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Chemical attributes of an Oxisol with the addition of conilon coffee straw biochar

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Abstract – The objective of this work was to evaluate the effects of increasing rates of biochar produced with coffee straw, at two pyrolysis temperatures, on the chemical attributes of an Oxisol cultivated with conilon coffee (*Coffea canephora*) and on the nutrient content of coffee tree leaves. Treatments consisted of pyrolysis at two temperatures (350 and 600°C) and of five biochar rates (0, 5, 10, 15, and 20 Mg ha^{-1}). The following soil chemical attributes were evaluated: pH in water; P, K, Ca, Mg, Al, H+Al, Zn, Cu, Fe, and Mn contents; effective and potential cation exchange capacity (CEC); sum of bases (SB); base (V) and aluminium (m) saturation; and N, P, K, Ca, Mg, Zn, Cu, Fe, and Mn contents in the leaves. The biochar produced at 600°C, at rates of 10 and 15 Mg ha^{-1}, promoted a greater K release into the soil. Regardless of temperature, coffee straw biochar increased K and P availability, sum of bases, base saturation, and CEC in the soil, but did not influence macro- and micronutrient contents in the leaves. The addition of increasing rates of coffee straw biochar in the soil increases P, K, Mg, SB, CEC, and V, regardless of pyrolysis temperature.

Index terms: *Coffea canephora*, coffee farming, organic residues, phosphorus, potassium, pyrolysis.

Atributos químicos de um Latossolo acrescido de biocarvão de palha de café conilon

Resumo – O objetivo deste trabalho foi avaliar os efeitos de doses crescentes de biocarvão produzido com palhada de café, em duas temperaturas de pirólise, nos atributos químicos de um Latossolo cultivado com cafeeiro conilon (*Coffea canephora*), e no conteúdo de nutrientes das folhas do cafeeiro. Os tratamentos consistiram de duas temperaturas de pirólise (350 e 600°C) e cinco doses de biocarvão (0, 5, 10, 15 e 20 Mg ha^{-1}). Avaliaram-se os seguintes atributos químicos do solo: pH em água; teores de P, K, Ca, Mg, Al, H+Al, Zn, Cu, Fe e Mn; capacidade de troca catiônica efetiva e potencial (CTC); soma de bases (SB); saturação por bases (V) e por alumínio (m); e teores de N, P, K, Ca, Mg, Zn, Cu, Fe e Mn nas folhas. O biocarvão produzido a 600°C, nas doses de 10 e 15 Mg ha^{-1}, promoveu maior liberação de K ao solo. Independentemente da temperatura, o biocarvão promoveu o aumento de P e K disponíveis, SB, V e CTC no solo, mas não influenciou os conteúdos dos macro e micronutrientes das folhas. A adição de doses crescentes de biocarvão da palha de café conilon no solo promove aumento de P, K, Mg, SB, CTC e V, independentemente da temperatura de pirólise.

Termos para indexação: *Coffea canephora*, cafeicultura, resíduos orgânicos, fósforo, potássio, pirólise.

Introduction

Coffee growing is the main agricultural activity in the state of Espírito Santo, Brazil and this state is the largest national producer of conilon coffee (*Coffea canephora* Pierre ex Froehner), occupying an area of approximately 460,000 hectares (Ferrão et al., 2021).

Coffee straw is one of the main residuals from coffee bean processing. Each processed coffee bag generates 50 to 60 kg of straw (Matiello et al., 2010). Giving adequate destination to this organic residual is one of the main challenges of the coffee production in Espírito Santo. A regulation published in Espírito Santo, "Portaria no. 23-R", of December 2, 2003 (IDAF, 2003), prohibits the return of raw coffee straw to the plantations because when this residual is disposed on the soil without previous treatment, it may favor proliferation of the stable fly (*Stomoxys calcitrans*). The stable fly irritates and debilitates the animals and is one of the main plagues of the Brazilian livestock farming.

One of the possibilities for the correct use of coffee straw is to transform it in biochar through pyrolysis. Biochars have a conditioning effect on the soil, enhancing chemical, physical and biological conditions and, consequently, plant development (Lehmann et al., 2011). The addition of biochar to the soil promotes increased cation exchange capacity (CEC) (Singh et al., 2015), pH and nutrient availability for the plants (El-Naggar et al., 2015). The low density of biochars reduces soil density, enhancing soil aeration and water infiltration (Uzoma et al., 2011). This highly porous structural conformation with elevated specific surface increases water retention capacity (Méndez et al., 2013) and biological activity, as it creates an excellent habitat for soil microorganisms (Lehmann et al., 2011).

The physical-chemical nature of biochars is highly dependent on the source of raw material and size of particles, as well as on pyrolysis conditions such as the used equipment, temperature, time of permanence at the desired temperature and heating velocity (Singh et al., 2015). Therefore, results obtained with other materials cannot be generalized.

The effect of coffee straw biochars on chemical changes of the soil is still an understudied theme in Espírito Santo. At the same time, coffee growing demands innovative research to generate knowledge and technology on better uses for coffee straw. Either for agronomical or environmental purpose, an economically viable and environmentally correct destination for this residual is necessary.

The objective of this work was to evaluate the effects of increasing rates of biochar produced with coffee straw, at two pyrolysis temperatures, on the chemical attributes of an Oxisol cultivated with conilon coffee and on the nutrient content of coffee tree leaves.

Material and Methods

The experiment was carried out in a greenhouse at the center for agrarian sciences and engineering of the Universidade Federal do Espírito Santo (CCAE-UFES), at the Alegre campus, in state of Espírito Santo, from September 2017 to March 2018. The experimental design was randomized blocks in a 2×5 factorial scheme. Treatments consisted of different pyrolysis temperatures of conilon coffee straw for biochar production (350 and 600°C) and rates of each biochar (0, 5, 10, 15, and 20 Mg ha^{-1}), with five repetitions. The conilon coffee, clone 02, was chosen due to its representativeness in planted areas in Espírito Santo.

Rates of biochar were estimated considering the incorporation of the material in the 0–20 cm soil layer, corresponding to the use of 0, 5, 10, 15, and 20 Mg ha^{-1}, equivalent to 0.0, 2.5, 5.0, 7.5, and 10.0 g dm^{-3}, respectively.

To standardize the effects of soil pH on nutrient availability, treatments were submitted to the incubation curve method, with increasing pH values of all experimental units to 6.1. After determining the necessary quantities of calcium carbonate ($CaCO_3$), the required quantities of calcium chloride ($CaCl_2$) per treatment were calculated, aiming to match the quantities of Ca added by calcium carbonate (Silva, 2017). The combination of necessary $CaCO_3$ and $CaCl_2$ quantities with rates of coffee straw biochar is presented in Table 1.

Conilon coffee straw was dried on cement terrace at the Instituto Federal do Espírito Santo (Alegre campus), in state of Espírito Santo, and was composed of peel, pulp, parchment and beans lost during processing. Coffee straw was carbonized in a pyrolysis reactor installed at the experimental area of the CCAE-UFES. Heating was performed through six electrical resistors of 2.5 w each and a gas condensation system model SPPT-V60. To standardize the material, the biochar was sieved and only the fraction between 1 and 2 mm was used in the experiment. Coffee straw and biochars were subjected to chemical characterization (Table 2).

The soil used was a Latossolo Vermelho-Amarelo distrófico, according to the Brazilian soil classification system (Santos et al., 2018), i.e., Oxisol, sampled at the 0–20 cm layer of a degraded pasture located at the municipality of Alegre, in the state of Espírito Santo. Soil sampling was air-dried, loosened and sieved at 2 mm to obtain air-dried fine soil for chemical and physical characterization analysis (Silva, 2009). The soil presented the following characteristics: pH in water, 4.8; organic matter, 2.02 dag kg^{-1}; P-Mehlich-1, 0.63 mg dm^{-3}; K, 20.3 mg dm^{-3}; Na, 18 mg dm^{-3}; Ca, 0.25 $cmol_c$ dm^{-3}; Mg, 0.23 $cmol_c$ dm^{-3}; Al, 0.51 $cmol_c$ dm^{-3}; H+Al, 3.99 $cmol_c$ dm^{-3}; sum of bases, 0.69 $cmol_c$ dm^{-3}; CEC (T), 0.69 $cmol_c$ dm^{-3}; m, 4.25%; base saturation (V), 14.75%; Zn, 0.491 mg dm^{-3}; Mn, 30.92 mg dm^{-3}; Fe, 27.11 mg dm^{-3}; Cu, 0.312 mg dm^{-3}; sand, 450 g kg^{-1}; silt, 50 g kg^{-1}; clay, 470 g kg^{-1}.

Table 1. Quantity of $CaCO_3$ and $CaCl_2$ added to each treatment (combined pyrolysis temperature and rate) to increase pH to 6.1 and to standardize the levels of Ca in all experimental units.

Rate (g dm^{-3})	Biochar at 350°C		Biochar at 600°C	
	$CaCO_3$	$CaCl_2$	$CaCO_3$	$CaCl_2$
	-------------------------(g kg^{-1})-------------------------			
0.0	1.23	0.00	1.23	0.00
2.5	1.01	0.24	0.95	0.31
5.0	0.94	0.32	0.73	0.56
15.0	0.87	0.40	0.35	0.98
20.0	0.63	0.66	0.00	1.37

Table 2. Total levels of macro- and micronutrients present in conilon coffee (*Coffea canephora*) straw and biochars produced under different pyrolysis temperature (350 and 600ºC)(1).

Variables	Coffee straw	Biochar	
		350ºC	600ºC
N (g kg^{-1})	33.0	31.0	27.0
P (g kg^{-1})	0.70	0.92	1.21
K (g kg^{-1})	60.5	45.0	49.2
Ca (g kg^{-1})	1.15	1.67	1.52
Mg (g kg^{-1})	0.24	0.32	0.38
Cu (mg kg^{-1})	17.1	23.7	36.35
Fe (mg kg^{-1})	92.0	50.0	124.2
Zn (mg kg^{-1})	20.0	22.2	27.2
Mn (mg kg^{-1})	5.20	5.80	8.40

(1)Data obtained through nitroperchloric digestion (Embrapa, 2009).

The experimental units consisted of plastic pots filled with soil (10 dm^3). The mixture of soil and biochar of each treatment was incubated for 21 days, keeping soil moisture close to 60% of the total pore volume. Then, the soil was once more dried and sieved at 2 mm, and conditioned in the vases, where coffee seedlings were planted. Fertilization and irrigation were carried out according to what was indicated for controlled environments (Novais et al., 1991).

From the seedlings planting, the experiment lasted for 180 days. After this period, coffee plants were collected and soil was sampled for analysis. The following soil chemical attributes were analyzed: pH in water, P, K, Ca, Mg, Al, H+Al, Zn, Cu, Fe, and Mn, according to Silva (2009). From the results of the soil chemical attributes the effective (t) and potential (T) cation exchange capacity (CEC) and base (V) and aluminium (m) saturation were calculated. The nutritional diagnosis of N, P, K, Ca, Mg, Zn, Cu, Fe, and Mn was performed on the plant leaves (Silva, 2009).

The effect of the interaction between treatments was subjected to analysis of variance, through the F test, at 5% probability. When interaction effects were significant, means were compared as a function of temperature at 5% probability, through the F test. Linear regression models were fitted for the rates of biochars. When there was not an interaction effect, a simple study of the applied factors was performed, following the same procedures for mean comparisons and regression model fitting. These models were selected based on the significance of the regression coefficients using the Student t test, at 5% probability, and the determination coefficient (R^2). For the analyses, the software SISVAR 5.6 (Ferreira, 2011) was used.

Results and Discussion

There was interaction between the biochars rate (combined with $CaCO_3$) and pyrolysis temperature only for K and isolated effects for the other variables. This result can be explained by the fact that the pyrolysis temperature can affect some chemical composition parameters of the biochars produced and by the chemical constitution of the raw material used for the biochar production, since each type of plant biomass has a different composition, which results in biochars with different chemical compositions (Veiga et al., 2017). Conilon coffee straw had high K concentrations (Table 2), which was a determining factor for the absence of interactions between treatments. Thus, an increase in pyrolysis temperature, combined with the rates, causes interaction only for those soil nutrients, as K, which occurs in higher concentrations in the raw material used for the production of the biochar. According to these results, pyrolysis temperature has a secondary effect on the concentrations of nutrients present in the biochars.

During the carbonization process of organic matter, there are losses of volatile compounds present in the chemical structure of the material, resulting in increased nutrient concentration (Figueredo et al., 2017). However, an increase in pyrolysis temperature causes non-significant increases in the concentration of those nutrients that are present in low concentrations in the raw material. According to these results, plant biomass used in the pyrolysis process is the determining factor in the concentrations of macro and micronutrients present in the chemical composition of biochars.

Coffee straw biochar produced at 600°C increased available K levels and, consequently, increased values of sum and saturation of bases in the soil; however, it did not influence levels of Ca, Mg, Na, P, Zn, Mn, Fe, and Cu available in the soil (Table 3). These results are related to low levels of macro and micronutrients, except for K, in biochars. As coffee straw had low concentrations of macro and micronutrients (Table 2), increasing pyrolysis temperature did not cause significant increases in the levels of these elements in biochar.

Other studies stated that increasing pyrolysis temperature increases nutrient levels in biochars and, consequently, in the soil. In Al-Wabel et al. (2013), increase in pyrolysis temperature promoted significant increases in levels of C, N, P, Ca, and Mg in biochars of shrub residuals. In Yuan et al. (2015), biochars of sewage sludge produced at 700°C promoted increases in Mg, Fe, Ca, and P in the soil, when compared to biochar produced at 350°C. This is an indication that pyrolysis temperature can promote an increase in the levels of macro and micronutrients present in the biochars, as long as the plant biomass used has significant levels of these nutrients in its chemical composition (Enders et al., 2012).

Higher levels of K available in the soil were found in the treatments that received biochar produced at 600°C (Table 3), confirming the assumption that increasing pyrolysis temperature alters only the concentration of those nutrients that are already present in high concentrations in the raw material.

To evaluate the isolated effect of the biochar rates, the regressions for P, Mg, SB, CEC, and V were adjusted. All of these variables showed linear fits, where increasing rates of biochar caused an increase of these variables in the soil (Figure 1). Nonetheless, potential acidity (H+Al) presented a quadratic fit, with the lowest values at biochar rate of 15 Mg ha^{-1}. In Xu et al. (2016), the authors found an increase in available P concentrations after the addition of biochar in weathered soil. However, it should be highlighted that the intensity of these results varies according to the chemical characteristics and applied quantity of the biochar (Zhang et al., 2016).

The increase in available phosphorus levels in the soil promoted by coffee straw biochars was not caused by direct action, as the material evaluated has low phosphorus levels (Table 1). In weathered soils, such as the one used in this experiment, increases in available P concentrations may be related to the presence at the surface of biochars of functional groups

Table 3. Soil chemical attributes with addition of biochars produced under pyrolysis temperatures of 350 and 600°C[(1)].

Soil attributes	350°C	600°C	Probability
pH	5.570a	5.590a	0.5023
P (mg dm^{-3})	17.32a	17.42a	0.7520
Ca (cmol$_c$ dm^{-3})	2.460a	2.470a	0.8760
Mg (cmol$_c$ dm^{-3})	0.285a	0.290a	0.0470
K (mg dm^{-3})	282.4b	299.20a	0.003
Na (mg dm^{-3})	0.056a	0.057a	0.6738
H+Al (cmol$_c$ dm^{-3})	1.900a	1.860a	0.0846
SB (cmol$_c$ dm^{-3})[(2)]	3.510b	3.570a	0.055
T (cmol$_c$ dm^{-3})[(3)]	5.420a	5.440a	0.3928
V (%)[(4)]	64.70b	65.65a	0.0101
Fe (mg dm^{-3})	0.523a	0.515a	0.566
Mn (mg dm^{-3})	9.632a	9.753a	0.610
Fe (mg dm^{-3})	26.18a	25.16a	0.069
Cu (mg dm^{-3})	0.237a	0.268a	0.084

[(1)]Means followed by equal letters in row, do not differ by F test, at 5% probability. [(2)]SB, sum of bases. [(3)]T, potential cation exchange capacity. [(4)]V, saturation of bases.

such as carboxylic and phenolic compounds. These groups may block the phosphorous adsorption sites, increasing the availability of this nutrient in the soil. The high specific surface of biochars and the presence of functional groups are the factors that promote the decrease in P adsorption by the soil (Jiang et al., 2019).

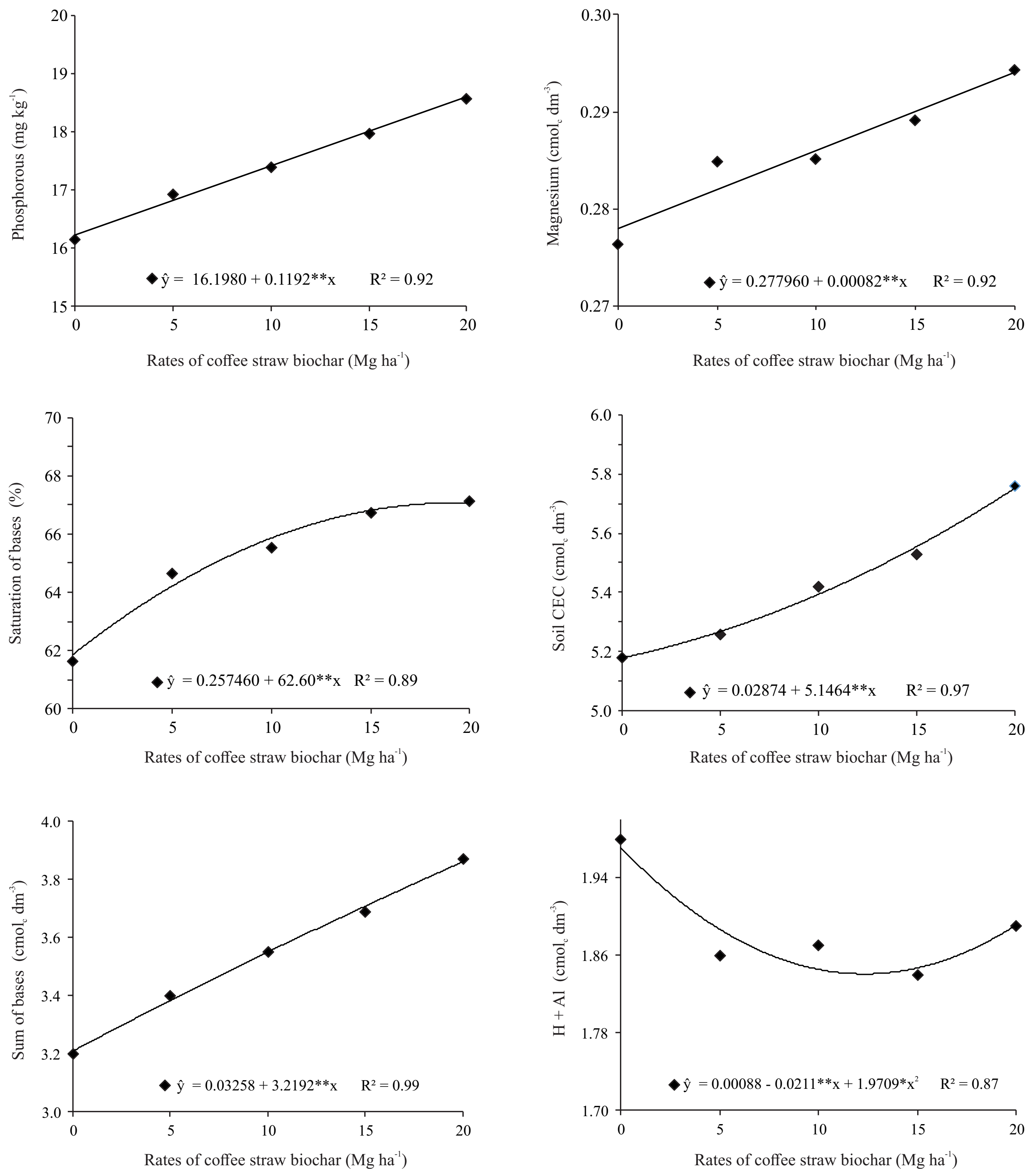

Figure 1. Values of phosphorous, magnesium, H+Al, sum of bases (SB), saturation of bases (V%) and cation exchange capacity (CEC) in the soil as a function of biochar rates. ** and *, significant at 1% and 5% probability, respectively, by t test.

According to the results of the present study, the availability of phosphorous is correlated with the rates of biochar applied. Increases in biochar rates in weathered soils promote increase in available levels of this nutrient (Blackwell et al., 2010). Thus, the effects of coffee straw biochar on the increase of phosphorus availability in the soil are especially important, since most Brazilian soils have a high phosphorus adsorption capacity and the natural sources of this nutrient are non-renewable. However, further studies are needed, especially with coffee straw biochar, for a real understanding of the processes involved in the increase in phosphorus availability caused by the use of biochars (Torres et al., 2020).

An increase in Mg, SB, CEC, and V was verified with increased coffee straw biochar rates, regardless of pyrolysis temperature (Figure 1).

Some authors explained why biochars have a high capacity to increase CEC in the soil. According to Silva et al. (2020), biochars have unique characteristics such as high specific surface area, high cation exchange capacity and abundant functional groups. To Nguyen et al. (2017), the increase in CEC in soils with the addition of biochar is related to the progressive oxidation of the oxygenated functional groups (hydroxyl, carbonyl and carboxyl) present in the aromatic rings, generating a negative charge on the surface of the biochars, and consequently increasing the CEC of the soil. In a situation of low nutrient concentration, this characteristic of biochar is of great importance for the maintenance of fertility (Cunha et al., 2022).

The application of biochars in degraded soils, such as in the present work, can increase CEC and nutrient availability in these areas. The surface charges of biochar compete with inorganic colloids in the soil for the adsorption of P, mitigating the intensity of the adsorption process and, consequently, increasing the availability of P. In addition, biochar has a high CEC and can increase the soil's ability to retain P, and an electrostatically available fraction of K and Mg, consequently increasing the sum of bases (Fonseca et al., 2021).

A different result was obtained for available K in the soil, which varied according to the interaction between biochar rate and pyrolysis temperature. Coffee straw biochar produced at 600°C promoted higher levels of available K in the soil for the rates of 10, 15, and 20 Mg ha^{-1} (Table 4). Among these, the rate of 15 Mg ha^{-1} promoted higher increases in available K, presenting 40 mg dm^{-3} more than the same rate at the temperature of 350°C. However, the highest levels of K in the soil occurred with the use of 20 Mg ha^{-1}. According to Fonseca et al. (2020), coffee straw biochar is responsible for elevating available levels of K in the soil. Without considering the levels of K found in the control, the above-mentioned rates are capable of elevating the levels of K in the soil to values above 200 mg dm^{3}. These values are considered ideal for the good development of the conilon coffee in the south region of Espírito Santo (Ribeiro et al., 1999).

Increased pyrolysis temperature intensifies the loss of volatile compounds of the coffee straw and increases nutrient levels (McBeath et al., 2015). Part of these nutrients may be in available form, therefore, when biochar is incorporated into the soil, there is an increase in the availability of these nutrients (Fonseca et al., 2021), as demonstrated in the present work, in which the biochars produced at 600°C had a greater capacity to increase the levels of K available in the soil.

The increase in available K levels in the soil was directly proportional to the applied rates of biochar produced at 600°C. Rice husk biochar produced at 800°C presented higher concentrations of potassium when compared to biochar produced at 400°C (Tan et al., 2017). According to Figueredo et al. (2017), during the carbonization process of an organic material, there is increase in nutrient concentration.

The ability of coffee straw biochars to increase the availability of nutrients (K and P), sum of bases, base saturation and CEC show that this type of material can be used in coffee plantations. However, further studies

Table 4. Mean values of potassium (K) in the soil as a function of pyrolysis temperature of 350 and 600°C for each biochar rate[(1)].

Rate (g dm^{-3})	K (mg kg^{-1})		Probability
	350°C	600°C	
0	177.7a	165.0a	0.0988
5	232.4a	235.4a	0.6866
10	276.8b	310.2a	0.0001
15	329.0b	369.4a	0.00001
20	395.8b	415.8a	0.0102

[(1)]Means followed by equal letters in row do not differ by F test at 5% probability.

are needed for a better understanding of the causes that led to the results obtained in the present work.

Regarding the nutritional results of the plants, the type of biochar used, produced at 350 or 600°C, did not change the values of macro and micronutrients in the coffee leaves. These data can be seen in Table 5, which presents an analysis of the single effect for the two types of biochar used.

In that Table, there is also the nutritional diagnosis of coffee plants, compared by the critical level method proposed by Costa & Bragança (1996) for conilon coffee leaves. The average values of macronutrients showed that the plants had Mg levels below the recommended. There was a similar finding for all micronutrients.

The inability of coffee straw biochars to change the levels of macro and micronutrients is related to the low concentration of these chemical elements in the biochar, except for potassium (Table 2). Generally, biochars have small concentrations of macro and micronutrients (Colotani et al., 2016). Furthermore, there is great variability in plant responses to the application of biochars to the soil, which are mainly dependent on the type of biochar used and the physical and biological chemical attributes of the soil used for cultivation (Silva et al., 2017). Another factor that cannot be discarded is the nutritional requirement of the plant species, as well as the phenological phase in the evaluation period. Thus, the type and granulometry of the biochar, soil attributes, climatic conditions and nutritional requirements of the crop together make up the pillars that determine the chemical interactions that can increase or even decrease the absorption of nutrients by plants.

Table 5. Means of macro- and micronutrient values in conilon coffee (*Coffea canephora*) leaves compared by the simple effect as a function of production temperatures (350 and 600°C) of the biochar and nutritional status of the coffee plant compared by critical levels[(1)].

Variable	350°C	600°C	Sufficiency range	
		Macronutrients		
N (g kg^{-1})	31.69 a	31.22 a	30.0	Yes
P (g kg^{-1})	1.412 a	1.501 a	1.2	Yes
Ca (g kg^{-1})	12.30 a	11.56 a	11.40	Yes
Mg (g kg^{-1})	2.280 a	2.212 a	3.2	No
K (g kg^{-1})	21.56 a	21.48 a	21.0	Yes
		Micronutrients		
Fe (mg kg^{-1})	107.8 a	108.80 a	131	No
Mn (mg kg^{-1})	54.21 a	54.50 a	69	No
Zn (mg kg^{-1})	11.37 a	12.55 a	12	No
Cu (mg kg^{-1})	8.980 a	8.591 a	11	No

[(1)]Means followed by the same letters in the lines for each variable, do not differ by the F test, at 5% probability.

Conclusions

1. The addition of increasing rates of coffee straw biochar in the soil promotes increase in P, K, Mg, SB, CEC and V, regardless of pyrolysis temperature.
2. The biochar produced at 600°C at rates of 10 and 15 Mg ha^{-1} promotes greater release of K to the soil, in direct proportion to the applied rate.
3. The pyrolysis temperature has a secondary effect on the concentrations of nutrients present in the biochars.

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