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# CHARACTERIZATION OF SAMPLES OF GREEN COCONUT HUSK (*COCOS NUCIFERA* L.) *IN NATURA* COLLECTED IN MARANHÃO, PIAUÍ, AND CEARÁ

# CARACTERIZAÇÃO DE AMOSTRAS DE CASCA DE COCO VERDE (*COCOS NUCIFERA L.) IN NATURA* COLETADAS NO MARANHÃO, PIAUÍ E CEARÁ

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## ABSTRACT

**Background**: To define the applicability of green coconut husk, it is important to understand its chemical and physical characteristics. **Objective**: Therefore, this study aims to evaluate the chemical composition and particle size of crushed green coconut husk from three states: Maranhão, Piauí, and Ceará. **Methods**: After crushing, we assessed the levels of cellulose, hemicellulose, lignin, and ash, and investigated the particle sizes of the coconut husk (9-325 mesh). Principal component analysis and Pearson correlation were also conducted. **Results**: Regarding particle size, there is greater retention at 48 mesh, and in terms of chemical composition, the levels of cellulose, hemicellulose, lignin, and ash showed values close to 30%. Principal component 1 is related to particle size, while principal component 2 is related to composition. We also observed a correlation between lignin and cellulose levels and particle size. **Conclusion**: The analysis of green coconut husk reveals a relationship between particle size and chemical composition.

**Keywords**: *Green coconut; PCA; chemical composition; Particle size distribution.*

## RESUMO

**Introdução**: Para definir a aplicabilidade da casca de coco verde, é importante compreender suas características químicas e físicas. **Objetivo**: Portanto, este estudo visa avaliar a composição química e o tamanho de partículas da casca de coco verde triturada de três estados: Maranhão, Piauí e Ceará. **Métodos**: Após a trituração,



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avaliamos os níveis de celulose, hemicelulose, lignina e cinzas, e investigamos os tamanhos de partículas da casca de coco (9-325 mesh). Análise de componentes principais e correlação de Pearson também foram conduzidas. **Resultados**: Em relação ao tamanho de partículas, há maior retenção em 48 mesh, e em termos de composição química, os níveis de celulose, hemicelulose, lignina e cinzas mostraram valores próximos a 30%. O componente principal 1 está relacionado ao tamanho de partículas, enquanto o componente principal 2 está relacionado à composição. Observamos também uma correlação entre os níveis de lignina e celulose com o tamanho de partículas. **Conclusão**: A análise da casca de coco verde revela uma relação entre tamanho de partículas e composição química.

**Palavras-chave**: *Coco verde; PCA; composição química; distribuição do tamanho de partículas.*

## 1. INTRODUCTION:

The green coconut (Cocos nucifera L.) is one of the most widely consumed fruits in the world, particularly in tropical and subtropical regions such as Brazil (Perera *et al*., 2009). Its composition includes vitamins, minerals, and antioxidants, and its use has been explored in a variety of food products (Mat *et al*., 2022) and cosmetics (Lestari *et al*., 2021). Brazil stands out as one of the main producers of green coconuts, especially in coastal regions, with an estimated annual production of 1.8 billion fruits and an annual revenue of 1.6 billion reais (IBGE, 2023).

Despite the large Brazilian production of coconut, the husk is usually neglected and treated as agricultural waste. The husk accounts for approximately 85% of the fruit (Ayrilmis *et al*., 2011) and is primarily composed of lignin, cellulose, hemicellulose, and ash (Arena *et al*., 2016). Some examples of applications for the husk reported in the literature include the production of concrete composites (Ali *et al*., 2012), reinforcement of polymer matrices (Das & Biswas, 2016), organic filters (Lo Monaco *et al*., 2009), or cement composites (Asasutjarit *et al*., 2007). After the removal of the pulp and coconut water, the husk is typically disposed of in landfills or public spaces, where it can take up to approximately 10 years to decompose in the environment, potentially causing pollution and the spread of diseases (Banerjee *et al*., 2013). Furthermore, the disposal of the coconut husk represents a technological waste, given its wide range of applications.

It is known that the composition and properties of coconut husk can vary depending on various factors, including the origin of the coconut (Afrande & Achaw, 2008). In this context, this research aims to contribute to the characterization of green coconut husk produced in three different Brazilian states, Maranhão, Piauí, and Ceará, through granulometric and chemical composition analyses.

## 2. MATERIALS AND METHODS:

### 2.1. Material Collection

Green coconut husks were collected fresh from local markets in different Brazilian states: São José de Ribamar in Maranhão, Jerumenha in Piauí, and Fortaleza in Ceará. After collection, the coconut samples were identified and then washed with running water to remove impurities. The samples were stored in a refrigerated environment until further use.

### 2.2. Sample preparation

Initially, the water and pulp were removed from the coconuts, and then the husks were shredded. The shredding was performed using a water coconut shredder (PI 0005184-5). After shredding, the samples were air-dried for 96 hours to reduce the moisture content to below 20%. Subsequently, the material was ground using a Willey knife mill (Solab, São Paulo, Brazil) at a rotation speed of 1750 RPM. The samples were dried at 80°C and weighed every 4 hours until a constant weight was achieved.

### 2.3. Granulometric Classification

After grinding, the samples were subjected to granulometric classification using a set of sieves arranged in the order of 9 to 325 mesh, with constant agitation for a period of 60 minutes. The retention (R) was determined using Equation 1.

$$\mathrm{R}(\%) = \left(\frac{\text{Sample mass(g)} - \text{Retained mass(g)}}{\text{Sample mass(g)}}\right) \times 100\% \quad \text{(Eq.1)}$$

### 2.4. Chemical characterization

The soluble and insoluble lignin content was determined using the standard Klason method (Tappi Standard Methods, 2011). A 1 g sample of green coconut husk was weighed and transferred to an Erlenmeyer flask, where 20 mL of 72% $H_2SO_4$ was added with constant stirring for 2 hours at room temperature (26 ± 5°C). The samples were then diluted with 560 mL of distilled water and subjected to reflux for 4 hours. The

residue was filtered and washed with water until a neutral pH was obtained and then dried in an oven at 103 ± 2°C until a constant mass was achieved. The insoluble lignin content (Li) was determined by the gravimetric method and calculated using Equation 2.:

$$\text{Li}\ (\%) = \left(\frac{\text{Mass of dry sample (g)}}{\text{Mass of dry residue (g)}}\right) \times 100\% \quad \text{(Eq. 2)}$$

The filtrate was diluted to 1000 mL for the determination of soluble lignin by spectroscopy according to the method of Sarkenen and Ludwig (1971). Absorbance was measured at wavelengths of 215 nm and 280 nm using a Beckman DU-7 spectrophotometer (Beckman, Krefeld, Germany). The lignin concentration was calculated using Equation 3 (Perez, 1996).

$$\text{Soluble lignin}\ \left(\frac{\text{g}}{\text{L}}\right) = \frac{4{,}53 \times \text{A}_{215} - \text{A}_{280}}{300} \quad \text{(Eq. 3)}$$

The cellulose content was determined through the analysis of holocellulose, a combination of cellulose with hemicellulose, according to Razera (2006). A mass of 3 g of the sample was mixed with 250 mL of distilled water, 1 mL of acetic acid, and 2.5 g of sodium chlorite. The mixture was heated to 70°C for 1 hour. Then, 1 mL of acetic acid and 2.5 g of sodium chlorite were added to the mixture. The mixture was heated for an additional 3 hours, then cooled to 10°C and filtered. The filtrate was washed with distilled water until a neutral pH was achieved, then washed with methanol and dried in an oven at 105°C until a constant mass was attained. It was then cooled in a desiccator. The holocellulose content (h) was calculated using Equation 4.

$$\text{h}\ (\%) = \left(\frac{\text{Mass of dry sample (g)}}{\text{Mass of dry residue (g)}}\right) \times 100\% \quad \text{(Eq. 4)}$$

For the determination of cellulose (Browning, 1967), 0.3 g of the sample was added to a beaker containing 3 mL of 17.5% NaOH and mixed for 3.5 minutes. The sample was then incubated in a water bath at 20°C for 20 minutes. After incubation, 3 mL of distilled water was added and stirred for 1 minute. The sample was allowed to stand for 5 minutes at room temperature (26 ± 5°C) and then filtered using a Büchner funnel for 1 minute under vacuum. The mixture was washed with distilled water and 10% acetic acid solution until a neutral pH was obtained. The sample was dried in an oven at 100°C until a constant weight was achieved, and then the mass was used to calculate the cellulose content (C) using Equation 5.

$$\text{C}\ (\%) = \left(\frac{\text{Mass of dry sample (g)}}{\text{Holocellulose mass (g)}}\right) \times 100\% \quad \text{(Eq. 5)}$$

The hemicellulose content was calculated as the difference between the holocellulose and cellulose contents.

The ash content was determined by the gravimetric method. 1 g of sample was weighed and then subjected to a temperature of 600°C for 4 hours. The sample was cooled in a desiccator at room temperature and weighed. The process was repeated until a constant mass was obtained. The ash content (C) was obtained using Equation 6.

$$\text{C}\ (\%) = \left(\frac{\text{Mass of ashed sample (g)}}{\text{Initial sample mass (g)}}\right) \times 100\% \quad \text{(Eq. 6)}$$

### 2.5. Statistical Analysis

A principal component analysis (PCA) was conducted to evaluate the distribution of the studied states on a biplot graph. Simple correlation was determined by Pearson correlation coefficients. The analyses were performed using Jamovi software (version 2.3.17). A statistical significance level of $p < 0.05$ was considered.

## 3. RESULTS AND DISCUSSION:

### 3.1. Results

Table 1 presents the results for the granulometry of green coconut shells. It is noted that at a 48 mesh thickness, there was a greater retention of green coconut shell (53.55 – 58.76%), followed by values at 80 mesh (12.50 – 19.17%). The granulometry values for all the states studied (Maranhão, Piauí, and Ceará) were similar, demonstrating minimal regional differences.

Table 2 shows the results of the composition of ground green coconut shells. The main components were carbohydrates (cellulose and hemicellulose), phenolic polymers (insoluble and total lignin), and inorganic materials (ash).

The PCA (Figure 1) displays two principal components, both explaining 100% of the data variability. PC1 is strongly correlated with the granulometry of the green coconut shell, where the values with the highest explained variance loadings were 16, 20, 48, 80, 150, and 325 mesh. Among the chemical composition values, only insoluble lignin exhibits significant variance. Additionally, the proximity observed in the scatter plot between insoluble lignin and 325 mesh granulometry indicates a correlation between these two properties. PC2 is associated with the

chemical composition of the green coconut, specifically cellulose and hemicellulose. Figure 1 also shows the distribution among the three states sampled, Maranhão, Ceará, and Piauí. This distribution suggests substantial differences in the granulometry and composition of the green coconuts.

Table 3 shows the correlation between the chemical composition and the granulometry of ground green coconut. It is noted that cellulose is positively correlated with retention at 9 mesh. Insoluble lignin has a negative correlation with the 20 mesh thickness and a positive correlation with the 325 mesh thickness.

### 3.2. Discussion

Green coconut shells, a byproduct of the coconut industry, hold potential as a source of biodegradable and renewable materials. This study aims to understand the properties of this material through granulometric and compositional analyses, supplemented by statistical evaluations. The analysis of the results indicates that both granulometry and composition vary depending on the region. The association between the presence of lignin and cellulose and particle sizes provides valuable insights for understanding the properties of this material and its potential applications.

According to the granulometric results, there is a higher retention of ground coconut shells at 48 mesh. This greater retention is associated with the shell's composition and physical structure, such as surface irregularities (Avnir *et al*., 1985), as well as the pre-processing method, in this case, grinding (Marková *et al*., 2016). For the composition of green coconut, the main components present are cellulose, hemicellulose, total lignin, and insoluble lignin, all of which are present in concentrations close to 30%. These results are in line with data from the literature (Arena *et al*., 2016; Ikumapayi *et al*., 2020), which indicate similar concentrations, except for the ash content, which showed values of 5%, significantly higher than those reported in the literature, ranging from 0.56% (Arena *et al*., 2016) to 1.03% (Endut *et al*., 2017).

This comprehensive understanding of the granulometric and compositional properties of green coconut shells underscores their potential for various applications. The study's findings on the correlation between particle size and chemical composition highlight key factors that can influence the material's suitability for specific uses, such as biodegradable packaging, composite materials, and other environmentally friendly products. Further research could explore the optimization of processing methods to enhance the material's properties and expand its applications.

The PCA reveals an association between the presence of lignin and finer granulometry (325 mesh), a result confirmed by Pearson correlation analysis, where insoluble lignin was significantly directly correlated with both 20-mesh and 325-mesh granulometries. This indicates that lignin influences both finer and coarser granulometries. The lignin content affects rigidity and strength (Mafuleka *et al*., 1993; Yang *et al*., 2022). Higher lignin content results in harder, more difficult-to-fragment shells, leading to coarser granulometries. In the analysis of PC2, we observe that the most significant variance loadings were attributed to cellulose and hemicellulose, i.e., compositional factors. Several factors, including the stage of maturation (Hua *et al*., 2007), genetics (Lampugnani *et al*., 2019), and regional characteristics such as environmental conditions (Sensuła & Pazdur, 2013) and farming practices (Chen *et al*., 2014), influence these compositional differences. These geographic composition differences enable the discrimination of the green coconut's geographic origin through discriminant analysis.

The variation in cellulose concentration in coconuts, depending on the region, correlates with coarser granulometry (9 mesh), as shown by the Pearson correlation results. Cellulose is a primary component in forming the fibrillar structure of the cell wall, providing strength and rigidity to the cell wall of the green coconut shell (Jakob *et al*., 2022), along with lignin. Therefore, an increase in cellulose concentration contributes to a more solid structure, resulting in larger particles.

We observe that the states studied are widely distributed in the PCA results, indicating differences in the chemical composition and granulometry of green coconut shells, which allows for their origin determination. The results suggest that regional factors, such as climate, soil, and agricultural practices, play a significant role in determining the characteristics of green coconuts (Chen *et al*., 2014; Sensuła & Pazdur, 2013). The distribution of states in the PCA results provides valuable insights into how the characteristics of green coconuts can vary geographically.

## 4. CONCLUSIONS:

The analysis of green coconut shells reveals a relationship between granulometry and chemical composition, as well as geographic location. These compositional differences enable the determination of the green coconut's origin

among the states of Maranhão, Ceará, and Piauí through discriminant statistical analysis. Furthermore, understanding the regional compositional variations of green coconut is crucial for developing more efficient management strategies and optimizing its applicability.

## 5. DECLARATIONS

### 5.1. Study Limitations

This study recognizes several important limitations, the most significant being the absence of experimental replicates, which restricts our ability to assess measurement uncertainty and limits the statistical power of our findings. The geographical scope, while covering three Brazilian states, represents a limited sampling strategy. Our collection was restricted to specific locations within Maranhão (São José de Ribamar), Piauí (Jerumenha) and Ceará (Fortaleza), which may not fully capture the regional variability in Husk coconut ownership in these states.


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### 5.4. Conflicts of Interest

The authors declare no financial or non-financial competing interests that could have influenced the work reported in this paper. The research was conducted independently without influence from commercial entities or organizations with vested interests in the outcomes. No author has received compensation or benefits from companies involved in coconut processing, agricultural equipment manufacturing, or related industries that might create conflicts of interest regarding the study findings.



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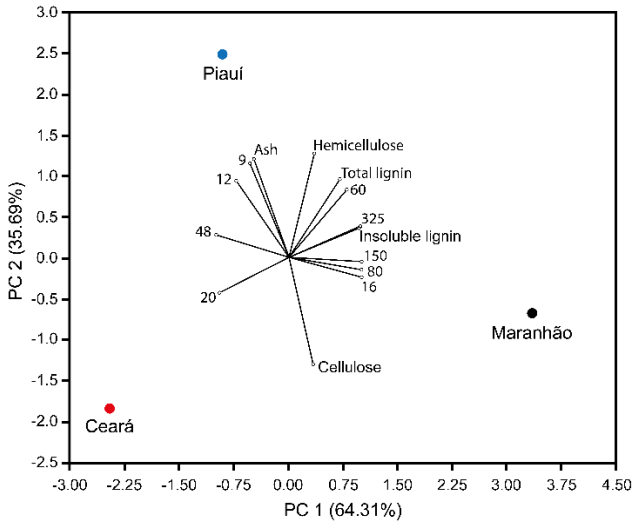


***Figure 1.*** *Principal Component Analysis of Granulometric and Chemical Composition Properties of Green Coconuts Collected in Maranhão, Piauí, and Ceará.*

***Table 1.*** *Granulometry of Green Coconut Shell Powder Collected from Maranhão, Piauí, and Ceará.Granulometria do pó de casca de coco verde coletados do Maranhão, Piauí e Ceará*

| | **Retention (%)** | | |
|---|---|---|---|
| **Mesh** | **Maranhão** | **Piauí** | **Ceará** |
| 9 | 0.00 | 0.07 | 0.02 |
| 12 | 0.05 | 0.15 | 0.10 |
| 16 | 1.74 | 1.33 | 1.28 |
| 20 | 3.13 | 6.22 | 9.93 |
| 48 | 53.55 | 58.47 | 58.76 |
| 60 | 9.06 | 8.86 | 7.61 |
| 80 | 19.17 | 13.64 | 12.50 |
| 150 | 6.12 | 4.58 | 4.11 |
| 325 | 4.67 | 4.09 | 3.45 |

***Table 2.*** *Composition of Green Coconut Shell Powder Collected from Maranhão, Piauí, and Ceará.*

| | **Composition (%)** | | |
|---|---|---|---|
| **Component** | **Maranhão** | **Piauí** | **Ceará** |
| Cellulose | 31.64 | 30.33 | 31.53 |
| Hemicellulose | 23.02 | 24.23 | 21.27 |
| Insoluble Lignin | 34.04 | 33.20 | 31.90 |
| Total Lignin | 2.37 | 2.23 | 2.52 |
| Ash | 5.74 | 6.18 | 5.84 |
| Others | 3.19 | 3.83 | 6.94 |
| Total | 100.00 | 100.00 | 100.00 |

***Table 3.*** *Pearson correlation matrix for the granulometric and chemical composition properties of green coconuts collected in Maranhão, Piauí, and Ceará.*

| | 9 | 12 | 16 | 20 | 48 | 60 | 80 | 130 | 325 | Cel. | HCel | Li | Ls | A |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| **A** | 0.971 | -0.812 | -0.888 | 0.864 | -0.999 | 0.682 | -0.775 | -0.732 | -0.475 | -0.901 | 0.407 | -0.480 | 0.004 | 0.954 |
| **Ls** | -0.648 | 0.454 | -0.999 | 0.840 | -0.942 | -0.127 | -0.775 | -0.732 | -0.475 | -0.901 | 0.407 | -0.480 | 0.004 | 0.954 |
| **Li** | 0.226 | 0.840 | -0.999 | 0.864 | -0.942 | -0.127 | -0.775 | -0.732 | -0.475 | -0.901 | 0.407 | -0.480 | 0.004 | 0.954 |
| **HCel** | 0.685 | -0.127 | 0.682 | -0.942 | -0.915 | -0.127 | -0.775 | -0.732 | -0.475 | -0.901 | 0.407 | -0.480 | 0.004 | 0.954 |
| **Cel.** | 0.115 | -0.775 | 0.682 | -0.915 | -0.939 | -0.985 | -0.994 | -0.985 | -0.876 | -0.523 | -0.154 | -0.879 | -0.539 | 0.639 |
| **325** | -0.601 | -0.732 | 0.992 | -0.939 | -1.000 | 0.933 | 0.725 | 0.769 | 0.933 | -0.315 | 0.854 | 0.931 | 0.991 | 0.177 |
| **130** | -0.547 | -0.475 | 0.899 | -1.000 | -0.876 | -0.985 | -0.994 | -0.985 | -0.876 | -0.523 | -0.154 | -0.879 | -0.539 | 0.639 |
| **80** | -0.601 | -0.775 | 0.998 | -0.915 | -0.942 | -0.127 | -0.775 | -0.732 | -0.475 | -0.901 | 0.407 | -0.480 | 0.004 | 0.954 |
| **60** | 0.115 | -0.127 | 0.682 | -0.942 | -0.915 | -0.127 | -0.775 | -0.732 | -0.475 | -0.901 | 0.407 | -0.480 | 0.004 | 0.954 |
| **48** | 0.685 | -0.127 | -0.999 | 0.864 | -0.942 | -0.127 | -0.775 | -0.732 | -0.475 | -0.901 | 0.407 | -0.480 | 0.004 | 0.954 |
| **20** | 0.226 | 0.840 | -0.999 | 0.864 | -0.942 | -0.127 | -0.775 | -0.732 | -0.475 | -0.901 | 0.407 | -0.480 | 0.004 | 0.954 |
| **16** | -0.648 | 0.454 | -0.999 | 0.864 | -0.942 | -0.127 | -0.775 | -0.732 | -0.475 | -0.901 | 0.407 | -0.480 | 0.004 | 0.954 |
| **12** | 0.971 | -0.812 | -0.888 | 0.864 | -0.999 | 0.682 | -0.775 | -0.732 | -0.475 | -0.901 | 0.407 | -0.480 | 0.004 | 0.954 |
| **9** | — | 0.971 | -0.648 | 0.226 | 0.685 | 0.115 | -0.601 | -0.547 | -0.250 | -0.979 | 0.614 | -0.255 | 0.244 | **0.998** |

Cel = Cellulose; HCel = Hemicellulose; Li = Insoluble lignin; Ls = soluble lignin; A = Ash; Green ($p < 0.05$)