



CAPÍTULO 13

Physicochemical characterization of cocoa Pod Husk (*Theobroma cacao* L.)

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ABSTRACT

This work aimed to carry out the physicochemical characterization of cocoa pod husk (*Theobroma cacao* L.) and propose possible technological applications for this biomass. The methodology involved physicochemical analyses, including moisture content, bulk and real density, porosity, ash content, volatile matter, fixed carbon, pH, and point of zero charge. The results showed values close to those found in the literature, indicating the reliability of the study. The potential of cocoa pod husk for use in adsorption and biosorption processes, especially in effluent treatment, is highlighted. Moreover, the utilization of this agro-industrial waste contributes to the reduction of environmental impacts and to sustainable development. In this way, cocoa pod husks present viability in environmental applications, encouraging the use of regional resources and the generation of innovative solutions in the Amazon.

Keywords: Agro-industrial waste. Cocoa pod husk. Biosorption. Adsorption. Sustainability. Wastewater treatment.

Introduction

According to Silva, Tagliaferro and Oliveira (2021), solid and semi-solid waste includes all those originating from residences, industries, hospitals, commerce, urban cleaning services, and agriculture. The improper management of these wastes causes various problems to human health, as inappropriate disposal can lead to the contamination of water bodies. In Brazil, Law No. 12,305 was enacted in 2010, which established the National Solid Waste Policy, addressing the guidelines for solid waste management and the management responsibilities of each generator.

After 20 years of deliberation in the National Congress, the aforementioned Law allowed the country to make the necessary progress in addressing the environmental, economic, and social problems resulting from inadequate waste management (Rauber, 2011). However, the generation of waste has become so high that the country is now in fourth place in the ranking, behind only the United States of America, China, and India (Martins et al., 2023).

When improperly managed, these wastes can cause several environmental problems, including soil and water contamination. Various methods can be used for water treatment, including coagulation, flocculation, adsorption, and biosorption (Abukhadra & Allah, 2019).

Brazil has fertile soils and considerable climatic diversity across its regions, resulting in a wide variety of biomass resources (Martins & Carrino, 2023). From this perspective, one of the biomasses produced by the country is cocoa pod husk (*Theobroma cacao* L.). According to Veríssimo (2012), cocoa belongs to the Sterculiaceae family and there are three main types of cocoa tree varieties, the most common being Forastero, which is responsible for 90% of the world's cocoa bean production.

In this context, this study aimed to determine the physicochemical characteristics of cocoa pod husk (*Theobroma cacao* L.) and propose potential technological applications.

Materials and Methods

Raw material

The cocoa pod husk was obtained from a private property located in Tracuateuazinho – PA (Figure 1). The location is situated in the mesoregion of Northeast Pará and the Bragantina microregion, under the geographical coordinates 1°13'37" S and 47°00'27" W (IBGE, 2019). The selected husks were collected from the same cacao tree and met a standardized size criterion. After collection, they were stored under refrigeration until the time of laboratory analysis.

Location of the analyses

The physicochemical analyses of cocoa pod husks were conducted at the Laboratory of Engineering and Environment of the Federal Rural University of the Amazon - Capanema Campus.

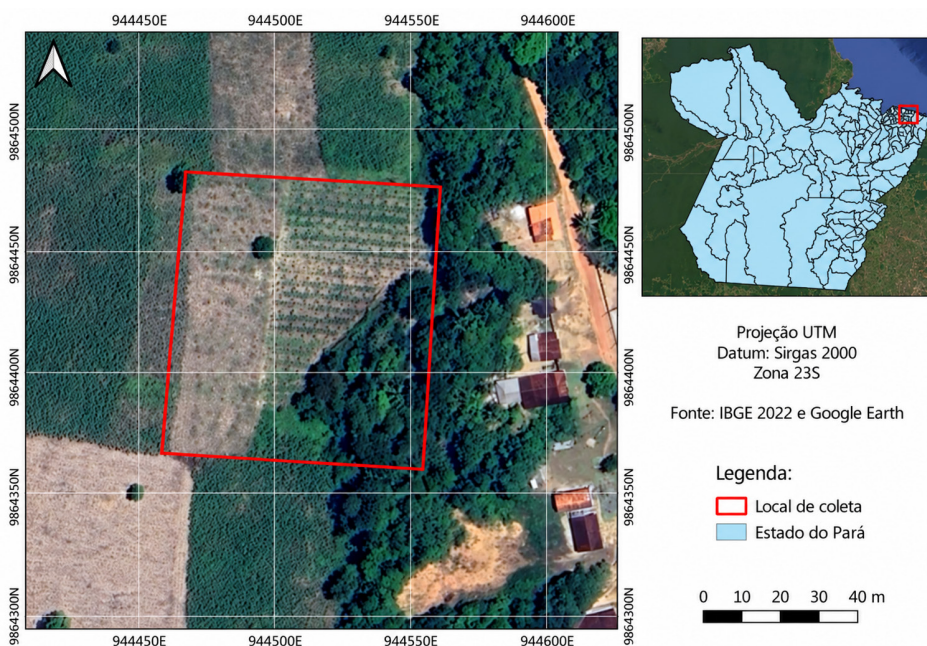


Figure 1 Map of the Location of the Cocoa Pod Husk Collection Property. *Source:* Authors (2026).

Physicochemical characterization of cocoa pod husk

The physicochemical characterization of the biomass was determined through analyses of moisture content, bulk and real densities, porosity, ash content, volatile matter, fixed carbon, pH and point of zero charge.

Moisture content

The moisture content analysis was carried out according to Silva et al. (2015) and calculated using Equation 1.

$$MC\% = \frac{W_w - W_d}{W_w} \times 100 \quad (1)$$

Where:

MC: Moisture content (%);

Ww: Wet weight (g);

Wd: Dry weight (after 24 h) (g).

Bulk Density

This analysis was conducted in accordance with the ASTM D2854-09 standard (Grauito et al., 2007; Namane et al., 2005) and Equation 2 was used for its quantification.

$$BD = m/v \quad (2)$$

Where:

BD: Bulk density (g/cm³);

m: Mass of biomass (g);

v: Occupied bulk volume (cm³).

Real Density

The tests for determining real density were conducted according to the NBR165 Standard and calculated following ASTM D2854-09 (Ramos, 2005), using Equation 3.

$$RD = ma/[ma - (mw - mw')] \quad (3)$$

Where:

RD: Real density (g/cm³);

ma: Mass of biomass (g);

mw: Mass of the volumetric flask + water + biomass (g);

mw': Mass of the volumetric flask + water (g).

Porosity

With the values obtained from the real and bulk density equations, the porosity percentage of the material was determined, following the ABNT NBR 9165 Standard (ABNT, 1985) and Equation 4.

$$\phi = \left[1 - \left(\frac{BD}{RD} \right) \right] \times 100 \quad (4)$$

Where:

Φ: Porosity (%);

BD: Bulk density (g/cm³);

RD: Real density (g/cm³).

Ash content

Ash content was determined according to ASTM E1755-01, "Standard Method for the Determination of Ash in Biomass," and calculated using Equation 5.

$$\text{Ash (\%)} = \left(\frac{m_3 - m_1}{m_2 - m_1} \right) \times 100 \quad (5)$$

Where:

Ash (%): Ash content (%);

m_1 : Mass of empty crucible (g);

m_2 : Mass of crucible + initial sample (g);

m_3 : Mass of crucible + ash (g).

Volatile Matter

For the determination of the volatile matter, the methodology of the ASTM D3175-07 Standard (ASTM INTERNATIONAL, 2007) was used, and the quantification was carried out using Equation 6.

$$\text{VM (\%)} = \left(\frac{m_2 - m_3}{m_1} \right) \times 100 \quad (6)$$

Where:

VM (%): Volatile matter (%);

m_1 : Mass of the initial sample (g);

m_2 : Mass of crucible + initial sample (g);

m_3 : Mass of crucible + final sample (g).

Fixed Carbon

The fixed carbon was determined according to the ASTM D3172-89 Standard (ASTM INTERNATIONAL, 1989), using Equation 7.

$$\text{FC (\%)} = 100 - (\text{AC} + \text{VM}) \quad (7)$$

Where:

FC: Fixed Carbon (%);

AC: Ash content;

VM: Volatile matter.

pH

The pH analysis in suspension was carried out using 50 mL beakers, where 3 g of biomass and 30 mL of distilled water were first added in a 1:10 (solid:solution) ratio (Singh et al., 2010). The pH was determined using the Tekna T-1000 pH meter.

Point of Zero charge

The analysis was conducted according to the method proposed by Regalbuto and Robles (2004), using seven pH values (4, 5, 6, 7, 8, 9 and 10). In each flask, 50 mL of distilled water and 0.05 g of biomass were added, and the pH levels were controlled using hydrochloric acid and sodium hydroxide, both with a molar concentration of 0.1 M. The resulting suspensions were subjected to agitation in a Luca-22 Shaker incubator for 24 hours at a temperature of 30 °C. The construction of the graph and the determination of the point of zero charge were obtained using the Origin software.

Results

Physicochemical characterization of cocoa pod husk

Moisture content

Pereira (2013) reported a moisture content of 84.2% for cocoa pod husk. In the present study, the moisture content was 85.1%, which is consistent with the value reported in the literature.

Bulk Density

The value of the bulk density of cocoa pod husk obtained in this study was 0.40 g/cm³. This result is consistent with values reported in the literature, where Bot et al. (2024) found a husk density of 0.40 g/cm³ in briquettes produced from cocoa pod husk, a value similar to that observed in this study. Similarly, Adebayo et al. (2022) reported a bulk density of 0.38 g/cm³ for materials derived from cocoa pod husk.

Real Density

The real density of cocoa pod husk was 0.69 g/cm³. Visvanathan et al. (2021), in a study of the physical properties of cocoa fruit, reported a real density of 0.66 g/cm³, which is close to the value obtained in the present study.

Porosity

The porosity of the cocoa pod husk was 40.61%, a value similar to that observed in studies that used cocoa pod husk residues as soil conditioners. According to Santos et al. (2013), lignocellulosic plant biomasses exhibit a high porous structure, with porosity values ranging approximately between 35% and 60%, depending on the moisture content, compaction and treatment applied to the material. Thus, it is noted that the biomass used in this study falls within this percentage range.

Ash Content

The ash content found in this study was 9.91%. This value is consistent with data found in the study conducted by Ozung et al. (2016), in which this analysis presented a value of 9.30%. Furthermore, in a study conducted by Alemaewor et al. (2009), the cocoa pod husk showed a value of 9.14% for ash content.

volatile Matter

The volatile matter content found for cocoa pod husk was 67.37%, a value close to that reported in the literature by Ghysels et al. (2022), who found a volatile matter of 67.13% in biomass characterization analyses of cocoa pod husk.

Fixed Carbon

The fixed carbon content obtained in this study was 23.57%. In research conducted by Pereira (2013), the same analysis showed a value of 23.34%. Thus, the immediate chemical composition of this biomass for this analysis showed a value similar to that found in the literature.

pH

The pH value found in this study was 7.75, a result that is relatively distant when compared to the values reported in the literature. Rozendo (2018) found a pH of 5.46 for cocoa pod husk, a value identical to that observed by Herrera-Rengifo et al. (2020), in the characterization of cocoa pod husk used for starch extraction. These differences may be related to the differences in the origin of the raw material used. Factors such as climatic conditions, moisture content, nutrient availability in the soil, and environmental characteristics of the cultivation site can directly influence the chemical properties of cocoa pod husks, including pH, which contributes to variations between the results found in different studies.

Point of Zero Charge

The value found for the point of zero charge was 6.9 (Figure 2). This value is comparable to the research conducted with adsorbent obtained from modified cocoa pod husk, which had a value of 6.3 for the same analysis, a value relatively close to that found in this study (Reyes & Campoverde, 2024).

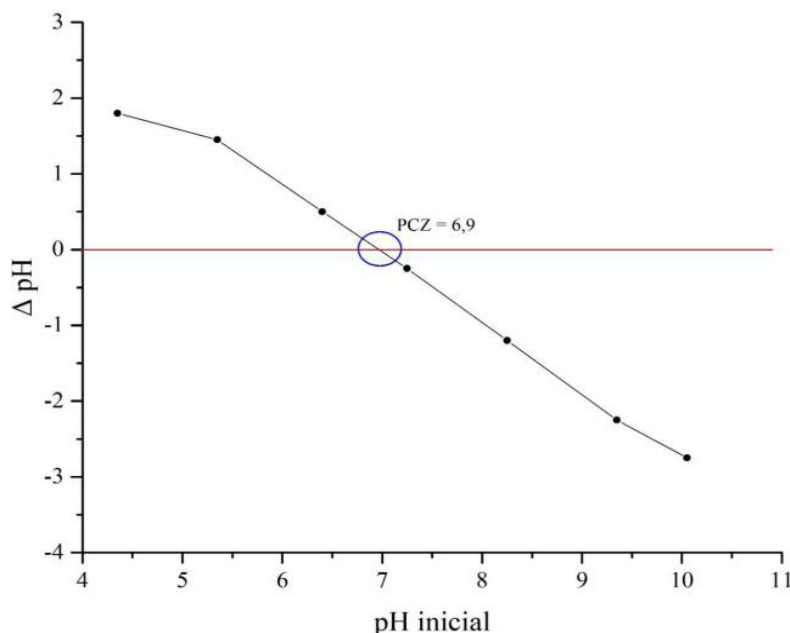


Figure 2 Graph of Point of Zero Charge. Source: Authors (2026).

conclusion

Based on the results obtained in this study, it was possible to more clearly understand the potential of cocoa pod husk (*Theobroma cacao* L.) as a biomass with relevant and promising physicochemical characteristics for technological applications. The analyzes conducted showed that the values for moisture content, bulk density, real density, porosity, ash content, volatile matter and fixed carbon remain close to those found in the literature, which reinforces the reliability of the obtained data and the consistency of the analyzed material.

Moreover, the pH value and the point of zero charge indicate that cocoa pod husk exhibits properties that can favor its use in processes such as adsorption and biosorption,

especially in wastewater treatment and the removal of contaminants from water bodies. In this sense, the study meets its main objective and points to viable pathways for the technological application of the analyzed biomass.

Another relevant aspect is the valorization of an agro-industrial waste abundant in the Amazon region. By proposing the utilization of cocoa pod husks, the work contributes not only to the reduction of environmental impacts associated with improper waste disposal, but also to the development of sustainable solutions aligned with the local reality. This approach reinforces the importance of integrating scientific knowledge and sustainable practices, especially in a region like the Amazon, where biodiversity and the availability of biomass represent great opportunities for innovation.

In this way, the results obtained expand the knowledge about the use of lignocellulosic waste and demonstrate that cocoa pod husk can be considered a viable and low-cost alternative for environmental applications. However, it is important to highlight that further studies are still necessary, especially regarding the efficiency of the material in real treatment processes, its regeneration capacity, and possible chemical modifications that could enhance its performance.

Thus, this study reinforces the importance of investing in innovative solutions based on regional resources, contributing to the sustainable development of the Amazon. By transforming waste into opportunities, a path is opened for new environmental technologies and the construction of a more balanced future, in which economic growth and environmental preservation go hand in hand.

References

ABUKHADRA, M. R.; ALLAH, A. F. S. Synthesis and characterization of kaolinite nanotubes (KNTs) as a novel carrier for 5-fluorouracil of high encapsulation properties and controlled release. **Inorganic Chemistry Communications**, v. 103, p. 30–36, 2019. Disponível em: <https://doi.org/10.1016/j.inoche.2019.03.018>. Acesso em: 16 mar. 2026.

ADEBAYO, A. S. et al. Characterizations of alpha-cellulose and microcrystalline cellulose isolated from cocoa pod husk as a potential pharmaceutical excipient. **Journal of Applied Pharmaceutical Science**, v. 12, n. 9, 2022. Disponível em: <https://pmc.ncbi.nlm.nih.gov/articles/PMC9457090/>. Acesso em: 16 mar. 2026.

ALEMAWOR, F. et al. Effect of *Pleurotus ostreatus* fermentation on cocoa pod husk composition: influence of fermentation period and Mn²⁺ supplementation on the fermentation process. **African Journal of Biotechnology**, v. 8, n. 9, p. 1950–1958, 2009. Disponível em: https://hero.epa.gov/hero/index.cfm/reference/details/reference_id/780075. Acesso em: 15 mar. 2026.

ASSOCIAÇÃO BRASILEIRA DE NORMAS TÉCNICAS. NBR 9165: Carvão vegetal - Determinação da densidade relativa aparente, relativa verdadeira e porosidade. Rio de Janeiro: **ABNT**, 1985.

ASTM INTERNATIONAL. **ASTM D3172-89**: Standard practice for proximate analysis of coal and coke. West Conshohocken, PA: ASTM International, 1989.

ASTM INTERNATIONAL. **ASTM D3175-07**: Standard test method for volatile matter in the analysis sample of coal and coke. West Conshohocken, PA: ASTM International, 2007.

BOT, B. V. et al. Physicochemical characterization of briquette fuel produced from cocoa pod husk in Cameroon. **Results in Engineering**, 2024. Disponível em: <https://www.sciencedirect.com/science/article/pii/S235248472400026X>. Acesso em: 16 mar. 2026.

GRATUITO, M. K. B. et al. Production of activated carbon from coconut shell: optimization using response surface methodology. **Bioresource Technology**, v. 99, n. 11, p. 4887–4895, 2007.

GHYSELS, S. et al. Study of cocoa pod husks thermal decomposition. **Applied Sciences**, v. 12, n. 18, p. 9318, 2022. Disponível em: <https://www.mdpi.com/2076-3417/12/18/9318> . Acesso em: 16 mar. 2026.

HERRERA-RENGIFO, J. D. et al. Extracción de almidón de cáscara de cacao (*Theobroma cacao* L.) como alternativa de bioprospección. **Revista ION**, v. 33, n. 2, p. 25–34, 2020. Disponível em: <https://doi.org/10.18273/revion.v33n2-2020002> . Acesso em: 16 mar. 2026.

INSTITUTO BRASILEIRO DE GEOGRAFIA E ESTATÍSTICA (IBGE). Divisão territorial brasileira (DTB). Rio de Janeiro: **IBGE**, 2019. Disponível em: <https://www.ibge.gov.br/geociencias/orga-nizacao-do-territorio/divisao-regional/23701-divisao-territorial-brasileira.html> . Acesso em: 25 set. 2025.

MARTINS, A. C. S. et al. Degradação ambiental nas favelas: projeto referente aos impactos dos resíduos sólidos no bairro Vila dos Pescadores, município de Cubatão-SP. Cubatão: ETEC de Cubatão – Centro Paula Souza, 2023. Disponível em: https://ric.cps.sp.gov.br/bits-tream/123456789/19556/1/etimma_2023_martins_degradacao-ambiental.pdf . Acesso em: 16 mar. 2026.

MARTINS, L. R.; CARRINO, A. L. Um estudo sobre a biomassa: uma fonte renovável de energia limpa. **Revista Interface Tecnológica**, v. 20, n. 2, p. 531–544, 2023. DOI: <https://doi.org/10.31510/inf.v20i2.1728> .

OZUNG, P. O. et al. Proximate composition and fibre fractions of raw and processed cocoa pod husk meal. **Agricultural and Environmental Sciences Research**, 2016. Disponível em: <https://www.asianonlinejournals.com/index.php/AESR/article/download/149/1082> . Acesso em: 15 mar. 2026.

PEREIRA, I. O. **Viabilidade da utilização da casca de cacau como combustível no aquecimento de ar para secagem de amêndoas de cacau**. 2013. Tese (Doutorado) – Universidade Federal de Viçosa, Minas Gerais, 2013.

RAMOS, M. A. B. **Remoção de íon Cr (III) de solução aquosa por adsorção e biossorção**. 2005. Dissertação (Mestrado) – Universidade Federal do Pará, Belém, 2005.

RAUBER, M. E. Apointamentos sobre a Política Nacional de Resíduos Sólidos, instituída pela Lei Federal nº 12.305, de 02.08.2010. **Revista Eletrônica em Gestão, Educação e Tecnologia Ambiental (REGET)**, v. 4, n. 4, p. 1–24, 2011. Disponível em: <https://periodicos.ufsm.br/reget> . Acesso em: 16 mar. 2026.

REGALBUTO, J. R.; ROBLES, J. **The engineering of Pt/Carbon catalyst preparation**. University of Illinois: Chicago, 2004.

ROZENDO, A. S. **Produção de pectinases a partir de cascas de cacau (*Theobroma cacao* L.) em fermentação submersa com aplicação na clarificação de suco de maçã**. 2018. Dissertação (Mestrado em Engenharia de Bioprocessos e Biotecnologia) – Universidade Federal do Paraná, Curitiba, 2018. Disponível em: <https://acervodigital.ufpr.br/handle/1884/70139> . Acesso em: 14 mar. 2026.

SANTOS, F. A. et al. Potencial da palha de cana-de-açúcar para produção de etanol. **Química Nova**, São Paulo, v. 35, n. 5, p. 1004-1010, 2013.

SILVA, I. O.; TAGLIAFERRO, E. R.; OLIVEIRA, A. J. Gerenciamento dos resíduos sólidos domiciliares no município de Jales – SP e sua relação com a Política Nacional de Resíduos Sólidos (PNRS). **Brazilian Journal of Development**, v. 7, n. 1, p. 11475–11499, 2021. DOI: <https://doi.org/10.34117/bjdv7n1-782>.

SILVA, D. A.; YAMAJI, F. M.; BARROS, J. L.; RÓZ, A. L.; NAKASHIMA, G. T. **Caracterização de biomassas para a briquetagem**. Floresta, Curitiba, v. 45, n. 4, p. 713-722, out./dez. 2015.

SINGH, V. et al. Sondagem de expansão térmica de grafeno e dispersão modal a baixa temperatura usando ressonadores de sistemas nanoeletromecânicos de grafeno. **Nanotecnologia**, v. 21, n. 16, 2010.

REYES, A. M. Z.; CAMPOVERDE, G. A. L. Evaluación del potencial de la cáscara de cacao modificada com tempo para la adsorción de cadmio en soluciones acuosas. **Machala**, 2024. Disponível em: https://repositorio.utmachala.edu.ec/bitstream/48000/23524/1/Trabajo_Titulacion_3648.pdf . Acesso em: 16 mar. 2026.

VERÍSSIMO, A. J. M. **Efeito da origem do cacau na sua qualidade comercial, funcional e sensorial**. 2012. Dissertação (Mestrado em Engenharia Alimentar) – Universidade de Lisboa, Lisboa, 2012. Disponível em: <http://hdl.handle.net/10400.5/5378> . Acesso em: 16 mar. 2026.

VISVANATHAN, R. et al. Effect of pod size on physical properties of cocoa pods (*Theobroma cacao* L.) with reference to farm level mechanization of cocoa processing. **Agricultural Engineering Today**, v. 45, n. 2, p. 13–19, 2021. Disponível em: <https://pub.isae.in/index.php/aet/article/view/2943> . Acesso em: 16 mar. 2026.

