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EFFECT OF ORANGE PEEL BIOCHAR ON THE FERTILITY OF THREE TYPES OF SOIL

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ABSTRACT

The orange juice production industry in Veracruz generates a large amount of peel waste, which is both an economic and environmental problem. One way to repurpose these residues is to create biochar, which can be used in agriculture to improve fertility and crop productivity. The aim was to determine how the addition of orange biochar affected the physical and chemical properties of different soils, as well as corn growth (*Zea mays* L.). The experimental design was completely randomized in factorial arrangement (3 x 4): three soil types (clay loam, sandy, and sandy loam), four biochar doses (0, 18, 90, and 180 equivalent to Mg ha^{-1}), and nine replicates per treatment (n = 108). The highest biochar dose presented significant differences ($p \leq 0.05$) in almost all soil physical and chemical variables, except for moisture holding capacity, which varied significantly only in the sandy loam soil with the medium and high doses. In the clay loam soil, the higher dose significantly reduced stem width, aerial biomass, and leaf area. In sandy soil, the medium dose increased stem height and root biomass. In addition, in the sandy loam soil, the highest dose increased stem height and width. The remaining growth variables showed no significant differences between doses or soil types. In conclusion, the use of this biochar altered the physical and chemical properties of the three different soils, as well as having a significant impact on some maize growth variables.

Keywords: utilization, pollution, management, mitigation, pyrolysis, sustainability.

INTRODUCTION

Orange (*Citrus sinensis* L.) is one of the most cultivated fruit species globally. Mexico ranks fifth in global orange production, with an annual total of 4.7 Gg (FAO, 2021). According to García-Salazar *et al*. (2021) 56 % of orange production in Mexico is consumed fresh in homes, 34 % is destined for industrial juice extraction, and the

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remaining 10 % is wasted. During the industrial juice extraction process, a large amount of waste is produced, representing approximately 50 % of the weight of the fruit on a wet basis (Alvarez *et al*., 2018). In Mexico, about 800 000 Mg of orange peel waste is generated each year as a result of the industrial processing of this fruit. The physical and chemical properties of orange peel waste, such as its strongly acidic pH, high moisture content, and the presence of essential oils, make its management complicated (Calabrò *et al*., 2020).

The management and final disposal of large volumes of orange peel waste (RCN) is an economic and environmental problem, particularly in regions where industrial processing of this fruit is a significant economic activity (Cypriano *et al*., 2018). For example, in the central zone of Veracruz, two of the three leading national companies in the industrial extraction of orange juice and other co-products operate. Every year, RCN generation represents a risk of environmental contamination. In 2021 alone, the state of Veracruz produced 50 % of the national supply of this citrus fruit (SIAP, 2021). Currently, open dumps (Figure 1) and biomass composting are the main alternatives for disposing of these wastes (Okino-Delgado and Fleuri, 2016). However, depending on the volume and frequency of discharges, the final disposal of RCN in the environment through these methods represents the emission of large amounts of CO_2, CH_4, and other greenhouse gases. Likewise, waste in open dumps has the potential to release toxic components and decrease air quality due to the decomposition of biomass (Wei *et al*., 2017).

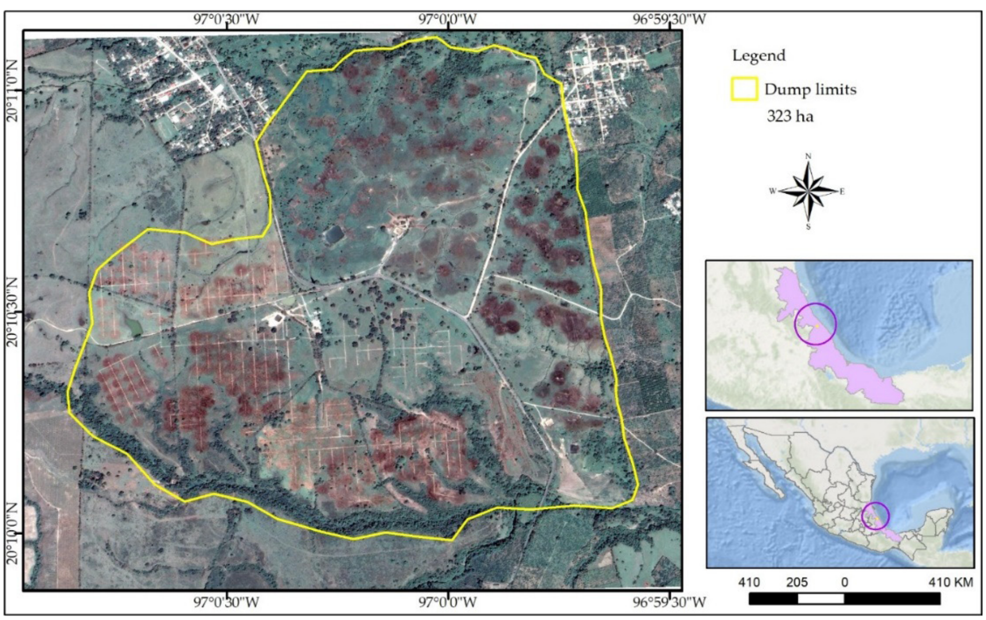

Figure 1. Open dumping site of orange peel waste in the ejido El Nuevo Faisán, San Rafael, Veracruz, Mexico, 2019. Source: Google Earth Pro.

In addition to the gases released, the leachate produced from the disposal of waste into the environment can be a source of contamination for soil and water bodies (Andiloro *et al*., 2021) due to the discharge of organic matter, highly acidic pH, large amount of suspended particles, and high chemical oxygen demand (Malini *et al*., 2018).

Organic wastes in general, particularly from orange peel, represent a biomass resource that can be exploited instead of being disposed of in open dumps (Ortiz-Sánchez *et al*., 2021). These wastes can be used in the production of biochar (Devens *et al*., 2018), which results from the thermochemical conversion of residual biomass in an oxygen-free environment. Biochar can be used as an edaphic amendment since, in synergy with natural soil processes, it counteracts soil degradation, improves fertility, promotes moisture retention, and mitigates climate change through soil carbon sequestration (Lehmann and Joseph, 2015).

Currently, research on the effect of RCN biochar on soil fertility and crop yields is scarce. Studies concerning this particular type of biochar are limited to its characterization in the laboratory, while crop response has only been tested on one type of soil. Therefore, a pot experiment was conducted to test the hypothesis that the application of orange waste biochar will have favorable effects in terms of soil fertility and plant growth. The objective was to determine the effect of orange biochar addition doses on the physical and chemical characteristics of three soil types, as well as the impact on corn growth.

MATERIALS AND METHODS

Biochar production and characterization

For the production of biochar, RCN collected from different juice outlets in the city of Martínez de la Torre, Veracruz, Mexico, was used. This raw material was dried in the sun for seven days until it reached a constant weight and the shells were crispy (with 15 % residual moisture). The biochar was produced at a temperature of 250–300 °C for 180 minutes. For this purpose, a manual pyrolytic rotary kiln with a capacity of 250 L was used. The temperature was recorded with a Raytek MiniTemp model MT2 non-contact thermometer (Santa Cruz, CA, USA). After cooling, the biochar was weighed and crushed with a hand mill. Finally, it was sieved with a 4 mm aperture sifter. The biochar yield was determined through the ratio of the mass of biochar and the feedstock mass:

$$Biochar\ yiel\ (\%) = (P2 / P1)\ x\ 100$$

where $P1$ is the dry weight of the feedstock before pyrolysis and $P2$ is the weight of the orange peel biochar.

Three biochar composite samples were used for characterization. The pH and electrical conductivity were determined by the potentiometric method in a 1:2 soil-water solution. The analysis of extractable metals (Fe, Cu, Zn, and Mn) was performed using the atomic absorption method. The organic matter content (MO, %) was determined by the Walkley and Black method, the nitrogen (N) content by the Kjeldahl method, and the phosphorus (P) content by the Olsen procedure. The determination of exchangeable cations (Ca, Mg, Na, and K) was carried out with ammonium acetate and their quantification using a SHIMADZU Model TOC_VCSN atomic absorption spectrophotometer (Kyoto, Japan). All analyses were performed in accordance with NOM-021-RECNAT-2000.

Experiment

The experiment was carried out at the facilities of the Postgraduate College, Campus Veracruz (19° 11′ 43.82′′ N, 96° 20′ 16.29′′ W, 63 m altitude). The mean temperature recorded in the area was 25.5 °C, with a mean annual precipitation of 1589.6 mm.

Three soil types were used for this study: clay loam, sandy, and sandy loam. The soils used came from agricultural areas and were collected from the top layer (0–30 cm) in three different locations. The clay loam soil was obtained from the lowlands of the Postgraduate College, Campus Veracruz; the sandy soil from the dunes near the city of Veracruz, and the sandy loam from a sugarcane field in the municipality of Zempoala, Veracruz (Figure 2).

The experimental design was completely randomized in a factorial arrangement (3 x 4), resulting in three soil types (clay loam, sandy, and sandy loam) and four doses of biochar application equivalent to 0 (control), 18, 90, and 180 Mg ha^{-1} and nine replicates per treatment (n = 108).

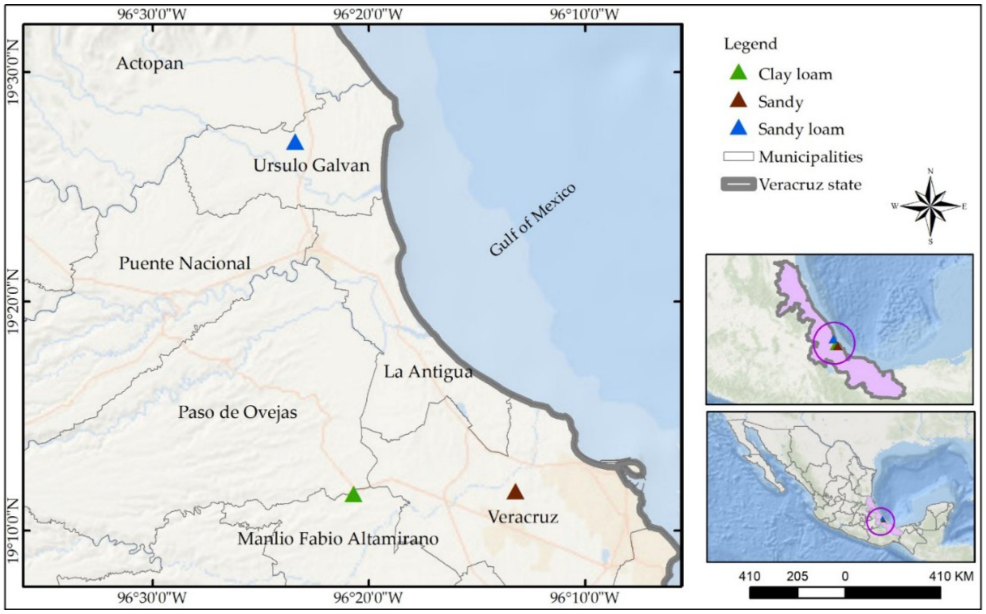

Figure 2. Geographical location of soil collection sites.

The biological material used as an indicator species was maize (*Zea mays* L.). Seeds were germinated in peat substrate in plastic trays. Once the plants reached a height of 15 cm, vigorous individuals of the same height were selected and transplanted into 5 L plastic pots containing the soil and biochar mixture according to each treatment. Subsequently, three composite samples of the substrate were taken for each treatment (n = 36) for physical and chemical characterization. Moisture retention capacity (CRH) was determined by the gravimetric method. The pH, electrical conductivity, cation exchange capacity, organic matter content, total nitrogen, phosphorus, and available potassium were determined by the methods described for the physical and chemical analysis of biochar (NOM-021-RECNAT-2000). Physical and chemical analyses were performed at the Soil Fertility and Environmental Chemistry Laboratory of the Postgraduate College, Campus Montecillo.

Experiment management

The same dose of N-P-K fertilizer (110-60-60) was added to each experimental unit (pot), divided into four similar periodic applications. A daily irrigation was applied at field capacity. The pots were rotated weekly to avoid the microclimate effect.

The corn plants were grown for 90 days (September to December 2021). During the time of the experiment, the average maximum temperature was 31.4 °C, and the average minimum temperature was 21.8 °C. The average humidity during the experiment was 68 %. At the end of this period, each plant was harvested, and the aerial biomass (stems and leaves) and root biomass (root) were separated.

Recording of corn growth variables

Plant height and stem thickness were recorded weekly using a tape measure and a vernier caliper. The leaf area was measured every two weeks. This value was calculated using linear measurements of the width and length of three true leaves per plant. Both values were multiplied, and the product was adjusted using the correction coefficient for corn (0.75), according to Montgomery (1911).

To determine leaf chlorophyll content, CCI (*Chlorophyll Concentration Index*) units were recorded using a CCM-200 plus chlorophyll content meter (Hudson, NY, USA). For each plant, the CCI value of three true leaves was recorded in three sections of the leaf lamina (bottom, middle, and top) because of the spatial variation of chlorophyll along the leaf. The individual CCI value of each plant corresponded to the arithmetic mean of the nine records. Subsequently, the value of leaf chlorophyll content was estimated through the statistical model ($y = -121+129*(CCI)^{0.42}$ $r^2 = 0.82$) according to Parry *et al*. (2014).

Aerial biomass (stems and leaves) and root biomass (root) were measured at the end. To this end, the stem was cut at ground level, and the stem, leaves, and root were separated. For the root system, the root was washed with running water, taking care not to destroy the tissue. The samples were placed on a shaded table to remove excess moisture. The fresh weight of the biomass was recorded on an analytical balance

(0.01 g). Subsequently, each plant part was placed inside labeled paper bags and dried to a constant weight in a forced air circulation oven for 24 h at a temperature of 70 °C. Finally, the dry biomass weight of each plant part was recorded for each experimental unit.

Statistical analysis

Analyses of variance were performed using STATISTICA software. Mean comparison tests were carried out for each edaphic and maize growth variable between each soil type by Tukey's method with a significance level of $p \leq 0.05$.

RESULTS AND DISCUSSION

Biochar characteristics

Biochar production had a 31.3 % yield. The orange peel biochar had a pH of 6.9 and an electrical conductivity of 4.4 dS m^{-1}. The organic matter concentration in the orange peel biochar was 93.2 %. Total carbon (Ct) was 54 % and total nitrogen (Nt) was 0.84 %. The C/N ratio was 68:1. Calcium oxide (CaO) had a concentration of 2.1 %, magnesium oxide (MgO) was 0.3 %, and sodium oxide (Na_2O) was 0.1 %. Potassium oxide (K_2O) and phosphoric anhydride (P_2O_5) had a concentration of 1.7 and 0.4 %, respectively. It is worth noting that the values determined are in the range reported by other authors (Table 1).

Table 1. Characteristics of orange peel biochar discovered in this study, as compared to results from other studies.

Properties	Value found in this study (250–300 °C, 150 min)	Gonzaga *et al.* (2018) (500 °C, 60 min)	Mireles *et al.* (2019) (450 °C, 60 min, atm N_2)	Sial *et al.* (2019) (350 °C, 180 min, atm N_2)
Yield (%)	31.3	34	29	32.5
Humidity (%)	4.9	1.7	-	6.2
pH	6.9	9.7	9.4	9.9
CE (dSm^{-1})	4.4	3.3	7.25	2.9
Ash (%)	6.8	16	5.2	3.4
MO (%)	93.2	-	-	-
Ct (%)	54	66	92	69
Nt (%)	0.8	2.6	-	2.2
C/N	68:1	28:1	-	30:1
CaO (%)	2.1	-	-	-
Na_2O (%)	0.9	-	-	-
K_2O (%)	1.7	-	-	0.05
P_2O_5 (%)	0.4	-	-	0.6

CE: electrical conductivity; MO: organic matter; Ct: total carbon; Nt: total nitrogen; C/N: carbon-nitrogen ratio; CaO: calcium oxide; Na_2O: sodium oxide; K_2O: potassium oxide; P_2O_5: phosphorus oxide.

According to Tomczyk *et al*. (2020), the pH of biochar ranges from 6 to 10, with a tendency to be mostly alkaline and varying based on the feedstock used for production, temperature conditions, and residence time during the pyrolysis process. Mireles *et al*. (2019) determined that orange peel biochar produced at 450 °C within 60 min resulted in a pH of 9.3, while at 600 °C it resulted in a pH of 10. In this study, the temperature was observed in a range of 250 to 300 °C, which explains the difference in pH. The concentration of carbon in biochar can vary from 17 to 88 % (Atkinson *et al*., 2010). The percentage of carbon in orange biochar was 54 %. Therefore, it is in a normal range.

Higher temperatures and longer residence times produce biochars with a higher carbon concentration (Cantrell *et al*., 2012), which may explain the higher carbon content in other studies. The low total nitrogen (Nt) content in orange biochar may be explained by the production method used. Other research involves biochar production in a nitrogenous environment or a pyrolysis process at higher temperatures and different residence times.

Biochars with a carbon concentration ≥ 50 % are considered an alternative to mitigate climate change (Camps-Arbestain *et al*., 2015). Normally, only a small portion (≤ 1 %) is easily degradable; the rest is highly recalcitrant inert material, with a residence time in the soil ranging from hundreds to thousands of years, depending on the biotic and abiotic factors of the site. Therefore, due to the carbon content of the orange peel biochar (54 %), it has the potential to immobilize carbon in the soil.

According to Stella *et al*. (2016), this result is important because it shows that this type of biochar is an effective alternative to increasing carbon in soil and that the pyrolytic process can be used to reduce residual biomass levels. This provides a significant solution for managing and disposing of this agro-industrial waste.

Effect of biochar on the physical and chemical characteristics of soils.

The addition of orange peel biochar at different application rates had a significant effect on the treatments (Table 2). In the clay loam soil, pH was significantly reduced at the 90 and 180 Mg ha^{-1} rates. The pH of the control group (without biochar) was moderately alkaline, while it became neutral when the biochar application rate increased.

The MO content in the control group and the 18 and 90 Mg ha^{-1} dose treatments remained at a medium level. However, with the 180 Mg ha^{-1} dose, the value became very high. The values of the macronutrients Nt, P, and K increased significantly as the biochar application rate escalated. Based on the classification of the standard, these soils have a low content of these elements. CE and CIC also recorded a significant increase as the biochar application rate climbed. As for CRH, there was no significant difference between treatments.

In the case of the sandy soil, pH decreased significantly with an increasing biochar application rate. The strongly alkaline pH of the control group was neutralized with the 180 Mg ha^{-1} dose. The control group and the 18 and 90 Mg ha^{-1} treatments had very low to low pH. However, with the highest dose, pH increased significantly and was

Table 2. Effect of biochar application rate on soil physical and chemical properties.

Soil	Dose (Mg ha^{-1})	pH	CE	MO	Nt	P	K	CIC	CRH
Clay loam	0	7.6 a	0.08 c	1.7 b	0.09 c	8.8 d	0.4 c	19.7 c	45 a
	18	7.4 a	0.13 b	1.3 b	0.08 c	12.7 c	0.7 c	24.7 b	50.7 a
	90	7.2 b	0.13 b	1.9 b	0.12 b	26.7 b	2 b	24 b	52 a
	180	7.1 b	0.75 a	8.5 a	0.17 a	54.4 a	3.1 a	27.8 a	55 a
Sandy	0	8.4 a	0.08 b	0.08 b	0.04 b	7.2 c	0.4 c	37.4 b	22.7 a
	18	7.9 b	0.13 b	0.9 b	0.03 b	8.1 c	0.6 c	39.7 a	23.5 a
	90	7.4 c	0.13 b	0.9 b	0.07 b	20.3 b	1.7 b	37.4 b	28.7 a
	180	7.0 d	0.66 a	7.35 a	0.17 a	55.5 a	3.3 a	39.6 a	32.3 a
Sandy loam	0	6.5 b	1.03 b	2.39 b	0.12 b	41.5 b	3.2 c	30.5 b	57 b
	18	6.9 ab	0.13 c	2.27 b	0.15 b	26.7 b	4.7 ab	34 b	54.3 b
	90	7.4 a	0.13 c	2.57 b	0.16 b	45.7 b	5.2 a	38.7 a	63.7 a
	180	7.1 a	1.47 a	9.8 a	0.28 a	85.8 a	4.2 b	33.5 b	65 a

Means with different letters for each variable indicate significant differences between treatments (Tukey, $p \geq 0.05$). pH: hydrogen potential; CE: electrical conductivity (dS m^{-1}); MO: organic matter (%); Nt: total nitrogen (%); P: available phosphorus (mg kg^{-1}); K: available potassium (mg kg^{-1}); CIC: cation exchange capacity (meq 100 g^{-1}). CRH: moisture retention capacity (%).

classified as soil with a very high MO content. For CE and Nt, statistical differences were observed with the 180 Mg ha^{-1} dose. Phosphorus and potassium concentrations increased with the 90 Mg ha^{-1} dose of biochar, while CIC increased with the 18 and 180 Mg ha^{-1} doses. As in the clay loam soil, CRH did not increase with the addition of biochar at different doses.

In the sandy loam soil, the 90 and 180 Mg ha^{-1} doses significantly increased pH compared to the control group. However, they always remained in a neutral classification. Soil CE increased significantly with the increase in the application rate, reaching values of 1.47 dS m^{-1} with the 180 Mg ha^{-1} rate. While with the control group and the 18 and 90 Mg ha^{-1} dose treatments, the effects of salinity were negligible, and with the highest dose, it became slightly saline. Regarding MO, the control group and the 18 and 90 Mg ha^{-1} treatments had medium levels of MO, but the higher dose increased this variable, resulting in a soil with very high MO content.

The averages for Nt and P increased with the 180 Mg ha^{-1} dose. However, in the case of K and CIC, they increased significantly from the 18 Mg ha^{-1} dose. Doses 90 and 180 Mg ha^{-1} increased moisture retention by 14 % in comparison to the control group. Abdelaal *et al*. (2021) report that the orange peel biochar doses of 36 and 108 Mg ha^{-1} in sandy soil did not increase CRH, and such absence of variation among the different doses can be attributed to the reduction of pore density in the biochar when it was crushed in the mill.

Effect of biochar on corn growth

In the clay loam soil, the addition of orange peel biochar at a dose of 180 Mg ha^{-1} significantly reduced stem width by 8 %, aerial biomass by 46 %, and leaf area by 13 %, compared to the control group. Stem height, root biomass, and chlorophyll content did not show significant differences with the addition of biochar at any application rate.

In the sandy soil, the 18 Mg ha^{-1} dose increased stem height by 20 % and root biomass by 68 % compared to the control group. For the rest of the variables, the addition of biochar had no significant effect. In the sandy loam soil, the dose of 180 Mg ha^{-1} increased plant height and stem thickness by 31 and 7 %, respectively, in contrast to the control group. However, for the rest of the variables, the addition of biochar had no significant effect (Table 3).

In general, the addition of biochar had no significant effect on leaf area or chlorophyll content. The latter, in maize plants, ranged from 298 to 433 μmol m^{-2}. These values were within the normal range, according to Parry *et al.* (2014) and studies on maize plant health (Brewer *et al*., 2022). In another study, the application of 20 Mg ha^{-1} of orange peel biochar increased corn biomass by 16 % compared to the control treatment (evaluating only loam soil), but when increasing the dose to 60 Mg ha^{-1}, biomass decreased by 15 % (Gonzaga *et al*., 2018). This biochar was produced at 500 °C for 60 min and the type of soil used was different.

Orange peel biochar produced at 200 °C for 8 h and applied at doses of 90, 180, and 270 Mg ha^{-1} did not promote maize growth or affect the moisture holding capacity of clay soil, and had a negative effect on germination (Kalderis *et al*., 2019). Given

Table 3. Effect of biochar application rate on growth indicators and chlorophyll content in maize plants grown in different soils and orange peel biochar applications.

Soil	Dose (Mg ha^{-1})	Height (cm)	Width (cm)	Aerial biomass (g per pot)	Root biomass (g per pot)	Leaf area (cm^{-2})	Chlorophyll content (μmol m^{-2})
Clay loam	0	223.7 a	1.3 ab	147.3 a	30 a	492.5 bc	433 a
	18	226.1 a	1.5 a	152.7 a	37.1 a	571 ab	414.6 a
	90	216.8 a	1.5 a	151.4 a	45.1 a	616.4 a	425.2 a
	180	186.3 a	1.2 b	78.9 b	27.8 a	430 c	416.5 a
Sandy	0	160 ab	0.9 a	38.7 a	14.8 ab	318.4 a	328 a
	18	191.8 a	1 a	66 a	24.8 a	400.6 a	332 a
	90	138.3 b	1 a	48 a	9.9 b	311.9 a	298 a
	180	162 ab	0.9 a	41.9 a	12.4 b	306 a	361.4 a
Sandy loam	0	200 b	1.5 b	166.8 a	26.4 a	512.7 a	407.2 a
	18	245 a	1.4 b	166.8 a	24 a	533.4 a	422.5 a
	90	231 ab	1.5 ab	182.5 a	37 a	581.6 a	404.4 a
	180	262.1 a	1.6 a	166.6 a	34.8 a	587 a	387 a

Means with different letters for each variable indicate significant differences between treatments (Tukey, $p \geq 0.05$).

the discrepancy in the recorded responses, further research on the application of this product in agriculture is recommended.

During the pyrolytic process, D-Limonene, an essential oil present in orange peel, contributes to the release of volatile organic substances that, in the cooling phase, condense as a liquid on the surface of the biochar or become trapped inside the pores. This compound can have a negative effect on plant growth (Han *et al*., 2022). To increase its long-term use in agricultural crop production, contaminants must be controlled and removed from the feedstock.

Negro *et al*. (2017) determined that managing 1 Mg of RCN on a dry basis in an open dump has the same cost as carbonizing it (100 € Mg^{-1}, equivalent to $1890 MXN at the current exchange rate). Carbonization of this waste can sequester 5.5 kg CO_2 eq Mg^{-1}, while in the open dump, 361 kg CO_2 eq Mg^{-1} are emitted to the environment. Moreover, carbonizing the waste reduces its volume by at least 80 % (Kim *et al*., 2020). Adeniyi *et al*. (2020) emphasize that, due to its porosity and high adsorption capacity, orange biochar is a high-quality product that can be used in a variety of applications, such as an adsorbent for aquatic pollutants or soil amendment.

Carbonization of RCN can be an alternative for the management and final disposal of waste generated by the juice industry. However, a life cycle analysis is required to determine the environmental and financial viability of this waste utilization strategy before its use is considered. In addition, it is necessary to determine the presence of toxic metals and other contaminants that could have a negative impact on the environment. In future research, it is necessary to pay attention to the particle size of biochar. Also, it is advisable to evaluate lower doses and their profitability for small and medium producers, as well as to experiment with other plant species that have different nutritional demands.

CONCLUSIONS

The application of biochar from orange peel residues at different doses significantly modified the physical and chemical characteristics of the three soils. Regarding the growth of corn plants, the addition of biochar at different doses had significant effects on growth. In the clay loam soil, the highest dose significantly reduced stalk width, aerial biomass, and leaf area. In sandy soil, the medium dose increased stem height and root biomass. Finally, in the sandy loam soil, the highest dose increased stem height and width. The rest of the growth variables did not show significant differences between doses and soil types.

In general, biochar from orange peel residues is an inefficient soil amendment for corn cultivation. However, producing biochar from orange peel waste and using it in agriculture can be a viable alternative to increasing soil carbon and reducing residual biomass volume via the pyrolytic process.

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