

SYNTHESIS OF LIGNOSULFONATE AS A BINDER EXTENDER IN PARTICLE BOARD MANUFACTURE

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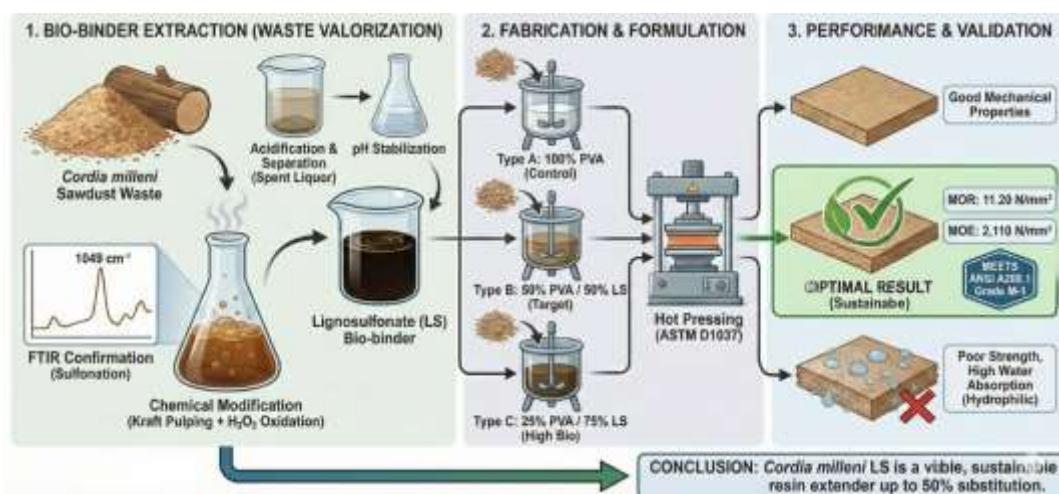
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Abstract:

Synthetic adhesives are currently in high demand in the particleboard industry and these adhesives are usually expensive and non-renewable, creating environmental concerns. This has necessitated the development of sustainable bio-derived alternatives such as lignosulfonates (LS) from agro-wastes which are abundant. Lignosulfonate is a polymer that can be used in many industries as concrete additives, wood preservatives, animal feed and oil well drilling. In this work, lignosulfonates were produced from lignin extracted from *Cordia millenii* (Omo wood) sawdust as a partial substitute of synthetic Polyvinyl Acetate (PVA) resin in the manufacture of particleboards. *Cordia millenii* sawdust was pulped using modified (0.375M) NaOH process at 120°C for 120mins. Afterwards, lignin was precipitated from the spent liquor by acidification and then separated. FTIR spectroscopy was used to confirm the production of the lignosulfonate. The isolated lignin was activated by hydrogen peroxide oxidation and the pH-stabilised to activate the binder. Three formulations of the particle boards were produced and tested per ASTM D1037: Type A (100% PVA), Type B (50% PVA/LS), and Type C (25% PVA/75% LS). FTIR spectroscopy confirmed the presence of lignosulphonate with a clear peak at 1049 cm⁻¹. The Type B met ANSI A208.1 Grade M-1 standards with a Modulus of Rupture (MOR) of 11.20 N/mm² and Modulus of Elasticity (MOE) of 2,110.00 N/mm². While a 50% substitution proved feasible, higher bio-binder levels reduced strength and increased water absorption due to sulfonate hydrophilicity. The study concludes that *Cordia millenii* lignosulfonate is a viable resin extender up to 50%.

Key words: Bio-based adhesives, *Cordia millenii* sawdust, Lignosulfonate, Particleboard, Polyvinyl Acetate.

Graphical Abstract



INTRODUCTION

Urbanisation and the increasing demand for cheap building materials have resulted in a steady increase in the demand of the wood-based panel industry in the world (FAO 2023). Particleboards form about 57% of the total world use of these panels, which are also used as key materials in furniture production, interior cabinetry and floor underlayment (Eshraghi *et al.* 2012). Today, synthetic petrochemical resins are largely used in the manufacture of these composites. These binders are usually between 8 and 15% of the total mass

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of the board (Antov *et al.* 2020). Synthetic resins have some benefits like quick curing and good adhesion however, like the fossil fuels, they are not renewable. The environmental issues surrounding the extraction and processing of these petrochemicals are also of high concern due to carbon emissions and high cost of production which is subject to the global fluctuations of oil prices. Accordingly, the world is in urgent need to come up with green, bio-based adhesives that can lower the carbon footprint and dependence on fossil fuel by the industry (Kamau-Devers *et al.* 2020).

Lignin, the second-largest natural polymer after cellulose, has become a promising candidate in this search (Aristri *et al.* 2021; Gadhav *et al.* 2023). Lignin is a natural adhesive in woody biomass, which gives structural rigidity and stability to plant fibres (Abdullah *et al.* 2021). Technical lignin which is a by-product of pulp and paper industry is mostly underutilised despite its abundance. The industry is estimated to generate nearly half a billion tons of lignin each year, but only approximately 2% is used in a commercialised manner, with the rest being burned to produce low-value energy output (Dessbesell *et al.* 2020). Recycling this "spent liquor" to high-value uses is consistent with the concept of the circular economy and lignosulfonates are anionic polymers that are soluble in water and thus can be used in adhesive applications. They have an unusual amphiphilic structure, which consists of a hydrophobic aromatic and hydrophilic functional groups, such as sulfonic acid (SO_3^-), and phenolic hydroxyl ($-\text{OH}$) groups (Gordobil *et al.* 2021). It is through this chemical structure that they can be used as surfactants and binders that can form good interfacial relationships with wood particles. However, the native lignin is not always reactive to the extent that it can be utilised in industries. The most recent innovations in green chemistry have focused on the lignosulfonates adjustment, such as oxidative activation of lignosulfonates by hydrogen peroxide (H_2O_2) to increase the number of active sites and enhance their ability to crosslink with synthetic resins (Zhou *et al.* 2021).

In addition to the technical lignin of the pulp industry, huge amounts of lignin are available in the by-products of mechanical wood processing. Timber industry produces huge amounts of lignocellulosic residues each year, with less than 60 percent of the log volume typically being converted to sawn timber, with the rest being in the form of sawdust, off-cuts, and shavings (Hassan *et al.* 2023). Traditionally, such residues are regarded as low-value waste and are often burned or buried on land, which causes pollution of the environment and greenhouse gases (Zhang *et al.* 2024). Nevertheless, this biomass is a valuable renewable source of native lignin. The conversion of these agro-industrial residues into lignin extraction is also in line with the principles of the circular economy, and the potential environmental hazard can be converted into a sustainable source of bio-adhesives (Tripathi *et al.* 2022).

In this study, the focus is on the valorisation of wastes of *Cordia millenii* (Omo wood), a tropical hardwood species that is rich and commercially processed in Nigeria. Mechanical treatment of this timber generates masses of sawdust, which are normally disposed of or burned, and pollutes the environment (Santos *et al.* 2019). In this research, modified Soda pulping process will be utilized in extracting and activating lignosulfonate to serve as a bio-binder using *Cordia millenii* sawdust. The intended aim is to examine the mechanical and physical properties of the manufactured particleboards with the help of a hybrid binder system, in which lignosulfonate is used partially as a substitute of Polyvinyl Acetate (PVA). The aim of this strategy is to identify an ideal level of substitution that is both ecologically sustainable and meets the high mechanical quality standards needed for industrial applications, and in effect, the local timber waste will be empowered in the production of a functional and an eco-friendly construction material.

MATERIALS AND METHOD

Materials

The sawdust residues of *Cordia millenii* were obtained at Bodija Sawmill, Ibadan, Nigeria. The sawdust was screened to obtain coarse (2mm sieve) and fine (0.5mm sieve) particles. Polyvinyl Acetate (PVA) resin of commercial grade was purchased to be used as the control adhesive and co-adhesive. Chemical processing was performed using analytical grade Sodium Hydroxide (NaOH), Sulfuric Acid (H_2SO_4), and Hydrogen Peroxide (H_2O_2).

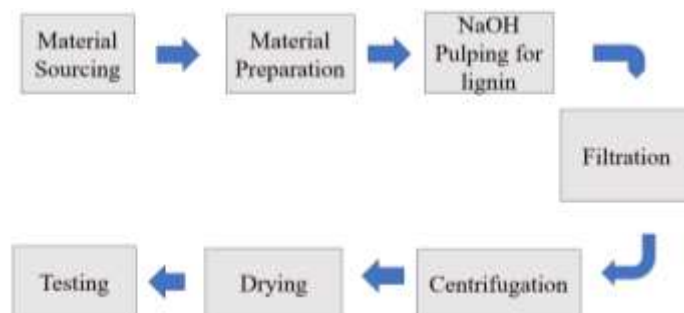


Fig. 1.
Flow chart of the methodology.

Characterisation of Raw Materials

The ultimate analysis of the elemental composition (Carbon, Hydrogen, Nitrogen, and Sulphur) of the *Cordia millenii* sawdust was carried out using PerkinElmer 2400 Series II CHNS/O Elemental Analyzer. The samples were heated in high temperatures and the gaseous products of combustion were tested to give the percent weight of each element. The Oxygen content was subsequently calculated by difference.

Lignin Extraction and Activation

The extraction of lignin was done by a modified version of the NaOH pulping process by first digesting 100 grams of screened sawdust, adopted from Akintola *et al.*, (2024). A solution that contained the sawdust was added to an alkaline cooking solution, which was made by dissolving 15g of Sodium Hydroxide (NaOH) in distilled water and heated at 120°C, a period of 120 minutes, using a chemical reactor constructed by Kolajo and Onilude (2016). After the digestion, the black liquor that had a pH of about 11 was filtered to eliminate the fibrous residue. To extract the lignin, the black liquor was acidified with the Sulfuric Acid (H₂SO₄) until the pH was reduced to 2. This acidification protonated the phenolic groups causing the precipitation of the lignin (Gao *et al.* 2023). The mixture was then subjected to a purification step to remove the traces of hemicellulosic sugars by fermentation of the acidified solution using *Saccharomyces cerevisiae* (baker's yeast) over a period of 24 hours. Separating the resultant precipitate was then done through centrifugation. Lastly, the produced lignosulfonate was activated oxidatively by using hydrogen peroxide in an alkaline condition which helped to enhance the number of reactive functional groups that would be useful in cross linking. This method was adapted from Balea *et al.*, (2022).

Chemical analyses of the Lignosulfonate

Characterisation of the produced binder was done to confirm that the substance is structurally stable and can be used as an adhesive. The Fourier Transform Infrared Spectroscopy (FTIR) using a PerkinElmer Spectrum Two™ spectrometer equipped with a UATR Two accessory was utilised to confirm the presence of the molecular fingerprint of the modified lignin, namely, the identification of sulfonate (SO₃⁻) and hydroxyl (-OH) functionalities. This spectral analysis played an important role in validating that the lignin backbone has been successfully introduced to hydrophilic functional groups that are critical in making the lignin water soluble and hence cross-linking.

Particleboard production

The particleboards, with a density ranging between 533.3 and 573.3 kg/m³, were produced by mixing coarse and fine particles in a ratio of 35:65, and the adhesive loading was 15% of the total weight of the particles (Gul *et al.* 2024). Three different formulations of binders were prepared to test the impact of the lignosulfonate replacement: Type A, which was the control group, was 100% Polyvinyl Acetate (PVA); Type B was a mixture of 50% PVA and 50% Lignosulfonate (LS); and Type C was a mixture of 25% PVA and 75% LS. After blending, the particles were coated with resin and moulded into mats in triplicates, which were subjected to a hot press at 180°C and 2.5 MPa pressure for 16 minutes. For stability, the resultant boards were conditioned at 20°C and 65% relative humidity over a period of seven days prior to testing.

Test Procedure on the Particle Board

Physical Properties

The physical stability of the particleboards was also tested for Water Absorption (WA) and Thickness Swelling (TS) according to the ASTM D1037-12. The test specimens were prepared to standard specification, weighed, and their thickness taken before being immersed. The samples were then immersed in room temperature distilled water. Short-term and long-term moisture resistance were measured after 2 hours and 24 hours, respectively.

$$\text{Water Absorption (\%)} = \frac{W_f - W_i}{W_i} \times 100 \quad (1)$$

where:

W_i = Initial dry weight (g), W_f = Wet weight after immersion (g)

$$\text{Thickness swelling (\%)} \text{ is given by } = \frac{T_f - T_i}{T_i} \times 100 \quad (2)$$

where:

T_i = initial thickness, T_f = Thickness after immersion

Mechanical Properties

The test of mechanical performance was conducted using a Universal Testing Machine (UTM) (Wincom WA-1000B Digital Display Hydraulic Universal Testing Machine, Wincom Company Ltd.) at constant cross-

head speed as per the ASTM D1037-96 standards. To determine the Modulus of Rupture (MOR) and the Modulus of Elasticity (MOE) of the boards, the three-point bending test was used. Also, the energy absorption of the dynamic loading boards was measured on the basis of Charpy Impact Test (ASTM D6110). In this test, the specimen was exposed to a pendulum strike and the energy consumed in the fracture was measured so as to determine the Impact Strength.

$$\text{MOR} = \frac{3PL}{2BH^3} \quad (3)$$

$$\text{MOE} = \frac{PL^3}{4BH^3Y} \quad (4)$$

where:

P = load (N), L = length (m), B = width (m), H = height (m), Y = deflection (m)

RESULTS AND DISCUSSION

All values presented in this section represent the mean of three replicates ($n=3$) \pm standard deviation (SD), unless otherwise stated.

Characterisation of Raw Materials

Ultimate Analysis

Table 1 shows the elemental composition of *Cordia millenii* sawdust. The content of carbon was measured as 48.50%, which falls within the range of 49.54–50.98% of the same tropical hardwoods by Dadile *et al.* (2020). This high organic carbon content albeit slightly lower makes it clear that the biomass is suitable in the production of lignosulfonate, and the hydrogen content of 6.10% is just within the standard literature range of 4.00 - 6.50% observed in the same study. Compared to a second study, the amounts of nitrogen (0.35%) and sulphur (0.08%) are remarkably low and agree with the results of Onochie *et al.* (2024) who found ranges of 0.03 - 0.24% of sulphur in clean biomass. This low level of heteroatoms is noteworthy since it shows that the thermal treatment of this sawdust will produce little NO_x and SO_x gases, which proves that it is an eco-friendly feedstock in contrast to polymers based on fossil fuels.

Table 1

Ultimate analysis of <i>Cordia millenii</i> sawdust		
Property (%)	Measured Value	Literature*
Carbon	48.50	47.00 – 51.00
Hydrogen	6.10	5.80 – 6.50
Nitrogen	0.35	0.10 – 0.50
Sulphur	0.08	< 0.10
Oxygen	44.97	40.00 – 45.00

* Dadile *et al.* (2020) and Onochie *et al.* (2024).

Lignosulfonate Binder Yield

The yield of the production process was measured using the mass balance. Out of the 100 g of screened sawdust, the soda pulping and acid precipitation process was modified to obtain 14.20 g of lignin that was precipitated. The oxidative activation step was followed by a high yield of functionalized lignosulfonate of 15.65 g. This is a final conversion yield of 15.65 percent on the raw biomass feedstock, which is a good comparison with the conventional soda pulping yields on hardwood species (Saber *et al.* 2020; Gao *et al.* 2023).

FTIR Analysis:

The spectra obtained from the lignosulphonate is as detailed in Table 2.

Table 2

FTIR Spectral Assignments for extracted Lignosulfonate Binder		
Wavenumber(cm ⁻¹)	Functional Group	Significance in Binder Performance
3341.08	O-H Stretching (Phenolic and Aliphatic Hydroxyls)	Primary sites for hydrogen bonding crosslinking with resin
1634.69	C=C Aromatic Ring Vibrations (Skeletal Stretching)	Confirms the preservation of the core lignin structure during extraction
1513.25	C=C Aromatic Ring Vibrations (Skeletal Stretching)	Further validation of the characteristic hardwood lignin structure.
1049.10	S=O Stretching (Sulfonate Group SO ₃ ⁻)	Confirmation of Sulfonation. Proves the lignin is now water-soluble and active.

The spectrum of the produced lignosulfonate showed a broad band at 3341 cm⁻¹ (hydroxyl groups) and aromatic skeletal vibrations at 1634 cm⁻¹. Crucially, a distinct peak at 1049 cm⁻¹ (S=O stretching) confirmed the successful sulfonation of the lignin (Hemmilä *et al.* 2017, Suryani *et al.* 2024).

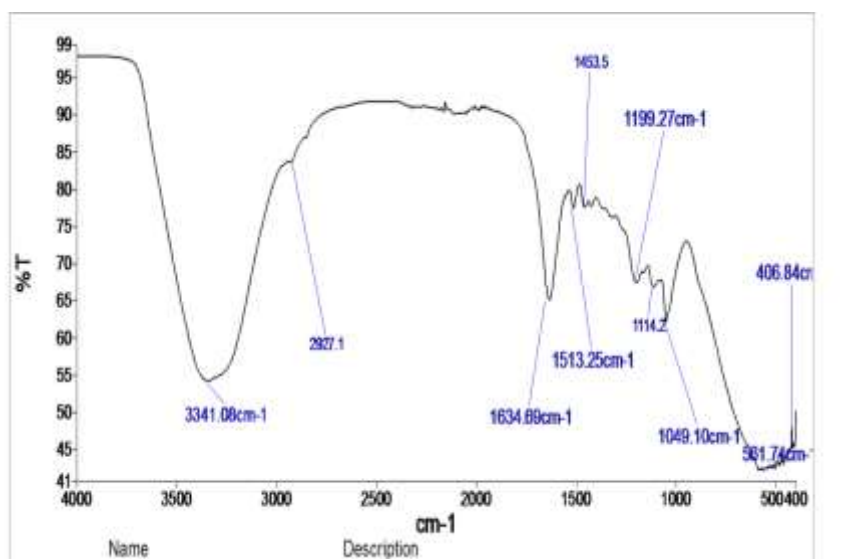


Fig. 2.
FTIR spectrum of the extracted and activated lignosulfonate binder.

Physical Properties

Table 3

Physical Properties of Particleboards (mean ± SD, n = 3)

Formulation	Density (kg/m ³)	Specific Gravity	WA 2h* (%)	WA 24h* (%)	TS 2h* (%)	TS 24h* (%)
Type A (100% PVA)	573.3	0.57	22.0 ± 0.35	55.8 ± 0.52	3.5 ± 0.17	8.5 ± 0.17
Type B (50% PVA / 50% LS)	560.0	0.56	18.0 ± 0.26	45.2 ± 0.36	3.2 ± 0.17	7.2 ± 0.17
Type C (25% PVA / 75% LS)	533.3	0.53	33.0 ± 0.35	82.5 ± 0.36	4.3 ± 0.17	12.1 ± 0.17

*Mean ± SD, WA = Water Absorption; Thickness Swelling; LS = Lignosulfonate

The particleboards had a density of between 533.3 and 573.3 kg/m³, falling within the conventional classification of medium-density particleboard. There was a slight drop in density with increasing lignosulfonate content (Type A to Type C), indicating that the bio-binder forms a slightly less compact structure compared to conventional synthetic resin. This tendency is consistent with reports by Antov *et al.* (2020), who reported that the slower curing and rheological diversity of lignosulfonate adhesives tend to produce a spring-back effect during hot pressing, preventing maximum densification compared to standard PVA resin.

For water absorption, the 50% substitution (Type B) recorded the lowest 2-hour absorption of 18.0 ± 0.26%, and 24-hour absorption of 45.2 ± 0.36%, outperforming the 100% synthetic control (Type A: 22.0 ± 0.35% at 2h; 55.8 ± 0.52% at 24h). This suggests a synergistic effect whereby the lignosulfonate fills voids within the composite matrix, reducing porosity and limiting moisture intake. This mechanism corresponds with observations by Kluska *et al.* (2018), who reported that lignin additives can enhance dimensional stability by physically blocking moisture pathways in the wood network when used in moderate proportions. However, at 75% substitution (Type C), water absorption increased significantly to 33.0 ± 0.35% at 2 hours and 82.5 ± 0.36% at 24 hours, indicating that excessive lignosulfonate introduces more hydrophilic sulfonate groups which, according to Hemmilä *et al.* (2017), cause hygroscopicity to rise rapidly once bio-binder content passes a critical limit.

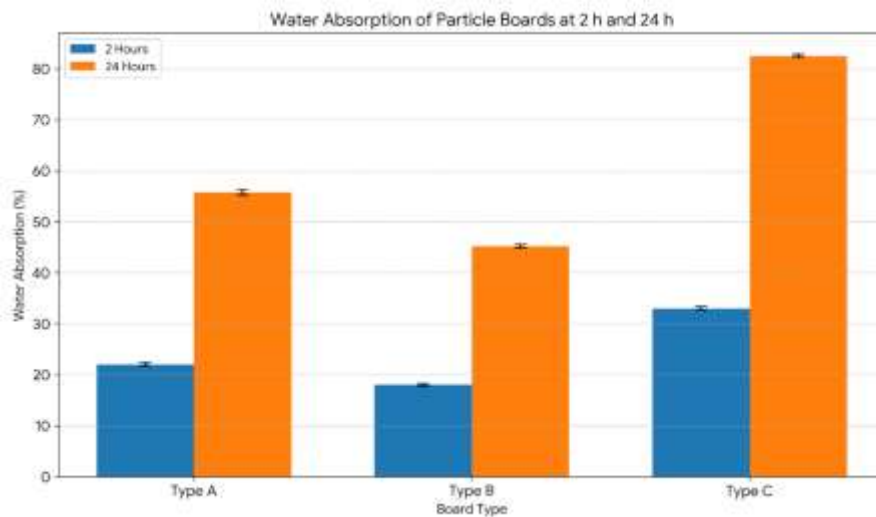


Fig. 3.
Bar chart showing percentage rise in water absorption at 2h and 24h.

Thickness swelling followed a similar pattern. At moderate substitution, Type B recorded the lowest swelling of $3.2 \pm 0.17\%$ at 2 hours and $7.2 \pm 0.17\%$ at 24 hours, compared to Type A ($3.5 \pm 0.17\%$ at 2h; $8.5 \pm 0.17\%$ at 24h). This improvement indicates that liginosulfonate effectively penetrates the wood-PVA interface and reduces void spaces. However, increasing the substitution to 75% (Type C) led to a significant increase in swelling to $4.3 \pm 0.17\%$ at 2 hours and $12.1 \pm 0.17\%$ at 24 hours, demonstrating that while moderate addition stabilizes the composite, excessive liginosulfonate compromises internal bond integrity under wet conditions. This aligns with findings by Lubis *et al.* (2019), who attributed higher swelling rates in liginin-rich composites to the hygroscopic nature of excess sulfonate groups and the subsequent release of compressive stresses within the board.

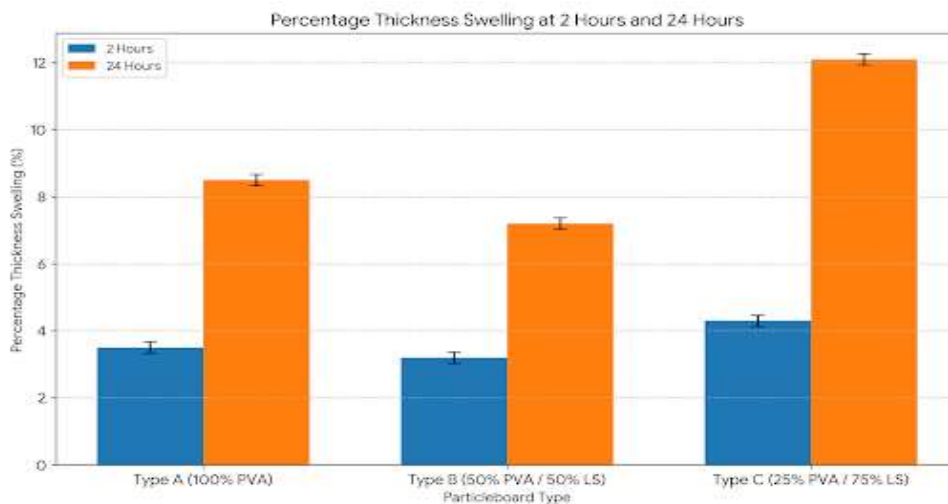


Fig. 4.
Bar chart showing percentage rise in thickness swelling at 2h and 24h.

Mechanical Properties (MOR & MOE)

Table 4

<i>Mechanical Properties of Particleboards</i>			
Formulation	Impact Strength (KJ/m ²)*	MOE (N/mm ²)*	MOR (N/mm ²)*
Type A (100% PVA)	4.25 ± 0.06	2450 ± 26.0	12.5 ± 0.17
Type B (50% PVA / 50% LS)	3.60 ± 0.05	2110 ± 21.4	11.2 ± 0.17
Type C (25% PVA / 75% LS)	2.85 ± 0.05	1600 ± 18.8	9.6 ± 0.17

*Mean ± SD, MOE = Modulus of Elasticity; MOR = Modulus of Rupture.

Impact strength is the capacity of the board to withstand shock without breaking. The control board (Type A) recorded the highest value of 4.25 ± 0.06 KJ/m². Partial replacement with lignosulfonate reduced impact strength to 3.60 ± 0.05 KJ/m² (Type B) and 2.85 ± 0.05 KJ/m² (Type C). The small standard deviations across all three formulations confirm the consistency and reliability of the measurements. The reduction in impact strength indicates that the bio-binder forms a more brittle internal structure that cannot resist physical impacts as effectively as the synthetic control. This is in line with findings by Lubis *et al.* (2019), who found that the stiff aromatic structure of lignin limits polymer chain mobility when mixed with more ductile resins like PVA, thereby increasing brittleness and reducing impact resistance.

Modulus of Elasticity (MOE) represents the rigidity of the board and its ability to resist bending. Type A was the stiffest at $2,450 \pm 26.0$ N/mm², followed by Type B at $2,110 \pm 21.4$ N/mm², and Type C at $1,600 \pm 18.8$ N/mm². Despite reduced stiffness, both Type A and Type B exceeded the ANSI A208.1 minimum standard of $1,550$ N/mm², confirming their commercial viability. The consistent standard deviations across replicates indicate reliable bonding during manufacture. The observed decrease in MOE with increasing bio-binder content is consistent with findings by Antov *et al.* (2020), who attributed reduced rigidity to the lower cross-linking density of lignosulfonate compared to synthetic resins, resulting in a more flexible but less stiff polymer network within the composite matrix.

Modulus of Rupture (MOR) represents the maximum load the board can sustain before breaking. Flexural strength decreased from 12.5 ± 0.17 N/mm² (Type A) to 9.6 ± 0.17 N/mm² (Type C). The 50% substitution (Type B) achieved 11.2 ± 0.17 N/mm², exceeding the ANSI A208.1 minimum of 10.0 N/mm². This is a significant result, confirming that half the synthetic resin can be replaced with *Cordia millenii* lignosulfonate without breaching the structural failure limit of the board. The tight standard deviations across all replicates further validate the reproducibility of this finding. This performance is consistent with the observations of Lubis *et al.* (2019), who reported that although technical lignin generally exhibits lower reactivity than pure synthetic resins, lignin–PVA blends are capable of maintaining adequate interfacial bonding and efficient load transfer at moderate substitution levels.

CONCLUSION

In this study, lignosulfonate binder using *Cordia millenii* sawdust was successfully synthesised. Final analysis showed that the feedstock contains a high level of organic carbon (48.50%) and a low level of heteroatoms, making it a clean and appropriate raw material. The presence of sulfonate (SO₃⁻) and hydroxyl (-OH) groups in the product was also confirmed by the FTIR analysis, which confirmed the structural integrity of the binder to be used in adhesive.

At 50% substitution of Polyvinyl Acetate (Type B), the manufactured particleboards were found to be of ANSI A208.1 Grade M-1 minimum with an MOR of 11.2 ± 0.17 N/mm² and MOE of $2,110 \pm 21.4$ N/mm² and also had a better moisture resistance than the 100 percent PVA reference. Nonetheless, further substitution to 75% (Type C) greatly reduced the dimensional stability and mechanical performance as the hydrophilicity of the sulfonate groups became overly high at high bio-binder loadings.

These results make *Cordia millenii* lignosulfonate a mechanically feasible and sustainable synthetic resin extender in the production of particleboard, and therefore, timber waste is valorised according to UN SDG 12 (Responsible Consumption and Production). In future studies, the use of hydrophobic additives like paraffin wax to enhance moisture resistance under increased levels of substitution needs to be studied with a view of complete industrial scale-up.

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