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Non-conventional biomasses for briquette manufacturing: densification parameters and quality

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ABSTRACT: The objective of this study was to produce and evaluate briquettes from residues of corn crops and from its association with cotton crop residues, rice husk or elephant grass for energetic use. Data on moisture, immediate chemical analysis, bulk density, higher calorific value (HCV), energetic density, volumetric expansion and mechanical strength were used to compare the treatments. Biomass was densified at 125ºC under 15 MPa of pressure for 8 minutes. HCV results were similar for all treatments. The immediate chemical analysis showed high VC and FCC for all treatments. The highest AC was observed in briquets with 20% of rice husk (4.22%) and the lowest in briquets from corn crop residues (2.67%). The apparent density of briquets ranged from 0.96 to 1.10 t.m^{-3} and the energetic density from 17.53 and 20.27 GJ.m-3. All treatments delivered moderate volumetric expansion, with a maximum of 14.5% by corn crop residue briquets. Water absorptions were similar for all treatments. Mechanical strength by diametrical compression revealed desirable behavior by undefined breaking points. Data demonstrate the energetic potential of solid biofuels produced by the densification of corn crop residues, as well as their mixtures in different proportions with cotton crop residues, rice husk and Elephant grass.
Keywords: alternative fuel; biomass; densification; energetic use.

Biomassas não convencionais para fabricação de briquetes: parâmetros de densificação e qualidade

RESUMO: O objetivo deste trabalho foi produzir e avaliar briquetes a partir de resíduos da cultura do milho e sua associação com resíduos da cultura do algodão, casca de arroz ou capim elefante para uso energético. Dados de umidade, análise química imediata, densidade aparente, poder calorífico superior (HHV), densidade energética, expansão volumétrica e resistência mecânica foram utilizados para comparação dos tratamentos. A biomassa foi densificada a 125ºC sob pressão de 15 MPa por 8 minutos. Os resultados do HCV foram semelhantes para todos os tratamentos. A análise química imediata mostrou VC e FCC elevados para todos os tratamentos. A maior AC foi observada nos briquetes com 20% de casca de arroz (4,22%) e a menor nos briquetes de resíduos da cultura do milho (2,67%). A densidade aparente dos briquetes variou de 0,96 a 1,10 t.m^{-3} e a densidade energética de 17,53 e 20,27 GJ.m^{-3}. Todos os tratamentos proporcionaram expansão volumétrica moderada, com máximo de 14,5% de briquetes de resíduos da cultura do milho. As absorções de água foram semelhantes para todos os tratamentos. A resistência mecânica por compressão diametral revelou comportamento desejável por ponto de ruptura indefinido. Os dados demonstram o potencial energético dos biocombustíveis sólidos produzidos pelo adensamento dos resíduos da cultura do milho, bem como suas misturas em diferentes proporções com resíduos da cultura do algodão, casca de arroz e capim elefante.
Palavras-chave: combustíveis alternativos; biomassa; adensamento; aproveitamento energético.

1. INTRODUCTION

Brazil is one of the world's largest producers of agricultural and forest biomass, with ongoing expansions in area and productivity, particularly in the Midwest Region. The increase in energy demand by the agroindustry sector is also notable, driven by the need for drying, storage, and processing of grains, as well as for biofuel processing (BRASIL, 2020). The production of firewood and charcoal has proven inadequate to meet the demands of agroindustries, leading to increased prices for reforested biomass and, consequently, a greater reliance on products from native forests (SIMIONI et al., 2017).

Energy from renewable sources has become a prominent topic, and the energetic use of crop residues has been identified as a viable option. This approach not only provides an alternative but also adds value to production chains (SILVA et al., 2017). However, residual biomass exhibits undesirable characteristics for direct fuel use, including high moisture content, heterogeneity, low bulk density, and low energetic density (NAKASHIMA et al., 2017; SOUZA; VALE, 2017; KEBEDE et al., 2022).

Energy densification of biomass can enhance its fuel properties (Gong et al., 2023). Briquettes are produced by compacting chopped lignocellulosic biomass under high pressure. This process generates frictional heat, which results in the plasticization of lignin and the formation of a stable, homogeneous block. The primary advantage of this technique lies in the logistical benefits of transporting and storing the compacted biomass (SOUZA; VALE, 2013; NAKASHIMA et al., 2017; SILVA et al., 2017; KEBEDE et al., 2022). Briquettes, as a concentrated solid biofuel, offer a viable alternative to firewood (DASS et al., 2019).

Biomass moisture is a critical parameter in the compaction process. Water acts as a natural binder, facilitating the solubilization of certain biomass components and enhancing particle interaction through Van der Waals forces, which contribute to compaction (QUIRINO; BRITO, 1991; SILVA et al., 2021). However, high moisture content can damage compaction equipment and reduce the burning efficiency of the briquettes due to lower calorific value. Ideally, biomass for densified fuel production should have a moisture content between 8% and 16%. Deviations from this range can result in unstable briquettes that may crumble during handling, transportation, and storage (SCHÜTZ et al., 2010; ABDEL AAL et al., 2023).

Desirable briquette characteristics for fuel use include high calorific value, high density, low ash content, dimensional stability, and good mechanical resistance (NAKASHIMA et al., 2017). Blocks with higher apparent densities are advantageous as they reduce costs and simplify transportation and storage due to the smaller product volume (YAMAJI et al., 2013; SILVA et al., 2015; KUMAR et al., 2021). Energetic density, defined as the amount of energy per unit volume, varies according to the biofuel's properties, such as chemical composition, calorific value, and ash content (SOUZA; VALE, 2013). The stackability of the blocks during storage is determined by the mechanical strength of the briquettes (PAULA et al., 2011).

Therefore, the objective was to produce briquettes from corn (*Zea mays* L.) crop residues, either alone or in combination with cotton crop residues (*Gossypium* L.), rice husk (*Oryza sativa*), and elephant grass (*Cenchrus purpureus* S.), and to evaluate their potential as fuel based on their physicochemical, energetic, and mechanical properties.

2. MATERIAL AND METHODS

Samples of corn and cotton crop residues, as well as elephant grass, were collected in the municipality of Itanhangá, Mato Grosso, during the 2020/2021 crop season. The crop residues (corn and rice) are largely produced in the region, and elephant grass has been cultivated for energetic purposes. The agricultural residue samples included the aerial parts of the plants, excluding the roots. The corn residues comprised straw, cob, stem, and leaves. The cotton residues included stalks, side branches, leaves, capulin remnants, and cotton fibers. The elephant grass samples (Var. BRS Capiaçú) include leaves and stems. Rice husks were obtained from an agroindustry in the city of Sinop. The biomasses were analyzed, and their characteristics are detailed in Table 1.

Table 1. Chemical and energetic characteristics of corn crop residues (Corn), cotton crop residues (Cotton), rice husk (Rice), and Elephant Grass (Grass).
Tabela 1. Características químicas e energéticas dos resíduos da cultura do milho (Milho), resíduos da cultura do algodão (Algodão), casca de arroz (Arroz) e Capim Elefante (Capim).

Properties	Biomass			
	Corn	Cotton	Rice	Grass
Higher Heat Value (MJ kg^{-1})	17.73	18.06	18.11	17.58
	80.76	83.86	75.70	75.42
	17.67	12.55	16.20	19.72
	1.56	3.59	8.10	4.86

The biomasses, except for rice husk, underwent two pre-processing steps before compaction: drying and fractionation. The samples were dried by exposure to ambient air and subsequently processed using a forage chopper (Trapp TRF-700), techniques commonly employed on an industrial scale. The preprocessed samples exhibited varying granulometry, predominantly below 2.0 cm.

Briquettes were produced by densifying corn crop residue, both alone and in combination with other biomasses, resulting in a total of 10 treatments, as outlined in Table 2.

Briquettes were produced using a hydraulic press with temperature and pressure control (Lipel LB-32). The pressure applied for biomass densification was 150 Bar (153 kgf cm^2), and the volume of material was constrained by the dimensions of the densification matrix (32 mm diameter and 300 mm height). The pressure was maintained for 8 minutes, with a temperature of 125°C. Twelve briquettes were produced per treatment, each weighing approximately 18 g.

Table 2. Composition of briquettes – treatments.
Tabela 2. Composição dos briquetes – tratamentos.

Treatment	Composition
C100	100% of corn crop residues
CC10	90% of corn crop residues + 10% of cotton crop residues
CC15	85% of corn crop residues + 15% of cotton crop residues
CC20	80% of corn crop residues + 20% of cotton crop residues
CR10	90% of corn crop residues + 10% of rice husk
CR15	85% of corn crop residues + 15% of rice husk
CR20	80% of corn crop residues + 20% of rice husk
CG10	90% of corn crop residues + 10% of Elephant grass
CG15	85% of corn crop residues + 15% of Elephant grass
CG20	80% of corn crop residues + 20% of Elephant grass

Immediately after production, the briquettes were placed in a desiccator to cool. Once cooled, the densified blocks were weighed using an analytical balance with a precision of 0.001 g. The volume of each briquette (cylinder) was calculated based on the average of the diameter and height, measured with a digital caliper (sensitivity of 0.01 mm). From the mass and volume of each briquette, the specific density was determined. The briquettes were stored in an open environment, protected from rain, to assess volumetric expansion and moisture absorption over time.

Mass and volume measurements were taken at 24-hour intervals, with measurements ceasing once the briquettes showed stability. Dimensional stability was evaluated based on volumetric expansion, as described by Equation 1.

$$VExp = \left[\frac{V_n - V_1}{V_1}\right].100 \qquad (01)$$

where: VExp – Volumetric Expansion of briquettes (%); V1 - Volume of briquette immediately compaction, cm³; Vn - Volume of briquette at n intervals of 24 hours, cm³.

Moisture content was determined by oven drying (Solab SL-102) at 105°C until a constant mass was achieved. The moisture behavior of the densified blocks was assessed over the stabilization period based on mass variations.

Samples were ground using a Wiley knife mill (Marconi MA-340) for preparation. Particles smaller than 0.84 mm were completely dried in a circulating oven and then analyzed. The higher heating value (HHV) was determined using a calorimeter operating in isoperibolic mode (IKA C-200), according to ASTM E711-87 (ASTM, 1987). Immediate chemical analyses were conducted for all treatments to measure volatile content (VC), fixed carbon content (FCC), and ash content (AC) through thermal degradation in a muffle furnace (Magnus) (ASTM, 1984). Energetic density was calculated by multiplying the higher heating value by the apparent density of the dry briquette (after stabilization).

Mechanical strength was evaluated for the treatments that exhibited the best physicochemical and energetic properties within each waste group. Three specimens per treatment were tested using a universal testing machine, applying a compression load of 300 kN at a constant speed of 0.5 mm s^{-1}, following the adaptation of the NBR 7222 standard (ABNT, 1994). The maximum number of blocks that can be stacked without causing damage was estimated by applying a safety factor of 3 to the tensile strength, as described by SILVA et al. (2015).

All data were subjected to the Shapiro-Wilk normality test. Analysis of variance (ANOVA) was performed on data with a normal distribution with $p< 0.05$. Differences between treatments were assessed using the Scott-Knott test for mean comparison at a level of 5% probability.

3. RESULTS

There was no statistical difference between treatments for the content of volatile materials, fixed carbon and higher heating value (VC: F= 0.59; p= 0.78; FC: F= 0.54; p= 0.82 and HHV: F=1.56; p= 0.19) (Table 1). However, the ash content was higher in the treatment with a higher percentage of rice husk. The apparent densities of the densified blocks, after their stabilization, as well as the energy densities (Figure 1).

The briquettes produced by mixing corn residue with Elephant grass and those with the highest level of rice husk in the corn residue showed higher bulk density and energetic density compared to the other treatments.

After stabilization, which was achieved after eight days of exposure to ambient air, the maximum expansion (Figure 2) observed was greatest for briquettes made from corn crop residues (14.52%) and least for briquettes produced with the highest percentages of cotton crop residues and elephant grass (CC20; CG20 = 8.10%). Moisture absorption was highest for briquettes made from corn crop residues, ranging from 2.59% to 8.58% during the stabilization period. Briquettes produced with rice husk exhibited the lowest moisture variations during the study period.

The briquettes showed low specific strains about their diameters (Figure 3), withstanding average stresses of 1.03 MPa for C100, 1.06 MPa for CC20, 1.05 MPa for CR20, and 1.07 MPa for CG20. These values represent the amount of stress that caused a region of yielding by increasing the strain without the addition of stress. The rupture of the briquette was not complete, showing a slight weakening in its structure, with cracks appearing. After this first stage of embrittlement, the briquettes reacted to an increase in tension, with maximum diametral compressive strengths of up to 1.62, 1.93, 1.81, and 2.04 MPa for C100, CC20, CR20 and CG20, respectively. In this new region, there was also no total rupture of the blocks, only embrittlement of their structure (Figure 4 a and b).

Table 4 shows the results for the maximum number of blocks that can be stacked and the consequent maximum stacking height that does not cause a breakdown in the briquettes.

Table 3. Immediate Chemical analysis and Higher Heating Value (HHV) (Mean ± Standard Deviation) of briquettes.
Tabela 3. Análise química imediata e poder calorífico superior (HHV) (média ± desvio padrão) dos briquetes.

Treatments	Volatile Content (VC%)	Fixed Carbon Content (FC%)	Ash content (AC%)	HHV (MJ kg⁻¹)
C100	77.71±0.03a	19.61±1.76a	2.67±0.02e	17.76±0.56a
CC10	77.60±0.90a	19.58±0.87a	2.82±0.04e	17.79±0.12a
CC15	76.60±1.36a	20.06±1.37a	3.34±0.05c	17.94±0.33a
CC20	76.64±1.75a	20.33±1.81a	3.03±0.12d	18.55±0.72a
CR10	77.95±1.11a	18.69±1.08a	3.36±0.03c	18.20±0.04a
CR15	77.76±0.76a	18.53±0.72a	3.71±0.06b	18.25±0.05a
CR20	76.27±1.05a	19.52±1.05a	4.22±0.02a	18.35±0.39a
CG10	77.09±1.59a	19.73±1.72a	3.17±0.18d	18.10±0.11a
CG15	76.94±1.21a	19.92±1.19a	3.14±0.06d	18.15±0.11a
CG20	76.80±1.14a	19.64±1.07a	3.56±0.22b	18.31±0.26a

Means followed by the same letter in the same column do not differ among themselves by the Scott-Knott test at the level of 5% probability.
Médias seguidas pela mesma letra na mesma coluna não diferem entre si pelo teste de Scott-Knott ao nível de 5% de probabilidade.

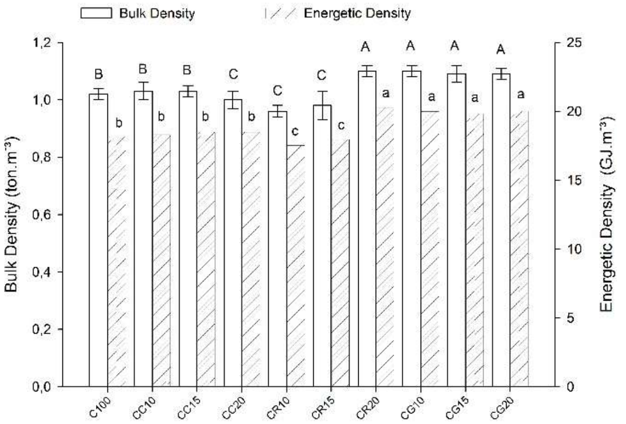

Figure 1. Bulk density and energetic density of briquettes. Means followed by the same capital letter for the bulk density column or by the same lower-case letter for the energetic density column do not differ by the Scott-Knott test at the level of 5% probability.
Figura 1. Densidade aparente e densidade energética dos briquetes. Médias seguidas da mesma letra maiúscula para a coluna de densidade aparente ou da mesma letra minúscula para a coluna de densidade energética não diferem pelo teste de Scott-Knott ao nível de 5% de probabilidade.

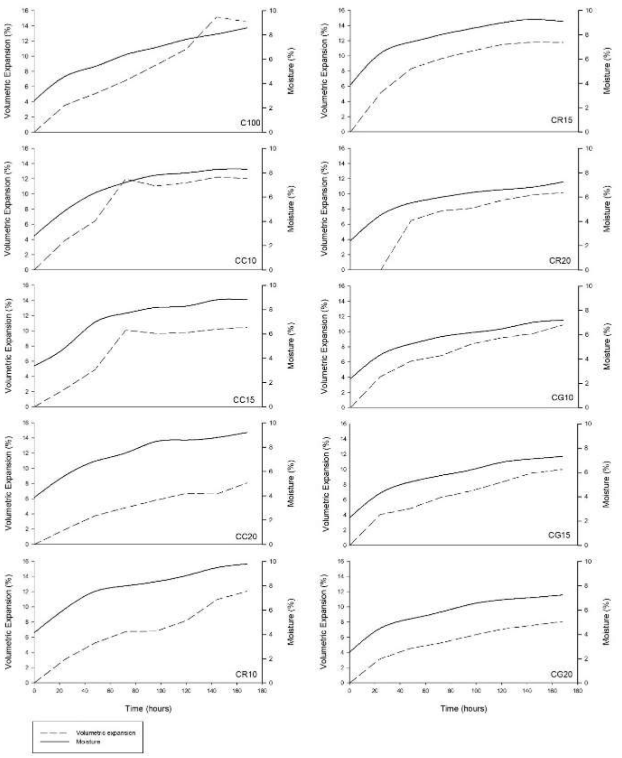

Figure 2. Volumetric expansion and moisture for treatments.
Figura 2. Expansão volumétrica e umidade dos tratamentos.

Table 4. Number of blocks (Nb) and maximum height of stack (Hstc) for selected treatments.
Tabela 4. Número de blocos (Nb) e altura máxima de pilha (Hstc) para tratamentos selecionados.

Treatments / Parameters	C100	CC20	CR20	CG20
N_b	1533	1586	1566	1605
H_{stc} (m)	49.08	50.77	50.12	51.36

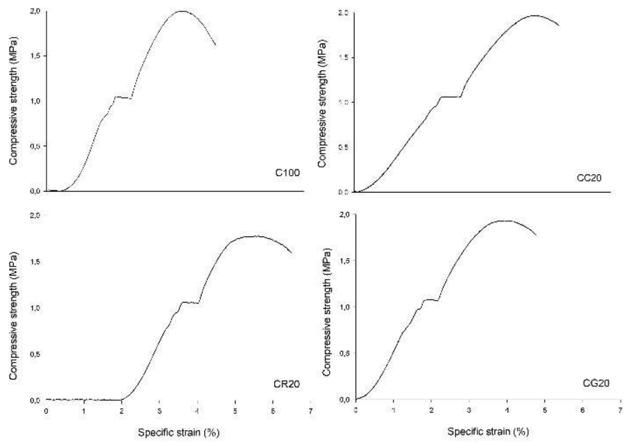

Figure 3. Diametral compressive strength for briquettes.
Figura 3. Resistência à compressão diametral para briquetes.

(a) (b)

Figure 4. Briquettes before (a) and after stress analysis (b).
Figura 4. Briquetes antes (a) e depois da análise de tensão (b).

4. DISCUSSÃO

The average HHV did not differ for the alternative biomasses added to the corn residue, not even for the concentration ranges. The values obtained are similar to the calorific values of the biomass used to make the blends and to the results found in the scientific literature for briquettes made from wood products and other lignocellulosic biomass (AMORIM et al., 2015; PADILHA et al., 2016).

The immediate chemical analysis showed high values of VC and values considered acceptable for FC and AC (AC< of the briquettes. Compared with the raw material, there was a slight increase in fixed carbon content, which may have occurred due to the loss of part of the volatile materials due to heating during compression and consequent fixation of part of the carbon (FILHO, 2013). These results demonstrate the potential of these products as fuel since the high content of volatile matter facilitates the ignition of the fuel, and the presence of fixed carbon makes the burning slower and more uniform, resulting in longer life in furnaces (AMORIM et al., 2015).

The average values obtained for AC are close to those found in the wood of *Pinus* sp. L. (3.20%), *Tabebuia impetiginosa* M. (4.68%) and *Daugerbia cearensis* D. (4.13%) (AMORIM et al., 2015). The higher ash contents for the treatments containing rice husk were expected due to the presence of silica in this residue. Ash contents of up to 22% have already been reported for the residue concerned (SOUZA; VALE, 2013). As the ash content in solid biofuels increases, less energy is available for the burning process, and the need for maintenance of the systems is greater (MCKENDRY, 2002).

The average bulk densities found in this work were close to those cited for briquettes produced under the same conditions of pressure, temperature, and compaction time for coffee parchment (0.95 t m^{-3}) and rice husks (1.08 t m^{-3}). However, briquettes produced from corn, soybean, sugarcane, and wood residues, compressed at the same pressure but without the action of temperature, showed lower values for bulk density (PAULA et al., 2011).

The diverse granulometry and the presence of particles larger than those typically used in the compression process did not cause losses to the final bulk density. The smaller particle sizes have larger surface areas and promote better interactions, acting as a binder for larger particles, which probably allowed the interweaving of the fibers contained in the mixtures during the manufacturing process. This can be explained by the efficiency in the action of pressure and temperature for the formation of a homogeneously compacted briquette. This can bring advantages to the industrial process since no complex operations to homogenize the particle size of the waste would be required (SOUZA; VALE, 2013).

The high apparent densities found, besides reflecting high energy densities, allow for better packaging since dense materials are less hygroscopic, which ensures greater stability and resistance to the structure of the densified block (YAMAJI et al., 2013; PAULA et al., 2011). The significant variation in the bulk density results of the blocks containing rice hulls suggests that the increment of 5% of this biomass in the mixtures (from 15% hulls in the mixtures) interfered positively with the bulk density of the blocks.

All treatments presented high values for energetic density, following the apparent densities of the blocks, since these parameters are directly proportional (SOUZA; VALE, 2013). All values obtained in this study are higher than those found by other authors, under similar conditions, for corn crop residue briquettes (15.73 GJ m^{-3}), eucalyptus sawdust briquettes (15.54 GJ m^{-3}) (PROTÁSIO et al., 2011), and for 56-month-old eucalyptus charcoal (11.44 GJ m^{-3}) (PROTÁSIO et al., 2014). Energetic density is one of the most widely used parameters to determine the quality of solid biofuels (SOUZA; VALE, 2013).

The tendency to increase the volumetric expansion for the briquettes with higher participation of corn crop residue in its composition can be explained by the greater hygroscopicity of this residue in comparison to the other residues studied, increasing the volumetric expansion of the block by water absorption. All treatments showed lower values than those found by other authors for volumetric expansion of briquettes produced at similar compressing conditions (temperature, pressure and time), from corn crop residues (35.30%) and *Eucalyptus* sp. (19.00%) (PROTÁSIO et al., 2011).

The higher volumetric expansion rate in the first days of stabilization in all treatments is consistent with the results published in previous studies. However, the occurrence of rain is decisive for the definition of the volumetric expansion behavior of the blocks. On days when heavy rains precede the measurements, the relative humidity of the air is high, thus increasing the absorption of moisture and consequent expansion of the blocks. All treatments achieved stability up to 168h after compaction, even when exposed to critical environmental conditions. High volumetric variations can reduce the apparent and energetic densities of the briquettes, as well as negatively affect the mechanical strength of the product. (PROTÁSIO et al., 2011).

The densification process was favored by the moisture of the studied biomasses, which were within the appropriate range. This also favored the formation of a product with low final moisture (SILVA, 2018). After stabilizing the blocks, the moisture content was lower than 10%. The blocks showed adequate moisture throughout the stabilization period, showing a good pattern for water absorption until its dimensional stabilization, even being kept in an environment without temperature and air humidity control.

The presence of short and long fibers in the raw material for the manufacture of briquettes may have been responsible for the non-rupture of the briquettes when subjected to high compressive stresses. However, the appearance of cracks in briquettes increases their surface area exposed to environmental conditions, favoring the absorption of moisture.

The maximum stacking heights found were close to 50 meters. These heights are lower than those found for briquettes produced from eucalyptus sawdust (87 m), higher than those of sugarcane bagasse briquettes (20 m), and close to briquettes made from pine sawdust (46 m) and sugarcane straw (59 m), considering the compressive strengths of 1.22 MPa, 0.27 MPa, 0.70 MPa, 0.70 MPa and 0.98 MPa, respectively (SILVA et al., 2015). Considering that conventional warehouses have heights between 10 and 12 m, therefore, all treatments present good conditions to support stacking without causing any damage to their structures.

4. CONCLUSIONS

Based on the results, it is possible to conclude that the corn crop residues, as well as the blends with cotton harvest residue, rice husks, and Elephant grass, in different proportions, present a potential for energetic use from the production of densified blocks without the addition of binders and with varied granulometry.

The briquettes produced the present potential for replacing the solid fuels currently used in furnaces, especially in agroindustries, which are installed in regions with great availability of the materials studied. The use of agricultural residues or short-cycle biomass as fuel can reduce the pressure on forest energy resources, which have been exploited at a rate incompatible with their natural restoration condition, especially in the agricultural and agro-industrial frontier regions in Brazil.

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