MODIFICATION OF LIGNIN FROM PULPING BLACK LIQUOR FOR PRODUCTION OF LIGNIN-PHENOL-FORMALDEHYDE ADHESIVE

By

LIM KAH YEN

Thesis submitted to School of Graduate Studies,
Universiti Putra Malaysia in Fulfilment of the Requirements for the Degree of Doctor of Philosophy

February 2023

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February 2023

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Black liquor, a complex pulping by-product, contains lignin residues, degraded carbohydrates, and inorganic constituents. To release cellulose fibres, lignin, hemicelluloses, and other wood extractives were removed from wood during pulping. It is a main pollutant from conventional paper mills. However, sustainability and environmental awareness have drawn attention to black liquor's main ingredient, lignin. Lignin could replace non-renewable chemical feedstocks. The study aimed to investigate the potential to utilise lignin from oil palm empty fruit bunch black liquor (OPEFB-BL) from Preconditioning Refiner Chemical- Recycle Bleached Mechanised Pulping (PRC-RBMP) and increase its chemical reactivity for the production of lignin-phenol-formaldehyde (LPF) adhesive. This study had a four-part design. The first part of the study determined OPEFB-BL composition and characteristics from PRC-RBMP. Secondly, lignin extraction from PRC-RBMP OPEFB-BL was optimised at pH 2.5-3.5, 40-60 °C, and 0.5-1.5 hours, and were characterised by phenolic hydroxyl content and FT-IR analysis. Thirdly, lignin was phenolated and microwave pyrolysed to increase its chemical reactivity. The lignin was phenolated and optimised in the condition range of lignin/phenol ratio (1:2 to 2:1), 80-120 °C, 30-110mins and H_2SO_4 catalyst dosage between 2-10%. For microwave pyrolysis, the yield of bio-oil produced was obtained at 7 different powers between 600-1200 W. Both modified-lignins were evaluated by their phenolic hydroxyl contents and FT-IR analysis. Lastly, LPF resins were synthesized using two types of modified lignin with different percentages of lignin to phenol replacement (5%, 10%, 15%, 20%, 25% and 30%). The selected adhesives were applied on rubberwood veneers to determine its shear strength. PRC-RBMP black liquor lignin had similar properties to hardwood and softwood lignin. EFB lignin was mostly guaicyl (G) and syringyl (S). Lignin extraction at pH 3.0, 1 hour, and 60 °C enables extraction of lignin with 1.268 mmol/g phenolic hydroxyl. Phenolation and microwave pyrolysis have increased the chemical

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reactivity of extracted lignin relative to phenolic hydroxyl content. The phenolic hydroxyl content of phenolated lignin under the optimised condition of 1:1 L/P ratio, 110 mins, 100 °C and 8% H_2SO_4 is five-folds that of extracted lignin; while that of bio-oil produced from 1000W microwave pyrolysis is 15.5 folds that of extracted lignin. PRC-RBMP EFB lignin showed promise as plywood adhesive for LPF resins. LPF and PF share functional groups and similar properties. Phenolated lignin had better viscosity and solid content than bio-oil in plywood adhesive synthesis, indicating that it reacts better with formaldehyde. Plywood bonded with 5% and 10% phenolated lignin resin had higher shear strength (1.61 to 1.78 MPa) than unmodified lignin (1.05 MPa) but lower than control PF (2.72 MPa). 5% and 10% phenolated lignin LPF (ELPF) yield satisfactory results. 5%ELPF performed better than 10%ELPF. This research helps us understand PRC-RBMP OPEFB-BL and modified lignins in LPF resin's properties. Lignin extracted from black liquor can be used in a variety of biopolymer applications, providing a second source of income to the pulping industry, reducing production waste, waste water treatment costs, petrochemical use, and environmental pollution. The industry would benefit economically, environmentally, and socially by completing the study and continuing research.

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Abstrak tesis yang dikemukakan kepada Senat Universiti Putra Malaysia sebagai memenuhi keperluan untuk ijazah Doktor Falsafah

MODIFIKASI LIGNIN DARIPADA AIR REBUSAN HITAM PULPA UNTUK PENGELUARAN PELEKAT LIGNIN-FENOL-FORMALDEHID

Oleh

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Air rebusan hitam merupakan sisa hasil sampingan selepas proses pulpa. Ia mengandungi sisa-sisa degradasi lignin, karbohidrat dan komponen bukan organik. Dalam proses pencernaan kayu menjadi pulpa, lignin, hemiselulosa dan ekstraktif kayu lain telah dikeluarkan dari kayu untuk membebaskan gentian selulosa. Ia sentiasa dikaitkan dengan isu pencemaran oleh kilang kertas konvensional. Namun begitu, isu kemampanan dan kesedaran alam sekitar telah menarik perhatian para penyelidik terhadap komponen utama yang dikandungi dalam air rebus hitam, iaitu lignin. Lignin berpotensi digunakan sebagai bekalan mampan untuk bahan kimia berharga yang merupakan sumber tidak boleh diperbaharui. Kajian ini bertujuan untuk menguji potensi penggunaan lignin daripada air rebusan hitam tandan kosong kelapa sawit (OPEFB-BL) daripada *Preconditioning Refiner Chemical-Recycle Bleached Mechanized Pulping* (PRC-RBMP) dan meningkatkan kereaktifan kimianya untuk penghasilan pelekat lignin-phenol-formaldehid (LPF). Kajian ini mengandungi empat bahagian. Bahagian pertama kajian menentukan komposisi dan ciri PRC-RBMP OPEFB-BL. Kedua, pengekstrakan lignin daripada PRC-RBMP OPEFB-BL telah dioptimumkan pada pH 2.5-3.5, 40-60 °C, dan 0.5-1.5 jam, dan dicirikan oleh kandungan hidroksil fenolik dan analisis FT-IR. Ketiga, lignin telah difenolasi dan dipirolisis gelombang mikro untuk meningkatkan kereaktifan kimianya. Lignin telah difenolasi dan dioptimumkan dalam julat keadaan nisbah lignin/fenol (1:2 hingga 2:1), 80-120 °C, 30-110minit dan dos pemangkin H_2SO_4 antara 2-10%. Untuk pirolisis gelombang mikro, hasil bio-minyak yang dihasilkan diperolehi pada 7 kuasa yang berbeza antara 600-1200 W. Kedua-dua lignin yang diubah suai dinilai oleh kandungan hidroksil fenolik dan analisis FT-IR. Akhir sekali, pelekat lignin-fenol-formaldehid (LPF) telah disintesis menggunakan dua jenis lignin yang diubah suai dengan peratusan penggantian

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lignin kepada fenol yang berbeza (5%, 10%, 15%, 20%, 25% dan 30%). Pelekat yang dipilih telah digunakan pada venir kayu getah untuk pengujian kekuatan ricihnya. Lignin yang diekstrak daripada air rebus hitam PRC-RBMP mempunyai sifat yang hampir sama dengan lignin kayu keras dan kayu halus. Lignin EFB terdiri terutamanya daripada lignin jenis *guaicyl* (G) dan *syringyl* (S). Pengekstrakan lignin pada pH 3.0, 1 jam, dan 60 °C membolehkan pengekstrakan lignin dengan 1.268 mmol/g hidroksil fenolik. Fenolasi dan pirolisis gelombang mikro berjaya meningkatkan kereaktifan kimia lignin yang diekstrak seperti yang ditunjukkan dengan perubahan kandungan hidroksil fenolik. Kandungan hidroksil fenolik lignin terfenolasi di bawah keadaan optimum nisbah 1:1 L/P, 110 minit, 100 °C dan 8% H_2SO_4 adalah lima kali ganda daripada lignin yang diekstrak. Manakala kandungan hidroksil fenolik tinggi dalam *bio-oil* yang dihasilkan daripada pirolisis gelombang mikro 1000W adalah 15.5 kali ganda lebih tinggi daripada lignin yang diekstrak. Lignin EFB PRC-RBMP berpotensi digunakan sebagai resin LPF bertujuan untuk melekat papan lapis. Kedua-dua resin LPF dan PF mempunyai kumpulan berfungsi yang agak serupa. Campuran lignin terfenolasi didapati mempunyai kelikatan dan kandungan pepejal yang lebih baik berbanding *bio-oil* dalam sintesis pelekat papan lapis. Permerhatian ini menunjukkan bahawa lignin fenolasi mempunyai kereaktifan yang lebih baik berbanding *bio-oil* terhadap formaldehid dalam penghasilan pelekat. Akhir sekali, papan lapis yang dilekat dengan resin lignin terfenolasi (ELPF) 5% dan 10% telah menunjukkan kekuatan ricih (1.61-1.78 MPa) yang lebih baik daripada papan lapis yang dilekat dengan lignin tidak diubah suai (1.05 MPa), walaupun lebih rendah berbanding dengan PF kawalan (2.72 MPa). 5%ELPF mempunyai prestasi pelekat papan lapis yang lebih baik daripada 10%ELPF, namun kedua-dua menghasilkan keputusan memuaskan. Penyelidikan ini menyumbang kepada pemahaman dari segi sifat PRC-RBMP OPEFB-BL dan juga sifat fizikal dan pelekat lignin yang diubah suai sebagai pengganti fenol dalam penghasilan pelekat LPF. Selain itu, lignin yang diekstrak daripada air rebusan hitam dalam kajian ini boleh digunakan dalam pelbagai aplikasi biopolimer dan berpotensi untuk menyumbang sumber pendapatan kedua kepada industri pulpa, mengurangkan sisa daripada pengeluaran dan kos rawatan air sisa, mengurangkan penggunaan petrokimia, dan mengurangkan pencemaran alam sekitar. Pihak industri akan mendapat manfaat dari segi ekonomi, alam sekitar dan aspek sosial dengan pencapaian objektif kajian dengan jayanya dan penyelidikan lanjut dapat diteruskan pada masa yang akan datang.

ACKNOWLEDGEMENTS

As a full-time working adult, it is never easy to complete a PhD in Environmental Biotechnology. However, I am fortunate enough that there is a list of people behind this achievement. First, I would like to express my utmost appreciation to my mentor, my supervisor Dr. Termizi Yusof, co-supervisor Professor Hidayah Ariffin, Professor Paridah Md Tahir, and Professor Li Xin Ping for their unconditional guidance throughout my PhD journey since the day I started my PhD on 08 Feb 2017. During these five years, it has been a very challenging period for me to juggle my work at the same time on my study. Their persistence encouragement, priceless time, and boundless patience have allowed me to pass through the thick and thin throughout the journey. I never thought that I could complete my PhD on time as I have been through the frustrations during my tough time. Their strong motivation has pushed me beyond my limit and unleashed my potential before I can see it myself. They mentor me more than my thesis and to some extent, such guidance applies to my work. No word can express how thankful I am to all my supervisors.

Next, I would like to thank to UPM Institute of Tropical Forestry and Forest Products, (INTROP) and Shaanxi University of Science and Technology (SUST) for the use of necessary facilities. My special thanks to Dr Lee Seng Hua and his team for their kind assistance and technical supports, especially during the lockdown and movement restriction in the unprecedented pandemic.

I also would like to extend my sincere thanks to my company Nextgreen Pulp & Paper Sdn Bhd and my team members (Dr Arisyah, Dr Shamila, Farhana and Hazwani) on their help and support throughout this work. The assistance and moral support from other colleagues and friends who are not mentioned here are also appreciated.

Lastly and most importantly, my heartfelt gratitude to my parents and my siblings. To complete this thesis, I have no option but to scarify quality time with my family even during the festive season, and commit to my PhD study. Their unconditional love and understanding have been my biggest motivation in my PhD study.

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This thesis was submitted to the Senafe of Universiti Putra Malaysia and has been accepted as fulfillment of the requirement for the degree of Doctor of Philosophy. The members of the Supervisory Committee were as follows:

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LIST OF ABBREVIATIONS

3FI	Three-factor interaction
AKE	Aldehydes, ketones and esters
ANOVA	Analysis of variance
AOX	Absorbable organic halides
BL	Black liquor
BLG	Black liquor gasification
BLS	Black liquor solids
BOD	Biochemical oxygen demand
BPF	Bio-oil phenol-formaldehyde
CAGR	Compound annual growth rate
$CDCl_3$	Deuterated chloroform (d-chloroform)
CEH	Chlorination, Alkaline extraction, and Hypochlorite
CH_4	Methane
COD	Chemical oxygen demand
COP-15	Conference of Parties-15
DMRT	Duncan multiple range test
DO	Dissolved oxygen
DOE	Department of Environment
DTG	Derivative thermogravimetry
EBPF	EFB Phenol-rich bio-oil- phenol-formaldehyde
ECF	Elemental chlorine free
EFB	Empty fruit bunch
ELPF	EFB Phenolated lignin- phenol-formaldehyde
ETS	Emission trading system

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EVA	Ethylene vinyl acetate
FAO	Food and Agriculture Organization
FE	Formaldehyde emission
FFB	Fresh fruit bunch
FT-IR	Fourier transform infrared spectroscopy
GC-MS	Gas chromatography–mass spectrometry
GDP	Gross domestic Product
GTP	Green technology park
GW	Groundwood
H_2O_2	Hydrogen peroxide
H_2SO_4	Sulfuric acid
HCl	Hydrochloric acid
HMF	5-hydroxymethylfurfural
KBr	Potassium bromide
$KBrO_3$	Potassium bromate
KI	Potassium iodide
KL	Kraft lignin
L/P ratio	The ratio of lignin to phenol
LiP	Lignin peroxidase
LPF	Lignin-phenol-formaldehyde
MAP/MP	Microwave-assisted pyrolysis/Microwave pyrolysis
MDF	Medium-density fibreboard
MDI	Diphenylmethane-4,4'-discocyanate
MF	Melamine-formaldehyde

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MnP	Manganese peroxidase
MPOB	Malaysian palm oil board
MUF	Melamine-urea-formaldehyde
MVR	Mechanical vapor recompression
N_2S	Sodium sulphite
Na_2CO_3	Sodium carbonate
$Na_2S_2O_3$	Sodium thiosulfate
NaOCl	Sodium hypochlorite
NaOH	Sodium hydroxide
NMR	Nuclear magnetic resonance
O/I	Organic/inorganic ratio
O_3	Ozone
OPEFB-BL	Oil palm empty fruit bunch-black liquor
OPF	Oil Palm frond
OSB	Oriented strand board
PF	Phenol- formaldehyde
PKS	Palm kernel shells
POME	Palm oil mill effluent
ppm	Parts per million
PRC-RBMP	Preconditioning refiner chemical- recycle bleached mechanised pulping
PRF	Phenol-resorcinol-formaldehyde
PVA	Polyvinyl alcohol
PVAc	Polyvinyl acetate
RMP	Refiner mechanical pulps

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SAS	Statistical analysis software
SBR	Sequential batch reactor
SBR	Styrene butadiene rubber
SiC	Silicon carbide
SO_2	Sulphur dioxide
SS	Suspended solid
TCF	Totally chlorine free
TG	Thermogravimetry
TGA	Thermogravimetric analysis
TG-DSC	Thermogravimetry-differential scanning calorimetry
TMP	Thermomechanical pulps
TMS	Tetramethylsilane
UF	Ultrafiltration
UF	Urea-formaldehyde
UNFCCC	United Nations Framework Convention on Climate Change
VOC	Volatile organic compounds

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CHAPTER 1

MODIFICATION OF LIGNIN FROM PULPING BLACK LIQUOR FOR PRODUCTION OF LIGNIN-PHENOL-FORMALDEHYDE ADHESIVE

1.1 Background

Green Technology Park, GTP (Pekan) was conceptualised by Nextgreen Global Berhad, a public listed company in Malaysia on a new sustainable industry model which in line with Malaysia's vision and sustainable development policies to develop a green technology industry. Malaysia has made a voluntary commitment at the United Nations Framework Convention on Climate Change (UNFCCC) Conference of Parties-15 (COP-15) in Copenhagen back in 2009, to reduce the carbon intensity of gross domestic product (GDP) by 40 Percent as compared to 2005 levels by the year 2020. Accordingly, the development of green technology and renewable energy innovations will enable Malaysia to achieve its carbon reduction goals and position the nation as a regional leader in reducing the emission of greenhouse gases. GTP project aims to achieve Malaysia's pledges and goals, emphasising not only the utilization of the biomass from palm oil industry but also integrating the growth of green technologies to increase variety of green products derived from its biomass. Malaysia is the second largest palm oil producer (Jaafar et al., 2015), produces an abundant supply of oil palm biomass, such as empty fruit bunches (EFB) and oil palm fronds (OPF) which have not been utilised (MPOB, 2016). However, challenges faced by the palm oil industry is causing environmental issues and negative social impacts. Hence, government and industries are putting efforts to enhance the sustainability of the palm oil waste in environmental, social and economic aspects by maximising the usage through by-products (Cheah et al., 2023, Ali et al., 2020).

Industries within GTP convert biomass into value added products, such as pulp and paper, animal feed, fertiliser, etc. As pulp and paper manufacturing is the major project in GTP, it utilises oil palm empty fruit bunch (EFB) integrated renewable energy to produce pulp and paper. The pulp and paper making in GTP is an advanced pulping process using patented 'Preconditioning Refiner Chemical- Recycle Bleached Mechanised Pulp (PRC-RBMP)' technology. It is a 'hybrid' of chemical, mechanical and thermal processes that can preserve the condition of EFB fibre for paper production. The ultimate goal of this park is to achieve 'zero-waste' in the whole line of technology. It is also aimed at leading the world towards achieving a higher level of environmental consciousness.

In order to reach the sustainable goal of GTP and solve the pollution issue, black liquor needs to be processed and comprehensively utilised it to develop a value-added product. Black liquor discharge is the largest water pollution in pulp and paper industry. Pollution control in this has been a difficult problem for many years, putting not only a large investment and also failed in producing high quality products. Organic materials such as lignin and its derivatives are difficult to degrade. One of the major pollution issues on black liquor in pulp and papermaking industry is due to its high chemical oxygen demand (COD) (Yang, 2003; Annie, 2008). Conventionally, black liquor was discharged to waterways as a method of disposal or directed to a recovery boiler to recover chemicals and generate bioenergy (Lora and Glasser 2002). Black liquor treatment is not technically and economically viable in small scale paper mills (Himadri, 2009), eventually black liquor is released to the river or other water resources. Its toxicity might cause chronic and sub-lethal toxic effects on fish, such as endocrine disruption or growth development (Ragsdale, 2011).

Nevertheless, lignin which is the major constituent of black liquor can be utilised for various bioproducts development, due to its variable functional groups (Ibrahim et al. 2011). In fact, numerous researchers agreed that the abundant industrial lignin produced from pulp and paper mills could be an alternative material to replace some petrochemical feedstocks in the future (Yang and Fang 2014; Podschun et al. 2015a; Kazzaz et al. 2019; Luo et al. 2020). Due to its complex structural unit, EFB lignin has a variety of reactive groups, and has the advantages of a wide range of renewable, non-toxic, harmless and easy to degrade, and has unique advantages in the synthesis of biomass-based adhesives. However, due to the different methods of separation and extraction of lignin, the structure of lignin products is complicated, the molecular weight is large and the properties are unstable, which also has certain restrictions on the utilization of lignin. Understanding of the compositional and structural changes of lignin in black liquor after pulping treatment is important to know for the improvement of lignin accessibility and accordingly an optimum process could be designed. At present, the development and research of lignin is mainly reflected in the following aspects: first, various modification treatments are carried out on traditional industrial lignin to improve the chemical reactivity of lignin and the physical and chemical properties of lignin, which to enhance its application in the field of adhesive. Therefore, the research on the modification and application of lignin is the research focus of most researchers at present. Secondly, research and developments of environmentally friendly pulping and papermaking methods and processes has been increased in countries with developed pulp and paper industry such as Canada, the United States and Northern Europe. New technologies for efficient separation of cellulose and lignin have been developed.

The third aspect is the development of new functions of lignin, especially industrial lignin which is considered by-product of bioprocessing. To utilise the by-product and turn it to valuable and saleable product, it mitigates pollution issue of the pulp and paper industry and gives additional income from the spill-over business. The raw material used for paper making in GTP is EFB instead of wood, the composition of black liquor released might be different according to the pulping method and the properties of the raw material used. The advanced hybrid pulping process might have different properties of lignin in certain extent due to EFB source and effect after pulping. Therefore, analyses of OPEFB-BL to determine the composition and characteristic of black liquor components become the key focus for any potential application. The separation of organic

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lignin by organic solvent method and its development and utilization will also be an important direction of lignin development and application.

Many researches on black liquor have been done to understand the components in the liquid content and its potential application. Most studies on the properties of lignin in wood, and many potential applications involved lignin-based materials have been discovered, such as dispersant agent, super plasticizer, additive in petroleum extraction, and wood adhesive (Bertaud et al., 2012). Lignin considered to be one of the most abundant and promising upcoming organic resources among the renewable and naturally occurring polymers (Zhu et al., 2020). It can be used to replace phenol in phenolic resin, which is the most widely used industrial adhesive. Phenol and formaldehyde used in the synthesis of phenolic resin are petrochemical by-products. The price is more expensive than lignin products, and the released free phenol and free formaldehyde are harmful to human body. As a substitute, lignin can replace phenol or formaldehyde as an alternative green resource to synthesize lignin-phenol-formaldehyde (LPF) adhesive by chemical modification, which can reduce production cost, reduce pollution and protect the ecological environment. Research has proven that formaldehyde emission has reduced significantly tested on wood particleboard with green adhesive based on lignin and tannin formulation (Bertaud et al., 2012). However, there is no pure lignin adhesive has succeeded to commercialized at an industrial level. Replacement of a smaller proportion of phenol and formaldehyde still can be done by using lignin as an alternative resource (Pfungen, 2015).

1.2 Justification and Problem statement

Black liquor is produced as a complex liquid by-product of the kraft pulping process, containing lignin residues, degraded carbohydrates and inorganic constituent (Annie, 2008). In the process of digesting wood into pulp, lignin, hemicelluloses and other extractives such as tall oil and hydroxy acids were removed from the wood to free the cellulose fibres with sodium-based alkali compounds, such as sodium hydroxide and sodium sulfide (Radoykova et al., 2013; Chao et al., 2007). Traditionally, black liquor from kraft pulping process was treated by evaporators and burned in a chemical recovery boiler to recover alkali and small scale of energy. This might not be viable if low alkali content and total solid concentration in black liquor. Pollution load can be reduced by lignin recovery from black liquor. Hence, the environmental concern has driven the technology advancement of black liquor treatment to lignin extraction. However, the exact composition of black liquor components varies according to the pulping method and the properties of the raw material used (Humpert, 2016), which may affect the characteristic of lignin and subsequently affect the extraction process (Nikolskaya et al., 2019). The conditions of lignin extraction (e.g. pH, temperature, time) may also affect the yield and characteristics of extracted lignin. Therefore, after determining the characteristic of the black liquor from OPEFB, it is important to optimise the process of lignin extraction. In this study, lignin extracted from PRC-RBMP black liquor produced by OPEFB is less reactive.

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Lignin has the potential to produce more environmental-friendly resins due to its phenolic structure and availability in black liquor. However, the drawbacks of using lignin alone as wood adhesives and the chemical reactivity of lignin have become the obstacles production of lignin-based copolymer adhesives for composite wood panels. The extracted lignin in this study must reach certain reactivity to replace phenol to react with formaldehyde in the production of PF resin. Hence, lignin extracted needs to undergo modifications (e.g. phenolation, microwave pyrolysis, demethylation, glyoxalation) to improve its reactivity for the production of lignin-phenol-formaldehyde (Ghaffar & Fan 2013, El Mansouri & Salvado 2006, Du et al. 2014; Podschun et al., 2015). Different methods give different effect on the reactivity and each has its own flow: either the reaction conditions are engraved or the cost is prohibitively expensive. Phenolation can be considered as a promising and practical method to modify lignin. Besides, microwave pyrolysis (MP) on lignin from biomass has been studied recent years and gave positive outcomes. However, there are not many research on MP from secondary source which is the current research direction of many biopolymers companies nowadays due to sustainability focus.

The chemical reactivity of lignin can be improved by phenolation and microwave pyrolysis of lignin to produce phenolated lignin and phenol-rich bio-oil, respectively. Manipulating the process conditions may improve the phenolic hydroxyl content which enhance the chemical reactivity for LPF production. On the other hand, microwave pyrolysis with different microwave powers may affect the yield of bio-oil and phenol content. In this research, both modified lignins will be tested on their properties and their level of chemical reactivity in relation of phenolic hydroxyl content. Lignin derivatives produced from phenolation and microwave pyrolysis, respectively may be suitable as a replacement to commercial phenol in the phenol-formaldehyde (PF) production for plywood application. Subsequently, both will be used as a replacement of phenol in the synthesis of PF adhesives. The properties of each adhesive will be characterized on their physical properties and thermal stability. Selected adhesive with better physical properties will be tested on its bonding strength on plywood. Hence, this study aims to establish not only the potential utilization of PRC-RBMP OPEFB lignin extracted from black liquor and also the modification of lignin for the production of LPF adhesive in the potential application as plywood adhesive.

The overall objective of this study is to offer a new approach to evaluate potential direction for pulping industry in utilising the lignin from black liquor in green phenolic resin market. The information produced from this research would help resolve pollution issue in papermaking industry, also to determine whether lignin is feasible and reactive enough to replace part of the phenol and in turn, the production of green adhesive provide an alternative source for Malaysia to diversify its downstream products from palm oil industry and paper industry. This new cutting-edge research on the development of zero-waste technology would revolutionize the pulp and paper industry, also brings a massive positive impact on the economy and environment.

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1.3 Objectives

1. To determine the composition and characteristic of oil palm empty fruit bunch black liquor (OPEFB-BL) components from PRC-RBMP.

2. To optimise the conditions of lignin extraction from PRC-RBMP OPEFB-BL and determine its properties.

3. To investigate the effect of phenolation and microwave pyrolysis on the chemical reactivity of lignin for the production of lignin-phenol-formaldehyde (LPF) adhesive.

4. To evaluate the physical and bonding properties of lignin-phenol-formaldehyde (LPF) adhesive derived from OPEFB-BL.

Hypothesis

i. In this study, lignin extracted from OPEFB-BL produced from PRC-RBMP might be different in properties and might give different effects in producing LPF adhesive.

ii. PRC-RBMP OPEFB-BL contains higher lignin and hence contributing to higher yield of lignin extracted from the black liquor.

iii. Manipulating phenolation conditions may improve the phenolic hydroxyl content which enhance the chemical reactivity for LPF production.

iv. Microwave pyrolysis with different microwave powers may affect the yield of bio-oil and phenol content.

v. Lignin derivatives produced from phenolation and microwave pyrolysis, respectively may be suitable as a replacement to commercial phenol in the phenol-formaldehyde (PF) production for plywood application.

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