



UNIVERSITI TEKNIKAL MALAYSIA MELAKA (UTeM)

**EFFECT OF CHEMICAL MODIFICATION ON TENSILE
PROPERTIES OF THERMOPLASTIC REINFORCED
COMMINUTION FOR OUT OF CONDITION AEROSPACE
GRADE CARBON FIBER PREPREG WASTE**

This report is submitted with requirement of the Universiti Teknikal Malaysia Melaka (UTeM) for the Bachelor Degree of Manufacturing Engineering (Engineering Materials) (Hons.)

by

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DECLARATION

I hereby, declared this report entitled 'Effect of Chemical Modification on Tensile Properties of Thermoplastic Reinforced Comminution for Out of Condition Aerospace Grade Carbon Fiber Prepreg Waste' is the results of my own research except as cited in the references.

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APPROVAL

This report is submitted to the Faculty of Manufacturing Engineering of UTeM as a partial fulfillment to the requirements for the degree of Bachelor of Manufacturing Engineering (Engineering Materials) (Hons.). The member of the supervisory is as follow:

.....

(Dr. Noraiham Binti Mohamad)

ABSTRACT

Nowadays, there are abundant of carbon fiber reinforced polymer (CFRP) wastes, especially in the automotive industries, which lead to environmental pollution. In order to overcome the problem, the CFRP was reused as recycle material. In this research, the effect of chemical modification on tensile properties of thermoplastic reinforced CFRP waste was studied. The objectives of this research are to evaluate the mechanical, physical and thermal properties as well as to analyse surface morphology of polypropylene (PP) reinforced carbon fiber (CF). This research involves preparation of raw materials, surface treatment of recycled CF, fabrication of composite as well as testing and analysis of the final product. For surface treatment recycled CF, the CF was treated by using chemical modification to enhance their fiber-matrix interaction. For the composite fabrication, PP and CF were undergoing a mixing process in the internal mixer and hot pressing machine to produce a Polymer Matrix Composite (PMC) blend. The mechanical and physical testing that involved in this research were tensile and density, respectively. The morphology was observed by using Scanning Electron Microscopy (SEM). Furthermore, thermal analysis was conducted by using Differential Scanning Calorimetry (DSC). While, Fourier Transform Infrared Spectroscopy (FTIR) was used for identifying types of chemical bonds (functional groups). It was observed, composite with chemical surface modification produced better mechanical properties than composite without chemical surface modification regarding to its higher interfacial interaction of fiber-matrix.

ABSTRAK

Pada masa kini, terdapat banyak bahan buangan polimer bertetulang gentian karbon CFRP terutamanya daripada industri automotif, yang membawa kepada pencemaran alam sekitar. Bagi mengatasi masalah tersebut, CFRP telah digunakan semula sebagai bahan kitar semula. Dalam kajian ini, kesan pengubahsuaian kimia ke atas sifat tegangan termoplastik diperkuatkan dengan sisa CFRP telah dikaji. Objektif kajian ini adalah untuk menilai sifat-sifat mekanikal, fizikal dan terma serta untuk menganalisis morfologi permukaan polipropilena (PP) diperkukuhkan bahan buangan karbon prepreg serat (CFP). Kajian ini melibatkan penyediaan bahan-bahan mentah, rawatan permukaan CF yang dikitar semula, fabrikasi komposit serta ujian dan analisis produk akhir. Bagi rawatan permukaan CF yang dikitar semula, CF telah dirawat dengan menggunakan pengubahsuaian kimia untuk meningkatkan interaksi diantara gentian-matriks. Bagi fabrikasi komposit pula, PP dan CF telah menjalani proses pengadunan di dalam pengadun dalaman dan mesin penekan panas untuk menghasilkan campuran komposit matriks polimer (PMC). Ujian mekanikal dan fizikal yang telah dijalankan di dalam kajian ini adalah tegangan dan ketumpatan. Analisis morfologi telah dijalankan dengan menggunakan Mikroskop Imbasan Elektron (SEM). Selain itu, analisis terma telah dijalankan dengan menggunakan Pengimbasan Perbezaan Kalorimeter (DSC). Sementara itu, Spektroskopi Inframerah Transformasi Fourier (FTIR) pula telah digunakan untuk mengenal pasti jenis ikatan kimia (kumpulan berfungsi). Didapati, komposit dengan pengubahsuaian permukaan bahan kimia mempunyai ciri-ciri mekanikal lebih baik daripada komposit tanpa pengubahsuaian permukaan kimia disebabkan interaksi antara muka yang lebih tinggi di dalam gentian-matrix.

DEDICATION

To my late father Jaafar Talib. His words of inspiration and encouragement in the pursuit of excellence, still linger on. To my mother, who always stood behind me and knew I would succeed. Thanks for all you did. This work is dedicated to them.

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LIST OF ABBREVIATIONS, SYMBOLS AND NOMENCLATURE

ASTM	-	American Society for Testing and Materials
CF	-	Carbon Fiber
CFRP	-	Carbon Fiber Reinforced Polymers
CMC's	-	Ceramic Matrix Composites
CO ₂	-	Carbon Dioxide
Corp.	-	Corporation
CRT	-	Cathode Ray Tube
CTRM	-	Composites Technology Research Malaysia
DSC	-	Differential Scanning Calorimetry
e.g	-	Example
EOL	-	End Of Life
FTIR	-	Fourier Transform Infrared Spectroscopy
g/cm ³	-	gram per cubic centimeter
g/10min	-	gram per 10 minutes
g/l	-	gram per litre
GPa	-	Giga Pascal
hPa	-	hector Pascal
Hg	-	Mercury
i.e	-	that is
ISO	-	International Organization for Standardization
Kg	-	Kilogram
Kg/cm ³	-	Kilogram per cubic centimeter
Kg-cm/cm	-	Kilogram centimeter per centimeter
Kgf	-	Kilogram force
Kg/L	-	Kilogram per liter
MA	-	Maleic Anhydride

mbar	-	millibar
min	-	minute
ml/min	-	milliliter per minute
Mm	-	millimeter
MMC's	-	Metal Matrix Composites
MPa	-	Mega Pascal
NHO ₃	-	Nitric acid
PEEK	-	Ketone
pH	-	power of Hydrogen
PMC's	-	Polymer Matrix Composites
PP	-	Polypropylene
PP/unt-CF	-	Polypropylene Reinforced Untreated Carbon Fiber
PP/t-CF-3%MA	-	Polypropylene Reinforced Treated Carbon Fiber with 3% MA
PP/t-CF-5%MA	-	Polypropylene Reinforced Treated Carbon Fiber with 5% MA
reCF	-	recycle Carbon Fiber
rpm	-	revolution per minute
SCF/PP	-	Short Carbon Fiber/Polypropylene
SEM	-	Scanning Electron Microscopy
T_c	-	Crystallization Temperature
T_g	-	Glass transition Temperature
T_m	-	Melting Temperature
UK	-	United Kingdom
US	-	United States
V_f	-	Volume fraction of Fiber
V_m	-	Volume fraction of Matrix
X_c	-	Crystallinity of the Polymer
%	-	Percent
°C	-	Degree celcius
ρ_c	-	Density of composites

ρ_f	-	Density of fiber
ρ_m	-	Density of matrix
ΔH_f	-	Heat of fusion
ΔH_{100}	-	Heat of fusion for 100%

CHAPTER 1

INTRODUCTION

1.1 Introduction

Demand for carbon fiber reinforced plastic (CFRP) increasing by year due to the demand for high-strength lightweight materials. Carbon fiber reinforced thermoplastics are advanced materials for future high-performance composites. A combination of characteristics such as light-weight, corrosion resistance, low to moderate cost, high thermal stability and easy material processability, make them attractive for many applications.

Carbon fiber prepregs are one of the most promising advanced materials. Currently there is growing interest in this high-modulus, high-strength filaments in developing technology for their usage reinforcement materials in reinforced plastics composites. Nowadays, the impregnated carbon fiber with a liquid polymer resin such as epoxy and polyester are commonly used for structural components. Almost 95% of the CF, used in composite materials is processed into carbon fiber reinforced plastic. Prepregs continue to dominate in the production of CFRP's parts. Around 54% of the carbon fiber produced worldwide is used for manufacturing prepregs. In 2004, an estimated 22,000 metric tons of CF were consumed worldwide, about 8400 tons (about 18.5 million pounds) in the United States alone. The worldwide carbon fiber consumption almost doubled in 2010 to approximately 40,000 tons and it is estimated that the total global demand will be approximately 65,000 tons by 2014.

There are no promising structural product from recycle carbon fiber, but one of the latest applications available is non-structural applications for recycling CFRP's e.g. industrial paints, construction materials, electromagnetic shielding, high performance ceramic brake discs and fuel cells (Curry, 2010; Howarth and Jeschke, 2009; Panesar, 2009; Pickering, 2006). But there several aeronautics industries is particularly interested in incorporating recycled CFRPs in the interiors of aircraft as long as the materials are traceable and their properties consistent (which may easily achieved when the feedstock is manufacturing waste).

1.2 Problem statement

The challenges that lies ahead is to value added the pre-impregnated carbon fiber waste (prepregs waste). Recycling and reuse makes sense from an economic and environmental perspective, however, the carbon fiber composite recycling industry is only just beginning and some success stories are not told in public as part of the confidential issue of business strategy. The problem with recycling and reusing of carbon fiber prepreg waste arises from the difficulties to separate carbon fibers with its thermostat matrix. It lies in the chemical structure of the component where crosslinks that took place in the waste would limit the interaction of carbon fibers with a fresh matrix for new reinforcing purposes.

In spite of the interest in recycling and reusing the CFRP's, there are fiber-matrix related interface obstacles that need to be encountered. The weak bond achieved between fresh matrix resin and recycled or reused CFRP's has been the subject of many investigations, but still the role of the many interacting factors is not fully understood and is the subject of much debate. Typically interlaminar shear strengths, which are a measure of the fiber-resin bond, treated carbon fiber-epoxy resin composites are greater than untreated carbon fiber. The general approach to the shear strength problem with carbon fiber has been through fiber surface treatments. Due to that, this study is focusing on chemical

modification and it is postulated that the tensile properties of CFRP waste reinforced thermoplastic will be improved due to increase interaction between fresh matrix and CFRP surface by chemical modification.

1.3 Objective of the research

The objectives of this study are as follows:

1. To prepare polypropylene reinforced chemically modified carbon fiber prepregs via melt compounding
2. To study the effect of chemical modification on tensile properties of polypropylene reinforced chemically modified carbon fiber prepregs
3. To correlate the tensile properties of polypropylene reinforced chemically modified carbon fiber prepregs with its morphological characteristics

1.4 Scope of study

The study is mainly focusing on the effect of chemical modification on tensile properties of thermoplastic reinforced comminution for out of condition aerospace grade carbon fiber prepreg waste. The chemical modification is to enhance the properties of the second generation polymer composite. The formulation of polypropylene and a particle of carbon fiber prepregs will be divided into five main components according to fresh matrix: waste weight ratio (percentage); - 100:0, 99.5:0.5, 99:1, 97:3, 95:5 and 93:7. The composites were compounded by using internal mixer machines. Then, it was fabricated using hot press machine to form composite panel. In this study, physical and tensile testing was carried out to evaluate the character and properties of resulted composites. The data is supported with morphological characteristics of the tensile fracture surfaces.

1.5 Thesis outline

This report consists of 5 main chapters which are the introduction, literature review, methodology, result and discussion also conclusion and recommendation. Chapter 1 gives an introduction of the project and briefly explained the problem statement of the project, objectives, scope of research and report content. Chapter 2 discusses about the literature and theory, composite which including of materials, processing and properties. Methodology for the whole research work is explained briefly in Chapter 3. It contains of the project flowchart, compounding process and method for final sample preparation. In Chapter 4 were discussed the observation, results, analysis and evaluation done throughout the study and recommendation or suggestion for future works and improvement of this research were discussed in Chapter 5.

CHAPTER 2

LITERATURE REVIEW

This chapter generally describes about material, processing and properties of a composite. Major factors controlling the performance of composites were briefly described. Special emphasis was given on the methods of chemical modification of carbon fiber as it plays an important role on mechanical properties of composites. This chapter also includes the related study by previous researchers on the carbon fiber prepregs reinforced polymer.

2.1 Composites

A composite material is made by combining two or more materials, often ones that have very different properties (Kalpakjian and Schmid, 2006). The constituent that is continuous and is often, but not always, present in the greater quantity in the composite is termed the matrix (Matthew and Rowling, 2002). A composite may have a ceramic, metallic or polymeric matrix. The second constituent known as reinforcing phase or reinforcement, as it enhances and reinforces the mechanical properties of the matrix. Many composite materials are composed of just two phases, one is termed the matrix, which is continuous and surrounds the other phase called the dispersed phase. The properties of composites are a function of the properties of the constituent phases, their relative amounts, and the geometry of the dispersed phase (Callister and Reswisch, 2011).

2.2 Matrix based on Polymer Composite

According to Avila *et al.* (2003), on the basis of matrix phase, composites can be classified into metal matrix composites (MMC's), ceramic matrix composites (CMC's), and polymer matrix composites (PMC's). They can be again divided on the basis of matrix, that is, nonbiodegradable matrix and biodegradable matrix (Nikoleta and Hickel, 2009).

Thomas *et al.* (2012) states that most commercially produced composites use a polymer matrix material often called a resin solution. There are many different polymers available depending upon the starting raw ingredients. The most common are known as polyester, vinyl ester, epoxy, phenolic, polyimide, polyamide, polypropylene, polyether ether ketone (PEEK), and others. The strength of the product is greatly dependent on this ratio. PMCs are very popular due to their low cost and simple fabrication methods. Figure 2.1 shows the classification of PMC's according to the type of matrix material (Pandey, 2004).

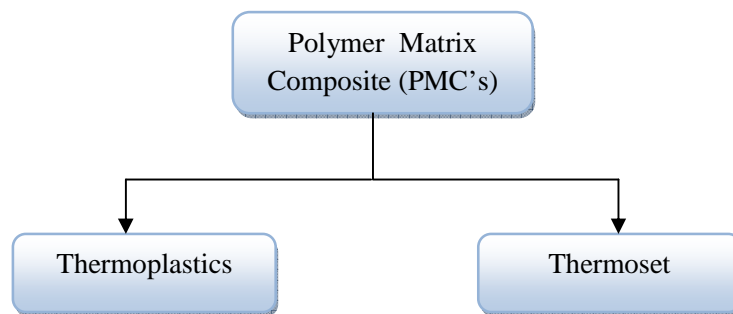


Figure 2.1: Classification of PMC's according to the type of matrix material (Pandey, 2004)

There are two main kinds of polymers which is thermosets and thermoplastics. Thermosets composite have qualities such as a well-bonded three-dimensional molecular structure after curing. They decompose instead of melting on hardening. Merely changing the basic composition of the resin is enough to alter the conditions suitable for curing and determine its other characteristics. Once cured, the materials cannot be

melted or re-shaped (Pandey, 2004). Thermoplastic composite readily flows under stress at elevated temperatures, so allowing them to be fabricated into the required component, and become solid and retain their shape when cooled to room temperature. These polymers may be repeatedly heated, fabricated and cooled and consequently scrap may be recycled, though there is evidence that this slightly degrades the properties because of a reduction in molecular weight (Matthew and Rowling 2002).

2.2.1 Polypropylene as Matrix in Thermoplastic Composite

According to Dikobe (2009), PP is one of the most important commodity polymers and is used in many areas, such as home appliances, automotive parts, construction and other important industrial applications. PP applications are often limited due to its low impact strength and Young's modulus properties, particularly at low temperatures and high temperature loading conditions. Blending PP with different polymers is an economic and effective way to improve these drawbacks. PP, supplied by has a density of $0.90 \text{ g}\cdot\text{cm}^{-3}$, a melting point of 165°C , a tensile strength of 30 MPa and a melt flow index of 12 g/10 min (230°C , 2.16 kg).

Polypropylene is a semi-crystalline polymer that is used extensively due to its unique combination of properties, cost and ease of fabrication. Polypropylene can be processed by a variety of fabrication techniques like film/sheet extrusion, multifilament, non-wovens, injection moulding, blow moulding and profile extrusion (Formosa Plastic, 2005). PP has an excellent resistance to stress and a low specific gravity. PP is readily able to be mechanically recycled several times using conventional equipment. Attempts have been made to reuse these waste plastics in order to reduce the environmental impact and consumption of the virgin plastics (Hannequart, 2004). Past studies have demonstrated that the recycled plastics possess similar mechanical properties, but are much cheaper than their virgin counterparts (Panthapulakkal *et al.*, 1991). Figure 2.2 shows the molecular structure of polypropylene (Cambray, 2006).