DEVELOPMENT OF BIO-ADHESIVES FOR COMPOSITES WOOD USING NATURAL RUBBER LATEX AND MODIFIED STARCH WITH CROSSLINKERS

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SUPERVISOR'S DECLARATION

I hereby declare that I have checked this thesis, and, in my opinion, this thesis is adequate in terms of scope and quality for the award of the degree of Doctor of Philosophy.

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I hereby declare that the work in this thesis is based on my original work except for quotations and citations which have been duly acknowledged. I also declare that it has not been previously or concurrently submitted for any other degree at Universiti Malaysia Pahang or any other institutions.

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ABSTRAK

Pelekat sintetik yang digunakan dalam industri kayu komposit adalah satu perkara yang membimbangkan kerana pelepasan formaldehid gas karsinogenik, peningkatan pencemaran alam sekitar dan kehabisan bahan api fosil. Oleh itu, membangunkan pelekat bebas formaldehid dan mesra alam yang diperoleh daripada sumber boleh diperbaharui adalah penting. Penyelidikan ini memberi tumpuan kepada pembangunan dan penggunaan bio-pelekat berdasarkan dua biopolimer: kanji beras bersilang (RS) dan lateks getah asli (NRL). Kanji beras telah dikaitkan silang dengan glioksal, polimer 4,4"-diphenylmethane diisocyanate (pMDI), dan dimethylol dihydroxy ethylene urea (DMDHEU) untuk memperbaiki sifat hidrofiliknya sebelum diadun dengan NRL. Interaksi kimia kanji beras dan penghubung silang telah disiasat menggunakan spektroskopi inframerah transformasi total dilemahkan-Fourier (ATR-FTIR). Hidrofobisiti kanji beras asli dan kanji beras bersilang diukur menggunakan ukuran sudut sentuhan. Dengan pengadunan, bio-pelekat dengan kandungan berat berbeza-beza RS dan NRL bersilang telah dibangunkan, dan sifat fizikalnya seperti masa gel, kelikatan dan kandungan pepejal telah dicirikan. Akibatnya, bio-pelekat yang dirumus digunakan untuk pembuatan papan lapis melalui penekanan panas pada 120°C selama 5 minit, dan spesimen papan lapis dicirikan secara fizikal dan mekanikal dari segi penyerapan air (WA), bengkak ketebalan (TS), modulus pecah (MOR), modulus keanjalan (MOE) dan ikatan dalaman (IB). Kestabilan haba dan sifat permukaan papan lapis diperhatikan melalui analisis termogravimetrik (TGA) dan mikroskopi elektron pengimbasan (SEM). Sifat fizikal dan mekanikal papan lapis diukur mengikut piawaian ASTM, ISO dan IS. Perisian pakar reka bentuk DX7 digunakan untuk menganalisis pembolehubah penting yang mempengaruhi formulasi bio-pelekat dalam ikatan dalaman papan lapis. ATR-FTIR mengesahkan kehadiran fungsi glioksal, isosianat dan DMDHEU dalam makromolekul kanji, menghasilkan prestasi bio-pelekat yang dipertingkatkan. Kanji bersilang glioksal, mempunyai sifat hidrofobik yang luar biasa, dengan CA 91.45° pada 3 minit. Bio-pelekat Iso A didapati mempunyai kelikatan tertinggi 8270 mPa.s. Jenis pelekat bio Gly B mempunyai masa gel terpendek 2.80 min dan kandungan pepejal tertinggi sebanyak 46%, kandungan pepejal yang lebih tinggi mempercepatkan masa gel. Papan lapis yang diikat dengan bio-pelekat Gly B menunjukkan pembengkakan ketebalan (TS) paling rendah sebanyak 11% dan penyerapan air (WA) sebanyak 35%. Papan lapis yang diikat dengan bio-pelekat Gly B mempunyai modulus pecah (MOR) tertinggi sebanyak 72 MPa, modulus keanjalan (MOE) sebanyak 9574 MPa, dan ikatan dalaman (IB) sebanyak 2.2 MPa sepadan dengan ISO 12466-2-2007 dan keperluan standard IS 303. Tambahan pula, analisis haba menunjukkan bahawa papan lapis yang diikat dengan bio-pelekat Gly A mempunyai suhu penurunan berat badan yang lebih tinggi, menunjukkan bahawa penambahan kanji beras bersilang meningkatkan kestabilan terma oksidatif bio-pelekat. Bagi sifat permukaan, papan lapis yang diikat dengan Gly B menunjukkan tompok lompang yang kurang dan kelihatan dalam taburan rawak di antara matriks gentian kayu. Berdasarkan model regresi RSM-CCD, parameter ideal formulasi bio-pelekat yang mempengaruhi ikatan dalaman papan lapis ialah 73.47°C, pH 7.33, dan 0.35% glioksal. Menurut penemuan, papan lapis yang diikat dengan bio-pelekat Gly B memberi kesan ketara kepada kualiti sifat lekatan berbanding dengan formulasi bio-pelekat yang lain. Kajian ini menunjukkan bahawa biopolimer seperti kanji beras silang silang dan NRL boleh membantu untuk menghapuskan penggunaan pelekat sintetik berbahaya sepenuhnya.

ABSTRACT

Synthetic adhesives used in the composite wood industries are a matter of concern due to the emission of carcinogenic gas formaldehyde, increased environmental pollution and the depletion of fossil fuels. Therefore, developing a formaldehyde free and eco-friendly adhesive derived from renewable resources is essential. The present research focuses on developing and applying bio-adhesives based on two biopolymers: crosslinked rice starch (RS) and natural rubber latex (NRL). The rice starch was crosslinked with glyoxal, polymeric 4,4"- diphenylmethane diisocyanate (pMDI), and dimethylol dihydroxy ethylene urea (DMDHEU) to improve its hydrophilicity nature before blending with NRL. The chemical interaction of rice starch and crosslinkers was investigated using attenuated total reflectance-Fourier transform infrared spectroscopy (ATR-FTIR). The hydrophobicity of native rice starch and crosslinked rice starch was measured using contact angle measurements. By blending, a bio-adhesive with varying weight content of crosslinked RS and NRL was developed, and its physical properties such as gel time, viscosity, and solid content were characterized. Consequently, formulated bio-adhesives were applied for plywood manufacturing via hot pressing at 120°C for 5 minutes, and plywood specimens were characterized physically and mechanically in terms of water absorption (WA), thickness swelling (TS), modulus of rupture (MOR), modulus of elasticity (MOE) and internal bonding (IB). The thermal stability and surface property of the plywood were observed through thermogravimetric analysis (TGA) and scanning electron microscopy (SEM). The physical and mechanical properties of the plywood are measured following ASTM, ISO, and IS standards. The design expert software DX7 was used to examine the significant variables that influence bio-adhesive formulations in plywood internal bonding. ATR-FTIR confirmed the presence of glyoxal, isocyanate, and DMDHEU functionalities in starch macromolecules, resulting in enhanced bio-adhesive performance. The glyoxal crosslinked starch, has outstanding hydrophobic nature, with an CA of 91.45° at 3 minutes. The bio-adhesive Iso A was discovered to have the highest viscosity of 8270 mPa.s. The bio-adhesive type Gly B has the shortest gel time of 2.80 min and the highest solid content of 46%, the higher the solid content, the faster the gel time. The plywood bonded with Gly B bio-adhesive shows the lowest thickness swelling (TS) of 11% and water absorption (WA) of 35%. Plywood bonded with Gly B bio-adhesive had the highest modulus of rupture (MOR) of 72 MPa, modulus of elasticity (MOE) of 9574 MPa, and internal bonding (IB) of 2.2 MPa corresponded to the ISO 12466-2-2007 and IS 303 standard requirements. Furthermore, the thermal analysis showed that plywood bonded with Gly A bio-adhesive has a higher weight loss temperature, indicating that the addition of crosslinked rice starch improves the oxidative thermal stability of bio-adhesive. As for the surface property, the plywood bonded with Gly B showed less void and visible patches in a random distribution in between the wood fibres matrices. Based on the RSM-CCD regression model, the ideal parameters of bio-adhesive formulation that influence the internal bonding of plywood were 73.47°C, pH 7.33, and 0.35% glyoxal. According to the findings, the plywood bonded with Gly B bio-adhesive significantly impacted the quality of adhesion properties compared to other bio-adhesive formulations. This study shows that biopolymers like crosslinked rice starch and NRL can help to eliminate the use of hazardous synthetic adhesives completely.

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K	Number of parameters
α-helix	Alpha helix
A	Area
B	Breadth of specimen
D	Average depth of specimen
L	Distance between knife edges on where the sample was placed
MPa	Megapascal
mPa. s	Millipascal second
Cm	Centimetre
kN	Kilonewton
°C	Degree Celsius
P	Breaking load
=	Equal
F	Force
G	Grams
Hr	Hours
W_1	Sample weight before soaking
W_2	Sample wight after soaking
p	Significant
3D	Three-dimensional surface
≤	Less than or equal to
≥	More than or equal to
L	Litre
ml	Millilitres
-	Negative sign
+	Positive sign
Min	Minutes
N	Normality
%	Percentage
Σ	Sigma
e	Standard error

T_1	Thickness before immersion in water
T_2	Thickness after immersion in water
mm	Millimetres
R^2	The coefficients of determination
Wt.%	Weight of solvent
Y	Response
Kg/m^3	Kilogram/metre cube
V	Volume
Λ	Wavelength

LIST OF ABBREVIATIONS

ASTM	American Society for Testing and Materials
ATCM	American Toxic Control Measure
API	Aqueous Polymer Isocyanate
CARB	California Air Resource Board
CA	Contact Angle
CCD	Central Composite Design
DMDHEU	Dimethylol Dihydroxy Ethylene Urea
ATR-FTIR	Attenuated Total Reflectance-Fourier Transform Infrared Spectroscopy
GA	Glutaraldehyde
HDF	High Density Fibreboard
HA	High Ammonia
HCl	Hydrochloric Acid
IB	Internal Bonding
ISO	International Organization for Standardization
IS	Indian Standard
IARC	The International Agency for Research on Cancer
KH570	Methacryloxypropyl, Trimethoxy Silane
LVL	Laminated Veneer Lumber
MOE	Modulus of Elasticity
MOR	Modulus of Rupture
MF	Melamine Formaldehyde
MDF	Medium Density Fibreboard
NRL	Natural Rubber Latex
NaOH	Sodium Hydroxide
OSB	Oriented Stranded Board
PRF	Phenol Resorcinol Formaldehyde
PB	Particle Board
pH	Potential of Hydrogen
PURs	Polyurethanes
PF	Phenol Formaldehyde

pMDI	Diphenylmethane Diisocyanate
PVAc	Polyvinyl Acetate
RS	Rice Starch
RSM	Response Surface Methodology
SEM	Scanning Electron Microscope
TGA	Thermogravimetric Analysis
TS	Thickness Swelling
UF	Urea Formaldehyde
VOC	Volatile Organic Compound
WA	Water absoprtion

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