OPTIMIZATION OF FLAME RETARDANT COMPOSITE PARAMETERS BASED ON OIL PALM TRUNK USING RESPONSE SURFACE METHODOLOGY AND ITS SUITABILITY FOR OTHER BIOMASS

MADIHAN BINTI YUSOF

UNIVERSITI SAINS MALAYSIA

2024

OPTIMIZATION OF FLAME RETARDANT COMPOSITE PARAMETERS BASED ON OIL PALM TRUNK USING RESPONSE SURFACE METHODOLOGY AND ITS SUITABILITY FOR OTHER BIOMASS

by

MADIHAN BINTI YUSOF

**Thesis submitted in fulfilment of the requirements
for the degree of
Doctor of Philosophy**

July 2024

ACKNOWLEDGEMENT`

Alhamdulillah, I would like to express my gratitude to Allah SWT for allowing me to help endlessly in finishing my Ph.D. studies. I would like to acknowledge all those who contributed and played an essential part in my studies.

Firstly, my beloved husband Ahmad Nurul Irwan, a significant person in my life, my backbone and strength, and my children Affan Wafiq and Adam Hafiy. Thank you for giving me support and encouragement to finish my studies. I also would like to express my deepest gratitude to my respectful supervisor, Dr. Mazlan Ibrahim, my co-supervisors Prof. Dr. Rokiah Hashim, Dr. Firdaus Yhaya, and Dr Fazita for their guidance and encouragement, and help throughout my studies.

Also, I am very thankful to have understanding and caring parents Yusof Harun and my late mother Marjinal Derahman, In-laws Adnan Latif and Sohana Yusoff, and my siblings Raihan, Azihan, my late brother Adihan, Sharhan, and Farhan.

To all my fellow friends, a big thank you for sharing all the challenging journeys and cherishing them together from the beginning until the end. And also, very much indebted to all that have been mentioned and not mentioned who had assisted along my journey. I will never forget all those experiences, knowledge, and moments gathered. Thank you.

TABLE OF CONTENTS

ACKNOWLEDGEMENT		**ii**
TABLE OF CONTENTS		**iii**
LIST OF TABLES		**vii**
LIST OF FIGURES		**ix**
LIST OF ABBREVIATION		**xii**
ABSTRAK		**xv**
ABSTRACT		**xvii**
CHAPTER 1	**INTRODUCTION**	**1**
1.1	Research Background	1
1.2	Problem Statement	8
1.3	Objectives	12
1.4	Scope of Study	13
CHAPTER 2	**LITERATURE REVIEW**	**14**
2.1	Composite	14
	2.1.1 Types of Composites and its Applications	16
2.2	Raw Material for Wood Composites	18
	2.2.1 Oil Palm Trunk	19
	2.2.2 Rubberwood	23
	2.2.3 Application of Rubberwood	24
2.3	Adhesive in Wood Composite	26
	2.3.1 Polyvinyl Alcohol (PVOH) as Binder	27

	2.3.2 Characteristic of PVOH……………………………….	28
2.4	Flame Retardant ……………………………………………...	30
	2.4.1 Types of Flame Retardant …………………………...	32
	2.4.2 Mechanism of Flame Retardancy…………………….	35
	2.4.3 Flame Retardant Additives…………………………...	38
	2.4.4 Sodium Chloride (NaCl) as Flame Retardant………..	38
	2.4.5 Calcium Carbonate ($CaCO_3$)…………………………	43
2.5	Design of Experiment ………………………………………..	45
	2.5.1 Response Surface Methodology (RSM) ……………..	45
	2.5.2 Central Composite Design ……………………….......	47
CHAPTER 3	**MATERIALS AND METHOD ……………………………….**	**50**
3.1	Overview ………………………………………………….....	50
3.2	Sample Preparation for Weight Gain ……………......................	53
	3.2.1 Composite Making…………………………………….	53
	3.2.2 The Adoption of Central Composite Design (CCD)…	55
3.3	Physical Properties of Board…………………………………….	58
	3.3.1 Moisture Content Test………………………………..	58
	3.3.2 Thickness Swelling Test ……………………………..	58
	3.3.3 Water Absorption test ………………………………..	59
	3.3.4 Density Test ………………………………………....	59
3.4	Mechanical Properties of Board ………………………………...	60
	3.4.1 Bending Strength Test………………………………..	60
	3.4.2 Internal Bonding Test ………………………………...	60

3.5	Flame Retardant Properties ……………………………………..		61
	3.5.1	Limited Oxygen Index ……………………………....	61
	3.5.2	UL 94 Fire Test ……………………………….......	61
3.6	Thermal Stability Properties …………………………………….		64
	3.6.1	Thermogravimetric Analysis (TGA)………………….	64
3.7	Fourier Transform Infrared Analysis (FTIR)…………………….		66
3.8	X-Ray Diffraction Analysis (XRD)…. ………………………….		66
3.9	Microscopy Morphology Analysis………………………………		67
	3.9.1	Image Fracture Observation…………………………..	68
	3.9.2	Scanning Electron Microscopy Analysis……………..	68
3.10	Preparation of Oil Palm Trunk Flame Retardant Composite Board and Rubberwood Flame Retardant Composite Board……		68
CHAPTER 4	**ANALYSIS OF OIL PALM TRUNK FLAME RETARDANT COMPOSITES BY RESPONSE SURFACE METHODOLOGY……………………………………………**		**69**
4.1	Result and Discussion ……………………………………………...		69
	4.1.1	Effect of Solutions Concentration on NaCl Adsorption	69
	4.1.2	Analysis of Variance (ANOVA) of LOI Based on CCD …………………………………………………	70
	4.1.3	Diagnostic Checking of the LOI of the Design ……	74
	4.1.4	Effect of Parameter on LOI of the Flame Retardant Composites………………………………………...	76
	4.1.5	Analysis of Variance (ANOVA) of MOR Based on CCD ………………………………………..........	81
	4.1.6	Diagnostic Checking of the MOR of the Design………………………………………………...	85

	4.1.7	Effect of the Parameter on MOR of the Flame Retardant Composites ………………………………..	88
	4.1.8	Analysis of Variance (ANOVA) of IB Based on CCD..	91
	4.1.9	Diagnostic Checking of the IB of the Design ……….	95
	4.1.10	Effect of the Parameter on IB of the Flame Retardant Composites……………………………………	98
	4.1.11	Optimization of Manufacturing Flame Retardant Oil Palm Trunk Composite Board by Response Surface Methodology (RSM) ……………………………….	101
CHAPTER 5	**COMPARATIVE STUDY ON FLAME RETARDANT COMPOSITE BOARD OF OIL PALM TRUNK AND RUBBERWOOD……………………………………………**		**104**
5.1	Introduction…………………………………………………...		104
5.2	Evaluation of Physical Properties …………………………….		104
	5.2.1	Moisture Content…………………………………..	104
	5.2.2	Thickness and swelling……………………………	105
	5.2.3	Water Absorption…………………………………	107
5.3	Evaluation of Mechanical Properties………………………….		108
	5.3.1	Bending Strength Test……………………………...	108
	5.3.2	Modulus of Elasticity……………………………….	110
	5.3.3	Internal Bonding Strength Test……………………….	110
5.4	Evaluation of Limited Oxygen Index ………………………….		111
5.5	Evaluation of Cone Calorimeter……………………………….		113
5.6	Evaluation of Thermogravimetric Analysis (TGA)……………...		118
5.7	Evaluation of UL-94 Fire Testing……………………………….		123
5.8	Evaluation of Fourier Transform Infrared Analysis……………...		127

5.9	Evaluation of X-Ray Diffraction………………………………...	129
5.10	Evaluation of Image Fracture……………………………………	130
5.11	Evaluation of scanning Electron Microscopy Analysis…………	131
5.12	Evaluation of Energy Dispersive X-Ray Spectroscopic (EDX) Analysis…………………………………………………………	133
5.13	Conclusion …………………………………………………...	135
CHAPTER 6	**GENERAL CONCLUSION AND RECOMMENDATIONS..**	136
6.1	Conclusion …………………………………………………...	136
6.2	Recommendation for Future Work …………………………...	138
REFERENCES ……………………………………………………………..		139
LIST OF PUBLICATION		

LIST OF TABLES

		Page
Table 2.1	Previous research on composite board conducted with assistance from RSM …………………………………	17
Table 2.2	Previous research on NaCl…………………………….	48
Table 2.3	Previous research on composite board conducted with assistance from RSM………………………………….	54
Table 3.1	Table of formulation…………………………………..	62
Table 3.1	Manufacturing ratio of the flame retardant composite board by Central Composite Design …………………	62
Table 4.1	Total number of experimental runs with actual and coded values ………………………………………….	76
Table 4.2	The actual and predicted value of responses from overall experiment runs ……………………………………..	78
Table 4.3	Analysis of Variance (ANOVA) for response surfaced reduced model of Limited Oxygen Index……………..	85
Table 4.4	Analysis of Variance (ANOVA) for response surfaced reduced model of MOR ……………………………….	86
Table 4.5	Analysis of Variance (ANOVA) for response surfaced reduced model of internal bonding (IB) ……………..	87
Table 4.6	Fit summary analysis for the LOI flame retardant composites …………………………………………..	89
Table 4.7	Fit summary analysis for the MOR flame retardant composites …………………………………………..	90
Table 4.8	Fit summary analysis for the IB flame retardant composites…………………………………………...	90
Table 4.9	RSM's Central Composite Design provides optimal manufacture of the flame retardant composite board through numerical optimization ………………………..	103

Table 5.1	Specimen classification according to LOI value ………	114
Table 5.2	Fire response parameters for OPT flame retardant composite board and rubberwood flame retardant composite board ……………………………………….	116
Table 5.3	UL-94 Fire Testing rating and criteria …………………	126
Table 5.4	UL-94 test result for OPT flame retardant composites board and rubberwood flame retardant composites board…………………………………………………	127

LIST OF FIGURES

		Page
Figure 2.1	Oil palm biomass (a) empty fruit bunch, (b) mesocarp fibers, (c) oil palm trunk, (d) palm kernel shell, (e) oil palm frond, (f) oil palm leaf …………………………….	22
Figure 2.2	Micrograph of an oil palm trunk in cross-section ………	24
Figure 2.3	The model of flame retardant systems is presented schematically ………………………………………...	36
Figure 2.4	The availability of heat, fuel, and oxygen as oxidizing agents all influence the combustion …………………….	42
Figure 3.1	Flowchart of the overall experiment with testing………………………………………………….	60
Figure 3.2	Oil palm trunk composite board ………………………..	52
Figure 3.3	Limited oxygen index testing …………………………..	67
Figure 3.4	The setup for sample evaluation in UL-94 ……………..	69
Figure 4.1	Percentage weight gain of treated Oil Palm Trunk ……..	74
Figure 4.2	The correlations between actual and predicted values of (a) limited oxygen index (b) bending strength (c) internal bonding ………………………………………………	80
Figure 4.3	Studentized residuals vs anticipated values for each response studied (a: LOI, b: bending strength, c: internal bonding) ………………………………………………...	81
Figure 4.4	Perturbation plot for LOI ……………………………	94
Figure 4.5	3D plot showing the effect of PVOH (B), and $CaCO_3$ on LOI for OPT flame retardant composite board …………	95
Figure 4.6	Perturbation plot for MOR ……………………………...	97
Figure 4.7	3D plot showing the effect of PVOH (B) and $CaCO_3$ on MOR for OPT flame retardant composite board ………..	98
Figure 4.8	Perturbation plot for IB …………………………........	100

Figure 4.9	3D plot showing the effect of NaCl (A), and PVOH (B) on IB for OPT flame retardant composite board ………..	101
Figure 5.1	Moisture content of OPT-1, OPT-2, RBW-1, and RBW-2 composite board ………...	106
Figure 5.2	Thickness swelling of OPT-1, OPT-2, RBW-1, and RBW-2 composite board ………………………………..	108
Figure 5.3	Water absorption of OPT-1, OPT-2, RBW-2, and RBW-2 composite board ………...	109
Figure 5.4	Bending strength (MOR) of OPT-1, OPT-2, RBW-1, and RBW-2 composite board ………………………………..	111
Figure 5.5	Modulus of elasticity (MOE) OPT-1, OPT-2, RBW-2, and RBW-2 ……………………………………………..	112
Figure 5.6	Internal bonding (IB) strength of OPT-1, OPT-2, RBW-1, and RBW-2 composite board ……………………….	113
Figure 5.7	Limited oxygen index (LOI) of OPT-1, OPT-2, RBW-1, RBW-2………………………..	115
Figure 5.8	Heat Release Rate (HRR) of OPT flame retardant composite board and rubberwood flame retardant composite board as a function time …………………….	119
Figure 5.9	Image sample before and after burnt (a) OPT flame retardant composite board, (b) rubberwood flame retardant composite board ………………………………	120
Figure 5.10	TGA curve of OPT-1, OPT-2, RBW-1 and RBW-2………………………………………………………	123
Figure 5.11	DTG of OPT-1, OPT-2, RBW-1 and RBW-2 …………..	124
Figure 5.12	FTIR of OPT-1, OPT-2, RBW-1 and RBW-2……………	129
Figure 5.13	XRD of OPT-1, OPT-2, RBW-1 and RBW-2……………	131
Figure 5.14	Fracture image of OPT and Rubberwood ………………	133
Figure 5.15	SEM of OPT at different magnification ………………..	134
Figure 5.16	SEM of Rubberwood at different magnification ……….	134

Figure 5.17 EDX analysis of (a) OPT flame retardant composite board and (b) Rubberwood flame retardant composite board 136

LIST OF ABBREVIATION

$CaCO_3$	Calcium Carbonate
CCD	Central Composite Design
DoE	Design of Experiment
EFB	Empty Fruit Bunch
FTIR	Fourier Transform Infrared
g	Gram
g/cm^3	Gram per centimetre cube
g/mol	Gram per mole
IB	Internal Bonding
LOI	Limited Oxygen Index
MOE	Modulus of Elasticity
MOR	Modulus of Rupture
MPa	Mega Pascal
NaCl	Sodium Chloride
OPF	Oil Palm Frond
OPT	Oil Palm Trunk
OPT-1	OPT+PVOH
OPT-2	OPT+PVOH+NaCl+$CaCO_3$
PF	Phenol-Formaldehyde
POME	Palm Oil Mill Effluent
PVC	Polyvinyl Chloride
PVOH	Polyvinyl Alcohol
RBW-1	RBW+PVOH
RBW-2	RBW+PVOH+NaCl+$CaCO_3$
RSM	Response Surface Methodology
UF	Urea Formaldehyde
XRD	X-ray Diffraction

PENGOPTIMUMAN PARAMETER KOMPOSIT TAHAN API BERASASKAN BATANG KELAPA SAWIT MENGGUNAKAN KAEDAH RANGSANGAN PERMUKAAN DAN KESESUAIANNYA UNTUK BIOJISIM YANG LAIN

ABSTRAK

Komposit tahan api yang dibincangkan dalam kajian ini telah dibuat dengan merawat batang kelapa sawit (OPT) dengan larutan natrium klorida (NaCl) dan kemudian mengikatnya dengan poli(vinil) alkohol (PVOH) dan kalsium karbonat ($CaCO_3$) sebagai bahan pengisi. Reka bentuk eksperimen menggunakan reka bentuk komposit pusat (CCD) dalam kaedah gerak balas permukaan (RSM) untuk mengoptimumkan kombinasi NaCl, PVOH, dan $CaCO_3$ dalam komposit OPT. Julat kombinasi untuk NaCl, PVOH, dan $CaCO_3$ adalah dari 10% hingga 30%, 10% hingga 20%, dan 4% hingga 10%, secara berurutan. Kriteria pengoptimuman adalah berdasarkan kesan mereka terhadap kekuatan lenturan (MOR), pautan dalaman (IB), dan indeks oksigen terhad (LOI). Melalui analisis, nisbah optimum untuk menghasilkan papan komposit tahan api dari OPT telah ditentukan sebagai NaCl 10%, PVOH 20%, dan $CaCO_3$ 4%. Nilai yang diramalkan dari model empirikal untuk MOR, IB, dan LOI adalah masing-masing 12.96 MPa, 4.19 MPa, dan 33.73%. Keputusan eksperimen hampir sejajar dengan ramalan ini, dengan MOR pada 11.13 MPa, IB pada 5.78 MPa, dan LOI pada 32.3%. Kajian ini juga menyiasat aplikasi nisbah optimum ini kepada zarah-zarah kayu getah, membandingkan papan komposit tahan api yang dihasilkan dengan OPT. Pelbagai analisis seperti Transformasi Fourier Infra Merah (FTIR), penyebaran sinar-X (XRD), analisis termografi (TGA), ujian kebakaran UL 94, ujian kalorimeter kon, pemerhatian gambaran kecacatan, dan mikroskop elektron imbasan (SEM) telah dijalankan pada papan komposit tahan api OPT. Kedua-dua papan komposit OPT dan kayu getah menunjukkan kestabilan termal yang baik, mencadangkan bahawa rawatan NaCl dan $CaCO_3$ adalah bahan pengisi yang berkesan untuk papan partikel tahan api, dengan PVOH berfungsi sebagai pengikat yang sesuai. Penyelidikan ini menunjukkan potensi penggunaan batang kelapa sawit untuk menghasilkan bahan komposit dengan penambahbaikan ciri-ciri tahan api.

OPTIMIZATION OF FLAME RETARDANT COMPOSITE PARAMETERS BASED ON OIL PALM TRUNK USING RESPONSE SURFACE METHODOLOGY AND ITS SUITABILITY FOR OTHER BIOMASS

ABSTRACT

The flame retardant composite discussed in the study was developed by treating oil palm trunk (OPT) with a sodium chloride (NaCl) solution and then bonding it with poly(vinyl) alcohol (PVOH) and calcium carbonate ($CaCO_3$) as a filler. The experimental design utilized central composites design (CCD) within the response surface methodology (RSM) framework to optimize the combination of NaCl, PVOH, and $CaCO_3$ in the OPT composites. The range of combinations for NaCl, PVOH, and $CaCO_3$ varied from 10% to 30%, 10% to 20%, and 4% to 10%, respectively. Optimization criteria were based on their effects on bending strength (MOR), internal bonding (IB), and limited oxygen index (LOI). Through analysis, the optimal ratio for producing flame retardant composite boards from OPT was determined to be NaCl 10%, PVOH 20%, and $CaCO_3$ 4%. Predicted values from the empirical model for MOR, IB, and LOI were 12.96 MPa, 4.19 MPa, and 33.73%, respectively. Experimental results closely aligned with these predictions, with MOR at 11.13 MPa, IB at 5.78 MPa, and LOI at 32.3%. The study also investigated the application of this optimal ratio to rubberwood particles, comparing the resulting flame retardant composite board with that of OPT. Various analyses such as Fourier Transform Infrared (FTIR), X-ray diffraction (XRD), Thermogravimetric Analysis (TGA), UL 94 Testing, Cone Calorimeter testing, image fracture observation, and Scanning Electron Microscope (SEM) were conducted on the OPT flame retardant composite board. Both OPT and

rubberwood composite boards exhibited good thermal stability, suggesting that NaCl and $CaCO_3$ treatments are effective fillers for flame retardant particleboards, with PVOH as a suitable binder. This research highlights the potential of using oil palm trunks to develop composite materials with improved flame retardant properties.

CHAPTER 1

INTRODUCTION

1.1 Research Background

Flame retardants are chemical compounds added to or incorporated into composite materials to give variable levels of flammability protection. Flame retardancy can be achieved in composite materials by blocking or altering the combustion cycle. The combustion cycle of composite materials can be stopped in two ways. First, incorporation of chemical compounds that prevent composites to reach a pyrolysis point when heated where these compounds disintegrate endothermically. Second, by introducing chemicals that generate more non-flammable byproducts and char during the pyrolysis cycle (Bar et al., 2015).

Composite material refers to new materials created by combining two or more materials and having properties that differ from the materials used (Kumar Sharma et al., 2022). Composite is designed to achieve targeted properties such as high strength, high durability, stiffness, flexural strength, and resistance to corrosion, wear impact, and also fire.

Fire resistance and smoke generation properties of composite materials have gained significant attention these days as they must pass a regulatory fire test depending on the application to guarantee public safety. Therefore, it is imperative to comprehend the combustion process of individual components of end-products and determine the most effective methods for enhancing their flame resistance while maintaining their advantageous characteristics of low weight and high mechanical ratios (Bar et al., 2015). Typically, this weakness can be mitigated or even eliminated by modifying the composite with specific chemicals, such a halogenated organic flame retardant (mainly brominated and chlorinated), phosphorus-containing flame retardants, nitrogen-containing flame retardant and inorganic flame retardant (Malucelli, 2019).

Oil palm plantations are known to generate a substantial quantity of biomass, primarily in the form of fronds and trunks. Oil palm fronds and trunks, being agricultural by-products, are often encountered lignocellulosic materials that are often found in substantial quantities on the field following pruning and replanting activities (Madusari et al., 2023). In other words, the oil palm trunk (OPT) is removed during replanting when the trunk is no longer useful. Utilizing OPT as a primary material to create products with added value will reduce overall production costs and increase economic returns (Nuryawan et al., 2022).

The expansion of the oil palm industry has a negative impact on the environment while improving the stability of the Malaysian economy. Agricultural waste such as oil palm trunks has skyrocketed due to the expansion of oil palm plantations. Due to this problem, various extensive studies were carried out by many researchers to determine the best methods to utilize the byproduct of crop into a functional product such as particleboard (Abd Karim et al., 2020), binderless particleboard (Baskaran et al., 2017), flame retardant composites (Selamat et al., 2019) and compressed veneer panel (Saari et al., 2020). The wood-based panel sector faces significant issues in terms of wood supply due to several factors such as rising global demand for wood raw materials, growing environmental concerns, and recent legislative requirements that prioritize value-added applications of wood resources. The implementation of strategies to mitigate these shortages is of utmost importance, especially in nations characterized by a limited forested landmass (Lee et al., 2022).

Rubberwood (Hevea brasiliensis), a byproduct of natural rubber tree cultivation, was selected as the biomass for this research, has emerged as an essential raw material for timbers in recent years. After 20 to 25 years, when the latex supply begins to diminish, this tree is typically cut down. Rubberwood is valued for its appealing wood grain, finds common use in furniture production, interior design, and toy making. However, its utility is somewhat restricted by its comparatively low strength and susceptibility to changes in size and shape, especially in humid conditions (Meethaworn et al., 2020). Despite these limitations, rubberwood is highly valued for crafting wooden items like furniture and sturdy flooring, thanks to its intricate textures and excellent machining properties. Yet, it is notably prone to mildew, moth infestation, and decay due to its elevated nutritional content, comprising around 8% starch and free sugars (Zhu et al., 2021).

Wood products are extensively utilized in building and construction due to their distinctive amalgamation of characteristics. These include the ease of modification, superior physical and mechanical capabilities, aesthetic appeal, environmental friendliness, and the ability to offer substantial strength while retaining a sustainable life cycle. Wood is remarkably sustainable and renewable resource due to its ability to replenished naturally. Additionally, it offers the advantage of being easily malleable, making it a popular choice for the construction of both residential and industrial structures, a practice that has persisted over an extended period (Mali et al., 2022). Despite these benefits, one of the disadvantages of using wood as a constructing material is its low fire resistance. Most composite materials are easily combustible, therefore, in order to ensure their secure use, they are typically combined with flame retardants (Wang et al., 2022). To enhance fire resistance, major timber products are generally treated with fire retardants. Current fire-retardant products are focused on enhancing fire performance but do not meet the rising demand for wood products (Mali et al., 2022).

The application of flame retardants to wood materials is a strategy for enhancing fire protection. The purpose of flame retardants is to prevent or delay ignition and to mitigate combustion's effects. Improving the fire resistance of wood can increase the use of wood in the construction industry (Elvira-León et al., 2016). Flame retardant treatments can provide composite materials with low ignitability, lowering flame propagation or even preventing the burning process.

Based on their chemical structure and composition, the flame retardant treatment particles can self-extinguish in this case (Karunakaran et al., 2016; Ma et al., 2021; Malucelli, 2019). Chemical treatment of natural fibers is the process of grafting composites on the surface of fibers to alter the nature of fiber cell walls permanently. Surface treatment of natural fibers can be done with a variety of chemicals. Chemical modification improves dimensional stability, decreases water absorption capacity, and increases fiber resistance to fungal decay (Sood & Dwivedi, 2017).

A previous study done by Manral and Bajpai (2020) showed that when kenaf fibers were treated with sodium acetate, the removal of non-cellulosic content from kenaf fibers after treatment is responsible for the improved thermal nature of treated fiber composites. Only a few studies have been conducted using salts as a pre-treatment agent (Das & Sarmah, 2015). Sodium chloride can penetrate the cell walls, and cure cell walls to enhance their dimensional stability (Jiang, Li, et al., 2015a). Nasir et al., (2019) concluded that particles treated with chemicals showed an impressive performance in flame retardancy but also had several weaknesses such as instability of board quality and insufficient mechanical strength.

Sodium chloride (NaCl), also known as salt or halite, is an ionic compound with direct or indirect uses in producing many chemicals. Previous researchers Jiang et al., (2016) investigated the effect of the ionic strength of salt on the rheological properties of a suspension of nanoparticles. They found that sodium chloride conveniently interacts and influences the surface charge, significantly affecting the dispersion of nanoparticles, viscosity, storage and loss modulus, and gelatinizing properties.

Calcium carbonate ($CaCO_3$) is the most extensively used as filler since it is abundant, inexpensive, and non-toxic. Due to the emission of carbon dioxide, it may also improve the flame retardancy of the materials (Hongzhen et al., 2017). Calcium carbonate is commonly used as an additive or filler in rubber, plastics, coatings, and various industrial applications nowadays (Srivabut et al., 2018). This compound, CaCO3, is non-toxic, cost-effective, and environmentally friendly. It exhibits excellent compatibility with polymer substrates and imparts numerous desirable properties to the substrate (Huang et al., 2018).

As society evolved, so did the demand for flame retardant building decoration materials. When compared to other forms of reinforcing materials, the use of calcium carbonate as a filler in wood composites is gaining appeal as a novel material due to its capacity to improve dimensional stability, mechanical and physical qualities, and lower costs. (Olakanmi et al., 2008; Srivabut et al., 2018). Because inorganic fillers are much smaller than wood fiber, they can easily be inserted into a polymeric matrix between wood fibers (Liang & Wang, 2015). Nanoclay, talcum, and calcium carbonate were found to positively impact the strength, modulus, processing efficiency, creep, and elastic recovery performance of wood composites in previous studies (Karumuri et al., 2017). Mineral incorporation into the composite affects cellulose thermal decomposition by reducing the formation of volatiles.

Phenol formaldehyde (PF) and Urea Formaldehyde (UF) are essential adhesives in various fields, including the wood industry, aerospace, and microelectronics (Granado et al., 2020). Currently, formaldehyde-based adhesives are used to bond the majority of commercially produced particleboard, which could have negative effects on the environment and human health owing to formaldehyde emission. The market is trending towards employing particle boards with little to no formaldehyde, according to the global trend (Hashim, 2011b). As a result, the global marketplace trend shows that particleboard with little or no formaldehyde is increasing, which leads to studies on the production of non-toxic particleboard or particleboard without the use of synthetic adhesions (Homkhiew et al., 2020a).

Formaldehyde is a carcinogenic chemical used in industrial processes and released into the environment. Formaldehyde emissions can come from synthetic-free formaldehyde that hasn't been polymerized into the network and will emit during or shortly after panel production and from adhesive hydrolysis, which will emit over the panel's lifetime depending on moisture and temperature (Solt et al., 2019). Using another material as a formaldehyde replacement is one way to avoid formaldehyde's harmful effects. Since formaldehyde has such a high carcinogenic effect, extensive research has been done to find a more environmentally friendly, cost-effective, and long-lasting alternative (Yaakob et al., 2022). Therefore, a variety binder of non-formaldehyde choices was investigated to replace the formaldehyde adhesive. Adhesives have played an important role in the efficient use of wood resources and the development and growth of the forest products industry and will continue to do so (Solt et al., 2019)

Polyvinyl alcohol (PVOH) has been considered as an adhesive and recognized as a biodegradable polymer because of its properties readily consumed by microorganisms and enzymes when exposed to the natural environment (Sin et al., 2010). The PVOH, a water-soluble synthetic polymer, has good chemical and thermal stability with many polymer applications (Yusof et al., 2020). PVOH is also a hydrophilic polymer that is non-toxic, biodegradable, and biocompatible (Thomas et al., 2018). Based on its applicant and facts, PVOH significantly impacts application innovations in domestic and specialty industries (Sin et al., 2010).

Although various works have investigated the properties of oil palm trunk composite board, there is currently no information on the flame retardant of composite produced from oil palm trunk treated with NaCl and $CaCO_3$ as fillers and PVOH as a binder. This research aimed to analyze the influence of individual parameters, with a focus on achieving high mechanical properties and a high limiting oxygen index (LOI) in a composite board mixture containing PVOH, NaCl, and $CaCO_3$. Following this, an optimized composite board was created using central composite design (CCD) as part of the response surface methodology (RSM). Lastly, a comparative assessment was carried out between the optimal combination of OPT, NaCl, PVOH, and $CaCO_3$ with rubberwood.

1.2 Problem Statement

A massive plantation area in Malaysia produced an abundance of oil palm trunks, which negatively impacted the environment due to improper handling. Increasing global demand for wood raw materials, elevated environmental concerns, and recent legislative requirements interacting to dropping wood consumption and prioritizing value-added wood uses have all brought significant wood supply challenges to the wood-based panel industry (Hua et al., 2022). Addressing these inadequacies may be critical in countries with scant woods. The attempt to optimize the utilization of existing wood and lignocellulosic resources, together in exploration of alternative natural feedstocks obtained from plentiful and renewable agricultural residues and wood by-products, represents a feasible approach to mitigate the adverse environmental consequences associated with the wood-based panel sector and enhance resource efficiency.

Most flammable furniture poses a safety risk because it is the first thing to be ignited by small flames such as candles and cigarettes. The release of smoke and toxic gases by composite materials during combustion, one of the critical issues which strongly affect the ability of the victim to evacuate a building on fire and can be the first cause of fire fatalities (Shi et al., 2022). It has been well-documented that residential fires have been a significant contributor to worldwide fires. Therefore, the creation of composite-based household stuff that is resistant to fire becomes both essential and challenging (Yi et al., 2023). Because of the use of natural fiber composites in various applications, the flammability behavior of natural fiber composites has become one of the most critical issues in recent years (Gholampour & Ozbakkaloglu, 2020). The loss of strength and stiffness at high temperatures is one of the weaknesses of natural fiber composites. When natural fiber composites are exposed to fire or other high-intensity heat sources, they undergo thermal decomposition and combustion (Faruk et al., 2012).

Hence, in this study, the oil palm trunk particles will undergo treatment with NaCl and will be mixed with $CaCO_3$ as a filler, while PVOH will be employed as a binder. It is anticipated that this combination will result in positive improvements in the flame retardant properties. Different natural fibers have different chemical compositions and microstructures, the flammability of natural fiber composites varies. The fire resistance of natural fiber composites can be improved by lowering the cellulose content of the fiber, increasing crystallinity, and decreasing polymerization. Hence, additives and fillers can enhance natural fiber composite fire resistance (Yiga et al., 2021). Further difficulties in comprehending the flame retardancy performance of additives in composite matrices have occurred as a result of the lack of a consistent criterion for experimental data analysis, as well as some arbitrary judgments based on short datasets (Vahabi et al., 2021). As a result, while composite from OPT has been produced in large quantities, flame retardant composite from OPT has yet to be produced.

Rubberwood, a versatile and sustainable material, has gained prominence in the production of composite boards due to its abundance and eco-friendly properties (Olaniran et al., 2021). However, despite its numerous advantages, the utilization of rubberwood in composite boards is hampered by several limitations. Firstly, the inherent low density and high porosity of rubberwood pose challenges in achieving desired mechanical properties, such as strength and durability, in composite boards. These characteristics often result in reduced load-bearing capacity and susceptibility to moisture absorption, limiting its applications in structural and outdoor settings (Khairun et al., 2019). Secondly, the uneven distribution of natural rubber latex within rubberwood fibers can lead to non-uniform bonding and uneven surface finishes in composite boards, compromising their aesthetic appeal and overall quality (Meethaworn et al., 2020). Addressing these limitations requires innovative approaches in material processing, adhesive formulations, and structural design to enhance the performance and versatility of rubberwood-based composite boards in various applications.

Efforts to overcome the limitations of rubberwood in composite board production face significant challenges in balancing cost-effectiveness with performance enhancement. Despite its eco-friendly credentials, the processing and treatment of rubberwood fibers for composite board manufacturing often incur additional expenses, affecting the overall competitiveness of the end product. Moreover, the variability in rubberwood's properties, influenced by factors such as tree age, growth conditions, and harvesting methods, complicates standardization efforts and quality control measures in composite board production. This variability can lead to inconsistencies in material performance and product characteristics, limiting the scalability and widespread adoption of rubberwood-based composite boards in the construction and furniture industries. Thus, addressing these challenges necessitates comprehensive research and development initiatives aimed at optimizing processing techniques, improving material homogeneity, and establishing stringent quality assurance protocols to unlock the full potential of rubberwood in composite board applications.

This work commenced with developing renewable flame-retardant composites using non-wood resources (oil palm trunks) and non-formaldehyde-based binder (polyvinyl alcohol). This research also tries to explore the potential of the opportunities of turning the oil palm trunk into a flame-retardant composite. This development has the immense potential of generating as much in reducing the oil palm biomass.

This research started with screening composites made from treated oil palm trunks with sodium chloride, then bonded with polyvinyl alcohol and citric acid as crosslinkers and calcium carbonate as additives. The composites were analyzed and optimized using the central composite design (CCD) of response surface methodology (RSM). The RSM is a statistical analysis used by many researchers to study the interactions between two or more parameters (Deng et al., 2018; Sulaiman et al., 2018). The RSM is also one of the useful tools that can help by reducing the number of experimental runs while maintaining enough information for statistically acceptable results (Jawad et al., 2014). The RSM can determine the optimum operating factors and find the specific region that satisfies the operating specifications. Moreover, it was a practical and systematic experimental design to present the overall effect of the investigated parameters coupled with CCD and then further subjected to regression analysis (Baskaran et al., 2015; Razak & Sharif, 2015). The optimization of flame-retardant composites focuses on the highest LOI and highest strength of composites mixture of PVOH, NaCl, and $CaCO_3$. Then, the comparative study with rubberwood was done using the optimization from OPT flame retardant composite board.

1.3 Objectives

This study investigates the potential of flame retardant composites with PVOH as binder and $CaCO_3$ as filler. The objectives are:

1) To investigate the factors that affect the mechanical and flame retardant properties of oil palm trunk (OPT) composites by response surface methodology (RSM).

2) To optimize the mechanical and flame retardant properties of combination NaCl, PVOH and $CaCO_3$ ratio of oil palm trunk (OPT) composites based on response surface methodology (RSM).

3) To study the mechanical, flame retardant and morphological properties of OPT flame retardant composites and rubberwood composites.

1.4 Scope of Study

This research details the production of flame-retardant composites from treated oil palm trunks with NaCl, $CaCO_3$ as filler, and PVOH as additives. Therefore, it provides data on optimizing sodium chloride, calcium carbonate, and polyvinyl alcohol production. Characteristic, properties and flame-retardant performance is improving. Besides that, this research also contributed to the environment as it helps to reduce the waste from oil palm trunks. To achieve these objectives, the work would be divided into the following steps: The first step was the preparation of pre-treatment. The oil palm trunk particles were screened to obtain 10mm to 20mm size. The OPT particles were then treated with NaCl by immersing them in the different concentration of NaCl solution for 6 hours. The second phase was optimisation, in which the treated particles were mixed together depending on the combination generated by RSM. This work optimised the production condition of flame retardant composites board from oil palm trunk based on the factors and responses explored previously. The comparison investigation using rubberwood was the final stage. The optimal OPT flame retardant composites board was used in this study to determine the ideal circumstances for creating flame-resistant composite boards, which were then compared to rubberwood boards made with the same ratios.

CHAPTER 2

LITERATURE REVIEW

2.1 Composite

Composite board, also known as engineered wood or composite wood, is a versatile material constructed from a blend of wood fibers or particles bonded together with adhesives and other additives. This composite material offers several advantages over traditional lumber, including enhanced durability, dimensional stability, and resistance to warping, splitting, and decay. By combining wood fibers with resins or glues under controlled conditions, manufacturers can create boards with consistent strength and structural integrity, making them suitable for a wide range of applications in construction, furniture making, and interior design. Composite boards can mimic the appearance of natural wood while offering superior performance characteristics, making them an attractive option for projects where traditional wood may not be suitable or practical.

One of the most common types of composite board is particleboard, which consists of small wood particles bonded together with resin and formed into panels under high pressure and heat. Another popular variety is medium-density fiberboard (MDF), made from fine wood fibers bonded together with resin under high pressure. Both types of composite boards are highly versatile and can be easily shaped, cut, and finished to meet specific requirements (Piekarski et al., 2017). Additionally, composite boards are often manufactured using recycled wood fibers and sustainable practices, making them an environmentally friendly choice for many applications. Overall, composite boards offer a cost-effective, durable, and environmentally conscious alternative to traditional lumber for a wide range of construction and woodworking projects.

Biomass composites represent a significant advancement in material science, combining the sustainability of renewable resources with the functional properties required for a wide range of applications. As technology and processing methods improve, the performance and versatility of these composites are expected to increase, making them a vital component of a more sustainable future. Composite materials are experiencing significant growth due to their favorable mechanical properties, chemical resistance, and corrosion resistance. However, the flame retardant characteristics of these materials provide a substantial safety concern in their application. When biomass composites are exposed to fire, they combust quickly, releasing substantial amounts of heat, flames, and smoke (Ren et al., 2022).

Composite materials are widely employed in contemporary culture owing to their lightweight nature, exceptional mechanical properties, chemical stability, resistance to corrosion, ease of production, and high specific strength. However, the inherent flammability of these substances, attributed to their chemical composition and organic constituents, imposes significant constraints on their viability for utilization in many sectors such as electrical and electronic, construction, automotive, transportation, textile, and aerospace industries (Wang et al., 2023). Therefore, research on flame-retardant biomass composites is quite important. Flame retardant chemicals are frequently used in the flame retardant modification of composite materials (Liu et al., 2023). Table 2.1 displays the earlier research on flame-retardant treatment composites.

Table 2.1 Previous study of flame retardant treatment composites.

Authors	Title
Setyayunita et al., 2022	Effect of different conditions of sodium chloride treatment on the characteristics of Kenaf Fiber-Epoxy composite board.
Jiang et al., 2015	Effect of flame retardant treatment on dimensional stability and thermal degradation of wood.
Yihua Ren et al., 2022	Mechanically strong, thermostable and flame retardant composite enabled by brown paper made from bamboo.
Vo & Navard, 2016	Treatments of plant biomass for cementitious building materials – A review
Cheng et al., 2019	A bio-resourced phytic acid/chitosan polyelectrolyte complex for the flame retardant treatment of wool fabric.

2.1.1 Types of Composites and its Applications

Composites, engineered materials formed by combining different components, come in various types, each tailored to specific applications, offering enhanced properties compared to individual constituents (Ashraf et al., 2019). Fiber-reinforced composites, consisting of fibers such as carbon or glass embedded in a polymer matrix, find widespread use in industries like aerospace for lightweight and strong components, in automotive applications for durable body parts, and in sports equipment where a balance of strength and weight is crucial. Metal matrix composites, combining metals like aluminum or titanium with reinforcing elements such as ceramics or carbon fibers, are employed in aerospace for structural components and in automotive and military applications due to their improved mechanical characteristics. These composites exemplify the versatility and strength achievable by integrating dissimilar materials.

A biomass composite is a material created by combining natural fibers or other organic substances with a matrix or binder, typically a polymer. These composites leverage the mechanical properties of both natural and synthetic components, resulting in materials that are often more sustainable and environmentally friendly than traditional composites. For instance, natural fiber composites, which incorporate materials like jute or flax into a polymer matrix, serve as eco-friendly alternatives in automotive interiors and construction. Carbon-carbon composites, consisting of carbon fibers within a carbon matrix, are used in aerospace for their high-temperature performance. Biomedical implants and structural components utilize functionally graded composites. Concrete composites reinforced with fibers or polymers are crucial in construction for their enhanced toughness and durability in structures, bridges, and infrastructure projects. The diverse range of composite materials highlights their essential role in addressing specific engineering challenges across various industries.

2.2 Raw Materials for Wood Composites

Wood composites are innovative materials that combine natural wood fibers with other constituents, providing enhanced properties and a sustainable alternative to traditional wood products. The primary raw material for wood composites is wood itself, sourced from various timber species. This natural component contributes to the composite's aesthetic appeal and structural integrity. Wood fibers, obtained through processes like chipping or sawing, are then combined with a binder, typically a polymer resin, to form the composite. This combination results in a material that retains the look and feel of wood while exhibiting improved durability, resistance to decay, and dimensional stability. Wood as a raw material in these composites aligns with sustainability principles, as it allows for using wood byproducts and waste from the lumber industry, reducing environmental impact.

In addition to wood, non-wood materials are increasingly employed as raw components in wood composites, expanding the range of available resources. Agricultural fibers such as rice husks, straw, or bamboo, are notable examples. These non-wood fibers offer unique properties, including high strength and low environmental impact, making them valuable additions to wood composites (Nuryawan et al., 2022). By incorporating a variety of raw materials, wood composites can be tailored for specific applications, from construction materials to furniture and decking, demonstrating the versatility and eco-friendly nature of these innovative materials.

2.2.1 Oil Palm Trunk

The oil palm (*Elaeis guineensis* Jacq.) is native to West Africa's tropical rainforest region. It's a perennial crop produced primarily for its palm oil. Palm oil comes from the mesocarp, whereas kernel oil comes from the kernel. Between the 14^{th} and 17^{th} centuries, palm fruit is brought to the Americas and later to the Far East. The plant appears to have thrived in the Far East, yielding the most significant commercial output far from its native land (Asyraf et al., 2021).

Oil palm stands out as a pivotal commercial crop in numerous nations, with its plantations extensively established globally. In Malaysia, specifically, oil palm assumes a noteworthy status as an agricultural commodity, contributing substantially to the nation's economic revenue, ranking fourth (Asyraf et al., 2021; Nordin et al., 2017). Annually, Malaysia generates millions of tons of oil palm biomass, owing to the exceptional productivity of its palm oil sector. Biomass derived from oil palms is readily available in substantial quantities, sourced from both plantations and processing mills.

Oil palm plantations yield biomass in the form of oil palm fronds (OPF), oil palm trunks (OPT) empty fruit bunch (EFB), fronds, palm-pressed fiber, and shells as shown in Figure 2.1 are all possible biomaterials derived from lignocellulosic biomass (Asyraf et al., 2021; Saadon et al., 2014). OPT, harvested during fresh fruit bunches collection and palm tree pruning in plantation areas, contributes significantly to this biomass (Selamat et al., 2019; Zahari et al., 2015). The palm oil industry has witnessed rapid expansion due to increasing demand, leading to a substantial rise in production over recent decades, with nearly threefold growth every ten years (Khatun et al., 2017; Lim et al., 2021). Palm oil and palm kernel oil together make up more than 60% of global exports, with palm oil experiencing significant growth in the global market over the past forty years. Malaysia, a key player, has averaged an annual palm oil production of 15.4 million tonnes between 2016 and 2020 (Asyraf et al., 2021), reflecting the industry's robust growth trajectory.

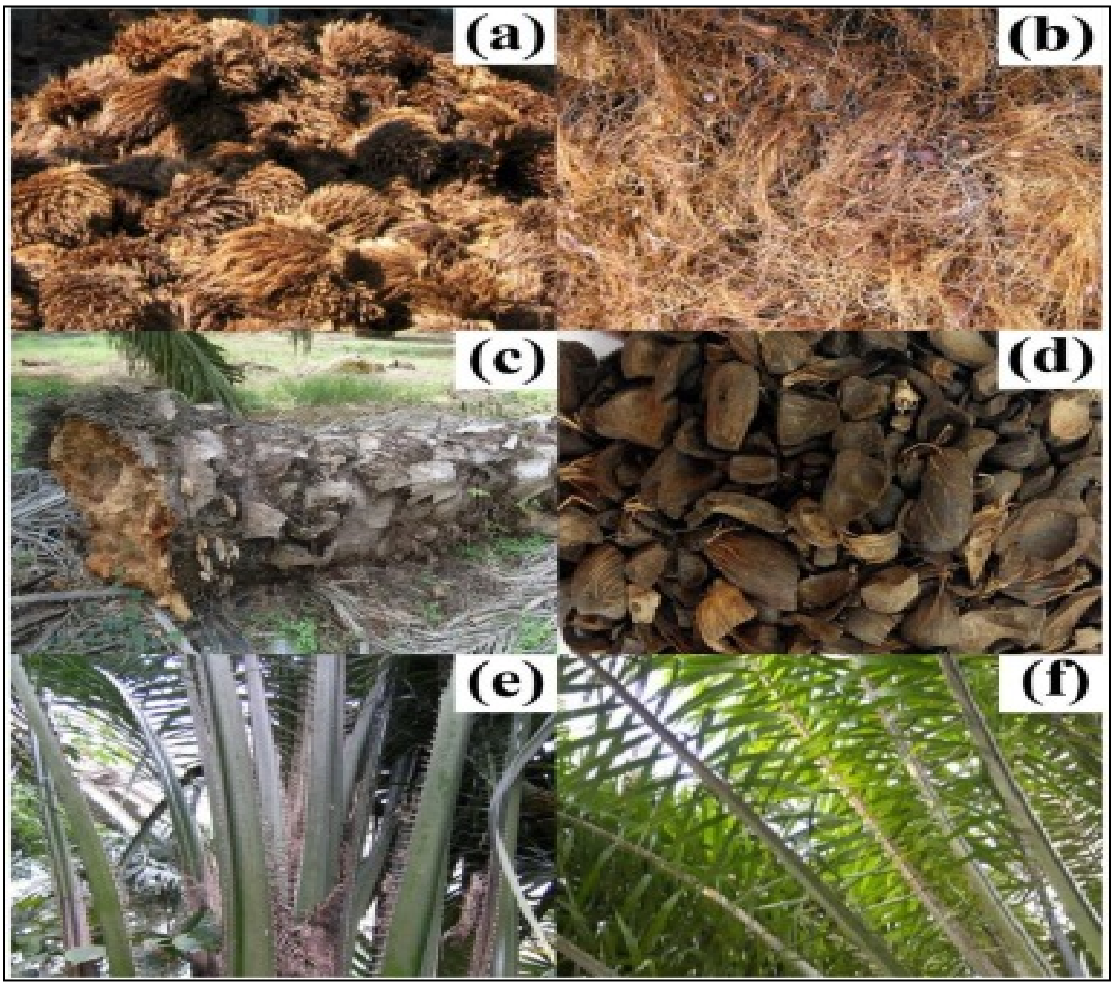

Figure 2.1: Oil palm biomass (a) empty fruit bunch, (b) mesocarp fibers, (c) oil palm trunk, (d) palm kernel shell, (e) oil palm frond, (f) oil palm leaf (Terry et al., 2021).

Hence, many researchers are interested in oil palm biomass utilization because it is abundant and can be transformed into value-added commodities such as bio-plastic, bio-sugars, nitrocellulose, energy, biogas, bio-hydrogen, adsorbents, and polymer composites (Ariffin et al., 2017; Asyraf et al., 2021; Norrrahim et al., 2021; Sukiran et al., 2017). The oil palm tree is a monocotyledonous species, meaning it lacks cambium, secondary growth, growth rings, ray cells, sapwood and heartwood, and branches and knots found in other timber species. Based on this criterion, the oil palm tree was different with wood species (Saari et al., 2020). The parenchymatous ground tissue of the oil palm trunk grew in diameter through cell division and cell enlargement. Aside from that, the increased size of the vascular bundle aids in the growth of the oil palm trunk (Othman et al., 2012). The oil palm trunk (OPT) consists of vascular bundles embedded in parenchymatous ground tissues and serves as a support system for the circulatory system and a storage organ.

The cortical, periphery, and central portions of the oil palm trunk are three separate regions. The outer bark of the oil palm comprises a narrow cortex 1.5–3.5 cm in diameter. Ground parenchyma tissues with numerous disordered and irregular longitudinal fibrous strands and vascular bundles encrusted in the region's majority make up this (Saari et al., 2020). Meanwhile, a limited layer of parenchyma makes up the periphery zone of the oil palm plant, which is responsible for providing mechanical support. Figure 2.2 show the micrograph of an oil palm trunk in cross-section. The sclerotic area comprises crowded vascular bundles and fibrous phloem sheaths. On the other hand, it seemed slightly more significant in the center zone, the vascular bundle near the core of the oil palm trunk, with a wide scattering distribution.

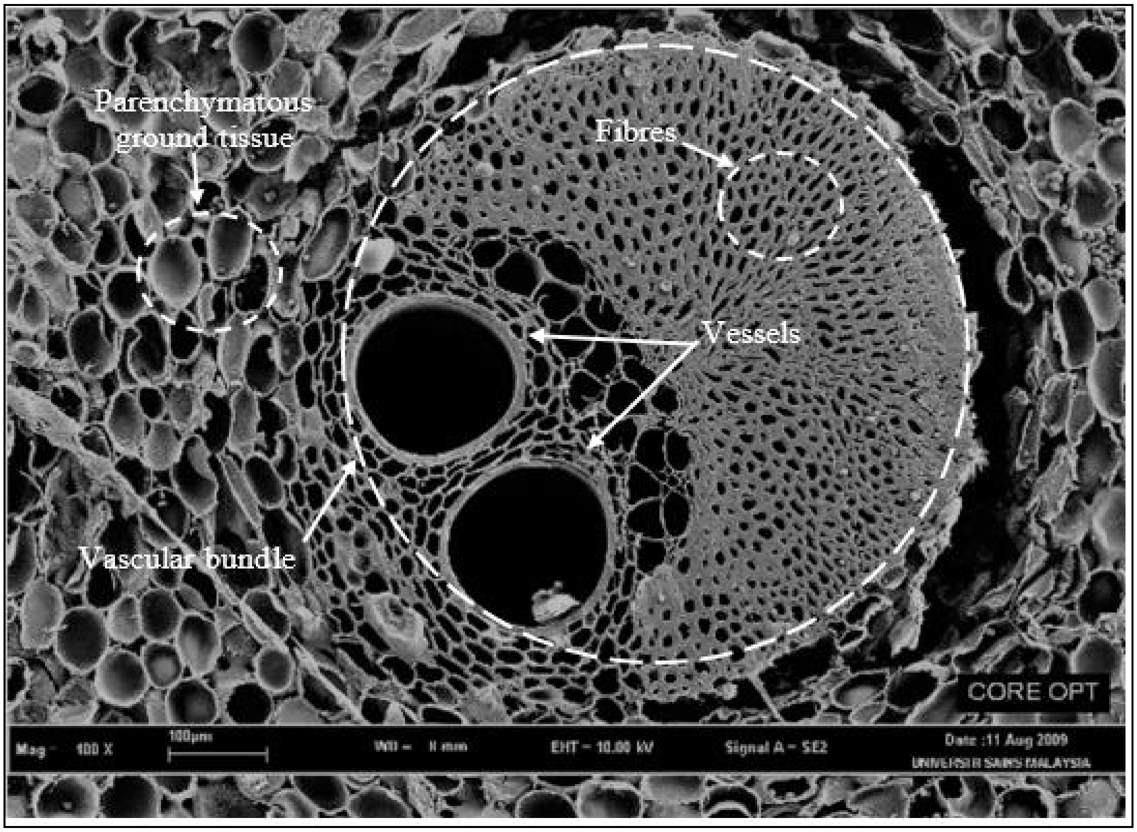

Figure 2.2: Micrograph of an oil palm trunk in cross-section (Hashim et al., 2012) with 100 x magnification.

OPT is one of the most potential lignocellulosic materials produced by oil palm farms (Bukhari et al., 2019). In 2019, 5.85 million hectares of oil palm plantations in Malaysia produced roughly 21.8 million tonnes of OPT (on a dry-weight basis) (Parveez et al., 2021). Many studies have been carried out to determine the potential of oil palm trunks as a raw material for wood-based products to replace timber supplies. Oil palm trunk has been used to make composites, binderless particleboard, laminated veneer lumber, and plywood (Khalid et al., 2015; Nadhari et al., 2014a; Nordin et al., 2017; Othman et al., 2012). Some research studies employ oil palm trunk products in various applications, including automobile parts, furniture, packaging, and sawn lumber (Saari et al., 2020). As seen by a small amount used for plywood manufacturing and the large amounts left unused at planting sites, such OPT is underutilized (Bukhari et al., 2019). Extensive research efforts have been dedicated to exploring the capabilities of oil palm trunks for wood-based products, aimed at replacing traditional timber sources. A wide range of products has been developed from oil palm trunks, including composite materials (Saari et al., 2020).

2.2.2 Rubberwood

Rubberwood *(Hevea brasiliensis)* (Willd. ex. Adr. de Juss Muell. Arg) is one of Malaysia's most important raw materials for making particleboard (Lee et al., 2015). Rubberwood, which originated as an indigenous species in Brazil's Amazon Forest, was first introduced to South East Asia in the mid-1800s (Teoh et al., 2011). The rubberwood (RBW) tree is a light hard-wood tree with low branching, a white to pale cream color, good machining and working properties, and a density that ranges from 0.48 to 0.65 g/cm3 depending on the tree's age. After the latex production has decreased, typically between 25 and 30 years, they must then be cut down after that period (Homkhiew et al., 2020b). The log was used for furniture and interior fittings, domestic flooring, stair components, and the manufacturing of kitchen cabinets due to the growth rings' attractive color and appearance. Moreover, the log was utilized for domestic flooring (Juliana et al., 2012).

Malaysia achieved considerable success using rubberwood in the manufacture of value-added products in the early 1990s, becoming the leader in South East Asia. Malaysia's rubberwood sawn timber industry is well established and makes efficient use of this resource. Previously thrown as waste, woody biomass has found new use as the main raw material for Malaysia's growing wood industry, especially after the availability of logs from the country's natural forests began to decline in the mid-1980s (Ratnasingam et al., 2015). Currently, waste materials from furniture and lumber manufacturing, as well as low-quality tiny logs, are the primary raw materials used by Malaysian composite panel manufacturers (Amini et al., 2013). Rubberwood timber is commonly used to make furniture, toys, and culinary utensils. Because its mechanical qualities are equivalent to or better than those of structural hardwood, the use of rubberwood for structural applications may raise demand for rubberwood (Srivaro et al., 2021). Rubber plantation acreage is decreasing throughout the country year by year due to land acquisition for housing and industrial expansion, as well as the conversion of rubber plantations to oil palm production (Abdullah et al., 2012).

2.2.3 Application of Rubberwood

Rubberwood's potential was seen as early as the 1950s, and it is currently considered to be the most significant raw material for Malaysia's wood industry. Rubberwood processing for wood goods was not widely accepted, nevertheless, due to the abundance of wood available in natural forests. This was made even more difficult by the wood's poor durability. In the mid-1970s, the Malaysian Timber Industry Board (MTIB)