CHARACTERISATION OF WOVEN KENAF/POLYESTER REINFORCED POLYLACTIC ACID HYBRID COMPOSITES

By

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

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IPTPH 2023 4

DEDICATION

To Al-Quran, the greatest source of knowledge

Bring me sheets of iron" - until, when he had leveled [them] between the two mountain walls, he said, "Blow [with bellows]," until when he had made it [like] fire, he said, "Bring me, that I may pour over it molten copper." (Al-Kahf:Verse 96)

To my beloved father and mother for their invaluable sacrifices, encouragement and support throughout my life

To my beloved wife for her love, patience and understanding

To my beloved daughter and son

To my awesome siblings

Abstract of thesis presented to the Senate of Universiti Putra Malaysia in fulfillment of the requirement for the degree of Doctor of Philosophy

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January 2023

Chairman : Mohd Sapuan bin Salit, PhD, PEng
Institute : Tropical Forestry and Forest Product

Growing concerns about the disposal of petroleum-based products and the demand for high-strength materials have stimulated interest in the use of biodegradable materials with higher strength for the production of hybrid composites. The objective of this study is to determine the mechanical, thermal, morphological and flammability properties of woven kenaf/polyester reinforced polylactic acid hybrid composites. The composites were fabricated by the hot-press method. The effects of different fibre contents and stacking sequences were examined. The first and second phases focused on the characterization of woven polyester-reinforced polylactic acid and woven kenaf-reinforced polylactic acid composites. The characterization focused on the effects of different fibre contents on both composites. Increasing the polyester fibre content in the composites resulted in the highest improvement in tensile and impact strength of the composites by up to 225% and 93%, respectively. For the woven kenaf/PLA composites, the results show that the addition of woven kenaf improved tensile properties by 47%. In the third and fourth phases, the effects of different fibre contents and stacking sequences of the hybrid composites were investigated. The sample with 4 layers of woven polyester and 1 layer of woven kenaf (S4) showed a significant percentage increase in tensile strength and elongation at break among the hybrid composites by 151% and 714%, respectively. However, the higher composition of woven kenaf in the hybrid laminated composites exhibited better flexural properties. The addition of woven polyester in the composites improved the thermal stability and degradation of the composites. In terms of flammability, the result shows that the addition of kenaf fabric was responsible for the high char yield and extended the burning time of the hybrid composites. The S5 sample with 4 layers of woven kenaf and 1 layer of woven polyester also shows the optimum viscoelastic properties such as storage modulus and loss modulus among the hybrid composites. In summary, the optimum composition for woven kenaf/polyester/PLA hybrid composites was determined for the S5 sample. All data were statistically analysed using a one-way analysis of variance and showed significant differences between the results. Overall, based on the results, these hybrid composites are suitable for use in non-load-bearing applications such as car headliners and door panels.

Abstrak tesis yang dikemukan kepada Senat Universiti Putra Malaysia sebagai memenuhi keperluan untuk Ijazah Doktor Falsafah

PENCIRIAN KOMPOSIT HIBRID ASID POLILAKTIK DIPERKUAT GENTIAN KENAF/POLIESTER TERTENUN

Oleh

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Peningkatan kesedaran tentang pelupusan produk berasaskan petroleum dan permintaan untuk bahan berkekuatan tinggi telah merangsang minat dalam penggunaan bahan terbiodegradasi dengan kekuatan yang lebih tinggi untuk pengeluaran komposit hibrid. Objektif kajian ini adalah untuk menentukan sifat mekanikal, termal, morfologi dan mudah terbakar bagi komposit hibrid asid polilaktik diperkuat fabrik kenaf dan poliester. Komposit telah dibuat dengan kaedah hot-press. Kesan kandungan serat dan urutan susunan fabrik yang berbeza telah diperiksa. Fasa pertama dan kedua tertumpu kepada pencirian komposit tenunan poliester memperkuat asid polilaktik dan komposit tenunan kenaf memperkuat asid polilaktik. Pencirian tertumpu kepada kesan kandungan serat yang berbeza pada kedua-dua komposit. Peningkatan kandungan serat poliester dalam komposit menghasilkan peningkatan tertinggi dalam kekuatan tegangan dan hentaman komposit masing-masing sehingga 225% dan 93%. Bagi komposit tenunan kenaf/PLA, keputusan menunjukkan penambahan tenunan kenaf meningkatkan sifat tegangan sebanyak 47%. Dalam fasa ketiga dan keempat, kesan kandungan serat yang berbeza dan urutan susunan komposit hibrid telah diselidik. Sampel dengan 4 lapisan fabrik poliester dan 1 lapisan fabrik kenaf (S4) menunjukkan peningkatan peratusan yang ketara dalam kekuatan tegangan dan pemanjangan putus antara komposit hibrid masing-masing sebanyak 151% dan 714%. Walau bagaimanapun, komposisi kenaf yang lebih tinggi dalam komposit berlamina hibrid menunjukkan sifat lentur yang lebih baik. Penambahan poliester dalam komposit meningkatkan kestabilan terma dan degradasi komposit. Dari segi kemudahbakaran, keputusan menunjukkan penambahan fabrik kenaf bertanggungjawab terhadap hasil arang yang tinggi dan memanjangkan masa pembakaran komposit hibrid. Sampel S5 dengan 4 lapisan fabrik kenaf dan 1 lapisan fabrik poliester juga menunjukkan sifat viskoelastik yang paling optimum seperti modulus penyimpanan dan modulus kehilangan antara komposit hibrid. Secara ringkasnya, komposisi optimum untuk komposit fabrik kenaf/poliester/PLA telah ditentukan untuk sampel S5. Kesemua data dianalisis secara statistik menggunakan analisis varians sehala dan menunjukkan perbezaan yang ketara antara keputusan. Secara keseluruhan, berdasarkan keputusan, komposit hibrid ini sesuai digunakan dalam penggunaan tanpa beban seperti siling kereta dan panel pintu kereta.

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In the name of Allah, the Most Gracious, the Most Merciful

In the Name of Allah, the Most Merciful, the Most Compassionate,

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

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TABLE OF CONTENTS

			Page
ABSTRACT			i
ABSTRAK			ii
ACKNOWLEDGEMENTS			iii
APPROVAL			iv
DECLARATION			vi
LIST OF TABLES			xi
LIST OF FIGURES			xii
LIST OF SYMBOLS			xv
LIST OF ABBREVIATIONS			xvi

CHAPTER

1	**INTRODUCTION**		1
	1.1	Background	1
	1.2	Problem statements	2
	1.3	Research Objectives	4
	1.4	Significance of Study	4
	1.5	Scope and limitation of the study	5
	1.6	Structure of the Thesis	5
2	**LITERATURE REVIEW**		9
	2.1	Natural fibres	9
		2.1.1 Plant fibres	9
	2.2	Synthetic fibres	11
		2.2.1 Polyester	12
	2.3	Polymer Matrix	13
		2.3.1 Polylactic acid (PLA)	14
	2.4	Composites	14
		2.4.1 Fibre reinforced composites	15
	2.5	Fibre reinforced Polylactic (PLA) composites	16
		2.5.1 Short fibres reinforced PLA composites	16
		2.5.2 Particles reinforced PLA composites	18
		2.5.3 Woven fabrics reinforced PLA composites	18
		2.5.4 Fibre reinforced PLA hybrid composites	19
	2.6	Processing methods	21
		2.6.1 Compression moulding	22
	2.7	Mechanical properties of fibre reinforced PLA composites	22
	2.8	Application of fibre reinforced composites	23
		2.8.1 Structural Application	24
		2.8.2 Automotive Application	24
	2.9	Summary	25
3	**MATERIALS AND METHODS**		27
	3.1	Introduction	27
	3.1	Materials	27
	3.2	Material preparation	27

3.3 Composite fabrication 28
3.3.1 Hand lay-up technique 28
3.3.2 Hot press method 29
3.3.3 Hot Press Temperature profile 29
3.3.4 Fabrication of woven polyester/polylactic composites 30
3.3.5 Fabrication of woven kenaf/polylactic composite 31
3.3.6 Fabrication of woven kenaf/woven polyester/ PLA hybrid composites 32
3.4 Characterization 33
3.4.1 Physical properties 33
3.4.2 Chemical composition of kenaf fibre 34
3.4.3 Mechanical properties 35
3.4.4 Thermal properties 36
3.4.5 Flammability 36
3.4.6 Statistical analysis 37

4 MECHANICAL, MORPHOLOGICAL AND THERMAL PROPERTIES OF WOVEN POLYESTER FIBRE REINFORCED POLYLACTIC ACID (PLA) COMPOSITES 38
4.1 Introduction 39
4.2 Experimental 40
4.2.1 Materials 40
4.2.2 Methods 41
4.2.3 Methods of testing 42
4.3 Results and discussion 44
4.3.1 Scanning Electron Microscope (SEM) Micrograph 44
4.3.2 Mechanical properties of WP/PLA composites 45
4.3.3 Thermal Properties 49
4.4 Conclusions 52
References 53
Proof of Publication 60

5 MECHANICAL, MORPHOLOGICAL AND THERMAL PROPERTIES OF WOVEN KENAF FIBRE REINFORCED POLYLACTIC ACID (PLA) COMPOSITES 61
5.1 Introduction 62
5.2 Experimental 64
5.2.1 Materials 64
5.2.2 Methods 64
5.2.3 Methods of testing 66
5.3 Results and discussion 68
5.3.1 Mechanical properties of WK/PLA composites 68
5.4 Conclusions 76
References 77
Proof of Publication 87

6 **EFFECT OF STACKING SEQUENCE AND FIBRE CONTENT ON MECHANICAL AND MORPHOLOGICAL PROPERTIES OF WOVEN KENAF/POLYESTER FIBRE REINFORCED POLYLACTIC ACID (PLA) HYBRID LAMINATED COMPOSITES** 88
6.1 Introduction 88
6.2 Material and Methods 91
6.2.1 Materials 91
6.2.2 Material preparation 92
6.2.3 Fabrication of hybrid composite laminates 92
6.2.4 Mechanical testing 94
6.3 Results and discussion 95
6.3.1 Tensile properties 95
6.3.2 Flexural properties 98
6.3.3 Impact strength 99
6.3.4 Morphological properties 100
6.4 Conclusions 102
References 103
Proof of Publication 111

7 **THERMAL STABILITY, DYNAMIC MECHANICAL ANALYSIS AND FLAMMABILITY PROPERTIES OF WOVEN KENAF / POLYESTER - REINFORCED POLYLACTIC ACID HYBRID LAMINATED COMPOSITES** 112
7.1 Introduction 113
7.2 Material and Methods 114
7.2.1 Materials 114
7.2.2 Material preparation 115
7.2.3 Fabrication of hybrid composite laminates 115
7.2.4 Thermogravimetric Analysis (TGA) 116
7.2.5 Differential scanning calorimetry (DSC) 116
7.2.6 Dynamic mechanical analysis (DMA) 116
7.2.7 Flammability (UL-94) 117
7.3 Results and discussions 117
7.3.1 Thermal-gravimetric analysis (TGA) 117
7.3.2 Differential scanning calorimetry (DSC) 120
7.3.3 Dynamic mechanical Analysis (DMA) 121
7.3.4 Flammability test 125
7.4 Conclusion 127
References 128
Proof of Publication 138

8 **CONCLUSION AND RECOMMENDATIONS** 139
8.1 Conclusions 139
8.2 Recommendations for Future Research 141

REFERENCES 142
APPENDICES 179
BIODATA OF STUDENT 183
LIST OF PUBLICATIONS 184

LIST OF TABLES

Table		Page
2.1	Chemical composition, and moisture content of plant fibres	9
2.2	Most common polymers used to make composites	13
2.3	Applications of natural fibres in the automotive industry	25
3.1	Characterization of WP, WK and PLA film	27
3.2	Formulation of WP/PLA composites	31
3.3	Formulation of WK/PLA composites	31
3.4	Formulation of the woven kenaf/woven polyester/ PLA hybrid composites	32
4.1	Characterization of WP	41
4.2	Composition of WP/PLA composites	42
4.3	Density of WP0 and WP/PLA composites	42
5.1	Composition of WK/PLA composites	65
5.2	Summary of the analysis of variance (ANOVA) of mechanical properties of WK/PLA composites	72
6.1	Fibre matrix ratio and thickness of the laminated composites	92
6.2	Summary of the analysis of variance (ANOVA) of mechanical properties	100
7.1	Chemical composition of woven kenaf	115
7.2	Fibre matrix ratio and thickness of the laminated composites	116
7.3	The transition temperature at 5% weight loss ($T_{5\%}$), initial degradation temperature (T_{max1}), major degradation temperature (T_{max2}), final degradation temperature (T_{max3}) and residue of laminated composites	119
7.4	The values of glass transition temperature (T_g), melting temperature (T_m) and the cold crystallization peak temperatures (T_c) of laminated composites	120
7.5	The UL94 combustibility properties of the laminated composites	126

LIST OF FIGURES

Figure		Page
1.1	Flow process of the method	8
2.1	Classification of synthetic fibres according to organic, inorganic and other fibres	11
2.2	Reinforcement form	15
2.3	Fabrication procedure of laminated composites	17
2.4	Kenaf/Bamboo/Coir/Coir/Bamboo/Kenaf (KBCCBK) composites	20
2.5	Schematic diagram of Compression moulding for composites	22
2.6	SmartFit PLA Bamboo	24
3.1	a) Woven polyester (WP) b) Woven kenaf (WK) c) Polylactic acid film (PLA)	27
3.2	a) Layered woven fabric and film b) Layered woven fabric and film in a mould	28
3.3	Layering of woven fabric/PLA film for each composite	28
3.4	Hot press method	29
3.5	Hot press temperature profile	30
3.6	a) Layered WP/PLA composite b) Layered WP/PLA film in a mould c) Hot compression process of WP/PLA composite	30
3.7	Schematic representation of the manufacturing process of WK/PLA composit	31
3.8	a) Layered WK/WP/PLA b) Layered WK/WP/PLA in a mould c) Hot press process of hybrid laminated composite d) Hybrid laminated composites	32
3.9	Stacking sequences of hybrid composites	33
3.10	Woven composite burning marks sample for flammability test	37
4.1	Woven polyester (WP)	41

4.2	a) Layered WP/PLA composite b) Layered WP/PLA film in a mould c) Hot compression process of WP/PLA composite	42
4.3	SEM micrograph of WP/PLA with different fibre content a) WP0, b) WP20 c) WP30 and d) WP40	45
4.4	Tensile strength of WP/PLA composites	46
4.5	Tensile modulus of WP/PLA composites	46
4.6	Elongation of WP/PLA composites	47
4.7	Flexural strength of WP/PLA composites	48
4.8	Flexural modulus of WP/PLA composites	48
4.9	Impact strength of WP/PLA composites	49
4.10	TG curves of WP0 and WP/PLA composites	50
4.11	DTG curves of WP0 and WP/PLA composites	51
4.12	DSC curves of WP0 and WP/PLA composites	52
5.1	(a) Woven kenaf/PLA, (b) Woven kenaf/PLA in a mould, and (c) WK/PLA composites	65
5.2	Schematic representation of the manufacturing process of WK/PLA composite	65
5.3	Hot compression temperature profile	66
5.4	Tensile properties of WK/PLA composites	69
5.5	Flexural properties of WK/PLA composites	70
5.6	Impact strength of WK/PLA composites	71
5.7	SEM micrograph of WK/PLA with different fibre fractions (a) WK0, (b) WK/PLA composite (c) 30% of WK/PLA composite (d) 40% of WK/PLA composite	73
5.8	TG thermogram of WK0 and WK/PLA composites	74
5.9	DTG thermogram of WK0 and WK/PLA composites	75
5.10	DSC thermogram of WK0 and WK/PLA composites	76
6.1	Stacking sequences of laminated composites	92

6.2 a) Layered WK/WP/PLA b) Layered WK/WP/PLA in a mould c) Hot press process of hybrid laminated composite d) Hybrid laminated composites 93

6.3 Hot compression temperature profile 93

6.4 The effect of stacking sequence and fibre content on the (a) tensile strength, (b) tensile modulus, (c) elongation at break (%) of composites 96

6.5 The effect of stacking sequence and fibre content on the (a) Flexural strength, (b) Flexural modulus of composites 98

6.6 Impact strength of the laminates 100

6.7 Morphological micrograph of hybrid laminated composites of S1, S2, S3, S4, and S5 tensile fracture 101

7.1 Stacking sequences and fibre content of laminated composites 115

7.2 TG thermogram of laminated composites 117

7.3 DTG thermogram of laminated composites 119

7.4 DSC thermogram of laminated composites 120

7.5 The storage modulus of different laminated composites 122

7.6 The loss modulus of different laminated composites 123

7.7 The Tan δ curves of different laminated composites 124

7.8 The burning rate of laminated composites 126

LIST OF SYMBOLS

^{o}C	Degree Celcius
ρ	Density
T_c	Crystallization Temperature
T_g	Glass transition
T_m	Melting temperature
T_{Onset}	Onset temperature
MPa	Mega pascal
wt.%	Weight percentage

LIST OF ABBREVIATIONS

ANOVA	Analysis of Variance
ASTM	American Society for Testing and Materials
DMA	Dynamic mechanical analysis
DTG	Derivative thermogravimetry
DSC	Differential Scanning Calorimetry
FESEM	Field emission scanning electron microscopy
PLA	Polylactic Acid
SEM	Scanning Electron Microscopy
TG	Thermogravimetry
TGA	Thermal-gravimetric analysis
WK	Woven kenaf
WP	Woven polyester

CHAPTER 1

INTRODUCTION

1.1 Background

The rapid growth of manufacturing has necessitated the development of stronger, stiffer, denser, and less expensive materials that are also more environmentally friendly. Composites are a type of material with improved properties that have the potential to be used in many different areas (Chukov et al., 2019; Linul et al., 2019; Sherif et al., 2019; Gowda et al., 2018). Composites are composed of several materials, one of which serves as the matrix phase, and the others may be in the form of particles or fibres.

In recent years, natural and synthetic fibres have been widely used to reinforce polymer composites. Many people are interested in fibre-reinforced composites because they have many desirable properties, such as lightweight, high strength, corrosion resistance, and durability. Fibre-reinforced composites can not only maintain their original properties but also improve or compensate for the weaknesses of a single material (Yang et al., 2019). The advantage of compensating for the weaknesses of a single material by combining different materials into hybrid composites. These hybrid composites offer improved and combined properties compared to typical single fibre composites.

Over the past two decades, the amount of plastic waste produced worldwide has doubled, but only 9% of it is properly recycled, with the majority ending up in landfills, incinerated, or released into the environment (*Global Plastics Outlook*, 2022). The rapid increase in landfills around the world has raised consumer awareness of the importance of protecting the environment. Consumers are becoming more environmentally conscious, and environmentally friendly products are preferred.

Various industries have contributed to the generation of plastic waste, such as textiles, electronics, consumer products, and even construction cannot avoid contributing to the waste problem. In addition, the polymer industry made product from synthetic fibres derived from fossil fuels. Synthetic fibres, which are byproducts of petroleum, are not biodegradable and will take a longer time to decompose, resulting in long-term pollution. Therefore, various initiatives have been taken to solve the problems related to environmental pollution.

Consumer awareness of environmental pollution has also led to natural fibres becoming an important material in the composites industry. Natural fibres offer several advantages over synthetic fibres, such as low density, sustainability, biodegradability, cost efficiency, and good mechanical properties.(Hazrati et al., 2021a; Kandola et al., 2018). The natural fibre kenaf has become one of the most important natural fibres in the production of green composites (Manral & Bajpai, 2020).

In the automotive industry, kenaf fibres are used to manufacture parts of automotive components such as interior trim (Sreenivas et al., 2020), dashboards and headliners. The use of natural fibres such as flax, kenaf, hemp, sisal and jute fibres leads to the production of lightweight products (Holbery & Houston, 2006). Reportedly, 80,000 to 160,000 tonnes of natural fibres are used annually in automotive production in Western Europe (Jamrichov & Akov, 2013). The German-based car manufacturer is a leader in the use of natural fibres in car parts. It is reported that Daimler-Chrysler is also initiating programs to develop natural fibre-based products to benefit the agricultural industry.

Cunha et al. (2006) reported that the 2003 Toyota RAUM model is among the vehicles that use natural fibres as a spare tire cover. The cover is made of kenaf fibres reinforced with polylactic acid to produce composite materials. During EcoInnovAsia 2008, the Mazda 5 used kenaf/polylactic acid composites as seat covers and claimed that nearly 30% of the Mazda 5's interior components are made of bio-based materials (Suddell, 2008). The use of natural and synthetic fibres in hybrid composites is becoming increasingly common in a variety of technical and engineering disciplines. The result is a better balance between performance and cost for products made from hybrid composites.

Insufficient research has been conducted on the combination of woven kenaf/polyester/polylactic acid hybrid composites. No study has been conducted on the mechanical, morphological, thermal, and flammability properties of the woven kenaf/polyester reinforced polylactic acid in hybrid composites. Therefore, this research focuses on the use of woven kenaf, woven polyester, and polylactic acid in hybrid composites.

1.2 Problem statements

Fibre-reinforced polymer composites are made by reinforcing the polymer matrix with natural or synthetic fibres or a combination of both. The choice of fibres and matrix is very important because the properties of the composites are highly dependent on the properties of the individual components (Azman et al., 2021; Karthi et al., 2019; Prashanth et al., 2017).

The high consumption of petroleum-based polymers by the industry has led to serious environmental problems, especially in the disposal phase. When these wastes are released into the environment, they do not biodegrade quickly, causing significant problems for the ecosystem. The environmental problem of the degradation of plastics associated with synthetic-based composites has attracted considerable critical attention. Manufacturers and scientists are under increasing pressure to find environmentally friendly and sustainable materials to replace existing synthetic fibres and reduce dependence on petroleum-based products. This is in response to environmental legislation combined with demands from industry and consumers around the world (Lotfi et al., 2021). Despite environmental concerns, other disadvantages of synthetic fibre composites include their high price, high energy consumption during processing and manufacturing, poor recyclability and lack of renewability, CO_2 emissions, and inhalation health concerns (Bichang'a et al., 2022; Nassar et al., 2017).

The problems with synthetic fibres have led to the use of natural fibres in the composite industry becoming the first choice to solve the problem. Due to the inherent properties of natural fibres, researchers face numerous challenges in the development and application of natural fibre reinforced composites. Even though natural fibres are obtained from renewable sources and the polymer composites based on them are environmentally friendly compared to the composites reinforced with synthetic fibres, there are also some disadvantages associated with the use of natural fibres in the production of the composites The disadvantages of natural fibres, especially high water absorption, deteriorate the mechanical properties by weakening the interfacial bond between the polymer matrix and the fibres (Khalid et al., 2021).

Although biocomposites using natural fibres and biodegradable polymers as materials can overcome the problem of plastic degradation, industry players face problems with natural-based materials, such as variable fibre quality, incompatibility with hydrophobic matrices, low thermal stability, water hydrophilicity, the tendency to agglomerate, and very limited mechanical properties (Li et al., 2020), which do not meet the requirements for high-strength applications. The biggest challenge for many industries is the demand for high-strength composites. The demand forces them to use synthetic materials due to their excellent properties in terms of durability and strength, thermal stability, impact resistance, chemical resistance, and friction (Rajak et al., 2019). The use of kenaf fibres in automotive applications has proven to be a viable and environmentally friendly substitute for synthetic fibres.

The growing demand for high-performance composites for various applications has simultaneously driven the growth of hybrid composites. For instance, lightweight products are preferred in the automotive industry to improve fuel efficiency (Mohammed et al., 2015), and they are also used for non-structural automotive applications (Cicala et al., 2010). Woven fibres from kenaf (Ismail & Che Abdul Aziz, 2015; Salman et al., 2015), bamboo (Ali et al., 2021; Widiastuti et al., 2020), jute (Ahmad & Rahim, 2017; Khan et al., 2016), banana (Gunge et al., 2019), curaua (Costa et al., 2020) and hemp (Corbin et al., 2020; Misnon et al., 2016) have been used as materials for hybrid composites. The production of hybrid composites from natural and synthetic fibres can reduce the use of synthetic fibres and help to reduce the problem of plastic waste. The selection of cheaper synthetic fibres such as polyester, which have excellent mechanical and thermal properties, can also minimise production costs in the manufacture of woven hybrid composites (Erturk et al., 2019; Islam et al., 2022).

In addition, the environmental problem of plastic degradation can be further reduced by using biodegradable polymers such as polylactic acid (PLA). Recently, extensive research has been conducted on PLA as a biodegradable plastic that can also reduce the negative environmental impact caused by the accumulation of non-biodegradable plastic waste (Hamad et al., 2014). PLA is a fascinating biopolymer, both from a commercial and environmental point of view because it has many special properties. Some of these properties include high stiffness, excellent processability, high transparency, and a glossy appearance. Inherent brittleness and low toughness are some of the main limitations that prevent its widespread use. PLA can replace non-biodegradable polymers when recycling is difficult or costly. Although PLA has some limitations due to its material properties, many of these problems can be solved by reinforcing the PLA matrix with natural or synthetic fibres that can compensate for the material's shortcomings.

The aim of this work is to produce hybrid composites of kenaf and polyester reinforced with biodegradable polylactic acid as a resin to meet the demand for environmentally friendly and high-strength products.

1.3 Research Objectives

The objective of this research is to develop and characterize hybrid composites of woven kenaf, woven polyester, and polylactic acid (PLA). The specific objectives are as follows:

1) To investigate the effects of different fibre contents on the mechanical, thermal, and morphological properties of woven polyester-reinforced PLA composites.

2) To determine the effect of different fibre contents on the mechanical, thermal, and morphological properties of woven kenaf reinforced PLA composites.

3) To examine the effect of different stacking sequences and fibre contents on the mechanical and morphological properties of woven kenaf/polyester reinforced PLA hybrid composites.

4) To evaluate the effects of fibre contents on the thermal properties and flammability of woven kenaf/polyester-reinforced PLA hybrid composites.

1.4 Significance of Study

As a result of environmental laws and pollution, many researchers have turned their attention to reducing the use of petroleum-based materials and products. This goal has led researchers to develop a natural material that has both economic and environmental benefits. Natural fibres have become an effective alternative to synthetic fibres in the automotive, aerospace, military, and marine industries due to their good strength-to-weight ratio, natural biodegradability, and low cost.

Few studies have documented the use of woven polyester fibres in the manufacture of composites, with most studies focusing on polyester as a resin. Synthetic fibres are known to have better mechanical and thermal properties than natural fibres. Hybridization of natural and synthetic fibres, on the other hand, combines the advantages of natural and synthetic fibres and results in hybrid composites with better properties.

1.5 Scope and limitation of the study

This research focuses on the fabrication of novel hybrid composites using a combination of woven kenaf/polyester/polylactic acid. Synergistic improvements in the properties of composites composed of two or more types of fibres are the result of using natural/synthetic hybrid composites. The properties of hybrid composites may be more amenable to manipulation, allowing a better balance between the advantages and disadvantages of composites in general. A hot pressing process for the production of the composites.

A series of experiments were conducted to develop and characterize the hybrid composites. The first phase is the material preparation and development of the composites to determine the optimum fibre content for fibre-reinforced composites. In the second phase, the effects of different stacking orders for hybrid composites on mechanical and morphological properties are investigated. In the third phase, the effects of fibre content of hybrid composites on thermal properties and flammability will be investigated.

This study of hybrid woven kenaf/polyester-reinforced PLA composites has some limitations for use in the commercial industry, such as the increase of woven polyester fibres, which reduces the flexural strength of the hybrid composites. This is due to the nature of the woven structure and the higher elasticity of polyester fibres. The reinforcing material in woven form affected the flexural properties of the composites because the structure of the woven reinforcing fibres consists of the gap between two adjacent yarns. The presence of a larger gap area may reduce the flexural properties of the woven fibre-reinforced composite (Adumitroaie & Barbero, 2012). However, the use of natural fibres as one of the materials in the manufacture of composites has limited the use of hybrid composites because they are not suitable for structural applications and outdoor applications and in areas where the water medium and flexural properties are important. The limitation of the study has restricted the potential application of this material. Therefore, appropriate modifications should be made to improve the properties of this material. Possible modifications include differentiating the stacking order and fibre content of woven kenaf and woven polyester.

1.6 Structure of the Thesis

The structure of the dissertation follows the Universiti Putra Malaysia alternative dissertation format based on publications. Each research chapter (4 - 7) represents a separate study that has its own sections: 'Introduction', 'Materials and Methods', 'Results and Discussion', and 'Conclusion'. More details about the structure of the paper are given below. Figure 3.1 shows the flow of the research methodology.

Chapter 1

This chapter contains a concise summary of the issues that prompted this investigation and the objectives of the research conducted. The important contribution made by the research and its overall scope is also highlighted in this chapter.

Chapter 2

This chapter provides a comprehensive review of the literature in the key areas related to the title of this thesis. This chapter also explains the research gaps identified in the literature review.

Chapter 3

This chapter describes all actions related to this study, from material preparation to processing, testing, and analysis of results.

Chapter 4

This chapter presents the first article entitled "Mechanical, Morphological and Thermal Properties of Woven Polyester Fibre Reinforced Polylactic Acid (PLA) Composites". In this article, the study of the effect of different fibre content on mechanical, thermal and morphological properties of the woven polyester reinforced polylactic acid composites, was accomplished.

Chapter 5

This chapter presents the second article entitled "Mechanical, Morphological and Thermal Properties of Woven Kenaf Fibre Reinforced Polylactic Acid (PLA) Composites". In this article, the study of the effect of different fibre content on mechanical, thermal, and morphological properties of the woven kenaf reinforced polylactic acid composites were evaluated.

Chapter 6

This chapter presents the third article entitled "Effect of stacking sequence and fibre content on mechanical and morphological properties of woven kenaf/polyester fibre reinforced polylactic acid (PLA) hybrid laminated composites". In this article, the influence of fibre content and different stacking sequences on the mechanical and morphological properties of woven kenaf/polyester fibre reinforced polylactic acid (PLA) hybrid laminated composites was investigated.

Chapter 7

This chapter presents the fourth article entitled "Thermal Stability, Dynamic Mechanical Analysis and Flammability Properties of Woven Kenaf/Polyester-Reinforced Polylactic Acid Hybrid Laminated Composites.". This article studied the effect of the influence of different fibre content on thermal stability, dynamic mechanical analysis, and flammability properties of woven kenaf/polyester-reinforced polylactic acid hybrid laminated composites.

Chapter 8

This chapter will elaborate on the general conclusions drawn from the entire study, as well as recommendations for improving this study in the future

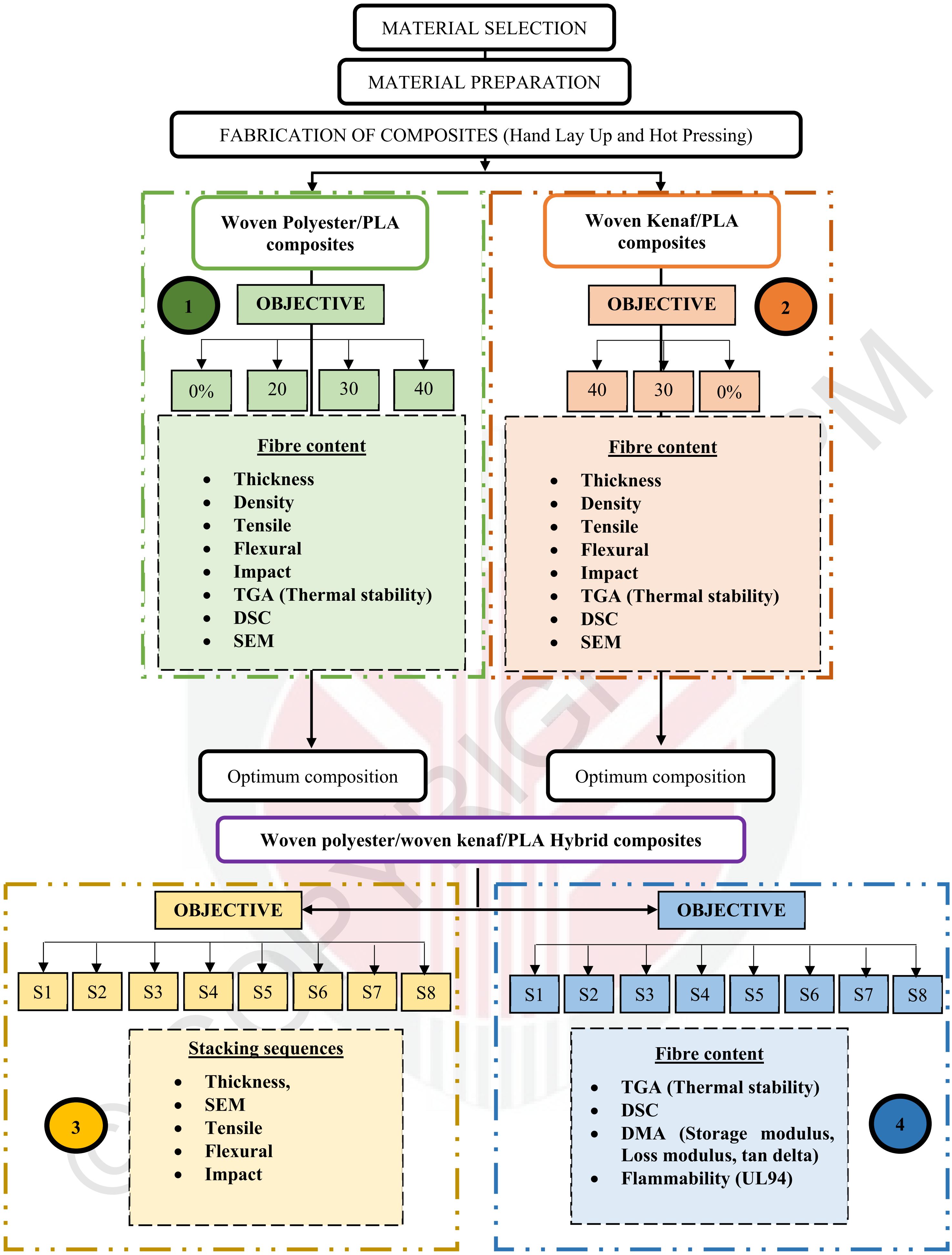

Figure 1.1: Flow process of the method

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