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Abstract

Spent coffee grounds (SCG) are a bioresidue generated in large amounts worldwide, which could be employed as either fresh or transformed organic soil amendment, by means of different treatments in order to improve its agronomic qualities. An *in vitro* experiment was conducted in order to evaluate the effect of using different bioamendments derived from spent coffee grounds (SCG) on biomass and Zn, Cu and Fe content of lettuces. Application of 7.5 % (w/w) fresh SCG, vermicompost, compost, biochars (at 270 and 400 °C; pyrolysis), SCG washed with ethanol and water, and hydrolysed SCG was carried out in an agricultural soil (Cambic Calcisol). In order to compare with conventional agriculture, the addition of NPK fertilizer was also assessed. Only vermicompost and biochar at 400°C overcome the growth limitation of SCG. However, these treatments diminished Zn, Cu and Fe concentrations in lettuce probably due to the destruction (microbial degradation/thermal treatment) of natural chelating components (polyphenols). Increase in mineral content was observed in those treatments that did not completely eliminate polyphenols. NPK fertilizer gave rise to lettuces with higher biomass but lower micronutrients content. The results lead us to the possible solution for the use of SCG as organic amendment by vermicomposting and biocharization in order to eliminate toxicity.

Keywords: spent coffee grounds, micronutrients, polyphenols, organic amendment, vermicompost, biochar.

1. Introduction

Coffee is a worthy primary product for its consumption as ready-to-drink beverage after an extraction process with hot water (Yamane et al., 2014). However, several by-products like spent coffee grounds (SCG) are generated after coffee beverage production at high amounts (15 million tons annually, according to Kamil et al., 2019). Generally, agricultural soils have low amounts of carbon. Thus, Rodriguez et al. (2016) stated that half the Spanish soils have less than 1% of soil organic carbon. This fact is in line with the “4 per mille” initiative launched by the 21st Conference of the Parties to the United Nation Framework Convention on Climate Change (COP21, November 30 to December 11, 2015) in order to enhance soil organic carbon (SOC) content of world soils 0.4% per year.

In SCG, the high content of organic matter represented by cellulose, hemicelluloses, lignin, proteins, lipids, polyphenols, chlorogenic acids and caffeine, are remarkable (Campos-Vega et al., 2015). However, the direct addition of fresh SCG as organic amendment to agricultural soils develops a negative effect on crop growth (Hardgrove and Livesley, 2016; Ribeiro et al., 2017). This effect could be related to the competition between roots and microorganisms for nitrogen in soils (Kuzyakov and Xu, 2013), as well as with the presence of toxic compounds in SCG (Leifa et al., 2000). Therefore, SCG composting, could be a profitable alternative that allows the degradation of caffeine, polyphenols and condensed tannins; this pre-treatment diminishes SCG anti-physiological and anti-nutritional effects on plant growth, as well as achieving a more transformed final product with a lower C/N (An et al., 2019; Campos-Vega et al., 2015; Gummadi et al., 2012; Leifa et al., 2000; Santos et al., 2017). In this sense, other authors reported that the type of compost had a significant effect on both yield and phenolic content of lettuce (Coria-Capuyán et al., 2009; Santos et al., 2017).

The absorption of micronutrients by plants (such as Zn, Cu or Fe) in relation to the addition of organic amendment has been widely described in the literature. Sofu et al. (2016) reported that the adoption of an organic management, when comparing three-different fertilization treatments (manure, rock dust and manure + rock dust) of agricultural soils, determined higher yields and

better lettuce Cu balance, although no changes were found for Fe and Zn when compared with controls (conventional managements). Moreover these authors found that Fe + Zn + Cu content in leaves of green lettuces were significantly and negatively correlated with total antioxidant capacity as well as total phenol content (Santos et al., 2017). Other researchers found that inorganic phosphorus fertilizer ameliorated maize growth by reducing Cu (66.3%) and Zn (91.9%) concentrations in roots and therefore metal uptake by the plant (Wu et al., 2017). On the other hand, Kelly and Bateman (2010) did not find differences in Zn, Cu and Fe content when compared commercially grown organic and conventional crops of lettuce.

In a preliminary study, the amendment of agricultural soils from the Vega of Granada with increasing percentages of SCG has been checked (from 1 to 15%, expressed as SCG g/100 g of soil) showing the exertion of a negative effect on lettuce growth and biomass, even in a noticeable way for the lower SCG percentage (Cervera-Mata et al., 2019). Contrarily, a positive aspect of the referred study was that the organic SCG amendment of soils enhanced the nutritional value of lettuces by increasing the levels of different micronutrients (Cervera-Mata et al., 2019). On the other hand, Ribeiro et al. (2017) reported that SCG could possibly be suitable for soil amendment if previously stabilized. In this sense, different stabilizing SCG treatments would be considered in this study, like the promising composting and vermicomposting (Murthy and Madhava Naidu, 2012), which could be used for recycling industrial wastes providing an enhancement of soil nutrients and finally a better lettuce growth. Santos et al. (2017) reported that SCG composting diminished total phenols and tannins and increased gallic acid. The group of Cruz et al. (2014) reported that the presence of 5% (v/v) of composted SCG significantly reduced the microelements Cu and Fe, whereas the direct composting in soil (during 4 months) of 30% (v/v) of fresh SCG increased the levels of Cu, Fe and Zn. Liu and Price (2011) found that different composting systems (in-vessel, static aerated pile or vermicomposting) can be used for SCG composting. The group of Ronga et al. (2016) reported that SCG compost could effectively replace peat moss (a highly demanded growing substrate) for the production of potted plants.

Other researchers (Vardon et al., 2013) have stated that the application of SCG biochar with fertilizer enhanced 2-fold the sorgum-sandgrass yields, remarking its capacity as soil amendment. However, without the concomitant addition of fertilizer to agricultural soils, the application of SCG biochar alone did not increase biomass yield (Vardon et al., 2013). Other authors reported that biochar reduced the compost toxicity and alleviated the inhibitory effect of seed germination capacity in treatments with maize straw composts and composts enriched with coffee grounds and yeast effluent (Kopec et al., 2018). Similarly, Kim et al. (2014) checked that SCG biochar (400°C for 20 min) facilitates the overcoming of the phytotoxic effect of the SCG alone when added to agricultural soils, and therefore enhances root elongation.

Taking into account all this information, the aim of the study was to select the most appropriate treatment of SCG that generates a bioproduct that, on the one hand, overcomes plant phytotoxicity and, on the other hand, takes advantage of the chelating capacity of SCG. In other words: a better and higher productivity of lettuce's biomass and a higher nutritional value in relation to Zn, Cu and Fe contents in plant. The following SCG treatments were investigated: composting, vermicomposting, biocharization at different temperatures, the fallow effect, and other possibilities such as washing with water or ethanol, or high temperature hydrolysis of SCG, from which no information is available in the literature. Another objective of the study was to compare the addition of bioamendments derived from SCG against conventional agriculture represented by an inorganic fertilization (NPK).

2. Material and methods

2.1. Soil, SCG and bioamendments derived from SCG

Soil samples were the arable layer (0-20 cm) of an agricultural soil of the Vega of Granada (Andalucía, Southern Spain), typical of the Mediterranean climate. This soil had a brownish-grey color (color Munsell: 10YR 5.5/2), and was classified as Cambic Calcisol (Aric, Ochric) (IUSS Working Group WRB, 2014). Soil samples were air dried and sieved (< 5 mm).

SCG were obtained from the cafeteria of the Faculty of Pharmacy (University of Granada). SCG were spread into a thin layer and dried at room temperature to remove residual moisture. Seven bioamendments were elaborated from SCG: two compost (vermicompost and conventional compost), two biochar (at 270°C and 400°C), SCG washed with ethanol, SCG washed with hot water and SCG hydrolysed at high temperature (180°C). The different composts derived from SCG were supplied by the organic natural fertilizer company AGROHULOVA S.L (Granada, Spain). The vermicomposting treatment of SCG was performed for 8 months using Californian red earthworm (*Eisenia foetida*). Conventional SCG composting with dolomite was performed during 8 months using the proportions of 28.72 kg of SCG and 8.5 kg of dolomite [$\text{CaMg}(\text{CO}_3)_2$] in order to neutralize the acidic pH of SCG. A vermicompost of goat manure obtained in the same conditions as the vermicomposted SCG was used as control, due to the increasing importance of this type of products in organic and ecological agriculture (Hussain and Abbasi, 2018). Two types of SCG biochar were performed by means of pyrolysis in a pyrolytic oven (Navertherm GmbH, Germany) in two different ways: 270°C for 60 min (modified from Comino et al. 2017), and 400°C for 30 min (Kim et al., 2014). SCG washing with ethanol or hot deionized water was performed in a percolated funnel until reaching discoloration of the percolated materials. Hydrolysed SCG was a residue obtained after the extraction of manooligosaccharides from SCG (Pérez-Burillo et al., 2019). A mix of SCG and water (10% w/w) was hydrolysed in a reactor (270 mL of distilled water and 30 g of spent coffee grounds) by applying 180 °C for 60 min.

2.2. Lettuce samples

The assay was carried out with 30-day-old lettuces (*Lactuca sativa* var. *Longifolia*) of the “Little Duende” variety coming from a commercial greenhouse in Southern Spain (Saliplant S.L., Granada). Lettuce ‘Little Duende’ is one of the small varieties, and so it is well suited for *in vitro* experiments performed in climatic chamber (Cervera-Mata et al., 2019). In this work, these lettuces are called “baby lettuces”.

2.3. Experimental design

The assay was carried out in octuplicate. Five controls and eight SCG treatments were used. The different controls and SCG treatments and their main research question are summarized in **Table 1**. The application rate to soil for all bioamendments was 7.5 % (w/w). After the soil and bioamendments were thoroughly mixed, the soil-bioamendment mixtures were transferred to PVC pots of 300 mL capacity closed with a mesh of fiberglass at the base to avoid the loss of fine particles and transplanted with 30-days-old baby lettuces (Cervera-Mata et al., 2019). Then the samples were incubated in a growth chamber under controlled conditions with a relative humidity of 50–60%, temperature of 22/18°C (day/night) and 12/12-h photoperiod during 40 days. To prevent leaching and water stress, pot moisture was maintained between field capacity and permanent wilting point. The irrigation requirements were calculated by weighting. The pots were irrigated every three days with distilled water. The incubated samples (Inc-, **Table 1**) were included to study the fallow effect and consisted of samples without SCG addition (Inc-NoBA) or added with 7.5 % of SCG (Inc-SCG); these samples were then incubated for 40 days previous to the assay under the same incubation conditions described above, but were not cultivated with lettuce. A final group consisting of eight samples of baby lettuces was included to compare the effect of SCG on the initial state of the lettuces.

After 40 days of cultivation, lettuce samples were harvested separating the root from the edible part (leaves) of the lettuce (Cervera-Mata et al., 2019). The edible part was weighed and subsequently washed with distilled water and finally dried at 65°C for 24h. The washing step was performed to mimic the cleaning process followed as a previous step to the eating of this vegetable by humans. Finally, lettuce samples were homogenized and frozen at –80°C until element analysis. Soil samples from microcosms were dried at room temperature and conserved in PVC flasks at refrigeration temperature (5°C).

2.4. Elemental analysis in lettuce samples

Two hundred milligrams of dried lettuce sample were mineralized by attack with HNO₃ and H₂O₂ of supra-pure quality (Merck, Darmstadt, Germany) in a microwave digester (CEM MARS, XP1500 Plus). The digest was diluted to 50 mL with Milli-Q water in order to obtain the analytical dissolution. The determination of total concentrations of Zn, Cu and Fe in lettuce samples was performed by an AAS instrument (Varian SpectraA, 140. Mulgrave Victoria, Australia). Calibration curves were previously prepared by diluting stock solutions of 1,000 mg/L in 1% HNO₃ for the analyzed elements (Merck, Darmstadt, Germany).

The accuracy and precision of Zn, Cu and Fe measurement procedures ($n = 10$) were verified by testing the certified reference standard Apple leaves powder of the National Institute for Standards and Technology (NIST) 1515 (Gaithersburg, MD, USA). No significant differences were found between the mean element concentrations determined in these materials and the certified concentrations (12.4 ± 0.43 and 12.7 ± 0.85 , 5.69 ± 0.13 and 5.48 ± 0.35 , and 82.7 ± 2.60 and 83.1 ± 3.00 , for Zn, Cu and Fe, respectively). Additionally, the accuracy of the methods was also tested on the basis of recovery experiments, after complete digestion of spiked lettuce samples with different amounts of elements from the standard solutions (Cervera-Mata et al., 2019). The calculated recoveries for each element were between 98% and 103% in all cases. The limits of detection (LOD) of the method for elements analyzed (3.7, 0.83 and 0.23 ng/ml, for Zn, Cu and Fe, respectively) were calculated as previously reported (Cervera-Mata et al., 2019). The concentration (mg/100g) in samples was obtained by linear calibration. Every element was analyzed in triplicate in each one of the lettuce samples. The concentration of these elements is expressed in fresh weight, which is the usual form of consumption.

2.5. Characterization of soil and bioamendments

The methods of soil analyses of the American Society of Agronomy and Soil Science Society of America (2014) were followed for analyses. Soil pH was measured in 1:2.5 (w/w) soil–

water suspension and in 1:5 (w/w) for organic amendments. Electrical conductivity (EC_{25}) was measured in the extract of the 1:5 (w/w) soil–water and the 1:10 (w/ w) bioamendment–water suspensions. Organic carbon and total Nitrogen was determined with a Truspec CN analyzer (LECO Corporation, Saint Joseph, MI, USA). Available phosphorus was determined by Olsen Watanabe’s method with a Helios alpha spectrophotometer (Thermo Fisher Scientific Inc., Waltham, MA, USA). Available potassium was extracted with 1N ammonium acetate ($pH = 7$), and determined with a PFP7 flame photometer (Jenway, Staffordshire, UK). Bioavailable Zn, Cu and Fe (extractable with DTPA) were determined by the Lindsay and Norvell’s method with AAS instrument (Varian SpectraA, 140. Mulgrave Victoria, Australia). The Folin-Ciocalteu assay was used to measure the content of total phenolic compounds as described by Singleton et al. (1999); total phenolic compounds were expressed as mg of gallic acid equivalents per g of soil (mg GAE/g).

2.6. Statistical analysis

The homogeneity of variance was assessed using the Levene test and the normal distribution of the samples with the Shapiro-Wilk test. The Student’s t-test was used to analyze parametric data and the Kruskal-Wallis test to analyze non-parametric data. Linear correlations were evaluated by computing the correlation coefficient by Pearson (for normally distributed data) or Spearman linear correlation (for non-normally distributed data). For the correlation analysis all the data matrix were considered. The significance level was set at 5% ($p < 0.05$) in all tests. SPSS 22.0 for Windows (IBM SPSS Inc., New York, USA) was used for data analyses. A principal component analysis (PCA) was performed to reduce and explain the variability of the system and to represent samples in the PC plot. The validation method used was cross validation leave one out. This statistical treatment was performed in MATLAB 2008R (MathWorks, Natick, USA) using the Eigenvector Research Inc. (Wenatchee, USA) PLS-toolbox.

3. Results and discussion

3.1. Effect of the SCG treatments on chemical and physicochemical soil properties

The chemical and physicochemical properties of soil and bioamendments are shown in **Table 2**. Vega soil had a basic pH (8.2) due to its carbonated character (Cervera-Mata et al., 2017). Fresh-SCG had an acidic character and high content of C. All composts of SCG were characterized by its salinity (high EC₂₅) and both SCGchar had contents of C higher than 50%. The higher total phenolic contents resulted in fresh-SCG, ethanol-SCG, water-SCG and hydro-SCG.

The influence of the different SCG treatments on soil chemical and physicochemical parameters is shown in **Table 3**. Despite the acidic character of some materials, such as fresh-SCG, water-SCG and hydro-SCG (**Table 2**), the pH values of microcosms were in the range of neutrality and basicity, which could be related to the tampon capacity of the carbonates content of Vega soil (Cervera-Mata et al., 2017). It has been previously reported that the biocharization of SCG increases the pH of the raw material (Kim et al., 2018), which could be due to the generation of ashes (Nigussie et al., 2012). Vermicomposting neutralized the pH of SCG, which coincides with Lui and Price (2011) and is attributed to the basic hydroxides generated during the first phases of the vermicomposting process (Nogales et al., 2014). Composting with dolomite also increased the pH because of the basic character of dolomite. In this sense, any bioamendment was harmful because of its acidity. However, the salinity of some bioamendments did influence negatively on the soil. For example, vermi-SCG, comp-SCG and vermi-goat increased significantly ($p < 0.05$) the EC₂₅ of the soil due to its high salinity (> 20 dS/m, **Table 2**), giving rise to salinities significantly higher than those obtained with SCG. Vermicomposting was the treatment that increased salinity to a greater extent, which is the opposite to what is commonly reported (Nogales et al., 2014). However, this property could be directly related with the specific processing conditions of vermicomposting. Soil samples added with vermi-SCG could be considered moderately saline, which could affect several cultivars (Navarro and Navarro, 2013). In this sense, some studies reported that EC exceeding 1.1 dS/m significantly reduces lettuce growth (Ünlükara et al., 2008). In the case of the C/N ratio, it ranged from 7 to 14, being the lower ratio ($p < 0.05$) vermi-SCG

samples, due to its highest content of N. In fact, vermi-SCG is the most humified material with a C/N of 7 (**Table 2**). Nevertheless, the bioamendments that provide the higher content of C to the soil (and can serve as carbon sinks) were SCG and both biochar, being the SCGchar₄₀₀ the SCG derivative that provided more C (**Table 2**). Vermi-goat and vermi-SCG increased significantly ($p < 0.05$) the amounts of available P and K, respectively, reaching values of 140 ppm of P and 1047 of K. This could be related to the high amount of K in fresh-SCG (3800 ppm), as stated by Liu and Price (2011).

3.2. Fresh weights of lettuces cultivated with the different SCG treatments

The addition of fresh-SCG diminished significantly ($p < 0.001$) the fresh weight of lettuce in relation to NoBA (**Fig. 1**), as previously reported for this vegetable (Cervera-Mata et al., 2019; Cruz et al., 2015; Hardgrove and Livesley, 2016), carrots and spinaches (Cruz and Marques dos Santos Cordovil, 2015). Other authors also found the same effect when different organic amendments related with SCG (fresh SCG mixed with compost and domestic ash) were directly added to soils (Kopec et al., 2018; Ribeiro et al., 2017). Contrarily, the inorganic fertilization with NPK significantly enhanced lettuce biomass in relation to NoBA lettuces and fresh-SCG lettuces (**Fig. 1**); the same effect was reported in a previous study (Cervera-Mata et al., 2019).

A positive *fallow effect* on lettuce biomass was found (**Fig. 1**) when agricultural soils without bioamendment were previously incubated for 40 days before the beginning of the experiment (Inc-NoBA). Fallow is the non-productive periods that leave essential elements in abundance for the upcoming agrosystem (Wojtkowski, 2010). In this sense, the fresh weights of Inc-NoBA lettuce increased significantly ($p < 0.001$) in relation to NoBA lettuces (19.9 ± 0.49 and 12.9 ± 0.28 , respectively; **Fig. 1**). Nevertheless, this positive fallow effect was not found when comparing the fresh weights of fresh-SCG with Inc-SCG lettuces (2.22 ± 0.41 and 1.89 ± 0.27 , respectively; **Fig. 1**). This finding could probably be related to the short-term incubation (only 40 days), which could be not enough to get the necessary degradation of the anti-physiological and

anti-nutritional components of fresh SCG like caffeine, polyphenols and condensed tannins (Campos-Vega et al., 2015; Gummadi et al., 2012; Santos et al., 2017). In this sense, Cruz et al. (2015) found that 4 months of SCG fallow (termed “direct composting” in their study) were enough to eliminate caffeine by means of degradation with microorganisms or lixiviation. The “poor” fallow effect of SCG (Inc-SCG, **Fig. 1**) could be related with the competition for nitrogen (N) between those microorganisms existing and the plant, which in turn could affect lettuce productivity (Cervera-Mata et al., 2017; Cruz and Marques dos Santos Cordovil, 2015; Kuzkayok and Yu, 2013). A positive aspect of the inhibitory effect on plant growth of SCG is the possibility of its use as weed control in a crop rotation system with fallow period (Yamane et al., 2014).

The negative effect on lettuce productivity disappeared when SCG were previously submitted to several treatments like vermicomposting and biocharization at 400°C, and in a lesser extent composting with dolomite and biocharization at 270°C (**Fig. 1**). It was also found (**Table 2**) that vermicomposting and biocharization at 400°C were the only SCG treatments that completely eliminated SCG phenols; therefore, the growth limitation could be directly related to the total phenols content in the bioamendments. In this sense, several authors reported that previous SCG treatments namely composting and vermicomposting (Liu and Price, 2011; Santos et al., 2017) or biocharization (Kim et al., 2014; Kopec et al., 2018; Vardon et al., 2013) could generate a more suitable product for agricultural proposals, facilitating the overcoming of the phytotoxic effect of SCG.

In this study the influence of composted SCG on lettuce productivity and a vermicomposted goat manure was also assessed and compared, a good organic fertilizer employed in organic farming (Hussain and Abbasi, 2018). Vermi-SCG significantly enhanced the fresh weight of lettuces (**Fig. 1**) although the addition of vermi-goat also enhanced significantly ($p < 0.001$) lettuce productivity (24.6 g) in a similar proportion to vermi-SCG (20 g). This finding opens the possibility of the use of vermi-SCG as a useful alternative to other organic amendments traditionally used in organic farming, and as well as a sustainable and ecological alternative to the inorganic fertilizers like NPK. Despite all this, lettuce productivity was significantly higher when

NPK was added to agricultural soils (49.5 g) in relation to the any other treatment (**Fig. 1**). Taking into account only fresh weights results, conventional agriculture (NPK) would be the most recommendable practice for crop production, but in later sections the nutritive capacity of lettuce in relation to its content in micronutrients will be discussed, which will generate a different criterion for the choice of a fertilizer.

The addition of comp-SCG slightly increased lettuce biomass in relation to fresh-SCG lettuces (3.61 ± 0.41 vs. 2.22 ± 0.41 g fresh weight, respectively, $p < 0.001$), although it was significantly lower than that for NoBA lettuces (12.9 ± 0.28 g, fresh weight; **Fig. 1**). During the elaboration of comp-SCG, dolomite containing Ca and Mg ions was added to neutralize SCG acidity. Mulia et al. (2019) also added dolomite to manure compost and found a three-fold increase in the number of tree leaves. Kammoun et al. (2017) also reported the positive effect of phosphogypsum supplemented compost (produced by mixing olive oil wastes and SCG, containing Ca and other ions) on potato plant growth and tuber yield.

In general, biocharization of residues can enhance plant growth and yield through the amelioration of soil chemical properties (Palansooriya et al., 2019). In our assay, the addition of SCGchar₂₇₀ and SCGchar₄₀₀ significantly increased lettuce productivity in relation to fresh-SCG lettuces (**Fig. 1**); however, in the case of SCGchar₂₇₀ such increase was scarce. Therefore, it would be necessary to apply higher temperatures to get a complete breakdown of organic matter. In this sense, 400°C for 30 min generated a fresh lettuce weight of 10.5 g and 270°C for 60 min 2.97 g (**Fig. 1**). In a phytotoxicity assay with *Raphanus sativus* L. seeds, Comino et al. (2017) reported that the thermal treatment of SCG at 275°C is enough to eliminate toxicity due to the decrease in hydrophobic compounds by mineralisation, volatilisation or transformation. Other authors²⁹ also reported that the addition of coffee husk biochar (530°C for 10 to 12 h under oxygen limited conditions) improved maize growth in a sandy soil. Similarly, Kim et al. (2014) reported that after 4 weeks of cultivation, bok choy roots had a higher elongation when treated with SCGchar (400°C for 30 min) compared to those cultivated with SCG only, although such elongation was lower than that of controls (without SCG). In sorghum-sandgrass, Vardon et al. (2013) found a similar result,

but they stated that the application of SCG biochar and an inorganic fertilizer was necessary to increase cultivar yield. Consequently, SCG-char could partially overcome the problems of phytotoxicity caused by SCG addition.

Fig. 1 depicts the behaviour of fresh lettuce weight treated with different SCG products. No significant differences ($p > 0.05$) were found for lettuces cultivated with fresh-SCG, ethanol-SCG, water-SCG or hydro-SCG. This finding points out that those SCG components related to the decrease in lettuce productivity (such as polyphenols), were not eliminated by a previous washing with ethanol, water or a hydrolysis treatment at high temperature (**Table 2**).

To sum up, SCG and the bioamendments derived from it, had two opposite behaviours regarding the growth of the lettuces (**Fig. 1**): i. When polyphenols were not detected (vermi-SCG, vermi-goat and SCGchar₄₀₀, **Table 2**), lettuces had a similar or higher growth to NoBA sample. ii. When polyphenols are present in the soil (fresh-SCG, comp-SCG, SCGchar₂₇₀, ethanol-SCG, water-SCG and hydro-SCG, **Table 2**) the growth limitation is evident.

3.3. Comparison between fresh-SCG and other bioamendments

The addition of fresh-SCG increased significantly ($p < 0.001$) the Zn, Cu and Fe concentrations in lettuces (**Table 4**) in relation to NoBA and Inc-NoBA treatments, as previously reported (Cervera-Mata et al., 2019). This is a positive effect of fresh-SCG addition as amendment to agricultural soils in organic farming since it increases the nutritional value of lettuce. Contrarily, Cruz et al. (2014) found a decrease in Zn and Fe levels in lettuces grown on soils amended with different concentrations of SCG, and an increase in Cu only at high doses (10 to 20%) compared to those cultivated in soils without SCG addition. These results are not comparable to those reported in the present paper since the group of Cruz et al. (2014) did not use real agricultural soils but added fresh-SCG to peat moss, which are known to influence the nutritional characteristics of plants (Ceglie et al., 2015).

In a previous study (Cervera-Mata et al., 2017), we reported that the content of available Cu, Zn and Fe increased significantly by SCG addition since this waste contains more Cu, Zn and Fe than the soils (Cervera-Mata et al., 2018) as other researchers also stated (Adriano, 2001). On the other hand, when the SCG are pyrolyzed, this procedure does not eliminate the mineral elements, but instead concentrates them, since the yields in both cases are ~70% for biochar270 and ~40% for biochar400. However, the plant is enriched when biochar270 is used, but the same cannot be said of biochar400. The main difference between both is that in the biocharization at 400 °C the polyphenols have disappeared. The facts undoubtedly lead us to a different hypothesis than the micronutrient supply.

Fallow effect had no influence on Zn, Cu and Fe concentration levels in lettuce leaves when agricultural soils (0% SCG) were previously incubated for 40 days before the beginning of the experiment ($p > 0.05$; **Table 4**). Similar results were obtained in fresh-SCG lettuces compared to Inc-SCG lettuces ($p > 0.05$; **Table 4**). Probably, the time used for the fallow effect was not enough to allow the decomposition of some SCG components in agricultural soils that would finally facilitate element uptake by lettuce.

The addition of vermi-SCG significantly decreased Zn and Fe ($p < 0.05$), and Cu ($p < 0.001$) concentrations in lettuce compared to fresh-SCG samples (**Table 4**). These results are similar to those reported by Cruz et al. (2014) for composted SCG and could be related to the reduction in the carbon structure of SCG, decreasing its metal-binding capacity. In this sense, vermicomposting probably caused the decomposition of some of natural organic components of SCG such as polyphenols (**Table 2**) and the generation of heat-processing related compounds like coffee melanoidins (Rufián-Henares, Guerra-Hernández and García-Villanova, 2006), which have been reported as chelating agents that facilitate elements uptake, and therefore the availability by plant (Morikawa and Saigusa, 2011). Consequently, vermicomposting would impair Zn, Cu and Fe availability to lettuce. Specifically for Fe, some researchers were able to chelate it with coffee melanoidins (Rufián-Henares and de la Cueva, 2009), which has been used as explanation to the Fe increase in the plant (Cervera-Mata et al., 2019). Similar results were stated by Coulibaly et al.

(2018), describing an increase in yield and a decrease of Zn a Cu levels in *Lagenaria siceraria* cultivated with vermicompost. Additionally, comp-SCG lettuces significantly diminished the Fe concentrations and with a tendency to signify the Cu levels ($p = 0.061$) in lettuces when compared with fresh-SCG lettuces (**Table 4**). This result could be explained in a similar way to that of vermi-SCG.

Similarly to that reported for vermi-SCG, the addition of SCGchar₄₀₀ significantly decreased Zn ($p < 0.01$) and Cu ($p < 0.001$) and Fe ($p < 0.05$), in relation to fresh-SCG samples (**Table 4**). This is in line with the results found by Kim et al. (2014) who stated that SCGchar at 400°C retained less amount of micronutrient by formation of metaling ligand complexes that precipitate minerals in the surface of soil particles, although other researchers found the opposite behaviour for SCGchar with similar conditions (at 450°C, Verdon et al., 2013). On this matter, Gunes et al. (2014) also reported a reduced Zn, Cu and Fe concentration in lettuce plants grown in alkaline soil added with poultry-manure-derived biochar at 300°C. However, the addition of SCGchar₂₇₀ increased (although not significantly) the concentration of Zn and Fe in lettuces compared to fresh-SCG lettuces (**Table 4**). This finding could probably be due to the presence (although scarce) of polyphenols (1.16 mg GAE/g, **Table 2**).

In order to assess if the vermi-SCG could be a good bioamendment to the organic farming, its effects were compared to manure vermicompost (vermi-goat). Zn, Cu and Fe concentrations were not significantly different ($p > 0.05$) between vermi-SCG and vermi-goat manure lettuces. Therefore, vermi-SCG could be considered as an alternative to the vermicomposted manures since it supplies more micronutrients and provides adequate biomass. This similar behaviour between both vermicomposts is also related to polyphenols, as they were not detected in both bioamendments (**Table 2**).

Table 4 shows that Zn, Cu and Fe concentrations in ethanol-SCG, water SCG and hydro-SCG lettuces were not significantly different ($p > 0.05$) in relation to fresh-SCG lettuce, except for Cu in ethanol-SCG whose levels decreased significantly. These findings point out that the SCG

components related with the uptake and availability of studied elements (Zn, Cu and Fe), namely polyphenols, were not eliminated by these treatments (**Table 2**).

Zn, Cu and Fe concentrations in fresh-SCG lettuces were also compared with baby lettuces (uncultivated). We found that Zn and Fe concentrations were significantly higher, while contrarily Cu concentrations were significantly lower (**Table 4**). The increase in Zn and Fe in fresh-SCG lettuces could be attributed to the high amounts provided by this bioamendment, as reported by Cervera-Mata et al. (2017); this could be related with the same mechanism previously mentioned: chelation by means of polyphenols and other chelating compounds provided by fresh-SCG and mobilization to the plant. Nevertheless, the behaviour of Fe is more complex.

Finally, the bioavailable Zn, Cu and Fe in soils added with the different bioamendments derived from SCG were also analysed subsequent to cultivation. A significant decrease in Zn amounts and a significant increase in Cu were found in the microcosms without bioamendment addition (NoBA, NPK and Inc-NoBA) in relation to fresh-SCG microcosms (**Table 4**). Moreover, fresh-SCG significantly increased the bioavailable Zn and Fe in the soil compared to SCGchar₂₇₀ and SCGchar₄₀₀, and Cu only for SCGchar₄₀₀ and water-SCG. On another note, the addition of vermi-goat, vermi-SCG and comp-SCG significantly increased the soil bioavailable Zn in relation to fresh-SCG treatment. In the literature, composting and vermicomposting are processes that can increase heavy metals concentrations, such as Zn and Cu, in the final product (Brunetti et al., 2019) although opposite results have also been described (Nogales et al., 2014). Regarding biochar, Kim et al. (2014) found that SCGchar₄₀₀ decreases the amounts of bioavailable Zn and Cu, as previously described in this study (**Table 4**). On the other hand, the addition of hydro-SCG significantly increased the contents of bioavailable Cu, which could be due to the acidic pH of this bioamendment (**Table 2**).

3.4. Comparison between NPK treatment and bioamendments

There are many assays in the literature that compare conventional agriculture vs. organic fertilization in relation to their impact on the nutritional value of crops (Gomiero, 2018). In this sense, Zn, Cu and Fe concentrations in NPK treatment with respect to the rest of treatments were

compared. Inorganic fertilization decreases the contents of the three micronutrients in all cases, but such a decrease was only statistically significant ($p < 0.05$) in the case of fresh-SCG, ethanol-SCG, water-SCG and hydro-SCG, and of Zn and Cu in the case of inc-SCG and SCGchar₂₇₀. Of particular interest is the comparison between fresh-SCG and NPK treatment, where fresh-SCG significantly increased Zn ($p < 0.001$), Cu ($p < 0.001$) Fe ($p < 0.01$) concentrations in lettuces compared to NPK treatment (**Table 4**). All these findings reinforce that those treatments which increase in a greater extent the nutritional value of lettuces have chelating substances that were not eliminated or where the transformation of these molecules did not take place. Others authors reported that inorganic P fertilizer as soil amendment ameliorates maize growth by reducing Cu and Zn concentrations in the roots (Wu et al., 2017). Pokhrel et al. (2015) also reported that organic fertilizers decreased the concentrations of all micronutrients in plants compared with the inorganic fertilizers. Previous findings (Cervera-Mata et al., 2019) reinforce the use of fresh-SCG as organic fertilizer in organic farming as a more nutritional, sustainable and friendly environmental alternative to conventional farming with inorganic fertilizers like NPK. In this sense, Kelly and Bateman (2010) compared Zn, Cu and Fe concentrations in commercially grown organic and conventional lettuce crops reporting no significant differences between them. Similarly, Sofo et al. (2016) also observed that Zn and Fe concentrations did not change in lettuces with organic fertilization, while higher levels were found for Cu. Nevertheless, the behaviour of organic amendments was not the same in all cases. Thus, in this study the type of organic amendment, vermicompost, biochar, hydrolysed, etc., influenced in a different way the levels of Zn, Cu and Fe in lettuces (**Table 4**).

Regarding bioavailable Zn, Cu and Fe concentrations in Vega soil after cultivation, compared to NPK addition, they showed an irregular behaviour among treatments (**Table 4**). The addition of fresh-SCG, vermi-goat, inc-SCG, vermi-SCG, comp-SCG, ethanol-SCG, hydro-SCG and water-SCG treatment increased the amounts of bioavailable Zn in relation to NPK treatment. Contrarily, for Cu NPK treatment increased bioavailable Cu in soils in comparison to SCG treatments with the exception of hydro-SCG (**Table 4**). For Fe, NPK treatment increased

bioavailable Fe in soils for water-SCG and hydro-SCG treatments and diminished for SCGchar₂₇₀ and SCGchar₄₀₀.

3.5. Correlations and principal components analysis

PCA (**Fig. 2**) and correlation coefficients (**Supplemental Table S1**) were calculated for a matrix containing lettuce mineral elements (Zn, Cu and Fe) and soil parameters (bioavailable Zn, Cu and Fe, pH, EC₂₅, C, total N, C/N ratio, available P and K). The scores for each sample were represented superimposed with the loadings of each parameter (**Fig. 2**). PC1 vs PC2 was represented in the scatter plot and both explain together a 59.37% of the variance. Zn, Fe and Cu contents in lettuces and C/N ratio in soils appeared together, with higher scores in PC1, near the fresh-SCG, ethanol-SCG, water-SCG, Inc-SCG, SCGchar₂₇₀; this could state a strong correlation between the three microelements in lettuces [Cu and Zn ($r = 0.777$), Fe and Zn ($r = 0.683$) and Fe and Cu ($r = 0.699$)] and also with C/N in soil [Cu and C/N ($r = 0.680$), Zn and C/N ($r = 0.565$) and Fe and C/N ($r = 0.585$)]. These results supports the idea that the treatments that increase in a greater extent the nutritional value of lettuces were those that had a higher C/N (**Table 2**) and consequently, increased the C/N ratio of soil (**Table 3**). Lower C/N corresponds to a more stabilized organic material, in which organic compounds with a greater number of free radicals disappear, as is the case of polyphenols. Therefore, higher C/N could be associated with a stronger chelating capacity of the organic material (Brunetti et al., 2019). Vermi-goat samples appear near available P, bioavailable Zn and EC₂₅, since these properties had higher values in this type of samples (**Table 4**); hence, high and significant correlations between these variables were found: P and Zn ($r = 0.498$), P and EC₂₅ ($r = 0.661$), Zn and EC₂₅ ($r = 0.768$). This corroborated that vermi-goat microcosms had higher values in these properties. On the other side of the graph, pH appears near the samples NoBA and InC-NoBA, since these samples had the more basic pH values. Near of these samples, Fe appeared together NPK samples due to the addition of this inorganic fertilizer seem to mobilize the Fe.

In summary, the addition of fresh-SCG and their derived bioproducts as soil organic amendment in an *in vitro* assay had different effects on the soil-plant system, which could be

mainly attributed to the different amounts of polyphenols in every type of processed SCG. These compounds disappeared completely when SCG were subjected to vermicomposting or biocharization at 400°C. In fact, those bioadmenments with high and medium polyphenol content (fresh-SCG, SCGchar₂₇₀, ethanol-SCG, water-SCG and hydro-SCG) limited plant growth, but improved Zn, Cu and Fe levels compared to the bioamendments with zero or low polyphenol content (vermi-SCG, comp-SCG and SCGchar₄₀₀). Conventional fertilization with NPK produces lettuces with a higher biomass, but with lower content in Zn, Cu and Fe compared to the tested bioamendments. Therefore, a high production and more nutritious foods are incompatible at the same time. These findings establish that future research in this field should be focused on the development of the appropriate mixtures of fresh-SCG or derived bioamendment with NPK (inorganic fertilizer) in order to concomitantly improve lettuce productivity and elements content to finally improve the nutritional value of lettuces.

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Figure captions

Fig. 1. a) Fresh weight of lettuces (different letters indicate statistically significant differences, $p < 0.05$). b) Images of lettuces after 40 days of cultivation. SCG, spent coffee grounds

Fig. 2. Superimposed graph of PCA scores obtained for samples (PC2 vs PC1) and loadings of soil parameters and mineral elements in lettuces. Zn_L, Zn in lettuce; Cu_L, Cu in lettuce; Fe_L, Fe in lettuce; Zn_S, Zn in soil; Cu_S, Cu in soil; Fe_S, Fe in soil.

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Credit author statement

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Declaration of interests

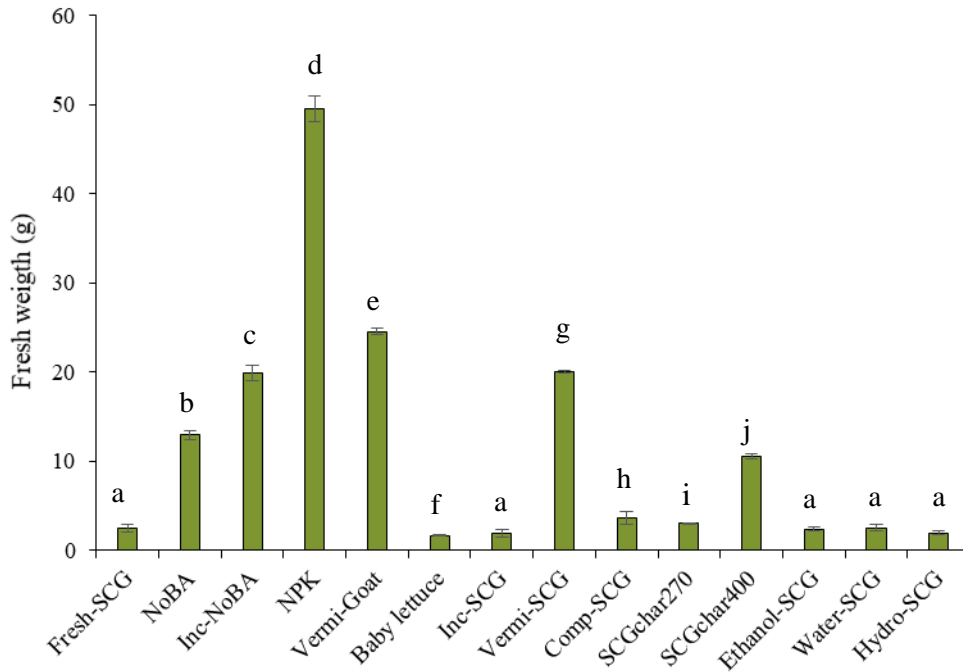
The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

The authors declare the following financial interests/personal relationships which may be considered as potential competing interests:

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Fig. 1

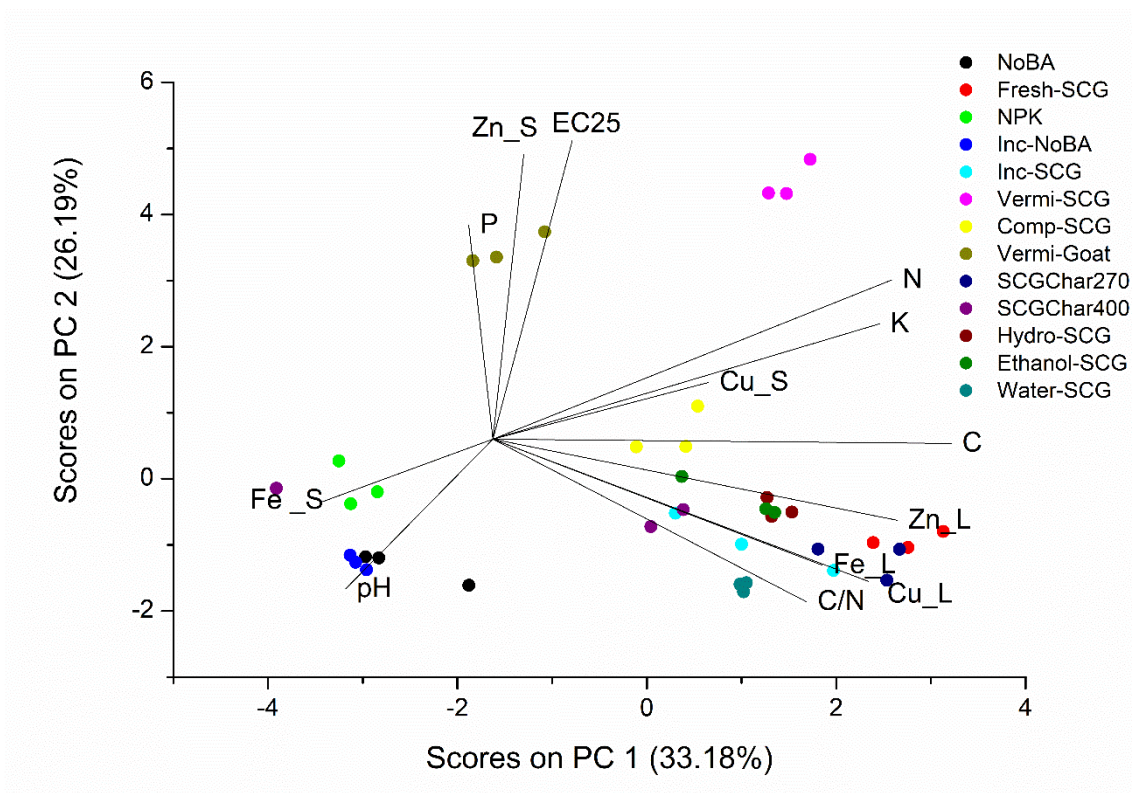
a)



b)



Fig. 2



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Table 1. Experimental conditions, nomenclature and objectives of each treatment.

	Microcosms	Nomenclature	Objective
Control samples	Vega soil with 7.5% of SCG	Fresh-SCG	Comparison with raw material
	Vega soil without bioamendment	NoBA	Comparison with no treatment
	Vega soil without bioamendment previously incubated for 40 days	Inc-NoBA	Fallow effect control
	Vega soil with NPK (15:15:15) (1.75 g/kg soil)	NPK	Comparison with inorganic fertilization
	Vega soil with vermicomposted goat manure	Vermi-goat	Comparison with conventional organic farming
SCG treatments to eliminate growth inhibition	Vega soil with 7.5% SCG previously incubated for 40 days	Inc-SCG	Fallow effect
	Vega soil with 7.5% vermicomposted SCG	Vermi-SCG	Biological mineralization of toxic components
	Vega soil with 7.5% composted SCG with dolomite [CaMg(CO ₃) ₂]	Comp-SCG	Biological mineralization of toxic components
	Vega soil with 7.5% biochar of SCG at 270°C	SCGchar ₂₇₀	Pyrolization of toxic components at low temperature
	Vega soil with 7.5% biochar of SCG at 400°C	SCGchar ₄₀₀	Pyrolization of toxic components at medium temperature
	Vega soil with 7.5% SCG washed with ethanol	Ethanol-SCG	Leaching of toxic components
	Vega soil with 7.5% SCG washed with hot water	Water-SCG	Leaching of toxic components
	Vega soil with 7.5% hydrolyzed SCG	Hydro-SCG	Treatment with high temperature water

Table 2. Characterization of soil and bioamendments.

Sample	Nomenclature	pH	EC ₂₅ (dS/m)	OC (%)	Total N (%)	C/N	Total phenolic compounds (mg GAE/g)
Vega soil	-	8.2	0.88	1.36	0.170	8	nd
SCG	Fresh-SCG	5.8	6.03	49.43	2.220	22	7.644
Vermicomposted goat manure	Vermi-goat	7.7	27.49	25.84	2.310	11	nd
Vermicomposted SCG	Vermi-SCG	7.8	48.28	40.63	5.770	7	nd
SCG-dolomite compost	Comp-SCG	7.3	22.55	29.33	2.653	11	0.008
SCG biochar at 270°C	SCGchar ₂₇₀	5.9	6.91	57.76	3.250	18	1.161
SCG biochar at 400°C	SCGchar ₄₀₀	7.2	11.95	69.22	4.400	16	nd
SCG washed with ethanol	Ethanol-SCG	5.4	11.25	45.70	2.443	19	14.264
SCG washed with water	Water-SCG	5.0	3.43	48.64	2.240	22	1.217
Hydrolyzed SCG	Hydro-SCG	3.8	5.42	54.29	2.162	25	13.975

SCG, spent coffee grounds; EC₂₅, electrical conductivity measured at 25°C; OC, organic carbon; GAE, gallic acid equivalents; nd, not detected.

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Table 3. Effect of the different SCG treatments on soil chemical and physicochemical properties (mean \pm SD).

Sample	pH	EC ₂₅ (dS/m)	OC (%)	Total N (%)	C/N	Available P	Available K
Fresh-SCG	7.9 \pm 0.1 ^{bc}	1.38 \pm 0.03 ^c	6.12 \pm 0.44 ^e	0.437 \pm 0.023 ^e	14 \pm 0 ^e	53 \pm 3 ^{ab}	747 \pm 14 ^e
NoBA	8.4 \pm 0.0 ^g	1.03 \pm 0.08 ^a	1.52 \pm 0.08 ^a	0.163 \pm 0.006 ^a	9 \pm 0 ^b	46 \pm 1 ^a	421 \pm 11 ^c
NPK	8.0 \pm 0.0 ^{de}	1.91 \pm 0.08 ^d	1.62 \pm 0.10 ^a	0.193 \pm 0.049 ^a	9 \pm 2 ^b	68 \pm 3 ^{cde}	305 \pm 13 ^a
Inc-NoBA	8.4 \pm 0.0 ^g	1.03 \pm 0.06 ^a	1.45 \pm 0.08 ^a	0.150 \pm 0.000 ^a	10 \pm 1 ^b	51 \pm 2 ^{ab}	347 \pm 6 ^b
Inc-SCG	8.2 \pm 0.1 ^f	1.37 \pm 0.03 ^c	4.17 \pm 0.24 ^d	0.347 \pm 0.006 ^{bc}	12 \pm 1 ^{cd}	61 \pm 2 ^{bcd}	766 \pm 18 ^{ef}
Vermi-Goat	8.0 \pm 0.0 ^{cde}	3.08 \pm 0.11 ^e	3.14 \pm 0.68 ^b	0.337 \pm 0.070 ^b	9 \pm 0 ^b	140 \pm 24 ^f	549 \pm 5 ^d
Vermi-SCG	7.9 \pm 0.0 ^b	4.76 \pm 0.23 ^f	4.24 \pm 0.24 ^d	0.613 \pm 0.031 ^f	7 \pm 0 ^a	79 \pm 4 ^e	1047 \pm 14 ^g
Comp-SCG	8.0 \pm 0.0 ^e	2.98 \pm 0.07 ^e	3.44 \pm 0.44 ^{bc}	0.383 \pm 0.068 ^{bcd}	9 \pm 0 ^b	59 \pm 5 ^{bc}	544 \pm 25 ^d
SCGchar ₂₇₀	8.0 \pm 0.0 ^{cde}	1.34 \pm 0.02 ^{bc}	5.71 \pm 0.83 ^e	0.417 \pm 0.055 ^{cd}	14 \pm 1 ^e	61 \pm 1 ^{bcd}	791 \pm 4 ^f
SCGchar ₄₀₀	8.4 \pm 0.0 ^g	1.16 \pm 0.05 ^{ab}	5.95 \pm 0.45 ^e	0.455 \pm 0.092 ^e	13 \pm 2 ^{de}	67 \pm 5 ^{cde}	749 \pm 22 ^e
Ethanol-SCG	8.0 \pm 0.1 ^{cd}	1.50 \pm 0.07 ^c	3.95 \pm 0.62 ^{cd}	0.333 \pm 0.031 ^b	12 \pm 1 ^c	70 \pm 3 ^{cde}	789 \pm 6 ^f
Water-SCG	8.8 \pm 0.0 ^h	1.34 \pm 0.11 ^{bc}	4.39 \pm 0.03 ^d	0.327 \pm 0.006 ^b	13 \pm 0 ^e	72 \pm 3 ^{de}	555 \pm 4 ^d
Hydro-SCG	7.7 \pm 0.0 ^a	1.98 \pm 0.28 ^d	3.75 \pm 0.16 ^{bcd}	0.327 \pm 0.006 ^b	11 \pm 0 ^c	75 \pm 10 ^e	766 \pm 53 ^{ef}

Different letters in the same column indicate statistically significant differences ($p < 0.05$). SCG, spent coffee grounds; EC₂₅, electrical conductivity measures at 25°C; OC, organic carbon.

Table 4. Zn, Fe and Cu contents in lettuces (mean \pm SD, fresh weight) and bioavailable amounts in soils (mean \pm SD) cultivated/treated respectively, with different SCG treatments.

Sample	Zn (mg/100g)	Cu (mg/100g)	Fe (mg/100g)
Lettuces			
Fresh-SCG	0.47 \pm 0.15 ^a	3.37 \pm 0.79 ^a	12.9 \pm 1.57 ^a
NoBA	0.18 \pm 0.03 ^{bc}	0.81 \pm 0.28 ^c	5.19 \pm 3.96 ^{bc}
NPK	0.14 \pm 0.05 ^b	0.11 \pm 0.06 ^b	2.82 \pm 1.11 ^b
Inc-NoBA	0.14 \pm 0.02 ^{bd}	0.26 \pm 0.06 ^d	4.91 \pm 2.79 ^{bd}
Vermi-Goat	0.22 \pm 0.04 ^{be}	0.31 \pm 0.19 ^{be}	3.21 \pm 1.99 ^{bd}
Baby lettuce	0.32 \pm 0.01 ^f	4.80 \pm 0.25 ^f	1.46 \pm 0.13 ^{bf}
Inc-SCG	0.39 \pm 0.11 ^a	2.69 \pm 0.42 ^a	12.3 \pm 7.47 ^{ab}
Vermi-SCG	0.26 \pm 0.04 ^g	0.35 \pm 0.08 ^g	5.74 \pm 2.83 ^{bg}
Comp-SCG	0.48 \pm 0.13 ^a	1.96 \pm 0.63 ^h	5.60 \pm 1.47 ^{bh}
SCGchar ₂₇₀	0.58 \pm 0.11 ^h	2.40 \pm 0.65 ^a	15.8 \pm 9.22 ^{ab}
SCGchar ₄₀₀	0.21 \pm 0.02 ^{bi}	0.65 \pm 0.11 ⁱ	5.46 \pm 0.80 ⁱ
Ethanol-SCG	0.38 \pm 0.07 ^a	2.26 \pm 0.52 ^a	10.6 \pm 4.52 ^a
Water-SCG	0.47 \pm 0.09 ^a	2.69 \pm 0.46 ^a	12.7 \pm 3.98 ^a
Hydro-SCG	0.44 \pm 0.09 ^a	3.12 \pm 0.86 ^a	17.2 \pm 6.37 ^a
Soils			
	Bioavailable Zn (ppm)	Bioavailable Cu (ppm)	Bioavailable Fe (ppm)
Fresh-SCG	0.189 \pm 0.029 ^a	0.711 \pm 0.021 ^a	0.293 \pm 0.021 ^{ab}
NoBA	0.131 \pm 0.006 ^{bc}	0.776 \pm 0.009 ^c	0.262 \pm 0.009 ^{abc}
NPK	0.132 \pm 0.005 ^b	0.836 \pm 0.017 ^b	0.256 \pm 0.005 ^{ab}
Inc-NoBA	0.126 \pm 0.010 ^{bd}	0.779 \pm 0.008 ^d	0.251 \pm 0.003 ^{bd}
Vermi-Goat	0.560 \pm 0.047 ^e	0.816 \pm 0.023 ^{be}	0.236 \pm 0.025 ^{be}
Inc-SCG	0.163 \pm 0.014 ^f	0.710 \pm 0.033 ^a	0.260 \pm 0.010 ^a
Vermi-SCG	0.419 \pm 0.075 ^g	0.574 \pm 0.050 ^f	0.314 \pm 0.023 ^a
Comp-SCG	0.239 \pm 0.015 ^h	0.623 \pm 0.021 ^g	0.257 \pm 0.009 ^{ab}
SCGchar ₂₇₀	0.134 \pm 0.005 ^{bi}	0.684 \pm 0.024 ^a	0.230 \pm 0.006 ^f
SCGchar ₄₀₀	0.123 \pm 0.002 ^{bj}	0.656 \pm 0.037 ^h	0.214 \pm 0.004 ^g
Ethanol-SCG	0.170 \pm 0.008 ^a	0.709 \pm 0.049 ^a	0.279 \pm 0.032 ^{ab}
Water-SCG	0.177 \pm 0.010 ^a	0.649 \pm 0.021 ⁱ	0.287 \pm 0.013 ^a
Hydro-SCG	0.178 \pm 0.006 ^a	0.958 \pm 0.025 ^j	0.290 \pm 0.010 ^a

For lettuces or soils samples, different letters for superscripts in the same column vs. superscript ^afor fresh-SCG and superscript ^bfor NPK, indicate statistically significant differences ($p < 0.05$). SCG, spent coffee grounds.

Graphical abstract

Highlights:

- Spent coffee grounds (SCG) generate bioamendments with different agronomical behavior.
- Bioamendment from SCG with polyphenols increases the mineral content of lettuces.
- Bioamendment from SCG rises lettuce elements vs. conventional farming (NPK).
- The SCG treatments that eliminate polyphenols increase lettuce biomass.
- Biocharization temperature modifies mineral uptake by lettuce.

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