

RESEARCH ARTICLE

Sequential effects of spent coffee grounds on soil physical properties

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Abstract

Spent coffee grounds are a bio-residue studied as soil organic amendment and it has been proven that it has short-term effects on soil physical properties. However, its sequential effects on the cultivation of clayey soils are little studied. Therefore, an in vitro experiment was carried out to evaluate the effect of increasing doses (1%, 2%, 2.5%, 5%, 7.5%, 10%, 12.5% and 15%) of spent coffee grounds on the physical properties of a clayey soil in the Spanish Mediterranean area which is rich in smectites. The addition of spent coffee grounds increased water retention at -33 and -1500 kPa proportionally to the added amounts, but the increase in the wilting point was much larger than the field water capacity, decreasing the plant available water content. A non-linear influence on the aggregate size is demonstrated. It increased total porosity and consequently reduced soil bulk density. This fact was reflected in the stereomicroscopy images where an increase in the pores analysed with image analysis was observed. Furthermore, SEM images corroborate that spent coffee grounds act intensely in the short-term due to the interaction between their particles and those of clay. The 5% dose acted as a threshold dose from which the greatest effects on soil physical properties occur. In general, the use of SCG as an organic amendment is a good sustainable solution because it supposes a reuse of this bio-residue (15 million tons per year), an increase in soil organic carbon (SCG contains $\approx 50\%$ carbon) and an improvement of the soil physical and chemical properties.

KEYWORDS

organic amendment, organo-mineral interaction, porosity, smectitic soil

Highlights

- Increased pore space is one of the most visible effects of spent coffee grounds (SCG)
- SCG linearly increases water retention and decreases available water content

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- In relation to the effects of SCG, the eight doses tested are reduced to low (<5%) and high (>5%)
- The results do not allow the establishment of an ideal dose of SCG
- The interaction between the SCG and clay particles seems key in its effects

1 | INTRODUCTION

Due to their high content of C organic amendments substantially influence soil physical properties (Ramzan et al., 2021). The influence on these properties depends in part on the type of amendment added. For example, Zhang, Wang, et al. (2022) added equal amounts of weathered coal, biochar and grass peat to a clay loam soil and they demonstrated that there is an increase in porosity and structural stability and a decrease in bulk density for the three amendments, but with different intensity according to the type of amendment added. Nevertheless, the effects of the amendments on the physical properties of the soil depend mainly on the characteristics of the soil, among which granulometry seems to be the most important (Gómez-Guerrero & Doane, 2018; Upadhyay & Raghubanshi, 2020). Thus, in the case of sandy soils, it inclines towards a greater state of aggregation, while clay soils have greater macroporosity and structural stability (Blanco-Canqui, 2017). In general, according to Kleber et al. (2021), most of the organic carbon in the biosphere takes place at solid earth interfaces with close proximity to some form of mineral material. In particular, soil clay minerals stabilize the organic matter in the soil through mineral-organic matter binding (Wattel-Koekkoek et al., 2001). Moreover, soils with a higher content of smectites, interact better with organic matter than illitic or kaolinitic soils (Six et al., 2002). Hence, the diverse effects on texturally different soils can be attributed to interactions with clay minerals.

Wang et al. (2021) showed that the effects of biochar on the physical properties of clayey soils rich in expandable clays (the cited authors include illites, vermiculites and smectites in this category) are closely related to the action time and the dose of biochar added. Regarding dose, there are many studies about the effect of the application rate of different organic amendments on the soil physical properties. Thus, Omondi et al. (2016) in a meta-analysis of data from the literature studied the effect of different doses of biochar: low (<20 t ha⁻¹), medium (21–40 t ha⁻¹), high (41–80 t ha⁻¹) and very high (>80 t ha⁻¹). They reported medium decrease in bulk density by 7% and medium increase in porosity, plant available water and saturated hydraulic conductivity, by 8.2%, 15.1% and 25% respectively. In another study, Aranyos et al. (2016)

studied in a long-term assay (in sandy soils) the effect of different composted sewage sludge doses (0, 9, 18 and 27 Mg ha⁻¹) on the bulk density. These authors observed a bulk density decreasing, but in the second year of the experiment, the positive effect of compost was observed only in the plots treated with the highest doses because of the quickly degradation of the organic matter. Hernández et al. (2015) also studied the effect of increasing doses (0, 150 and 450 t ha⁻¹) of sewage sludge compost on the water retention capacity and the stability of the aggregates. In this study, they observed how these properties increased linearly with the applied residue dose. In the same way, Yazdanpanah et al. (2016), studied the effect of the addition of 0, 10 and 30 Mg ha⁻¹ of alfalfa residues and municipal waste compost on the physical properties of a clay soil and a silty one. Moreover, the physicochemical properties, specifically the adsorption–desorption of nutrients, are affected by the doses of bio-products added to different types of soil. In this way, Peng et al. (2021) tested a range of doses (0.5% and 4%) of biochar modified by the adsorption of Fe/Al oxy-hydroxides (added to a calcareous crop soil). They found that 2% is the dose that most decreased the leaching of P and at the same time maintained an appropriate level of bioavailable P. Peng et al. (2022) also studied the effects of these biochars modified with oxy-hydroxides on P dynamics in a lateritic soil. These last authors showed that these are not indirect effects through microorganisms but rather abiotic processes. Zhang, Yin, et al. (2022) studied the curious effect of different doses of bio-products with the simultaneous addition of two of them. They found that low doses of biogas slurry without addition of 2% biochar decreased P leaching, while high doses of biogas slurry with simultaneous addition of 2% biochar were also favourable for P retention in the soil. In short, it is observed how the application rate is one of the most important factors and has to be taken into account in the study of the effect of organic amendments on soil physical and other properties.

Spent coffee grounds (SCG) are a waste generated in large quantities around the world (15 million tons annually, Kamil et al. 2019). Stylianou et al. (2018) review the literature on the environmental benefits of the reuse of SCG including its employment as organic soil amendments. These authors emphasize that despite the phytotoxic effects of SCG, its proven effects on the physical and

chemical fertility of soils, as well as its influence on the soil microbiota, make this form of SCG reuse advisable. Previously, our research group has evaluated the effect that the addition of this residue has on Mediterranean agricultural soils in relation to its physical, chemical, physicochemical and biological properties (Cervera-Mata et al., 2018, 2021, 2022; Cervera-Mata, Martín-García, et al., 2019; Comino et al., 2020; Vela-Cano et al., 2019). In summary, the main results obtained by the aforementioned authors were: (a) SCGs modify the C cycle by increasing its recalcitrant forms and increasing the content of humic and fulvic acids; (b) SCGs increase the contents of total N and the available forms of K and P; (c) they increase water retention at -33 and -1500 kPa, decrease bulk density, modify size and shape of soil aggregates and, increase total porosity and structural stability; (d) as negative effects of the addition of SCGs, there is a decreasing plant growth and a decrease in plant available water. All these effects are due to: (i) the composition of the SCGs, rich in C, N and other nutrients and for its content in phytotoxic compounds (mainly polyphenols); (ii) the increase in all hydrophobicity parameters; (iii) the stimulation of microbial activity (bacteria and fungi) even modifying the bacterial community structure; (iv) the incorporation of SCG particles in the soil matrix and their interaction with mineral particles, mainly with those of the clay fraction. Globally, these authors point out that the addition of SCG to the soil improves the soil quality.

There is little research on the effects of SCG dosage on soil properties. Turek et al. (2019) studied the sequential effect of increasing doses of SCG (5%, 10%, 15% and 20%), but did so on a sandy loam soil from Brazil. These authors found a 31% increase in water retention capacity with the addition of SCG, although it decreases 15 and 20% between the doses which they associate with pore geometry, which has been moulded by the SCG, modifying its shape, tortuosity, continuity and conductivity. Another test carried out on the soil physical properties was that of Kasongo et al. (2011), although these authors use other coffee residues (mixture of pulp and husk), observing how their addition increased water retention at levels of 53%–60%. These authors also obtained an increase in plant available water content as the addition of coffee residues increases by up to 15%. Hardgrove and Livesley (2016) also found an increase in water retention when adding fresh SCG in sandy and silty soils. In addition, they also reported an increase in the gravimetric moisture content in the soil when SCG doses greater than 10% were added. On the other hand, according to Sena da Fonseca et al. (2014), a study on the use of SCG as an additive in construction materials, showed how SCG increased water absorption,

bulk porosity and decreased the bulk density of samples added to SCG.

Within Europe, the Mediterranean region occupies 11% of the territory and is currently the most susceptible area to soil degradation and desertification; processes that have been aggravated by climate change (Ferreira et al., 2022). According to these authors, among the main processes of soil degradation are those of a physical nature: erosion, sealing and compaction, which are due, among others, to the low content of soil organic matter ($<2\%$ on average). Cerdà et al. (2021) confirm this problem in the European Mediterranean region and report that between 2007 and 2020 (13 years) the soil bulk density of some soils increased from 1.05 to 1.33 g cm^{-3} . In this Mediterranean region, clay soils rich in smectites are abundant (Sandler et al., 2015); The presence of expansive clay minerals causes a great capacity for expansion and contraction, so they become very hard during the dry season and very sticky when wet (Brierley et al., 2011). Poor soil structure prevents these soils from agricultural as well as engineering use and makes management difficult (Brierley et al., 2011; Dinka et al., 2013; Millán et al., 2012). Despite these shortcomings, soils rich in smectites are widely utilized for crop production in the world.

Taking all these considerations into account, the objective of this work is to investigate the sequential effects of increasing doses of SCG on the physical properties of an agricultural Mediterranean clay soil rich in smectites. The following physical parameters of the soil will be studied: bulk density (BD), water retention at -33 kPa (field capacity) water retention at -1500 kPa (permanent wilting point), plant-available water content (AW), macroaggregates ($>1000 \mu\text{m}$), mesoaggregates ($1000\text{--}250 \mu\text{m}$) and microaggregates ($<250 \mu\text{m}$), total porosity and soil microstructure. The study was carried out under 'in vitro' conditions in a climatic chamber due to two fundamental reasons: (1) it allows testing very high doses (up to 15%) which are not feasible in field experiments. In addition, the use of high doses allows the establishment of the effects of the amendment on the physical properties of soils that are not evident in low doses; (2) Investigating a very wide range of additions, allows us to determine if there is an ideal dose of SCG from the point of view of physical properties. The interest of this research lies in several aspects: (i) the abundance of SCG as residue, the scarcity of organic matter in Mediterranean soils, (ii) the abundance of clay soils for cultivation in that region and their physical degradation, (iii) the still scant bibliography on the effects of the SCG on the physical properties of soils and (iv) the sequential effect of SCG on these properties in clay soils has not been studied.

2 | MATERIALS AND METHODS

The soil was sampled from the arable layer (<20 cm) of a soil from the Vega de Granada (Spain) classified as a Calcic Cambisol (Aric, Ochric, Vertic) (IUSS Working group WRB, 2014), belonging to the Mediterranean area. Its main physical, chemical and physicochemical characteristics are shown in Table 1 (Comino et al., 2020). Regarding the characteristics of the soil, the abundance of carbonates (39%) and clay (58%) stands out. The clay fraction is composed mainly of illite (29%), smectite (17%) and calcite (14%) and other minerals such as paragonite, interstratified, phyllosilicates >1.4 nm, kaolinite, quartz, feldspars and dolomite (40% in total). The abundance and mineralogy of the clay are what determine the vertic nature of this soil.

The SCG were collected from the cafeteria of the Faculty of Pharmacy at the University of Granada. The coffee used was Arabica 100%. They were later spread, air-dried and stored in a dry place. Their main characteristics are shown in Table 1 (Cervera-Mata et al., 2021). SCG are characterized by their high content of C (48 g kg⁻¹) and K (3426 mg kg⁻¹). In addition, they are acidic and saline waste.

The lettuce used in the cultivation corresponds to *Lactuca sativa* var. *longifolia*. The lettuces used in the cultivation were 30 days old and were supplied by the company Saliplant S.L. (Granada). Seeds were not used but lettuce seedlings.

TABLE 1 Properties of the materials used in the assay

Soil properties	SCG properties		
CF (g kg ⁻¹)	20.0		
Clay (g kg ⁻¹)	580.0	pH	5.4
Silt (g kg ⁻¹)	299.0	EC ₂₅ (dS m ⁻¹)	9.0
Sand (g kg ⁻¹)	121.0	C (g kg ⁻¹)	480.0
pH	8.2	N (g kg ⁻¹)	22.90
EC ₂₅ (dS m ⁻¹)	1.3	C/N	21
OC (g kg ⁻¹)	13.6	P av. (mg kg ⁻¹)	405
N (g kg ⁻¹)	1.1	K av. (mg kg ⁻¹)	3426
C/N	13	WSC (mg kg ⁻¹)	25.92
CO ₃ Ca eq. (g kg ⁻¹)	390.0	Ash (g kg ⁻¹)	14.5
BD (g cm ⁻³)	1.20	Volatile matter (g kg ⁻¹)	845.5
W ₃₃ (g kg ⁻¹)	264.0		
W ₁₅₀₀ (g kg ⁻¹)	156.0	Fixed C (g kg ⁻¹)	139.7
AW (mm cm ⁻¹)	1.30		

Note: CF, coarse fragment; EC₂₅, electrical conductivity measured at 25°C; OC, organic carbon; BD, bulk density; W₃₃, water retention at -33 kPa; W₁₅₀₀, water retention at -1500 kPa; Water retention data (W₃₃ and W₁₅₀₀) are shown in weigh/weight; AW, plant available water content; av, available; WSC, water soluble carbon.

The experiment assay was carried out with eight replications and eight increasing doses of SCG (1%, 2%, 2.5%, 5%, 7.5%, 10%, 12.5% and 15%). Eight samples without the addition of SCG were used as controls. The SCG in different doses were mixed with the soil (<5 mm) until obtaining a soil-SCG mixture of 400 g. Subsequently, the mixtures were transferred to 300 ml pots (top diameter 11 cm, base diameter 7.5 cm, height 8.5 cm, number of holes in the base 19), closed at the base with a fibreglass mesh to avoid the loss of fine particles. Subsequently, 30-day-old lettuces were transplanted. All samples were incubated in a climatic chamber under controlled conditions of atmospheric humidity (50%-day, 60%-night), temperature (22°C-day, 18°C-night) and 12/12 h photoperiod. The soil moisture control of the samples was done by weighing, keeping the moisture between the field capacity and the permanent wilting point. Seventy-two pots were used. The trial was conducted in January-February 2019.

After 40 days, the soil samples were spread out and air dried. Later, they were stored in a dry place. Lettuce samples were sampled and the results corresponding to the analysed properties have been published by Cervera-Mata, Navarro-Alarcón, et al. (2019).

The Methods of Soil Analysis of the American Society of Agronomy and Soil Science Society of America (Soil Survey Staff, 2014) were followed. Soil pH was measured in 1:2.5 (w/w) soil-water suspensions. Electrical conductivity at 25°C (EC₂₅) was measured in the extract of the 1:5 (w/w) water suspension. Available phosphorus was determined by the Olsen Watanabe's method with a Helios alpha spectrophotometer (Thermo Fisher Scientific Inc., Waltham, MA, USA). Available potassium was extracted with 1 N ammonium acetate (pH = 7), and determined with a PFP7 flame photometer (Jenway, Staffordshire, UK). Organic carbon (OC) was determined by hot wet oxidation (Tyurin's method). Total nitrogen was determined by the Kjeldhal method.

Granulometry was determined by the Robbinson pipette method. Bulk density (BD) was determined by measuring the weight of the dry air sample contained in a cylinder of 223.4 cm³ (Bourger's method). Water retention at -33 kPa (field capacity) and -1500 kPa (permanent wilting point), W₃₃ and W₁₅₀₀, respectively, was determined using the Richards membrane method, and plant-available water content (AW) was obtained from the difference between water retention at -33 and -1500 kPa, employing the Cm coefficient. The classification of aggregates by size was performed with 250 and 1000 μm sieves, differentiating between macroaggregates (>1000 μm), mesoaggregates (1000-250 μm) and microaggregates (<250 μm). Total porosity was estimated from the particle and bulk density, and macroporosity from total porosity less that of microporosity, the latter was measured as the water content at field capacity (Sánchez-Marañón et al., 2002).

For the study of soil microstructure, an integrated microscopy chain by reflection was applied, which consisted of stereomicroscopy and scanning electron microscopy (SEM). Stereomicroscopy images (Olympus SZX12) were captured in air-dried soil aggregates (approx. 2 mm). An image analysis was performed using Image J 1.46r software on a macroaggregate fraction (1–5 mm). To quantify the porosity, pores were isolated from the background following the method of Calero et al. (2009). SEM images were captured in soil aggregates (1–5 mm) fixed with carbon double-sided adhesive tape, metallized with carbon and analysed with a variable pressure and high-resolution scanning electron microscope (HITACHI S-510), equipped with an energy dispersive X-ray (EDX) elemental analyser. For the observation of bacteria, the samples were first fixed with 2.5% glutaraldehyde in 0.1 M cacodylate buffer, and subsequently fixed with 1% osmium tetroxide, dehydrated with alcohol, dried using the critical point method and finally they were coated with carbon (Kuo, 2007).

Means between groups were compared by variance analysis (ANOVA) at $p < 0.05$ confidence level (Tukey test) with SPSS 22.0 for Windows (IBM SPSS Inc., New York, USA). Linear correlations were evaluated by computing the correlation coefficient by Pearson. Principal component analysis (PCA) was performed for clustering samples and relating them to the different parameters used in the study. This statistical treatment was performed in Origin b9.5.5409 (OriginLab Corporation, Northampton, MA, USA).

3 | RESULTS

As shown in Table 2, the sequential addition of SCG significantly ($p < 0.05$) modified the soil properties, as a function of the starting amounts of SCG (Table 1). Thus,

the SCG significantly decreased the soil pH in proportion to the amounts added. With respect to EC_{25} , they increased the salinity of the soil, reaching a dose of 15% at 1.18 dS m^{-1} . A very positive aspect of the addition of this organic residue is the increase in OC in the soil, obtaining an adjustment in the linear regression SCG dose vs OC with an r of 0.9622. In the same way, SCG increased the N content in soils. Regarding assimilable P, SCG did not significantly affect ($p > 0.05$) this property, although they did significantly ($p < 0.05$) increase the K content in soils, because this residue is very rich in K (3426 ppm, Table 1).

As can be seen in Figure 1a, the addition of SCG significantly decreased ($p < 0.05$) the bulk density (BD) and particle density (PD) of the mixtures progressively with the amounts added with respect to the control sample, except for the 2.5% SCG dose in the case of BD. Thus, for example, the addition of 5% produced a decrease of 36%, the addition of 10% a reduction of 39% and the addition of 15% a reduction of 48% of the BD, with respect to the control sample. It is observed how the significant effect of the addition of SCG is seen from the dose of 1%, requiring very small doses to reduce the density of the soils. Figure 2a corresponds to SEM images of the surface of an aggregate with 10% SCG. This image demonstrates the proliferation of bacteria in the vicinity of the SCG particles (at the bottom of the image). This bacterial activity is not observed in soil aggregates without SCG addition.

Regarding the total porosity (Figure 1b), it is observed how the addition of SCG significantly ($p < 0.05$) increases these percentages, reaching a maximum total porosity with the dose of 12.5% SCG (65%). This increase is inversely related to the BD. Regarding macroporosity, there is a significant increase ($p < 0.05$) in all doses compared to the control sample, but there are no significant differences between them, reaching a mean macroporosity of

TABLE 2 Soil chemical and physicochemical properties

Sample	pH	EC_{25} (dS m^{-1})	OC (g kg^{-1})	Total N (g kg^{-1})	C/N	P av. (mg kg^{-1})	K av. (mg kg^{-1})
Control	8.3 ± 0.0 f	0.86 ± 0.10 a	17.4 ± 0.17 a	1.42 ± 0.031 a	13 ± 2 ab	121 ± 6 a	491 ± 15 a
1% SCG	8.3 ± 0.0 ef	0.94 ± 0.08 ab	21.4 ± 0.10 ab	1.94 ± 0.006 ab	11 ± 1 a	118 ± 2 a	557 ± 8 b
2% SCG	8.2 ± 0.0 ef	1.03 ± 0.04 ab	27.0 ± 0.35 b	2.03 ± 0.033 ab	13 ± 2 ab	118 ± 1 a	585 ± 8 b
2.5% SCG	8.2 ± 0.1 de	0.96 ± 0.06 abc	27.7 ± 0.11 b	2.49 ± 0.016 abc	11 ± 1 a	122 ± 12 a	601 ± 0 b
5% SCG	8.1 ± 0.1 cd	1.13 ± 0.13 bcd	40.9 ± 0.36 c	2.54 ± 0.020 abc	16 ± 2 abc	121 ± 7 a	662 ± 8 c
7.5% SCG	8.0 ± 0.0 bc	1.29 ± 0.01 d	55.9 ± 0.38 d	3.03 ± 0.002 bc	18 ± 1 c	117 ± 6 a	713 ± 10 d
10% SCG	7.9 ± 0.0 b	1.31 ± 0.10 d	50.9 ± 0.37 d	3.48 ± 0.020 cd	15 ± 2 abc	123 ± 2 a	768 ± 12 e
12.5% SCG	7.8 ± 0.0 a	1.30 ± 0.04 d	75.2 ± 0.24 e	4.67 ± 0.129 de	17 ± 4 bc	122 ± 6 a	780 ± 20 e
15% SCG	7.8 ± 0.1 a	1.18 ± 0.06 cd	78.6 ± 0.33 e	5.32 ± 0.050 e	15 ± 1 abc	119 ± 21 a	788 ± 36 e

Note: SCG, spent coffee grounds; EC_{25} , electrical conductivity measured at 25°C ; OC, organic carbon; av, available. Different letters denote statistically significant differences ($p < 0.05$).

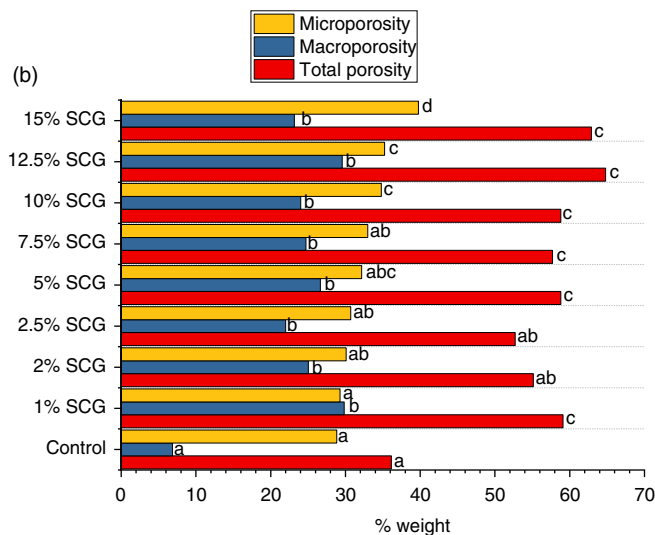
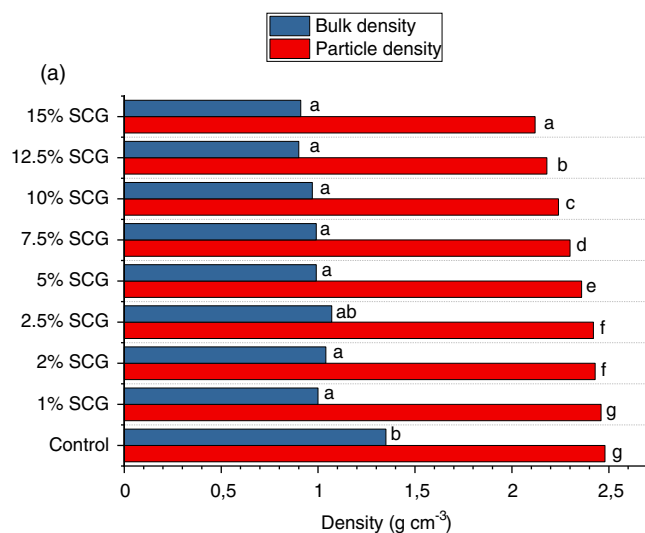


FIGURE 1 (a) Bulk and particle density. (b) Microporosity, macroporosity and total porosity. Different letters indicate statistically significant differences ($p < 0.05$).

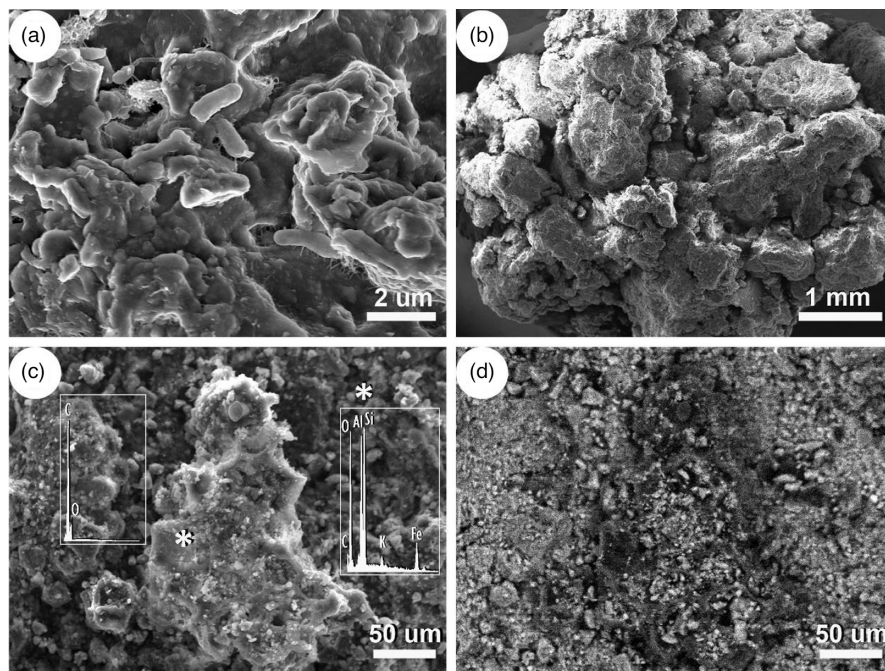


FIGURE 2 Scanning electron microscopy images. (a) 10%SCG, secondary electrons. (b) 2.5% SCG, secondary electrons. (c) 7.5%SCG, secondary electrons with EDX. (d) 7.5%SCG, backscattered electrons. Asterisks show the exact point in the sample where EDX analysis were performed.

26%. Microporosity also increased significantly ($p < 0.05$) with the addition of SCG but from high amounts (10%).

Regarding the percentage of macro, meso and microaggregates, no significant differences were observed between the different treatments (Figure 3), however, we can highlight some trends. The sample with an addition of 10% SCG is the one with the highest percentage of microaggregates and mesoaggregates, and therefore it was the sample with the least amount of macroaggregates. It seems that as SCG are added, there is an increase in micro and mesoaggregates and a decrease in macroaggregates until reaching the 10% SCG dose, where a threshold occurs. From here, there is a decrease in micro and mesoaggregates and a slight increase in macroaggregates.

Regarding the water retention capacity (Figure 4), a significant increase ($p < 0.05$) is observed both at -33 kPa (W_{33}) and at -1500 kPa (W_{1500}). The addition of 15% SCG increases W_{33} by 38% and W_{1500} by 129%, which indicates a greater relative increase at -1500 kPa than at -33 kPa. This results in a significant decrease in plant available water content as the SCG dose increases (for 0%, 1%, 2%, 2.5%, 5%, 7.5%, 10%, 12.5% and 15%, this corresponds to a quantity of usable water of 1.70%, 1.34%, 1.22%, 1.20%, 0.80%, 0.60%, 0.35%, 0.28% and 0.24 mm cm⁻¹ respectively). Since, as can be seen in Figure 4, the distance between the two curves decreases and therefore the plant available water content decreases (since this corresponds to the difference between the water at -33 kPa and -1500 kPa). Regarding

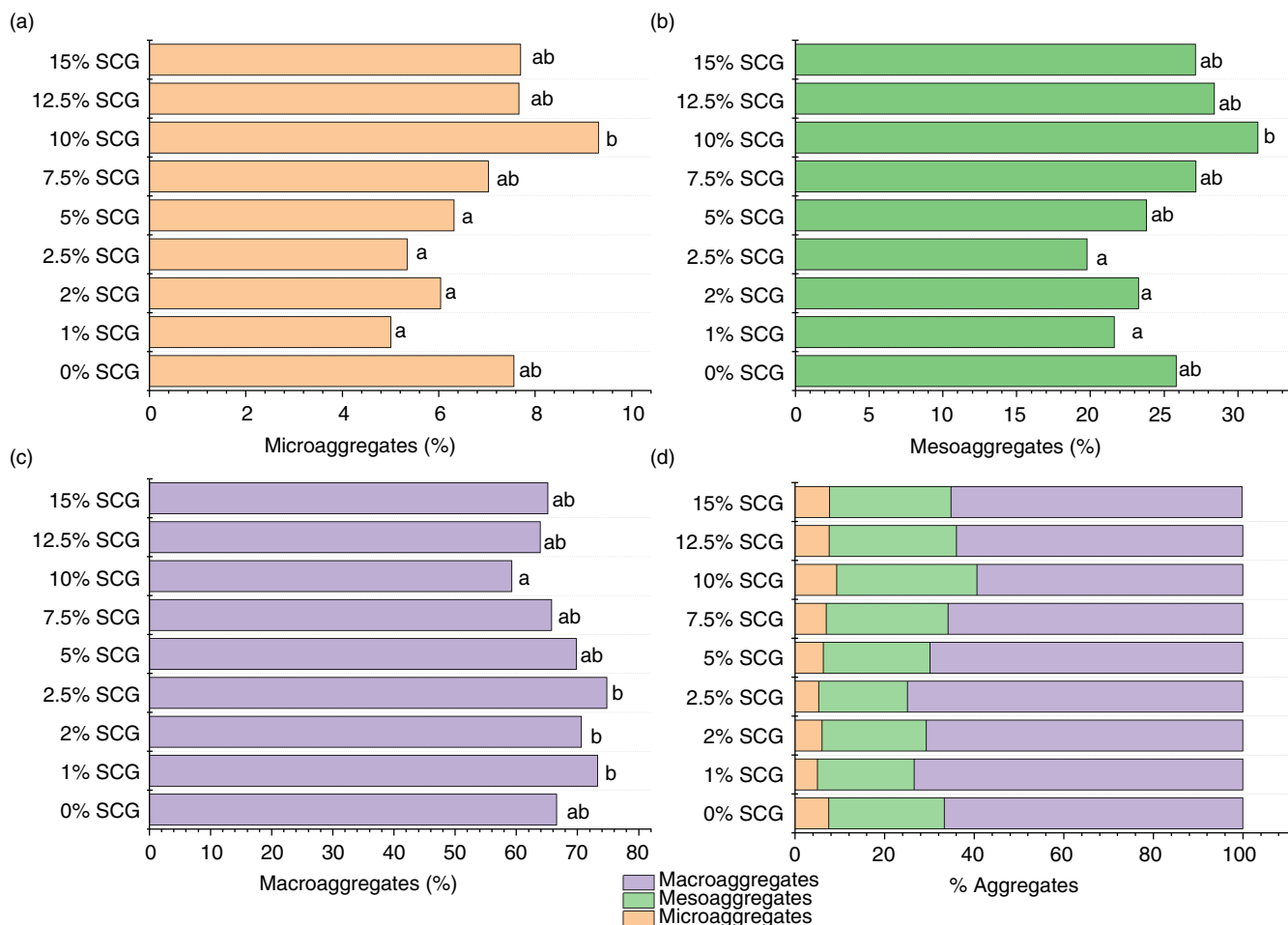


FIGURE 3 Percentage of macro, micro and mesoaggregates. Different letters indicate statistically significant differences ($p < 0.05$).

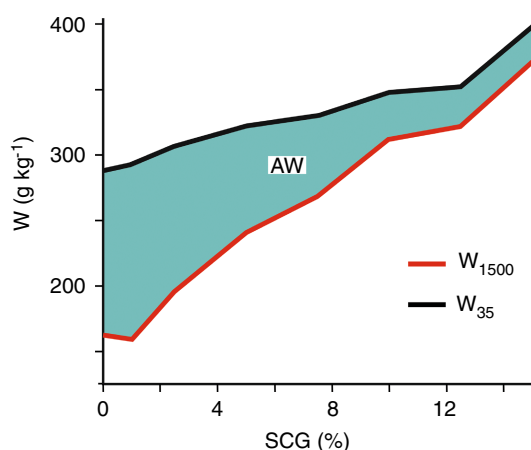


FIGURE 4 Water retention capacity at field capacity (W_{33}) and at permanent wilting point (W_{1500}) versus SCG dose. The distance between both straight lines is equal to plant available water content (AW).

the relationship between SCG doses, water retention and AW, the effect is totally linear. The higher the SCG dose, the higher the water retention and the less plant available

water content. In our case (clay soil with smectite), the SCG are integrated into the soil structure due to the cracking pattern and are surrounded by clay particles (as will be discussed later with the SEM images, Figure 2), increasing the porosity of the aggregates as observed in Figure 5. In the case of the sandy loam soil, the interaction between the SCG with the aggregates and the sand-silt particles will be different and this can be attributed to the differences between behaviour amid both soils.

As seen in Figure 5, both the addition of SCG and the simple incubation of the samples increase the individual porosity of the aggregates. Porosity increased 430% in the case of the 5% SCG sample (Figure 5c) and 639% in the 15% SCG sample (Figure 5d) compared to the control sample (Figure 5a). Simple incubation of the sample for 40 days (Figure 5b) also increased porosity due to small changes in humidity during incubation in the climatic chamber. As can be seen in Figure 5, the incorporation of SCG appears to be intra-ped in the case of the sample added with 5% SCG. However, when high amounts of SCG are added, they seem to form a mass which covers the mineral particles of the soil, facilitating the union between them.

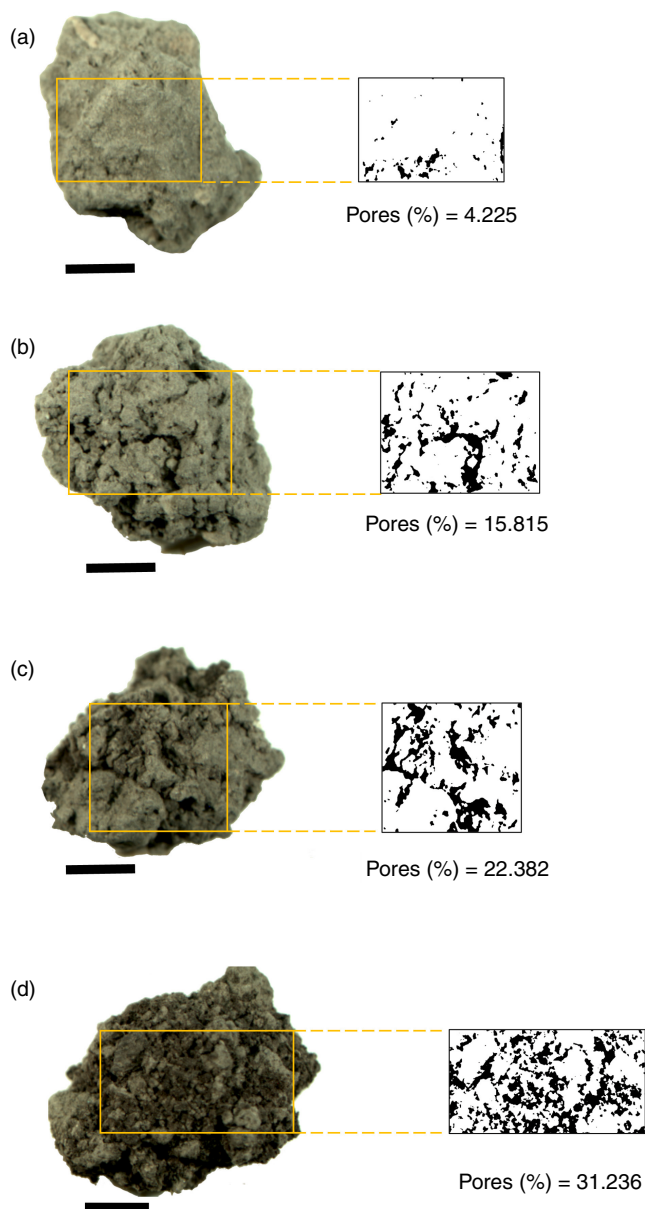


FIGURE 5 Stereomicroscopic images of the macroaggregates. (a) Native soil. (b) Control. (c) 5% SCG. (d) 15% SCG. The measuring bar corresponds to 1 cm

Another proof of the modification of porosity by the addition of SCG is provided by SEM images. Thus, in Figure 2b how the addition of small amounts of SCG (2.5%) stimulates the soil pedality is observed. The rounded macroaggregates of size close to 5 mm are clearly divided by a pore pattern into mesoaggregates of a size close to 1 mm.

Figure 2c shows us that SCG particles are arranged on the surface of the aggregates and that they also enter the cracks generated by changes in soil moisture (clay with smectites). The EDX diagrams corroborate the nature of the SCG particles that appear in the image. The clay particles cover the SCG particles as shown in Figure 2d, taken with backscattered electrons, where the darkest colours

correspond to the carbon-rich particles, with lower atomic numbers. Those of light colours, are the minerals that contain silicon, aluminium ... This interaction between the organic particles of the SCG and the minerals of the soil, justifies the effects of the SCG in the short term.

4 | DISCUSSION

The addition of SCG in increasing doses to a Mediterranean clay soil also modifies its chemical and physicochemical properties (Table 2). The increase in pH coincides with that reported by Hardgrove and Livesley (2016), since they found a decrease in pH when they added SCG in the field at doses of 2.5%, 5%, 10% and 20%. Although SCG increase EC_{25} , the limit of a saline soil, established at 2.4 dSm^{-1} (Liu et al., 2014), is not reached, which could negatively affect plant growth. The increase in OC is favourable, since the world's soils have lost 116 Gt of OC since agriculture began (Rumpel & Chabbi, 2021). The increase in N in soils could be a very positive aspect for crops. However, in the work of Cervera-Mata, Navarro-Alarcón, et al. (2019), corresponding to this same trial, but focused on the properties of the plant, it was reported that the addition of SCG inhibited plant growth from the lowest dose (1%).

As already indicated, one of the substantial effects of soil organic amendments is on the soil physical properties due to these residues contain large proportions of organic carbon. Thus, in recent years, numerous articles have been reported on the influence of different organic amendments (biochar of different sizes and temperatures, compost from different sources, poultry manure, etc.) on soil physical properties and how the application dose is one of the factors that most influence these effects.

The decrease in BD has been reported for the addition of biochar, sewage sludge compost, composted olive peel or poultry manure (Aranda et al., 2016; Aranyos et al., 2016; Garbuz et al., 2021; Khaliq & Abassi, 2015; Moreno et al., 2016; Omondi et al., 2016; Zhang, Wang, et al., 2022). Furthermore, Aranyos et al. (2016) also observed how the BD decreased with the compost application rate. This decrease in BD is directly related to the biological activity of the soil as well as to porosity (Bronick & Lal, 2005), a result that we will comment on in the next section. This increase in the biological activity of the soil due to the addition of SCG has been recently reported by our research group (Cervera-Mata et al., 2022), seeing how SCG significantly increased CO_2 emissions into the atmosphere. In other works (Cervera-Mata et al., 2021), it has been shown that the addition of SCG stimulates the activity of fungi.

The increase in total porosity coincides with those of Zhang, Wang, et al. (2022) and Fu et al. (2021) who

found that the addition of weathered coal, biochar and grass peat increased the soil total porosity. Zhang, Wang, et al. (2022) found that the addition of weathered coal increased the macroaggregates. Cervera-Mata, Martín-García, et al. (2019) in a test on two clayey Mediterranean agricultural soils added different SCG doses (2.5% and 10%) and found the following: (a) with respect to the macroaggregates, the original soils had an average of 23% that after 60 days of incubation reached up to 68% (average), which meant an increase in almost 200%; (b) regarding the microaggregates accounted for 30% (average) and after incubation they decreased to 4% of the total, which represented a decrease of 650%; (c) the mesoaggregates had an irregular behaviour, increasing their percentage in some samples and decreasing in others.

The increase in W_{33} agrees with that established by Turek et al. (2019) for the addition of SCG in doses of 5%, 10%, 15% and 20%, attributing it to the changes in the OC contents in the soil. They also coincide with the results of Badaou and Sahin (2021) for the addition of sewage sludge (150 Mg ha^{-1} , corresponding to the addition of 2.5%). The tendency to increase the micro and mesoaggregates with the addition of SCG (Figure 3) could also be related to the increase in W_{33} , since this water is more related to the structure than to granulometry (Rajkai et al., 2015). However, Turek et al. (2019) observe a decrease between 15% and 20% SCG attributing it to the modification of the microporous structure when adding SCG to the soil. In our case, we have not reached these SCG contents, but the trends in aggregate sizes (Figure 3) indicate that from 10% the micro and mesoaggregate contents decrease and those of macroaggregates increase, which would coincide with what is indicated by these authors. Aranda et al. (2016) also reported an increase in W_{33} after the addition of olive mill pomace compost. The increase in W_{1500} has also been observed for other types of waste such as composted olive peel and compost and poultry manure (Forge et al., 2016; Moreno et al., 2016). However, with respect to plant available water content, Turek et al. (2019) observed an increase in it after the addition of SCG. Our results contradict those of these authors, which may be due to the different type of soil used, mainly in terms of clay content: clayey in our case and sandy and silty in the case of Turek et al. (2019). On the contrary, our results coincide with those of Forge et al. (2016) and Zhang, Wang, et al. (2022) by adding compost of bird manure for 1 year in a Podzol type soil, that is, sandy and peat grass in a loamy soil respectively. In contrast, Parker et al. (2021) in a field experiment investigating the short-term effects of rice straw biochar (10 and 20 t ha^{-1} rates) on soil aggregate stability in an Acrisol in Ghana, found that the addition of biochar did not affect water retention characteristics. Turek et al. (2019) using pots filled with a mixture of a sandy loam soil with 0%, 5%,

10%, 15% and 20% of SCG, observed a decrease in water retention with high doses of SCG (15% and 20%), which they attribute to the fact that the pores are moulded by the SCG, modifying their shape, continuity and tortuosity.

The modification of the physical properties of the soil by the addition of SCG seems to be related in part to the hydrophobicity. Thus, Cervera-Mata et al. (2021) compared the effect of SCG on the physical properties of a red soil rich in illite and kaolinitic clays with those of an alluvial soil rich in smectites. In both soils, these authors found that SCG modified soil parameters related to hydrophobicity: water drop penetration times, contact angle and surface free energy components. These modifications, in the sense of greater hydrophobicity, are because the SCGs increase the content of a soil organic carbon with a lower humus quality index and a higher proportion of labile components. The increase in hydrophobicity leads to a significant change in physical properties: increases in structural stability, saturated hydraulic conductivity, water retention and total porosity and a decrease in available water content. The SEM images reported by these authors revealed the differential behaviour of the SCG in the two soils tested. The soil rich in smectites and carbonates (alluvial soil) showed SEM images where the SCG particles were inserted into the soil matrix (probably due to their greater cracking in the dry periods) and were covered by smectite particles. This supposes a greater stabilization of the organic matter to which the carbonates probably contribute. The greatest interactions between smectites and the organic matter have been described by other researchers (Six et al., 2002).

Another interesting question about the effects of SCGs on soil physical properties is the intensity of their effects and the time needed to be appreciated. In a previous research (Cervera-Mata, Martín-García, et al., 2019), our working group compared SCG with various types of biowaste (cited in the bibliography) in order to evaluate the intensity and time of their effects. These authors found that SCGs act more intensely and therefore require less interaction time than other types of waste. Thus, using the response ratio (RR) established by Omondi et al. (2016), we found, for example, that the RR reached by Forge et al. (2016) for structural stability adding 290 Mg ha^{-1} of compost and poultry manure during 1 year was 1.06, while with the addition of 270 SCG Mg ha^{-1} over 60 days was 1.7 (according to RR, the higher this index is, the greater the effect).

A principal component analysis was carried out to analyse how the different doses of SCG affect the properties of the soil. A matrix containing both the chemical and physicochemical and physical properties was selected. Figure 6 shows the space defined by PC1 and PC2 (which capture 76.61% of variance) with the score values of the samples. The highest values on PC1 (60.14% of variance) correspond

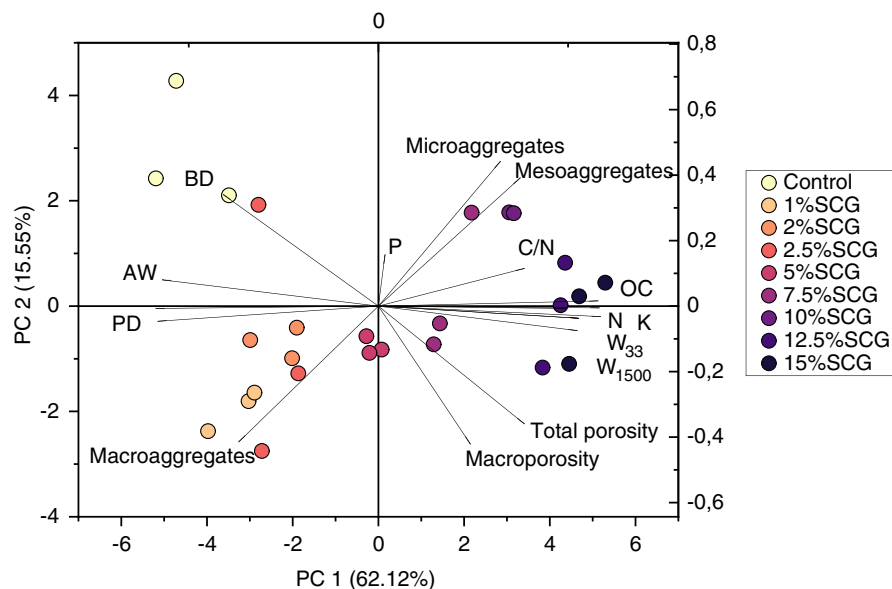


FIGURE 6 Superimposed graph of principal component analysis (PCA) scores obtained for samples and loadings of each parameter analysed. BD, bulk density; PD, particle density; AW, plant available water content; W_{33} , water retention at field capacity; W_{1500} , water retention at permanent wilting point; OC, organic carbon

to the highest doses of SCG (7.5%, 10%, 12.5% and 15%) with high values of OC, N, K, EC_{25} , W_{33} and W_{1500} . This is demonstrated by the high correlations between OC and N ($r = 0.912$; $p < 0.001$), OC and W_{33} ($r = 0.892$; $p < 0.001$), OC and W_{1500} ($r = 0.948$; $p < 0.001$), total porosity and macroporosity ($r = 0.805$; $p < 0.01$), microporosity and W_{1500} ($r = 0.938$; $p < 0.001$). Furthermore, it is important to note the negative correlations between BD and total porosity: $r = -0.993$; $p < 0.001$. On the contrary, the negative values on this PC correspond to the lowest doses of SCG (1%, 2%, 2.5% and 5%) with high values of macroaggregates, pH and AW. This is demonstrated by the high correlations between macroaggregates and pH ($r = 0.613$; $p < 0.01$); pH and AW ($r = 0.905$; $p < 0.001$); BD and AW ($r = 0.780$; $p < 0.001$). PC2, which explain the 16.47% of variance does not seem to group the samples according to a specific criterion, hence it explains a very small percentage of the variance of the system. It is also observed how the dose of 5% is a threshold for all the physical properties of the soil, finding these samples in the middle of the scatter plot.

5 | CONCLUSIONS

1. The addition of SCG to the Vega soil had a positive effect on all soil physical properties except for the plant available water content.
2. The water retained at field capacity (-33 kPa) and at the permanent wilting point (-1500 kPa), as well as the total porosity of the soil, was increased proportionally to the amounts added, thus decreasing the bulk density.
3. The effects of SCG on the soil physical properties are very intense in the short term due to the interaction between the particles of this residue with the mineral particles of the soil, specifically with smectite.

4. It is observed that the 5% SCG dose is a threshold in terms of the effect on the physical properties, although there are some properties that respond to the lowest doses tested (1% SCG).

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CONFLICT OF INTEREST

The authors do not declare any conflict of interest.

DATA AVAILABILITY STATEMENT

The data that support the findings of this study are available from the corresponding author upon reasonable request.

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