







Articles

Influence of grain size on the energy properties of briquettes made from grape stalks: a sustainable approach for Brazilian viticulture

Influência da granulometria nas propriedades energéticas de briquetes produzidos com engaço de uva: uma abordagem sustentável para a viticultura brasileira

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ABSTRACT

This study proposes a sustainable approach to meeting growing global energy demands, which are mainly made up of fossil fuels. In this context, grape stalks, an abundant waste product from viticulture, have emerged as a promising alternative for generating bioenergy. Initially, the chemical properties of the raw material were determined, such as the content of total extractives and holocellulose. Subsequently, briquettes were produced in a laboratory briquetting machine with four different grain sizes (2, 3, 5 and 8 mm). The energy quality of the briquettes produced and the impact of particle size on their physical and mechanical properties were evaluated. Detailed analyses of the briquettes' physical and mechanical properties, including bulk, apparent and energy density, compressive strength and volumetric expansion was realized. The size of the grape pomace particles had a significant influence on the physical, mechanical and energy properties of the briquettes. Remarkably, the smallest grain size (2 mm) provided more effective material accommodation, resulting in higher strength and density. The results achieved in terms of physical and mechanical properties show that briquettes made from residual biomass from viticulture emerge as compacted materials of high energy quality. Furthermore, this study goes beyond the technical dimension by presenting itself as a proposal that can be adapted to other production chains in which lignocellulosic waste is still little used. By integrating technological innovation, environmental sustainability and socio-economic inclusion, the research outlines a viable strategy towards carbon neutrality, especially in areas with an agricultural vocation, thus contributing to the goals of sustainable development.

Keywords: Bioenergy; Energy properties; Granulometry; Residual biomass

RESUMO

Esse estudo propõe uma abordagem sustentável para suprir as crescentes demandas energéticas globais, compostas principalmente por combustíveis fósseis. Nesse contexto, o engaço da uva, um resíduo abundante da viticultura, surge como uma alternativa promissora para a geração de bioenergia. Inicialmente foram determinadas as propriedades químicas da matéria-prima, como os teores de extrativos totais e holocelulose. Posteriormente, briquetes foram produzidos em uma briquetadeira laboratorial com quatro distintas granulometrias (2, 3, 5 e 8 mm). Foram avaliados a qualidade energética de briquetes produzidos e o impacto da granulometria das partículas nas suas propriedades físicas e mecânicas. Análises detalhadas das propriedades físicas e mecânicas dos briquetes, incluindo densidade à granel, aparente e energética, resistência à compressão e expansão volumétrica foram realizadas. O tamanho das partículas do engaço da uva exerceu significativa influência nas propriedades físicas, mecânicas e energéticas dos briquetes. Notavelmente, a menor granulometria (2 mm) propiciou uma acomodação mais eficaz do material, conferindo-lhe maior resistência e densidade. Os resultados alcançados das propriedades físicas e mecânicas evidenciam que os briquetes provenientes da biomassa residual da viticultura emergem como materiais compactados de alta qualidade energética. Além disso, esse estudo vai além da dimensão técnica ao se apresentar como uma proposta que pode ser adaptada a outras cadeias produtivas, nas quais resíduos lignocelulósicos ainda são pouco aproveitados. Ao integrar inovação tecnológica, sustentabilidade ambiental e inclusão socioeconômica, a pesquisa traça uma estratégia viável rumo à neutralidade de carbono, sobretudo em áreas com vocação agrícola, contribuindo assim para os objetivos de desenvolvimento sustentável.

Palavras-chave: Bioenergia; Biomassa residual; Granulometria; Propriedades energéticas

1 INTRODUCTION

The energy production sector is responsible for around 75% of global greenhouse gas emissions (IEA, 2021). Despite the growing expansion of renewable energy sources, non-renewable sources still account for a large part of global supply, with fossil fuels being the main contributors to carbon dioxide emissions, one of the main gases related to climate change (OSEI OPOKU *et al.*, 2024). Given this scenario, the energy transition to less polluting sources is becoming necessary and is a fundamental step towards tackling the climate crisis. In addition, this change is essential for meeting the Sustainable Development Goals (SDGs), especially SDG 7 - Clean and affordable energy, and SDG 13 - Action against global climate change (OSEI OPOKU *et al.*, 2024).

Among renewable sources, biomass stands out as a renewable energy source with wide popularity and growth potential. Its prominence is due to the ease with which

it can be obtained, since it is generated as waste in various agricultural and industrial activities, which favors its use in different regions of the world (KIBRIA *et al.*, 2024). In Brazil, viticulture plays a fundamental role in the sustainability of small rural properties, contributing to the socio-economic development of various regions, especially given the growing demand for wine products (MELO, 2021). However, the expansion of cultivation areas and wine agro-industries is leading to a significant production of biomass waste, making it essential to find solutions to reduce environmental impacts. Grape stalk - the woody part of the bunch, made up of the stem and its branches - is the main by-product of winemaking, accounting for between 2.5% and 7.5% of the total weight of the fruit (PUJOL *et al.*, 2013). In this context, the efficient management of this waste is essential to promote the sustainable use of natural resources, reduce waste and contribute to SDG 12, which aims to ensure more responsible consumption and production patterns.

One of the challenges related to the use of biomass is its disposal in nature, predominantly in dispersed form, resulting in a relatively low energy density (OBI; PECENKA; CLIFFORD, 2022). Therefore, compaction technologies, such as briquetting, can be adopted to maximize its energy efficiency (TUAN HOANG; VIET PHAM, 2021). During the briquette production process, when the particles are subjected to friction with the force of the briquetting press die, heat is generated, leading to a decrease in the moisture content of the biomass. This effect results in an increase in the apparent and energy density of the briquettes compared to the original material (NONES *et al.*, 2017).

In the briquetting process, grain size is a crucial variable in the production of briquettes for energy purposes, directly impacting on compaction and the maximum strength supported by them (RIBEIRO *et al.*, 2022). Studies report that smaller grain sizes favor efficient biomass accommodation, provide greater surface area, strengthen the particle-particle bond and give briquettes optimized physical and mechanical properties (RIBEIRO *et al.*, 2022). Despite the growing number of studies on the effect of grain size on the briquetting of different types of biomass, there are still few studies specifically focused on grape stalks, which reinforces the need for further research into this waste.

Given the growing demand for sustainable solutions in the energy matrix and the need to make better use of the waste generated by viticulture, this study proposes the use of grape stalks to produce briquettes. Although residual biomass represents a promising alternative on the world stage, the stalk is still little used in Brazil, indicating a gap in the literature and reinforcing the relevance of its investigation. Therefore, briquetting stalks is a viable strategy for recovering waste, offering a solution in line with SDG 7 and SDG 12. The research evaluated how grain size distribution influences the physical and mechanical properties of briquettes, based on the hypothesis that reducing grain size can improve their energy quality.

2 MATERIALS AND METHODS

2.1 Preparation and characterization of the material

For the production of the briquettes, grape stalks obtained from commercial wineries located in the municipality of Santa Maria, Rio Grande do Sul, Brazil, were used, with no control over the species used. The lignocellulosic biomass was air-dried to a final moisture content of approximately 11%, homogenized and stored in polyethylene bags in an air-conditioned chamber with a controlled temperature of $20 \pm 2^\circ\text{C}$ and a relative humidity of $65 \pm 5\%$. A sample of the lignocellulosic biomass was collected for chemical analysis, following the recommendations of the TAPPI standard (T 264-om-88). Through this analysis, the lignin (T222 om-98), total extractives (T264-CM-97) and holocellulose (expressed as the difference between the components) contents were obtained.

Bulk density was determined for each grain size fraction of the lignocellulosic biomass used to produce the briquettes. Bulk density was determined in accordance with ABNT standard NBR 6922 (1981) and with three replicates for each treatment.

2.2 Briquette production

Different grain sizes of grape stalks (2, 3, 5 and 8 mm) were used to produce the briquettes, making up treatments T1, T2, T3 and T4, respectively, obtained by grinding in a Willey-type mill and sieving. These grain sizes were determined based on preliminary studies. Compaction took place at 100 Bar in a briquetting machine (LB 32, LIPPEL, Brazil), with the volume of the mold being filled with 40 g of homogeneous lignocellulosic biomass. The residence time was five minutes, followed by three minutes of cooling. Seven briquettes from each treatment were produced and used in the analysis, with an average height of 35 mm and a diameter of 32 mm. After compaction, the briquettes were placed in an air-conditioned chamber at 20°C and 65% relative humidity until they reached a constant mass.

2.3 Analysis of briquette properties

To assess the effect of grain size on briquette properties, samples from each treatment were analyzed in terms of apparent and energy density, volumetric expansion and mechanical strength.

2.3.1 Apparent and energetic density of the briquettes

The apparent density of the briquettes was determined using the ratio between mass and volume. The volume was obtained by measuring the diameters at three points on the briquettes (top, middle and bottom) using a digital caliper, and the mass was determined on an analytical balance with a precision of 0.001 g.

The energy density was determined for each treatment based on the useful calorific value and the apparent density. The useful calorific value was obtained from the upper calorific value measured in a pump-type adiabatic calorimeter.

2.3.2 Volumetric expansion

The volumetric expansion was determined by measuring the diameter and height of each briquette using a digital caliper. The volume was then calculated to form

the expansion curve. Measurements were taken immediately after manufacture and then at 1, 2, 3, 4, 5, 12 and 24 hours.

2.3.3 Compression resistance

The briquettes were subjected to a mechanical compressive strength test on a universal testing machine (EMIC DL 30000N), at a speed of 3 mm min⁻¹, to obtain the maximum force withstood until the briquette completely broke, in accordance with ABNT standard NBR 11093-9 (2009).

2.4 Statistical analysis

The experimental design used was a completely randomized design. The data was subjected to the Bartlett and Kolmogorov-Smirnov tests to verify the homogeneity of the variances and the normality of the errors. All response variables in this study were subjected to analysis of variance, and when the significance of the effects indicated by the analysis of variance was observed, a comparison of means analysis was performed using the Tukey test ($P < 0.05$), using the statistical software R Studio (R Core Team, 2017).

Additionally, to verify the correlation effects between the response variables and the distribution of treatments, the data were subjected to a multivariate analysis of principal components (PCA), using the FactoExtra package (KASSAMBARA; MUNDT, 2017), with the assistance of R software (R Core Team, 2017). PCA is performed according to a set of principal components (in this case PC1 and PC2), which are composed of a set of standardized, orthogonal linear combinations that collectively explain the original variance of the data.

3 RESULTS AND DISCUSSIONS

3.1 Chemical composition of grape stalks

The grape stalks had a 38.82% holocellulose content, 28.91% lignin and 32.27% extractives. The significant presence of lignin and extractives is indicative of the

material's potential for energy application, considering that these components are associated with greater thermal stability, mechanical resistance and higher calorific value (HANSTED *et al.*, 2016; KUMAR *et al.*, 2020). Higher amounts of lignin are considered beneficial because, in addition to contributing significantly to the energy value, it acts as a natural binder, promoting greater cohesion between the particles and giving the solid biofuel strength (DE SOUZA *et al.*, 2020).

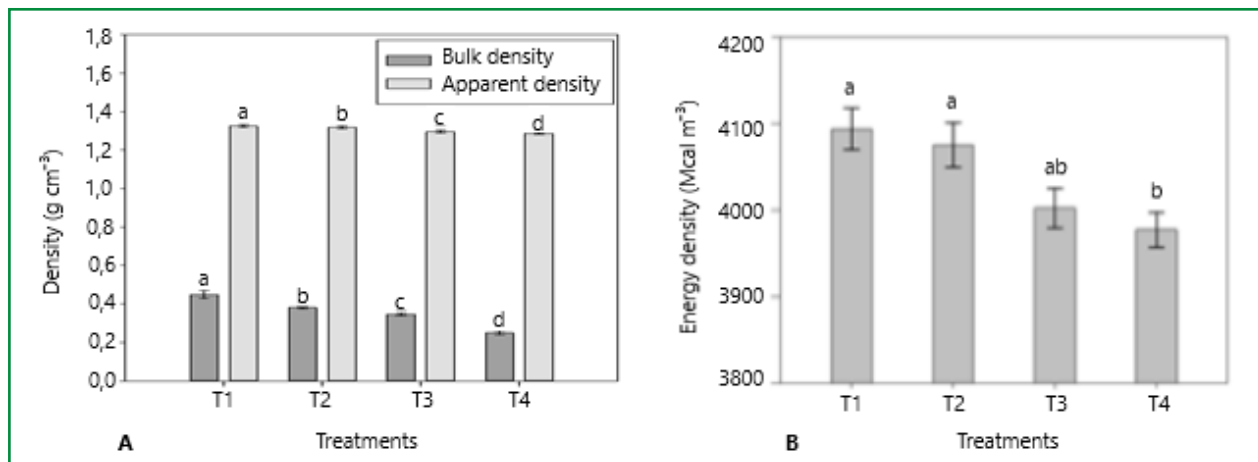
According to the studies by Spigno *et al.* (2008) and Ping *et al.* (2011), the lignin content in grape stalks ranged from 33 to 47%, results that are close to the values obtained in this study. In addition, the lignin content of the stalks is comparable to that of other biomasses, such as coconut fiber (33.44%), sawdust (24.15%), sugarcane bagasse (22.89%) and bamboo (22.12%), which reinforces its potential as an alternative raw material (NAM *et al.*, 2020). In this study, the main focus was on grape stalks, leaving for future studies the exploration of combinations with other biomasses or the use of pre-treatments that could increase their viability.

3.2 Bulk, apparent, and energetic density

The Figure 1A shows that the bulk density of the 2mm particles (T1) was the highest (0.4500 g cm⁻³) compared to the others. The lowest values were observed in the particles from treatment T4, due to their larger size. No statistically significant differences were found between the particles from treatments T2 and T3. The apparent density and energy density also followed this trend, being higher in treatments T1 and T2. These findings are in line with the studies by Abazarpour *et al.* (2020) and Labbé *et al.* (2020), which highlight the importance of particle size in the durability and performance of solid biofuels.

The increase in the apparent density of the briquettes leads to a reduction in the volume of the original biomass, reflecting the effectiveness of the briquetting process and providing useful information for logistics and transportation (RIBEIRO *et al.*, 2022).

Figure 1 – Bulk density of grape stalk particles and apparent density of briquettes (A) and energy density of briquettes (B) in different treatments



Source: Authors (2023)

In where: T1 - 2 mm particles (a); T2 - 3 mm particles (b); T3 - 5 mm particles (c); and T4 - 8 mm particles (d). Horizontal bars represent the standard error (n = 7). Means followed by the same letter did not differ significantly by Tukey's test ($P < 0.05$).

In the national context, biomass from sugarcane cultivation is widely recognized as a prominent agricultural residue for the production of briquettes for heat generation. Mixtures made up of sugarcane bagasse, sugarcane straw and cassava stalks had an apparent density of between 0.87 and 0.95 g cm⁻³ (SILVA *et al.*, 2021), lower than the briquettes produced in this study, which highlights the quality of this waste for energy purposes. In other studies, Brasil *et al.* (2015) found higher values of 0.9 g cm⁻³, using particle sizes of around 0.4 mm, while Brunerová, Hynek Roubík and Brožek (2018) reported apparent densities for briquettes produced with sugarcane husk and bamboo fiber of 1.067 g cm⁻³ and 0.98 g cm⁻³, respectively.

In assessing the quality of briquettes, bulk density contributes to obtaining a high-quality biofuel. In terms of bulk density, the briquettes produced with grape stalks show adequate quality and are viable for generating heat. In the case of grape stalks, the values obtained show that the briquettes are suitable for energy purposes. In addition, a positive correlation was observed between bulk density and energy

density: denser briquettes tend to have higher energy per volume, reflecting better burning performance.

High energy density values are desirable in solid fuels (PETRICOSKI, 2017). The grape stalk briquettes produced in this study had an energy density of 4,094 Mcal m⁻³ (T1) and 3,977 Mcal m⁻³ (T4), similar to those produced from mixtures of bagasse and sugarcane straw and cassava branches, which vary between 3,769 and 5,545 Mcal m⁻³ (SILVA *et al.*, 2021). Similar values were also recorded for *Dipteryx odorata* forest biomass (4,610 Mcal m⁻³), rice husks (3,550 Mcal m⁻³) and sugarcane bagasse (4,330 Mcal m⁻³) (SOUZA and VALE, 2016).

The grape stalk briquettes produced with all grain sizes remained intact, without imperfections, during all the stages prior to the application of the destructive methodology. This suggests that, aesthetically, the material is suitable for use, as there was no disintegration of the particles after compaction.

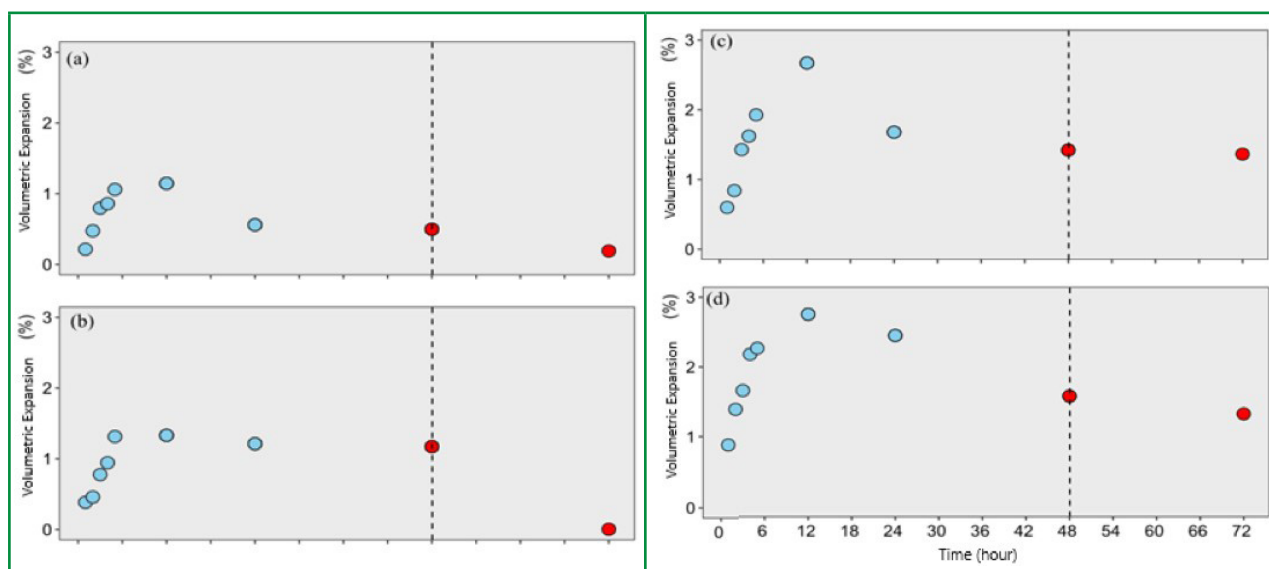
3.3 Volumetric expansion

The maximum expansion occurred in the first 12 hours after the briquetting process for all treatments. After this period, small variations were observed, and after 48 hours the briquettes stabilized, with no significant difference.

The volumetric expansion of compacted products is related to the initial moisture content and the chemical composition of the lignocellulosic material. In general, when the moisture content is very low, the volumetric expansion tends to be lower, as the compacted material has less capacity to absorb moisture from the environment, resulting in lower expansion (MORENO; FONT; CONESA, 2016; SETTER *et al.*, 2021). As can be seen in Figure 2, the relationship between the different grain sizes proved to be directly proportional, since larger particles resulted in greater expansion. The treatments with smaller particles (T1 and T2) had an average volumetric expansion of 0.49% and 0.63%, respectively, a result that reinforces the hypothesis that finer particles favor better pressing and lower absorption of environmental moisture.

Briquettes from T4, with larger compacted biomass, had an average expansion of 1.63%. The values in this study are lower than those found by Brasil *et al.* (2015), in which sugarcane briquettes had an average expansion of 22%, showing the dimensional stability of the material studied, so the volumetric expansion of grape stalk briquettes indicates satisfactory dimensions compared to other biomasses used for the same purpose.

Figure 2 – Spatial variation (hours) of the mean volumetric expansion (%) of grape stalk briquettes



Source: Authors (2023)

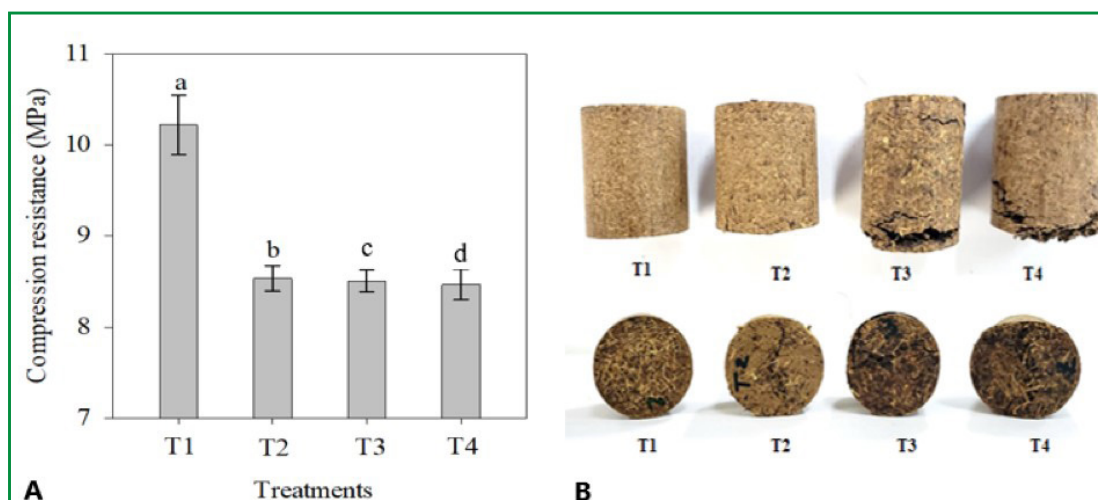
In where: T1 - 2 mm particles (a); T2 - 3 mm particles (b); T3 - 5 mm particles; and T4 - 8 mm particles (d). Red dots - Stabilization period. Means followed by the same color did not differ significantly by Tukey's test ($P < 0.05$).

3.4 Compression resistance

The briquettes from treatment T1 had the highest compressive strength, reaching 10.21 MPa, indicating better adhesion of the smaller particles, which corroborates the study's hypothesis. It should be noted that these briquettes also had the highest apparent density of 1.3246 g cm^{-3} , which contributed to this result. The size of the particles has an impact on the strength of the briquettes due to the

more effective mechanical interlacing, and the reduction in grain size results in an increase in the available surface area, providing greater cohesion and strength of the final products (SETTER *et al.*, 2021).

Figure 3 – Average compression strength of grape stalk briquettes (A) and post-mechanical resistance test of grape stalk briquettes (B)



Source: Authors (2023)

In where: T1 - 2 mm particles (a); T2 - 3 mm particles (b); T3 - 5 mm particles; and T4 - 8 mm particles (d). Means followed by the same letter did not differ significantly by Tukey's test ($P < 0.05$).

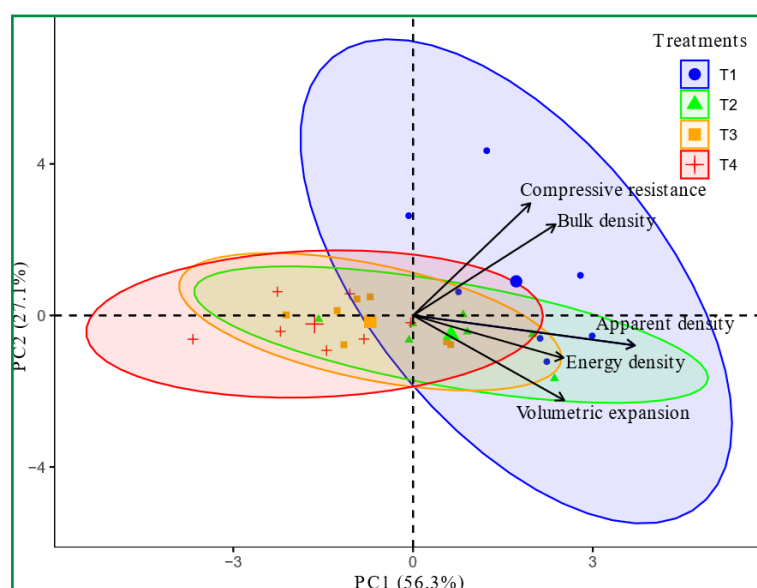
Briquettes with smaller particles tend to have greater mechanical strength compared to those produced with larger particles, as observed in this study. As a result, the compressive strength test (Figure 3B) showed that larger particles led to the appearance of cracks, which compromised the quality of the material.

The high lignin content present in the grape stalks (28.91%) also contributed to better compaction of the particles in the briquette. The cohesion of compacted biomass is influenced by the application of binders. Zobel and Van Buijtenen (1989) emphasize that lignin is a macromolecule responsible for the strength of the cell wall, so materials rich in lignin do not need external binders, because during exposure to high temperatures, the lignin undergoes plasticization, acting as a binding agent and forming a moisture-resistant protective layer in the briquettes, resulting in a reduction in manufacturing costs (BONFATTI JÚNIOR *et al.*, 2019).

3.5 Principal Component Analysis (PCA)

The Principal Components Analysis (PCA) only considered the first two components, since the sum of CP1 and CP2 explained 83.4% of the variability of the original data, as can be seen in Figure 4. CP1 explained 56.3% of the variability, allowing the effect of briquette grain size on the response variables to be observed. Briquettes produced with finer particles (T1 and T2) stood out for their higher correlations with bulk density, bulk density, energy density, compressive strength and lower volumetric expansion. This grouping corroborates the results presented earlier, indicating that fine grain size favors various properties of interest in briquetting.

Figure 4 –Relationship between PC1 and PC2 for bulk density, apparent density, energy density, compressive strength, and volumetric expansion of grape stalk briquettes (T1 - 2 mm particles; T2 - 3 mm particles; T3 - 5 mm particles; and T4 - 8 mm particles)



Source: Authors (2023)

The second principal component (PC2) accounted for 27.1% of the variability in the data and stood out for differentiating the behavior of the response variables. In this context, T1 briquettes were predominantly influenced by the variables bulk density, bulk density, energy density, compressive strength and volumetric expansion.

In addition, an inverse relationship was observed between strength and volumetric expansion, suggesting that stronger briquettes tend to have greater dimensional stability.

4 CONCLUSIONS

This study has shown that grape stalks, when processed with a grain size ≤ 2 mm, have a superior performance in the production of briquettes when compared to conventional biomass, achieving high bulk density, mechanical strength and good energy power. This alternative contributes to the diversification of lignocellulosic sources for bioenergy, reducing dependence on fossil fuels. It also promotes the valorization of agro-industrial waste, generates income for small producers and strengthens the circular economy in viticulture, in line with global carbon neutral targets and SDG 12. It is recommended that the methodology be replicated in other production chains, with adaptations to the scale and local socio-economic context.

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