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Thermal treatment of poultry litter: Part I. Characterization by immediate analysis and gravimetric yield[1]

Tratamento térmico da cama de aviário: Parte I. Caracterização por análises imediatas e rendimento gravimétrico

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HIGHLIGHTS:

Pyrolysis temperature influences biochar properties, with CPL250 showing the highest gravimetric yield.
CPL250 promotes the highest contents of volatile material and fixed carbon, while PPL350 and PPL450 high ash contents.
PPL450 treatment promotes the highest pH and EC values.

ABSTRACT: Producing biochar from poultry litter through slow pyrolysis is a farm-based, value added approach to recycle organic residues. Experiments were conducted to examine how heat treatments affected the final composition of carbonized and pyrolyzed material in relation to immediate analysis and gravimetric yield to assess agronomic performance value. These processes were carried out in porcelain containers containing samples of poultry litter and placed in a muffle furnace. The treatments corresponded to three temperatures: samples carbonized at 250 °C (CPL) and pyrolyzed at 350 and 450 °C (PPL). Samples were analyzed in relation to the volatile content, fixed carbon, ash, moisture, pH, electrical conductivity (EC), and zero charge point (ZCP). The PPL350 and PPL450 biochars had the highest ash contents and the lowest fixed carbon, moisture and gravimetric yields. The increase in temperatures used in the process resulted in higher pH and electrical conductivity values. In addition, the ZCP value, with the exception of CPL250, showed the presence of negative charges on the surface of the biochars.

Key words: slow pyrolysis, biochar, pyrolysis temperature, carbonization process

RESUMO: A produção de biocarvão a partir de cama de aviário por meio de pirólise lenta é uma abordagem de valor agregado baseada na fazenda para reciclar o resíduo orgânico. Os experimentos foram conduzidos para examinar como os tratamentos térmicos afetaram a composição final do material carbonizado e pirolisado em relação à análise imediata e rendimento gravimétrico para avaliar o valor do desempenho agronômico. Esses processos foram realizados em recipientes de porcelana contendo amostras de cama de aviário e acondicionados em mufla. Os tratamentos corresponderam a três temperaturas: amostras carbonizadas a 250 °C (CPL) e pirolisadas a 350 e 450 °C (PPL). As amostras foram analisadas em relação ao teor de voláteis, carbono fixo, cinzas, umidade, pH, condutividade elétrica (CE) e ponto de carga zero (PCZ). Os biocarvões PPL350 e PPL450 apresentaram os maiores teores de cinzas e os menores teores de carbono fixo, umidade e rendimento gravimétrico. O aumento das temperaturas utilizadas no processo ocasionou maiores valores de pH e condutividade elétrica. Além disso, o valor de PCZ, com exceção de CPL250, mostrou a presença de cargas negativas na superfície dos biocarvões.

Palavras-chave: pirólise lenta, biocarvão, temperatura de pirólise, processo de carbonização

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Introduction

With the expansion of the poultry sector in Brazil, in 2018, around 5.7 billion poultry were produced (IBGE, 2018) and, considering that one produces an average of 1.8 kg of waste (Rogeri et al., 2016), approximately 10.2 million tons of poultry litter is generated per year in Brazil, increasing the potential environmental problem if it is discharged in the field without treatment.

An option to recycle poultry litter in agriculture is to obtain biochar through pyrolysis process in anoxic or oxygen-limited environments (Dhyani & Bhaskar, 2018; Sajjadi et al., 2018). This product has been used as a soil amendment due to its ability for long term improvements in soil physical and chemical properties with potentially important effects on soil biota. Among the various factors that affect the pyrolysis of biomass is the temperature, which can vary between 300 and 1000 °C (Souza et al., 2021). Biochar can be produced by farmers, who obtain, depending on the temperature, a carbonized product (< 250 °C) or biochar (> 350 °C).

Poultry litter biochar, once subjected to different heat treatments, can be characterized through ‘’immediate analysis”, which determines the fixed carbon, moisture, volatiles, ash content, in addition to the pH, electrical conductivity and zero charge point of biochars (Aller et al., 2017). As for the temperature used in the pyrolysis of poultry litter, some studies have already been carried out, such as Sobik-Szołtysek et al. (2021), who studied the sorption capacity of heavy metals in biochars produced at 425, 525, and 752 °C. Tsai & Chang (2021; 2022) evaluated the impacts of biochars pyrolyzed at 200, 300, 400, 500 and 600 °C, on soil carbon mineralization and on the preservation of nutrients and immobilization of contaminants.

As the relationship between poultry litter or production conditions and biochar properties designed to improve soil properties is still little known, the purpose of this study was to examine how thermal treatments affected the final composition of the carbonized and pyrolyzed material with respect to immediate analysis and gravimetric yield to evaluate agronomic performance value.

Material and Methods

The raw material used for the production of the biochar was broiler poultry litter collected at UEPB experimental farm, in the municipality of Lagoa Seca (07° 09’ 22.42” S; 35° 52’ 09.64” W). The choice was based on the abundant availability of the material, since the poultry activity leads to a considerable production of poultry litter, an organic residue that can be a valuable input due to the high concentration of nutrients.

In addition, the presence of compounds contained in sugarcane straw, such as lignin (19%) and cellulose (44.5%), favors the formation of biochar (Santos et al., 2014). Poultry litter is composed of sugarcane bagasse, chicken feces and feed (about 4% wastage considering the whole production cycle comprising from receipt of the chick to slaughter). The ingredients used in the formulation of the feed in the different stages of development of the broilers, as well as the chemical composition, are described in Tables 1 and 2.

Table 1. Quantitative estimate of the ingredients used in the formulation of 100 kg of broiler feed

Ingredients	Development phase			
	Initial 1-10 days	Growth 11-24 days	Fattening 25-39 days	Termination 40-51 days
	(kg)			
Corn	44.74	50.54	52.53	59.39
Dicalcium Phosphate	0.64	0.46	0.64	0.19
Core	5.00*	5.00*	5.00*	5.00**
Soy oil	0.62	1.66	4.07	2.80
Methionine	0.27	0.24	0.23	0.17
Threonine	0.06	0.05	0.02	0.03
Semi-whole soy	48.67	42.06	37.52	32.41

*Chemical composition of the core used between the initial and fattening phases: Ca = 26.94%; P = 5.38%; Na = 5.7%; Fe = 0.09%; Cu = 0.03%; Mn = 0.21%; Zn = 0.15%; I = 0.003%; Co = 0.0006%; Se = 0.00105%; **Chemical composition of the core used in the termination phase: Ca = 9.6%; P = 1.5%; Na = 3.8%; Fe = 0.0656%; F = 0.015%; Cu = 0.0129%; Mn = 0.0845%; Zn = 0.0656%; I = 0.00151%; Se = 0.000947%

Table 2. Chemical composition of feed supplied at different stages of development of broiler chickens

Nutrients	Development phase			
	Initial 1-10 days	Growth 11-24 days	Fattening 25-39 days	Termination 40-51 days
Metabolizable energy	3.00	3.10	3.20	3.22
	(Mcal kg^{-1})			
	(%)			
Crude protein	24.77	22.58	19.49	19.40
Calcium	1.13	1.07	1.20	0.99
Phosphorus	0.48	0.44	0.39	0.37
Crude fiber	4.09	3.77	3.51	3.30
Sodium	0.21	0.21	0.19	0.21

The poultry litter, approximately 4 cm thick, was collected in the poultry house after slaughter of the chickens (52 days after arrival of the chicks). Thereafter, it was air-dried under shade to < 30% moisture, milled and sieved to 3.8 “mesh (9.50 mm). After this process, it was transported to the Laboratory of Irrigation and Salinity, Federal University of Campina Grande, where drying was finished, by conditioning the material in open trays and keeping it in the oven (BioPar S150SD) at temperature of 50.0 ± 1.0 °C until constant mass.

The chemical characterization of the poultry litter was performed in the Laboratory of Soil and Leaf Analysis according to Ministério da Agricultura, Pecuária e Abastecimento (MAPA) (2014) and the results were: pH = 8.24; Total N = 2.78%; C = 37.57%; Total P_2O_5 = 2.08%; Total K_2O = 2.48%; Ca = 2.40%; Mg = 0.68%; S = 0.17%; B = 0.01%; Zn = 0.02%; Cu = < 0.01%; Mn = 0.02%; Fe = 0.26%; Moisture = 13.29%.

The carbonization experiments were performed in porcelain containers filled with poultry litter samples and placed in a muffle furnace with digital microprocessor controller (Jung J200). Each container was filled with about 400 g, compressing it to reduce the O_2 concentration during the thermal degradation, then the lid of the container was set to allow only the output of volatiles. In this study, the volatiles produced were neither collected nor quantified. The thermal process used in this study was slow and the treatments corresponded to three temperatures of thermal degradation, 250, 350 and 450 °C, in triplicate (replicate), following a heating rate of 10 °C min^{-1}. After reaching the temperature, the samples remained in the muffle furnace for three hours. When heated to 250 °C, the sample is carbonized to a limited extent and labeled as CPL250 (carbonized poultry litter at 250 °C). The material produced after samples were subjected to temperatures of 350 and 450 °C is labeled as: PPL350 and PPL450 (poultry litter pyrolyzed at 350 and 450 °C, respectively).

For each replicate of the biochars CPL250, PPL350, and PPL450 produced, two samples were collected, totaling 18 plots, and then the chemical analysis was performed according to ASTM (2007), elaborated for wood charcoal.

The moisture content was determined according to Silva (2009), by the following Eq.:

$$\text{Moisture}(\%) = \frac{A - B}{A} 100 \tag{1}$$

where:

A - grams of air-dried sample used; and,
B - grams of sample after drying at 105 °C.

To determine the volatile matter in the samples, 1 g of sample in a crucible was taken to a muffle furnace and kept for 6 minutes at 950 °C.

The percentage of volatile matter in the sample was calculated as follows:

$$\text{Volatile matter}(\%) = \frac{B - C}{B} 100 \tag{2}$$

where:

B - grams of sample after drying at 105 °C; and,
C - grams of sample after drying at 950 °C.

For analysis of ash content, an uncovered crucible with about 1 g of biochar was heated in the muffle furnace at 750 °C for six hours.

The percentage of ash in the sample was calculated as follows:

$$\text{Ash}(\%) = \frac{D}{B} 100 \tag{3}$$

where:

B - grams of sample after drying at 105 °C; and,
D - grams of residue.

To obtain the fixed carbon content (FCC), the following equation was applied:

$$\text{FCC}(\%) = 100 - (\text{M} + \text{VMC} + \text{AC}) \tag{4}$$

where:

M - moisture content, in percentage;
VMC - volatile matter content, in percentage; and,
AC - ash content, in percentage.

To obtain the gravimetric yield (GY) values of the treatments, the Eq. 5 below was used:

$$\text{GY}(\%) = \frac{\text{BMP}}{\text{DCSM}} 100 \tag{5}$$

where:

BMP - mass after heat treatment (g); and,
DCSM - initial mass of poultry litter (g).

To determine the pH of the samples, 1 g of sample to be analyzed and 20 mL of deionized water were put in an Erlenmeyer® flask (125 mL). The mixture was stirred on an orbital shaking table for 1.5 hours. After stirring, the digital pH meter previously calibrated with standard solutions of pH 4.0 and 7.0 was used to determine the pH values of the samples. To determine the electrical conductivity, the same samples were left at rest for approximately 12 hours, when the EC was determined with the digital conductivity meter previously calibrated with a standard EC solution of to KCl 0.01 N (1.4 mS cm^{-1}), according to the methodology recommended by Rajkovich et al. (2011).

The determination of the zero charge point (ZCP) was carried out using the methodology called the “11 point experiment”, described by Regalbuto & Robles (2004).

The results of fixed carbon, ash, volatile matter, moisture and gravimetric yield were subjected to analysis of variance (ANOVA). The means obtained in the different treatments (250, 350 and 450 °C) were compared to each other using the Tukey test, at the significance level of 0.05. All statistical calculations were performed using the SISVAR 5.4 program (Ferreira, 2014).

RESULTS AND DISCUSSION

The relative amount of the volatile, ash, fixed carbon and moisture portions contained in the carbonized poultry litter (CPL250) and in the biochars pyrolyzed at 350 °C (PPL350) and 450 °C (PPL450) are shown in Figure 1.

In general, it is observed that the percentages of the components volatile matter and fixed carbon in the CPL250 treatment were higher than those in the biochar produced at

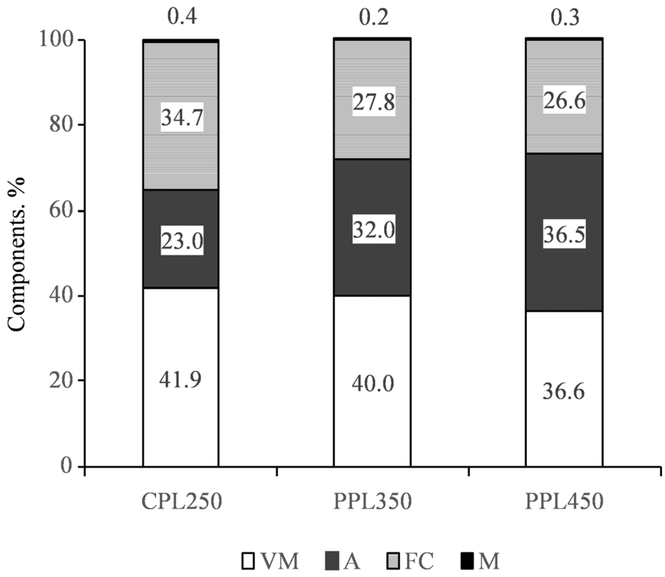


Figure 1. Relation of the volatile matter (VM), ash (A), fixed carbon (FC), and moisture (M) proportions of the carbonized poultry litter (CPL250) and in the biochars pyrolyzed at 350 °C (PPL350) and 450 °C (PPL450)

temperatures 350 and 450 °C, contrary to the percentage of ash, which was lower in the CPL250 (Figure 1).

The poultry litter underwent thermal modifications with increasing temperature, resulting in the loss of moisture bound to the structure of the biochar. Regardless of pyrolysis temperature, moisture content in biochar was not more than 1% (Figure 1).

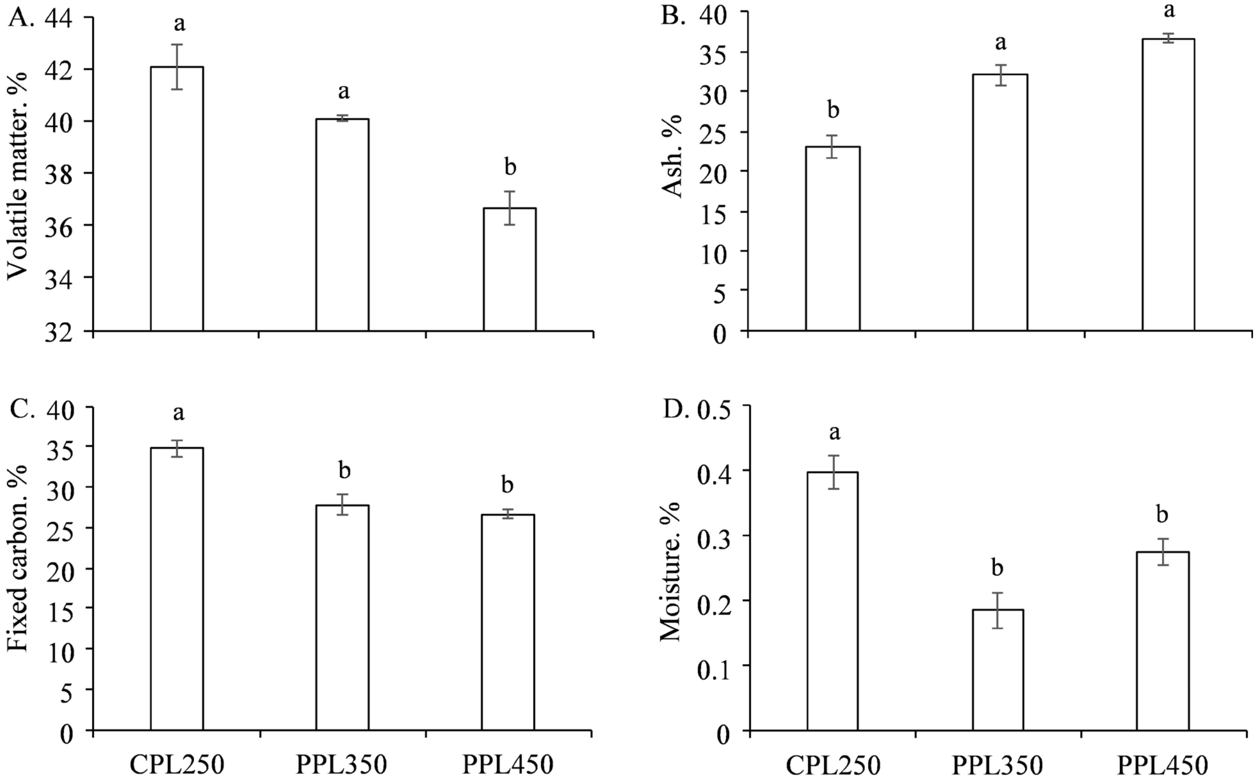
The volatile material is the portion of the mass of the original material lost during pyrolysis, being composed of labile materials. For this reason, as the pyrolysis temperature increases, the portion of these materials decreases (Figure 1), since they are easily released (Devens et al., 2018). The volatile contents vary depending on the raw material used in the pyrolysis process. Biochars from wood have a higher content of volatile material when compared to those from crop and animal waste. However, in general, the greatest variation of these volatile contents is due to the different temperatures in the production of bio-carbon as can be observed in Figure 1, that is, in CPL250, there were reductions of 1.9 and 5.3 units of volatiles when compared to PPL350 and PPL450, respectively (Zhao et al., 2013).

According to the analysis of variance for all the components of the analyzed treatments, there was a significant difference between at least one of the treatments, whose Pr > Fc values were: 0.0024; 0.0004; 0.0022, and 0.0024 for volatile matter, ash, moisture, and fixed carbon, respectively, and by Tukey's test, it was possible to know which treatments differed significantly (Figure 2).

It can be seen that volatile contents decreased with increasing temperature, corroborating Jindo et al. (2014); however, CPL250 and the PPL350 showed statistical similarity and higher contents than that observed in the PPL450 treatment (Figure 2A).

The raw material subjected to the process of pyrolysis (subjected to heating) gradually loses the chemical substances. According to Amonette & Joseph (2009), between 120 and 300 °C the formation of partially carbonized material predominates with loss of volatile molecules (methanol, acetic acid, CO, H_2, and CO_2), molecules with low molecular masses (fatty acids, oils, and resins), as well as the decomposition of the hemicelluloses and the release of water. Between 300 and 600 °C, there is the formation of tar, methane, phenolic compounds due to holocellulose breaking and total lignin, with a consequent decrease in biochar production.

Ash content is related to the content of nutrients present in the raw material. This part of the matter is determined after its complete combustion, when all the organic elements are volatilized (Devens et al., 2018). In this study the ash content increased as a function of the pyrolysis temperature employed; the means obtained in treatments PPL350 and PPL450 did not differ statistically, with increases corresponding to 38.96 and 58.44%, respectively, when compared to that observed in the treatment CPL250 (Figure 2B). The lower ash content is justified by the lower temperature used in the process and the composition of this biochar, mainly composed of volatile material and fixed carbon. These results corroborate those of Domingues et al. (2017), who verified an increase in ash content as a function of the pyrolysis temperature (350, 450, and 750 °C). According to these authors, this increase is correlated with the considerable concentration of inorganic elements (K, P, Ca, and Mg) present in poultry litter, which accumulated after volatilization of C, O, and H.



Same lowercase letters indicate no significant differences between treatments by the Tukey test at p<0.05. Vertical bars represent the standard error (n = 6 repetitions)

Figure 2. Volatile matter (A), ash (B), fixed carbon (C), and moisture (D) analyzed in the poultry litter carbonized at 250 °C (CPL250) and poultry litter pyrolyzed at 350 °C (PPL350) and 450 °C (PPL450)

It is important to note that the poultry litter used in this study was composed of sugarcane bagasse, chicken feces, and feed, waste at a ratio of 1:0.165, wet basis. By analyzing Tables 1 and 2, it is verified that the chicken feed consists of different ingredients, including dicalcium phosphate and the core that provides phosphorus (P), calcium (Ca), sodium (Na), among others.

The concentration of these ingredients varied according to the stage of development of the chickens, with calcium content close to 1% during the stay of the hens in the poultry house and the phosphorus content between 0.48 and 0.44% in the first two stages, decreasing to 0.37% in the termination phase.

The provision of feeds and the time spent by the chickens in the poultry house (52 days) favored an ash content of 36.6% when the chicken litter was pyrolyzed at 450 °C. This result was superior to that verified by Cantrell et al. (2014); these authors verified ash contents in the biochar from poultry litter of 4.9, 6.2, and 7.4% at temperatures of 350, 500, and 700 °C, respectively. The knowledge of the ash content in the biochar is important for the agricultural use, due to the presence of considerable concentrations of nutrients for the plants, besides having a corrective effect on acidity (Shetty & Prakash, 2020).

The fixed carbon content (FC) is the portion of the material in its most resistant form that remains in the biochar after the volatile materials are removed, usually arranged in aromatic chains (Devens et al., 2018). In this study, the highest FC content (34.82%) was found in CPL250, statistically differing from the means obtained at temperatures of 350 and 450 °C (Figure 2C). This result differs from that observed by Cantrell et al. (2012), who verified, for poultry litter biochar, a positive correlation between the fixed carbon content and the pyrolysis temperature. This result suggests that the variation of the fixed carbon content as a function of the temperature depends on the biomass used and, in the case of the poultry litter, on the handling and period in which the litter remains in the poultry house. The poultry litter used by Cantrell et al. (2012) was collected in a poultry flock in the upper part of 5 to 7.5 cm deep consisting of wood chips and chicken droppings accumulated during a year, while the one used in this study was collected after 52 days of chicks arrival in the poultry house and biomass was sugarcane bagasse. These differences influence the ash content of the biochar produced and consequently the fixed carbon content. According to Jindo et al. (2014), this component increases as a result of pyrolysis temperatures when the biochar contains less than 20% ash; however, the fixed carbon content decreases when biochar has an ash content exceeding 20%, probably due to interactions between the constituents of organic and inorganic raw materials during pyrolysis.

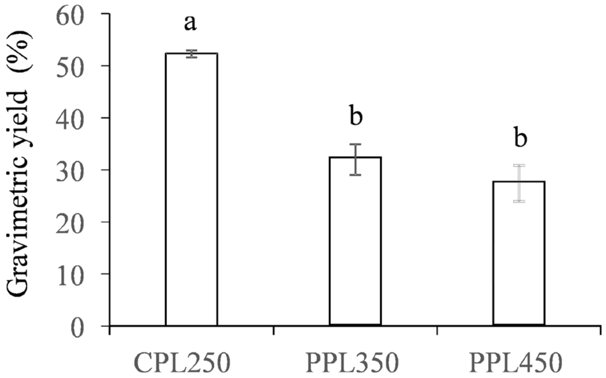
The average moisture content in the poultry litter (wet basis) used in this study was 29.7%. Under thermal treatment, biomass usually begins to loose moisture at temperatures from 50 °C, characterizing the first stage of the decomposition, so its fraction in the biochar is low (Sadaka et al., 2014). In this study, regardless of the treatment used, the samples had mean moisture content of less than 1%, with the highest content (0.4%) at 250 °C, significantly differing from the 0.19 and 0.27% levels recorded in the PPL350 and PPL450, respectively.

The masses obtained after the thermal conversion process show a gradual decrease in the yield with the increase of the pyrolysis temperature (Figure 3). According to Shahbaz et al. (2020), this is due to the increase in the degradation of cellulose, hemicellulose, and lignin generated under the pyrolysis conditions as a function of the temperature increase. Furthermore, the ash content present in the raw material (poultry litter) also influences yield of the produced biochar.

Also when analyzing Figure 3, it can be seen that the CPL250 treatment promoted a yield of 52.3%, significantly higher than the masses pyrolyzed at temperatures of 350 and 450 °C, with yields of 32.03 and 27.49%, respectively.

The samples analyzed showed a continuous increase in electrical conductivity (EC) associated with process temperatures, whose values corresponded to 7.270, 7.226 and 7.675 dS m^{-1} when applying 250, 350, and 450 °C, respectively. This behavior can be correlated with the loss of volatile material, resulting in the concentration of elements in the ash fraction (Cantrell et al., 2012). Also according to these authors, due to the incomplete assimilation of nutrients by hens, the mineral elements present in the poultry litter considerably increase the EC values in biochars produced from this biomass. The temperatures 250, 350, and 450 °C also influenced the pH of the samples, which showed alkaline values, that is, 7.55, 8.69, and 9.66, respectively. These results corroborate those of Cantrell et al. (2012), who verified pH values of 8.7 and 10.3 in the poultry litter biochar, pyrolyzed at 350 and 700 °C, respectively.

The determination of the zero charge point (ZCP) is important since the pH variation of the solution directly influences the surface charges of the biochar and consequently the electrostatic interactions between the adsorbent and the adsorbate (Chaves et al., 2020). The pHZCP values in the treatments CPL250, PPL350 and PPL450 corresponded to 7.95, 8.48, and 8.73, respectively. With the exception of CPL250, which had a pH of 7.55, the PPL350 (pH = 8.69) and PPL450 (pH = 9.66) biochars had a negatively charged surface and, under natural conditions, can be used as a cation adsorbent (Liao et al., 2015; Zhao, et al., 2019).



Same lowercase letters indicate no significant differences between treatments by the Tukey test at $p < 0.05$. Vertical bars represent the standard error (n = 6 repetitions)

Figure 3. Gravimetric yield of the poultry litter carbonized at 250 °C (CPL250) and pyrolyzed at 350 °C (PPL350) and 450 °C (PPL450)

Conclusions

1. The PPL350 and PPL450 biochars had the highest ash contents and the lowest contents of fixed carbon, moisture and gravimetric yield;

2. The increase in temperatures used in the process caused higher pH and EC values. In addition, the ZCP value, with the exception of CPL250, showed the presence of negative charges on the surface of the biochars.

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