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Performance Evaluation of Borax/Citric Acid-Modified OPEFB Lignin Bio-Composite Wood Adhesive: Water Resistance, Shear Strength, and Spectroscopic Characteristics

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ABSTRACT

The development of an environmentally friendly wood adhesive from oil palm empty fruit bunch (OPEFB) waste is essential to replace toxic synthetic phenol-formaldehyde (PF) adhesives. This study evaluated the bonding performance of a wood composite adhesive derived from OPEFB lignin-PVA bioresin synthesized through a dual crosslinking network (30% citric acid and 0–2.5% borax variation). Physical characterization of the bioresin showed a stable neutral pH (7.0) and an optimum thick-gel texture at sample B2 (1% borax). Adhesive performance evaluation, referring to SNI 0460-1989, showed a dry shear strength of 1.85 MPa and a wet shear strength (after 24-hour water immersion) of 1.42 MPa for formulation B2. These values exceed the minimum SNI shear-strength threshold (>1.0 MPa) under the 24-hour cold-water immersion condition tested, and the effect of borax concentration on shear strength was statistically significant (one-way ANOVA, $p < 0.001$). FTIR spectra showed an ester carbonyl band at 1760.99 cm^{-1}, consistent with citric acid esterification. Borax addition above 1.5% caused a sharp decline in bonding strength due to poor substrate wettability. Formulation B2 is a promising formaldehyde-free plywood adhesive, although longer-term water durability and benchmarking against commercial adhesives remain to be evaluated.

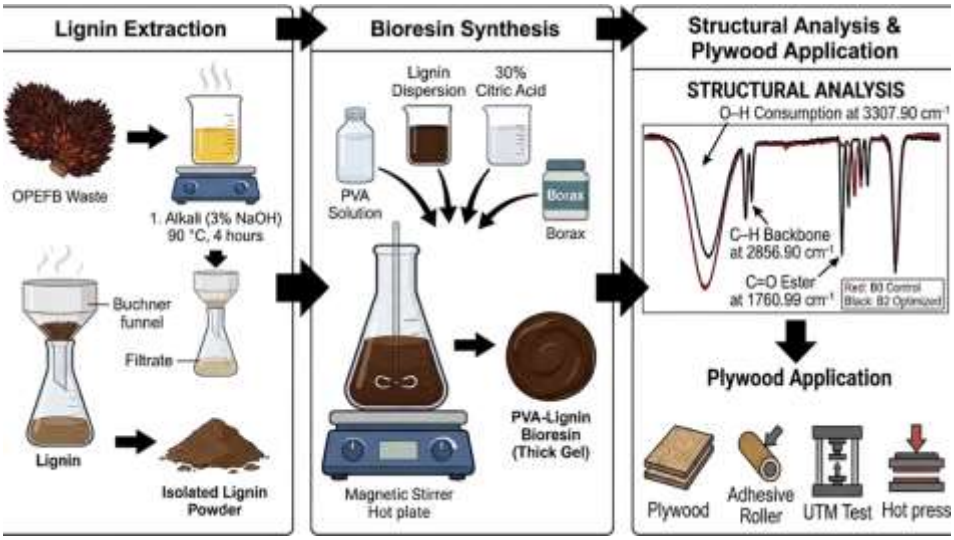
Keyword: Bioresin, OPEFB Lignin, Shear Strength, Water Resistance, Wood Adhesive

ABSTRAK

Pengembangan perekat kayu ramah lingkungan dari limbah Tandan Kosong Kelapa Sawit (TKKS) sangat krusial untuk menggantikan perekat sintetis beracun Fenol-Formaldehida (PF). Penelitian ini bertujuan menguji efektivitas performa perekat kayu komposit dari bioresin Lignin TKKS-PVA yang disintesis menggunakan jaringan crosslinking ganda (asam sitrat 30% dan variasi boraks 0%–2,5%). Karakterisasi fisik bioresin menunjukkan kestabilan pH netral (7,0) dan konsistensi tekstur gel kental optimum pada sampel B2 (1% boraks). Evaluasi kinerja perekat (mengacu pada standar SNI 0460-1989) menunjukkan kekuatan geser (shear strength) kondisi kering mencapai 1,85 MPa dan ketahanan air (wet shear strength setelah perendaman 24 jam) sebesar 1,42 MPa pada formulasi B2. Nilai ini melampaui ambang batas minimum kekuatan geser SNI (>1,0 MPa) pada kondisi perendaman air dingin 24 jam yang diuji, dan pengaruh konsentrasi boraks terhadap kekuatan geser signifikan secara statistik (ANOVA satu arah, $p < 0,001$). Spektra FTIR menunjukkan pita karbonil ester pada bilangan gelombang 1760,99 cm^{-1} yang konsisten dengan terjadinya esterifikasi asam sitrat. Penambahan boraks di atas 1,5% menyebabkan penurunan daya rekat akibat masalah pembasahan substrat (poor wettability). Formulasi B2 berpotensi sebagai perekat kayu lapis bebas formaldehida, meskipun ketahanan air jangka panjang dan pembandingan dengan perekat komersial masih perlu dievaluasi.

Kata Kunci: Bioresin, Lignin TKKS, Shear Strength, Ketahanan Air, Perekat Kayu

Graphical Abstract



1. Introduction

The wood adhesive industry still relies heavily on formaldehyde-based synthetic adhesives, particularly phenol-formaldehyde systems, which have long been used for their good bonding performance. However, dependence on fossil-based raw materials and emission concerns have driven growing research toward green adhesives or bioresins that are more environmentally friendly and derived from renewable sources [1–3]. In this context, lignin has become one of the most widely studied materials because it is an abundant natural aromatic polymer with a chemical structure that shows strong potential as a phenol substitute, and it has been extensively investigated in the development of wood-based adhesives and resins [3–7].

The utilization of lignin from oil palm biomass, including oil palm empty fruit bunch (OPEFB), is also becoming increasingly attractive because this material is available in large quantities as agro-industrial waste and has been reported to be isolable into lignin with properties suitable for biorefinery applications and wood-based materials [8–10]. Several studies have shown that lignin from oil palm biomass possesses chemical and thermal characteristics relevant for phenol substitution as well as for adhesive material development, so that its utilization not only adds value to the waste but also supports a more sustainable materials approach [4,9,10].

Nevertheless, the use of lignin as an adhesive material still faces several major obstacles, particularly its not-yet-optimal reactivity, limited bonding strength, and inadequate water resistance for wood applications that demand high stability [2,3,7]. For this reason, lignin is often combined with polyvinyl alcohol (PVA), a polymer well known for its ease of film formation, compatibility with biomass-based materials, and ability to help improve the flexibility and bonding performance of composite systems [11–14]. Various studies have also shown that PVA/lignin blends can be further developed to obtain better mechanical properties and stability through composition adjustment and material structure engineering [11,13–15].

However, PVA- and lignin-based systems still require reinforcement of the polymer network so that their performance does not readily decline upon exposure to moisture. Therefore, crosslinking strategies become an important part of bioresin development. The use of citric acid has been reported to improve the physical, thermal, mechanical, and adhesive performance properties of both PVA- and lignin-based systems through the formation of more stable bonds [11,16]. Meanwhile, borax/borate-based systems have also been shown to influence network structure, viscosity, and the adhesive properties of PVA-based materials, and thus have the potential to strengthen the character of the developed bioresin [17,18]. Accordingly, lignin-PVA modification through the addition of a

crosslinker is a relevant approach for producing a stronger, more stable, and more water-resistant formulation [11,14,16,17].

Based on these considerations, research on lignin-PVA bioresin is important to address the need for a more environmentally friendly, biomass-based wood adhesive that still delivers adequate performance. In addition, the utilization of lignin from OPEFB adds value to local biomass waste while simultaneously reinforcing the direction of sustainable materials research [3,4,9,10]. Therefore, research that varies the composition of lignin, PVA, and crosslinking agents such as borax or citric acid is highly relevant for identifying the optimum formulation suitable for wood bioresin applications [11,14,16,17].

Although lignin-based wood adhesives have been widely reported, most previous systems rely on technical or chemically modified lignins combined with a single crosslinking chemistry, such as lignin–glyoxal [5], lignin–PVOH blended with epoxy [6], phenolated kraft lignin [7], or citric acid-crosslinked hardwood lignin [2]. Dual crosslinking that combines a covalent ester network with borate complexation has so far been reported mainly for starch-based adhesives [16] and for boron-crosslinked starch–PVA and PVA–tannin blends [4,19], but, to the best of our knowledge, not for PVA blended with alkaline-extracted OPEFB lignin. The novelty of the present work therefore lies in (i) the use of OPEFB lignin isolated by a simple alkaline extraction without further chemical modification, (ii) the combination of citric acid esterification and borate complexation in a single water-based lignin–PVA formulation, and (iii) the systematic identification of the borax concentration window that balances crosslink density and substrate wettability on sengon plywood. Accordingly, this study aimed to evaluate the effect of borax concentration (0–2.5% w/w) in the presence of 30% citric acid on the physicochemical properties, dry and wet shear strength, and FTIR characteristics of an OPEFB lignin–PVA bioresin used as a wood adhesive.

2. Research Methodology

2.1. Materials and Bioresin Synthesis

Lignin was extracted from OPEFB using 3% NaOH at 90°C for 4 hours and precipitated at pH 3 with 10% H_2SO_4 (yield 13.71%). The bioresin was synthesized by mixing a 10% PVA solution with the lignin dispersion (7:3 w/w ratio on a dry-solids basis, i.e., solid PVA : solid lignin; sample PSL). Borax (sodium tetraborate decahydrate, $Na_2B_4O_7 \cdot 10H_2O$) variations (0%, 0.5%, 1.0%, 1.5%, 2.0%, and 2.5% w/w relative to the solids of PSL) were dissolved and added into the PSL, followed by the addition of a 5% citric acid solution (citric acid, $C_6H_8O_7$; final concentration 30% w/w relative to the solids of PSL) under constant stirring.

The formulation variables were selected as follows. The PVA/lignin ratio of 7:3 was chosen so that PVA remained the continuous, film-forming phase that governs wetting and film continuity, while lignin was incorporated at a level sufficient to contribute aromatic rigidity and renewable content. The citric acid level of 30% was selected to supply an excess of carboxyl groups for ester crosslinking of both PVA and lignin hydroxyls, in line with the use of citric acid as a crosslinker for PVA-based wood adhesives [4,20]. The borax range of 0–2.5% w/w was designed to span the uncrosslinked control (B0), the gelation region, and an over-crosslinked state, so that both the optimum and the onset of performance loss could be identified.

2.2. Adhesive Performance Testing on Wood Substrate

The bioresin was applied to the surface of sengon wood (lab-scale plywood) at a glue spread of 180 g/m^2 and hot-pressed at 140 °C under a pressure of 1.0 MPa (10 kg/cm²) for 5 minutes. Dry- and wet-condition lap shear strength (after 24-hour immersion in water at 25°C) was analyzed using a Universal Testing Machine (UTM) in accordance with SNI 0460-1989 and ASTM D906. For each formulation and test condition, five replicate specimens were tested.

2.3. Statistical Analysis

Shear strength data are reported as mean ± standard deviation (n = 5). The effect of borax concentration on dry and wet shear strength was evaluated separately by one-way analysis of variance (ANOVA), followed by Tukey's honestly significant difference (HSD) post-hoc test to identify significantly different formulations, at a significance level of $\alpha = 0.05$. Statistical analyses were performed using SPSS.

3. Results and Discussion

3.1. Physicochemical Properties and Adhesive Water Resistance Test

Observational data on the physical properties of the bioresin, together with the wood bonding strength evaluation results, are presented in Table 1.

Table 1. Physicochemical characteristics and plywood bonding performance of the bioresin

Formulation	Borax (% w/w)	Bioresin pH	Texture / Gelation	Dry Shear Strength (MPa)	Wet Shear Strength (MPa)	Evaluation Standard (SNI 0460-1989)
B0	0.0	9.0	Dark Brownish, Liquid	0.42 ± 0.05^{a}	0.00^{a} (Delamination)	Does Not Meet Standard
B1	0.5	7.0	Thick Gel	1.35 ± 0.08^{d}	0.95 ± 0.06^{d}	Meets Interior Grade Requirement
B2	1.0	7.0	Thick Gel (Optimum)	1.85 ± 0.10^{f}	1.42 ± 0.09^{f}	Meets Exterior Grade Requirement (>1.0 MPa)
B3	1.5	7.0	Dense Thick Gel	1.60 ± 0.09^{e}	1.18 ± 0.07^{e}	Meets Exterior Grade Requirement
B4	2.0	7.0	Solid / Rigid	0.92 ± 0.07^{c}	0.55 ± 0.05^{c}	Does Not Meet Standard
B5	2.5	7.0	Hard Solid	0.58 ± 0.06^{b}	0.30 ± 0.04^{b}	Does Not Meet Standard

Note: Values are mean ± SD (n = 5). Different superscript letters within the same column indicate significant differences between formulations (Tukey's HSD, $p < 0.05$).

Table 1 shows a consistent pattern among borax concentration, the physical character of the bioresin, and the resulting bonding performance, with relatively small standard deviation values across all formulations (ranging from 0.04–0.10 MPa), indicating that the synthesis and testing process was reasonably reproducible. One-way ANOVA showed that borax concentration had a significant effect on both dry shear strength ($F(5, 24) = 276.81$, $p < 0.001$) and wet shear strength ($F(5, 24) = 428.15$, $p < 0.001$). Tukey's HSD test further showed that all pairwise differences between formulations were significant ($p < 0.05$), as indicated by the different superscript letters in Table 1. The formulation without a crosslinker (B0) underwent total delamination upon water immersion, with its wet shear strength dropping to 0.00 MPa even though its dry shear strength of 0.42 MPa was still measurable. This total failure occurred because the bonds holding the PVA-lignin matrix together in B0 consisted solely of hydrogen bonds between hydroxyl groups, which are reversible, low in bond energy, and very easily broken (hydrolyzed) once water molecules diffuse into the adhesive layer. Without a permanent covalent network holding the resin structure together, water rapidly displaces/substitutes the intermolecular hydrogen bonds between PVA and lignin, causing the adhesive layer to lose structural integrity and the plywood to delaminate completely. The physical character of the B0 bioresin, still a thin, dark brownish liquid, further confirms that no three-dimensional network (gelation) had yet formed at this stage. This condition is also reflected in the still-alkaline pH of the B0 bioresin (9.0), a result of residual NaOH from the lignin extraction step that had not yet been neutralized by an acidic crosslinker; this alkaline condition also slows the esterification reaction rate between the carboxyl groups of citric acid and the hydroxyl groups of PVA/lignin, so that without the addition of a crosslinker, the likelihood of covalent bond formation becomes very small.

The addition of citric acid and borax (B1–B3) lowered the bioresin pH to neutral (7.0) and significantly increased the shear strength values compared to B0, under both dry and wet conditions. This improvement occurs through two crosslinking mechanisms that work simultaneously and complement each other. First, citric acid, as a tricarboxylic acid, undergoes an esterification reaction with the abundant –OH groups on the PVA chain as well as with the aliphatic and phenolic hydroxyl groups of lignin during hot-pressing at 140 °C. Among the lignin hydroxyls, the aliphatic –OH groups located on the propanoid side chains (mainly at the Cα and Cγ positions) are generally more reactive toward esterification than the phenolic –OH groups, which are sterically hindered by adjacent methoxy groups and less nucleophilic because the oxygen lone pair is delocalized into the aromatic ring [21]; esterification of lignin is therefore expected to proceed predominantly through its aliphatic hydroxyls. This reaction forms covalent ester bonds that are permanent and far more resistant to hydrolysis than the hydrogen bonds in B0, because the bond energy of a covalent C–O bond is much higher than that of a hydrogen bond. In addition, because citric acid has three carboxyl groups, a single citric acid molecule can potentially bind more than one polymer chain at once, thereby also contributing to the formation of a three-dimensional network. Second, borax ($Na_2B_4O_7 \cdot 10H_2O$) hydrolyzes in water to boric acid and tetrahydroxyborate ions, $B(OH)_4^-$, which are known to complex with the 1,3-diol units of the PVA chain to form six-membered cyclic borate esters (di-diol complexes) that are reversible yet fairly stable under neutral conditions [4,19]. This interaction markedly increases the solution viscosity and the crosslink density of the resin, as seen in the change in texture from a thin liquid (B0) to a thick gel (B1–B3). The combination of these two mechanisms is proposed to generate a dual network, consisting of a permanent covalent ester network from citric acid and a more dynamic borate network, which may be described as an interpenetrating-type polymer network. It should be noted, however, that the present mechanical and FTIR data do not allow the interpenetrating network structure or the borate–diol complexes to be conclusively established; confirmation would require complementary techniques such as ^{11}B NMR spectroscopy, gel-fraction and swelling measurements, or rheological analysis. Such a dual network would render the resin matrix less hydrophilic, denser, and more resistant to water penetration than the B0 resin, which relies solely on hydrogen bonding. The wet-strength retention ratio (wet/dry shear strength) for B1 was recorded at 70.4% (0.95/1.35 MPa), a drastic improvement over B0's 0% retention, and serves as a direct quantitative indicator of the effectiveness of this dual crosslinking system in resisting water-induced degradation.



Figure 1. Physical appearance of the dark, thick-gel texture of the B5 sample (2.5% borax formulation)

Peak performance was achieved by sample B2 (1.0% borax), with a dry shear strength of 1.85 MPa and a wet shear strength of 1.42 MPa, both the highest values among all formulations tested and exceeding the minimum SNI 0460-1989 shear-strength threshold (>1.0 MPa) under the 24-hour cold-water immersion condition used in this study. The wet-strength retention ratio for B2 reached 76.8% (1.42/1.85 MPa), the highest among all samples, showing that for this formulation the strength loss caused by 24-hour water immersion could be limited to only about a quarter of its initial dry strength. At this 1.0% borax concentration, the bioresin texture sits at the "optimum thick gel" point — viscous enough to have a high crosslink density, yet still fluid enough to wet and effectively penetrate the pores and vascular structure of the relatively porous and soft sengon wood substrate. This penetration of resin into the wood pores produces good mechanical interlocking between the adhesive and the substrate, while the crosslink network is still dense enough to provide high internal cohesive strength and water resistance within the adhesive layer itself. In other words, B2 represents the optimum trade-off point among three competing factors: resin viscosity, crosslink density, and substrate wetting ability — three factors that, as explained in the following paragraph, cannot all be

increased simultaneously without limit, because improving one factor ultimately comes at the expense of another.

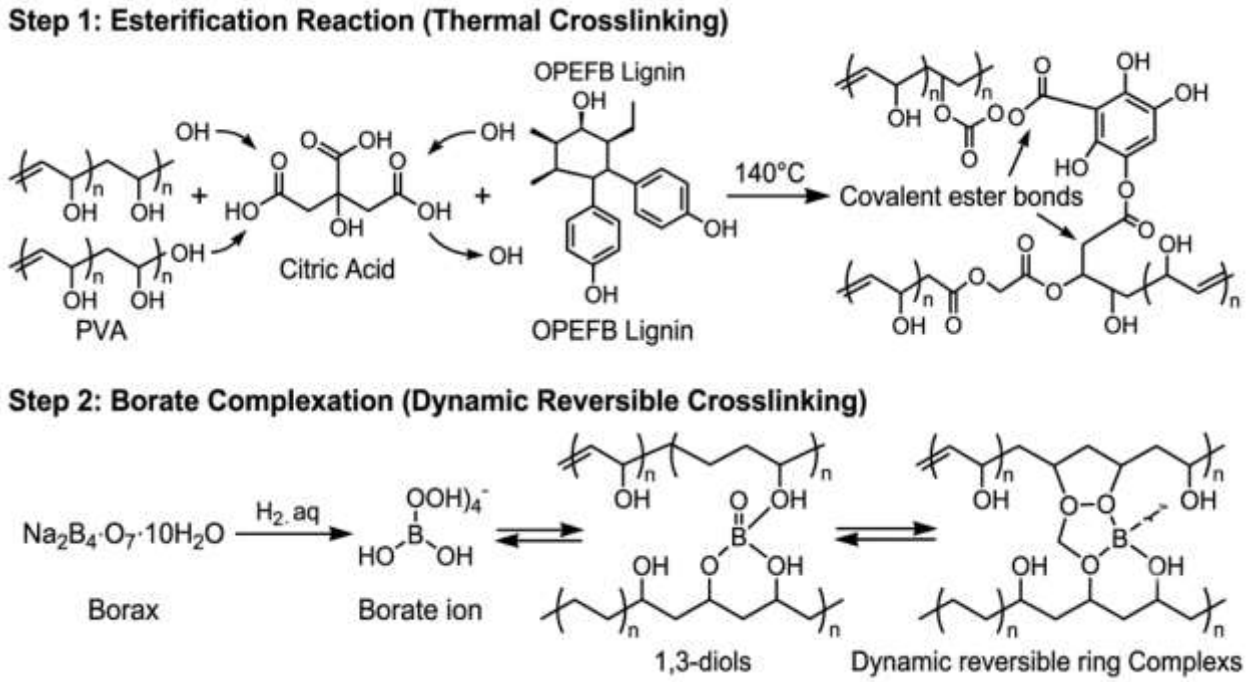


Figure 2. Schematic of the dual-crosslinking mechanisms: (Step 1) Thermal esterification crosslinking at 140°C, and (Step 2) dynamic reversible borate complexation.

In contrast, at a borax concentration of 1.5% (B3), shear strength began to decline (dry: 1.60 MPa; wet: 1.18 MPa; retention 73.8%), although this formulation still met the SNI exterior-grade requirement. The decline continued sharply for B4 (2.0%; dry 0.92 MPa, wet 0.55 MPa, retention 59.8%) and reached its lowest point at B5 (2.5%; dry 0.58 MPa, wet 0.30 MPa, retention 51.7%), both of which even failed to meet the minimum SNI standard for either exterior- or interior-grade adhesive. This progressive decline is caused by the resin texture becoming increasingly dense and rigid (from a "dense thick gel" at B3 to "solid/rigid" at B4 and "hard solid" at B5) as the borax concentration increases, as a result of excessive borate-bridge density along the PVA chain. Mechanistically, two factors explain this decline. First, a resin that is too viscous or dense prior to application is unable to flow and penetrate effectively into the pores of the sengon wood (poor wettability); an increase in viscosity with increasing boric acid/borate content has likewise been reported for boron-crosslinked PVA–starch and PVA–tannin wood adhesives [4,19], so that the interfacial contact area between the adhesive and the substrate is drastically reduced and mechanical interlocking between the adhesive and substrate becomes very poor — in other words, the high intrinsic strength of the polymer network can no longer be effectively transferred to the wood substrate. Second, an excessively dense borate network tends to make the adhesive layer more brittle and less flexible, making it more prone to microcracking under shear loading compared with the more elastic network in B2. The much sharper decline in wet shear strength relative to dry shear strength for B4 and B5 (seen in the continuously decreasing retention ratio: from 76.8% at B2 to only 51.7% at B5) also indicates that at high borax concentrations, the reversible borate-diol bonds become more susceptible to hydrolytic reversal upon prolonged contact with water, because the proportion of dynamic borate bonds is far more dominant than the permanent covalent ester bonds from citric acid in these formulations. Overall, these findings confirm that the relationship between crosslinker concentration and adhesive performance is non-linear and resembles an optimum curve peaking at B2, making the selection of an appropriate borax concentration a critical factor: excessive borax addition is in fact counterproductive, as it sacrifices substrate wettability and network flexibility for a higher crosslink density that is not proportionally matched by an increase in performance.

From a practical perspective, two limitations of the present evaluation should be noted. First, a commercial adhesive was not included as a control in the present test series. As an indirect benchmark, the wet shear strength of B2 (1.42 MPa) is comparable to the value of 1.22 ± 0.06 MPa reported for a dual-crosslinked (ester/borate) starch-based wood adhesive after water soaking, whereas its dry shear strength (1.85 MPa) is lower than the 5.34 ± 0.24 MPa reported for that system [16]; such comparisons must be interpreted with

caution because of differences in wood species, specimen geometry, and test protocols. Second, water resistance was evaluated only after 24-hour immersion at 25 °C. Because the borate–diol bonds are reversible and the retention ratio decreased at higher borax contents, longer-term water exposure (e.g., 72-hour cold-water soaking, boiling-water, or cyclic soak–dry tests) is required before long-term exterior durability can be claimed. Further work should therefore include a commercial adhesive (e.g., PVAc, UF, or PF) bonded and tested under identical conditions, together with extended water-exposure tests.

3.2. FTIR Spectroscopic Characterization Analysis

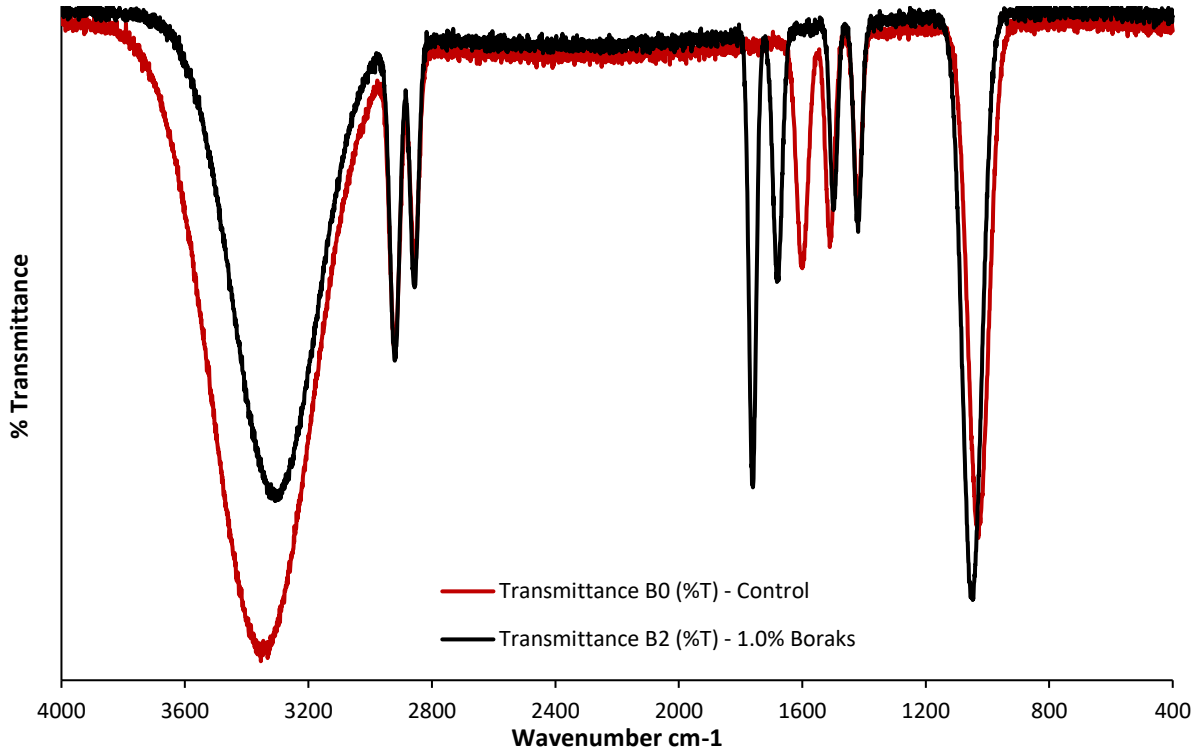


Figure 3. FTIR spectrum of the optimal B2 formulation (1.0% borax), with the specific ester carbonyl peak highlighted at 1760.99 cm^{-1}.

To examine the chemical mechanism proposed based on the mechanical data in Section 3.1, FTIR spectroscopic analysis was performed on formulation B2 as the sample with the optimum performance. The FTIR spectrum of B2 shows a specific absorption band for the ester carbonyl group (C=O) at a wavenumber of 1760.99 cm^{-1}. This band lies at the high-wavenumber end of the carbonyl stretching region of aliphatic esters (approximately 1715–1760 cm^{-1}) and is consistent with esterification of citric acid with the –OH groups of lignin and PVA during hot-pressing at 140 °C. Because the C=O stretching of free carboxylic acid groups (approximately 1700–1720 cm^{-1}) and of cyclic anhydride intermediates of citric acid (approximately 1780–1850 cm^{-1}) occurs nearby, this band is interpreted as supporting evidence of ester formation rather than as conclusive proof. This finding supports the covalent ester bond formation mechanism proposed in Section 3.1 as a major contributor to the increased water resistance of formulations B1–B3. Besides the ester band, the aliphatic C–H stretching absorption at 2856.90 cm^{-1} indicates that the backbone structure of the polymer matrix (PVA and lignin) remained well preserved and did not undergo significant thermal or oxidative degradation during either the synthesis or hot-pressing process; this is important because it shows that the observed increase in bonding strength was not caused by detrimental structural changes to the base polymer. Furthermore, the bands at 1680.21 cm^{-1} (conjugated C=O of lignin units, overlapping with aromatic skeletal vibrations) and 1498.30 cm^{-1} (aromatic skeletal C=C stretching) reflect the preservation of the lignin aromatic framework after the synthesis and hot-pressing process. The presence of these aromatic rings provides additional aromatic cohesive strength to the adhesive layer through inter-ring (π–π stacking) interactions and is consistent with the role of lignin as a renewable aromatic component with the potential to replace phenol in conventional phenolic resins [2,4]. Overall, this FTIR data reinforces the structure-property relationship observed in the mechanical testing in Section 3.1: the formation of a covalent ester network (supported by the 1760.99 cm^{-1} band), likely acting together with borate complexation, without compromising the integrity of the polymer backbone or the lignin aromatic structure, appears to be the main factor behind the high shear strength and water resistance of

formulation B2. The consistency between the spectroscopic evidence and the mechanical data suggests that the optimum performance of B2 can be rationalized by the proposed dual-crosslinking mechanism.

The broad O–H stretching band at 3307.90 cm^{-1} provides complementary information on the proposed crosslinking. Because both citric acid esterification and borate complexation consume hydroxyl groups of PVA and lignin, a reduction in the relative intensity of this band would be expected for the crosslinked formulations compared with the uncrosslinked control. However, an FTIR spectrum of B0 was not recorded under identical conditions in the present study, so this reduction could not be quantified. The persistence of a strong O–H band in B2 indicates that a substantial fraction of hydroxyl groups remained unreacted, which is consistent with partial crosslinking and with the reversible nature of borate–diol bonds. Comparative FTIR analysis of B0–B5, supported by techniques such as ^{11}B NMR spectroscopy and gel-fraction measurements, is therefore recommended to confirm hydroxyl consumption and borate complexation. Accordingly, the formation of borate–diol complexes and of an interpenetrating-type network is presented here as the most plausible interpretation rather than as a conclusively established structure.

4. Conclusion

An OPEFB lignin- and PVA-based bioresin modified with a dual crosslinker system (30% citric acid and 1% borax) significantly improved ($p < 0.05$) plywood bonding quality compared with the bioresin without a crosslinker. Formulation B2 was shown to produce the best water resistance (wet shear strength of 1.42 MPa), a stable neutral pH (7.0), and an ester carbonyl band at 1760.99 cm^{-1} consistent with citric acid crosslinking. Borax concentrations above 1.5% instead reduced adhesive performance, attributed mainly to reduced substrate wettability and a more brittle network, making the selection of an appropriate crosslinker concentration a key factor in formulation success. This bioresin shows potential as a formaldehyde-free alternative to PF adhesives; however, long-term water durability testing and direct benchmarking against a commercial adhesive under identical conditions are required before industrial application.

6. Acknowledgments

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7. Conflict of Interest

The authors declare that there is no conflict of interest in this research.

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