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Analysis of the Chemical Content of Coconut Husk as a Raw Material for Furfural Production

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Abstract. *Coconut-based production generates waste such as coconut husk, which contains up to 35% lignocellulose—a valuable raw material for furfural production. This study aimed to determine the impact of coconut maturity level and coconut husk section on the lignocellulose content in coconut husk. This study used a randomized group design with two factors: coconut maturity level and coconut husk section. The variables observed were moisture, extractive, cellulose, hemicellulose, and lignin contents. Analysis of Variance (ANOVA) was performed for data analysis, followed by Duncan's Multiple Range Test (DMRT). The coconut fruits used were immature and mature ones. This study analyzed the husk at the proximal end, equator, and distal end of the coconut fruit. The results show significant effects of coconut maturity level on the chemical composition of coconut husk at the three sections of the coconut, indicating coconut husk's potential as a raw material for furfural production.*
Keywords: *coconut; level of maturity; coconut husk; hemicellulose.*

Type of the Paper: Regular Article.

1. Introduction

Furfural is an important solvent characterized by a furan ring and an aldehyde functional group. It can be used directly as an organic solvent. It enhances the selectivity of aromatics and unsaturated compounds in related chemical reactions [1]. Currently, it is in high demand in the plastic, food, pharmaceutical, and agricultural industries [2]. It can be produced from materials containing lignocellulose, including agricultural residues, grass, wood, and wood residues, among other waste materials [2–4]. Converting lignocellulosic biomass into chemicals, such as furfural, has attracted significant attention as a pathway toward a sustainable society [5,6].

Plant biomass is generally composed of three main organic fractions: lignin, cellulose, and hemicellulose [7]. Lignin consists of cross-linked polymers of phenolic monomers and has a high molecular weight. Primary cell walls utilize lignin to provide structural support, impermeability, and resistance to microbes [8]. Cellulose, which is the main structural component in plant cell walls, consists of long chains of cellobiose units linked to D-glucose subunits through b-(1,4)-glycosidic bonds. Hydrolysis, catalysed by cellulose or acid, terminates these bonds [9].

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Hemicellulose comprises short lateral monosaccharide branches such as uronic acid, hexoses (glucose, mannose, and galactose), and pentoses (xylose, rhamnose, and arabinose). Long-chain cellulose polymers bind the cellulose in the cell wall into microfibrils through hydrogen and van der Waals bonds. Together with lignin, hemicellulose protects these long-chain cellulose polymers.

Hemicellulose is one of the lignocellulosic components that plays an important role in the production of furfural [10–12], making up around 20–35% of lignocellulosic biomass [13]. Research results show that the yield of furfural from biomass waste increases with the hemicellulose content in the biomass. Studies have measured furfural yield from a variety of sources, including ± 29% from sugarcane bagasse with a hemicellulose content of ± 21% [14], ± 42% from rice straw waste with a hemicellulose content of ± 33% [15], ± 26.34% from oil palm fronds [16], and 30.2% from corncobs [17]. Sugarcane bagasse and corncobs are the most dominant raw materials in the furfural industry worldwide. More than 98% of all furfural production uses corncobs as a primary raw material, with China being the largest producer of furfural on the global stage [18].

The coconut palm (*Cocos nucifera* L.) is frequently dubbed the "tree of life" as it sees a multitude of applications, including as food, fuel, and fiber source [19]. Some of the coconut derivative products are coconut milk, virgin coconut oil, copra, and nata de coco. Both immature and mature coconuts are widely used, with a surge in the consumption of pure young coconut water for health being reported [20]. However, widespread use of coconuts poses the issue of abundant waste generation. As each coconut fruit consists of 35% husk, 12% shell, 28% fruit flesh, and 25% coconut water [21], coconut husk contributes profusely to the waste produced for each fruit. Assessing its chemical composition will thus reveal opportunities for its utilization in various industries, including furfural production.

Where in the plant lignocellulose is found determines the lignocellulose content. AOAC [22], who examined lignocellulose content in bamboo, and Rezekinta et al. [23], who did in mensiang grass, demonstrated that lignocellulose content differed in the upper, middle, and lower parts of the plants. To identify which part of coconut husk is the most potential to be used as a source of furfural, it is necessary to conduct an analysis of lignocellulose content in several parts of coconut husk. Based on the foregoing, this study conducted a chemical analysis of coconut husk based on coconut maturity levels (immature and mature) and coconut husk sections (proximal end, equator, and distal end).

2. Materials and Methods

2.1. Tools and Materials

Experiments were conducted using coconut (*Cocos nucifera*) husk from both immature and mature coconuts, obtained from Padang Pariaman Regency. Immature coconuts are characterized by a green skin color with brownish spots, soft flesh, and copious coconut water. Mature coconuts are characterized by a brown skin color, drier coir, harder flesh, and little coconut water. The coconuts were cut with a cutting tool and then separated into three sections: proximal end (stem end), equator (midsection), and distal end (blunt end). The husk in the three sections was cleaned and reduced in size to 0.5 cm, oven-dried at 60°C for 60 minutes, and ground to powder. The coconut husk powders were then stored in zipper storage bags for further analysis. The materials used in this research were 72% H_2SO_4, alcohol, and $NaClO_2$ (Merck, Germany), acetone, glacial acetic acid (SmartLab), and distilled water. Meanwhile, the tools used were an oven (Memmert) and an analytical scale (Fujitsu).

2.2. Experimental Design

The experiments in this study used a factorial randomized group design with two factors. The first factor was coconut maturity level, consisting of immature and mature levels. The second factor was coconut husk section, consisting of the section near the stalk (proximal end), the middle section (equator), and the tip away from the stalk (distal end). As a result, there were six treatment combinations in total. Data were analyzed using Analysis of Variance (ANOVA). If a treatment had a significant effect, the analysis was followed by Duncan's Multiple Range Test (DMRT).

2.3. Research Implementation

2.3.1. Moisture Content [22]

The aluminum cups used for moisture content measurement were first dried in an oven at 105°C for one hour, and then the temperature was lowered in a desiccator. Afterward, each aluminum cup was filled with 3 g of sample and re-heated in the oven at 105°C until reaching a constant weight. Moisture content was calculated using equation (1):

$$Moisture\ Content\ (\%) = \frac{(B - A)}{C}\ x\ 100\% \tag{1}$$

Where A is the weight of the aluminum cup (g), B is the weight of the aluminum cup and the sample after oven-drying (g), and C is the initial weight of the sample (g).

2.3.2. Extractive Content [23]

Coconut husk powder was weighed at 10 grams (a) and put in a filter paper thimble with a known weight. The thimble was put into an extraction tube and positioned so that the cup became immersed in the solvent. Extraction was carried out for six hours. Upon completion, the thimble was removed from the extraction tube. The sample was washed with 50 mL of ethanol for benzene removal and then oven-dried at 105 ± 3°C for two hours. It was then let to cool in a desiccator and weighed to a constant weight (b). Extractive content was calculated using equation (2):

$$Extractive\ Content\ (\%) = \frac{(a-b)}{a}\ x\ 100\ \% \tag{2}$$

2.3.3. Holocellulose Content [23]

Extracted powder sample (weighing approximately 5 g) was put into a 200 mL Erlenmeyer flask fitted with a lid. It was then added with 160 mL of distilled water, 1.5 g of $NaCO_3$ (sodium carbonate), and 10 drops of CH_3COOH (glacial acetic acid). The sample-filled Erlenmeyer flask was heated over a water bath for four hours at 70–80°C. During heating, the Erlenmeyer flask was shaken periodically and added with 10 drops of acetic acid every hour, followed by 1.5 g of $NaClO_2$. After four hours, the Erlenmeyer flask was placed in ice water, and its content was filtered using a glass filter. The sample material was washed with distilled water and later with acetone. The material was dried in a vacuum oven at 40°C until it reached a constant weight before being cooled in a desiccator and weighed.

2.3.4. Cellulose Content [23]

A total of 2 g of holocellulose was weighed (B), then heated in a 500 mL beaker glass with 200 mL of 1.3% H_2SO_4 for two hours over boiling water in a water bath. After two hours, the mixture was filtered and washed with 150 mL of distilled water until neutralized. Upon reaching a neutral state, the mixture was then washed with ethanol. It was then dried in an oven at 105°C until it reached a constant weight (A). The value of cellulose content was calculated using equation (3):

$$Cellulose\ Content = \frac{A}{B}\ x\ 100\% \tag{3}$$

2.3.5. Hemicellulose Content

Holocellulose in plants is composed of hemicellulose and cellulose. Thus, hemicellulose content can be determined by reducing the total weight of holocellulose (a) by the weight of cellulose (b), as expressed in equation (4):

$$Hemicellulose\ Content = a - b \tag{4}$$

2.3.6. Lignin Content [23]

A 2-g portion of extracted sample (b) was put into a 500 mL beaker glass, added with 25 mL of 72% H_2SO_4. It was left to stand for two hours at room temperature, with frequent stirring during this time. Subsequently, it was diluted with 500 mL of distilled water and heated to boiling for approximately four hours. After four hours, it was let to cool, and the precipitate was filtered using a filter paper and washed with distilled water to remove acid. The precipitate on the filter paper was dried in an oven at 105°C until reaching a constant weight (a). The value of lignin content was calculated using equation (5):

$$Lignin\ Content\ (\%) = \frac{a}{b} x\ 100\% \tag{5}$$

3. Results and Discussion

3.1. Raw Material Preparation

This study used both mature and immature coconuts. Mature coconuts are characterized by brown, hardened outer skin and thickened flesh, whereas immature ones are characterized by green skin and thin flesh (Fig. 1).

(a) (b)

Fig. 1. Coconut cross-sections: (a) mature coconut; (b) immature coconut

Furthermore, coconut husk consists of three sections, namely, proximal end, equator, and distal end (Fig. 2). The proximal end has finer fibers, while the middle section has coarser ones. However, compared to the previous sections, the distal end contains fewer fibers and a higher proportion of cork. According to Nikhonta et al. [24], coir fiber content rises slightly between 1 and 7 months after flowering and then increases more sharply, by about 12%, afterward.

(a) (b) (c)

Fig. 2. Coconut husk sections: (a) proximal end; (b) equator; (c) distal end

3.2. Chemical Composition of Coconut Coir by Maturity Level and Coconut Husk Section

Chemical composition analysis was carried out to determine the chemical composition (moisture, extractive, holocellulose, cellulose, hemicellulose, and lignin contents) of coconut husk based on coconut maturity levels (mature and immature) and coconut husk sections (proximal end, equator, and distal end).

3.2.1. Moisture Content

Moisture content in coconut husk decreased with maturity. The results of the moisture content measurement show that immature coconuts had high moisture content in their husk, ranging from 13.09% to 24.75%, whereas mature coconuts had much lower moisture content, ranging from 5.26% to 7.70%. The research results on the moisture content of coconuts were reported by Adeyi [25] (5.43%). Statistical results show that there was a significant relationship between coconut maturity level and moisture content. However, different results were found for the relationship between coconut husk section and moisture content.

From observations of the samples, it was found that mature coconut husk tends to be drier and lighter compared to immature coconut husk, which contains more water. According to Nikhonta et al. [24], during the first six months, only the skin and shell develop rapidly, with the cavity filled with water. In the next 6–8 months, the skin and shell harden and thicken. In the ripening process, the sugar content in the liquid part of the endosperm increases from about 1% during the first few months to a maximum of 5–8% in the sixth to eighth month, before decreasing to about 2%. At full maturity, the husk begins to dry naturally, browning and hardening, with a significant decrease in its moisture content.

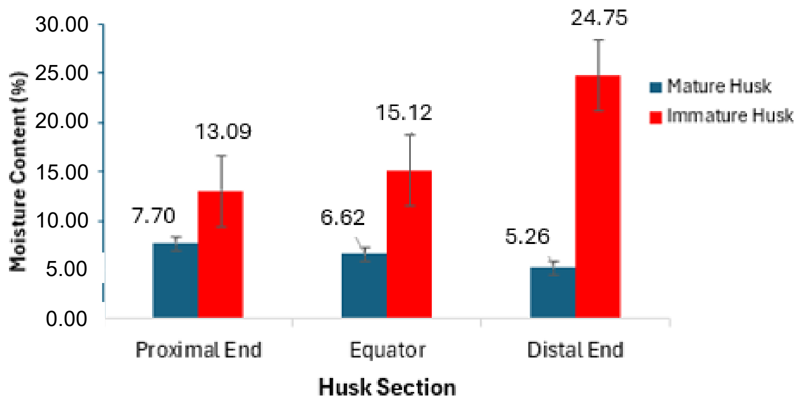

Fig. 3. Moisture Content

Fig. 3 shows decreases in moisture content with the distance of husk section from the stalk. The highest water content was found at the proximal end (top), which gradually declined in the equator (middle) and finally at the distal end (bottom). Meanwhile, in immature coconuts, the water content increased with distance from the stalk, which means the highest proportion of moisture/water should be at the distal end. This is because while the proximal end and equator are formed of fibrous material, which grows stiffer and longer as it moves toward the middle section, the distal end contains fewer fibers and more cork.

3.2.2. Extractive Content

Extractives are the products of secondary metabolism which are composed of both organic and inorganic components. The solubility of husk extractives in ethanol–benzene solution shows the ease with which semi-polar and polar compounds are extracted from coconut husk. Extractive content tends to decrease toward the distal part of husk in mature coconuts. However, the opposite is true for immature coconuts, where the extractive content is found to be highest.

Based on the results of extractive content measurement in ethanol–benzene solution, mature coconut husk had extractive content of between 9.13% and 17.78%, compared to the 3.6–5.76% content in immature coconut husk. As shown in Fig. 4, the extractive content in mature coconut husk increased from the proximal end to the distal end. On the other hand, in immature coconut husk, extractive content at the distal end was lower than in the proximal end.

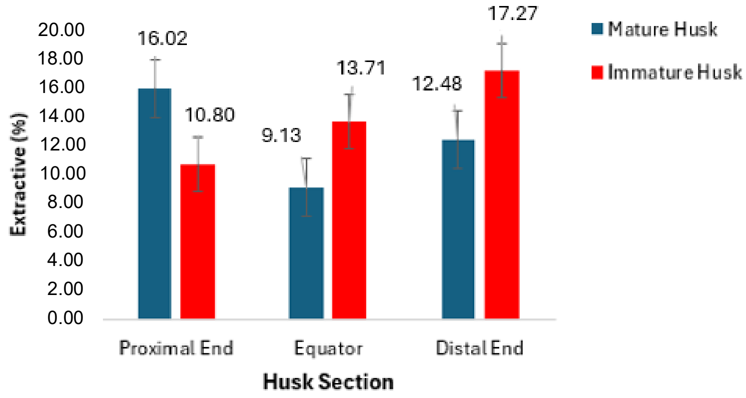

Fig. 4. Extractive Content

Latif et al. [26] has previously found that coconut husk from Sungai Petani, Kedah, Malaysia, had extractive content of 9.16%. A similar value (9.10%) was found by Ram and Mondal [27].

3.2.3. Cellulose Content

Holocellulose is the standard for quantifying cellulose and hemicellulose in plant cell walls. It is obtained from raw materials by delignification method, using strong oxidizing agents or acidic or alkaline solutions at high temperatures [28]. Holocellulose content varies depending on the species, biological variations (such as within-species genetic differences), and growing conditions [29].

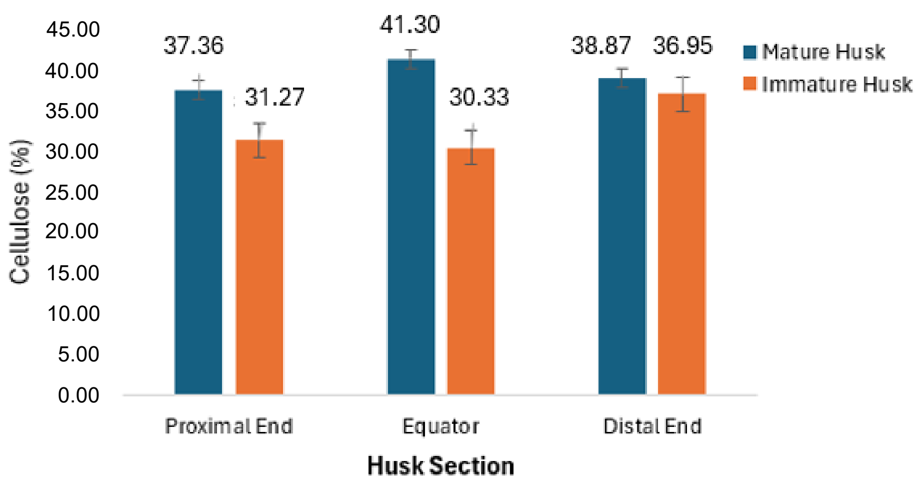

Fig. 5. Cellulose Content

The principle of holocellulose analysis is to remove lignin, extractives, and ash, but keep the cellulose and hemicellulose components intact. Cellulose is a linear polymer composed of D-glucose units linked by β-(1→4) glycosidic bonds and is strongly associated with hemicellulose and lignin [30]. The results of the cellulose content measurement are illustrated in Fig. 5.

The results of the study show that the cellulose content of coconut husk ranged from 31.27% to 41.30%. Lertwattanaruk and Suntijitto [31] reported 48.10% cellulose content in coconut husk, which is higher than the values reported previously by other studies (27.19% [26], 34.90% [31], and 42.30% [27]. The cellulose composition varied by age and variety of coconut used in the studies.

3.2.4. Hemicellulose Content

Hemicellulose is one of the constituents of plant cell walls, consisting of a collection of several sugar/heteropolysaccharide units and grouped based on the sugar residue main components, such as xylan, mannan, galactan, and glucan [30]. Hemicellulose content measurement results show that immature coconuts had higher hemicellulose content than mature ones. Hemicellulose measurement was carried out at the three sections of coconut husks. The results reveal that coconut husk contained hemicellulose at 17.36–25.12% (mature coconuts) and 16.31–25.93% (immature coconuts), which fluctuated from proximal to distal ends (Fig. 6). Bajpai [29] previously found that coconut husk contained hemicellulose at 12.69%, while Adeyi [25] found it at 23.70%.

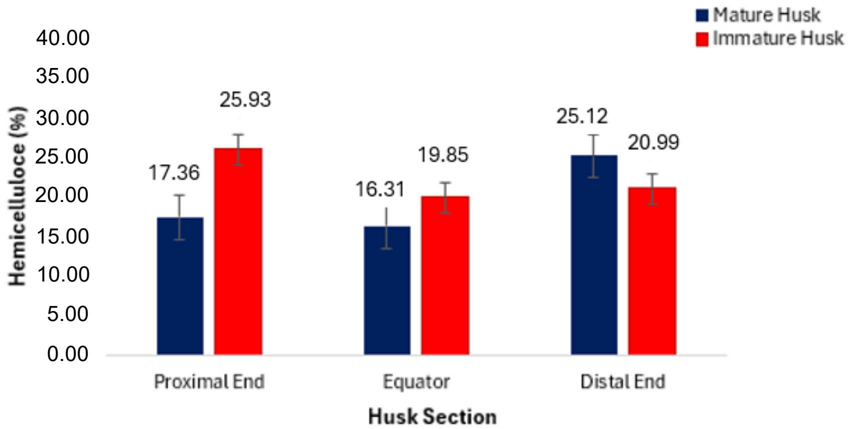

Fig 6. Hemicelluloce Content

The research results show that there was no relation between coconut maturity level and the hemicellulose content in coconut husk, but a relationship was found between coconut maturity level and coconut husk section.

Hemicellulose is the most important component in the manufacture of furfural. Hemicellulose hydrolysis primarily produces pentose (xylose), while furfural makes up a smaller portion of the products. Pentose (xylose) is formed from the depolymerization of hemicellulose contained in lignocellulosic materials. The utilization of all pentosans containing fibrous materials as raw materials for furfural production is a theoretical possibility. However, industrial production of furfural necessitates a minimum pentosan content of approximately 15–20% [17]. Consequently, the conversion of pentosans in raw materials into furfural is only feasible at a rate of approximately one-third through existing production processes.

3.2.5. Lignin

Lignin is the second most abundant polymer in plant cell walls, after cellulose [32]. The results of lignin content measurement show that mature coconut husk had lignin content of 36.16–48.85%, while immature coconut husk had 19.45–31.42%. Statistical results show that coconut maturity level and coconut husk section were related to lignin content, as shown in Fig. 7. Similar results were reported by Kondo et al. [33], who obtained lignin content of 39% in coconut husk previously soaked in 50% NaOH. Others obtained varying values, including Latif et al. [26] (37.93%), Lertwattanaruk & Suntijitto [31] (29.80%), and Ram & Mondal [27] (47.5%).

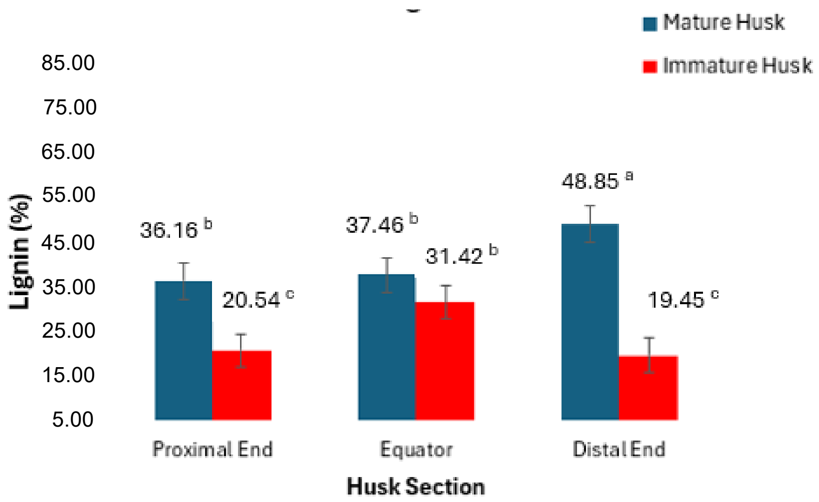

Fig. 7. Lignin Content

This study shows that the lignin content in mature coconut husk was higher than in immature coconut husk. Additionally, the lignin content in the distal end of mature coconut husk was higher than in the proximal end, because the distal end contains dust and cruder husk fibers than other parts. In immature coconuts, the highest lignin content was found in the equator of the husk. The fibers in this part of the husk are coarser than those in both the proximal and distal ends, resulting in a higher amount of lignin.

4. Conclusion

From the results of the analysis, it is concluded that coconut maturity levels (mature and immature) and coconut husk sections (proximal end, equator, and distal end) did not result in significant differences in cellulose and hemicellulose contents, indicating that all types of coconut husk have the potential to be used as a raw material for furfural production.

Abbreviations

Not applicable.

Data availability statement

Data will be shared upon request by the readers.

Author's Contribution

Dewi Arziyah: writing – original draft, conceptualization, methodology, resources, formal analysis, investigation, data curation. **Anwar Kasim**: writing – review & editing, validation, data curation, formal analysis, conceptualization, supervision, funding acquisition. **Alfi Asben**: conceptualization, supervision, data curation, writing – review & editing. **Munzir Busniah**: formal analysis, investigation, writing – review & editing.

Declaration of Competing Interest

The authors of this manuscript declare no conflict of interest or competing interest

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