# CHARACTERIZATION AND PROPERTIES OF PALM FIBER FILLED UNSATURATED POLYESTER COMPOSITES



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# Characterization and Properties of Palm Fiber Filled Unsaturated Polyester Composites



by

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## LIST OF ABBREVIATIONS AND SYMBOLS

| | |
|---|---|
| µm | Micron meter |
| $\delta$ | Energy dissipation |
| AA | Acrylic acid |
| AC | Acetic anhydride |
| ASTM | American Society for Testing and Material |
| BAK 1095 | Biodegradable plastic |
| C | Carbon |
| $C_5H_5N$ | Pyridine |
| $C_6H_{11}O_5$ | $_D$-anhydroglucose |
| $C_{18}H_{33}OCl$ | Oleoyl chloride |
| $CH_2Cl_2$ | Dichloromethane |
| $CO_2$ | Carbon dioxide |
| CPO | Crude Palm Oil |
| DMA | Dynamic mechanical analysis |
| DMAP | 4-Dimethylaminopyridine |
| E’ | Storage modulus |
| EA | Elemental analysis |
| EFB | Empty fruit bunches |
| FFB | Fresh fruit bunches |
| FRPs | Fiber-reinforced plastics |
| FTIR | Fourier Transform infrared spectroscopy |
| GFRP | Glass fiber reinforced plastics |

| | |
|---|---|
| H | Hydrogen |
| HDPE | High-Density Polyethylene |
| HFRUPE | Hemp fiber reinforced unsaturated polyester |
| ILSS | Interlaminar shear strength |
| MA | Maleic anhydride |
| MAPP | Maleic anhydride grafted polypropylene |
| MEKP | Methyl Ethyl Ketone Peroxide |
| mm | Milimeter |
| NaOH | Sodium hydroxide |
| O | Oxygen |
| OH | Hydroxyl |
| OPF | Oil Palm Fiber |
| PALF | Pineapple leaf fiber |
| Pd | Palladium |
| PF | Palm fiber |
| php | Part per hundred polymer |
| ppt | Parts per thousand |
| RH | Rice husk |
| RTM | Resin transfer moulding |
| S | Styrene |
| SEM | Scanning electron microscopy |
| TEM | Transmission electron microscope |
| Tg | Glass transition temperature |

**Pencirian dan Sifat-sifat Komposit Poliester Tak Tepu Terisi Gentian Sawit**

## ABSTRAK

Penyelidikan ke atas sifat-sifat dan pencirian komposit poliester tak tepu (PTP) terisi gentian sawit (GS) telah dikaji. Komposit disediakan dengan menggunakan teknik pelapisan tangan sebelum dimatangkan di dalam oven selama 10 minit pada suhu 90˚C. Kesan pembebanan pengisi gentian sawit terisi komposit PTP ke atas sifat-sifat mekanikal seperti sifat-sifat tensil dan kelenturan, morfologi telah dikaji. Keputusan menunjukkan elastik modulus dan modulus kelenturan komposit PTP/GS meningkat dengan semakin meningkatnya pembebanan gentian sawit tetapi kekuatan tensil, kekuatan lenturan dan pemanjangan pada takat putus berkurangan. Kajian mikroskop elektron penskanan (SEM) terhadap permukaan patahan tensil komposit PTP/GS menunjukkan interaksi permukaan yang lemah di antara gentian sawit dan PTP. Kesan saiz gentian sawit yang berbeza menunjukkan bahawa komposit PTP/GS dengan saiz 75 µm mempunyai kekuatan tensil dan kelenturan, pemanjangan pada takat putus, elastik modulus dan modulus kelenturan yang lebih tinggi berbanding dengan komposit saiz 150 µm. Rawatan kimia gentian sawit dengan oleoil klorida, 4-dimethilaminopiridina (DMAP) dan air laut digunakan untuk meningkatkan sifat-sifat mekanikal komposit. Kesan oleoil klorida telah meningkatkan kekuatan tensil dan kelenturan, elastik modulus, modulus kelenturan kecuali pemanjangan pada takat putus komposit yang dirawat. Komposit yang terawat dengan DMAP menunjukkan kekuatan tensil dan kelenturan, elastik modulus, modulus kelenturan yang lebih tinggi tetapi pemanjangan pada takat putus yang rendah berbanding komposit tanpa rawatan. Kesan rawatan gentian sawit dengan air laut telah meningkatkan sifat-sifat tensil dan kelenturan kecuali pemanjangan pada takat putus komposit. Mikrograf SEM menunjukkan kesemua komposit rawatan mempunyai interaksi antara muka dan perlekatan yang lebih baik diantara gentian dan matrik. Perubahan kumpulan berfungsi komposit rawatan telah dibuktikan dengan analisis spektroskopi FTIR.

**Characterization and Properties of Palm Fiber Filled Unsaturated Polyester Composites**

## ABSTRACT

The research on characterization and properties of palm fiber (PF) filled unsaturated polyester (UP) was investigated. The composites were prepared by using hand lay up technique before cured on oven about 10 minutes at 90˚C. The effect of filler loading on mechanical, flexural properties and morphology of palm fiber filled UP composites was studied. The modulus of elasticity and flexural modulus of UP/PF composites increase with increasing palm fiber loading while decrease the tensile strength, flexural strength and elongation at break. Scanning electron microscopy (SEM) study on tensile fracture surface of UP/PF composites indicates that poor interfacial interaction between palm fiber and UP matrix with increasing filler loading. Different sizes of palm fiber composites show that the UP/PF composites with size 75 µm have higher tensile and flexural strength, elongation at break, modulus of elasticity and flexural modulus compared the composites with size 150 µm. The chemical treatment of palm fiber with oleoyl chloride, 4-dimethylaminopyridine (DMAP) and seawater was used to improved mechanical properties of composites. The effect of oleoyl chloride has increased the tensile and flexural strength, modulus of elasticity, flexural modulus except elongation at break of treated composites. The treated composites with DMAP exhibit higher tensile and flexural strength, modulus of elasticity, flexural modulus but lower elongation at break compared untreated composites. The treatment with seawater of palm fiber has increases tensile and flexural properties, except elongation at break of composites. The micrographs SEM showed the all treated composites have better interfacial interaction and adhesion between fiber and matrix. The change of functional group of treated composites was evidenced by FTIR spectroscopy analysis.

# CHAPTER 1

# INTRODUCTION

## 1.1 Potential of Natural Fiber

Nowadays, development of natural fiber reinforced composites is highly attractive research lines (Corrales et al., 2007). Polymers have replaced many conventional materials because of their advantages in ease processing, low cost material generation. A natural fiber provides interesting properties for composites, especially capacity of recycling, renewable raw material, which is less abrasive and harmful to mankind (Mohanty et al., 2000).

Cellulosic fibers, like henequen, sisal, coconut fiber (coir), jute, Palm Fiber (PF), bamboo, wood, paper in their natural condition, as well as, several waste cellulosic products such as shell flour, wood flour and pulp have been used as reinforcement agents of different thermosetting and thermoplastic resins (Rana et al., 1997). During the decortications of the henequen leaves, and also during the transformation of the raw fibers into cordage approximately a 10% of waste is produced. These waste fibers could be profitably used in the manufacture of short fiber polymer reinforced composites because they posses attractive physical and mechanical properties (Hornsby et al., 1997). These waste fibers are composed of approximately 60% of cellulose pulps which are easily obtained and could also be used to reinforce polymeric materials. Unlike the traditional engineering fibers, e.g. glass and carbon fibers, and mineral fillers, these lignocellulosic fibers are able to impart the composite certain benefits such as low density, less machine wear during processing than that produced by mineral reinforcements, no health hazards, and a high degree of flexibility. The later is especially true because these fibers, unlike glass fibers, will bend rather than fracture during processing (Herrera-Franco et al., 2005). The glass fiber can cause acute irritation to the skin eyes, and upper respiratory tract. Concerns for long-term development of lung scarring (i.e., pulmonary fibrosis) and cancer have been raised because fibrous glass and other synthetic vitreous fibers, when disturbed, release fibers that can become airborne, inhaled, and retained in the respiratory tract. Research has been done to find a safer, cheaper, and maybe better fiber than glass fiber. In this case, natural fibers are the obvious choice (Sapuan et al., 2003; Arib et al., 2006).

Composites reinforced with glass fibers have found applications in many fields, but their non-recyclability becomes a significant disadvantage at the end of their lifetime (Corrales et al., 2007). Glass fiber reinforced plastic provides excellent thermal and mechanical properties. However, these properties make it difficult to carry out suitable disposal processing. Furthermore, it is necessary to reduce environmental impacts, such as global warming, that are generated by consumption of petroleum, a non-renewable resource. The use of natural fiber reinforced plastics represents an attractive and suitable method for replacing glass fiber reinforced plastic (Wambua et al., 2003; Joshi et al., 2004).

Thermoset is a hard and stiff crosslinked material that does not soften or become moldable when heated. Thermosets are stiff and do not stretch the way that elastomers and thermoplastics. Several types of polymers have been used as matrices for natural fiber composites. Most commonly used thermoset polymers are epoxy resins and other resins Unsaturated polyester (UP), Vinyl Ester, Phenolic Epoxy, Novolac and Polyamide) (Hull & Clyne, 1996; Bledzki & Gassan, 1998; Chawla, 1987). Unsaturated polyester matrices (Mahdi et al., 2004) were have been popular thermoset and widely used as the polymer matrix compared to epoxy (Gassan & Bledzki, 1999) plastic and thermoset based composites, the applications concern mainly automotive industry or domestic objects (Wo¨tzel et al., 1999) as they possess many advantages compared to other thermosetting resins including room temperature cure capability, good mechanical properties and transparency. Curing of unsaturated polyester is due to a polymerization reaction that causes crosslinking among individual linear polymer chains (Aziz & Ansell, 2005). In contrast to other thermosetting resins, no by-product is formed during the curing reaction; hence resins can be moulded, cast and laminated at low pressures and temperatures. The reinforcement of polyesters with cellulosic fibers has been widely reported such as jute, sisal, coir, banana-cotton, straw, pineapple leaf and cotton-kapok are some of the promising systems (Roe & Ansell, 1985; De Alburquerque et al., 1999; Pal et al., 1988; Owolabi et al., 1985; Satyanarayana et al., 1983; White & Ansell, 1983; Devi et al., 1997; Mwaikambo & Bisanda, 1999).

Incorporation of these fibers in polymers and other matrices can be one such use. Increased utilization of these may solve pollution problems as they go to waste in rural and other areas. 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. There are, however, a number of limitations, including cost factor and their performance over a long time duration, which need further research (Satyanarayana et al., 1990).

When natural fiber-reinforced plastics are subjected, at the end of their life cycle, to combustion process or landfill, the released amount of carbon dioxide of the fibers is neutral with respect to the assimilated amount during their growth (Wambua et al., 2003). The abrasive nature of fiber is much lower which leads to advantages in regard to technical process and recycling process of the composite materials in general. Natural fiber-reinforced plastics, by using biodegradable polymers as matrices, are the most environmental friendly materials, which can be composed at the end of their life cycle. Natural fiber composites are used in place of glass mostly in non-structural applications. A number of automotive components previously made with glass fiber composites are now being manufactured using environmentally friendly composites (Larbig et al., 1998). Although natural fibers and their composites are environmental friendly and renewable (unlike traditional sources of energy, i.e., coal, oil and gas), these have several disadvantages. They have: poor wetability, incompatibility with some polymeric matrices and high moisture absorption (Vazquez et al., 1999). Composite materials made with the use of unmodified plant fibers frequently exhibit unsatisfactory mechanical properties. To overcome this, in many cases, a surface treatment or compatibilizing agents need to be used prior to composite fabrication. The properties can be improved both by physical treatments (cold plasma treatment, corona treatment) and chemical treatments (maleic anhydride organosilanes, isocyanates, sodium hydroxide permanganate and peroxide) (Luo & Netravali, 1999). Mechanical properties of natural fibers are much lower than those of glass fibers but their specific properties, especially stiffness, are comparable to the glass fibers (Raczs & Hargitai, 2000). Due to this scenario, natural fiber such as palm fiber was considered in this research.

One of the major problems associated with the use of natural fibers in composites is their high moisture sensitivity leading to severe reduction of mechanical properties and delamination. The reduction in mechanical properties may be due to poor interfacial bonding between resin matrices and fibers. It is therefore necessary to modify the fiber surface to render it more hydrophobic and also more compatible with resin matrices. An effective method of chemical modification of natural fibers is graft copolymerisation. The resulting co-polymer displays the characteristic properties of both fibrous cellulose and grafted polymer (Dale & O'Dell, 1999; Patil et al., 2000). One of the most explored chemical modifications is the acetylation-esterification of cellulose-OH, by reaction with acetic anhydride. This reaction reduces hydrophilicity and swelling of lignocellulosics and their composites (Bledzki & Gassan, 1998).

Many researchers were reported on saturated polyester reinforced with lignocellulosic fibers obtained from sisal (Pal et al., (1988), kenaf (Sharifah et al., 2005), banana (Satyanarayana et al., 1983), palm tree (Hamid et al., 2006), cotton (Mwaikambo et al., 1999), jute (Roe & Ansell, 1985), straw (White & Ansell,1983), coir (Owolabi et al., 1985). and pineapple leaf (Devi at al., 1997).

In this study, UP/PF composites and treated palm fiber composites was developed and the mechanical properties such as tensile and flexural properties, morphology and FTIR analysis was reported. For treated fiber; oleoyl chloride, DMAP and seawater was chosen because it is inexpensive and effective. The aim of this work is to study the surface treatment of palm fiber with a reagent and seawater able to react with hydroxyl groups of cellulose.

## 1.2 Research Objectives

This research was to carry out a study on the potential of palm fiber as a filler for unsaturated polyester. The objectives of this research are;

1) To study the effect of filler loading and particle size on the properties of palm fiber filled unsaturated polyester composites.
2) To study the effect of oleoyl chloride of palm fiber on properties of UP/PF composites.
3) To study the effect of 4-Dimethylaminopyridine (DMAP) of palm fiber on properties of UP/PF composites.
4) To study the effect treatment with seawater of palm fiber on properties of UP/PF composites.

# CHAPTER 2

# LITERATURE REVIEW

## 2.1 Polymer Composites

### 2.1.1 Definition

The term composite can be defined in various ways. A composite can simply be defined as a combination of two or more dissimilar materials having a distinct interface between them such that the properties of the resulting material are superior to the individual constituting components. Composites materials can also be defined as two or more dissimilar materials that are intimately bonded to form integrated structure (Bhargava, 2005). Composites are produced when two or more materials or phases are used together to give a combination of properties that cannot be attained otherwise. Composites materials maybe selected to give unusual combination of stiffness, strength, weight, high-temperature performance, corrosion, resistance, hardness, or conductivity (Meyers & Chawla, 1998; Harper, 2001).

Peply, (1987) and Herakovich, (1998) has described that a composite is defined as a combination of two or more components differing in form or composition on a macroscale with two or more distinct phases having recognizable interfaces between them. Proper combination of materials into composites gives rise to properties which