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Characterization of Sawdust Wastes as Precursor for Particleboard Production using Fourier Transform Infrared Spectroscopy and X-ray Diffraction

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Abstract

The extraction and disposal of wood chips, shavings from sawmills, or fine sawdust in developing countries with no established or sustainable methods is an increasing problem, frequently posing environmental challenges. Several furniture-making phases, from cutting to processing to polishing, produces a significant volume of sawdust of differing grades. Sawdust is still regarded as one of the most underused wood waste portions, thus, causing ecological concerns. This study uses NREL Standard Techniques, Fourier Transform Infrared (FTIR) Spectroscopy and X-ray Diffraction (XRD) to investigate the potentiality of sawdust as a sustainable precursor for particleboards. NREL Standard Technique is used to determine the chemical composition, FTIR spectroscopy analysis delved into the chemical and molecular structure and X-ray diffraction (XRD) revealed cellulose content and crystallinity. The main chemical components found are cellulose (53.87%), lignin (24.78%), hemicellulose (19.78%), ash (1.5%) and extractives (0.87%). Distinct peaks are seen at 2200 cm-1, 2000 cm-1, 1700 cm-1, 1300 cm-1, and 800 cm-1 in the FTIR spectrum of the sawdust. Cellulose, hemicellulose, and lignin have functional groups that line up with these peak values. The XRD analysis of the sawdust specimen demonstrated the presence of two significant peaks at diffraction angles (2θ°) of 15.6° and 22.4°, which correspond to the (101) and (002) lattice planes of cellulose I. The sharp and intense nature of these peaks suggest the high crystallinity of cellulose in the sample. Higher crystallinity levels can lead to improved particleboard strength and durability. The study establishes that sawdust, which is often a nuisance at sawmills, can be put into more beneficial use, as it has adequate compositions for quality properties necessary for particleboard manufacture.

1.0. Introduction

Biomass refers to living materials derived from plants, animals, and microorganisms, including algae and fungi, found in diverse environments across the Earth's surface [1]. Forest biomass residues primarily consist of cellulose, hemicellulose, and lignin, with proportions varying depending on the species. For terrestrial plants, biomass can be categorized into two components:

lignocellulosic and non-lignocellulosic materials. Lignocellulose comprises non-starch and the fibrous structural elements of plants, encompassing cellulose, hemicellulose, and lignin [2]. Non-lignocellulosic material primarily consists of low-molecular-weight cytoplasmic and structural components, including sugars, lipids, and proteins [3], along with inorganic molecules, ions, and a soluble fraction of lignin and cellulose [4]. In the context of energy consumption, contemporary society has increasingly aimed to reduce reliance on fossil fuels and mitigate greenhouse gas emissions. This has led forest operations and wood manufacturers to explore the production of biofuels and biomaterials as sustainable alternatives, fostering the advancement of renewable technologies and the bio-economy [5]. The wood industry continually embraces technological advancements to address environmental concerns [6], actively engaging in the recycling of industrial residues from wood processing and their conversion into value-added products. This aligns with the principles of the circular economy, emphasizing the preservation of product and resource value while minimizing waste generation [7]. This becomes especially crucial when considering the diverse array of sources for wood waste, ranging from timber harvesting and wood processing residues to construction and demolition waste, wood packaging, and even private households or railway construction. The primary components of particleboards are wood particles and adhesive, setting the raw materials used in particleboard production apart from other forms of wood composites. Common types of wood particles utilized for particleboard manufacturing include agricultural wood-wool, softwood, and medium-density hardwood shavings, flakes, chips, sawdust, strands, and fibers. Small-sized wood waste, such as sawdust and bark, poses particular challenges as residues [5, 8, 9, 10]. Additionally, certain characteristics, such as the low bulk density and combustibility of wood sawdust, raise concerns regarding logistics and storage. These economic and environmental considerations underscore the compelling need to repurpose this type of residual biomass and provide it with a second life. Figure 1 illustrates various key applications of sawdust.

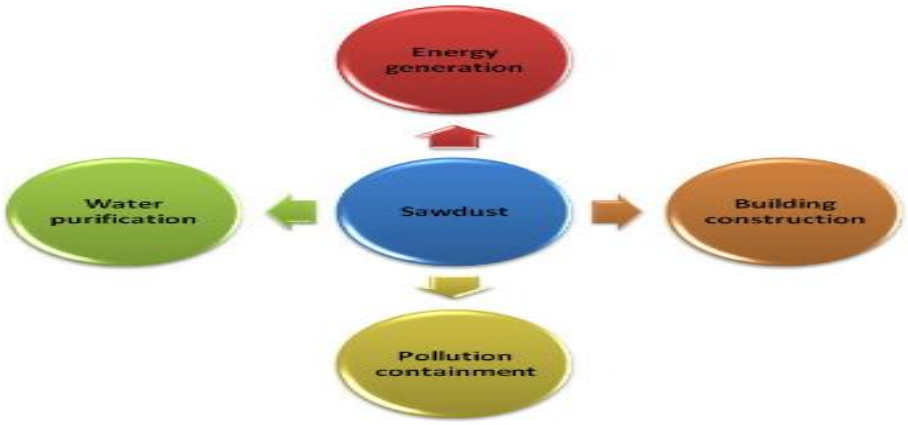

Figure 1: Primary Applications of Sawdust [11]

Nigeria produces around 1.8 million tonnes of sawdust and 5.2 million tonnes of wood waste annually [12, 13, 14]. Sawmills contribute significantly to this wood waste, with as much as 93 percent being in the form of sawdust [15]. Inappropriate disposal of waste wood has a substantial impact on both terrestrial and aquatic environments, with the added consequence of emitting greenhouse gases through wood waste burning, leading to various health issues. Repurposing and recycling these wood wastes can alleviate pressure on rapidly depleting forests, reduce pollution,

and generate employment and revenue. A sound sawdust management approach should be rooted in the three-pronged strategy of recovery, reuse, and recycling. The current study employs the Fourier Transform Infrared Spectroscopy (FTIRS) and X-ray diffraction (XRD) to characterize mahogany sawdust wastes aimed at ascertaining its use as a precursor for particleboard production.

2.0.Methodology

The sawdust was sourced from wood wastes obtained from commercial wood mills in the Ugbowo metropolis of Benin City, Edo state, Nigeria. The specie of woods in Benin, which is the geographical location where the sawdust was sourced is the Mahogany specie. Hence, the results from this single sawdust sample can be extrapolated to characterize other sawdust wastes species more generally by establishing a standard protocol for this particular characterization. The Mahogany Sawdust shown in Figure 2, was selected since it is environmentally sustainable in addition to being readily available and being less expensive.

Figure 2: (A) The biomass material (Mahogany sawdust)

The equipment used in the study are weighing balance, standard sieve, metal container, Steel caul plate, hammer mill, universal testing machine phenom Pro X scanning electron microscope, shimadzu fourier transform infrared spectrometer.

2.1 Material Preparation

The size of the dried feedstock was reduced by milling using a laboratory hammer mill followed by sieving to obtain 0.8 mm particles [16]. The starting saw dust material is characterized to determine its chemical composition. The raw sample was analyzed for structural carbohydrates (cellulose and hemicellulose), lignin, ash content, and extractives using the National Renewable Energy Laboratory's (NREL) standard techniques.

2.2 Characterization Approaches

2.2.1 Cellulose

The National Renewable Energy Laboratory (NREL) in the United States conducts compositional analysis on various materials, particularly biomass feedstock. Compositional analysis involves determining the chemical composition of a substance, and in the context of NREL, it often refers to analyzing the components of biomass samples. NREL employs various analytical techniques for compositional analysis on biomass to study its potential as a feedstock for bioenergy

production. This analysis helps in assessing the content of key components such as cellulose, hemicellulose, lignin, moisture content, extractives and ash content. The compositional analysis methodology at the National Renewable Energy Laboratory (NREL) typically involves a combination of wet chemical methods, instrumental techniques, and sometimes near-infrared spectroscopy (NIRS) for rapid analysis. Though, NREL continually refine and update their methodologies to improve accuracy and efficiency in assessing biomass composition, the following outlines the general methodology used for biomass compositional analysis: (i) Sample Preparation (ii) Moisture Content Analysis (iii) Extractives Analysis (iv) Ash Content Analysis (v) Cellulose, Hemicellulose, and Lignin Analysis (vi) Data Analysis (vii) Report the results of the compositional analysis (including the percentages of cellulose, hemicellulose, lignin, moisture, ash).

The amounts of cellulose and hemicellulose in each specimen were determined by quantitative acid hydrolysis of the extractive-free material, followed by the use of High-Performance Liquid Chromatography (HPLC) equipped with an Aminex HPX-87 P column and a refractive index (RI) detector. The methodology described by NREL was adhered to [16]. The total solid content (T_f) will be calculated initially using NREL techniques [18].

Weighed and put into a test container was a certain quantity of sawdust (0.3 g). The biomass's weight is shown by W_1. 4.92 g of 72% H_2SO_4 was mixed with the material in the test tube, and the plant matter was hydrolyzed for two hours at room temperature. Sugar recovery standards (SRS) were developed to make up for the sugars lost while hydrolyzed. After the breakdown of matter, the material was allowed to cool for about 20 minutes at room temperature before being diluted with deionized water to an acid content of 4%. Following calcium carbonate neutralization, the resultant solution was filtered and put through HPLC analysis to check for reducing sugars. To determine the amount of sugar in the sample, Equation (1) was used.

$$\% \text{ Sugar} = \frac{0.1 C_{corr} \times V_f}{W_1 \times T_f} \times 100\% \quad (1)$$

where:

W_1 = initial weight of sample

T_f = solid content in the initial sample

C_{corr} = concentration of sugar in hydrolyzed sample corrected for loss on hydrolysis, in mg/mL

The amount of cellulose was obtained from Equation 2 and 3 by using a correction factor of 0.90 for glucose (i.e. cellulose) and 0.88 for xylose (i.e. hemicellulose) as follows:

$$\% \text{ Cellulose} = \frac{0.9 \times 0.1 C_{corr} \times V_f}{W_1 \times T_f} \times 100\% \quad (2)$$

$$\% \text{ Hemicellulose} = \frac{0.88 \times 0.1 C_{corr} \times V_f}{W_1 \times T_f} \times 100\% \quad (3)$$

2.2.2 Determination of lignin

Following the quantitative acid hydrolysis procedure, the acid-insoluble and acid-soluble lignin concentration was determined. According to [17], the following approach was used. T_f was determined to be the total solid content. One gram of biomass, the predetermined amount, was measured and placed in a test tube. The sign W_1 stood for the amount of mass of the biomass. After

adding 15 milliliters of 72% H_2SO_4 to the material in the test tube, the biomass was hydrolyzed for two hours at room temperature. Deionised water (560 mL) was used to dilute the hydrolysate to 3% acid content. The resultant solution was filtered through a filtration anvil after being refluxed for four hours inside a condenser. A 10 mL portion of the resultant filtrate was used to determine the amount of acid-insoluble lignin. After cooling and weighing, the residue in the crucible was dried until a constant mass was reached at 105 ^oC. The weight that was acquired was labeled as W_1. The residue-containing crucible was heated to 575 ± 25 ^oC, cooled, weighed, and recorded as W_2 in order to account for acid-soluble ash. To determine the proportion of lignin in the biomass sample, Equation (4) was used.

$$\% \text{ Acid insoluble lignin} = \frac{W_1 - W_2}{W_i \times \frac{\%T_f}{100\%}} \times 100\% \quad (4)$$

where:
W_1 = weight of crucible + acid insoluble residue
W_2 = weight of crucible + ash
W_i = initial sample weight
T_f = solid content in the initial sample

Using the remaining filtrate from the acid insoluble lignin step, the amount of lignin that was soluble in acid was ascertained. The absorbance of the filtrate at 205 nm was measured using a UV-Vis mass spectrophotometer (T80 PG Instruments), with a 3% H_2SO_4 solution acting as a reference blank. To find the amount of acid soluble lignin in the initial biomass sample, Equation (5) was applied. The total amount of lignin in the sample was calculated by adding the amounts of lignin that were soluble and insoluble in acid.

$$\% \text{ Acid Soluble Lignin} = \frac{\frac{A}{b \times a} \times df \times V}{1000 \times W} \times 100\% \quad (5)$$

where:
A = absorbance at a wavelength of 205 nm
df = dilution factor
b = cell path length of 1 cm
a = absorptivity value of 100 L/(gcm)
V = filtrate volume, in mL
W = initial sample weight

2.2.3 Determination of extractives and acetyl groups

Ethanol extraction in a Soxhlet extraction equipment was used to determine the amount of extractives in the samples. According to [19], the following approach was used. First, the moisture content of raw sawdust samples was evaluated. Following that, 0.5 g of biomass was quantified and transferred into a 250 mL single-neck round-bottom reaction flask. The reaction flask was equipped with a distillation apparatus. Subsequently, a mixture of sodium methoxide (20 mL, 0.2N) and anhydrous methanol (40 mL) was introduced into the reaction flask through the graduated separatory funnel. To collect the distillate from the condenser, a 500 mL two-neck round-bottom flask placed in an ice bath was employed. 40 mL of anhydrous methanol was added

to the reaction flask via the funnel twice after the majority of the liquid in the flask had been distilled. 25 mL of 0.1 N NaOH was introduced through the side neck of the reaction flask after the majority of the liquid had been distilled, and the flask was then quickly sealed with a glass stopper. After being taken out of the ice bath, the round-bottom flask was heated for 20 minutes under reflux in a hot water bath. After the flask had cooled to room temperature, 50 mL of phenolphthalein indicator was added. After that, the solution was titrated with 0.1 N HCl until it lost color, and the volume of HCl used was noted. To get the biomass's acetyl content, Equation (6) was applied. Moreover, 0.1 N HCl was used to titrate a blank that contained solely methanol.

$$\% \text{ Acetyl content} = \frac{\Delta V \times N \times 0.043}{W} \times 100 \quad (6)$$

where:
ΔV = mL of HCl for blank – mL of HCl for sample
N = normality of HCl solution
W = dry weight of sample

2.2.4 Determination of ash content

The ash content of raw samples was determined using the methods outlined by [20]. An empty crucible with its lid was positioned in a muffle furnace set at 600 °C for four hours. Subsequent to extraction from the furnace, the crucible was transferred to a desiccator and allowed to cool for an hour. Two grams of the material to be analyzed, previously weighed, were placed into the cooled crucible. The crucible, along with the sample, was then weighed and recorded after being positioned in a drying oven set at 105 °C until a constant weight was achieved. Once again, after extraction from the furnace, the crucible was placed in a desiccator to cool for an hour. After placing the crucible and its contents into the muffle furnace, they were heated to 600 °C in order to eliminate all of the carbon. After being removed from the furnace, the crucible was placed in a desiccator to cool for an hour. Until the weight of the crucible plus the sample remained consistent, the heating procedure was repeated three times. To prevent burning and protect the crucible from strong drafts and sample loss through mechanical means, the sample was heated to a low temperature. After being removed from the furnace, the crucible was placed in a desiccator to cool for an hour. The following calculations were made using Equation (7) to determine the sample's ash content.

$$\% \text{ Ash content} = \frac{W_2}{W_1} \times 100 \quad (7)$$

Where: W_1 = Weight of ash (grams), W_2 = weight of oven-dry sample (grams)

2.2.5 X-ray diffraction analysis of the sawdust

Using a Philips diffractometer fitted with Cu-Kα radiation, the particleboard precursor (sawdust) crystalline phase were evaluated. By comparing the intensity and diffraction lines of the XRD machine's patterns to the Joint Committee on Powder Diffraction Standards (JCPDS), the patterns were however, interpreted.

2.2.6 FTIR analysis of feedstock

The chemical structures of the sawdust were determined through the Fourier transform infrared spectroscopy. This was accomplished by employing a Shimadzu FT-IR spectrometer to generate the biomass infrared spectra. The 400–4000 cm–1 scan range was used for the sample.

3. Results and Discussion

3.1 Chemical Composition Analysis of Feedstocks

Table 1 shows the sawdust's chemical composition, highlighting its properties as feedstock for the manufacturing of particleboard.

Table 1: Chemical composition analysis of the feedstock

Component	**Sample (%)**
	Sawdust
Cellulose (%)	53.87
Hemicellulose (%)	19.78
Holocellulose (%) (Cellulose + Hemicellulose)	73.65
Lignin (%)	24.78
Extractives (%)	0.87
Ash content (%)	1.57

The makeup of the sawdust sample utilized in this investigation is displayed in Table 1. The main ingredients were lignin (24.78%), hemicellulose (19.78%), and cellulose (53.87%). Insignificant amounts of ash (1.5%) and extractives (0.87%) were present. This composition provides insights into the capacity of sawdust as an initial ingredient for making particleboard and maybe as a means to synthesize particles. Sawdust's large amount of cellulose is advantageous for the manufacture of particleboard. This, as previously reported, is because cellulose acts as a natural binder, providing structural strength and stability to the boards. Thus, with its high cellulose content, sawdust shows great promise as a suitable material for particleboard production, creating durable and robust boards for various applications. The hemicellulose content of sawdust can help to reduce dimensional instability in the resulting particleboard which makes it less susceptible to moisture-induced issues, ensuring long-lasting and high-quality products. In addition, in the context of particleboard production, the low extractives content in sawdust has an important advantage. Low extractives content mean that the particleboard will not suffer from poor adhesion between the sawdust particles and the binder, ensuring strong bonding between particles, contributing to the overall quality of the particleboards. The low ash content recorded is also desirable for particleboard production. To assess the suitability of sawdust for particleboard production, Fourier Transform Infrared Spectroscopy (FTIR) and X-ray Diffraction (XRD) are commonly used techniques. These methods provide information about the chemical composition and crystalline structure of the sawdust, which are crucial factors in determining its suitability for particleboard manufacturing.

3.2 Fourier Transform Infrared Spectroscopy of Feedstocks

The FTIR is used to identify functional groups and chemical bonds present in the sawdust. The distinct peaks seen at 2200 cm-1, 2000 cm-1, 1700 cm-1, 1300 cm-1, and 800 cm-1 in the FTIR spectrum of the sawdust mixture used in this experiment are displayed in Figure 3. The three primary components of the sawdust, which are cellulose, hemicellulose, and lignin, have functional groups that line up with these peak values.

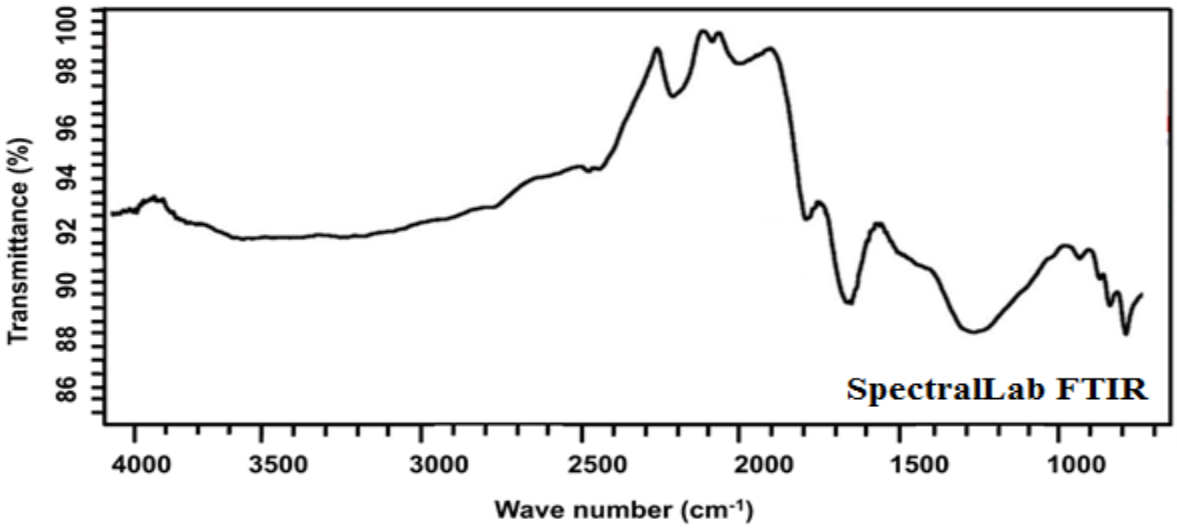

Figure 3: FTIR spectra of sawdust

Sawdust's lignin concentration is associated with the strong peak detected at 2200 cm-1, which was attributed to the stretching vibrations of C-C triple bonds [20]. As mentioned earlier, lignin constitutes a significant portion of woody biomass such as sawdust, characterized by its composition of aromatic rings containing the C≡C bonds. The amount of lignin in the sample can be determined by measuring the strength of this peak. [22] linked the presence of nitrile groups in the hemicellulose fraction of sawdust to the peak found at 2000 cm-1, which was attributed to the stretching vibrations of C-N triple bonds. As observed previously, hemicellulose is a complex polysaccharide that can contain different functional groups, such as nitrile groups. The intensity of this peak as seen in Figure 3 gives an idea of the hemicellulose content of the sawdust. A major peak was also identified at 1700 cm^{-1}, and this is consistent with the C=O bond's expanding vibration. The C=O bond suggests the presence of carbonyl groups in both cellulose and lignin. It is known that cellulose contains acetyl groups, which have carbonyl functionalities, hence the functional group identified at this peak. Furthermore, because lignin's structure contains carbonyl groups, it may possibly contribute to this peak. The intensity of this peak, as indicated in Figure 3, can also be used to estimate the sawdust sample's total cellulose and lignin content. The presence of ether groups in the cellulose and hemicellulose fractions was demonstrated by the peak at 1300 cm-1, which has been connected to the vibrations that stretch of C-O bonds. According to [23], cellulose is made up of glucose units joined by β-1,4-glycosidic linkages with ether functionalities. Hemicellulose, with a variety of structures, can contribute to this peak through its ether groups. The strength of this peak can reveal information about the combined cellulose and hemicellulose content of sawdust. Lastly, the peak at 800 cm^{-1}, was ascribed to the bending vibrations of C-H

bonds, which was attributed to the aliphatic hydrocarbons present in both lignin and hemicellulose. Lignin contains aliphatic side chains, while hemicellulose can also contain acetyl groups with aliphatic hydrogens. Previous studies have also shown similar FTIR spectra for sawdust [24, 25, 26].

3.3 X Ray Diffraction Analysis of Feedstocks

Figure 4 displays the findings of the XRD analysis of the sawdust specimen utilized in this investigation. The data obtained demonstrated the presence of two significant peaks at diffraction angles (2θ°) of 15.6° and 22.4°, which correspond to the (101) and (002) lattice planes of cellulose I. A significant percentage of sawdust, cellulose I is crucial to the manufacturing of particleboard.

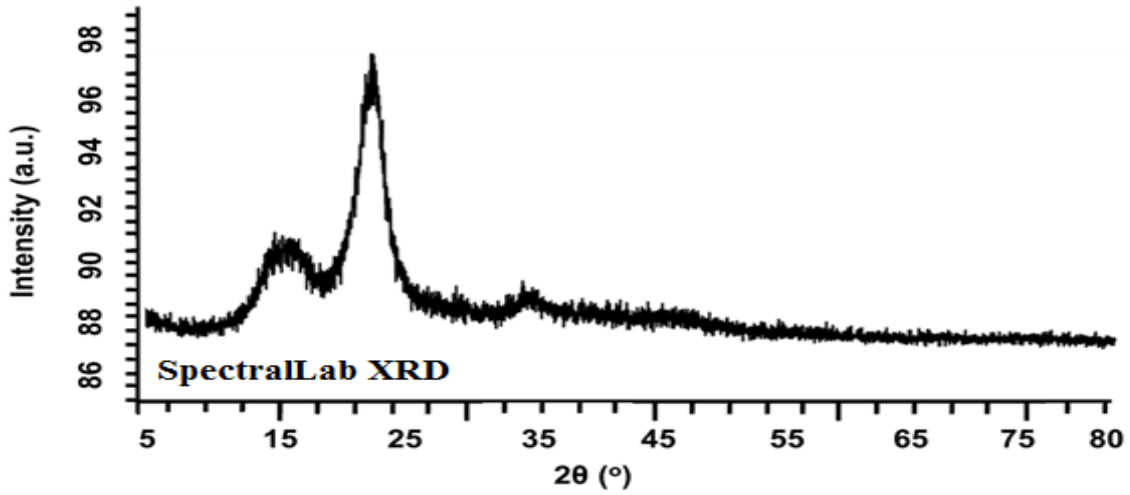

Figure 4: XRD pattern of sawdust

The presence of the (101) and (002) peaks confirm the abundance of cellulose in the sample, essential for its potential as a natural binder in particleboard production. Specifically, the (101) peak corresponds to the periodic stacking of cellulose chains within the sawdust. The sharp and intense nature of this peak suggests the high crystallinity of cellulose in the sample. Higher crystallinity levels can lead to improved particleboard strength and durability. In the same vein, the (002) peak describes the periodic arrangement of cellulose chains along the (002) plane. This peak corroborates the crystalline nature of cellulose in the sawdust specimen. Crystalline cellulose has a stronger binding capacity, contributing to better interlocking and adhesion between the particles during particleboard formation. Previous studies have also observed similar XRD patterns for sawdust [27, 28, 29].

4. Conclusion

This study aimed at characterizing sawdust as a precursor, to study its potentiality to be used as an engineered wood product (particleboard). The FTIR spectra of sawdust provided valuable insights into their composition, indicating their potential for particleboard production. The results, detailed in Table 1, support observations on the chemical composition of this material. The hemicellulose and lignin composition of sawdust present both advantages and challenges, highlighting the need for effective pretreatment processes to ensure optimal board quality. Similarly, XRD results for

sawdust revealed essential factors for particleboard manufacture, such as cellulose content and crystallinity. Strong cellulose I peaks at 15.6° and 22.4° were observed in sawdust, contributing to particleboard strength and durability. The study's conclusions are drawn from these findings. Sawdust composition analysis indicated its suitability for particleboard production. The analysis unveiled compositions which can influence the characteristics and quality of the final particleboard. Understanding sawdust composition allows for optimal particleboard production, promoting the development of eco-friendly and sustainable building materials. In conclusion, the composition suggests that sawdust is a viable choice as a raw material for manufacturing particleboards, contributing to their competitiveness in the global market.

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