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Synthesis of Lignin–Urea Formaldehyde (LUF) Wood Adhesive Based on Lignin Extracted from Oil Palm Empty Fruit Bunches (EFB)

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Abstract. Oil palm empty fruit bunches are one of the underutilized plantation wastes. Lignin is one of the components of plant cell walls besides cellulose and hemicellulose that can be used as a raw material for the synthesis of lignin-based adhesives, namely lignin-urea-formaldehyde (LUF) adhesive resins. The polymerization reaction of LUF formation was confirmed from the shift and change in the FTIR wavenumber of the -C-O-C- ether group of LUF compared to UF and LF obtained during the synthesis process. The tensile strength test results of all adhesives produced exceeded the tensile strength value of commercial UF adhesives, and it was found that increasing the lignin concentration in the synthesized adhesives was directly proportional to the tensile strength value. Increasing the lignin concentration was also able to reduce formaldehyde emissions from all variations of adhesives produced, but was unable to exceed the lower formaldehyde emission value of UF adhesives.

Keywords : Palm Oil EFB, Lignin, Wood Adhesives, LUF

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Introduction

Oil palm is one of Indonesia's major and strategic agricultural commodities, as reflected in the vast plantation area nationwide [1]. According to Statistics Indonesia (BPS) 2023 data, the total oil palm plantation area reached 16,833,985 ha, comprising 577,937 ha owned by the government, 8,614,259 ha managed by private enterprises, 6,736,516 ha cultivated by smallholders, and 905,273 ha designated as land subject to verification (LAD). Meanwhile, North Sumatera ranks among the top five province in Indonesia in terms of land area, covering approximately 2,018,727 hectares [2].

During the processing of Crude Palm Oil (CPO) in the industry, approximately 230 kg of empty fruit bunches (EFB) are generated from every 1 ton of fresh fruit bunches processed, which is equivalent to about 23% [3]. EFB is one of the largest waste streams generate by the plantation industry, whose utilization is still considered limited, as it is generally used only for compost production [4], burned in boilers or incinerators [3], or simply buried in soil as material for reinforcing plantation roads [5], [6]. Chemical composition analysis of EFB indicates that it contains approximately 33-44% cellulose [7], 12-24% hemicellulose [8], and 21-26% lignin [9], where the presence of these constituent materials suggest a strong potential for EFB to be processed into functional material feedstocks.

One of the functional materials with potential to be produced is a lignin-based urea-formaldehyde (UF) adhesive resin, which in this study is referred to as lignin-urea-formaldehyde (LUF) resin. UF resin itself has long been recognized in the plywood and composite wood industries, however, this resin has a major drawback, namely the high formaldehyde emission it produces, particularly when exposed to elevated temperatures [10]. Resin with formaldehyde emissions have negative effects on air quality, particularly in indoor environments, and may pose health risks as a result of exposure to the volatile nature of formaldehyde [11]. This condition has ultimately led to the implementation of stringent regulation governing the use of conventional formaldehyde-based resins [12], thereby necessitating the development of more environmentally friendly formaldehyde-based resin alternatives that remain suitable for plywood and

related wood-based industries [11]. It is well known that lignin is a natural polymer that acts as a natural adhesive, binding cellulose and hemicellulose within the structural framework of plant wood [13]. Previous related studies using lignin extracted from sugarcane bagasse [14] and lignin derived from bamboo fibers [15] reported that the adhesive strength of the resin increased with increasing lignin composition in the mixture, while simultaneously reducing the resulting formaldehyde emissions. In this study, we synthesize LUF adhesive resin using lignin isolated from EFB. The addition of lignin is expected to enhance bonding strength due to the presence of phenolic and hydroxyl functional groups [16] that promote strong interactions with the urea-formaldehyde matrix and wood surfaces, thereby improving resin cohesion and adhesion [17]. Simultaneously, lignin acts as a formaldehyde scavenger and partially replaces UF components, which reduces free formaldehyde and contributes to lower formaldehyde emissions [18].

Experimental

The Materials and Tools. The materials and equipment used in this study are briefly described as follows. Oil-palm empty fruit bunches (EFB), sodium hydroxide (NaOH), sulfuric acid (H_2SO_4), urea, formaldehyde solution (≈ 37 wt%), formic acid (HCOOH), deionized water, ammonium hydroxide (NH_4OH , 10%), hydrochloric acid (HCl, 0.1 N and 1 N), and a mixed indicator (methyl red/methylene blue) were used. The equipment included cutting tools, a grinder, sieves (60- and 100-mesh), a hot plate with magnetic stirring, a reflux setup, a water bath, a pH meter, filtration apparatus, an analytical balance, a Fourier transform infrared (FTIR) spectrometer, a universal testing machine, and hot and cold presses

Lignin Isolation from Oil-Palm Empty Fruit Bunches (EFB). EFB was cleaned, chopped to reduce size, sun-dried to constant mass, and milled to approximately 60-mesh. Delignification was performed using 5% (w/v) NaOH at a solid-liquid ratio of 1:10 (EFB:NaOH). The slurry was heated at 100 °C for 30 min, cooled, and the resulting black liquor was separated by filtration. The solid residue was washed with deionized water to neutrality (pH ≈ 7). The black liquor was heated and stirred at 60°C, while H_2SO_4 was added dropwise until the pH reached 2 and a brownish color was formed. The mixture was the allowed to stand for 24h to allow

lignin precipitation. Subsequently, the precipitate was filtered, dried, ground, and sieved to approximately 100-mesh to obtain lignin powder [19].

Urea Hydroxymethylation. Urea and formaldehyde were mixed at a molar ratio of 1:2. The pH was adjusted to ~8.0 using 50% (w/w) NaOH. The mixture was refluxed at 40 °C under stirring for 30 min, then the temperature was raised to 90 °C and maintained for 60 min. Afterward, a few drops of formic acid (HCOOH) were added to adjust the pH to 5.0-5.5 [20].

Lignin Hydroxymethylation In a beaker, lignin and formaldehyde were combined; the pH was adjusted to 12.5 with 50% (w/w) NaOH and stirred until the lignin dissolved. The mixture was refluxed at 60°C for 90 min. The pH was then adjusted to 5.0-5.5 with formic acid and stirred at room temperature for 90 min. Three lignin-to-formaldehyde molar ratios were prepared: LUF-1 (0.25:1), LUF-2 (0.50:1), and LUF-3 (0.75:1) [21].

Synthesis of Lignin-Urea-Formaldehyde (LUF) Adhesive. The two prepolymers (hydroxymethylated urea and hydroxymethylated lignin) were combined and refluxed at 90 °C for 90 min with magnetic stirring. The mixture was cooled to room temperature and neutralized to pH ~7 with 50% (w/w) NaOH. The resulting LUF adhesive was then ready for testing [22].

FTIR Analysis. Fourier transform infrared (FTIR) spectroscopy was employed to analyze lignin-formaldehyde (LF) resin and its precursors (lignin and formaldehyde), urea-formaldehyde (UF) resin and its precursors (urea and formaldehyde), as well as the lignin-urea-formaldehyde (LUF) resin. The analysis was conducted to identify changes in absorption band intensities and wavenumber shifts, thereby confirming the formation of the LUF resin through comparative evaluation of its FTIR spectrum with those of the LF resin, UF resin, and their respective precursors.

Lap-Shear Bond Strength Test. Wood adherends (10 cm × 1 cm × 0.4 cm) with an overlap (shear) area of 4 cm × 1 cm (4 cm²) were used. Adhesive was applied uniformly at a spread rate of 190 g m⁻² to one bonding surface. Cold pressing was conducted for 10 min, followed by hot pressing at 130 °C for 5 min. The laminates were conditioned at ambient temperature for 7 days. Dry-state bond strength was measured; a commercial adhesive was tested in parallel as a

reference [23].

Formaldehyde Emission Test (Titrimetric Method). An adhesive sample (5.00 g) was placed in an Erlenmeyer flask, mixed with 15 mL deionized water, and homogenized. Two to three drops of mixed indicator (methyl red/methylene blue) were added. Subsequently, 5 mL of 10% NH₄OH and 5 mL of 1 N NaOH were added; the mixture was neutralized with 0.1 N HCl, capped, shaken, and incubated in a water bath at 30 °C for 30 min. The solution was titrated with 1 N HCl to the endpoint indicated by a color change from green to bluish-gray and finally to reddish-purple. A blank (without adhesive) was processed identically. Formaldehyde emission was calculated using Eq. (1).

$$\text{Formaldehyde Emission (\%)} = \frac{(V_{\text{blank}} - V_{\text{sample}}) \times N_{\text{HCl}} \times 30.03}{m_{\text{sample}} \times 1000} \times 100\% \quad (1)$$

Where V is the HCl volume (mL), N_{HCl} is HCl normality (eq L⁻¹), m_{sample} is sample mass (g), and 30.03 is the molar mass of formaldehyde (g mol⁻¹) [24].

Result and Discussion

FTIR Analysis. FTIR analysis is essential for identifying changes in functional groups and chemical bonding during resin formation. Figure 1 shows the FTIR spectra of lignin and formaldehyde in comparison with the corresponding lignin-formaldehyde (LF) resin (A), as well as urea and formaldehyde in comparison with the urea-formaldehyde (UF) resin (B), enabling evaluation of molecular interactions and structural evolution in the resin systems.

The FTIR spectrum of the isolated lignin exhibits characteristic absorption bands that are consistent with those reported for reference lignin in the literature. The presence of a broad absorption band at a wavenumber of 3425.40 cm⁻¹, attributed to -OH stretching, confirms the presence of phenolic structures, which are characteristic features of lignin molecular structure [25]. The presence of aromatic lignin groups was confirmed by absorption bands at a wavenumber of 2925.49 cm⁻¹, attributed to C-H stretching, and at 1637.48 cm⁻¹ attributed to C=C stretching. This indicates that the backbone of the lignin structure is predominantly composed of aromatic compounds [26], [27]. Furthermore, the absorption band at wavenumber of 1222.65 cm⁻¹ indicates vibrations of the aromatic ring attributed to the C-O stretching, which is characteristic of guaiacyl

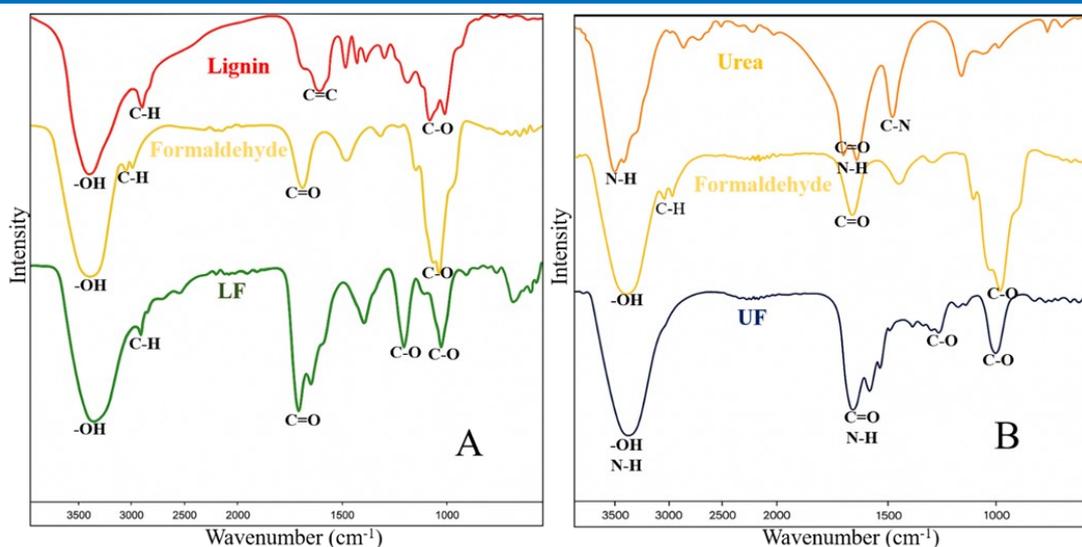


Figure 1. FTIR spectra of lignin-formaldehyde, LF (A) and urea-formaldehyde, UF (B) systems

units, while absorption band at 1328.80 cm^{-1} corresponds to aromatic ring vibrations originating from C-O stretching enhanced by the presence of methoxy groups in syringyl units [28], [29]. The C-O vibrations associated with methoxy ($-\text{OCH}_3$) ether groups were also identified in the $1222.65\text{--}1046.00\text{ cm}^{-1}$ region [29], [30]. The good agreement between the present results and the reference spectra provides strong evidence for the successful isolation of lignin. Although no quantitative purity measurement was conducted, the absence and significant reduction of cellulose and hemicellulose-related bands, typically observed around $\pm 1730\text{ cm}^{-1}$ and within the $1420\text{--}1030\text{ cm}^{-1}$ region, qualitatively indicate a relatively high lignin purity [31].

The FTIR spectrum of formaldehyde ex-

hibited an $-\text{OH}$ stretching band at 3340.75 cm^{-1} , which is likely associated with the alcoholic solvent present in the 37% formaldehyde solution. An absorption at 2912.71 cm^{-1} indicates the presence of $-\text{CH}$ groups [32], [33]. The aldehyde carbonyl was assigned to a band at 1641.38 cm^{-1} ($-\text{C}=\text{O}$), supported by a signal at 989.91 cm^{-1} corresponding to the symmetric stretching of the C-O-C linkage [34]. FTIR analysis of urea showed the $-\text{NH}_2$ functionality, identified by N-H stretching at 3442.56 cm^{-1} [35], [36]. An overlapping absorption of $-\text{C}=\text{O}$ and $-\text{NH}$ was observed at a wavenumber of 3332.48 cm^{-1} , and a band at 1623.06 cm^{-1} was identified, originating from C-N bending [35].

FTIR spectroscopy was also employed to determine chemical interactions and condensation phenomena occurring during resin formation by

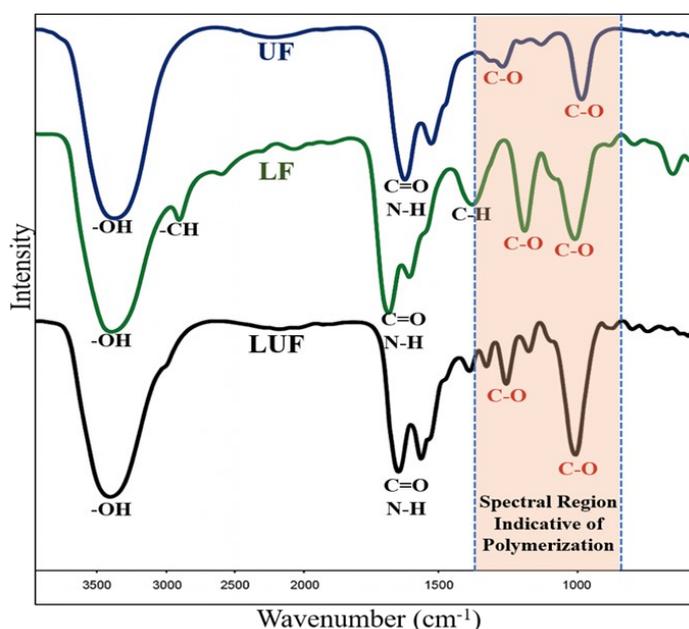


Figure 2. FTIR spectra acquired during LUF formation

examining changes in intensity and shifts in wavenumbers from the individual reactants to the final polymer system, as shown in Figure 2.

Evidence of polymerization through condensation processes was identified by observing shifts in wavenumbers and/or the emergence of new C-O absorption bands, which confirm the occurrence of condensation reaction between lignin-formaldehyde (LF) and urea-formaldehyde (UF) species. The specific region analysed was the wavenumber range of $1300\text{--}1100\text{ cm}^{-1}$, which corresponds to the presence of C-O stretching vibrations of ether linkages [34]. The phenomenon of a reduced presence of free formaldehyde associated with formaldehyde emissions can also be observed from this spectrum. During LF formation, a decrease in absorption band corresponding to symmetric C-O-C stretching was observed, indicating the consumption of a portion of formaldehyde during the condensation process. It should be noted that two ether C-O-C absorption bands appeared during this process, suggesting that lignin is also capable of accommodating a portion of free formaldehyde. This phenomenon is consistent with the expectation of this study, in which the addition of lignin is able to reduce the resulting formaldehyde emissions [37], [38]. A similar phenomenon was also observed during the formation of UF, where the C-O band intensity diminished even further, indicating that urea more effectively scavenges free formaldehyde than lignin, consistent with lignin's comparatively lower reactivity as a reactant [39].

In the lignin-urea-formaldehyde (LUF) system, additional shifts and intensity changes of C-O bands relative to the LF and UF spectra signify the formation of new ether C-O linkages, confirming condensation between LF and UF to produce LUF [34]. Based on the previous FTIR analysis results, the condensation phenomena are schematically represented through chemical reaction as shown in Figure 4. It should be noted that this reaction provides only simplified conceptual description of the formation of new ether linkages as evidence of condensation occurring within the LUF system. The process is initiated by hydroxymethylation (stage 1.), followed by condensation (stage 2).

Tensile Strength Test. Tensile strength testing was conducted to evaluate the bonding strength of the synthesized adhesive resin, as well as to assess its ability to transfer stress across the wood-adhesive interfacial surface, as shown in Figure 5.

Urea-formaldehyde is a thermosetting resin that function by penetrating the wood surface and hardening through heating, forming strong polymeric bonds between the bonded wood surfaces [40]. Meanwhile, lignin adheres to the wood surface through hydrogen bonding with cellulose [41], [42]. The combination of these two adhesion mechanisms subsequently results in an adhesive with high bonding strength, as demonstrated by tensile strength testing, in which all synthesized adhesive variations exhibited bonding strengths exceeding that of commercial UF adhesive. This clearly demonstrates that increasing lignin concentration within a certain threshold has a direct correlation with the

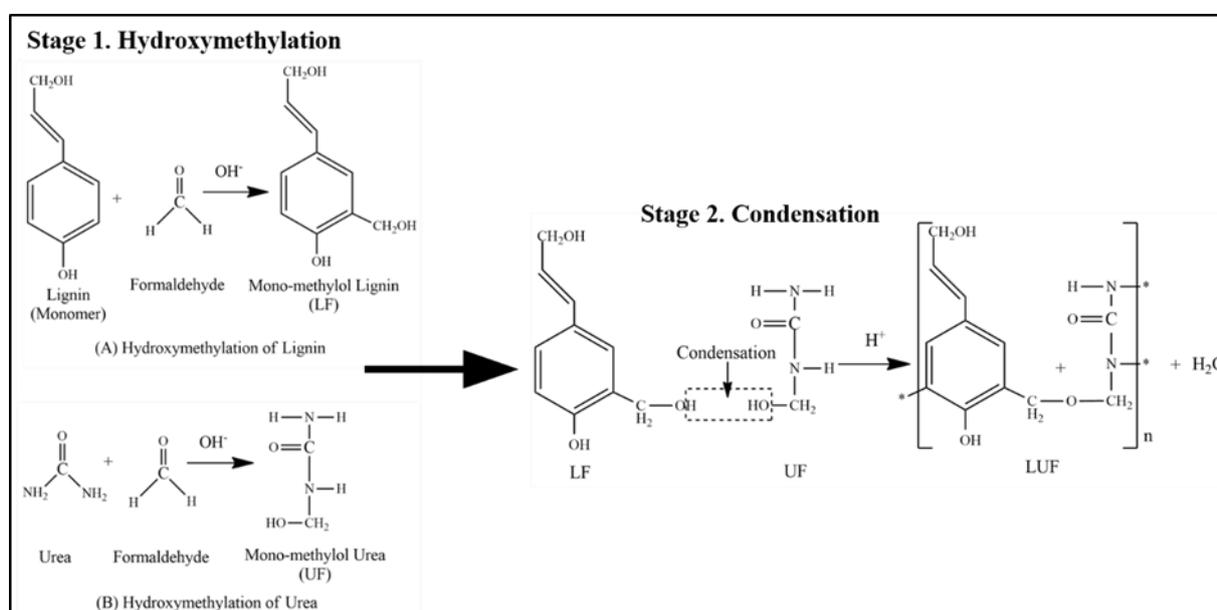


Figure 3. Proposed reaction scheme for LUF formation

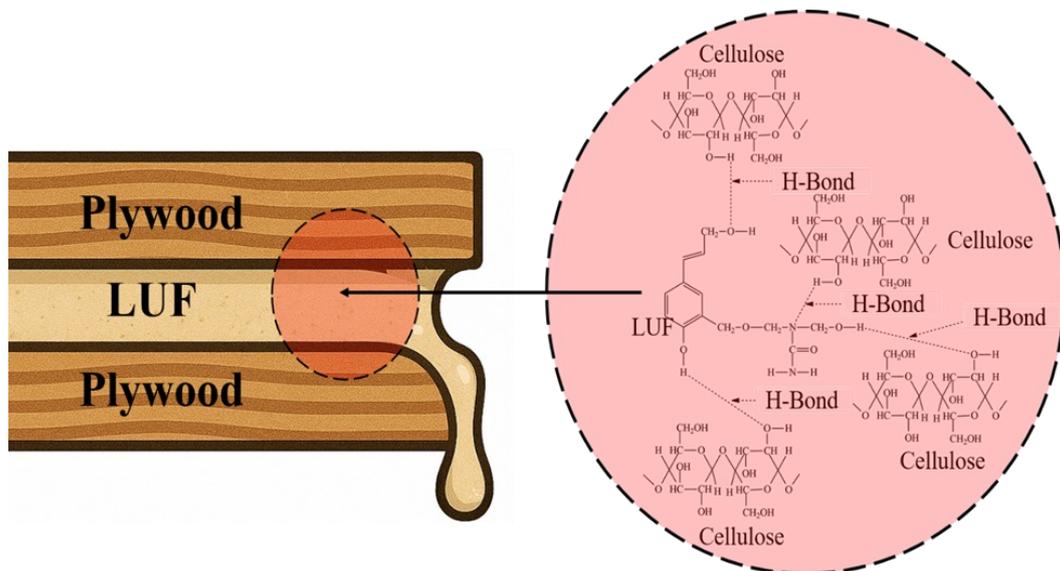


Figure 4. Illustration of the molecular interaction between LUF and cellulose on

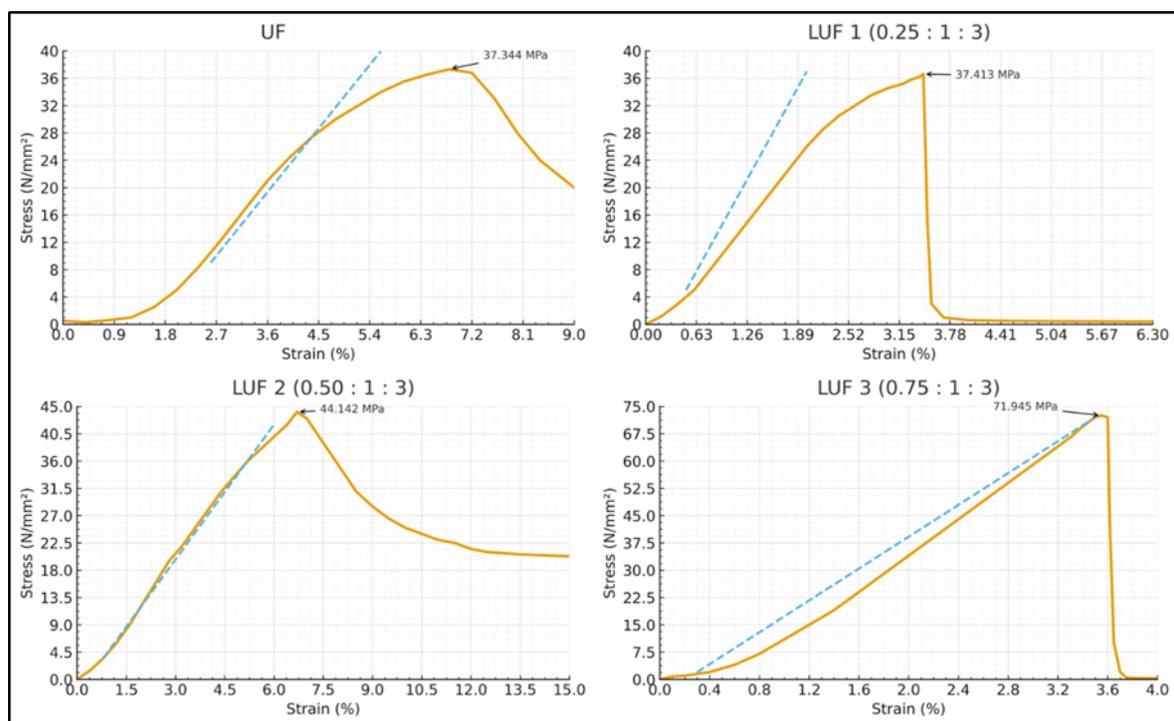


Figure 5. Tensile strength curves for all adhesive variations

resulting tensile strength.

This is directly reflected in the tensile strength values of each variation, namely 37.414 MPa (LUF 1), 44.142 MPa (LUF 2), and 71.945 MPa. The initial increase in tensile strength (37.414-44.142 MPa) indicates the initial reinforcing effect of lignin, where under these conditions lignin acts as an active component providing additional intermolecular interactions within the adhesive matrix [43]. The further increase in tensile strength to 71.945 MPa at the highest

lignin concentration (LUF 3) indicates that lignin within the adhesive has transformed into a more rigid and highly interconnected polymeric network [44]. This enhancement phenomenon implies a more uniform distribution of stress across the wood-adhesive interface, which may occur through increased formation of hydrogen bonds with cellulose as well as an improvement in mechanical properties in the form of greater stiffness [45]. These results indicate that lignin does not merely function as a filler, but rather acts as an effective reinforcing agent that significantly enhances the mechanical

performance of the LUF adhesive system at higher loadings.

Formaldehyde Emission Test Results.

Formaldehyde emission testing showed that increasing lignin content in the formulation resulted in a gradual decrease in formaldehyde emissions, likely because the phenolic-hydroxyl groups in lignin can act as scavenging sites for free formaldehyde through hydroxymethylation and subsequent condensation reaction [46]. This trend indicates that a higher availability of formaldehyde will promote more effective interaction with reactive sites on lignin, thereby partially immobilizing free formaldehyde within the polymer network.

Nevertheless, the emissions from all synthesized LUF adhesives remained significantly higher than those of commercial UF adhesive, which is associated with the large and heterogeneous macromolecular structure of lignin and its inherently lower reactivity toward formaldehyde compared to urea [13], [47]. As a result, a portion of formaldehyde is not fully consumed during the curing process and may be released through the cleavage or hydrolysis of labile ether linkages within the cured network [48]. These findings indicate that although lignin incorporation contributes to the scavenging of a portion of free formaldehyde, additional treatments or procedures, such as chemical activation of lignin,

es. Increasing lignin content significantly enhanced tensile strength and reduced formaldehyde emissions due to its scavenging effect. However, the emission levels remained higher than those of commercial UF resin, suggesting that further optimization is required

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Author Contributions

All authors made substantial contributions to the work, including conceptualization, data acquisition, supervision, and manuscript preparation. All authors reviewed and approved the final manuscript.

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Table 1. Formaldehyde emission test results for all adhesive variants

Variant	Formaldehyde Emission (%)
LUF 1 (0,25 : 1 : 3)	11.15
LUF 2 (0,50 : 1 : 3)	10.61
LUF 3 (0,75 : 1 : 3)	9.86
Commercial UF	3.74

timization of curing conditions, or the addition of scavenging agents, are still required to further suppress the formaldehyde emissions [49].

Conclusion

In this study, lignin extracted from oil palm empty fruit bunches (EFB) was successfully incorporated into lignin–urea–formaldehyde (LUF) adhesive resin, as confirmed by FTIR analysis indicating the formation of new ether linkag-

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