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Thermal treatment of poultry litter: Part II. Evaluation of structural and morphological characteristics¹

Tratamento térmico da cama de aviário: Parte II. Avaliação das características estruturais e morfológicas

Josely D. Fernandes², Hugo O. C. Guerra², Lúcia H. G. Chaves^{2*},
Laysa G. de S. Laurentino², Antônio R. Cavalcante² & Gustavo T. M. Kubo²

¹ Research developed at Universidade Federal de Campina Grande, Centro de Tecnologia e Recursos Naturais, Campina Grande, PB, Brazil

² Universidade Federal de Campina Grande/Unidade Acadêmica de Engenharia Agrícola, Campina Grande, PB, Brazil

HIGHLIGHTS:

Mesoporous structures predominate in all samples.

Surface area and pore volume increase due to the heat treatment.

Poultry litter subjected to 250, 350 and 450 °C shows similar physical, chemical and structural characteristics.

ABSTRACT: This study aimed to investigate the effect of heat treatments on the total pore volume, surface area of pores, structural characteristics, and functional groups of the materials which derive from the carbonization and pyrolysis of poultry litter. These processes were carried out in porcelain containers containing poultry litter samples, which were then placed in a muffle furnace. The treatments corresponded to three temperatures: samples carbonized at 250 °C and pyrolyzed at 350 and 450 °C. The surface porosity of biochars provides a suitable dimension to improve the water holding capacity, and surface functional groups may help to improve soil fertility. The influence of the temperatures on the pores of the biochar was investigated by using scanning electron microscopy with energy-dispersive x-ray spectroscopy, X-ray diffraction, and Fourier-transform infrared spectroscopy. The characterization of the biochar shows mesoporous structures, as well as increased surface area and pore volume. The chemical composition has potassium at higher concentrations than other metals, while similar surface functional groups were found in the biochar, such as phenolic, aliphatic, conjugated quinones, and OH-phenolic. The thermal treatments applied to poultry litter generated samples with similar physical, chemical, and structural characteristics. Thus, the production of biochar by the farmer and its use as a soil conditioner can contribute to the reduction of agricultural pollution, management, and efficient disposal of residual biomass.

Key words: BET surface area, pore volume and surface, chemical composition, functional groups

RESUMO: Este estudo teve como objetivo investigar o efeito dos tratamentos térmicos no volume total dos poros, na área superficial dos poros, nas características estruturais e nos grupos funcionais dos materiais derivados da carbonização e pirólise da cama de aviário. Esses processos foram realizados em recipientes de porcelana contendo amostras de cama de aviário e colocados em uma mufla. Os tratamentos corresponderam a três temperaturas: amostras carbonizadas a 250 °C e pirolisadas a 350 e 450 °C. A porosidade superficial dos biocarvões fornece uma dimensão adequada para melhorar a capacidade de retenção de água, e os grupos funcionais da superfície podem ajudar a melhorar a fertilidade do solo. A influência das temperaturas nos poros do biocarvão foi investigada por meio de microscopia eletrônica de varredura com espectroscopia de energia dispersiva, difração de raios-X e espectroscopia de infravermelho com transformada de Fourier. A caracterização do biocarvão mostra estruturas mesoporosas, aumento da área superficial e volume dos poros. A composição química evidencia potássio em concentrações mais elevadas do que outros metais, enquanto grupos funcionais de superfície semelhantes foram encontrados no biocarvão, como fenólico, alifático, quinonas conjugadas e OH-fenólico. Os tratamentos térmicos aplicados à cama de aviário geraram amostras com características físicas, químicas e estruturais semelhantes. Assim, a produção de biocarvão pelo agricultor e seu uso como condicionador do solo podem contribuir para a redução da poluição agrícola, manejo e disposição eficiente da biomassa residual.

Palavras-chave: área de superfície BET, volume e superfície dos poros, composição química, grupos funcionais

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* Corresponding author - E-mail: lhgarofalo@hotmail.com

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INTRODUCTION

Poultry litter (PL), a solid waste resulting from chicken rearing, refers to the mixture of poultry manure and bedding material from poultry farms. This material is considered an organic fertilizer; however, it can pollute agricultural soils due to the presence of pathogens, antibiotics, heavy metals, and pesticides. Therefore, poultry litter can be converted into biochar using pyrolysis, resulting in a safer and more effective alternative to utilize this type of agricultural waste (Joardar et al., 2020).

Pyrolysis temperature is the most crucial parameter in biochar production due to the release of volatiles and formation and volatilization of intermediate melts (Lehmann & Joseph, 2015). These affect the porosity, surface structure, and forms of the functional group of biochar, which are deciding factors for its ability to adsorb pollutants (Nguyen et al., 2019).

The exact chemical and structural composition is dependent on the combination of raw material and pyrolysis conditions. It is known that the incorporation of biochar into the soil, including poultry litter biochar, produced at 350 and/or 450 °C, can influence its physicochemical properties by decreasing the acidity and density of the soil, increasing the nutrient content, porosity, and available water, besides acting as an adsorbent of heavy metals (Chaves et al., 2018; 2020; 2021). However, it is important to check whether the material carbonized at 250 °C has the same effect on soils as the biochars produced at the above-mentioned temperatures.

Since the temperature used in the thermochemical conversion of poultry litter influences the characteristics of the materials produced (carbonized poultry litter (250 °C) and pyrolyzed poultry litter (350 and 450 °C)), this study aimed to investigate the effect of temperature on the total pore volume, surface area of pores, structural characteristics and functional groups of these materials which derive from poultry litter.

MATERIAL AND METHODS

The raw material used to produce the biochar was broiler poultry litter collected at the State University of Paraíba experimental farm, in the municipality of Lagoa Seca (07° 09' 22.42" S; 35° 52' 09.64" W). The preparation of the raw material and the thermal process used in this work with three temperatures of thermal degradation, 250, 350 and 450 °C, in triplicate, following a heating rate of 10 °C min⁻¹, as well as the carbonization procedure, were published in Part I of this research (Fernandes et al., 2022).

CPL250 (poultry litter carbonized at 250 °C), PPL350, and PPL450 (poultry litter pyrolyzed at 350 and 450 °C, respectively), were analyzed by SEM, EDS, N₂ adsorption-

desorption isotherms (surface area, pore volume, and diameter), and FTIR.

Scanning Electron Microscopy - SEM (Hitachi TM-1000) was used to visualize the morphology of the characters to obtain an impression of the pores of the biochar. Thus, samples obtained in the different thermal processes were analyzed with point scans using energy-dispersive x-ray spectroscopy (EDS) (Clemente et al., 2018). In this way, it was possible to check the elements present on the surface of the biochars.

The textural property of the biochar was analyzed via nitrogen adsorption-desorption isotherms. The apparatus used was the Micromeritics ASAP 2420 LAMM at CETENE. The surface area was calculated according to the Brunauer - Emmett- Teller (BET) method, while the pore-size distribution was obtained according to Barrett et al. (1951).

Fourier Transform Infrared Spectroscopy (FTIR) analysis was performed on the Perkin Elmer FTIR, Spectrum 400 series, with the Perkin Elmer software. The analysis was conducted in the medium infrared, and the samples were prepared in potassium bromide (KBr) tablets. For this, the sample was macerated to facilitate the preparation of the pellets and for better interaction of the infrared beam with the sample, thus obtaining an optimal wavelength range and resulting in more reliable results.

X-ray diffraction analyses were conducted at room temperature in an XRD-7000 Shimadzu apparatus, using copper K- α radiation (1.5418 Å), 40 kV voltage, and 30 mA current. The biochar was examined at an interval of 2 θ between 10 and 80.0 degrees at a speed of 1° min⁻¹.

RESULTS AND DISCUSSION

The thermal degradation process influenced all parameters described in Table 1. With the exception of pore size, all other parameters were increased with the temperature rise from 250 to 450 °C. As a result, the samples CPL250, PPL350, and PPL450 had surface area (SA) of 1.301, 3.375, and 3.444 m² g⁻¹, respectively. These results corroborate those reported by Song & Guo (2012), who studied poultry litter biochar and obtained SA of 2.68 and 3.94 m² g⁻¹ at 300 and 400 °C, respectively.

The pyrolysis of the raw material leads to a loss of mass in the form of volatile compounds that include the loss of hemicellulose, cellulose, and lignin, forming compounds with high carbon content, as can be seen in Part I of this paper (Fernandes et al., 2022). With the loss of these compounds, macro-, meso-, and micropores are formed, which increase the specific surface area of the biochar. Therefore, the SA increase as a function of temperature rise was expected due to the release of volatiles (Liu et al., 2017). In addition, biochar samples with larger surface areas, due to their porosity, when applied

Table 1. Brunauer-Emmett and Teller (BET) surface area and porosity of the poultry litter carbonized at 250 °C (CPL250) and pyrolyzed at 350 °C (PPL350) and 450 °C (PPL450)

Sample	BET surface area (m ² g ⁻¹)	Total pore volume (cm ³ g ⁻¹)	Microporous surface area (m ² g ⁻¹)	Microporous pore volume (cm ³ g ⁻¹)	Average pore width (nm)
CPL250	1.301	0.0041	1.237	0.00050	29.06
PPL350	3.375	0.0129	0.238	0.00003	14.83
PPL450	3.444	0.0128	1.453	0.00060	16.10

to the soil, improve its structure, significantly increasing the total water retention capacity and serving as a habitat for soil microorganisms (Lehmann & Joseph, 2015).

The specific surface area of biochar, in sandy soils, can also somewhat provide cation exchange capacity (Zhou et al., 2020), which is highly dependent on chemically reactive sites. These sites, in biochar, are formed over the years as the particles are attacked by microorganisms in the soil, changing the chemical and physical characteristics of the surface (Cohen-Ofri et al., 2006).

The presence of pores in the poultry litter carbonized at 250 °C (Table 1) indicated that volatile gases trapped within the original biomass were released (Zhao et al., 2017). With the temperature rise (250 to 450 °C), the porous structure and the total pore volume increased by 212.19%.

The lower values of surface area and pore volume of micropores in the PPL350, when compared with CPL250 and PPL450 treatments, indicated that the formation of these structures does not always occur in a uniform and

homogeneous order, or because, when the raw materials were partially carbonized, the volatile material is still contained in these structures, not increasing pore volume.

The presence of micropores may help to improve the moisture content in the soil. The mesopores and macropores (pores with medium and large diameter) primarily favor liquid-solid adsorption (Shaaban et al., 2013).

The morphological analysis allowed the samples to be analyzed for mass, size, shape, and structure. When analyzing the scanning electron microscopy (SEM) image of the surface of the poultry litter carbonized at 250 °C (CPL250) and pyrolyzed at 350 °C (PPL350) and 450 °C (PPL450), it was possible to observe an evolution of microstructural rearrangements such as cracks and pores due to the increase in temperature used in the process (Figures 1A, 1B, 1C, 1D, 1E, 1F, 1G, 1H, 1I, 1J, 1K, 1L).

The presence of pores in the CPL250 treatment (Figures 1A, 1D, 1G, 1J) indicated that at 250 °C, there was a release of

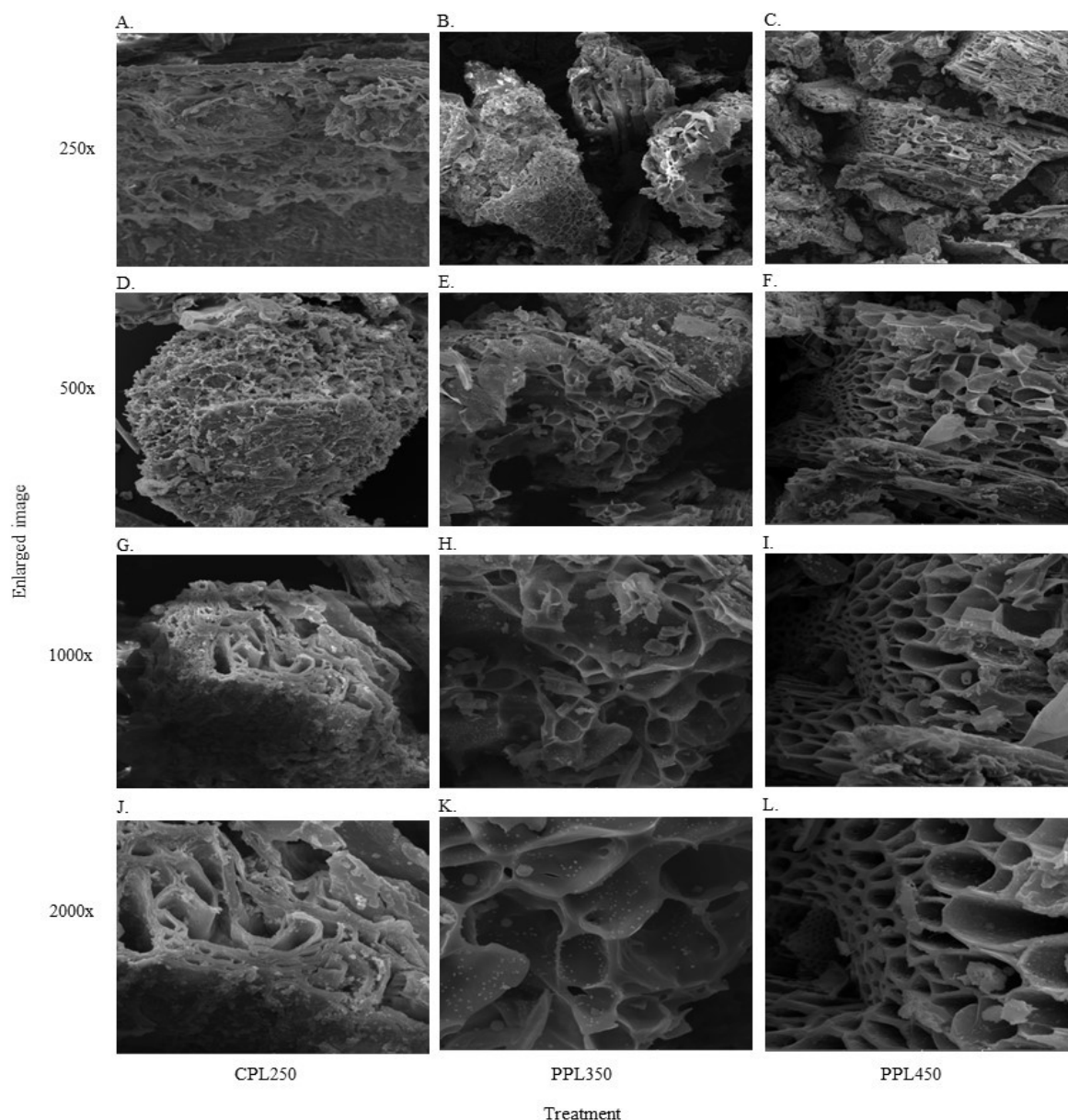


Figure 1. Scanning electron microscopy of the poultry litter carbonized at 250 °C (A, D, G, and J) and pyrolyzed at 350 °C (B, E, H, and K) and 450 °C (C, F, I, and L). Image magnified 250x (A, B, and C), 500x (D, E, and F), 1000x (G, H, and I) and 2000x (J, K, and L)

volatile gases exhausted by the biomass of origin, corroborating the findings of Zhao et al. (2017). In addition, the porous structure increased as a function of the temperature employed, as described in Table 1, but not in uniform and homogeneous order, possibly also indicating volatile material attached to the structure as mentioned in the item earlier about porosity.

The more significant pores observed in the biochar produced at 450 °C (PPL450) resulted from the more significant loss of volatile materials (VM) (Figures 1C, 1F, 1I, and 1L). The lower VM content (36.7%) verified in PPL450 showed its more significant loss during pyrolysis, whose value differed when compared to the other treatments, that is, 42 and 40.1% of VM in CPL250 and PPL350, respectively. According to Downie et al. (2009), the difference in pore sizes in the biochar as the temperature increases is a consequence of the release of volatile materials and the resistance of the structures of the poultry litter to thermal degradation, such as cell walls and conductive vessels.

The presence of pores in all the analyzed treatments is important data since these structures influence the transport of water and consequently the adsorption of metals, aeration, and water retention, as well as allowing the movement of plant roots (Restuccia et al., 2019). Regardless of the sample, biochar

has a pore size of 2-50 nm, characteristic of mesopores, which are helpful for liquid-solid adsorption.

According to Chaves et al. (2018), increasing doses of biochar produced from poultry litter wastes, subjected to pyrolysis at 350 °C, influenced soil physical characteristics, that is, improved these characteristics by modifying the granulometric analysis, led to a decrease in bulk density, an increase in total pore volume as well as an increase in water content mainly at matric potential of 0.5065 MPa.

The samples obtained in the different thermal processes were analyzed via energy-dispersive x-ray spectroscopy (EDS) using point scans (Figures 2A, 2B, and 2C) to check the elements present on the surface of the biochars (Figures 2D, 2E, and 2F) (Clemente et al., 2018).

In this type of analysis, the identified elements can be contained in the surface wall of the samples and in the ashes generated during the thermal degradation. It is also important to note that, during this process, the elements present in the original substrate (poultry litter) are concentrated due to the loss of C, H, and O (Figueredo et al., 2017).

The elements sodium (Na), magnesium (Mg), chlorine (Cl), potassium (K), calcium (Ca), manganese (Mn), iron

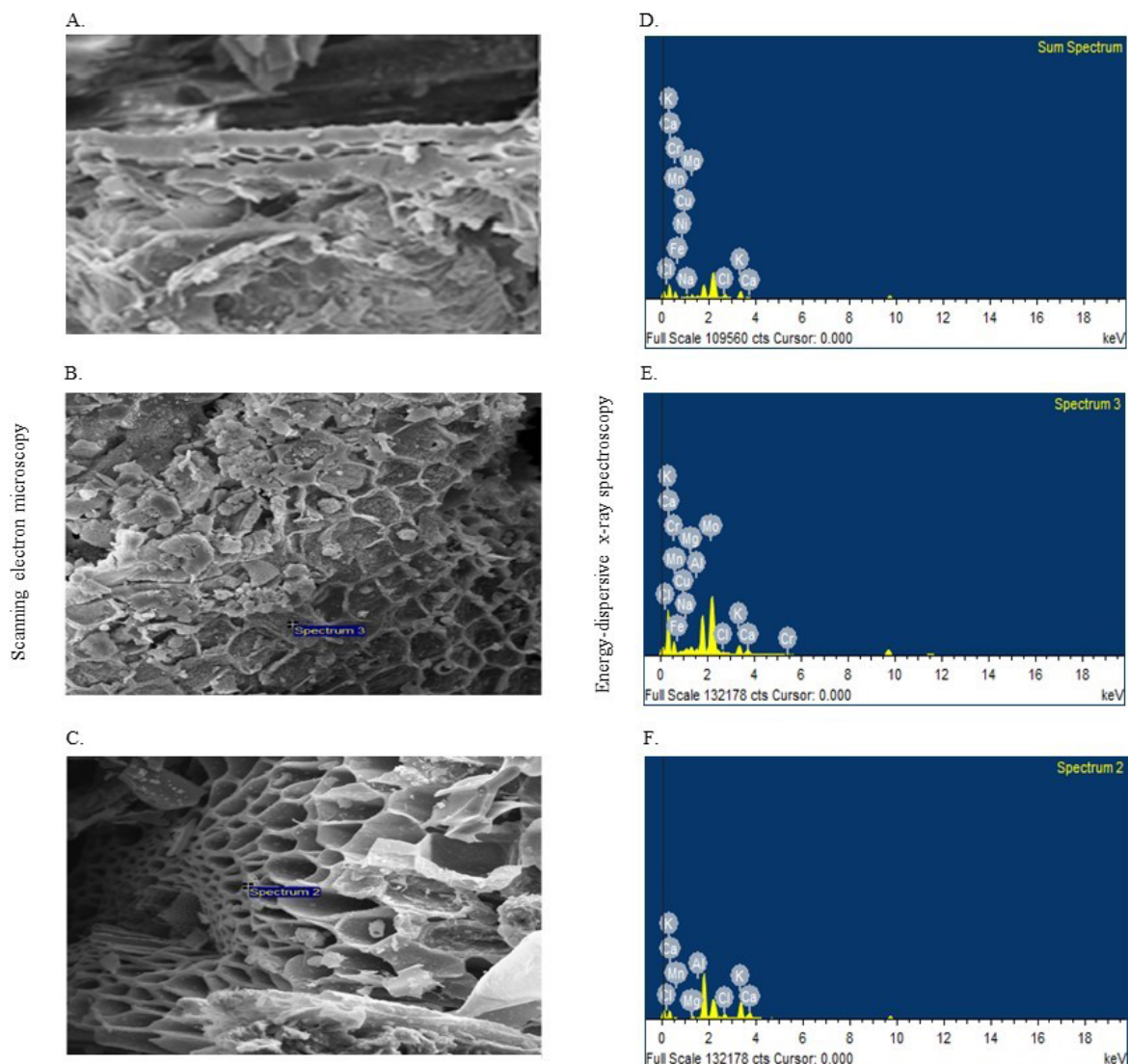


Figure 2. Scanning electron microscopy and energy-dispersive x-ray spectroscopy corresponding to point scans marked for the poultry litter carbonized at 250 °C (A and D) and pyrolyzed at 350 °C (B and E) and 450 °C (C and F)

(Fe), copper (Cu), and aluminum (Al) were identified in the different treatments (Figure 3). Regardless of the temperature employed, the samples showed a high potassium content in their constitution, whose values increased as a function of temperature, varying between 43.67 and 54.34% at 250 and 450 °C, respectively. The other elements also showed variation in their contents as a function of the temperature used, having the highest values at the following temperatures: Cl (23.96%) and Al (2.64%) at 450 °C; Ca (17.98%), Na (9.83%), and Cu (5.0%) at 350 °C and Mg (13.0%) and Fe (4.70%) at 250 °C. Manganese corresponded to only 0.38, 0.25 and 0.41% of the

elemental composition of the samples obtained at 250, 350, and 450 °C, respectively.

These results corroborate those reported by Sikder and Joardar (2019), who found that poultry litter biochar has mineral elements, such as P, K, Ca, Fe, and Mg, in its constitution. On the other hand, phosphorus (P), despite having a high content in poultry litter biochar (Chaves et al., 2020), was not identified by EDS, regardless of the temperature used. This result suggested that P may be associated with complex organic compounds within the cell, relatively stable at low degradation temperatures (Lehmann & Joseph, 2015). However, after 100 days of incubation with increasing doses of biochar in the Ultisol and Oxisol, these soil samples, analyzed for available soil phosphorus, showed that biochar is a potential source of phosphorus, particularly for weathered soils (Mendes et al., 2015). Furthermore, regarding the study of heavy metals adsorption by biochar, it was observed in these same samples that biochar increased zinc adsorption after this same incubation period (Lima et al., 2017). Likewise, the presence of biochar in the soil influences the adsorption of cadmium (Chaves et al., 2021) and increases soil fertility, influencing the growth, development, and yield of crops (Mendes et al., 2021).

According to X-ray diffraction, the XRD patterns of the poultry litter carbonized at 250 °C (CPL250) and pyrolyzed at 350 °C (PPL350) and 450 °C (PPL450) were similar to each other, showing broad peak patterns with some degree of short order. This result indicates amorphous material with overlapping peaks (Figure 4). Such characteristics made it difficult to assign smaller peaks to specific minerals, corroborating the results obtained by Clemente et al. (2018).

It is observed that the strongest peaks at 2θ 28.346° ($d = 3.146 \text{ \AA}$) and 40.509° ($d = 2.225 \text{ \AA}$) indicate the presence of inorganic components with the potassium element in its constitution such as sylvite [KCl, Powder Diffraction File (PDF) 041-1476] and at 2θ 28.790° ($d = 3.098 \text{ \AA}$) for potassium

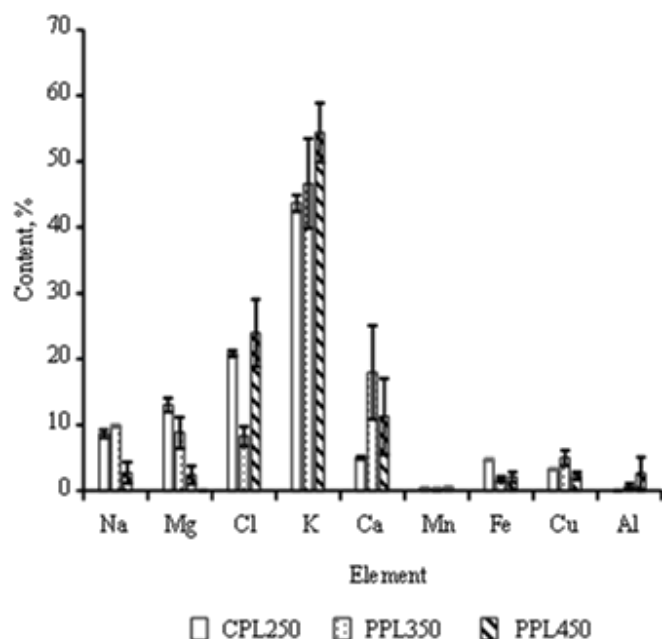


Figure 3. Semi-quantitative analysis by EDS of the surface elements of the poultry litter samples carbonized at 250 °C (CPL250) and pyrolyzed at 350 °C (PPL350) and 450 °C (PPL450)

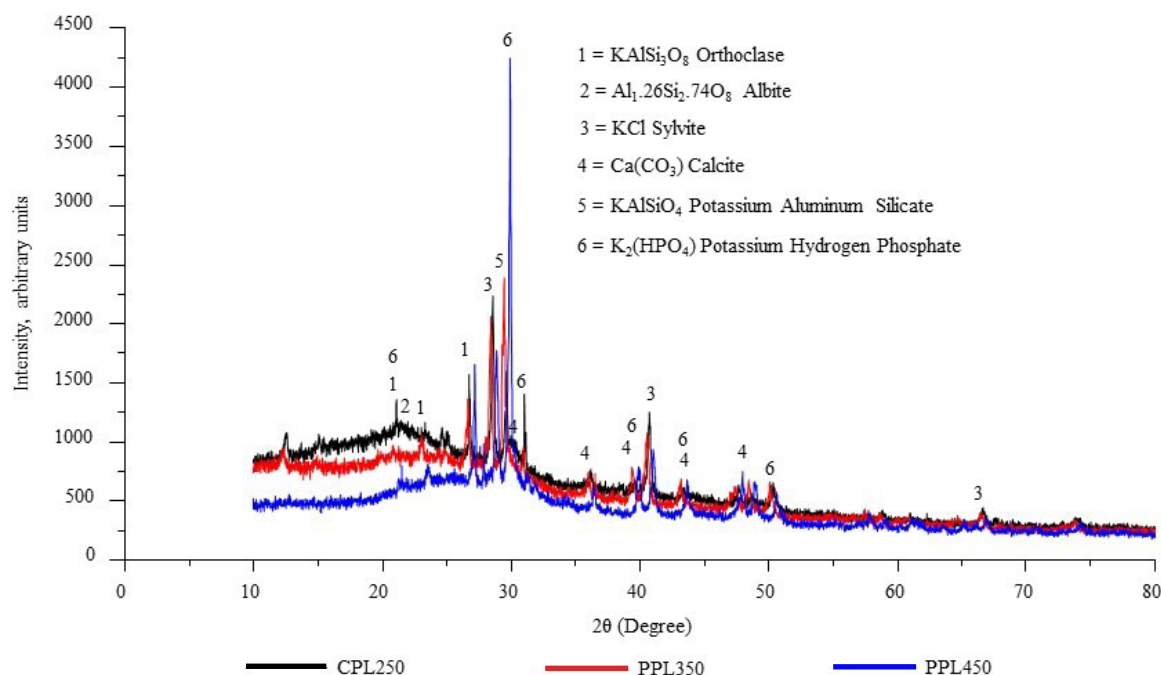


Figure 4. X-ray diffraction pattern of samples of poultry litter carbonized at 250 °C (CPL250) and pyrolyzed at 350 °C (PPL350) and 450 °C (PPL450)

aluminum silicate (KAlSi_3O_8). The presence of potassium minerals justifies the high concentration of this element in the treatments CPL250, PPL350, and PPL450, corroborating what was previously discussed through the EDS analysis. The calcite (CaCO_3) in the samples shows the alkaline character of the poultry litter biochar, and its presence is attributed to the use of calcitic limestone in the chickens' diet (Domingues et al., 2017; Chaves et al., 2020).

Feldspar compounds [Orthoclase (KAlSi_3O_8) and Albite ($\text{Al}_{1.26}\text{Si}_{2.74}\text{O}_8$)] and potassium phosphate ($\text{K}_2(\text{HPO}_4)$) (Figure 4) were also identified, confirming the result of the chemical analysis of the poultry litter biochar, which showed considerable contents of K, Ca, and Mg (Figure 3). The present patterns of XRD were quite similar to the pattern of biochar derived from chicken litter reported by Chaves et al. (2020).

The crystallinity observed in this study may be associated with the presence of inorganic minerals from the biomass used as poultry litter contaminated with soil, due to the waste of the supplied feed (around 3%) and bird feces (which metabolize approximately 70% of the nutrients) (Chaves et al., 2020).

Fourier-transform infrared spectroscopy (FTIR) spectra were similar between the treatments CPL250, PPL350, and PPL450 (Figure 5). After the biomass was subjected to the thermal process, the intensity of all organic functional bands remained practically unchanged, regardless of the treatment. This behavior may be associated with the high ash content found in the poultry litter, which protects organic compounds against thermal degradation (Domingues et al., 2017).

The wideband observed at wavelength of 3200-3400 cm^{-1} is attributed to -OH from H_2O , or phenolic groups (Chen et al., 2008; Melo et al., 2013). However, samples PPL350 and

PPL450 did not show hydroxyl peaks in these regions; the loss of OH functionality is a consequence of reactions of decarbonylation, decarboxylation, and dehydration promoted with the increase of the pyrolysis temperature (Cuixia et al., 2020). The absorption in the region between 2920 and 2885 cm^{-1} (CH stretching) was attributed to aliphatic functional groups (Melo et al., 2013), and the increase in band intensity in the region of 1600 cm^{-1} showed the presence of ketones and conjugated quinones ($\text{C}=\text{O}$ and $\text{C}=\text{C}$). The intense bands at 1270 cm^{-1} are attributed to OH-phenolic groups (Chen et al., 2008), and the strong band at 1030 cm^{-1} was due to the stretching of CO and associated with oxygenated functional groups of cellulose, hemicellulose, and methoxylated lignin groups (Cantrell et al., 2012). The appearance of weak bands between 885 and 750 cm^{-1} (aromatic CH outside the plane) was attributed to an increasing degree of condensation of the organic compounds present in the biochar. Spectra smaller than 601 cm^{-1} are associated with the presence of inorganic metals. Chaves et al. (2020) characterized the poultry litter biochar and obtained a spectrum similar to that found in this research.

Specific knowledge about the chemistry of the functional groups of the samples CPL250, PPL350, and PPL450 is essential to predict and understand how they will react in the soil. For example, it is known that functional groups such as carboxylic, phenolic, hydroxyl, carbonyl, or quinones present in poultry litter biochar increase the sorption of heavy metals (Lu et al., 2018); carboxylic, hydroxyl, and aromatic groups have enhanced cation exchange capacity (CEC). Also, as hydrophobic substances, these have a greater affinity for organic compounds (Ok et al., 2016). Furthermore, Fernandes et al. (2018) found an increase in soil CEC after applying poultry litter biochar, probably caused by negative charges due to the deprotonation of H^+ present in the hydroxyl phenolic groups.

CONCLUSIONS

1. The results showed the presence of mesoporous structures in the CPL250, PPL350, and PPL450 samples and an increase in surface area and pore volume as a result of heat treatment.
2. Point scan analyses of all samples by the SEM-EDS technique revealed the presence of heavy metals and macro and micronutrients. However, potassium, in the constitution of sylvite, potassium aluminum silicate, in addition to calcite and potassium phosphates, is present at higher concentrations, confirmed by XRD analysis.
3. The samples have surface functional groups similar to each other, such as phenolic, aliphatic, conjugated quinones, and OH-phenolic. There was only a loss of the hydroxyl group in samples PPL350 and PPL450.
4. The different thermal treatments applied to poultry litter generated samples with similar physical, chemical, and structural characteristics.

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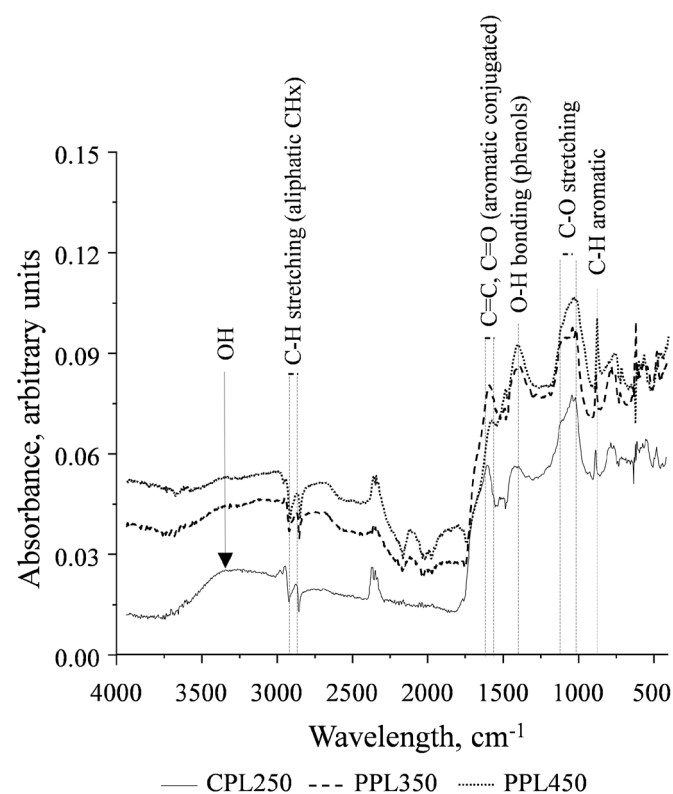


Figure 5. Fourier-transform infrared spectroscopy of samples of poultry litter carbonized at 250 °C (CPL250) and pyrolyzed at 350 °C (PPL350) and 450 °C (PPL450)

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