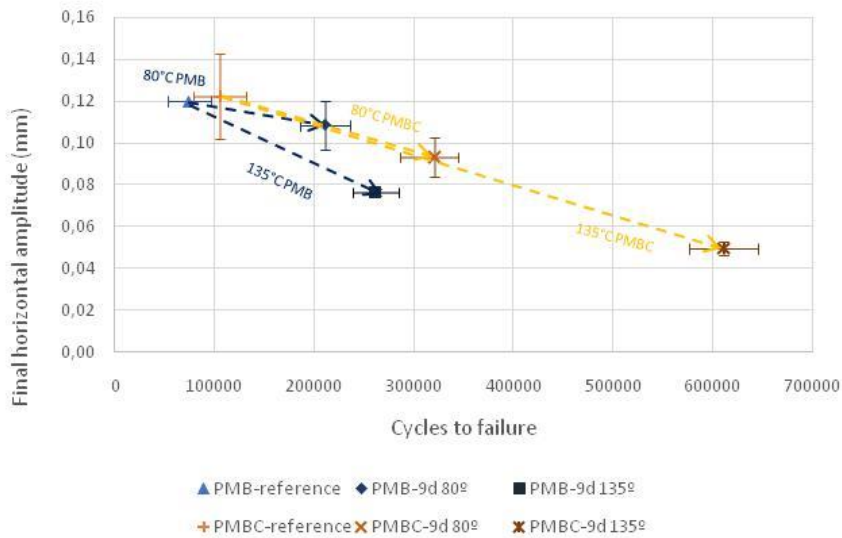


Figure 11 Results of durability and capacity to deform of the mixtures after ageing at different temperatures.

5.1.2 Influence of time on the ageing laboratory process

Figure 12 examines the effects of conditioning time (at 135°C, chosen as the most unfavorable from previous results) on stiffness and susceptibility to deformation for both asphalt mixtures after 4 hours (selected as short-term) versus 9 days (selected as long-term) (long-term). When the specimens were aged for 4 hours, there was little fluctuation in stiffness and deformation, with stiffness variations of around 7% and 8% for the mixtures containing bitumen type PMB and

type PMBC, respectively. When the exposure time is increased to 9 days, the rigidity of both cases increases significantly (more so for the PMBC, whose stiffness increased up to three times), and the tendency to deform under repeated loads decreases by around 28% for the mixture with PMB and close to 82 percent for the case with PMBC. As a result, the results show that conditioning time is important in simulating the ageing of mixtures, as evidenced by previous research (Curtis et al., 1993; Sol-Sánchez et al., 2015) that focused on bitumen and required extended periods to reproduce significant changes in material properties. Furthermore, as demonstrated in the case of temperature conditioning, selecting an appropriate ageing time in the laboratory simulation could be critical in distinguishing the long performance of different combinations, as short periods were seen to result in very comparable alterations.

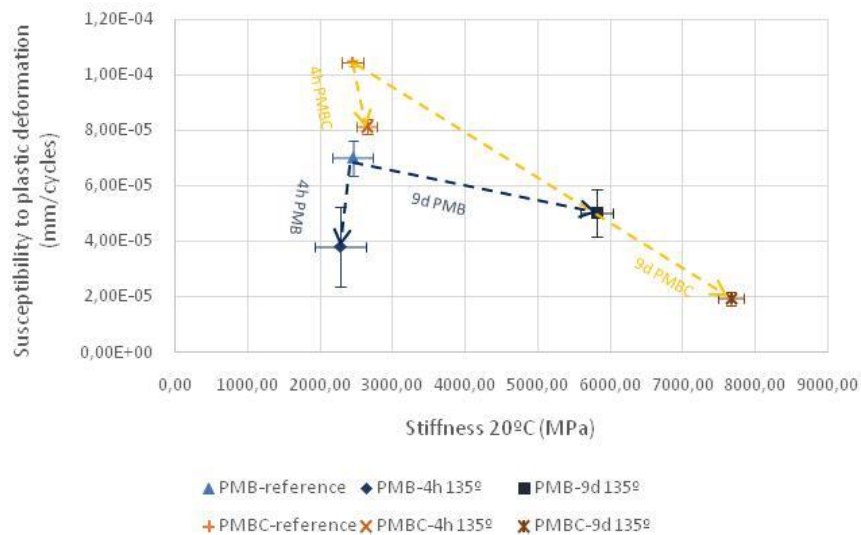


Figure 12 Influence of time for ageing simulation on material stiffness and susceptibility to permanent deformations.

Figure 13 shows the results of the UGR-FACT test for both mixtures, correlating the variation of the initial horizontal displacements (at cycles 100) with the mean values of number of cycles to failure, after 4 hours and 9 days of ageing at 135°C, with the goal of evaluating the influence of conditioning time on the change of mixture durability due to the ageing phenomenon. Short times had little effect on material flexibility (close to 42 percent for the mixture with PMB and 35 percent for the mixture with PMBC) and durability, whereas longer times resulted in significant changes in material performance, as well as a differentiated fatigue life depending on the type of bitumen used. The conditioning for 9 days caused a reduction in initial horizontal amplitude (at the specimen bottom) of close to 54%, resulting in an increase in cycles to failure of close to 3 times, while the conditioning for 9 days caused a reduction in initial horizontal displacements of close to 63%, resulting in an increase in cycles to failure of close to 6 times (double than the case with PMB). When choosing long conditioning periods, this emphasizes the importance of the ageing time, which has varying effects based on material properties/design.

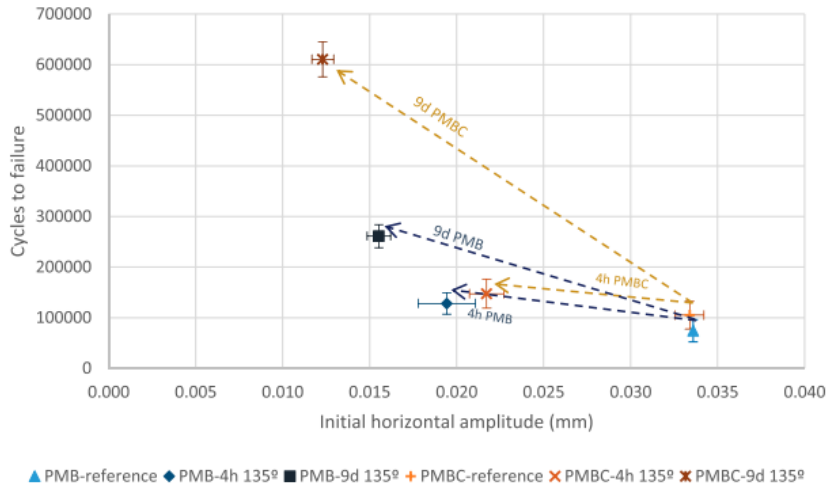


Figure 13 Results of fatigue test for different ageing periods applied in laboratory.

When evaluating the impact of this factor on material ductility, Figure 14 illustrates that, similar to the results in the previous section when evaluating the influence of temperature, material stiffening resulted in a decrease in the ability to deform without breaking, which was accentuated by increasing the exposure time, as short periods were shown to have a reduced effect. As a result, despite the increase in cycles, long-term ageing may result in a significant reduction in material ductility, and therefore its capacity for deformation adaptation, which must be addressed in connection to the material's durability depending on service conditions.

In terms of the bitumen's influence, it's worth noting that PMBC was found to be more susceptible to stiffness changes after long periods of ageing, but the results in Figure 14 show that it has a longer fatigue life and similar capacity to deform before cracking than PMB, indicating that it has a higher potential for longer durability despite having higher stiffness variations due to ageing. As a result, it's critical to investigate the impact of ageing on bitumen type, not just in terms of material

stiffness, as in earlier research (Sol-Sánchez et al., 2020), but also in terms of fundamental qualities like fatigue cracking.

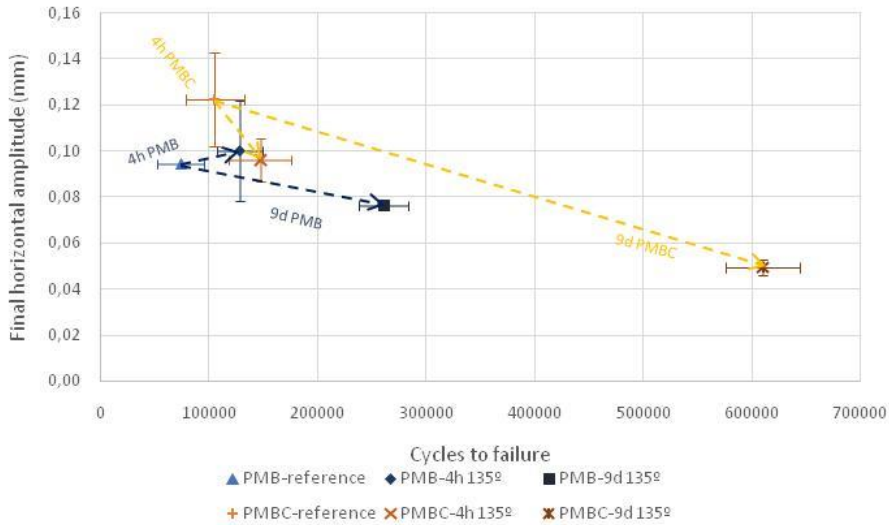


Figure 14 Effect of ageing time on the durability of the mixtures, and their reduction in capacity to deform before cracking.

5.1.3 Influence of pressure on the ageing laboratory process

Figure 15 shows the effect of applying pressure (in this case 2.1MPa at 80 °C) as an additional method for the intermediate cases of the previous processes studied (4 hours - 135 °C: short-term; 9 days - 80 °C: lower temperature), with the goal of determining whether the ageing of these intermediate cases was increased by using the SATS equipment. For both asphalt mixtures, the figure is showing the change in material stiffness and susceptibility to permanent deformations (with PMB and PMBC).

The results show that increasing the pressure factor reduces the stiffness of both combinations while decreasing their resistance to permanent deformations in general. Figures 16 and 17, which show how the pressure process increases the deflections at the bottom of the mixture layer and thus leads to less durability, correlate the change in

material flexibility (through the initial horizontal displacements) with its durability as well as the capacity to deform before cracking (material brittleness). In comparison to previous situations in which just temperature and time were controlled in an oven, introducing pressure via the SATS approach made the mixture less rigid, resulting in higher initial deformation values at the bottom of the layer and, as a result, a shorter material life due to fatigue cracking. This could be linked to material degradation during the pressure process, as observed in previous research (Wang et al., 2020), resulting in an anti-ageing effect in asphalt mixtures.

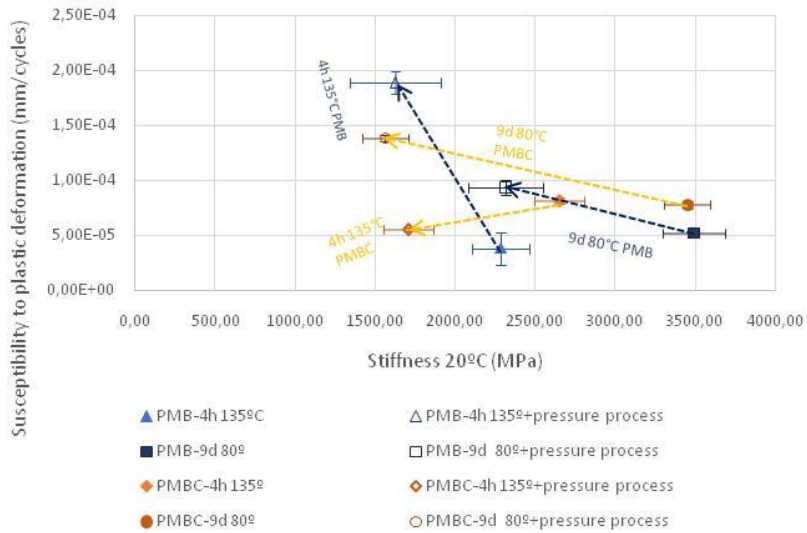


Figure 15 Results of stiffness and trend to permanent deformation after applying pressure with SATS test for both mixtures.

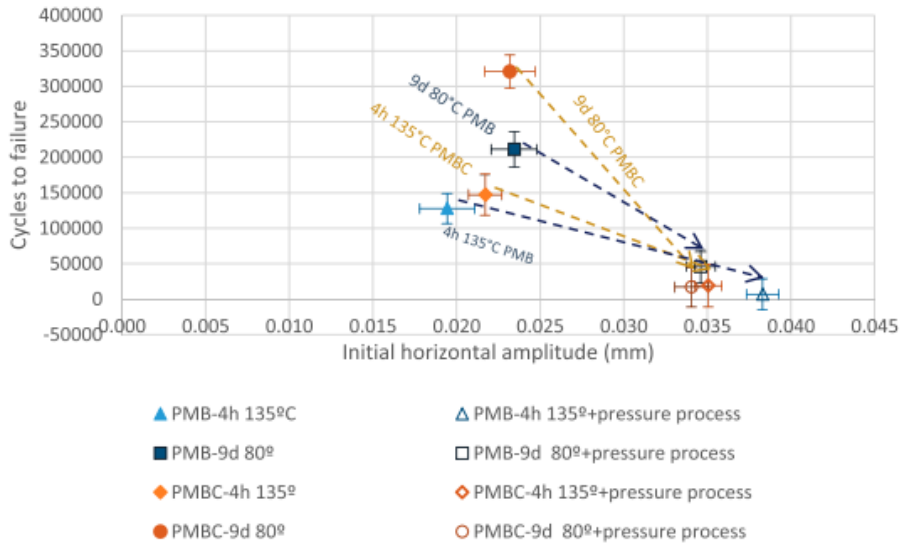


Figure 16 Changes in initial horizontal displacements and durability of the mixtures after pressure process.

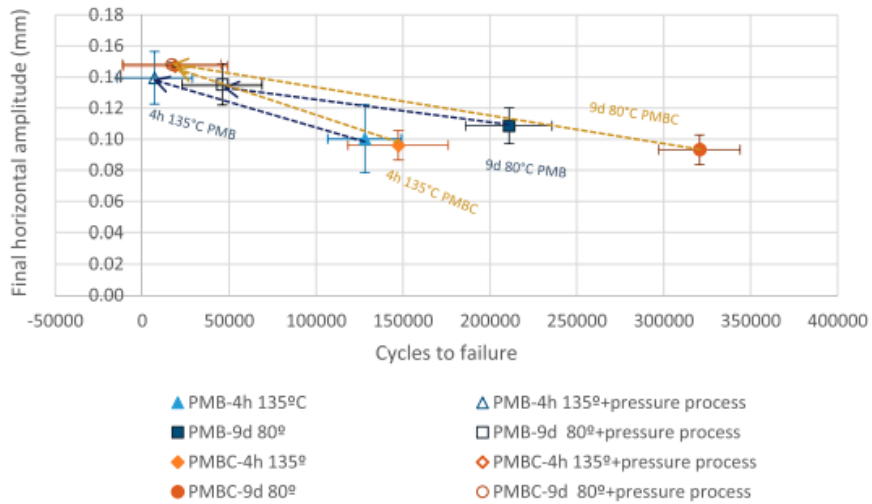


Figure 17 Cycles to failure and final horizontal amplitudes before cracking for the specimens after applying pressure with SATS test for both mixtures.

5.1.4 Comparison between laboratory procedures and field experience
 The Percentage change of the parameters tested in the PMB and PMBC mixtures after being subjected to the ageing processes in the laboratory and from field cores is shown in Figures 18 and 19. The cycles to failure (from fatigue testing) and rigidity were the parameters with the greatest alterations due to ageing, with the 9-day specimen at 135°C being the most important. In this way, it was determined that this method best represented long-term ageing, resulting in material changes comparable to those seen at 60 months for all parameters examined in both mixtures. As a result, this laboratory procedure may be suitable for simulating unfavorable ageing conditions. The 9-day specimen at 80°C was found to be the next method that came closest to field ageing data. However, the 4-hour specimen at 135°C did not match the field samples, indicating that the time variable is critical for reproducing realistic ageing in the laboratory.

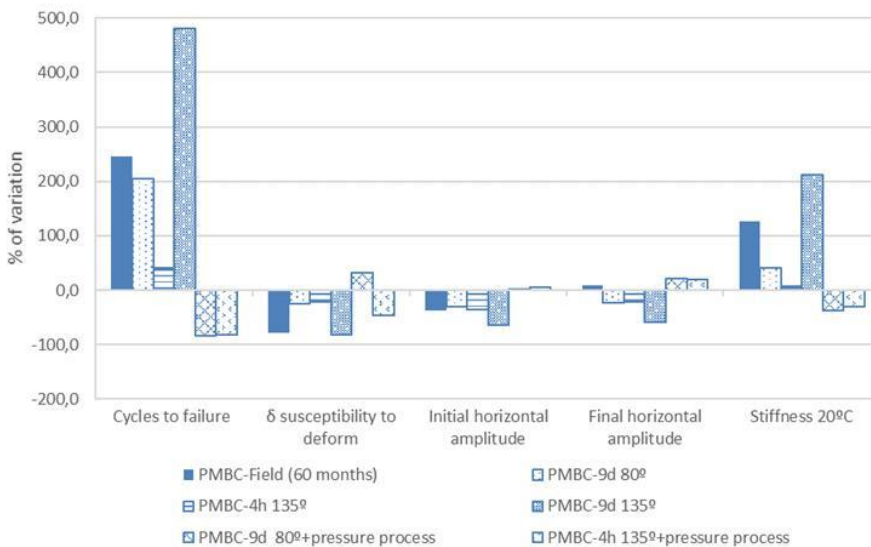


Figure 18 Percentage of variation of different parameters of fatigue cracking of bituminous mixture with PMB binder.

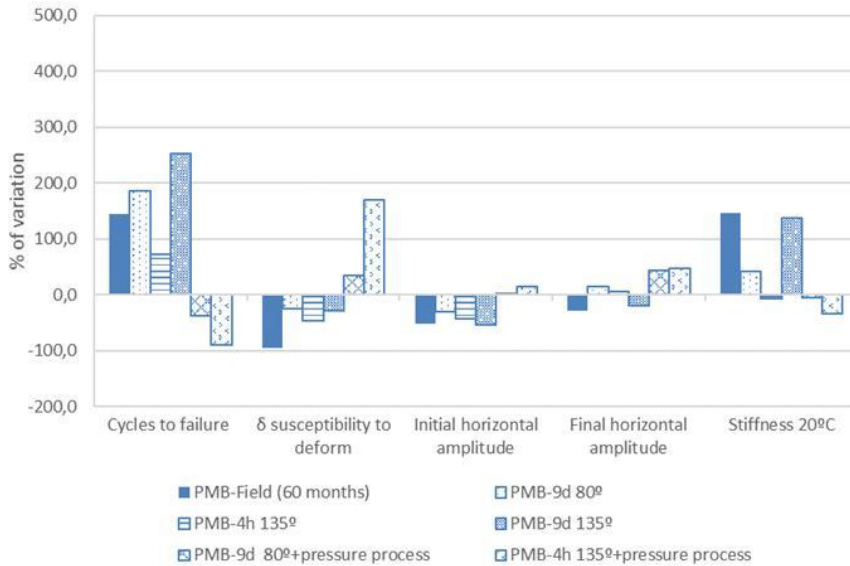


Figure 19 Percentage of variation of different parameters of fatigue cracking of bituminous mixture with PMBC binder.

5.2 Analysis of evaluation of the weight of designing parameters

5.2.1 Influence of type of bitumen on the ageing resistance

Figure 20 illustrates the stiffness modulus (measured in the stiffness test) and susceptibility to plastic deformations (measured at the start of the fatigue test) for two mixtures with similar design factors but different types of binder to investigate the impact of this parameter on ageing resistance by comparing the behavior of both mixtures before and after 9 days of conditioning at 135°C in the laboratory. The specimens that were not subjected to the ageing process were marked "reference," whereas those that were subjected to the conditioning procedure were labeled "aged."

For both forms of mixing, the tendency to alter was similar. Ageing increased the stiffness of both mixtures (with varying bitumen) while lowering sensitivity to plastic deformations due to material hardening,

which is consistent with prior research (Aiery, 2003; Naskar et al., 2012; Moreno-Navarro et al., 2015). It should be noted, however, that the amount of change differed depending on the type of binder. While the PMB mixture increased stiffness by roughly 130 %, the rubber mixture increased stiffness by more than 200 %, resulting in a greater change in susceptibility to deformations (around 80 % against the near 30 % of the PMB). This suggests that the modification of a bitumen mostly by rubber particle, might lead to a considerable variation in the ageing process, resulting in higher hardening of the material, according to these studies. This is in consonance with previous research (Curtis et al., 1993, Gallego et al., 2016, Sol-Sánchez et al., 2020) showing the impact of binder composition on mixture performance.

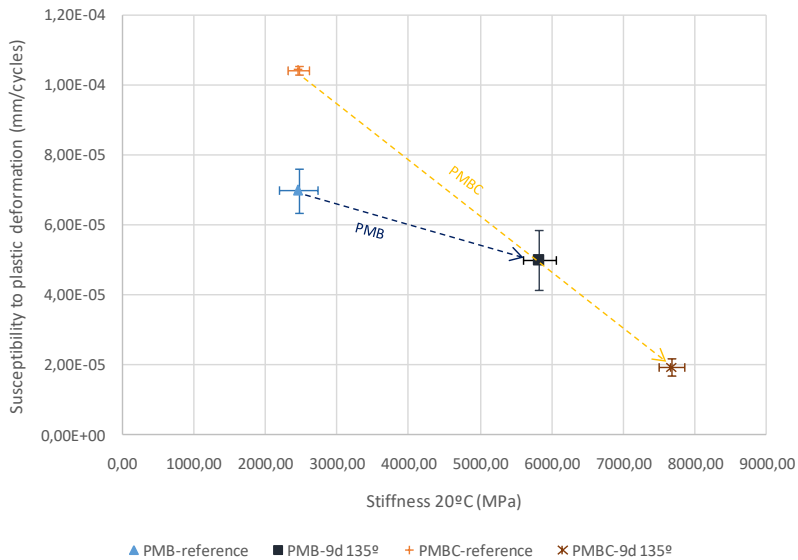


Figure 20 Influence of type of bitumen for ageing simulation on material stiffness and susceptibility to permanent deformations.

Figure 21 calculates the mean cycles to failure for both bitumen mixtures, as well as the link between the initial horizontal amplitude (measured at cycle 100 during the fatigue process in the UGR-FACT

test) and the ageing process. It has been discovered that stiffening reduces the initial horizontal deflections at the layer's bottom, resulting in a higher number of failure cycles for both mixtures. Nonetheless, despite the fact that both bitumens resulted in similar initial material behavior (the reference cases, without thermal conditioning), the values following the ageing process varied depending on the bitumen type (Moreno-Navarro et al., 2015). The reduction in horizontal flexibility at the specimen bottom was around 63% for the mixture with PMBC binder, while it was close to 54% for the mixture with PMB binder. These variations were lower than those measured in the previous parameters, as can be shown (tensile stiffness modulus and susceptibility to deformations). However, because the variations in cycles to failure were more than double in the case of the rubberized binder compared to the case of PMB, this variation has a greater impact on material durability, indicating once again the impact of binder type on the mixture's sensitivity to ageing.

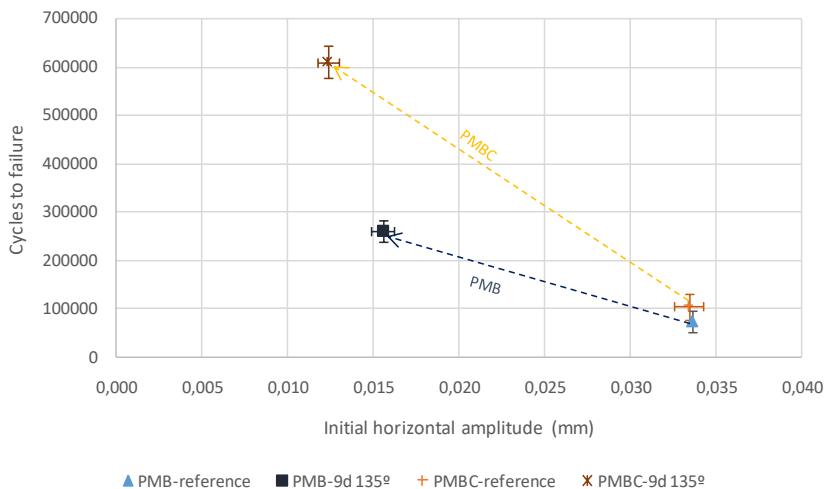


Figure 21 Results measured in the UGR-FACT for the mixtures with different type of bitumen.

Figure 22 illustrates the link between variations in cycle number and plastic deformations at the end of the test for a more in-depth investigation of the influence of binder on long-term performance of the mixture. Despite the fact that the aged mixture with rubber had a higher number of cycles to failure, it must be noted that this mixture also had a higher reduction in final plastic deformation, indicating a more brittle behavior than the same aged mixture with PMB, which could lead to premature failure under in-service conditions requiring deformation of this materials. Moreover, In reality, it was discovered that the rubberized mixture initially had a higher capacity to adapt deformation (ductility) during the fatigue process, but that this capacity was lost as the specimens aged, highlighting the critical importance of bitumen type on the ageing process and the importance of evaluating this phenomenon in the laboratory, rather than just the initial properties of the mixtures.

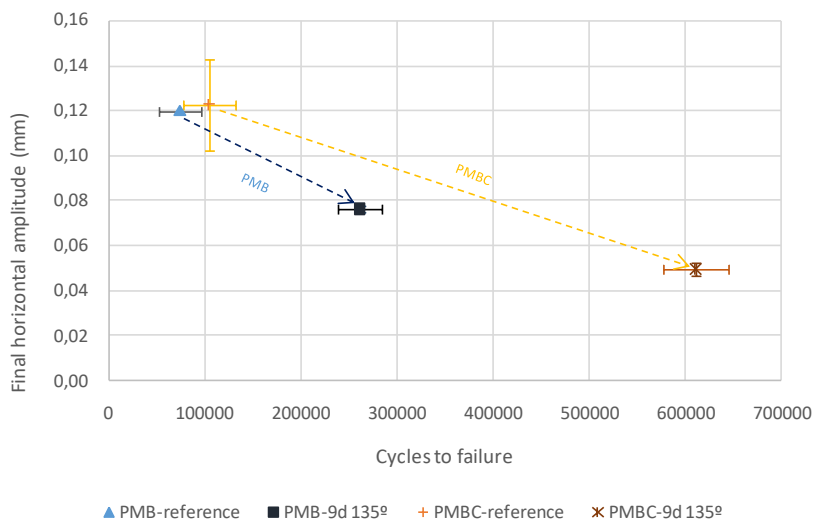


Figure 22 Results of durability and capacity to deform of the mixtures after ageing with different type of bitumen.

5.2.2 Effect of percentage of bitumen on the ageing process

Following figure 23 shows the influence of bitumen percentage on the change in stiffness and susceptibility to deformation for asphalt mixtures aged for 9 days at 135°C (long-term ageing) with 4.75% and 5.5% bitumen, respectively (both cases with PMB type). When looking at the effect of bitumen dosage on ageing, it was observed that a higher proportion of bitumen resulted in a greater rise in material stiffness (around 200% against the 130% of the mixture with lower bitumen content). This could be related to the fact that the bitumen is the component of an asphalt mixture most vulnerable to ageing, so utilizing a higher dose of this component could result in greater variances in long-term material performance. Even though, the mixture with a higher bitumen content restored a higher capacity to deform (even after ageing) than the other case with a lower dosage, despite the fact that the trend to change the susceptibility to deform after ageing was very similar in both cases, regardless of the bitumen content.

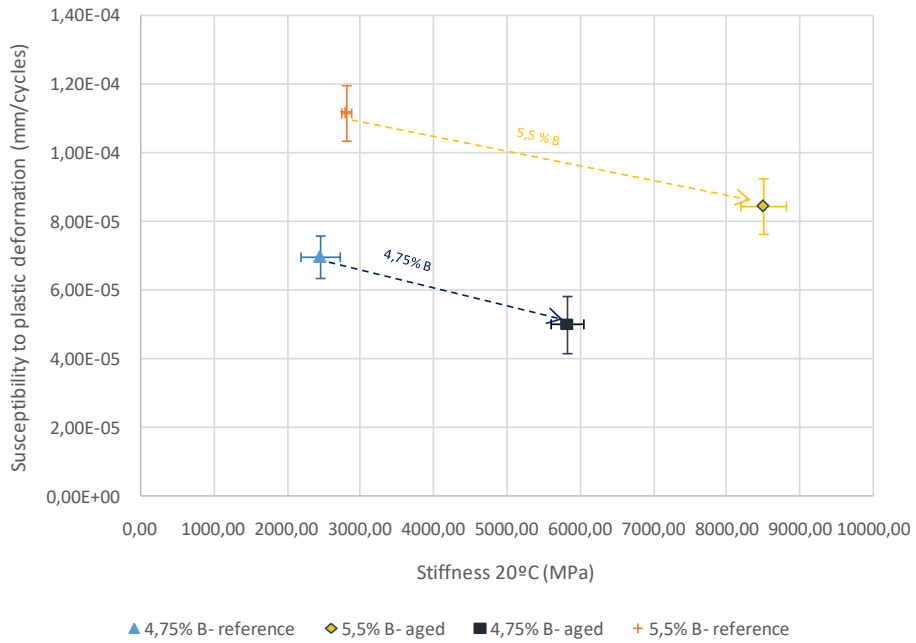


Figure 23 Influence of percentage of bitumen for ageing simulation on material stiffness and susceptibility to permanent deformations.

Figure 24, which shows changes in initial horizontal amplitude (deflexion) at specimen bottom and cycles to failure based on bitumen content, shows that the mixture with higher binder dosage caused a slight increase in variations due to the ageing process, reducing horizontal flexibility by around 70% when using 5.5% bitumen, compared to near 54% when using 4.75% bitumen. Nonetheless, as previously stated, these differences resulted in significant changes in the number of cycles to failure owing to ageing: roughly 250% for the 4.75% B combination and close to 300% for the 5.5% B example, with the latter retaining higher durability even after ageing. As a result, the bitumen content has a significant impact on the mixture performance, not only for the initial properties of the original material, but also for aged mixtures due to varying levels of property modification over time, making it critical to consider this aspect during material design and

manufacturing.

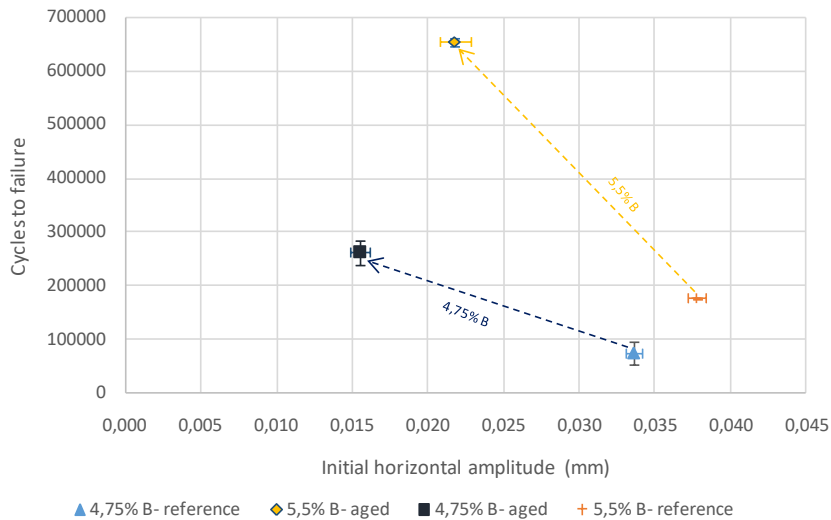


Figure 24 Results measured in the UGR-FACT for the mixtures with different percentage of bitumen.

Figure 25 demonstrates that, similar to the results obtained in the prior section when examining the effect of bitumen type on material ductility, material stiffening resulted in a decrease in the ability to deform without breaking, regardless of the binder amount. Even after ageing the specimens with higher bitumen content, it was observed that the usage of higher bitumen content allowed for greater ability to deform before failing at the end of the test, compared to 4.75% bitumen. As a result, higher bitumen content, despite causing higher variations in mixture properties and stronger ageing due to the higher quantity of bitumen, could provide a material with greater deformability (both at the start of the tests, as shown in Figure 6, and at the end of the tests), as well as greater crack resistance. In comparison to mixtures with lower bitumen content, this could be true both at the start and during the ageing process, resulting in a more ductile material during the long-term service life of the mixture.

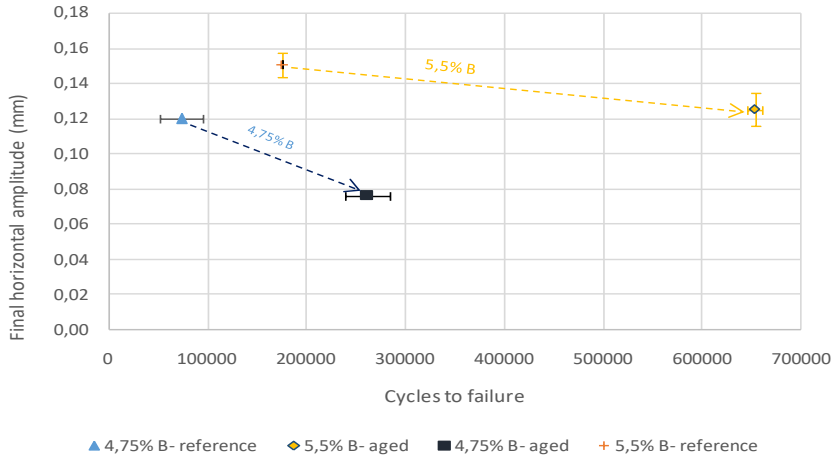


Figure 25 Results of durability and capacity to deform of the mixtures after ageing with different percentage of bitumen.

5.2.3 Impact of percentage of filler on the ageing process

Figure 26 represents the effect of filler content on ageing stiffness variations in a mixture, as well as measuring variations in sensitivity to plastic deformations at the start of the fatigue phase. According to the findings, this parameter (filler content) could have a significant impact on the mixtures' resistance to ageing, as varied levels of variation were seen depending on the filler quantity (rest of designing parameter being constant). The mixture with 6% filler had a stiffness increase of approximately 130%, while the mixture with 4% had a stiffness increase of more than 700%, which was a greater variation than the prior components studied (type and quantity of binder). This could be due to the fact that the lower the filler percentage, the more susceptible the asphalt mastic is to ageing, whereas a larger filler/bitumen ratio could allow for greater long-term deterioration resistance.

Furthermore, the material's susceptibility to deformation is reduced by almost 28% in a mixture with 6% filler, and by around 75% in a mixture with 4% filler, indicating higher variation in material

deformations (stronger ageing impact) due to an asphalt mastic with lower filler, and thus more bitumen available to be aged. This could necessitate a careful design and manufacturing procedure for asphalt mixtures that takes this issue into account, as well as a long-term examination of the ageing phenomenon.

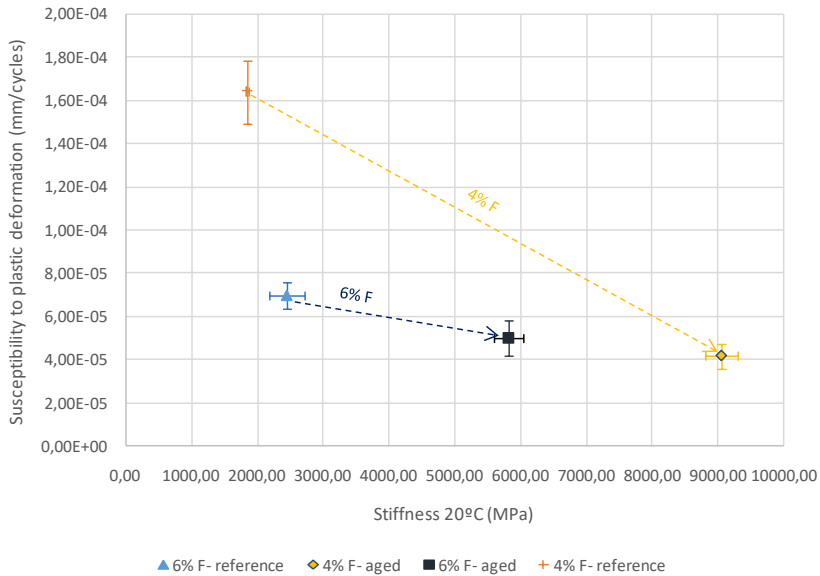


Figure 26 Influence of percentage of filler for ageing simulation on material stiffness and susceptibility to permanent deformations.

Figure 27 compares the difference between the initial horizontal displacements (at cycle 100) and the mean values of the number of cycles to failure in mixtures with 4% and 6% of filler. The higher the proportion of filler in the total weight of the mixture, the lower the material flexibility, which responds to a more rigid mixture, according to the results. Nonetheless, as seen in the previous results, Figure 10 shows that using 4% filler resulted in a 350% increase in the number of cycles, while using 6% filler resulted in a 250% increase in the number of cycles, indicating that using higher filler dosage results in lower changes.

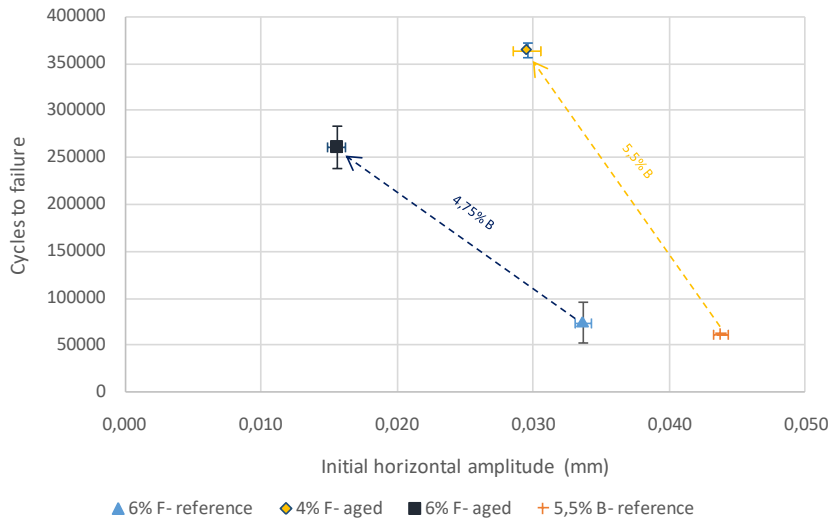


Figure 27 Results measured in the UGR-FACT for the mixtures with different percentage of filler.

Figure 28 proves that the mixture with 6% filler resulted in a more rigid material with lower capacity to deform during the fatigue process, and that this fact remained even after the ageing process, despite the mixture with lower filler showing higher susceptibility to change due to the ageing process (reduction close to 30% against the limited 20% for the case with higher filler content).

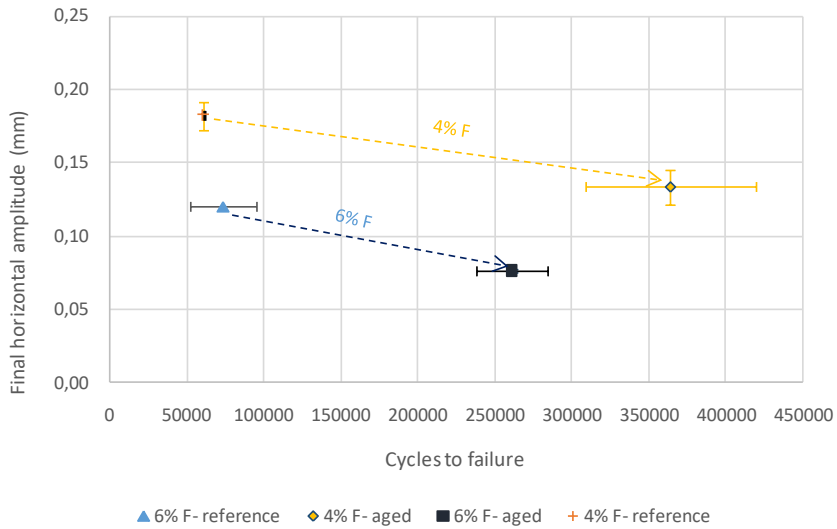


Figure 28 Results of durability and capacity to deform of the mixtures after ageing with different percentage of filler.

5.2.4 Influence of percentage of sand on the ageing process

Figure 29 show the effect of sand percentage (and therefore aggregate gradation) on material ageing by illustrating changes in stiffness and susceptibility to early deformations due to ageing after utilizing 20% and 35% of sand, respectively. The increased sand content resulted in a stiffer material due to the use of a denser gradation (and hence a reduction in air void content), which was exacerbated after the ageing process. Nonetheless, when the ageing rate was calculated using the percentual variation in stiffness, it was discovered that both cases (with 20% and 35% sand) resulted in a variation of around 130% due to ageing, but the mixture with higher sand dosage had a greater reduction in deformation susceptibility (around 70% against 29% of the mixture with lower sand %).

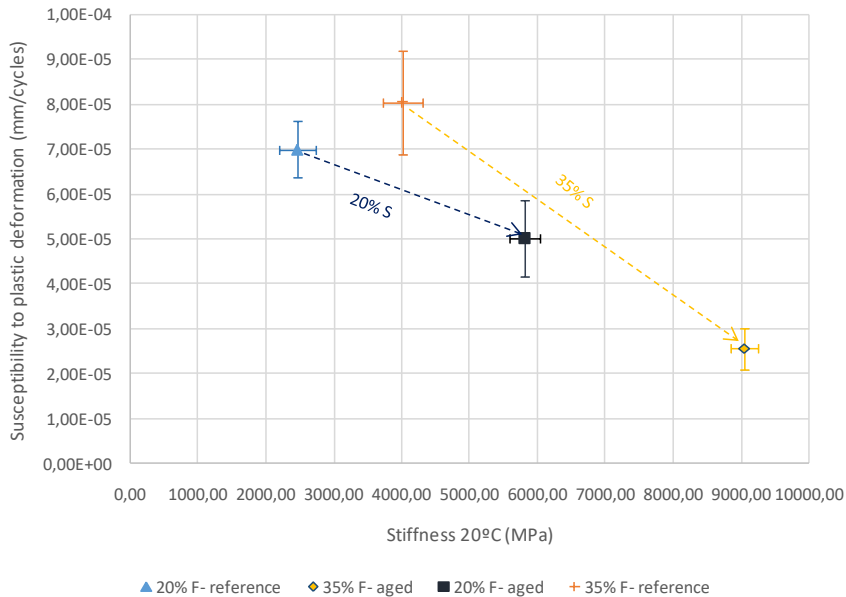


Figure 29 Influence of percentage of sand for ageing simulation on material stiffness and susceptibility to permanent deformations.

Figure 30 show the effect of sand dosage on ageing-related changes in material durability and initial flexibility. In comparison to the instance with 20% of sand, the combination with 35% of sand had lower initial horizontal deformations due to a stiffer reaction, which resulted in a significant increase in the number of cycles to failure, which remained after the ageing process. Also, the percentage of voids was reduced from around 18% in the control mixture (with 20% sand) to close to 6 percent in the case with higher sand dosage, which must be related to a lower air void content, as indicated by other authors (Valdés et al., 2014; Harrigan 2007), because the percentage of voids was reduced from around 18% in the control mixture (with 20% sand) to close to 6%.

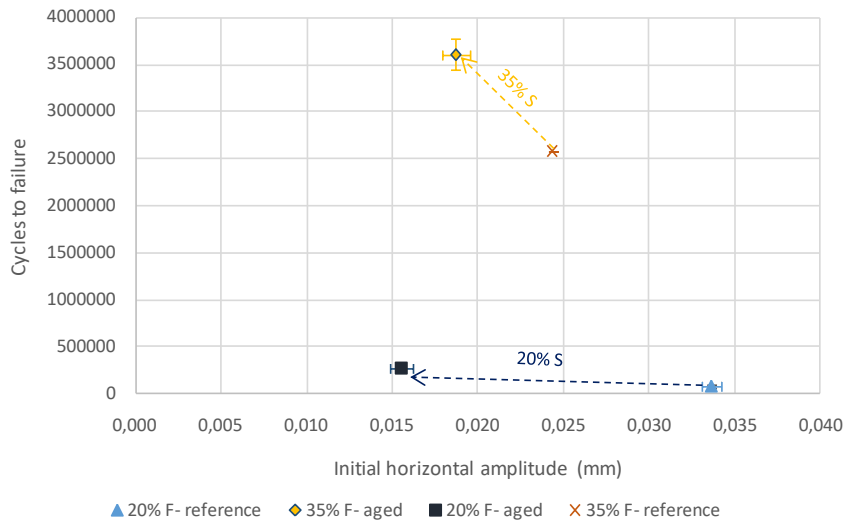


Figure 30 Results measured in the UGR-FACT for the mixtures with different percentage of sand.

According to Figure 31, these results could indicate that using a dense graded mineral skeleton could be crucial to reducing material ageing while also providing increased durability and ductility. The changes in material deformation at the end of the fatigue tests in relation to the variation in the number of cycles due to the ageing process are shown in this graph for both examples with 20% and 35% sand. The results show that the case with higher sand dosage had a lower ageing-related reduction in the capacity of the material to deform before failing (around 7%), compared to the reference case with 20% sand (with a variation close to 20%), resulting in a material with higher ductility even after the ageing process.

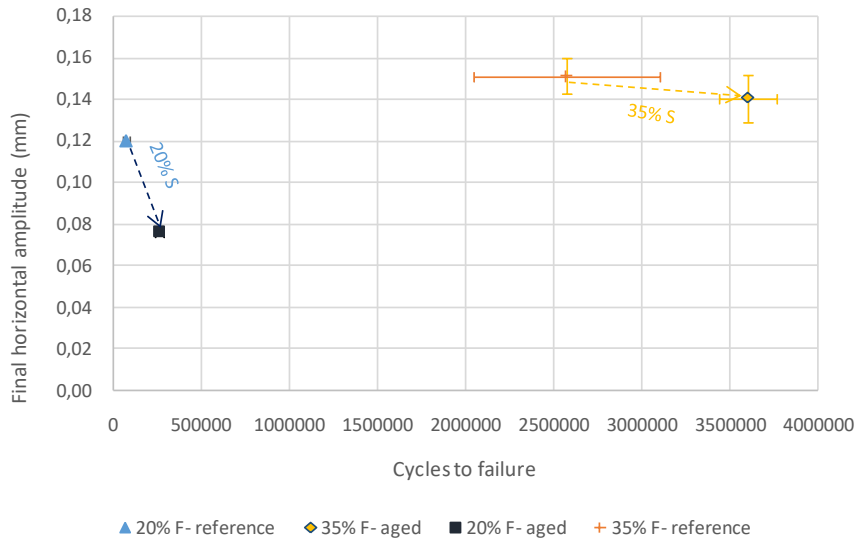


Figure 31 Results of durability and capacity to deform of the mixtures after ageing with different percentage of sand.

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5.2.5 Weight of factors on ageing process

Figures 32 and 33 show the perceptual changes in various properties due to the ageing process for the different mixtures studied. In comparison with the reference PMB 4.75%B6%F20%S (with 4.75% of PMB bitumen, 6% cement and 20% sand), it can be seen the impact when increasing binder content to 5.5 percent, reducing filler dosage to 4%, increasing sand up to 35%, and using a PMBC instead of the PMB, with the goal of evaluating the impact of varying the designing factors on the ageing rate of the control mixture (that with 4.75% PMB bitumen, 6% filler, and 20% sand).

Since reduced air void content was measured, the results show that raising the sand dose resulted in the lowest percentages of variance, implying that this approach may be appropriate to reduce material ageing. Also, it was discovered that varying this element (sand dosage)

resulted in comparable rates of change to those measured by the control mixture, implying that this designing factor has a lower impact than other factors such as bitumen type and filler dosage. These last two parameters, which included decreasing the filler content (and thus the amount of asphalt mastic in the combination) and utilizing rubberized bitumen, had a negative impact because the change rate due to ageing was larger in contrast to the control mixture. As a result, determining these elements has a substantial impact on the manufacturing of asphalt mixtures, as the ageing process can result in a significant drop in material durability.

In terms of bitumen content, it can be said that this aspect has a middle impact between sand dosage (the least influential according to these results) and filler dosage and bitumen type. The higher the proportion of bitumen, the greater the change in material qualities due to the greater quantity of binder available to be modified during the ageing process, according to the findings. However, as shown in Figures 8 and 9, despite increasing the ageing rate by employing a greater bitumen dose, the qualities of the aged materials are still superior to those of a mixture with a lower bitumen component, at least according to the parameters tested in this study.

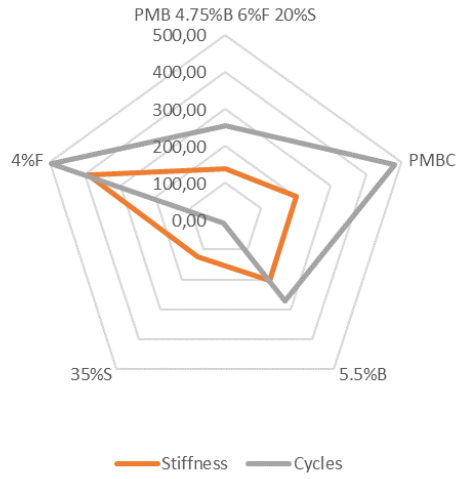


Figure 32 Weight of factors on variations of stiffness and cycles to failure after ageing process.

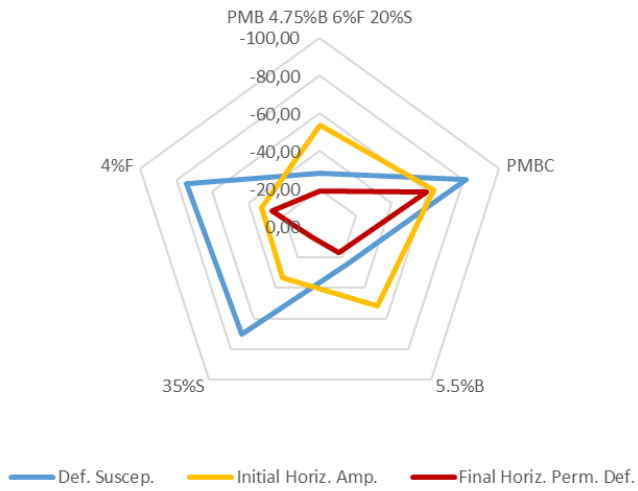


Figure 33 Impact of designing parameters on ageing rate.

6. Conclusions

The Thesis focuses on the ageing phenomenon in asphalt mixtures by examining how fundamental long-term mechanical properties (stiffness and fatigue cracking resistance) change due to such phenomenon, assessing the impact of mixture design factors. For this purpose, the thesis assessed the impact of various ageing conditions (temperature, time and pressure) on gap-graded mixtures with different type of binder. In addition, the impact of design factors (type and quantity of bitumen, filler dosage, and the impact of mineral skeleton) on the ageing rate of asphalt mixtures was examined when such parameters were varied within permissible ranges for the selected mixture due to possible manufacturing process modifications. The following conclusions can be taken from the results obtained:

- When the temperature of ageing simulation in laboratory was increased, higher stiffness and lower deformations (permanent and elastic) was obtained, regardless of the type of binder studied. According to the findings, temperatures around 135°C should be used at laboratory level when studying the resistance to ageing on different types of asphalt materials.
- The duration of the ageing process in laboratory is also an important variable. Little effects were observed in the mechanical properties of bituminous mixtures when exposed to short term ageing conditions (4 hours of conditioning). On the contrary, it was revealed that increasing laboratory ageing conditionings for longer periods (9 days), results in more significant changes of the mechanical properties of the materials studied (which would help to compare the performance between

two materials).

- The application of pressure during the laboratory simulation resulted in a reduction in material stiffness while increasing its sensitivity to deformation, in contrasting to the temperature and exposure parameter during the ageing process for surface course asphalt mixtures. This could be due to the mixture degradation as a result of the pressure process, resulting in the opposite result as seen with the preceding ageing parameters.
- In comparison to temperature and pressure, the results showed that time had the greatest impact on reproducing laboratory ageing. Based on the results obtained, 9 days at 135°C were selected as the laboratory ageing conditions to conduct the second part of the thesis.
- The type of bitumen and binder content influence the susceptibility of mixture ageing. The results obtained demonstrated that some binders offer higher hardening rates (moving from ductile to brittle cracking) than the others. In the same way, higher bitumen contents could result in materials with greater deformability and crack resistance, even after the ageing process.
- The less the filler content, the greater the changes in mechanical properties as a result of the ageing process. Thus, for the same binder content, asphalt mixtures with the lower filler content are more susceptible to this phenomenon. Similarly, the use of high sand contents allows for a reduction in the impact of ageing phenomenon. This meant that moving from an open/gap graded to a dense mixture improves material ageing resistance.

7. Future lines of investigation

From the research done in this Thesis, some remaining questions have been identified which can be further investigated in future research efforts:

- In order to reduce the effect of ageing of asphalt mixtures, it is possible to use others anti-ageing additives. Research on a variety of antioxidant additives is warranted in order to obtain a more effective and sustainable asphalt mixture that can perform equally well both at high and low temperatures.
- Also, it could be interesting to considerate other ageing factors like UVA rays on the change in mixture properties.
- Additional mixtures and binders extracted and recovered from aged mixtures (more than 60 months) should be analysed in order to get a wide variety of information on the influence of ageing on both mixture and binder properties and to build a large database.

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