

SEPARATION AND MODIFICATION OF
LIGNIN FROM OIL PALM EMPTY FRUIT
BUNCH AND ITS APPLICATION AS A
PHENOLIC LIGNIN ADHESIVE IN WOOD
COMPOSITES

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ABSTRAK

Lignin ialah molekul polifenol yang boleh digunakan sebagai pengganti separa fenol dalam komposisi resin fenolik. Oleh kerana lignin mempunyai struktur fenolik dan boleh didapati dengan mudah dan banyak, ia menjadikan lignin komponen berasaskan bio yang baik untuk menggantikan fenol dalam resin fenolik. Walau bagaimanapun, kereaktifan lignin adalah rendah dengan kehadiran formaldehid apabila resin fenol formaldehid (PF) disediakan. Kajian ini dijalankan bertujuan untuk meningkatkan kereaktifan lignin melalui pengubahsuaian kimia (fenolasi) dan menggunakan lignin dan lignin yang diubah suai sebagai pengganti separa untuk fenol dalam penghasilan pelekat kayu PF. Dalam kajian ini, kraft lignin (KL) telah diekstrak melalui proses pulpa kraf daripada Tandan Buah Kosong Kelapa Sawit (OPEFB). KL diekstrak menggunakan dua pelarut, 19% sodium hidroksida dan 25% sodium sulfida berdasarkan bahan mentah, dengan nisbah gentian sawit kepada pelarut ialah 1:8, suhu dan masa pepulpaan masing-masing ialah 170°C dan 3 jam. Kemudian, KL diubah suai menggunakan kaedah fenolasi, di mana KL bertindak balas dengan fenol dalam nisbah yang berbeza (1:1, 1:1.5, dan 1:2) dalam medium berasid (sulfurik asid), mengakibatkan penurunan yang ketara dalam kumpulan alifatik hidroksil dan peningkatan kumpulan hidroksil fenolik lignin tersebut. Untuk penyediaan lignin kraf terfenolasi (PKL), suhu tindak balas dikaji dari 90 hingga 130°C, dan masa tindak balas dikaji dari 100 hingga 140 minit. KL dan PKL dianalisa menggunakan FTIR, ¹HNMR, dan analisa unsur. Ciri-ciri fizikal seperti kandungan kelembapan, kandungan pepejal, dan kandungan abu KL dan PKL turut dikaji. Kedua-dua KL dan PKL telah ditindakbalas kepada resin PF dengan menggantikan peratusan tertentu fenol (10-50%). Resin kraft lignin fenol formaldehid (KLFF) yang disintesis dan resin kraft lignin fenol formaldehid (PKLFF) terfenolasi juga dibandingkan dengan resin PF. FTIR dan DSC digunakan analisa karakter semua resin dihasilkan. Sifat fizikal semua resin, seperti kelikatan, masa gel, kandungan pepejal, kandungan fenol bebas, dan kandungan formaldehid bebas telah dikaji. Penyerapan air, ketebalan swelling, kandungan formaldehid bebas, dan kekuatan ricih panel papan lapis yang diikat dengan pelekat ini dianalisa mengikut kaedah piawai. Keputusan kajian menunjukkan bahawa proses fenolasi meningkatkan kereaktifan lignin serta sifat pelekat PLFF. Kekuatan ricih ikatan papan lapis dengan resin PKLFF memenuhi kriteria spesifikasi piawaian yang berkaitan dan jauh lebih baik daripada ikatan panel dengan resin PF. Kesan penggantian KL dan PKL dalam resin fenolik untuk kekuatan ricih dan pelepasan formaldehid papan lapis telah juga dikaji. Kesan penggantian fenol dengan lignin dan lignin terfenolasi, suhu penekan panas dan masa penekanan semasa pencamtuman papan telah dikaji dan dioptimumkan untuk mendapatkan masa dan suhu penekanan yang terbaik untuk prestasi pelekat papan lapis. Program metodologi permukaan tindak balas (RSM) telah digunakan bagi mengoptimumkan suhu tekan panas dan masa tekan untuk proses penyediaan papan lapis. Eksperimen telah dijalankan dalam julat masa menekan dari 50 saat hingga 250 saat dan pada julat suhu 120 hingga 200°C. Keputusan eksperimen menunjukkan peningkatan ketara dalam kekuatan ricih papan lapis dan penurunan dalam kandungan formaldehid bebas. Peratusan optimum penggantian fenol dengan PKL dalam pelekat PF ialah 30% dan dengan KL ialah 20%. Kekuatan ricih maksimum 3.2 MPa dan pelepasan formaldehid minimum 0.333 mg/L didapati dalam pelekat PKLFF. Penyelidikan ini menunjukkan penggunaan PKL sebagai pengganti fenol yang boleh diperbaharui dalam formulasi resin fenolik dan amat dicadangkan bahawa lignin terfenolasi boleh digunakan sebagai produk bernilai tambah tinggi dalam aplikasi pelekat kayu.

ABSTRACT

Lignin is a polyphenolic molecule that can be used as a partial phenol substitution in phenolic resin compositions. Due to the phenolic structure and huge available quantities of lignin, lignin is an excellent bio-based component for phenol substitution in phenolic resin. However, the reactivity of lignin is low with formaldehyde when phenol formaldehyde (PF) resin is prepared. The present work aims to increase the lignin reactivity by chemical modification (phenolation) and utilize the lignin and modified lignin as a partial substitute for phenol in PF wood adhesive. In this work, lignin was extracted by the kraft pulping process from oil palm empty fruit bunch (OPEFB), and the extracted lignin is known as kraft lignin (KL). In the kraft pulping method, two solvents were used, 19% NaOH and 25% Na₂S based on raw materials, with fiber to solvent ratio of 1:8, the pulping temperature and time were 170°C and 3 h, respectively. Then, KL was modified using the phenolation method, in which the KL was reacted with phenol in different ratios (1:1, 1:1.5, and 1:2) in an acidic medium (H₂SO₄), resulting in a considerable drop in aliphatic hydroxyl groups and a rise in phenolic hydroxyl groups of that lignin. For the preparation of phenolated kraft lignin (PKL), the reaction temperature varied from 90 to 130°C, and the reaction time varied from 100 to 140 min. KL and PKL were characterized using FTIR, ¹HNMR, and elemental analysis. The physical properties such as moisture, solid, and ash content of KL and PKL were also studied. Both KL and PKL were introduced to the PF resin by substituting a certain percentage (10-50%) of phenol. The synthesized kraft lignin phenol formaldehyde (KLPF) and phenolated kraft lignin phenol formaldehyde (PKLPF) resins were also compared to PF resin. FTIR and DSC were used to characterize all the resins. The physical properties of all resins, such as viscosity, gel time, solid content, free phenol content, and free formaldehyde content, were studied. The water absorption, thickness swelling, formaldehyde emission, and shear strength of the plywood panels bonded with these adhesives were measured according to the standard method. The outcomes demonstrated that the phenolation process improved the lignin's reactivity as well as the PLPF's adhesive properties. The shear strength of plywood bonding with PKLPF resin meets the criteria of the relevant standards specifications and is substantially superior to that of panels bonding with PF resin. The effects of KL and PKL substitutions in resin for shear strength and formaldehyde emission of plywood were studied. The effects of phenol substitution with lignin or phenolated lignin, the hot press temperature and the pressing time were investigated and optimized to obtain the best possible pressing time and temperature for the performance of plywood adhesive. The response surface methodology (RSM) was used to optimize the hot-press temperature and press time for the plywood preparation process. The experiments have been conducted in the pressing time range from 50 s to 250 s and at temperatures ranging from 120 to 200°C. The experimental results showed a significant increase in plywood shear strength and a decrease in formaldehyde emission. The optimized percentage of phenol substitution with PKL in the PF adhesive was 30% and with KL it was 20%. The maximum shear strength of 3.2 MPa and minimum formaldehyde emission of 0.633 mg/L were found in the PKLPF adhesive. This research demonstrated the use of PKL as a renewable replacement of phenol in phenolic resin formulation and strongly suggested that phenolated lignin can be used as a high-value-added product in wood adhesive applications.

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