

UNSATURATED POLYESTER COMPOSITES FILLED WITH  
UNTREATED AND SURFACE TREATMENT SUGARCANE  
BAGASSE BY HOT PRESS TECHNIQUE

MUHAMMAD SALIHIN ZAKARIA

**UNIVERSITI MALAYSIA PERLIS**

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Unsaturated Polyester Composites Filled with Untreated  
and Surface Treatment Sugarcane Bagasse by  
Hot Press Technique

By

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## ABSTRAK

Matlamat penyelidikan ini adalah penambahbaikan sifat-sifat mekanikal termoset yang diperkuat gentian semulajadi, sebagai satu keputusan pengoptimuman sifat-sifat gentian hampas tebu menggunakan proses rawatan NaOH. Tert-butyl perbenzoate telah digunakan sebagai pemangkin pada suhu tinggi untuk pemprosesan komposit poliester yang efisien. Gentian hampas tebu telah dialkalkan dengan 1, 3 dan 5% kepekatan larutan NaOH, digabungkan dengan empat formula muatan gentian, dan ditekan panas untuk membentuk komposit gentian semulajadi. Modulus dan kekuatan lenturan komposit gentian tebu yang dialkalkan dengan 3% larutan NaOH memberikan nilai yang tertinggi dan gentian tebu yang tak dirawat memberikan nilai yang terendah. Seperti yang dijangka, haluan yang sama juga diperoleh untuk modulus dan kekuatan tegangan. Walau bagaimanapun, dengan meningkatkan larutan NaOH, sifat-sifat mekanikal menurun disebabkan oleh fibrilasi yang membawa kepada pemindahan tegasan yang tidak efektif. Sementara itu, takat putus pemanjangan komposit menurun kerana peningkatan lekatan matriks-gentian (yang memberi lebih kekakuan kepada komposit). Analisis dinamik mekanikal menunjukkan dengan rawatan 3% larutan NaOH, komposit hampas tebu memberikan nilai modulus penyimpanan yang tinggi dan nilai  $\tan \delta$  yang rendah. Pemerhatian SEM ke atas permukaan patah komposit menunjukkan pengubahsuaian permukaan gentian terhasil dan meningkatkan lekatan matriks-gentian. Ujian penyerapan air juga menunjukkan dengan rawatan larutan 3% NaOH, komposit hampas tebu memberikan ikatan dan lekatan yang paling kuat berbanding komposit hampas tebu yang lain.

## ABSTRACT

*The aim of this research is the improvement of the mechanical properties of natural fiber reinforced thermoset, as a result of optimization of the properties of sugarcane bagasse fibers by the use of NaOH treatment process. Tert-butyl perbenzoate was used as catalyst at elevated temperature for efficient processing of polyester composites. Sugarcane bagasse fibers were alkalized with 1, 3 and 5% NaOH solution, combined with four different fiber loading formulation, and hot-pressed to form natural fibers composites. The flexural modulus and strength of bagasse fiber composites alkalized with 3% NaOH solution gave the highest value and the untreated bagasse fiber gave the lowest. The same trend was obtained for the tensile modulus and strength as expected. However, by increased the NaOH solution, the mechanical properties decreased as a result of fibrillation lead to uneffective stress transfer occurred. Meanwhile, the break elongation of composites was decreased due to the improved fiber-matrix adhesion (to give more stiffness to the composites). Dynamic mechanical analysis (DMA) showed that with 3% NaOH solution treatment, bagasse fiber composites gave the highest storage modulus ( $E'$ ) values and the lowest  $\tan \delta$  values. SEM observations on the fracture surface of composites showed that the surface modification of the fiber occurred and improved fiber-matrix adhesion. A water absorption test also showed that with 3% NaOH solution treatment, bagasse fiber composites gave higher bonding and adhesion compared to all another bagasse fiber composites.*

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## LIST OF ABBREVIATIONS, SYMBOLS AND NOMENCLATURE

%	percent
E	dynamic modulus
AA	acrylic acid
APTS	1, 3 - amino propyl triethoxy silane
ASTM	american society for testing and materials
DMA	dynamic mechanical analysis
DP	degree of polymerization
DSC	differential scanning calorimetry
DTA	differential thermal analysis
E''	loss modulus
E'	storage modulus
FRP	fiber reinforced polymer
FTIR	fourier transform infra red
g	gram
GFRP	glass fiber reinforced plastic
GMA	glycidyl methacrylate
GP	general purpose
h	hour
ha	hectare
H <sub>z</sub>	hertz



MA	maleic anhydride
Mc	adjacent chemical junctions
MEKP	methyl ethyl ketone peroxide
mgKOH	mass of potassium hydroxide in milligrams
mm	millimeter
MMA	methyl methacrylate
Nc	number of chemical junction
°C	degree celcius
OH	hydroxide
PCM	polymeric composite materials
php	per hundred part
PP	polypropylene
psi	pound per square inch
RTM	resin transfer molding
SEM	scanning electron microscope
tan	tangent
$T_g$	glass transition temperature
Tert	tertiary
UPR	unsaturated polyester resin
wt	weight
XRD	x-ray diffraction
$\alpha$	alpha
$\beta$	beta
$\gamma$	gamma
$\delta$	delta

## CHAPTER 1

### INTRODUCTION

#### 1.1 Fiber Reinforced Plastic Composites

Fiber-reinforced plastic composites have played a dominant role for a long time in a variety of applications for their high specific strength and modulus. Fiber incorporated plastics have been very popular due to their flexibility, their lightness and the ease of fabrication of complicated shapes with economic savings in contrast to fiber reinforced metals/ alloys. In addition, these composites can easily substitute for conventional materials in several areas such as the building industry, transportation and consumer goods. Some of the attempts made in recent times for the utilization of natural fibers through composite material technology have indicated their potential as substitutes for conventional materials such as wood and glass fiber reinforced plastics (GFRP) in many applications (Satyanarayana et al., 1990).

The composites reinforced with filler have received crescent interest from industries for application in components with individual mechanical properties.

Reinforcement of polymer matrix using inorganic fillers is widely used, resulting in materials with excellent mechanical and thermal properties (Chawla, 1998).

In recent years, organic fillers, such as cellulose-based fibers have slowly taken root in the market to replace synthetic materials (Pandey et al., 2000). Cellulose-based fibers (natural fibers) come from renewable and relatively inexpensive resources, and also by the addition of natural fibers, such as wood fibers, flax or sisal to polymeric matrices can result in feasible composites concerning mechanical and economic points of view (Luyt & Malunka, 2005). Composites based on natural fibers are environmentally superior to those based on synthetic fibers, such as fiberglass. Recent studies reported that the incineration of components with natural fibers uses 45% less energy, and results in lower air emissions (Joshi et al., 2004).

It is well known that plant fibers also known as lignocellulosic fibers have been one of the attractive fillers for different types of polymers including rubbers as well as for ceramic matrices due to some of their unique characteristics unparalleled with any other reinforcing/filler materials. They include biodegradability contributing to a healthy ecosystem, low cost and higher stiffness to those of glass fibers, which would be attractive for the applications requiring stiffness dominant ones such as the automotive applications (Rijswijk & Brouwer, 2002). Additional motivation for their use in composites to receive greater attention in recent times is the increasing ecological considerations with many governments such as European Union passing laws for the use of about 95% recyclable materials with about 85% renewable materials in them in all new automotives to achieve the “end of life” required by 2015 (Netravali & Chabba, 2003; Peijs, 2003). In addition, use

of their composites have established comparable performance with those of glass fiber composites with possibility for their use as structural components as well (Bledzki & Gassan, 1999; Burgueno et al., 2004; Corbiere-Nicollier et al., 2001; Dweib et al., 2004; Holbery & Houston, 2006; Joshi et al., 2004;). When such materials are used in composites, developing countries, which produce these, become part of global composite industry as developer and manufacturer leading to increased revenues and creation of jobs (Rijswijk & Brouwer, 2002).

However, lignocellulosic fibers are used only to a limited extent in industrial practice due to the difficulties associated with surface interactions. The primary drawback of agro-based fibers is associated with their inherent polar and hydrophilic nature and the hydrophobic characteristics of most of thermoplastics. It resulting in difficulties during compounding the filler and the matrix, and therefore in achieving acceptable dispersion levels, which yields composites with low performances. This hydrogen bonding phenomenon is best exemplified in paper where these secondary interactions provide the basis of its mechanical strength. It also results in high moisture absorption and swelling of the fibers. Poor fiber–matrix interface induces a decrease in mechanical properties.

The properties of the lignocellulosic composites are dominated by the interfacial interaction between the lignocellulosic filler and polymer matrix. Generally, there are two types of interaction at the interfacial region, i.e., primary and secondary bondings. Primary and secondary bondings include covalent and hydrogen bondings, respectively. While covalent bondings at the interfacial region exist in thermoplastic–wood composites with the incorporation of a coupling agent, such bondings are more prevalent in the thermoset–

lignocellulosic composites. This is because lignocellulosic hydroxyl (OH) groups could serve as reaction sites with various functional groups in the thermoset system.

Thermal analysis of polymers is an important study, covering a broad field of applications and is a method used for understanding the structure–property relation and mastering the technology for industrial production of different polymeric materials, especially fiber-reinforced composites.

The properties of composite materials can be determined by the characteristics of the polymer matrices, together with reinforcements and the adhesion matrix-fiber interface and the bonding strength at the interface (Hull, 1981). As a consequence of these characteristics, sensitive techniques must be used, such as dynamic mechanical analysis (DMA), in which monitor changes in the mechanical properties, and serves as an important thermal analysis technique for characterizing the fiber-matrix interface (Keusch & Haessler, 1999; Saha et al., 1999; Murayama, 1978).

Dynamic mechanical analysis is a sensitive technique that is used in studying the effect of temperature on the mechanical properties of materials including polymers and composites. The technique separates the dynamic modulus  $|E|$  of materials into two distinct parts: an elastic (storage) part ( $E'$ ) and a viscous (loss) component ( $E''$ ). The elastic storage modulus  $E'$  is the component of the dynamic modulus  $|E|$  where the strain is in phase with the applied stress and the loss modulus  $E''$  is the component of the dynamic modulus  $|E|$  where the strain is  $90^\circ$  out of phase with the applied stress. The ratio of  $E''$  to  $E'$  gives the tangent of the phase angle  $\delta$  and  $\tan \delta$  is known as the damping and is a measure of energy

dissipation. Such parameters provide quantitative and qualitative information about material behavior (Aziz & Ansell, 2004).

## 1.2 Problem Statement

Bagasse is the residue after the crushing of sugar cane for juice extraction and on average, about 32% of bagasse is produced from every tonne of sugar cane had been processed. The total plantation area of sugarcane in Malaysia is nearly 22,500 ha. About 1,111,500 tonnes of sugar cane is produced in 2002, hence bagasse can be easily obtained in Malaysia. Increasing concern of disposal of bagasse residual creates interest to explore the potential application of this material (Lee & Mariatti, 2008).

Nowadays, sugarcane bagasse leftover still has a very harmful effect upon the environment around the world (de Souza et al., 2003). If conveniently modified and treated, sugarcane bagasse can be converted into a variety of chemicals, such as furfural, bagasse composite, or else be used to fabricate paper. Usually, methyl ketone peroxide (MEKP) was used as a catalyst in producing thermoset composites at room temperature. But its obtain times or days to cured. This is not efficient to apply in industrial scale. Therefore, in order to produce value added of sugarcane bagasse at efficient time, thus the idea of introducing NaOH treatment sugarcane bagasse filled unsaturated polyester composites with tert-butyl perbenzoate was born since there was no previous work have been done using tert-butyl perbenzoate as catalyst in natural fiber composites. By using tert-butyl perbenzoate as a catalyst, elevated temperature was needed for the curing process. Hence, time efficiency is gained and value added product was produced.

### **1.3 Objectives of Study**

The primary objectives of this study are:

- 1) To improve properties of natural fibers composites using NaOH surface modification. Preliminary study was carried out using only untreated bagasse fibers, in order to obtain a basic understanding of their characteristic and their effect when incorporated into unsaturated polyester. The mechanical properties of the treated and untreated natural fibers composites were evaluated. This evaluation of surface modification was carried out to determine the suitable concentration to modify the sugarcane bagasse that yields the best of overall properties.
- 2) To study the dynamic mechanical analysis of the sugarcane bagasse composites.
- 3) To study the morphological of the treated and untreated sugarcane bagasse composites to support the mechanical properties results.

### **1.4 Outline of Thesis Structure**

Chapter 1 starts with a brief introduction on lignocellulosic reinforced plastic. Issues that were of concern, which generate the ideas and energies to this research work, are also stated. The primary objectives and the general flow of the whole research program are also carefully outlined.

Chapter 2 relates the role of polymer matrix, filler and reinforcement with particular interest focused on alkali treatment. Subsequently, a literature survey was done on various published works on natural fiber/ polymer composites, particularly those that are closely related to this work. Works on other polymer composites was also extensively reviewed.

Chapter 3 describes step-by-step the experimental procedures employed, details of lab equipments used as well as any other processing techniques involved in generating any data that were used and presented in the course study.

Chapter 4 reports the effect of NaOH solution concentration with filler loading of treated and untreated sugarcane bagasse filled unsaturated polyester. Data graphs on the mechanical, thermal, swelling and morphological properties of these composites would be presented here and a detailed analysis would be carried out based on the data collected.

Chapter 5 presents some concluding remarks on the present work as well as some suggestion for future work.