UNIVERSITI PUTRA MALAYSIA

PERFORMANCE OF PINEAPPLE LEAF/GLASS FIBRE-REINFORCED VINYL ESTER COMPOSITES UTILIZING AUTOMATED SPRAY UP TECHNIQUE

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By

MOHD HANAFEE BIN ZIN

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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April 2019

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This research is aimed at developing optimized process parameters to fabricate pineapple leaf fibre (PALF)-vinyl-ester-based hybrid biocomposites utilising the industrial robotic arm integrated with fibre spray up mechanism. The optimized process parameters are developed through a series of testing using different variable process parameters controlled by the integrated spray gun-robotic arm system.

In the preliminary study, material characterization of pineapple leaf fibre (PALF) of 273 tex, was conducted, which include physical, morphological, mechanical, and thermal testing and analysis.

PALF yarn fibre with different robot linear travel speed (0.15, 0.23, 0.30, 0.38, and 0.45) m/s reinforced vinyl ester composites are fabricated and characterized based on mechanical (tensile and flexural), thermal (TGA and DMA) and surface morphology. For each mechanical result, coefficient of variance (COV) is calculated to measure the variability of the mean distribution. The optimum robot linear travel speed obtained is 0.23 m/s, which produced composite with density of 1.12 g/cm^3 and fibre volume fraction of 27.4%. The COV for 0.23 m/s robot speed sample is 9.51%, with the highest tensile strength of 28.70 MPa.

The analysis of spray angle showed optimum spray angle of 70°, whereby spray distribution of the chopped fibre showed highest degree of uniformity (COV 5.84%), as well as high mechanical strength.

Linear spray pattern produced more uniform fibre and resin distribution as compared to circular and cross, whereby a lot of overlap fibre distribution occurred, which affect thickness consistency throughout the sample. Thermal analysis showed that linear pattern resulted in the highest Onset Oxidation Temperature (OOT) at 397.56°C, while circular pattern recorded the maximum char residue at 5.78%.

The length of pineapple yarn consists of 3 different combination, namely as L1 (130 - 150) mm, L2 (530 - 550) mm and L3 (130 – 550) mm. The mechanical analysis depicted that L1 resulted in the highest tensile strength (29.20 MPa), while L3 resulted in better mechanical properties consistency.

All optimized parameters are later used to fabricate the glass-PALF-VE hybrid biocomposites. The mechanical analysis showed that the hybrid biocomposites recorded 171.61% higher in tensile strength as compared to PALF-VE composite alone. The investigation of optimized process parameters will pave the way towards greater usage of PALF as input for robotic spray up process and potentially to be scaled up in industrial mass production.

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

PRESTASI KOMPOSIT GENTIAN DAUN NENAS / KACA DIPERKUATKAN OLEH VINIL ESTER YANG MENGGUNAKAN TEKNIK SEMBURAN AUTOMATIK

Oleh

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Kajian ini bertujuan untuk membangunkan parameter-parameter proses teroptimum untuk menghasilkan biokomposit hibrid berasaskan gentian daun nanas (PALF)-vinil ester yang menggunakan lengan robotik industri yang diintegrasikan dengan mekanisme semburan gentian. Parameter-parameter proses teroptimum dibangunkan menerusi ujian bersiri yang menggunakan parameter proses boleh ubah yang berbeza yang dikawal oleh sistem pistol semburan-lengan robotik berintegrasi. Dalam kajian awal, pencirian bahan gentian daun nanas (PALF) dengan 273 tex telah dijalankan, termasuklah ujian dan analisis fizikal, morfologi, mekanikal, dan terma.

Gentian bebenang PALF bertetulang komposit vinil ester dengan kelajuan perjalanan linear robot yang berbeza (0.15, 0.23, 0.30, 0.38 dan 0.45 m/s) telah difabrikasi dan dicirikan berdasarkan morfologi mekanikal (tegangan dan lenturan), terma (TGA dan DMA) dan permukaan. Untuk setiap keputusan mekanikal, pekali varians (COV) dihitung untuk menilai kebolehubahan taburan min. Kelajuan perjalanan linear robot yang diperoleh ialah 0.23 m/s, yang menghasilkan komposit yang mempunyai ketumpatan 1.12 g/cm3 dan pecahan isi padu gentian sebanyak 27.4%. COV untuk sampel kelajuan robot 0.23 m/s ialah 9.51% yang mempunyai kekuatan tegangan tertinggi sebanyak 28.70 Mpa. Analisis sud ut semburan menunjukkan sudut semburan yang optimum ialah 70° dan taburan semburan potongan gentian menunjukkan darjah keseragaman yang tertinggi (COV 5.84%) dan juga kekuatan mekanikal yang tinggi.

Corak semburan linear menghasilkan gentian dan taburan resin yang lebih seragam berbanding corak bulat dan silang, iaitu terdapat banyak pertindihan dalam taburan gentian yang berlaku, dan memberi kesan kepada konsistensi ketebalan sepanjang

sampel. Analisis terma menunjukkan corak linear menghasilkan suhu pengoksidaan permulaan yang tertinggi (OOT) iaitu 397.56°C, manakala corak bulat mencatatkan baki arang yang maksimum iaitu 5.78%. Panjang bebenang nanas terdiri daripada 3 jenis kombinasi, iaitu L1 (130 -150) mm, L2 (530 - 550) mm dan L3 (130 – 550) mm. Analisis mekanikal menunjukkan bahawa L1 mempunyai kekuatan tegangan yang tertinggi (29.2 MPa) manakala L3 mempunyai sifat-sifat mekanikal yang lebih konsisten.

Kesemua parameter teroptimum telah kemudiannya digunakan untuk memasang siap biokomposit hibrid gelas-PALF-VE. Analisis mekanikal menunjukkan bahawa biokomposit hibrid mencatatkan kekuatan tegangan yang lebih tinggi sebanyak 171.61% berbanding komposit PALF-VE. Penyelidikan tentang parameter-parameter proses teroptimum akan membuka jalan ke arah peningkatan dalam penggunaan PALF sebagai input untuk proses semburan robotik dan berpotensi untuk dikembangkan dalam pengeluaran besar-besaran secara industri.

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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 18:96)

This thesis is dedicated for my parents, Hj Zin Ali and Hjh Chek Mah Omar,
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LIST OF ABBREVIATIONS

ADF	Acid detergent fibre
ADL	Acid detergent lignin
AFP	Automated fibre placement
ANOVA	Analysis of variance
ASTM	American Society for Testing and Materials
ATL	Automated tape lay up
BADGE	Bisphenol A - diglycidyl ether type
CAD	Computer aided design
CaO	Calcium Oxide
CI	Confident interval
CO_2	Carbon dioxide
COPVs	Composite overwrapped pressure vessels
COV	Coefficient of variance
DFC	Directed fibre compound
DFP	Direct fibre preformed
DMA	Dynamic mechanical analysis
DSC	Differential scanning calorimetry
DTG	Derivative thermogravimetric
E*	Complex modulus
E'	Storage modulus
E"	Loss modulus
E-CR	Electrical / Chemical Resistance
EPN	Epoxy phenol novolac type

EU	European union
E_f	Fibre modulus
E_m	Matrix modulus
E_c	Composite modulus
FRP	Fibre reinforced polymer
FTIR	Fourier transform infrared
F_m	Mass flowrate
GRP	Glass-reinforced plastic
IFSS	Interfacial shear stress
ILSS	Interlaminar shear stress
LDPE	Low density polyethylene
LNG	Liquefied Nitrogen Gas
MEKP	Methyl ethyl ketone peroxide
MgO	Manganese Oxide
NaOH	Sodium hydroxide
NDF	Neutral detergent fibre
NF	Natural fibre
NFRC	Natural fibre reinforced composites
OH	Hydroxyl group
OOT	Onset Oxidation Temperature
PALF	Pineapple Leaf Fibre
PC	Polycarbonate
PE	Polyethylene
PLC	Programmable logic controller
PP	Polypropylene

PS	Polystyrene
PU	Polyurethane
PVC	Polyvinylchloride
RRIM	Reinforced Reaction Injection Molding
RTM	Resin transfer moulding
SEM	Scanning electron microscopy
SPF	Sugar palm fibre
Tan δ	Tan delta
T_g	Glass transition temperature
TGA	Thermogravimetric analysis
UD	Unidirectional
UV	Ultra violet
VE	Vinyl Ester
V_f	Fibre volume fraction
V_m	Matrix volume fractions
VOC	Volatile organic compound
wt.%	Weight percent
η_d	Fibre diameter distribution factor (FDDF)
η_l	Fibre length distribution factor (FLDF)
η_o	Fibre orientation distribution factor (FODF).
κ	Fibre area correction factor (FACF)
σ_c	Unidirectional composite tensile strength

LIST OF UNITS

cPs	Centipoise
°C	Degree celcius
°C/min	Degree celcius per minute
°	Degree
GPa	Gigapascal
gsm	gram square meter
tex	gram per 1000 meters
g/cc	gram per cubic centimeter
g/cm^3	gram per cubic centimeter
g/s	gram per second
Hz	Hertz
kN	kilo Newtons
kg/m^3	Kilogram per cubic meter
kV	kilovolts
kPa	kilopascal
MPa	Megapascal
m/s	meter per second
mm	millimeter
mmHg	milimeter of mercury
mm/min	milimeter per minute
min	minute
MT	Metric tonne
%	Percentage
rpm	rotation per minute

CHAPTER 1

INTRODUCTION

1.1 Background

The rising on environmental awareness has led to fabrication of aircraft parts from sustainable renewable resources. Natural fibre reinforced composites (NFRC) have been used in construction and automotive application for quite some time, and now becoming more substantial for secondary structures in the next generation of aircrafts (Mohd Nurazzi et al., 2017).

In the recent years, NFRC have been in the limelight of research to be applied in industry such as automation and aerospace. The first application of natural composite in aircraft manufacturing was from the first flight of Wright Brothers' Flyer 1, in North Carolina on December 17, 1903 (Soutis, 2005). In the aerospace engineering industry, one of the main concerns is regarding reliability of using new materials for structural components. Challenging environment such as low temperature, low pressure, high UV exposure intensity and high stress, contribute to faster degradation of NFRP composites. Since 'defect-free' is the aim of aerospace structural parts design, substitution of synthetic fibre by natural fibre seems to be difficult process. The presence of moisture in laminar level and interfacial bonding of NFRP, would greatly vary the fibre-matrix adhesion, hence affecting the mechanical properties (Susheel et al., 2009). However, there is still opportunity for NFRP to enter aerospace application that is on the internal cabin part, which requires lower threshold for mechanical strength as compared to structural components. Of the potential substitution seen for interior cabin part include food tray, seat frame parts, window frame, galley and lavatories. This initiative has been put in place by Boeing through the application of flax non-woven mat in the window frame component since 2014 (Boeing, 2014)

There are various types of NFRP fabrication methods available, which can be divided into two main categories, namely as closed and open mould method. For closed mould, both top and bottom surfaces of composite part are confined to enable the part to have controlled finishing for both surfaces, while for open mould, the top surface is exposed which enables only one side to have controlled surface (Gurunathan et al., 2015). Some of the closed mould method includes hot compression, resin transfer molding, and injection molding, while the open mould fabrication technique consists of manual hand lay up, vacuum assisted resin infusion, spray up, filament winding and vacuum bagging.

The advantages of using natural fibre composites are environmental gains, reduced energy consumption, light weight, insulation and sound absorption properties and reduce dependency on petroleum-based materials. However, there are some limitation that restrains the application of natural fibre in mass production, such as inconsistent properties resulting in quality variation (Fiore et al., 2012). High dependency on human skill is another factor contributing to low usage of natural fibre in industry. In general, natural fibre possessed lower mechanical properties compared to their counterpart of petroleum-based source such as carbon fibre, making them limited to light loading non-structural parts. Component fabrication using natural fibre is subject to several type of deficiencies like porosity, void, layer disbonding, and weak fibre-matrix interfacial shear stress.

1.2 Problem Statement

Spray up or also known as direct fibre preformed (DFP) is a well-known process whereby chopped fibre and low-viscosity resin are manually sprayed onto a mould surface. The spray up process has been long used in the industry to fabricate light loaded structural component such as bathtub, boat and luxury yacht (Ecobiz, 2019).

In general, spray up process uses synthetic fibre as the main input. Natural fibres has been in the limelight of research for the past few decades and has great potential to substitute synthetic fibres in the spray up process fabrication (Kikuchi et al., 2014). However, there were very few attempts to use natural fibre, due several factors such as difficulty in getting the suitable input form (roving with consistent diameter or width), variation in physical properties, and difficulty in getting consistent output from this process. Spray up required material with consistent width or diameter to ensure smooth cutting and material spray out and this has been a big challenge for natural fibre in general.

There were a few studies trying to use natural fibre as the input for spray up fabrication. However, the study showed that the spray output varies significantly with human skill and expertise (Kikuchi et al., 2014). Another issue related to the usage of natural fibres in the manual spray up process is poor part consistency due to uneven distribution of fibres on the mould surface during the spray up process (Harper et al., 2007). There were a few attempts in research that have been conducted to investigate the parameters and behavior of the spray up composite such as randomization effect (Harper et al., 2007), discontinuous fibre composite strength (Qian et al., 2015), critical fibre length (Qian et al., 2012), spray pattern (Harper et al., 2007) and fibre volume fraction (Evans et al., 2016). However, these studies were conducted manually using synthetic fibres such as carbon or glass fibres.

One of the main reasons that contribute to the variation and inconsistency of the spray up output is the inability to control spray up process parameter manually. The spray up process parameters i.e. spray pattern, robot linear travel speed, spray angle, and fibre length are critical in controlling the variety of the product quality for spray up process. To date, very few systematic studies being conducted in automating the spray up process parameters for natural fibres and investigating how these parameters affect the properties of the spray up biocomposites. Issue such as low fibre volume fraction was also not being addressed effectively (Harper et al., 2007). Without proper study in these domains, it is difficult to use natural fibre as an input for spray up process, and to obtain consistent output, which eventually applied in mass rapid production. There is also lack of investigation in the performance of hybrid biocomposites combining natural fibres and synthetic fibres fabricated using automated spray up process. This is where automation is needed to better control the process parameter and enhance the consistency and repeatability of the output from spray up process using natural fibres. Automation can solve a few major issues such as material planning, cycle time, product quality and process efficiency. There are a few companies around the world that already automated the spray up process for industrial application, such as Matrasur Composites (Didier Barbini, 2018). However, the automation is only limited and tailored for the use of synthetic fibre as the input material, and generally very costly. This research works aims to explore automation of the spray up process, and in the same time to understand the effect of different spray up process parameter on the behavior of natural fibre composite properties. By automating the spray up method (integration with industrial 6-axis robotic arm), the spray output can be controlled automatically, simulation can be run to ensure correct spray parameters are achieved, process error and output defect can be minimized, consistency of output can be enhanced, and waste material can be reduced. This exploration work is vital in determining the suitability of natural fibres being used as the replacement for current synthetic fibre-based composite for various industrial application.

1.3 Significance of Study

1. A novel study on the viability of natural fibre (PALF) to be used as the input for automated spray up process.
2. A novel study of new fabrication process of automated spray up process for PALF-vinyl ester biocomposite.
3. A novel study in developing feasible production process parameters resulting in consistent properties for hybrid biocomposite mass production.

1.4 Objectives of the Research

1. To determine physical, morphological, mechanical and thermal properties of PALF yarn.
2. To investigate the effect of various robot linear travel speed on mass flowrate, volume fraction, mechanical, thermal and morphological properties of pineapple leaf fibre (PALF)-vinyl ester automated spray up biocomposite.
3. To investigate the effect of various spray angles, spray patterns and PALF fibre lengths, on mechanical, thermal and morphological properties of PALF-vinyl ester automated spray up biocomposite and determine optimized process parameter.
4. To investigate the effects of spray up process parameters on physical, mechanical, thermal, and morphological properties of PALF/glass hybrid biocomposites.

1.5 Scope and Limitation

The research scope is focusing on the fabrication process parameters of PALF-vinyl ester bio-composite using spray gun integrated with robotic arm. The PALF fibre used in this study is purchased from India, purchased from Mecha Solve Engineering, that comes with single ply, in roving form with 273 tex of twisting. This material is used as it is suitable for chop spray process, resulting in consistent fibre chop length. The thermosetting polymer used is Polymal Vinylester MFE 711P with MEKP Mepoxe hardener (100:3 ratio), purchased from Luxchem Trading Sdn Bhd. The resin is chosen, as it has low viscosity (250 – 450 cPs) at 25°C, as required by the spray system, and has higher strength compared to polyester resin. The vinyl ester is reinforced with chopped PALF fibre and the parameter investigated are the robot linear travel speed, spray angle, fibre length and spray pattern.

The robot linear travel speed studied are 0.5, 0.75, 1.0, 1.25 and 1.50 m/s. The speeds are chosen based on the capability of the robotic arm, whereby the maximum speed is 2.0 m/s. However due to safety issue, the maximum limit of robot speed set for this study is capped at 1.50 m/s. All the robot speed is limited to 30% actual robot speed, which gives the actual value of 0.15, 0.23, 0.30, 0.38 and 0.45 m/s. For speed below 0.15 m/s, the spray resulted in excess fibre pile up on the mould surface, hence affecting the wettability and composite strength. The spray angle set for this research are 60, 65, 70, 75 and 80°. The spray pattern consists of linear, circular and cross spray.

The fibre length investigated are L1 (130-150) mm, L2 (530-550) mm and L3 (130-550) mm. The restriction of fibre length is due to the rotary cutter of spray gun that has the perimeter of 10.4 mm, 8 equal distance cutting blade, and distance between each blade is 13 mm. For each process parameter, analysis is mainly made on their mechanical (tensile & flexural), supported by thermal (TGA & DMA) and morphological (SEM) properties. The optimized parameters are then used to fabricate hybrid glass-PALF-vinyl ester composite, and the mechanical, thermal and morphological properties are investigated.

1.6 Thesis Outline

The thesis consists of 5 chapters. The first chapter is an overview of the NFRP, the application in various industry, significance of study, problem statements, and objectives, scope and limitations of the research. Chapter two touched on the literature review of natural fibres and their properties, synthetic fibres and hybrid bio-composites, thermosetting matrix and its application, fabrication methods for bio-composites, and manufacturing of directed fibre compound (DFC). In chapter three, the research methodology was outlined, starting from preliminary study, material characterization, research design for different process parameters including robot linear travel speed, spray angle, spray path and fibre length. At the end of chapter three, the glass-PALF hybrid biocomposite was developed based on the optimized parameters obtained from earlier analysis. Chapter four discussed about the results obtained and divided into six sections. The sections cover the material selection, preliminary analysis on PALF fibre, the effect of robot linear travel speed, spray angle, spray pattern and fibre length on morphological, mechanical and thermal properties of biocomposite. The last section in chapter four explains about the characteristics of glass-PALF hybrid bio-composite fabricated utilizing optimized parameters. The final chapter concludes the research findings and recommendations for future works.

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LIST OF PUBLICATIONS

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