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Scientific Article

QUALITY OF BLACK PELLETS OF CHARCOAL FINES PRODUCED WITH DIFFERENT MOISTURE CONTENT AND DRYING TIME

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ABSTRACT

Charcoal fines are co-products of the wood carbonization process, unwanted because they reduce the gravimetric yield of the main product, charcoal. It can be produced due to the carbonization process, during the storage and transport of charcoal. Pelletizing charcoal fines increases the energy density of this material, producing a homogeneous fuel with lower moisture content, enabling burning in equipment with high energy efficiency, in addition to desirable properties in energy products such as high fixed carbon content and low moisture content. This study evaluated the effect of moisture content and drying time on the production of black pellets from charcoal fines bound with rice starch (*Oryza sativa*) on the physicochemical and mechanical characteristics. Black pellets were produced using two proportions of water (10 or 20%) and a drying time of 4 or 6 hours. Black pellets were evaluated for their proximate analysis, elemental composition, moisture content, size, apparent density, calorific value and energy density. The addition of water did not affect the content of volatile matter, ash, and fixed carbon, while the treatment with addition of 20% water and four hours of drying showed a higher moisture content (3.34%). The addition of water allowed for better heat conduction and particle arrangement, producing pellets with a durability greater than 98%. The better arrangement promoted by the addition of 20% water also increased the density of the pellets, resulting in greater energy density. Black pellets can be considered an alternative for residential heating, with T3 treatment (20% - 4h) with a high energy density and better performance in terms of desirable properties for use and commercialization according to the standard EN 14961-6.

Keywords: Residential heating; Bioenergy; Charcoal residue.

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QUALIDADE DE BLACK PELLETS DE FINOS DE CARVÃO VEGETAL PRODUZIDOS COM DIFERENTES UMIDADES E TEMPO DE SECAGEM

RESUMO – Finos de carvão vegetal são coprodutos da carbonização da madeira, indesejados por diminuir o rendimento gravimétrico em carvão vegetal. São produzidos devido ao processo de carbonização, durante o armazenamento e transporte do carvão vegetal. A pelletização de finos aumenta a densidade energética, produzindo um combustível homogêneo e com menor teor de umidade, possibilitando a queima em equipamentos com alta eficiência energética, além de propriedades desejáveis em produtos energéticos como alto teor de carbono fixo e baixo teor de umidade. Este estudo avaliou o efeito do teor de umidade e do tempo de secagem na produção de black pellets a partir de finos de carvão vegetal ligados com amido de arroz (*Oryza sativa*) nas características físico-químicas e mecânicas. Os black pellets foram produzidos utilizando duas proporções de água (10 ou 20%) e tempo de secagem de 4 ou 6 horas. Os black pellets foram avaliados quanto à análise química imediata, composição elementar, teor de umidade, tamanho, densidade aparente, poder calorífico e densidade energética. A adição de água não afetou o teor de voláteis, cinzas e carbono fixo, enquanto o tratamento com adição de 20% de água e quatro horas de secagem apresentou maior teor de umidade (3,34%). A adição de água permitiu melhor condução de calor e disposição das partículas, produzindo pellets com durabilidade superior a 98%. A melhor disposição promovida pela adição de 20% de água também aumentou a densidade dos pellets, resultando em maior densidade energética. Os black pellets podem ser considerados uma boa alternativa para aquecimento residencial, com tratamento T3 (20% de umidade - 4h de secagem) apresentando elevada densidade energética e melhor desempenho em termos de propriedades desejáveis para utilização e comercialização de acordo com a norma EN 14961-6.

Palavras-Chave: Aquecimento residencial; Bioenergia; Resíduos do carvão.

1. INTRODUCTION

Brazil stands out on the world stage for being the largest producer and consumer of charcoal, accounting for 12% of world production, and for being the only country in the world that uses this product on a large scale in the pig iron and steel industries (IBÁ, 2022). In 2022, 6.933 thousand tons of charcoal were produced in Brazil (BEN, 2023).

The generation of fines during the production and transport of charcoal is around 25% of the total production of charcoal (Ramos et al., 2023), and studies show that this total can reach 40% in processes with a high degree of mechanization (Somerville and Jahanshahi, 2015; Rudolfsson et al., 2017; Pascoal et al., 2022), resulting in large stocks in charcoal companies. Factors such as raw material variability, lack of control over the carbonization process, inadequate cooling of kilns, storage, sieving and transportation are identified as the main factors causing the increase in the amount of fines (Assis et al., 2016). Despite the use of this co-product in blast furnace tuyeres in the carbonization process or as a binder for iron ore fines, such uses do not meet all the demand for material generated, making it necessary to implement new processes that are economically viable to solve this challenge. The densification of charcoal fines can be a promising alternative for the disposal of these fines, resulting in new products with added value such as pellets and briquettes (Arteaga-Pérez et al., 2017; Lima et al., 2023).

Pellets made from carbonized and torrefied material are known as black pellets due to their dark color. As it is a raw material with desirable characteristics for the energy sector, such as high calorific value, low moisture content and high energy density (Lima et al., 2022; Rodrigues and Junior, 2019), the production of black pellets is of increasing commercial interest, mainly for export. However, pellets formed from these materials can easily break during transportation due to their high friability. Consequently, the addition of binders would be necessary to improve the physical-mechanical properties due to the cohesive force generated between the particles, resulting in solid bonds and/or inducing chemical reactions between the binders (Ghebre-Sellassie, 2022; Zhao et al., 2023).

Organic additives or binders such as starches from corn (*Zea mays*), cassava

(*Manihot esculenta*), pea (*Pisum sativum*), potato (*Solanum tuberosum*), sweet potato (*Ipomoea batatas*), rice (*Oryza sativa*), wheat (*Triticum* sp.) or yam (*Dioscorea* sp.), as well as kraft lignin can be used to reduce the breakage and disintegration of pellets (Amaya et al., 2015; Surdi et al., 2021). Rice starch, which also belongs to the class of organic binders, is a material of natural, renewable origin and easy to obtain due to high Brazilian production (Conab, 2023). One of the possible applications of rice starch is associated with the gelatinization mechanism with heated water, which can be used in the manufacture of densified materials (Chakraborty et al., 2023), such as pellets.

Despite studies reported in the literature investigating black pellets, information related to their production is scarce regarding the amount of water in the manufacturing process and drying time, which impact not only the quality of the final product, but also in costs and competitiveness in relation to other energy sources. Thus, the objective of this study was to evaluate the combined effect of moisture content and drying time on the physical-chemical and mechanical characteristics of black pellets produced from charcoal fines bound with rice starch.

2. MATERIAL AND METHODS

2.1 Obtaining charcoal fines and rice starch

The charcoal used was obtained from *Eucalyptus* sp. wood, with dry basis moisture content of approximately 35%. The carbonizations were carried out in a pilot kilns-furnace system, according to the procedure described by Siqueira (2021), with the maximum temperature of 350 ºC. After wood carbonization, the gravimetric yield was determined on charcoal, semi-carbonized wood and charcoal fines (charcoal with a particle size of less than 10 mm), presenting average values of 33.5, 4.1 and 7.2%, respectively. The fines generated were stored in plastic bags and subsequently ground and sieved to adjust the particle size to 20 mesh. Finally, black pellets were produced using commercial rice starch from Glúten Free Alimentos (São Paulo, Brazil) as a binder.

Elemental chemical analysis was carried out on charcoal fines, before adding the binder. The fines were classified between 40 and 60 mesh, and analyzed using the TruSPec Micro equipment (Leco, São Paulo, Brazil) for CHN and the TruSpec S module for sulfur content. The oxygen content was obtained by difference. Charcoal fines presented 87.2; 2.72; 0.04; 0.02; and 10.05% for carbon, hydrogen, nitrogen, sulfur and oxygen contents, respectively.

The charcoal fines and starch were classified between 40 and 60 mesh sieves, and then dried in a forced air circulation oven until reaching constant mass values to determine the contents of volatile matter, ash and fixed carbon according to the standard D1762-84 (ASTM, 2021). Charcoal fines have 20.85% volatile matter, 1.86% ash content, and 77.29% fixed carbon content. The rice starch has 86.88% volatile matter, 1.45% ash content and 11.67% fixed carbon content.

2.2 Black pellets production

For every treatment, 2 kg of charcoal fines, 7.5% (150 g) of rice starch and water in different amounts were mixed and added to a pellet mill. Proportions of 10 or 20% water (200 or 400 mL of water) and 4 or 6 hours of drying time were evaluated, resulting in the combination of treatments that were evaluated in CDR (Completely Randomized Design) (Table 1).

The black pellets were produced in a laboratory pellets mill manufactured by Amandus Kahl, model 14–175 (Germany), with a production capacity of 10 kg.h-1. The average temperature was 100±5 °C. The pellets die was preheated in oil at 200 °C for approximately 20 minutes. Approximately 2 kilograms of each treatment were produced.

The drying process was carried out in a forced air circulation oven with a temperature of 100 °C ± 5, for 4 or 6 hours. Then, the black pellets were removed from the oven and cooled to room temperature, and stored in a dry, covered place (Figure 1) until analysis was carried out.

2.3 Characterization of black pellets

After the drying stage, before storage, the moisture content of the black pellets, wet basis, was determined in an air circulation oven, in triplicate in accordance with EN 14774-2 (EN, 2010).

A sample of black pellets was crushed

Table 1. Experimental design of the study.

Tabela 1. Desenho experimental do estudo.

Treatment	Water proportion (%)	Drying time (h)	Code
1	10%	4	10 - 4h
2	10%	6	10 - 6h
3	20%	4	20 - 4h
4	20%	6	20 - 6h

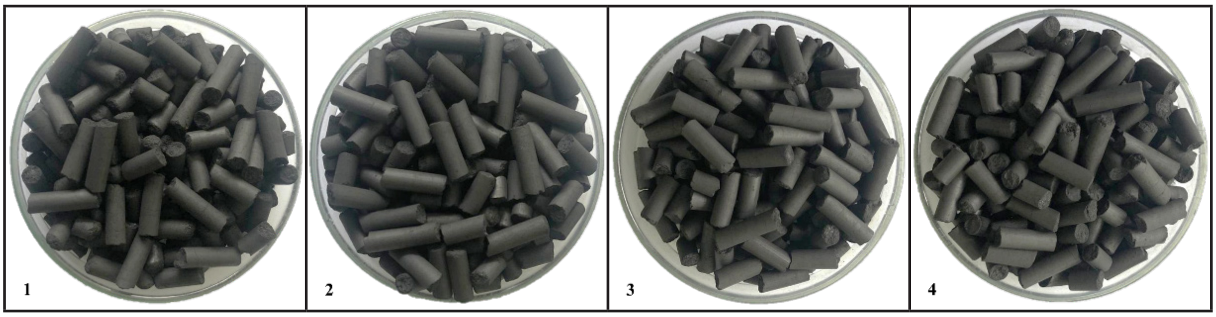

Figure 1. Black pellets produced from established treatments.

Figura 1. Black pellets produzidos a partir dos tratamentos estabelecidos.

manually and classified between 40 and 60 mesh sieves, and then dried in a forced air circulation oven until reaching constant mass values to determine the contents of volatile matter, ash and fixed carbon according to the standard D1762-84 (ASTM, 2021). The gross calorific value (GCV) was determined using an adiabatic bomb calorimeter model IKA300 (Danon, Rio de Janeiro, Brazil) in accordance with Brito (1986). The net calorific value (NCV) was calculated according to Equations 1 and 2.

$$NCV_{dry} = GCV - 600\left(\frac{9H}{100}\right) \qquad (Eq.\ 1)$$

$$NCV = NCV_{dry}\,(1 - M) - 600M \qquad (Eq.\ 2)$$

Where:

NCVdry (kcal/kg): net calorific value dry basis;

NCV (kcal/kg): net calorific value as received;

GCV (kcal/kg): gross calorific value;

H (%): Hydrogen content;

600 (kcal): Average value of energy absorbed/kg of water to reach evaporation temperature;

9: Multiple of the weight of the hydrogen H2, contained in the material, which provides the weight of the water formed during combustion;

M (%): Moisture content, wet basis.

The bulk density of black pellets was obtained using a wooden box with dimensions (10 x 10 x 10 cm), in accordance with the EN 15103 standard (DIN EN, 2010) and the energy density was calculated by multiplying the bulk density and the GCV of each treatment. The dimensions of the black pellets produced were obtained with a caliper, in accordance with EN 16127 (DIN EN, 2012) and the mechanical durability and the percentage of fines were obtained in Ligno-tester equipment (TekPro Limited, Willow Park, England) in accordance with ISO17831 -1 (ISO, 2015). The pellets were classified according to DIN 14961-6 (2012) for the commercialization of this material. The experimental scheme of the study is represented in Figure 2.

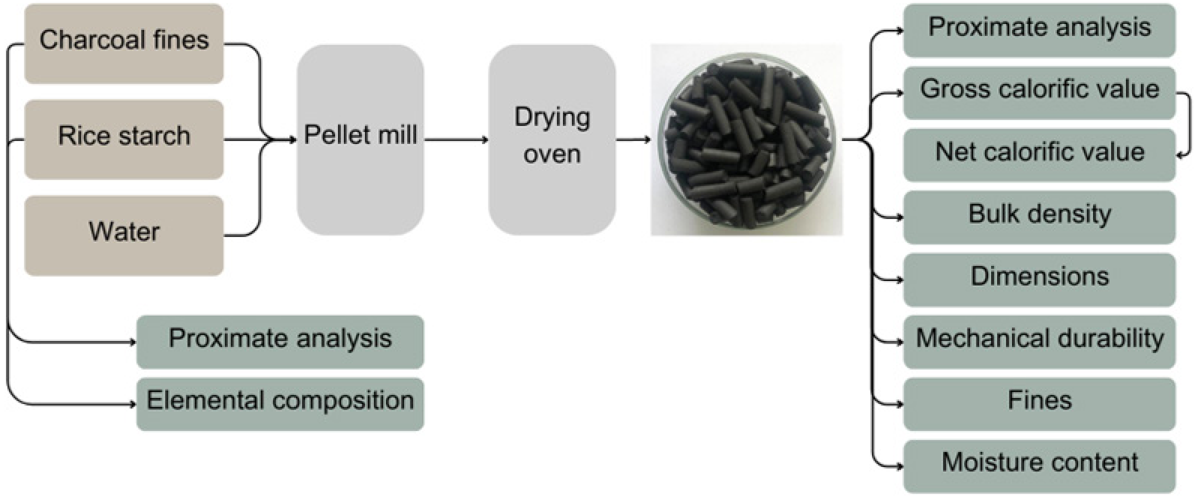

Figure 2. Experimental scheme of the study.

Figura 2. Esquema experimental do estudo.

2.4 Statistical analysis

The data were subjected to analysis of variance, and when the difference between treatments was significant, they were compared using the Tukey test at a significance level of 5%. Statistical analyzes were performed using the software SISVAR (version 5.6).

3. RESULTS

The variation in moisture content and drying time did not affect the contents of volatile matter, ash and fixed carbon, however, the treatment that used 20% water and 4 hours of drying showed a higher moisture content (Table 2).

The results for volatile matter (24.19-26.85%), ash content (1.37-1.55%) and fixed carbon (71.61-74.25%) were similar between the evaluated treatments. The length and diameter also did not vary between treatments, with the values from 19.76 to 20.38 mm and 5.94 to 6.03 mm, respectively. The moisture content (wet basis) was higher in T3 (3.34%), similar in T1 (1.63%) and T4 (1.52%), and lower in T2 (1.05%). Durability varied from 93.74% in T2 and 98.16% in T4. The values of fines and bulk density were similar in T1 and T2, with more suitable values found in T3 and T4. The highest values of GCV and NCV are in T4 and the smaller ones were found in T1. T3 and T4 stood out with highest energy density values.

4. DISCUSSION

Wood has 50% carbon. During the carbonization process, the components rich in oxygen, such as cellulose and hemicelluloses, are degraded by temperature resulting in a material with a higher concentration of carbon (Trubetskaya et al., 2020; Massuque et al., 2021). Furthermore, carbon and hydrogen are the chemical elements desired in material intended for bioenergy as they have a direct relationship with calorific value (Souza et al., 2020). However, hydrogen is much more energetic, which is why low values in the C/H ratio promote a high gain in the caloric value of biofuels (Hu et al., 2019).

The sulfur content was extremely low (0.02%) and produces sulfur oxides (SOx) when reacting with atmospheric oxygen during combustion. These oxides are toxic and have a low dew point (≈150 ºC) and can form acids that cause corrosion of parts and equipment (Sakulkit al., 2020).

The nitrogen content was low (0.04%) and associated with the high carbon content, the C/N ratio was 2180. Nitrogen is undesirable for energy generation, as it has no relationship with the calorific value and releases nitrogen oxides with combustion. These gases cause wear and tear on burning equipment and are highly toxic to the environment (Vonk et al., 2019). A low C/N ratio reduces the amount of nitrogen to be released into the atmosphere with combustion, in addition, low values for this ratio result in rapid release of CO_2 into the

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Table 2. Volatile matter content (VM), ash content (AC), fixed carbon content (FC), moisture content, wet basis (MC), length, diameter, durability, fines content, bulk density (BD), gross calorific value (GCV), net calorific value (NCV) and energy density (ED) (mean ± standard error) of black pellets.

Tabela 2. Teor de material volátil (VM), teor de cinzas (AC), teor de carbono fixo (FC), teor de umidade, base úmida (MC), comprimento, diâmetro, durabilidade, teor de finos, densidade aparente (BD), poder calorífico bruto (GCV), poder calorífico líquido (NCV) e densidade energética (DE) (média ± erro padrão) dos black pellets.

Parameter	T1 (10% - 4h)	T2 (10% - 6h)	T3 (20% - 4h)	T4 (20% - 6h)
VM (%)	24.19 ± 1.05 a	25.82 ± 4.16 a	24.35 ± 0.97 a	26.85 ± 5.60 a
AC (%)	1.37 ± 0.03 a	1.55 ± 0.11 a	1.40 ± 0.04 a	1.54 ± 0.07 a
FC (%)	72.44 ± 1.02 a	72.63 ± 4.26 a	74.25 ± 0.94 a	71.61 ± 5.67 a
MC (%)	1.63 ± 0.06 b	1.05 ± 0.07 c	3.34 ± 0.06 a	1.52 ± 0.08 b
Length (mm)	19.76 ± 0.69 a	20.38 ± 0.44 a	19.85 ± 0.88 a	20.16 ± 0.80 a
Diameter (mm)	6.03 ± 0.05 a	6.01 ± 0.04 a	6.02 ± 0.04 a	5.94 ± 0.04 a
Durability (%)	94.03 ± 2.69 bc	93.74 ± 2.84 c	98.14 ± 0.06 ab	98.16 ± 0.04 a
Fines (%)	1.75 ± 0.74 a	1.81 ± 0.88 a	0.29 ± 0.41 b	0.47 ± 0.22 b
BD (kg.m^{-3})	505.80 ± 5.99 b	522.12 ± 6.52 b	556.41 ± 9.06 a	541.92 ± 5.33 a
GCV (MJ.kg)	27.3 ± 0.046 c	29.1 ± 0.024 b	29.7 ± 0.018 ab	29.8 ± 0.042 a
NCV (MJ.kg)	26.15 ± 0.045 c	28.13 ± 0.023 b	28.0 ± 0.017 ab	28.6 ± 0.041 a
ED (MJ.m^3)	13801.71 ± 163.70 c	15203.10 ± 189.87 b	16502.52 ± 268.96 a	16131.63 ± 158.76 a

T1: 10% water and 4 hours of drying; T2: 10% water and 6 hours of drying; T3: 20% water and 4 hours of drying; T4: 20% water and 6 hours of drying. Means followed by the same letter, on the line, do not differ significantly ($p < 0.05$) by Tukey's test.

T1: 10% de água e 4 horas de secagem; T2: 10% de água e 6 horas de secagem; T3: 20% de água e 4 horas de secagem; T4: 20% de água e 6 horas de secagem. Médias seguidas de mesma letra na mesma linha, não diferem significativamente ($p < 0,05$) pelo teste de Tukey.

atmosphere during combustion, enhancing the negative effects of the greenhouse effect (Xu et al et al., 2021). In this sense, black pellets have excellent potential for burning and generating energy.

During the densification process for the production of black pellets, the temperature used was 100°C, thus, the lignocellulosic constituents that could be degraded by such temperature were already thermally degraded during carbonization, resulting in the same chemical and elemental composition in all the treatments. As for the proximate analysis, the results for volatile matter, ash content and fixed carbon were similar between the evaluated treatments because the polar extractives and hemicelluloses were volatilized or thermally degraded with increasing temperature (Esteves et al., 2022) and the amount of water and drying time did not interfere with these parameters. The high content of fixed carbon is due to the thermal degradation of oxygen and hydrogen-based constituents, such as cellulose and hemicelluloses, leaving lignin derivatives, which have a higher carbon content (Lima et al., 2023). The ash content of charcoal fines and rice starch were 1.86% and 1.45% respectively. After making the pellets, it was observed that the addition of rice starch did not negatively influence the ash content, so that all treatments tested (Table 2) presented values below 2%, which is recommended by the EN 14961-6 standard.

The higher moisture content in the pellets produced in the T3 treatment is due to the greater mass of water used in the process (20%) and the shorter drying time (4 hours). Thus, adsorbed water has a high energy demand for its removal in the form of vapor, which requires high temperature and/or longer drying time (Thybring et al., 2022). Treatment T2, with the lowest water mass and longest drying time between treatments, presented lower moisture content in the black pellets. On the other hand, the low moisture content of the mixture of charcoal and binder makes it difficult to compact the material, because water assists in heat transfer and promotes the gelatinization of the binder with the charcoal particles. Moisture content wet basis lower them 12% (A) and 15% (B) is ideal according to European standard EN 14961-6 (2012). All the pellets produced have less them 12% of moisture content, which is required for A pellets.

The pellets produced in the different treatments had the same length, diameter, and fines content, with only the durability varying (Table 2). The length and diameter of the pellets are determined by the pellets mill itself, with little or no influence from the raw material, therefore all treatments presented similar values for these two parameters. The constant pressing temperature of 100 °C brought together the starch granules, which acted as an adhesive, increasing contact between the charcoal particles and reducing expansion due to lower hygroscopicity and, consequently, not altering the diameter and length of the black pellets (Furtado et al., 2010).

The durability of the pellets was greater in treatments that used higher moisture content in the material (T3 and T4), as water acts as a binding agent, allowing softening and fluidity between the particles of the binding agent (rice starch) and charcoal, acting under the heat and pressure of pelletization, hardening and forming solid bridges when cooled, thus increasing the bonding strength between particles and, consequently, the durability of black pellets (Liu et al., 2014). The positive effect of adding water was observed for fines, with treatments with higher moisture content (T3 and T4) showing lower values and smaller variations, which is desired for black pellets.

In the study by Surup et al. (2019), in which charcoal fines bound with tar were tested in the production of pellets in a manual press, similar results for durability were found. As seen for T3 and T4, this study obtained durability results above 97.5%, meeting commercialization requirements. Greater durability values and smaller fines values means that the pellets can be transported, stored, and used with less breakage.

The generation of pellets fines was less than 2% in treatments with greater addition of water and, therefore, met the commercialization standard EN 14961-6. The lower fines content values of pellets produced with greater amounts of water are possibly due to the densification process of the pellets matrix with higher water contents, generating pellets with better bonding characteristics between particles and, consequently, less fines. Pellets with lower fines production during handling and transport should be preferred commercially (DIN EN, 2010). The fines content can increase with the moisture content of the material, generating cracks to exhaust gases, mainly water vapor, and, consequently, reducing its mechanical resistance during handling (Boschetti et al., 2019). However, for the present study, the higher moisture content in the production of black pellets reduced the fines content. The mechanical durability and fines content of the T3 and T4 treatments, where the values were above 97.5% and less than 2%, respectively, met the requirements of the EN 14961-6 standard (DIN EN, 2012).

The greater addition of moisture in the pellets production stage (T3 and T4 treatments) affected the density and calorific value of the product and, consequently, the energy density positively. The water facilitated the arrangement of the charcoal particles among themselves and with the binding agent, resulting in a more compact material, increasing the density and, consequently, greater durability and lower fines content of the pellets from these treatments. The drying time did not affect the density of the pellets, since the carbonization process degrades the hygroscopic components of wood, such as cellulose and hemicelluloses, resulting in charcoal, a material with low water adsorption capacity and zero volume variation due to moisture. Due to this, the interaction between charcoal and moisture, and whether it is removed by drying, does not influence the volume of the pellets and, consequently, its density. The lower density of pellets produced with less water explains an increase in surface area and less contact and cohesion between particles (Boschetti et al., 2019). The apparent density determines the storage and transport conditions of pellets and is directly linked to the concept of energy density, that is, the amount of energy transported per unit volume (Sotannde et al., 2010).

The GCV found in this study for T4 (29.8 MJ.kg) was similar to that found by Amaya et al. (2015), in which charcoal fine pellets bound with potato starch were tested, with a value of 30.8 MJ.kg. When selecting materials for energy purposes, a higher net calorific value is one of the main factors, especially for producing pellets to release the maximum amount of energy upon combustion. The fixed carbon content of material must be high due to its direct positive relationship with the calorific value (Peng et al., 2013). To comply with the EN 14961-6 standard (DIN, 2012), the net calorific value must be greater or equal to14.1 MJ.kg for A pellets and greater or equal to 13.2 MJ.kg for B pellets, which was achieved by all treatments for the black pellets.

Treatments T3 and T4 showed higher energy density. This parameter is the product of the density and gross calorific value of the material. The addition of moisture in the pellets production process facilitated its compaction and increased its density. Furthermore, these treatments presented high calorific values, which resulted in high energy density. High values of energy density are important for optimizing transportation and use of the furnaces. In both cases, the space to be occupied by the biofuel is limited, and it is interesting that it presents the greatest amount of energy possible in this confined space (Lima et al., 2023).

5. CONCLUSION

Pellets produced from charcoal have production and commercialization potential, being an important alternative for using charcoal fines. The content of volatile matter, ash and fixed carbon were the same between the different treatments. The drying time did not influence the quality of the pellets, with a time of four hours being recommended, due to the lower energy expenditure of the drying operation. Treatments with 20% water addition showed better compaction and arrangement of particles, resulting in higher density and energy density and lower generation of fines. Therefore, the treatment with 20% water and 4 hours of drying showed better results.

All treatments met the standard regarding diameter, length and moisture content (wet basis) and ash content EN 14961-6 (DIN, 2012). For bulk density, no treatment presented satisfactory results, with values lower than those established by the standard (above 600 kg/m³). Regarding mechanical durability, treatments T3 and T4 presented satisfactory results that met the minimum stipulated by the standard (97.5% for A). All treatments expressed satisfactory values for the percentage of fines generated, however T1 and T2 present higher values, close to what is accepted for A pellets (less them 2%). The net calorific value of the pellets, for all treatments, was above that expected by the standard. The fines used to produce the pellets also meet the standard values for nitrogen and sulfur contents. Finally, the T3 and T4 treatments were the one that best suited the commercialization standard, but further testing is required for the elements that were not tested, such as chlorine content. However, T3 uses less drying time, with equally satisfying results when compared to T4, making it the best treatment.

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AUTHOR CONTRIBUTIONS

FBC: Conceptualization, methodology, investigation, original draft preparation, writing. CCNM: Writing, review, editing. LCP: Writing, review, editing. LVLL: Methodology, investigation, review. AJVZ: Writing, review, editing. ACOC: Methodology, investigation, original draft preparation, review. VRC: Supervision, review, editing. SOA: Funding, review, editing. IFD: Supervision, review, editing.

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