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JUDUL: **The Study of Mechanical Properties and Microstructure of Thermal-Curing Process on Natural Fiber (Kenaf) Reinforced Thermoset Composite**

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UNIVERSITI TEKNIKAL MALAYSIA MELAKA

The Study of Mechanical Properties and Microstructure of Thermal-Curing Process on Natural Fiber (Kenaf) Reinforced Thermoset Composite

Thesis submitted in accordance with the requirements of the Universiti Teknikal Malaysia Melaka for the Degree of Bachelor of Manufacturing Engineering (Manufacturing Design) (Honours)

By

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May 2007

DECLARATION

I hereby, declare this thesis entitled "The Study of Mechanical Properties & Microstructure of Thermal-Curing Process on Natural Fiber (Kenaf) Reinforced Thermoset Composite" is the results of my own research except as cited in the reference.

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APPROVAL

This thesis submitted to the Faculty of Manufacturing Engineering of UTeM and has been accepted as partial fulfillment of the requirement for the degree of Bachelor of Manufacturing Engineering (Honours) (Manufacturing Design).

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ABSTRACT

This paper contains the report of the study of mechanical properties and microstructure of thermal-curing process on natural fiber (kenaf) reinforced thermoset composite. The aims of this paperwork are to investigate the influence of the various exposure times and the various curing temperature on the mechanical properties and microstructure of the unsaturated polyester resins/kenaf composite and also to find the optimum parameter of curing times and curing temperatures in order to produce good mechanical properties of polymeric-matrix composites. The specimens are been differentiate by the curing temperature and curing time. The curing temperatures are 50ºC, 70ºC and 100ºC. Meanwhile the curing times are 30, 60 and 90 minutes. The composition of the compound are 240g unsaturated polyester, 10g kenaf and 2.4g of catalyst. The mixture is then stirred using mixer at the speed of 500rpm. Before curing with heat, the specimen was put on the vacuum oven to suck out all the air bubble trapped in the specimen. For study purpose the specimen was tested with tensile test, impact test and hardness test. Besides that, the interfacial microstructure was also observed using the SEM. From the study, as the curing temperature and curing time increases, the mechanical properties of the specimen decreases. But exception can be made for specimen 5030 (which cured at 50ºC and 30 minutes) because this specific specimen gives the best result compared to the rest of the specimen and this also including the reference specimen which cured at ambient temperature.

DEDICATION

For My family and friends

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LIST OF ABBREVIATIONS

ASTM	-	American Standard Test Method
SEM	-	Scanning Electron Microscope
UTM	-	Universal Testing Machine
UPR	-	Unsaturated Polyester Resins
PC	-	Polycarbonate
PET	-	Polyethylene terephthate
PMMA	-	Polymethyl methacrylate
PMCs	-	Polymer-matrix composites
MMCs	-	Metal-matrix composites
CMCs	-	Ceramic-matrix composites
T_g	-	Glass transition temperature
T_m	-	Crystalline melting point

CHAPTER 1
INTRODUCTION

1.1 Problem Statement and Background

Thermal curing process is a technique which used the heat as the main medium to cure the polymeric material compound. In this research, bast from Kenaf (natural fiber) is cured using the thermal cure technique. Seems recently, there is a high demand in finding the substituted of conventional composites, scientists and engineers is turning their attention to natural fibers which can almost reach the same mechanical properties as the conventional composites. Heat generated by the oven can increase the reaction rate for any chemical reaction for several different, although interrelated, reasons. Since molecules must collide in order to react, when are heated they move more and therefore the chance that they collide is increase. This enhanced polymers to crosslink to each other and will produce higher strength due to crystalline structures. To make it more interesting, catalyst is added to thermoset resin in order to increase it curing rate under the heat oven. It can also help the polymeric material to undergo curing process even after exposed with heat.

Polyester resin was used because Polyesters are versatile because of their capacity to be modified or tailored during the building of the polymer chains. They have been found to have almost unlimited usefulness in all segments of the composites industry. The principal advantage of these resins are the balance properties (including mechanical, chemical and electrical) dimensional stability, cost and ease of handling or processing. Besides that, more reason to study and give more attention to polyester is unsaturated

polyester resins (UPR) are the workhorse of the composites industry and represent approximately 75% of the total resins used. Meaning, polyester resin is widely used all around the world and applied many sector, for examples in the aerospace, automotive, boats, bus shelters, storage tank, cooling tower, dustbin, safety helmet, sorting equipment and many more. More than that, composites made with polyester possess excellent mechanical strength and have good rigidity and outstanding durability.

Thermal curing can cut the curing time short. Upon heating, the matrix viscosity is reduced and therefore, the molecules move more freely and colliding likelihood is increased. Making the chemical reaction go even faster than usual. But careful studies have to be done because if the cure proceeds to quickly because too much heat generated, the part could be too brittle. Also, the crosslinking might proceed so quick that all of the styrene is not used and the resulting part could have residual styrene content. If the too much peroxide was used, incomplete crosslinking would likely result and that would lead to poor physical and mechanical properties. The best measure is to understand, monitor and control the various components in the matrix and the variables that control the rate. Therefore for this research the varied parameter are the curing temperature and the curing time.

In this research, polyester resin is used as the matrix and mixed together with the kenaf. Natural fiber (kenaf) is being used in this research as the replacement for glass fiber which is dangerous if accidentally inhaled. In the past decades, scientists and engineers have been shifting from monolithic material to fiber-reinforced polymeric materials (notably aramid, carbon and glass fiber reinforced plastic). Conventional and traditional fiber reinforced composites are composed of carbon fibers, glass fibers, which are incorporated into unsaturated polyester or epoxy. These composites show high mechanical and thermal properties. And because of that, although there are many different types of resin in use in the composite industry, the majority of structural parts are made with three main types, namely polyester, vinyl ester and epoxy. (www.azom.com, 2007). However these advantages have serious drawbacks, for

example; environmental problems in disposal by incineration and dangerous if accidentally inhaled. Because of that scientists and engineers turn to natural fibers which are low in density, cost and energy consumption compared to glass fibers. Besides that, natural fiber are renewable, recyclable, no abrasion to machine and biodegradable. This was proven by the article stated in compositesworld.com, which mention that the U.S government who realizes the potential of natural fibers is promoting it to all the manufacturers in U.S.

For this research, the main raw materials used are kenaf (natural fiber), polyester resin (SHCP 268 BQT) and catalyst (BUTANOX M-50). To identify which combination of curing times and temperatures work the best and give the good result, the curing time are varied into 30, 60 and 90 minutes. Meanwhile for the curing temperatures is 50ºC, 70ºC and 100ºC. Hopefully all this parameter will provide clearer and better result compare to the previous research. According to the research made by Chaowasakoo, T and Sombatsompop, T (2007) in "Mechanical and morphological properties of fly ash/epoxy composites using conventional thermal and microwave curing methods", the curing temperature and curing time was used are 70ºC and 80 minutes and better result is shown by the specimen cured using microwaves. Only one curing parameter was introduced and as for that, for this research, 10 different parameters were set. Hopefully it can answer the mystery of which combination of mixture provides better result compare to the others. Samples characteristic will be analyzed and study through several of test like hardness, density, peel, impact and monitored using the Scanning Electron Microscope (SEM). All this testing will be conduct according to the ASTM standard. Below are how the specimen is been varied from each other.

Table 1.1 Specimens variation and coding indicator

Curing Temperature (Celsius) / Curing Time (Minutes)	50	70	100
30	5030	7030	10030
60	5060	7060	10060
90	5090	7090	10090

1.2 Objectives and Scope of Project

Using the thermal curing process technique, samples will be monitored and evaluates. This is to identified, whether the thermal cure manage to cure the polymeric- matrix composites effectively and improve its mechanical properties. Below are the research objectives:

1. To investigate the influence of the various exposure time and the various curing temperature on the mechanical properties and microstructure of the unsaturated polyester resins/kenaf composite.
2. To analyze the mechanical properties of the thermal cured unsaturated polyester resin/kenaf composite with the one cured using the ambient temperature
3. To find the optimum parameter of curing times and curing temperatures in order to produce good mechanical properties of polymeric- matrix composites.

Several series of research activities will be conducted, these include:

1. Characterization of raw materials: natural fiber (Kenaf) ,thermoset resin (polyester) and catalyst
2. Fabrication of natural fiber reinforced polymeric-matrix composites
3. To study the influence of thermal curing process to the mechanical properties and microstructure of the composites

1.3 Study Benefits

Through this research and study, hopefully useful information can be produced and benefit to other scientists and engineers in the same field of research. Below are the lists of benefits that can be obtained from this research:

1. Result from experiment and testing can provide information which answers all or some doubt regarding the application of thermal curing process on the kenaf (natural fiber) product and composites.
2. Valuable information like this can lure more researchers to keep on going improving the thermal curing process technique and also move their attention towards the potential of natural fibers replacing or substitute for the normally used composite and fiber glass.

Through this research also, hopefully it can promote others to continue this study, incorporate to find the other advantages that can be provided when applying thermal curing process in curing unsaturated polyester resins/kenaf composites. By doing this it can also promote join venture among the developer, institution and government to make use of natural fibers which been assume as a waste over the decades, into useful product and industry applications.

CHAPTER 2

LITERATURE REVIEW

2.1 Thermal Curing Process

Thermal curing process is a technique which utilizes the heat generated by the conventional oven as the main medium to cure the polymeric material compound.

2.1.1 What happens during curing?

During the curing reaction, medium-sized molecules link together to form enormous molecules and resulting material becomes solid with dramatic increase in strength, stiffness, hardness and other desirable mechanical, physical and chemical properties. As a result, solid material cannot be remelted or reshaped, so any molding (shaping) of the material must be done while curing takes place. Reinforcing fibers must also be introduced before curing so that the fibers can be wetted by the liquid resin and can be properly encapsulated and positioned in the resulting solid product. The creation of the links (bonds) during curing involves a series of chemical reactions. By examining these reactions, you should gain a greater insight into the basics of curing reactions in general and, thereby, understand which variables are important to control and how they should be controlled with the particular resin system you employ (Strong, 1996).

2.1.2 Curing polyester fundamental

Usually the polyester resin used is generally a liquid consisting of medium molecules. These molecules are in form of short-chain polyester polymers which have been synthesized by the resin manufacturer. In processing the resin, the manufacturer varies the components and conditions to create a wide range of resin grades and types. These various grades and types allow great variations in suggested cure conditions, final part properties and resin costs (Strong, 1996).

The curing reaction for polyester resins can be initiated by adding small quantities of a catalyst for example organic peroxide or an aliphatic azo compound, to the polyester resin. With the presence of heat, the catalyst rapidly decomposes into free radicals and react with the styrene molecules and break their [C = C] bonds. Meanwhile the freed styrene radicals reacted with the polyester molecules at their unsaturation points and eventually form cross-links between them. Finally, the result from this interaction produces a solid polyester resin.

The above process is only going to stop when the free radical molecules reacted with other molecules besides the styrene and [C = C] bond. The unwanted molecules can include oxygen, contaminants and other peroxide molecules. Therefore, if these other reactants present in large amount, the crosslinking reactions process could be disturb and worse terminated earlier than it should be, making only small amount of crosslinks formed. Because of this, it is important to control the peroxide and the other substances concentration.

In actual practice, several peroxide molecules will react with several [C = C] bonds to begin these crosslinking reactions throughout the mix. Only a few peroxides are needed to get the reactions started. Hence, the peroxides are only needed in small quantities, usually 1 to 2% of the total resin weight (Strong, 1996).

According to A. Brent Strong (1996) in each bond that formed heat was being generated. The heat is generated because of the molecules in the matrix, moved and crashed to each other in fast motion. This rising temperature incidence is known as exotherm. Normally, thicker specimen generated higher exotherms compared to thin specimen. Because the heat is retained by the polymer, causing the reactions to occur even faster and therefore even more heat produced.

2.1.3 Heat increase reaction

Energy caused by heat can increase reaction rate of a chemical reaction. This is because, when a temperature of a system rises, it produces an increase in the speed of the particles of the system (Wikipedia, 2007).

In the present of heat, the heated molecules move even more and molecules must collide in order to react, therefore the chance that molecules collide to each other increased. In the same time the viscosity the compound is reduced by heat (Strong, 1996) and therefore, the molecules move more freely and the chances of them colliding among them is increased. At higher heat the energy of the moving molecules increases, meaning that when it collides they collide harder and as a result it creates an effective collision. Below is the equation of bonding energy of an atomic system (Callister Jr, W.D, 2001).

$$E_N = E_A + E_R$$

Where by, E_N, E_A and E_R are represent the net, attractive and repulsive energies for two isolated and adjacent atoms.

But referrers to A. Brent Strong (1996), not all collision result in bonding. The heating of the molecules also increase the overall energy in the system, thus making it easier for

the molecules to overcome the energy of activation, Q [The energy required to initiate a reaction, such as diffusion (Callister Jr, W.D, 2001).

$$Q_d = -(R\ T\ D / D_o \exp)$$

Where

Q_d = the activation energy for diffusion (J/mol)

D_o = a temperature-independent preexponential (m^2/s)

R = the gas constant, 8.31 J/mol-K

T = absolute temperature (K)

which is a threshold barrier for effective reactions to occur. Yet, another effect of heating is to increase the break-up of the peroxide molecules, thus increasing the number of chains that are started and therefore the rate of the curing reaction. Heat can also cause direct formation of free radicals at the carbon-carbon double bonds in the polyester molecules. It is this possibility that lead to addition to inhibitors to prevent crosslinking from occurring prematurely.

2.1.4 Polymer reaction due to heat cure.

Based on the study made by Prof. McCullough R.L (1986), a preliminary temperature cycle should be selected which minimizes the peak temperature observed in the center of the composite, to ensure the composite cures inside out once the gel point is reached and maintains the curing time less than 90 minutes.

Currently, the dominant form of composite materials utilizes the thermoset polymeric materials as the matrix phase. In the presence of catalyst, heat, radiation and pressure, thermoset resins solidify through an irreversible exothermic chemical reaction or

"cure". Prior to cure, the polymer is a viscous fluid that can be made to flow under pressure. As the cure reactions proceed, the molecules react to form covalent bonded three-dimensional network. The increasing molecular weight is accompanied by an increase in viscosity. At gel-point, a loose three-dimensional network pervades the system, the polymer exhibits the behavior of a gel and flow ceases. However, the reactions continue and form a tightly cross-linked structure with the characteristics of a glassy solid (McCullough et al., 1986).

Below are the list of a minimal successful cure cycle is defined in terms of several non chemical criteria:- (McCullough et al., 1986)

- All of the polymer must have achieved a minimum acceptable degree of cure.
- The formation of voids must be prevented such that the number and size of all voids are below an acceptable limit.
- The temperature must remain below some critical value at which damage to the structure may occur through polymer degradation.
- The part must be cured uniformly such that large gradients in degree of conversion or temperature are avoided. This is because the presence of such gradients will result in residual stresses which detract from the performance of the part.

Parts that meet these minimal standards are subsequently subjected to mechanical characterization to evaluate performance characteristics such as rigidity, strength, fracture toughness, damage tolerance and many more (McCullough et al., 1986).