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Research Article

Development of formaldehyde-free wood adhesive resins using oil palm's nano lignin particles

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Abstract

The quest for green, sustainable substitutes for petroleum-derived adhesives has accelerated amid rising environmental concerns and the rapid depletion of fossil fuels. This work investigated the potential application of lignin nanoparticles (LNPs) derived from oil palm empty fruit bunch (OPEFB) in a bio-based adhesive system. The organosolv technique was effective in extracting relatively pure lignin from the biomass, as evidenced by the extracted lignin's 88.94% purity and 75.10% recovery efficiency. The lignin was successfully converted to LNPs via antisolvent precipitation. Stable nanoparticle formation was indicated by Dynamic Light Scattering (DLS), which showed an average particle size of 163.38 nm and a zeta potential of -15.6 mV. At loadings of 10%, 30%, and 50%, the LNPs were evaluated as reinforcements in resin. At 30% loading, tensile strength reached a maximum of 19.15 MPa. This highlights the promise of OPEFB-derived lignin as an effective reinforcing agent in adhesives and a renewable constituent for environmentally friendly polymer products.

Keywords: OPEFB, Organosolv, Nano lignin, Tensile strength, Adhesion

Introduction

Wood has historically been considered a vital material utilised across several industries, such as construction, furniture production, and floor coverings, which are essential to fostering emissions reductions and sustainable growth. The global annual production of wood-based panels corresponds to approximately 480 million cubic meters, requiring substantial amounts of wood adhesives (Zhu *et al.,* 2024). Adhesives facilitate the bonding of wood components by forming stable joints through interlocking and chemical interactions. The qualities exhibited by adhesives are of utmost importance for their application. A comprehensive understanding of these characteristics, such as mechanical shear strength (Huveners *et al.,* 2007) and environmental resistance (Wang *et al.,* 2024), is important for choosing a suitable adhesive.

The growing demand for manufactured furniture and building supplies has driven the need for more reliable and versatile adhesives. Urea-formaldehyde (UF) resin adhesives are primarily used to produce wood-based panels due to their cost-effectiveness, durability, superior performance, and ease of application. The production of medium-density fiberboard (MDF), largely based on UF adhesives, surpassed 100 million cubic meters in 2020, underscoring its widespread use (Rovira *et al.,* 2016). Additionally, phenol formaldehyde (PF) resin is a polymeric condensate formed by the reaction of phenol and formaldehyde, catalyzed by an alkaline catalyst. PF resin has been widely utilized in the moulding and composite laminate sectors (Neiva *et al.,* 2020). These conventional adhesives gained prominence due to their exceptional durability, scalability, and affordability. The global demand for adhesives has increased substantially across various industries alongside their ongoing use.

Additionally, the rise of health and ecological concerns has contributed to an anticipated increase in bio-based wood adhesives. This is because formaldehyde-based adhesives are significant contributors to volatile organic compound (VOC) emissions (Bertheau *et al.,* 2024). The International Agency for Research on Cancer (IARC) has categorised formaldehyde as "Group 1, carcinogenic to humans" (Jia *et al.,* 2020). Therefore, bio-based adhesives derived from renewable polymers such as lignin offer an ecologically friendly alternative.

Despite lignin's potential as a sustainable resource, research focused on developing lignin-based adhesives without formaldehyde remains limited. Specifically, the importance of adding OPEFB-derived LNPs to lignin-phenol-glyoxal (LPG) resin systems regarding mechanical performance has not been thoroughly explored. Therefore, this work aims to extract organosolv lignin (OL) from OPEFB and convert it into LNPs using the antisolvent precipitation method. The lignin purity, colloidal dispersion stability, and mechanical properties were analyzed through various quantitative tests. The performance of lignin-phenol-glyoxal (LPG) wood adhesives was evaluated to understand the effect of different LNPs loading from OL.

Materials and methods

Materials

Oil palm empty fruit bunch (*Elaeis guineensis*) was collected from a palm oil plantation in Penang, Malaysia. Ultrapure water was obtained from a Milli-Q Plus water purification system (Nancy, France), and absolute ethanol was purchased from VWR. The chemicals used are sulphuric acid (H_2SO_4, 97.0%), phenol (99.5%), and glyoxal (40% wt% solution in water). The chemicals were purchased from Sigma-Aldrich. Acetone (99.8%) was purchased from Carlo Erba reagents. All the chemicals were used without further purification.

Macro Lignin Particles (MLP) extraction using organosolv

This method was adopted from a previous study with modifications (Girard *et al.,* 2024). About 600 g of oven-dried biomass was combined with a 40:60 (v/v) water-ethanol solvent at a solid-to-liquid ratio of 1:8 to extract the organosolv lignin (OL). The mixture was treated for an hour at 200°C and 25 bar in a 10 L reactor. After the reaction, the solution was filtered and rinsed with 60% aqueous ethanol. Distilled water was progressively added to the filtrate in a 3:1 (v/v) ratio to induce lignin precipitation. The mixture was allowed to settle at room temperature overnight. The precipitated lignin was then filtered, thoroughly rinsed with distilled water, and air-dried at room temperature. The lignin was further ground into a fine powder.

Soxhlet extraction of macro lignin

Cellulose thimbles and wool were first dried at 105 °C for 72 hours, then weighed in a vacuum desiccator to determine the extractive content. A KERN® DAB 100-3 thermobalance was used to precisely weigh about 1 g of lignin and measure its moisture content accordingly. After loading the material into the thimble and sealing it with wool, 175 mL of a 2:1 v/v toluene-ethanol mixture was used for a 6-hour Soxhlet extraction (about 20 cycles) (Girard *et al.,* 2024).

Preparation of Lignin Nano Particles (LNPs) using the antisolvent method

This procedure was adopted from previous work with modifications (Girard *et al.,* 2024). Around 5.00 g of OL was dissolved in 120 mL of pure acetone to generate lignin nanoparticles. The volumetric flask was filtered through a 0.45 µm nylon membrane after immersion in an ultrasonic bath for 1 hour to ensure complete dissolution. Using a KF Technology NE-1010 syringe pump, the filtered lignin solution was subsequently converted into nanoparticles via antisolvent precipitation. A regulated flow rate of 2 mL/min was used to inject 120 mL of the solution into 450 mL of ultrapure water while stirring continuously at 350 rpm. The final lignin nanoparticles were obtained by freeze-drying the aqueous suspension after excess acetone was removed using a rotary evaporator (Büchi Rotavapor).

Formulation of LPG resins

The procedure was adapted from a previous study (Huzyan *et al.,* 2021). Different lignin concentrations (10%, 30%, and 50% w/w) were used to produce the resin. A measured amount of phenol was added to the flask and followed by lignin addition. The mixture was stirred continuously until a homogeneous phase was achieved. Upon complete mixing, a 0.5% wt NaOH solution was added dropwise to the flask. Then ethanol was added to the mixture as a solvent. The resulting mixture was refluxed at 80 °C with continuous stirring for 10 minutes. Next, a 40% (w/w) glyoxal solution was slowly added to the phenolated lignin mixture at a glyoxal-to-phenol (G/P) molar ratio of 1.5:1. The mixture was refluxed at 80 °C for 2 hours with constant stirring. After completion of the reaction, the remaining ethanol was removed from the reaction mixture using the rotary evaporator. The evaporator used was Heidolph Laborota 4011 digital.

Application of LPG resin adhesive to plywood

The method was adapted and carried out based on a procedure reported elsewhere (Huzyan *et al.,* 2021). The prepared LPG resin samples were applied on wood panels by applying 1115 g/m^2 to a single side of three-ply veneer wood, measuring 12.0 cm x 12.0 cm × 0.6 cm. The resin-coated wood panels underwent a two-phase curing procedure. Initially, the wooden panels were assembled and placed in the cold press. The process was conducted at room temperature, and a pressure of 2100 psi was applied for 7 minutes. After cold pressing, the wooden panels were conveyed to the hot press for further curing. The pressure was maintained at 2100 psi at 140 ℃ for 7 minutes.

Characterizations

Klason Lignin

A 50 mL centrifuge tube was filled with about 0.175 g of dry powdered OPEFB lignin to calculate the amount of acid-insoluble lignin. To avoid agglomeration, the sample was treated with 1.5 mL of 72% H_2SO_4 and incubated in a water bath at 30 °C for 60 minutes while being manually stirred every five minutes. To make sure all of the biomass was suspended in the liquid phase after hydrolysis, the mixture was diluted with 42 mL of ultrapure water. After that, the suspension was autoclaved for 60 minutes at 121 °C. Following the cooling process, a vacuum flask was used to filter the mixture via a Gooch crucible, and ultrapure water was used to properly wash the solid residue. The solid residue-containing crucible was kept in an oven set at 105 °C for 24 hours. About 5 mL of the filtrate was kept in Eppendorf tubes for further analysis. Lastly, the mass of the dried residue was measured using an analytical balance to determine the overall lignin yield (Girard *et al.,* 2024).

High-performance anion-exchange chromatography with pulsed amperometric detection (HPAEC-PAD)

Monomeric sugars in liquid fractions were analyzed using the High-Performance Anion Exchange

Chromatography coupled with Pulsed Amperometry Detection (HPAE-PAD, ICS-3000 Dionex™) with analytical column Dionex™ CarboPac PA-20 (3 × 150 mm). Acid-insoluble lignin and monosaccharide content were measured using extractive-free biomass in accordance with NREL methods (NREL/TP-510-42618 and NREL/TP-510-42623). The following composition was used to elute monosaccharides at 35 °C at a flow rate of 0.4 mL/min: 99.2% ultrapure water/250 mM NaOH 0.8%: 0-20 minutes; 75% ultrapure water/250 mM NaOH 20%/NaOAc (1 M) – NaOH (20 mM) 5%: 20-37 minutes; and 40% ultrapure water/250 mM NaOH 20%/NaOAc (1 M) – NaOH (20 mM) 40%: 37-41 minutes. Following each run, the column had been rinsed and re-equilibrated, and sugar concentrations were measured using external calibration standards (Girard *et al.,* 2024).

Dynamic Light Scattering (DLS) and Zeta Potential (ζ-potential)

A Malvern Zetasizer ULTRA was used to measure the average particle size and zeta-potential of the lignin nanoparticles. Approximately 1.5 mL of the nanoparticle suspension was put into optical polystyrene cells. Three measurements in 3-D were performed in dynamic light scattering (DLS) mode at an angle of 174°. Finally, the same equipment was used to measure the zeta-potential at 25 °C using precisely folded capillary zeta cells (DTS 1070) (Girard *et al.,* 2024).

Tensile strength test

The wood panels were maintained at ambient temperature before testing for bond durability. The test machine was configured to mount panels centrally between two clamps. The standard used was ASTM D903: Standard Test Method for Peel or Stripping Strength of Adhesive Bonds. The tensile strength and elasticity modulus of the wood panels were measured with Instron UTM 5582 (Instron, Norwood, MA, USA) (Hussin *et al.,* 2017).

Results and discussion

Characteristics of OPEFB lignin

The residue composition, lignin recovery efficiency, lignin yield, and purity of the extracted OPEFB lignin are presented in Table 1.

The data above indicate that OPEFB-OL demonstrated high lignin purity with minimal residual cellulose and hemicellulose. The remaining carbohydrate proportion was comparatively low, showing successful delignification. This suggests potential interactions between lignin and polysaccharide fragments during extraction. The organosolv technique is able to selectively solubilise lignin while reducing carbohydrate contamination. As a result, the high yield and recovery efficiency are evident. The production of high-purity lignin recovery was primarily facilitated by the organosolv method, notwithstanding the effectiveness of other techniques in addressing the resistance of lignocellulosic biomass (Nair *et al.,* 2023).

This extraction method provides an effective alternative, producing lignin with substantially reduced levels of residual sugars and ash. During this process, high-purity lignin and hemicellulose-derived compounds are simultaneously generated and readily isolated from cellulose pulp. The pulping process significantly alters lignin structure, primarily by the substantial degradation of the unstable β-O-4 linkages (Constant *et al.,* 2016). Lignin structures predominantly undergo degradation in the presence of polar protic solvents such as methanol and ethanol (Sarkar & Adhikari, 2000). The elimination of carbohydrates from hemicellulose enhances the delignification process and increases the yield of precipitated lignin. The purity of lignin is preserved, as supplementary solubilised oligomeric sugars do not bind to it during extraction, thereby averting carbohydrate interference (Adamcyk *et al.,* 2023).

DLS and zeta potential

The LNPs were produced using the antisolvent method. The mean particle size and zeta potential values of the LNPs are recorded in Table 2.

The OPEFB-OL exhibits a particle size with a mean diameter of 160.06 nm, thus indicating effective nanoscale production through antisolvent precipitation. Effective nucleation and self-assembly during solvent exchange are indicated by the production of particles smaller than 200 nm. The crosslinked lignin contributes to the formation of smaller LNPs by creating a robust network (Liu *et al.,* 2019). The effectiveness of the antisolvent precipitation technique utilized matches the values reported in earlier studies (Makri *et al.,* 2022). The measured zeta potential values of the OPEFB-OL particles suggest a moderate colloidal stability. Values in this range suggest a possible inclination toward aggregation over time. The lignin may exhibit reduced electrostatic repulsion despite its increased heterogeneity, which provides fewer ionizable groups to generate surface charges. Additionally, Pereira *et al.* (2022) suggested that LNPs tend to be smaller in size when they possess a strong negative surface charge. This prominent negative surface charge results from the high presence of phenolic groups and adsorbed hydroxyl (-OH) groups, indicating how hydrophobic molecules interact with water (Pereira *et al.,* 2022). Additionally, the nanoscale particle size will enhance stress transmission and surface interactions between the LNP and the resin network by promoting

Table 1: Residue composition, lignin yield, recovery efficiency, and purity of OPEFB lignin after extraction

Index	Residue composition (%)		Lignin yield (%)	Lignin Recovery Efficiency (%)	Lignin purity (%)
	Cellulose	Hemicellulose			
OPEFB-OL	4.70±0.01	0.05±0.01	17.80	75.10	88.94±0.52

greater dispersion of the LNP within the LPG resin matrix. However, uniform dispersion may be hindered by higher nanoparticle loadings due to excessive aggregation resulting from moderate colloidal stability. This may eventually affect the mechanical performance of the adhesive.

Tensile strength

Figure 1 below shows the comparison of tensile strength and elastic modulus for each adhesive sample. The LNPs loading had a significant impact on the mechanical performance of the adhesive compositions. The samples that correspond to 10%, 30%, and 50% of lignin substitution in the LPG resin are OPEFB-OL 10, OPEFB-OL 30, and OPEFB-OL 50, respectively.

It is noticeable that when the LNPs loading increased, the OPEFB-OL resin's mechanical characteristics also improved. The tensile strength rose from 13.81 MPa for OPEFB-OL 10 to 19.15 MPa for OPEFB-OL 30, suggesting that the modest addition of LNPs enhanced the mechanical performance of the resin. Better stress transport within the material may be facilitated by improved interfacial contacts between the lignin nanoparticles and the polymer matrix. Besides, Khan and Ashraf (2006) further showed that cross-linking density affects the resin's bonding strength (Khan & Ashraf, 2006).

Nevertheless, the tensile strength dropped to 14.63 MPa when the LNPs loading was raised to OPEFB-OL 50. This decrease suggests that inadequate dispersion or particle agglomeration within the matrix may result from excessive nanoparticle loading. Aziz *et al.* (2019) have observed that the largest levels of polycondensation and cross-linking formation occur in PG resin at 30% lignin substitution. Moreover, inadequate polycondensation and cross-linking formation do occur at 50% lignin substitution. Thus, 30% was shown to be the optimal lignin substitution rate when producing LPG resins (Aziz *et al.,* 2019).

On the other hand, the elastic modulus improved steadily as the LNPs loading increased. This pattern suggests that the resin's stiffness was much higher due to the inclusion of lignin nanoparticles. Although major loading may adversely influence tensile strength due to dispersion constraints, the stiff structure of lignin nanoparticles probably limits the flexibility of the polymer chains, enabling the material to be stiffer even at greater loadings.

Table 2: The mean particle size (nm) and zeta potential values of the lignin

Sample Name	Mean Particle Size (nm)	Zeta Potential (mV)
OPEFB-OL	160.06±1.38	-15.6±0.2

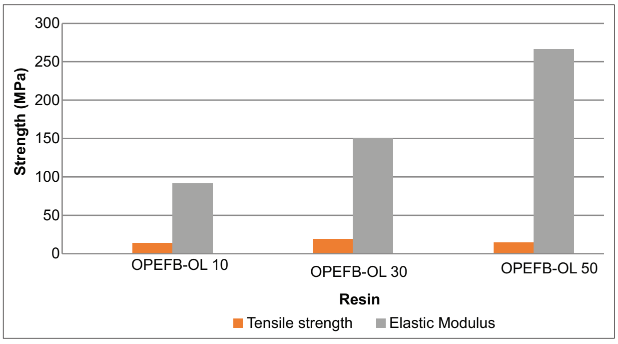

Figure 1: The tensile strength and the elastic modulus of the resin samples

In addition, the interactions between the polymer matrix and LNPS could potentially be responsible for the enhanced mechanical performance shown in the adhesive system. During the polycondensation reaction with phenol and glyoxal, lignin nanoparticles can engage in hydrogen bonding and possible covalent interactions due to the abundance of phenolic and hydroxyl functional groups on their surface. Research has demonstrated that lignin and glyoxal can be cross-linked under a condensation reaction. This is due to the presence of G units in lignin and phenol, which are structurally similar (Faris *et al.,* 2017). Methylene and acetal crosslinks are generated when two aldehyde groups in glyoxal react with the ortho- and para-positions of lignin phenolic rings. Glyoxal can produce a 3-D polymeric network having an adequate crosslinking density to alter the resin's mechanical characteristics and structure (Sharulnizam *et al.,* 2026). The LNPs improve the surface area, which facilitates greater dispersion in the polymer matrix and allows the LNPs to act as reinforcing components, enhancing the mechanical properties and the flexibility of the adhesive produced.

Conclusion

The research concludes that OPEFB lignin is a sustainable alternative for producing LNPs and their application in a bio-based adhesive system. High-purity lignin was extracted using the organosolv method and successfully converted into LNPs via the antisolvent technique. The integration of LNPs into the adhesive formulations significantly improved the mechanical performance, confirming their function as an essential reinforcing filler within the polymer network. Moreover, OPEFB-OL 30 showed the best mechanical performance. The LPG resin exhibited the maximum tensile strength at this loading, indicating that a suitable amount of LNPs improves the mechanical characteristics. This sample has achieved an ideal distribution, which provides a stronger bridge between polymer chains to withstand higher tension. In summary, the results show that lignin nanoparticles generated from OPEFB are a viable renewable material for innovative and eco-friendly adhesive applications.

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Author contributions

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