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IDENTIFICATION OF AVAILABILITY AND LIGNOCELLULOSIC PROPERTIES IN COCONUT DREGS WASTE

Wenny Surya Murtius*[,1,2], Bambang Dwi Argo[3], Irnia Nurika[4], Sukardi[4]

[1]Doctoral Program in Agricultural Industrial Technology, University of Brawijaya, Malang, Indonesia
[2]Department of Agricultural Industrial Technology, University of Andalas, Padang, Indonesia
[3]Department of Agricultural and Biosystem Engineering, University of Brawijaya, Malang, Indonesia
[4]Department of Agricultural Industrial Technology, University of Brawijaya, Malang, Indonesia

***Corresponding author**
Email: wenny.murtius@gmail.com

***Abstract**. Agricultural waste, including coconut pulp, contains lignocellulose and is a very important, renewable and sustainable industrial raw material. Many of the food, textile, pharmaceutical, paint and resin, agrochemical, oil processing, and other sectors utilize lignocellulosic derivatives. The objectives of this study were to determine the availability of coconut pulp in Padang City-West Sumatra, analyse the lignocellulosic components contained and cell surface morphology, and observe the chemical elements in coconut pulp waste. An exploratory approach was used in this study to achieve these objectives. The results showed that there were 98 coconut milk entrepreneurs spread across traditional markets in Padang City, West Sumatra. Every day the coconut milk squeeze business examined produces ± 1.18 tonnes of coconut pulp. Coconut waste also contains 47.18% cellulose, 10.58% lignin, and 12.10% hemicellulose. Based on the XRD results, the crystal size of coconut pulp obtained from XRD observation is 11.8 nm.*
***Keywords:** coconut dregs; lignocellulose; potential; raw materials; West Sumatra*

1. Introduction

Based on the availability of raw materials, Indonesia has the potential to produce various bio-products with high economic value, such as biofuels, biochemical, biodiesel, bioethanol, aromatic compounds, and materials that can be used to replace fossil-based derivative products (Raj *et al*., 2022). These products can be produced from agricultural waste, namely lignocellulosic components or biomass. Biomass is one of the world's most cost-effective, environmentally benign, and widely available resources. Cellulose, lignin, extractives, and traces of hemicellulose are all found in biomass resources (Pirah *et al*., 2022).

Lignocellulose is the main component of the plant cell wall that belongs to polysaccharide compounds and is available in the world (Vydrina *et al*., 2023). Plant cell walls are divided into three layers: primary walls that enclose growing cells or cells with the potential to grow, secondary walls that include thickened lignin structures and specialized cells that surround them, such as fiber cells, and intercellular layers. The primary wall is made up of cellulose, hemicellulose, and pectin, and it has multilayered nanostructures (Wu *et al*., 2022). Over the years, lignocellulose biodegradation research has grown in importance (Nurika *et al*., 2020). Lignocellulose can also be made to produce adsorbents from agricultural biomass (waste product) (Tanasă *et al*., 2020).

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Lignocellulosic particles from *Eucalyptus*, coffee husk, banana *pseudosterm*, and coconut shell could be utilized to make fiber cement (Teixeira *et al*., 2020). Furfural is produced from biomass containing xylan (hemicellulose) (Peleteiro *et al*., 2016). Pentose sugar, xylose, is the main substrate in furfural production (Xia *et al*., 2019). This means that hemicellulose, as one of the components of lignocellulosic biomass, is the main raw material for furfural production. Hemicellulose is commonly found in agricultural waste, including waste from coconut processing. The content of cellulose, hemicellulose, and lignin in coconut dregs allows it to be processed and produce products with high economic value (Gonçalves *et al*., 2019; Leesing *et al*., 2022).

Indonesia, as the country with the highest coconut production in the world, also has abundant coconut processing waste that has not been optimally utilized. West Sumatra is a province in Indonesia that uses coconut milk as a raw material in various dishes, so the coconut milk squeeze business is also widely found in traditional markets and residential areas. The current utilization of coconut dregs, based on observations, is generally used as an animal feed additive, with a selling price of IDR 15,000 per sack. However, based on their lignocellulosic component, coconut dregs have the potential to be processed into other products with high economic value. Several studies related to the utilization of lignocellulosic components in coconut dregs have also not been widely developed, including as a raw material for 2nd generation and 3rd generation bioethanol (Leesing *et al*., 2022). Studied related to sugars production from coconut dregs by hydrothermal pretreatment and acid hydrolysis have also been reported (Mariano *et al*., 2020). Research related to the extraction of galactomannan in coconut dregs also revealed several derivatives of lignocellulose contained in coconut dregs (Barlina, 2015).

As a result, the purpose of this study was to determine the availability of coconut dregs in Padang City-West Sumatra, as well as the analysis of lignocellulose components, cell surface morphological features, and chemical elements found in coconut dregs. Characterization methods included X-ray diffraction, Fourier transform infrared, and FESEM. Therefore, this research can be used as a reference for the next stage of research related to the utilization of coconut dregs waste.

2. Methods

2.1. Tools and Materials

The tools used in this research were interview attributes, reflux (pyrex), water bath (memmert), oven (memmert), FESEM (FEI Inspect F50 FESEM instrument with a 12.5 mm WD lens), X-ray Diffraction (Advance Bruker (Germany) XRD D8 using Cu Kα radiation at an operating voltage and current of 40 kV and 20 Ma), and FT-IR (ATR Platinum Diamond Bruker

Universal). The materials used are coconut dregs (obtained from coconut milk squeezing waste in Padang City), H_2SO_4 1N, H_2SO_4 72%, and H_2O pH 7

2.2. Methods

The method used is an exploratory method, which reveals facts related to the availability and potential of lignocellulose components in coconut dregs consisting of cellulose, hemicellulose, and lignin content, cell surface morphology (FESEM), FTIR, and XRD. The research was conducted in two stages, namely, the identification of the availability of coconut dreg waste based on primary data in Padang City as stage one. The number of samples was determined based on the Isaac Michael equation. The sample size determination table from Isaac and Michael makes it easy to determine the number of samples based on an error rate of 1%, 5%, and 10% (Hendryadi, 2021). Stage two is the identification of potential lignocellulosic components (carried out three times) in coconut dregs based on the content, composition, and morphological structure of cells, as well as the chemical elements contained.

2.3. Lignocellulose Content Test by Chesson Method (Nurika *et al*., 2022).

Dried coconut dregs (sample) of 1 gram (weight a) was weighed and 150 ml of H_2O was added and reflux at 100°C with a water bath for 1 hour. The reflux process with a water bath was carried out to stabilize the mixing volume of raw materials with H_2O. The residue was filtered and washed with distilled water until neutral. The neutralised residue was dried in an oven until constant. Drying was carried out at 105 °C with a time of 1 hour. After the constant material was weighed as value (b), the value of solubility in hot water (HWS) was calculated by (1). HWS is used to determine the starch dissolved in hot distilled water.

$$\text{HWS (\%)} = \frac{a-b}{a} 100\% \quad (1)$$

2.3.1. Hemicellulose Content Procedure

The residue was added to 1 N H_2SO_4 and refluxed in a water bath at 100°C for 1 hour. The filtered residue was washed until neutral, then the residue was oven dried until constant at 105°C for 1 hour and weighed as (c) value. The hemicellulose content was calculated by (2).

$$\text{Hemicellulose} = \frac{b-c}{a} x100\% \quad (2)$$

2.3.2. Cellulose Content Procedure

The residue was added 10 ml of 72% H_2SO_4 soaked at room temperature for 4 hours. 150 ml of 1 N H_2SO_4 was added and refluxed on a water bath 1 hour on counter cooling. The residue was filtered and washed with H_2O until neutral. It was then heated in an oven at 105°C for 1 hour.

After 1 hour and considered constant the residue was weighed as value (d). The cellulose content was calculated by (3).

$$\text{Cellulose} = \frac{c-d}{a} x100\% \quad (3)$$

2.3.3. Lignin Content Procedure

The residue was burnt by putting it into an electric furnace or heat it up to 500^0 C for 2 hours, let it cool slightly then put it in a desiccator for ½ hour. After that, constant residue weighed as value (e). The lignin content was calculated by (4).

$$\text{Lignin} = \frac{d-e}{a} x100\% \quad (4)$$

2.4. FTIR observation (Pratama *et al*., 2019)

Observations using FTIR were made to determine the functional groups present in coconut dregs. Infrared spectra were recorded with an ATR Platinum Diamond Bruker Universal. Samples were placed on the surface of the FTIR diamond for analysis with transmittance measured at wave numbers 4000-400 cm-1.

2.5. X-Ray *Diffraction* (X-RD) observation (Pratama *et al*., 2019)

It is one of the oldest and most frequently used material characterization methods to date. This technique is used to identify the crystalline phase in the material by determining the lattice structure parameters as well as to obtain the particle size. X-Ray equatorial diffraction profiles of the samples were collected by an Advance Bruker (Germany) XRD D8 using Cu Kα radiation at an operating voltage and current of 40 kV and 20 Ma, respectively. Diffraction intensities were recorded between 5 and 80θ (2θ angle range).

2.6. FESEM Observation (Pratama *et al*., 2019)

Morphological analysis was performed using a FEI Inspect F50 FESEM instrument with a 12.5 mm WD lens and magnification at 100 and 1000.

3. Results and Discussion

3.1. Identify the Availability of Coconut dregs

Indonesia is the country with the highest coconut production in the world. In addition to meeting export needs, there are also many small to large industries related to coconut processing. In general, coconut is processed by the Indonesian people as a cooking ingredient (coconut milk), coconut oil raw material, virgin coconut oil (VCO) raw material, copra, and so on. Generally, the coconut meat component reaches 30% of the weight of the whole coconut fruit, or around 300–500 g per fruit. (Barlina, 2015; Barlina, 2022). The processing process applied produces coconut waste in the form of coconut dregs, coconut fiber, and coconut shells. As one of the wastes or by-products of coconut meat processing that is generally discarded, coconut dregs are even left to accumulate to cause a rancid (Mariano *et al*., 2020) odor that, if left unchecked, can pollute the environment (Syahputri & Faridah, 2023). According to data obtained from coconut milk squeeze entrepreneurs in Padang City, West Sumatra, the availability of coconut dregs is abundant, but its utilization is still limited. Coconut dregs are usually only used as animal feed or discarded. Based on interviews conducted with coconut milk squeezing entrepreneurs in ten traditional market locations in Padang City, West Sumatra, data on the number of coconuts squeezed daily (Figure 1), the amount of coconut dregs each entrepreneur squeezes daily (Figure 2), and the amount of coconut dregs based on traditional market locations is shown in Figure 3.

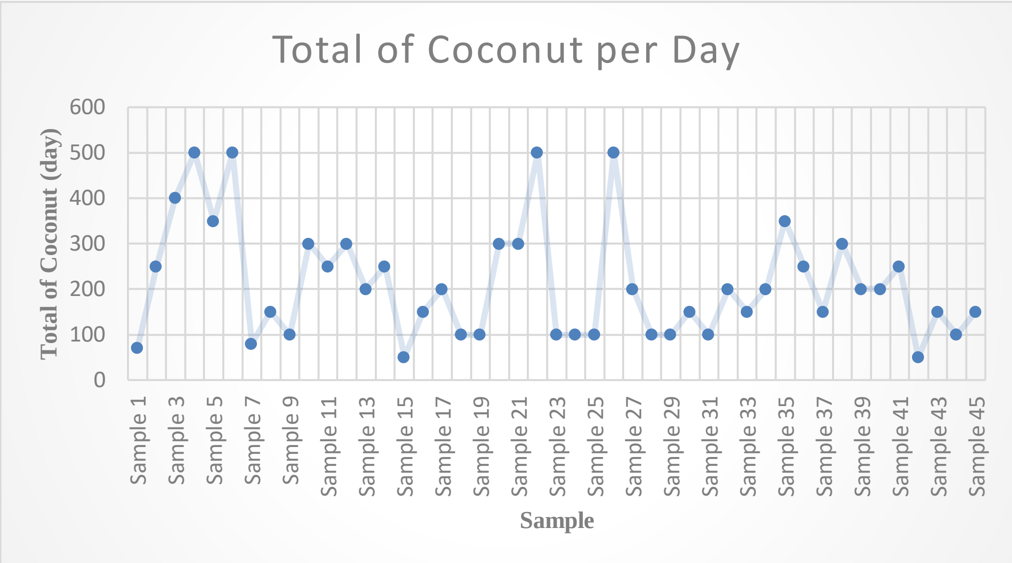


Figure 1. Total of Coconuts Squeezed for Coconut Milk per Day

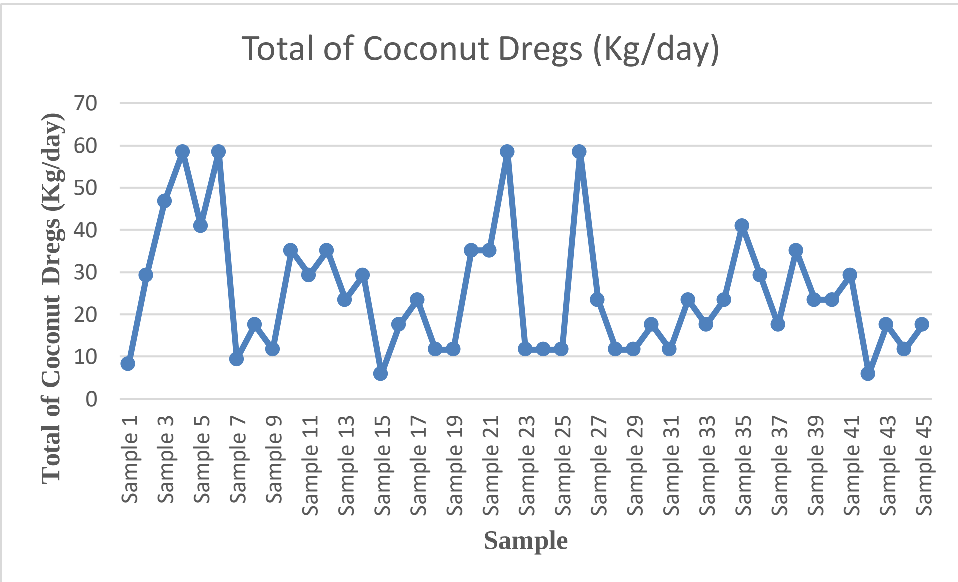


Figure 2. Amount of Coconut Dregs Produced (Kg/day)

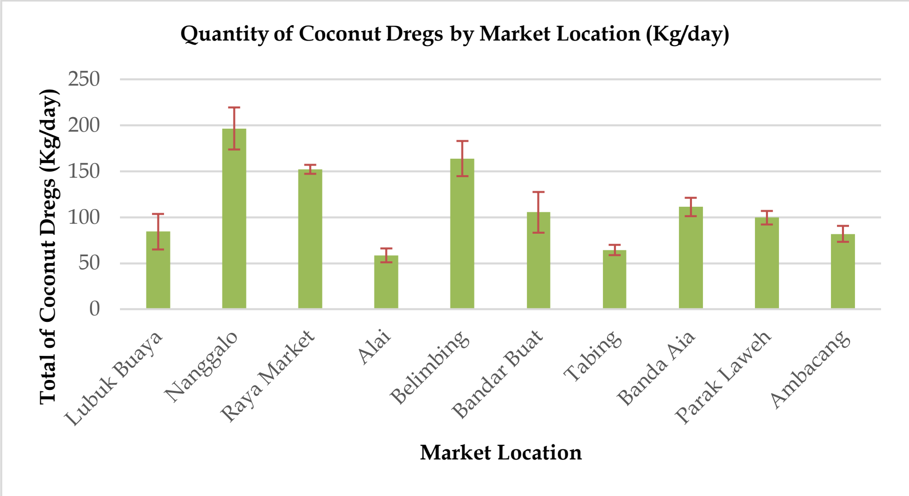


Figure 3. Quantity of Coconut Dregs by Traditional Market Location

The interview results also show that coconut milk squeeze entrepreneurs in Padang City, West Sumatra, have a productive age range of 19–58 years. The length of time running the business also varies from just 1 year to some up to 25 years. The number of coconuts used each day ranges from 50 to 500 grains (Figure 1), and the handling of coconut dregs is generally put into sacks. The utilization of coconut pulp as animal feed, either for personal livestock or sold to be used as animal feed. The selling price also varies from IDR 10,000 to IDR 15,000 per sack (50kg sack size). Coconut suppliers come from Padang Pariaman District, Pariaman City, Solok City, and Pesisir Selatan District. These areas are known as coconut-producing areas and have extensive coconut plantations in West Sumatra. In addition, the districts and cities from which the coconuts originate are the ones with the closest distance to Padang City.

Interviews were also conducted to determine the amount of coconut dregs produced each day by the coconut milk squeeze business (Figure 2). This was done to determine the availability of coconut dregs in Padang City. The amount of coconut dregs depends on the number of coconuts that are grated and squeezed every day, so each entrepreneur will produce a different amount of pulp. Thus, data on the amount of coconut dregs produced based on the location of traditional markets was obtained (Figure 3). The standard deviation indicates an imbalance in the amount of coconut dregs produced each day, depending on the number of coconuts used by each coconut milk squeezing entrepreneur. However, there is a tendency for the standard deviation to be small at some market points. This is thought to be because the number of consumers who use coconut milk squeezing services tends to visit the market more, and entrepreneurs g*et al*most the same opportunity.

3.2. Lignocellulose Assay

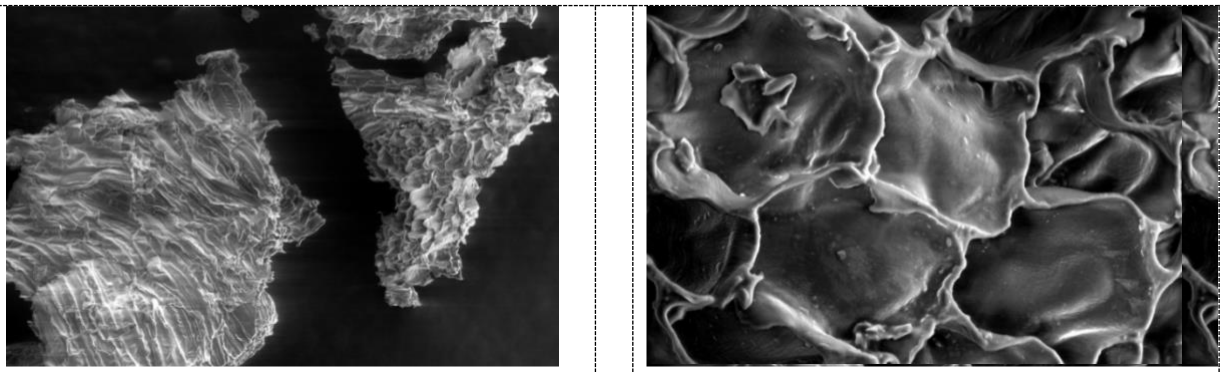
Lignocellulose is the main component of plant cell walls that are classified as polysaccharide compounds. The distribution of cellulose, hemicellulose, and lignin changes significantly between plant cell layers. The secondary wall is often thicker and comprises thick cellulose, consisting of secondary layer 1, secondary layer 2, and secondary layer 3. Furthermore, the middle lamella, which binds adjacent cells, is almost entirely composed of lignin (Menon & Rao, 2012). Research showed that the lignocellulose component in coconut dregs contains 47.18% cellulose, 12.10% hemicellulose, and 10.58% lignin (Table 1). Other research discuss about the utilization of coconut dregs as a raw material for 2nd and 3rd generation bioethanol (Mariano *et al*., 2020), coconut dregs after hydrothermal pretreatment and acid hydrolysis to produce sugar (Leesing *et al*., 2022), as well as research related to the extraction of galactomannan from coconut dregs (Barlina, 2015). The lignocellulose content of coconut dregs has also been tested. The content of cellulose, hemicellulose, and lignin in coconut dregs allows them to be processed and produce products with high economic value (Cardoso & Gonçalez, 2016; Leesing *et al*., 2022).

Table 1. Results of Lignocellulose Test on Coconut Dregs

No	**Component**	(Leesing *et al*., 2022)	(Mariano *et al*., 2020)	(Barlina, 2015)	**Result**
1.	Cellulose (%)		14.8	3.32	47.18
2.	Hemicellulose (%)		27.7	1.50	12.10
3.	Lignin (%)		12.4	1.73	10.58
4.	Fructose (%)	14		27.93	
5.	Glucose (%)	1	0.114*	20.38	
6.	Galactose (%)	15.6		10.64	
7.	Xylose (%)	17.1	2.358*	12.91	
8.	Mannose (%)	19.9		1.96	
9.	Rafinose (%)	23.7			
10.	Maltotriose (%)	3		4.08	
11.	Arabinose (%)		0.236*	8.53	
12.	Rhamnose (%)			5.96	
13.	Cellobiose (%)			4.91	

3.3. Morphological Observation of Coconut Dregs by *Scanning Electron Microscopy* (FESEM)

Characterization of coconut dregs surface morphology was carried out by Scanning Electron Microscopy (FESEM). FESEM is one of the observations made on the cell surface or structure of a solid object. Morphology and structure of cells can be analyzed by FESEM (Lu *et al*., 2022). The results of the morphological observation of the coconut dreg surface are presented in Figure 4.



(a) (b)

Figure 4. (a) Overview of Coconut Dregs Morphology Observed with FESEM 100 x mag, (b) Structure of Coconut Dregs Observation with FESEM 1,000 x mag

Figure 4a shows that the morphological shape of the coconut dregs surface is uneven and looks like an irregular hollow sponge at 100 x magnification. After continuing with the observation at 1,000 x magnification, a visible square-shaped cell surface was arranged. This is in accordance with the explanation of the results of the analysis of coconut pulp powder using FESEM magnification of 3000 x from the actual size of 426–600 µm. The surface of coconut dregs fiber powder is uneven, and the cell shape tends to be irregular (Erminawati *et al.*, 2017). Morphological observation of the rice husk cell surface showed a similar type to the surface morphology of coconut dregs cells. Where there is thickening of the secondary cell wall, is part of the plant cell with the main composition of cellulose, xylan, glucomannan (hemicellulose), and lignin. In some cell types, the secondary cell wall thickens and undergoes composite lignification, which is interconnected between cellulose, glucuronoxylans, and other hemicelluloses with a network of phenylpropanoid acids and lignin (Carpita & McCann, 2020). The secondary cell composition of plants differs, which can be differentiated based on the species and also the sclerenchyma cells of the plant. For example, the hemicellulose component in angiosperms is dominated by xylan and gymnosperms by gluco-galactomannan (Zhong *et al*., 2019).

Observation of the structure and surface of coconut dregs was also carried out by energy dispersive X-ray spectroscopy (EDS) analysis to determine the distribution of elements contained in coconut dregs, as presented in Figure 5. The results of EDS analysis (Figure 5 and Figure 6) show that coconut dregs are dominated by carbon and oxygen elements, followed by aluminum, kalium, phosphor, calcium, and silicon, respectively. Minerals such as aluminum, silicon, iron, and calcium have been identified in the fiber (Moghaddam, 2023). The EDS map's color dots, which represented the identified carbon, oxygen, aluminum, silica, and kalium, clearly covered the fiber sections.

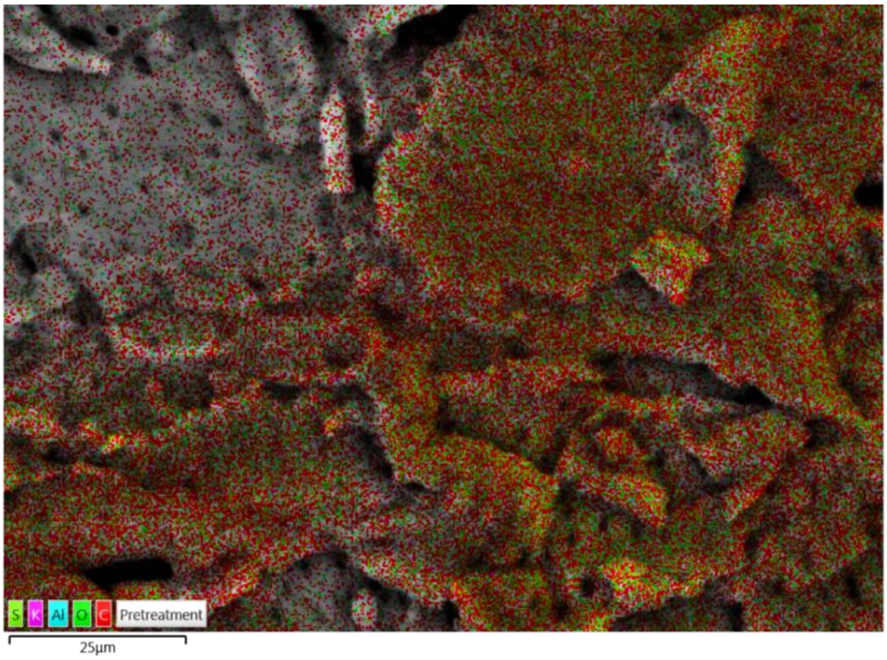


Figure 5. Elemental Distribution of Coconut Dregs EDS Analysis Results

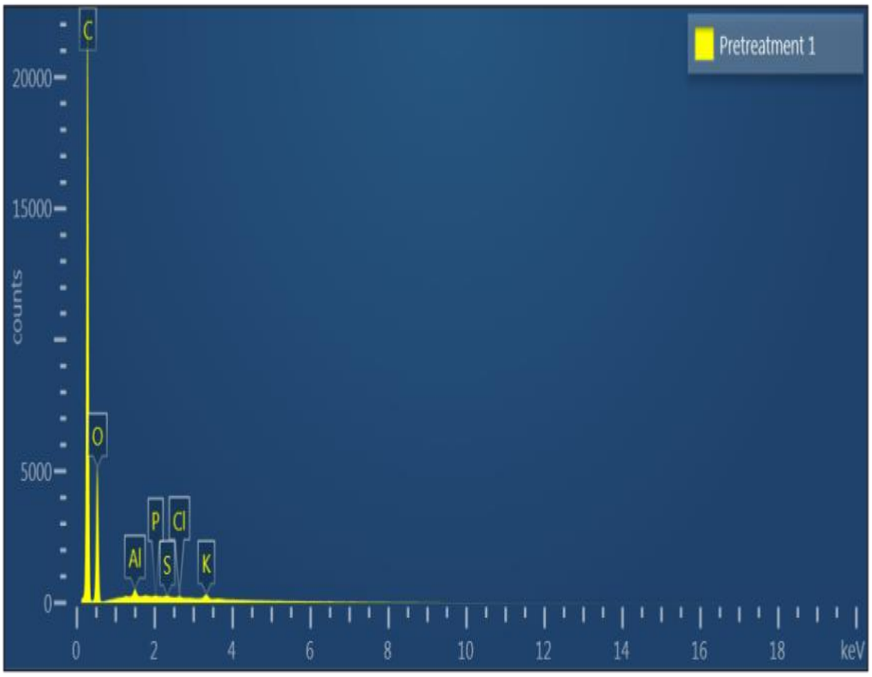


Figure 6. Elemental Composition of Coconut Dregs EDS Analysis

3.4. Fourier Transform Infra-Red (FT-IR)

Furthermore, to determine the functional groups contained in coconut dregs, observations were made using Fourier Transform Infra-Red (FT-IR). Generally, lignocellulose bands are very broad, making it difficult to distinguish peak (Pancholi *et al.*, 2023). The test results (Figure 7) revealed the presence of O-H, C-H, C=O, C-O, and other bonds. The high O-H (hydroxyl) indicates the presence of cellulose, hemicellulose, and lignin. Cellulose is indicated by the presence of -OH and C-O bonds; hemicellulose is indicated by the presence of C=O (aldehyde) bonds; and lignin is indicated by the number of C=C (aromatic ring), -O-CH3 (methoxyl), and C-O-C stretch bonds (Budiman *et al*., 2019).

Two main regions (region 1 (500–2000 cm1) and region 2 (2000–4000 cm1) show on FT-IR spectra in Figure 7. On Figure 7, Region 1 has a strong absorption spectrum; the absorbance spectra of 1316, 1242, 1148, 1058, and 1028 cm1 indicate cellulose. β-glycoside linkage of cellulose, with an absorbance spectrum around 1320–897 cm1 (Bhunia *et al*., 2023). In addition, FT-IR is also used to determine the crystallinity structure, namely at peaks of 700–1500 cm1 as a sensitive measure of crystalline cellulose structure (Vydrina *et al*., 2023). C-O stretching can be seen in the 950–1200 cm1 range (Pirah *et al*., 2022). Absorbance spectrum 1148 cm-1 indicated C-O-C stretching at the β-(1-4)-glycosidic linkages is cellulose, absorbance spectrum 1316 cm-1 indicated C-C and C-O skeletal vibration is cellulose, absorbance spectrum 1419 cm-1 indicated CH2 bending is cellulose, lignin, and hemicellulose, absorbance spectrum 2922 cm-1 indicated C-H stretching in methyl and methylene groups is cellulose, lignin, and hemicellulose, and absorbance spectrum 3318 cm-1 indicated H-bonded OH groups stretching (Maceda *et al*., 2020) is cellulose, lignin, hemicellulose (Pancholi *et al*., 2023).

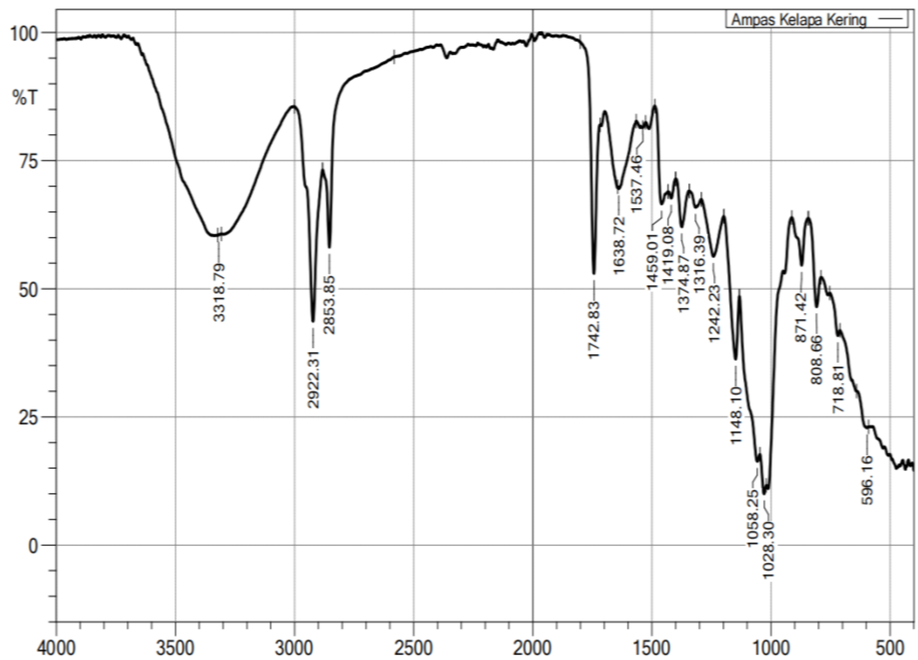


Figure 7. FT-IR Spectra of Coconut Dregs

3.5. *X-Ray Diffraction* (XRD)

One of the techniques in mineral analysis is X-Ray Diffraction (XRD), where the wavelength is almost equal to the distance between atoms in the crystal (between 0.5-2.5 Å). Electromagnetic radiation that has energy between 200 eV-1 MeV is also owned by X-rays. (Jamaluddin, 2016). XRD is an analytical technique that provides information on the atomic structure of substances, based on X-ray sample diffractions (Habibunnisa *et al.*, 2022). XRD was used to examine variations in the crystalline and amorphous areas of cellulose (Zhang *et al.*, 2022). According to XRD, there are numerous methods for calculating the crystallinity degree of a substance. Essentially, the diffractogram is conditionally separated into two sections (phases), with one crystalline and the other amorphous (Vydrina *et al.*, 2023). Figure 8. show that there are two prominent peaks: the first spectrum contains at $2\theta = 20.09^0$, which corresponds to the crystallography phase of cellulose-I, and the second at $2\theta = 16.01^o$, which corresponds to the presence of amorphous cellulose IV; both peaks exhibit monoclinic structures. These peaks are more noticeable when amorphous materials are present (Habibunnisa *et al.*, 2022). The size of the coconut dregs crystal obtained from XRD observations is 11.8 nm. Coconut dregs contains a fairly high cellulose component, but the crystallinity of the cellulose tends to be easily broken down by various treatments, both chemical, mechanical and biological.

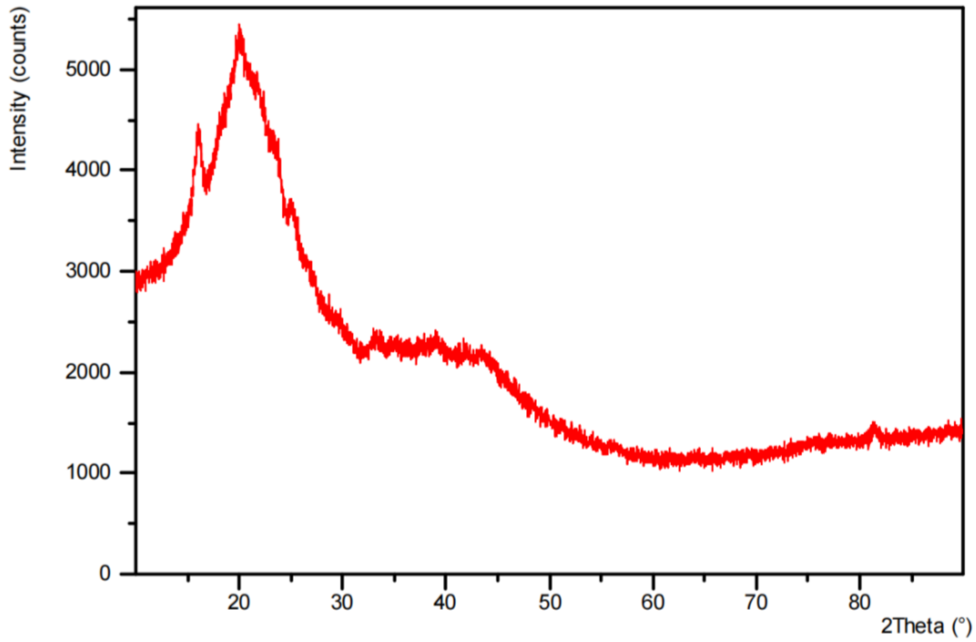


Figure 8. XRD Results of Coconut Dregs

4. Conclusions

Coconut dregs, a solid waste with abundant availability in Padang City, West Sumatra (in particular) produced 1.18 tons/day, has the potential to be utilized for its cellulose (47.18%), hemicellulose (12.10%) and or lignin (10.58%) fraction components. Based on XRD results, the size of the coconut dregs crystal obtained from XRD observations is 11.8 nm. The crystallinity of the lignocellulosic components in coconut dregs can be easily to breakdown by pretreatment, either physically, chemically or biologically. It is suggested that the utilization of lignocellulosic components in coconut dregs to produce environmentally friendly products should be done through bio-processing or environmentally friendly processes.

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