

Faculty of Manufacturing Engineering

THERMAL STABILITY EFFECT ON PHYSICAL AND FLEXURAL PROPERTIES OF HYBRID GLASS/JUTE FIBER REINFORCED EPOXY COMPOSITES

Mohd Fadli bin Hassan

Master of Science in Manufacturing Engineering

2017

THERMAL STABILITY EFFECT ON PHYSICAL AND FLEXURAL PROPERTIES OF HYBRID GLASS/JUTE FIBER REINFORCED EPOXY COMPOSITES

MOHD FADLI BIN HASSAN

A thesis submitted in fulfillment of the requirements for the degree of Master of Science in Manufacturing Engineering

Faculty of Manufacturing Engineering

UNIVERSITI TEKNIKAL MALAYSIA MELAKA

2017

DECLARATION

I declare that this thesis entitled "Thermal stability effect on physical and flexural properties of hybrid glass/jute fiber reinforced epoxy composites" is the result of my own research except as cited in the references. The thesis has not been accepted for any degree and is not concurrently submitted in candidature of any other degree.

Signature

Name

Mohd Fadli bin Hassan

Date

25/9/2077

APPROVAL

I hereby declare that I have read this thesis and in my opinion this thesis is sufficient in terms of scope and quality for the award of Master of Science in Manufacturing Engineering.

Signature

Name

Prof Madya Dr. Noraiham binti Mohamad

25/9/2017 Date

DEDICATION

I dedicate this thesis to my family for nursing me with affections and love and their dedicated for success in my life. Thank you. My love for you all can never be quantified.

ABSTRACT

Introduction of natural fibers with partial replacement of synthetic fibers in tooling materials could lead to new venture in composite tooling industries. This is due to the environmental concern on the recyclability limitation of synthetic fiber made composites as well as their potential in weight and cost reduction. The primary goal of this research is to fabricate a green hybrid glass/jute fibre reinforced epoxy composite for potential tooling material via vacuum infusion technique and evaluate the thermal cycle effect on its flexural properties. In the first stage, the processing parameters of the vacuum infusion technique; i) supply pressure, ii) soaking time and iii) the utilization of flow media (with or without) were optimized by response surface methodology for maximum flexural properties of the hybrid glass/jute fibre reinforced epoxy composites. The flexural strength of ~195 MPa and flexural modulus of ~13412 MPa were achieved at optimum parameters of 100 kPa pressure and 120 minutes soaking time with the utilization of flow media during the vacuum infusion process. In the second stage, the hybrid glass/jute fiber reinforced epoxy laminated composite was prepared with the optimum process paramaters and exposed to 50 times thermal cycles at three different temperature; i) room temperature, ii) 120 °C and iii) 200 °C and tested for flexural properties. The reduction in flexural properties of around 40% was recorded composites exposed to 200 °C thermal cycles when compared with those at room temperature and at 120 °C. It was resulted from partial degradation of natural fibers at 200°C. The composites which exposed to thermal cycle at 120 °C showed stable flexural properties as well as physical properties even at 50 thermal cycles. The findings indicate that the hybridization between glass and jute fiber exhibit promising potential to be used as tooling materials with weight reduction of almost 25%.

ABSTRAK

Pengenalan gentian asli melalui penggantian sebahagian daripada gentian sintetik ke dalam bahan acuan membawa kepada suatu cubaan baru dalam industri acuan komposit. Hal ini disebabkan kebimbangan terhadap isu alam sekitar bagi had kitar semula gentian sintetik dalam penghasilan komposit di samping potensinya dalam penjimatan berat dan kos. Matlamat utama kajian ini adalah untuk menghasilkan komposit epoksi diperkuat gentian hibrid kaca / jut mesra alam untuk potensinya sebagai bahan acuan melalui teknik infusi vakum dan mengkaji kesan kitaran haba ke atas sifat lenturannya. Pada peringkat pertama, parameter pemprosesan bagi teknik infusi vakum; i) tekanan bekalan, ii) masa rendaman dan iii) penggunaan media aliran (dengan atau tanpa) telah dioptimumkan dengan kaedah permukaan sambutan (RSM) untuk sifat lenturan maksimum komposit epoksi diperkukuh gentian hibrid kaca / jut. Kekuatan lenturan ~ 195 MPa dan modulus lenturan ~ 13412 MPa telah dicapai pada parameter optimum iaitu tekanan setinggi 100 kPa dan masa rendaman selama 120 minit dengan penggunaan media aliran semasa proses infusi vakum. Pada peringkat kedua komposit epoksi diperkukuh gentian hibrid kaca / jut disediakan mengikut paramater proses optimum yang dijana dalam peringkat pertama dan didedahkan kepada 50 kali kitaran haba pada tiga suhu yang berbeza; i) suhu bilik, ii) 120°C dan iii) 200°C dan diuji untuk sifat lenturan. Pengurangan dalam sifat lenturan sekitar 40% dicatatkan bagi komposit yang terdedah kepada kitaran haba pada 200°C berbanding dengan yang berada pada suhu bilik dan pada 120°C. Ia adalah disebabkan degradasi sebahagian daripada gentian asli pada 200°C. Komposit yang terdedah kepada kitaran haba pada 120°C menunjukkan sifat lenturan dan sifat-sifat fizikal yang stabil walaupun pada 50 kitaran haba. Dapatan kajian menunjukkan bahawa penghibridan antara gentian kaca dan jut menpamerkan potensi yang meyakinkan untuk digunakan sebagai bahan acuan dengan pengurangan berat badan hampir 25%.

ACKNOWLEDGEMENTS

First of all, my gratitude to Allah S.W.T for giving me the strength to undertake this Master of Science by Research. I would like to express my heartiest appreciation and deep gratitude to my supervisor, Prof Madya Dr. Noraiham binti Mohamad for her supervision, advice, guidance, assistance, encouragements, and constant dedication during my period of study. I believe that her diligence to motivate is of paramount importance to the foundation of this research work. I also value highly her time and effort to evaluate my research work. Her willingness to revise the thesis prior to submission will always be appreciated.

I would also like to express my sincere gratitude to Universiti Teknikal Malaysia Melaka (UTeM) especially to Faculty of Manufacturing Engineering (FKP) and Centre for Graduate Studies (PPS) as well as Kolej Kemahiran Tinggi MARA Masjid Tanah for giving me this opportunity to further my study. Thank you so much to my co-supervisor Prof. Dr. Qumrul Ahsan for his constant help and precious advice on his expert view on pursuing this research. Furthermore, my deepest appreciation to Miss Chang Siang Yee and regards to all of my family, my friends (Dr. Mohd Asyraf Kasim, Mohd Aziz Rashid, Mustafa Ali Azhar, Azam Adnan, Anisah Abd Latif, Mazlin Aida Mahamood and Dr. Jeefferie Abd Razak), staffs of Universiti Teknikal Malaysia Melaka (UTeM) as well as staffs of Kolej Kemahiran Tinggi MARA Masjid Tanah for their support.

Finally, I would like to thank UTeM for sponsoring my research work under the short term grant PJP/2012/FKP(35A)/S01039 which made my research possible and fulfilled my desire to pursue this Master of Science.

TABLE OF CONTENTS

			PAGE
DEC	CLARA'	ΓΙΟΝ	
	ROVA		
DEI	DICATI	ON	
ABS	TRACT	r	i
ABS	TRAK		ii
ACI	KNOWI	LEDGEMENTS	iii
TAE	BLE OF	CONTENTS	iv
LIS	T OF TA	ABLES	vi
LIS	T OF FI	GURES	vii
LIS	LIST OF APPENDICES		
LIS	LIST OF ABBREVIATIONS		xii
LIS	T OF PU	UBLICATIONS	xiv
CHA	APTER		
1.		RODUCTION	1
	1.1	Background of study	1
	1.2	Problem statement	3
	1.3	Objective of study	5
	1.4	Scope of study	5
2.	LITERATURE REVIEW		7
	2.1.	Tooling	7
	2.2.	Polymer Matrix Composites	9
		2.2.1. Matrix	9
		2.2.2. Reinforcement	13
	2.3.	Hybrid Composites	17
		2.3.1. Epoxy Based Hybrid Composites	19
	2.4.	Processing of Thermoset Composites	19
		2.4.1. Hand Lay Up and Spray Up	19
		2.4.2. Vacuum Bagging	23
		2.4.3. Vacuum Infusion	25
	2.5.	Flexural Properties of Hybrid Composites	28
		2.5.1. Synthetic/Natural Hybrid Composites	30
	2.6.	Thermal Stability of Thermoset Composites	32
3.	MET	THODOLOGY	39
	3.1	Introduction	39
	3.2	Materials	41
		3.2.1 Epoxy	41
		3.2.2 Glass Fiber	42
		3.2.3 Jute Fiber	43
	3.3	Fabrication of Hybrid Laminated Composites	44
		3.3.1 Apparatus for Vacuum Infusion Technique	44
		3.3.2 General Process Flow for Fabrication Hybrid Laminated	50
		Composites via Infusion Technique	

	3.4	Testing and Analysis of Hybrid Laminated Composites	51
		3.4.1 Physical Properties	51
		3.4.2 Mechanical Testing	54
		3.4.3 Thermal Analysis	57
		3.4.4 Morphological study	58
	3.5	The Conducted Studies	59
		3.5.1 Stage 1	59
		3.5.2 Stage 2	61
4.	RES	ULT AND DISCUSSION	62
	4.1.	Introduction	62
	4.2.	Characterization of raw materials	62
		4.2.1. Physical properties	62
	4.3.	Stage 1: Optimization of Process Parameters	65
		for Hybrid Laminated Composites	
		4.3.1. Flexural Properties	65
		4.3.2. Determination of the Optimum Process	72
		Parameter for Hybrid Laminated Composites using	
		the Response Surface Methodology (RSM)	
	4.4.	Stage 2: Effect of Thermal Cycling on Flexural Properties	74
		of Hybrid Laminated Composites	
		4.4.1. Flexural Properties of Hybrid Laminated Composites	74
		4.4.2. Physical Properties of Hybrid Laminated Composites	80
		4.4.3. Thermal Analysis of Hybrid Laminated Composites	86
		4.4.4. Morphology Analysis of Hybrid Laminated Composites	92
5.	CON	ICLUSION AND RECOMMENDATIONS	101
	5.1	Conclusion	101
	5.2	Recommendations for Future Work	104
REF	EREN	CES	105
APP	APPENDICES		117

LIST OF TABLES

TABLE	TITLE	PAGE
2.1	Raw materials used in hand lay-up	21
3.1	Specification of Epoxy Resin DM15 (F3)	42
3.2	Design matrix of two level factorial designs for three factors.	60
3.3	Thermal Cycling for Hybrid Laminated Composite	61
4.1	Density Value for Raw Materials	63
4.2	Regression model	68
4.3	Selection of optimum parameters	73
4.4	Density of Laminated Composite	81
4.5	Summary for thermal degradation profile weight loss and	90
	residue analysis on the temperature stabilization from	
	TGA thermogram	

LIST OF FIGURES

FIGURE	TITLE	PAGE
2.1	Schematic representation of a molecule of diglycidyl ether of	12
	bisphenol A (DGEBA) epoxy resin	
2.2	Three-dimensional network structure of solid epoxy	13
2.3	Schematic representation of hand lay-up process	22
2.4	Schematic representation of spray lay-up technique	23
2.5	A basic diagram of the vacuum infusion process	28
2.6	Hardness for different composites after exposure to thermal	36
	cycling	
2.7	Degradation of Charpy impact energy of composites after	37
	exposure to the thermal cycling	
3.1	Flow chart of research activities	40
3.2	E- glass fiber	43
3.3	Jute Fiber Mat	43
3.4	Infusion Technique Process	44
3.5	Schematic diagram for Infusion Technique	44
3.6	Equipment used in Infusion Technique	45
3.7	The Resin Catch Pot	46
3.8	The Vacuum Hose	46

3.9	The Infusion Spiral (Plastic Spiral Tube)	47
3.10	The Infusion Mesh or Flow Media	48
3.11	The Vacuum Bagging Films	48
3.12	a) The Peel Ply Stitch type, b) The Teflon Peel Ply	49
3.13	The Sealing Tape	49
3.14	The stacking of plies into an interplay hybrid laminated composite	51
3.15	Electronic Densimeter	52
3.16	Water Bath Machine	53
3.17	a) Table Saw Machine b) Disc cutter (power air)	54
3.18	Dimension of flexural test specimen	55
3.19	GOTECH Humidity Chamber Machine	56
3.20	INSTRON 50 kN Universal Testing Machine (UTM)	57
3.21	(a) Stereo Microscope Stemi 2000-C, (b) Digital Camera	58
4.1	Interaction graph for flexural strength of (a) hybrid laminated	67
	Composite under constant pressure (b) hybrid laminated	
	composite nder constant soaking time (c) hybrid laminated	
	composite without using flow media and (d) hybrid laminated	
	with using flow media	
4.2	Effect of the soaking time and pressure on the elastic modulus	70
	of hybrid laminated composites with/without presence of	
	flow media	
4.3	Image of resin starvation defect which obvious in	71
	(a) Experiment 4 then (b) Experiment 7 of hybrid	
	laminated composites	

4.4	Sample under flexural load	75
4.5	Flexural strength of hybrid laminated composites under	76
	the thermal cycling	
4.6	Flexural elastic modulus of hybrid laminated composites under	77
	the thermal cycling	
4.7	Morphologies of fracture surfaces of composites exposed to	79
	120°C for 50 cycles (a) at 500 x magnifications and	
	(b) 50 x magnifications. Morphologies of fracture surfaces	
	of composites exposed to 200°C for 50 cycles	
	(c) at 50 x magnifications and (d) 500 x magnifications	
4.8	Density of hybrid laminated composite	82
4.9	Water absorption of hybrid laminated composites	84
4.10	Water absorption of hybrid laminated composites at room	85
	temperature for five days	
4.11	(a) TGA thermogram for hybrid laminated composite	88
	$(120^{\circ}\text{C}\ /\ 0\ \text{Cycle}),$ (b) TGA thermogram for hybrid laminated	
	composite (200°C / 0 Cycle)	
4.12	Two steps slide of thermal degradation profile weight	89
	loss with increasing temperature	
4.13	TGA thermograms of weight loss as a function of temperature	91
	for hybrid laminated composites after thermal cycling	
4.14	Scanning electron micrographs of flexural fracture surfaces	93
	for hybrid laminated composite exposed to thermal cycles	
	at 120oC at magnification of 50X	

4.15	Scanning electron micrographs of flexural fracture surfaces	94
	for hybrid laminated composite exposed to thermal cycles	
	at 200°C at magnification of 50X	
4.16	Scanning electron micrographs of flexural fracture surfaces	95
	for hybrid laminated composite exposed to 120 °C and	
	200 °C thermal cycles at magnification of 250X	
4.17	Scanning electron micrographs of flexural fracture surfaces	97
	for hybrid laminated composite exposed to thermal cycles	
	at 200°C at magnification 250X	
4.18	Image of flexural induced fracture for hybrid laminated	98
	composite exposed to thermal cycles at 120°C	
4.19	Image of flexural induced fracture for hybrid laminated	99
	composites exposed to thermal cycles at 200°C	

LIST OF APPENDICES

APPENDIX	TITLE	PAGE
A	Paper Publication	117
В	Raw Data: The Flexural Testing for Samples in Stage 1	118
C	Raw Data: The Flexural Test for Sample at 0 cycle	123
D	Raw Data: The Flexural Test for Sample at 120°C and	124
	12 cycles	
E	Raw Data: The Flexural Test for Sample at 200°C and	125
	12 cycles	
F	Image of flexural induced fracture for hybrid laminated	126
	composite exposed to thermal cycles at 120oC	
G	Image of flexural induced fracture for hybrid laminated	127
	composites exposed to thermal cycles at 200oC	

LIST OF ABBREVIATIONS

ASTM - American Society for Testing and Materials

BFP - Woven Basalt prepreg

BCFP - Basalt and carbon fiber prepreg

CFP - Woven Carbon prepreg

CFRP - Carbon Fiber Reinforced Polymer

CTE - Coefficient of Thermal Expension

DGEBA - Diglycidyl Ether of Bisphenol A

DOE - Design of Experiment

DSC - Differential Scanning Calorimetry

FRC - Fiber Reinforced Composites

FRP - Fiber Reinforced Polymer

GFRP - Glass Fiber Reinforced Polymer

JRP - Jute Reinforced Polymer

MEKP - Methyl Ethyl Ketone Peroxide

MPa - Mega Pascal

oC - degree celsius

PLA - Polylactic Acid

PMC - Polymer Matrix Composite

RC - Reinforced Concrete

RSM - Response Surface Methodology

RTM - Resin Transfer Molding

SEM - Scaning Electron Microscopy

SFRC - Sisal Fabric Reinforced Polymer

TGA - Thermagravimetric Analysis

UTM - Universal Testing Machine

VIP - Vacuum Infusion Process

WA - Water Absorption

LIST OF PUBLICATIONS

Journals

 N. Mohamad, M. F. Hassan, S. Y. Chang, Q. Ahsan, Y. Yaakob, H. E. Ab Maulod, "Correlation between Process Parameters with Flexural Properties of Hybrid Glass/Jute Fibre Reinforced Epoxy Composite Fabricated via Vacuum Infusion Technique", Applied Mechanics and Materials, Vol. 761, pp. 531-535, 2015

Proceedings

 Noraiham Mohamad, Mohd Fadli Hassan, Chang Siang Yee, Qumrul Ahsan, Yuhazri Yaakob, Hairul Effendy Ab Maulod, "Correlation between Process Parameters with Flexural Properties of Hybrid Glass/Jute Fibre Reinforced Epoxy Composite Fabricated via Vacuum Infusion Technique", 3rd International Conference on Design and Concurrent Engineering 2014 (iDECON 2014), 22-23
September 2014, Avillion Legacy Melaka Hotel, Malaysia, #144.

CHAPTER 1

INTRODUCTION

1.1 Background of study

Fiber-reinforced polymer (FRP) made from glass, aramid and carbon fiber is widely utilized as construction materials in composite tooling technology. It possesses high strength and stiffness at a relatively low weight. Unfortunately the use of synthetic fiber-reinforced polymer leads to a serious disposal problem due to structure stability of thermoset matrix and synthetic fibers once the structure made is ready to be decommissioned. It is worse when simple landfill disposal of this material is becoming increasingly impossible due to problems related to environmental sensitivity. An interesting option might be where construction materials made of renewable resources that consist of natural fibers are embedded in so-called biopolymers or natural polymer, as these also involve economically and ecologically acceptable manufacturing technologies.

The idea of the combination of synthetic fibers and natural fibers is an interesting idea to counter the disadvantage of synthetic fibers. This is due to concerns about environmental awareness about the limitations of recycled synthetic fiber composites, but it is also due to the potential of such materials in cost reduction.

Hybridization of two or more different continuous fibres in a matrix is a versatile approach to improve strength or stiffness of structural composites (Matthews and Rawlings, 1999). Performance of a hybrid composite is a combination of weighed amounts of the individual components, of which balances between the strength and weakness of the

individual components (Al-Mosawi et al., 2012b). The selection of suitable components is determined by the desired properties required for the final products. Despite the excellent performance of synthetic fibres such as high strength and stiffness (Yuhazri et al., 2014) as well as good chemical resistance (Jennise et al., 2014), issues on production cost and disposal of after-use products of non-biodegradable materials are crucial. Therefore, the utilization of either solely natural fibres or in-hybridization with synthetic fibres has attracted the attention of scientists and technologists. Natural fibres such as banana, cotton, coir, sisal and jute have found their applications in consumer products, low-cost housing and other civil structures (Gowda et al., 1999). It has been observed that natural fibre composites have compatible electrical resistance, good thermal and acoustic insulation properties as well as good resistance to fracture (Gowda et al., 1999). Furthermore, the use of green composites offers several advantages such as light weight, low production cost and low thermal mass (Cicala et al., 2009, Arrakhiz et al., 2013, Yuhazri and Sihombing, 2010).

In addition, the most widely used fiber for fiber reinforced composite is called E-glass or electrical glass. E-glass is low cost, high density, low modulus fiber that has good corrosion resistance. However, glass fiber with superior mechanical properties known as S-glass or structural glass is used for higher-strength fiber for filament wound pressure vessels and solid rocket motor castings (Campbell, 2004).

As other part of reinforcement for hybrid laminated composites, jute fibers possess huge potential as fibrous reinforcement in polymeric composites. The fibers are extracted from jute plant Corchorus, from the Malvaceae family. Jute fiber extracted from bast or skin of plant, is off-white to brown in colour and measured 1-4m long. The fiber thickness varies between 40 and 80 µm which leads to a variation in the tensile strength between

1000 and 480MPa. In addition, jute fibers can withstand up to 100°C (Chand and Fahim, 2008).

1.2 Problem statement

In aerospace industry, production tooling made of synthetic fiber (carbon fiber or glass fiber) reinforced thermoset composite is facing difficulties to be disposed or recycled. Most of the production tooling in the market produces by wet hand layup technique or high quality prepreg. Tooling components by wet hand layup technique are heavy and bulky where prepreg are not cost effective. Therefore a vacuum infusion technique was utilized in this study. This technique is proven by several studies to be an efficient technique in producing high performance composites at high production rate and low cost. In addition, the uses of synthetic fibers as tooling materials are very expensive, thus increasing the overall cost of composite products. The hybridization of natural fiber and synthetic fiber in these tooling materials could lead to new venture in tooling industries. These new generation hybrid green composites offer several advantages such as light weight, low production cost and low thermal mass (Cicala et al., 2009, Arrakhiz et al., 2013, Yuhazri and Sihombing, 2010) as well as having high potential to be biodegradeble.

For tooling manufacturer, thermoset resins such as epoxy is widely used as matrix phase in the fabrication of mould tool for composite parts. Rao et al., (2011) also stated the epoxy is most commonly used polymer matrix with reinforcing fibers for advanced composites applications. Epoxy resin is more viscous than polyester with curing temperature up to 180°C. The shrinkage of epoxy is smaller than that of polyester, which is by 1-5%. Generally epoxy is stiffer and stronger and posseses to maintain its properties to higher temperature compared to polyester (Matthews and Rawlings, 1999).

This study initially fabricates the hybrid composite (glass / jute fibre) via vacuum infusion technique. This technique is common for the production of synthetic fiber based composites however; the application of this technique to prepare natural fiber based composites is limited. This technique offers lower resin to fiber ratio and produces stronger and lighter composites (Yuhazri and Sihombing, 2010). However, the process parameters need to be optimised to ensure appreciable amount of impregnated resin to fibers for better wetting.

In the industries, the tooling material subjected to thermal cycling in service. Thermal cycling is defined as alternate heating and cooling of materials. Low thermal cycle means the time taken for completion of the cycle is large enough to cool the component. On the other hand, high thermal cycle means time involved is in milliseconds and the heating and cooling is influenced by the thermal inertia of the system under consideration (Agbadua et al., 2011). During heating and cooling, the material experiences expansion and contraction. Therefore, the dimension of the material changes when subjected to thermal cycling.

Tooling material is subjected to repeated thermal cycling during in service and is likely to induce distortion on the tooling due to the difference in thermal mass and coefficient of thermal expansion. Despite the potential of these green composites, studies on thermal effect on its properties are crucial. Therefore, this study addresses the potential of hybrid glass/jute fibers reinforced composite to be fabricated via a vacuum infusion technique and evaluates its physical and flexural properties under thermal effect.

1.3 Objective of study

In this research, the main objectives are:

- To optimize process parameter of vacuum infusion technique for fabrication of hybrid glass/jute fiber reinforced epoxy composites
- To determine the physical and flexural properties of hybrid glass/jute fiber reinforced epoxy composites
- iii. To evaluate thermal cycle effect (temperature and number of cycle) on the physical and flexural properties of hybrid glass/jute fiber reinforced epoxy composites

1.4 Scope of study

In this research, a hybrid laminated composite of woven glass fibers / jute fibers reinforced epoxy was fabricated via a vacuum infusion technique. In the first stage, the process parameters of the vacuum infusion technique (pressure, soaking time and flow media) were optimized through a response surface methodology (RSM). Two level factorial design for three independent variables (pressure (X₁), soaking time (X₂) and flow media (X₃) were used to design the experiment using response surface methodology (RSM). The optimum parameters were determined from flexural properties as well as physical properties and support by several analyses. The testing was conducted on 7 plies hybrid laminated composites at constant stacking sequence and orientation. In the second stage, the hybrid laminated composites were produced using the selected optimum parameters and tested for their performance under thermal cycle effect (temperature and number of cycles). The observations were supported with several analyses (SEM, TGA etc). Tested samples of the hybrid laminated composite were investigated for fracture