

**COMPOSITES FROM POLYPROPYLENE (PP) REINFORCED WITH
OIL PALM EMPTY FRUIT BUNCH (OPEFB) FIBRES**

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JUDUL : **COMPOSITE FROM POLYPROPYLENE (PP) REINFORCED WITH OIL PALM EMPTY FRUIT BUNCH (OPEFB) FIBRE**

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PALM EMPTY FRUIT BUNCH (OPEFB) FIBRE**

NORA'ASHEERA BINTI MOHD NAZIF

**A thesis submitted in fulfillment of the
requirement for the award of the degree of
Bachelor of Chemical Engineering**

**Faculty of Chemical and Natural Resources Engineering
University Malaysia Pahang**

MAY 2011

“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 the degree of Bachelor of Chemical Engineering”

Signature : _____
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Date : 2 MAY 2011

I declare that this thesis entitled '*Composites from Polypropylene (PP) Reinforced With Oil Palm Empty Fruit Bunch (OPEFB) Fibre*' is the result of my own research except as cited in references. The thesis has not been accepted for any degree and it is not concurrently submitted in candidature of any degree.

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With the name of Allah, the most gracious the most merciful...

“

*Allah! There is no God but Him;
The living, the Self-subsisting, the Eternal.
No slumber can seize Him, nor sleep.
All things in heaven and earth are His.
Who could intercede in His presence without His permission?
He knows what appears in front of and behind His creatures.
Nor can they encompass any knowledge of Him except what he will.
His throne extends over the heavens and the earth,
and He feels no fatigue in guarding and preserving them,
for He is the Highest and Most Exalted.*

”

(Surah Al-Baqarah: 255)

To my beloved parents:

Mr. Mohd Nazif Bin Mohd Esa and Mrs. Safiah Binti Kassim

Who gave me everlasting inspiration, never-ending encouragements and priceless support towards the success of this study.

To my best friend:

Nor Azlina and Nur Asilah

Who gave me everlasting support and help towards the success in my Undergraduate Research Project

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ABSTRACT

The use of natural fibers obtained from plants and trees are reinforcing materials has attracted many researcher to widen their applications. Natural fibers are low cost, low density, have high specific properties, biodegradable and non-abrasive. Oil palm fiber (OPF) can be obtained directly from natural resource; it is cheap and also has advantages due to its renewable nature, low cost and easy viability. The study aims to give the improvement to composite from plastic reinforced with oil palm empty fruit bunch along with to determine the mechanical performances of the composite. In this research, the sample will through the alkali treatment using sodium hydroxide (NaOH) follow by hydrogen peroxide (H_2O_2) and the last is using acetic acid ($C_2H_4O_2$). NaOH is use as a solvent extraction and it will break the lignin bonding. Remove lignin will decrease the absorption of water in composite. Addition different type plasticizer and MAPP will give better improvement to mechanical performance to composite. From the analysis that have done, compatibilized and plasticizers systems an enhanced fibre dispersion and interfacial adhesion has been observed, supporting the occurrence of effective interactions between the functional groups on the copolymer chains and the polar groups of the fibres, as demonstrated by FT-IR analysis. Besides that, the density and melt flow index of composites with different plasticizer was determined and from the all test that have been done it showed that polyethylene glycol is the good plasticizers compared to sorbitol and glycerol.

ABSTRAK

Gentian asli didapati daripada tumbuhan dan pokok, ia digunakan sebagai bahan penguat yang kini menarik perhatian penyelidik untuk meluaskan lagi aplikasinya. Gentian asli mempunyai ketumpatan yang rendah, harga yang murah, memiliki sifat-sifat yang khusus, boleh diurai secara semulajadi dan tidak kasar. Gentian kelapa sawit boleh didapati terus dari sumber semulajadi dan mempunyai kelebihan seperti sumber semulajadi yang boleh diperbaharui dan mudah didapati. Matlamat kajian ini adalah untuk membuat penambahbaikan pada komposit plastik yang diperkuatkan dengan gentian tandan buah kelapa sawit kosong,(OPEFB). Selain itu, untuk menentukan keupayaan mekanikal bagi komposit. Dalam kajian ini, sampel akan melalui rawatan berkali menggunakan Natrium Hydroxide,(NaOH), diikuti oleh proses pemutihan menggunakan Hydrogen Peroxide,(H₂O₂) dan yang terakhir ialah proses peneutralan menggunakan asetik asid,(C₂H₄O₂). NaOH digunakan sebagai pelarut pengestrakan and ia akan memutuskan ikatan lignin. Membuang lignin dari gentian akan mengurangkan penyerapan air dalam komposit. Penambahan plasticizer yang berbeza dan MAPP akan meningkatkan keupayaan mekanikal bagi komposit. Daripada analisis yang telah dibuat MAPP dan plasticizer sistem akan menambah penyebaran dan pelekatan akan diperhatikan. FTIR analisis akan menunjukkan kumpulan berfungsi yang wujud dalam komposit manakala ketumpatan dan MFI bagi komposit dengan plasticizer yang berbeza juga ditentukan. Daripada semua analisis yang telah dilakukan, ia menunjukkan Polyethylene Glycol adalah plasticizer yang terbaik jika dibandingkan dengan Sorbitol dan Glycerol.

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LIST OF ABBREVIATIONS

ABS	Acrylonitrile Butadiene Styrene
ASTM	American Standard Testing Materials
EFB	Empty Fruit Bunch
EPDM	Ethylene Propylene Diene Monomer
FTIR	Fourier Transform Infrared
FRP	Fibre-Reinforced Plastic
GFRP	Glass Fibre Reinforced Polymer
H ₂ O ₂	Hydrogen Peroxide
HDPE	High Density Polyethylene
LDPE	Low Density Polyethylene
MFI	Melt Flow Index
MFR	Melt Flow Rate
MTBE	Methyl Tertiary Butyl Ether
MgOH	Magnesium hydroxide
MAPP	Maleic Anhydride Polypropylene
NaOH	Sodium Hydroxide
OPT	Oil Palm Trunks
OPF	Oil Palm Fronds
OPEFB	Oil Palm Empty Fruit Bunch
PPF	Palm Pressed Fibres
PP	Polypropylene Plastic
PVC	Poly Vinyl Chloride
YM	Young's Modulus

LIST OF SYMBOLS

°C	-	Degree Celcius
%	-	Percentage
wt. %	-	Weight percentage
mm		Milimeter
µm		Micrometer
Tg		Glass Transition
Rpm		Rate Per Minute
cal./cc		Calories Per Centimeter Cube

LIST OF APPENDIX**APPENDIX****TITLE**

A	FTIR analysis
B	Density Analysis

CHAPTER 1

INTRODUCTION

1.1 Background of study

Over the last decade, polymers reinforced with natural fibres composites have attracted attention from the academic world and various industries. The rapid growth in the consumption of plastic products, persistence of plastics in the environment, the shortage of landfill space, the depletion of petroleum resources and entrapment by the ingestion of packaging plastic by marine and land animals have spurred efforts to look for better alternatives (Khalid *et al.*, 2008). It has relatively high strength and stiffness and has no skin irritations effects (Oksman *et al.*, 2003).

Composite are combinations of two or more materials with different composition or form. The constituent retain their identities in a composite and do not dissolve or merge, but act together. A composite may have a ceramic, metallic or polymeric (thermoset or thermoplastic) matrix. The fibres can also be ceramic, metallic or polymeric, however, a more common classification relates to whether they are synthetic (e.g. glass fibre, carbon fibre, Kevlar fibre) or natural (wood fibre, hemp fibre, flax fibre, oil palm fibre, jute fibre, etc). Therefore, the number and variety of composites available

are very large. Fibre-reinforced composite materials commonly consist of fibres of high strength and Young's modulus embedded in, or bonded to, a matrix with a distinct interface between them. In general, the fibres are the principal load carrying members, while the surrounding matrix holds them in the desired location and orientation, acting as a load transfer medium between them (Dalour, 2007).

Natural fibres are gaining importance and increasingly in demand across a wide range of polymer-composite materials and in the manufacture of bio-composites for various applications due to their desirable properties (Bledski *et al.*, 1999). They originate from plants, crops, animals, agro waste or other natural sources that are natural, renewable and biodegradable after their end use. Global environmental concerns such as climate change and sustainability are encouraging development of totally green materials that can deliver eco-social system change. Synthetic fibres are discouraged, while conversely natural fibres are the best available alternative reinforcement or fillers to make polymer-composite materials partially biodegradable. Currently the major thrust of on-going research in the development of bio-composite is on flax, hemp, jute, coir, palm and other natural fibres. The properties of natural fibre depend on factor like fibre length, maturity as well as processing method adopted for the extraction of fibres. Properties such as density, electrical resistivity, ultimate tensile strength, Young's modulus and many others, are related to the internal structure and chemical composition of fibres (Mohanty *et al.*, 2001). For now days, there has been a renewed interest in using natural fibres as reinforcement materials in the plastics for industry. More recently, critical discussion about the preservation of natural resources and recycling has led to further interest concerning natural materials with the focus on renewable raw materials. As mentioned earlier, there is a wide variety of different natural fibres which can be applied as reinforcement.

Wood fibre is the most widely used lignocellulosic fibre for reinforcing plastics. It can be obtained from variety of process, namely: thermomechanical, soda, sulphite, sulphate (Kraft) and semi-chemical pulping. Wood fibre composite gives several advantages over synthetic fibre composites such as: low density, improved acoustic

properties, favorable processing properties (e.g. low wear on tools), occupational health benefits compared to glass fibres as well as positive effects on environment and agriculture with comparable mechanical properties. Lignocelluloses are the main structural component of wood plant and non wood plant such as palm oil empty fruit bunch. The chemical properties of the components of lignocellulosics make them a substrate of enormous biotechnological value (Howard *et al.*, 2003). Malaysia is the largest exporter of palm oil in the world, the main source of lignocelluloses comes from palm oil empty fruit bunch as a palm oil residue. Now days, no more technology obtained to dispose this residue and the lack of disposal place for this waste. Because of that, many palm oil mill use incineration process to dispose this residue. Effect of that, can contribute to the environmental pollution. Many researchers were run in order to convert the palm oil empty fruit bunch as waste to become various value added product.

Oil palm empty fruit bunch (OPEFB) is one of the lignocellulosic materials, which has great relevance to Malaysia, as a large quantity of the biomass is generated by oil palm industries (Bakar *et al.*, 2005). The fresh oil palm fruit bunch contains about 21% palm oil, 6-7% palm kernel, 14-15% fibre, 6-7% shell and 23% empty fruit bunch (Umikalsom *et al.*, 1997). The incorporation of empty fruit bunch (EFB) into polymers to obtain cost reduction and reinforcement has been reported by various workers. Rozman *et al.*, (2001) investigated the mechanical properties of polypropylene/EFB composites and found that EFB has high tensile modulus, but low tensile strength. Abdul *et al.*, (2001) reported that modified EFB has improved the mechanical properties and water resistance of the polyester/EFB composites. Ishak *et al.*, (1998) found that the tensile modulus of high density polyethylene (HDPE)/EFB showed an increase compared to neat HDPE, whereas tensile strength and impact strength decreased.

Oil palm industries generate at least 30 million tons of lignocellulosic biomass annually in the form of oil palm trunks (OPT), empty fruit bunches (EFB), oil palm fronds (OPF) and palm pressed fibres (PPF). At present, the biomass is either left to rot in the plantations to provide organic nutrients to the oil palm trees (mulching) or used as solid fuel in the boilers to generate steam and electricity at the mills. An oil palm

plantation produces about 55 tones ha⁻¹ yr⁻¹ of total dry matter in the form of fibrous biomass as compared to only about 5.5 tones ha⁻¹ yr⁻¹ of palm oil and palm kernel oil. The fibrous biomass is yet to be commercially exploited. Technology development in the industry is still focused on process development and improvement rather than creating and inventing newer products for value-added application. The need for materials not harmful to the body but having appropriate properties has increased due to a lack of resources and increasing environmental pollution. Thus, composite prepared from recycled materials are actively being sought after. Many synthetic polymeric materials are produced by combining with various reinforcing fillers to improve their mechanical properties and obtain the desired properties. Among the reinforcing fillers, active research is under way concerning the use of lignocellulosic materials, which are among the most environmentally friendly agro waste, as a substitute for synthetic materials. Lignocellulosic materials offer many environmental benefits when used as reinforcing fillers for plastics, including their making the final product lightweight, decreasing the erosion of the manufacturing machinery, low cost, biodegradability, and absence of production of residue or toxic by-products when burnt. In Malaysia, the construction of early roads and bridges was a necessity to facilitate the movements of peoples and goods. There are now more than 10,000 bridges in Malaysia of various sizes and forms, out of which approximately 2,500 are located on federal roads. The early bridges were mainly made of steel in the form of trusses, arches and beam, which was a subsequent product out of the 18th century Industrial Age in England. Concrete and steel have been replaced by other materials such as FRP, GFRP and others fibre polymer materials. This offers a good opportunity for fibre reinforced polymer composite to be used for maintenance of old bridge structures.

1.2 Problem Statements

In today's world energy scenario, petroleum resources are becoming depleted and demand is increasing along with increase in their hazardous effect on the environment. Therefore, there is a growing awareness for an integrated approach considering global environmental factors, sustainability, industrial ecology and engineering for developing alternative materials and products. Bio-composite are one of the new alternative structural materials that are developed by combining natural fiber and petrochemical based polymer. The more usage of natural fiber makes the product less hazardous and reduces usage of petroleum-based polymers. Because of that, the purpose of this research can pressure on industry to use environmental friendly and sustainable materials to replace materials that can contributing to petroleum hydrocarbon pollution.

Palm oil empty fruit bunch is the one of palm oil mill residue 'waste'. Malaysia is one of the palm oil producers around the world. But the residue of the palm oil has still not been completely used. Palm oil empty fruit bunch was disposed by incineration process and directly contributing to the environmental pollution. In this research, the purpose is to convert the waste palm oil empty fruit bunch to the natural fibre through a several treatment and then after treatment it will through a several process to become a new bio-composite. From that, it can helping Malaysia economic to provide a lower cost raw material to replace the expensive raw material like glass fibre and carbon fibre.

Current use of plastics in daily life is very important. Production of plastic by industry also increase based on increasing the usage of plastics. But at the same time plastic can contribute to pollution, particularly plastic which unbiodegradable will cause soil pollution. The purpose of this research is natural fibre is added to the thermoplastic to improve the structural of plastic from unbiodegradable to biodegradable plastic. Mechanical properties of natural fibre are biodegradable. Addition of natural fibre to thermoplastics can reduce of lignin hence increase the water absorption in soil so from that the plastic can biodegrade faster and reduce the pollution to soil.

1.3 Research Objectives

- I. To produce composite from polypropylene plastic (PP) reinforced with oil palm empty fruit bunch (OPEFB) fibre with different type of plasticizer.
- II. To determine the mechanical performance of oil palm empty fruit bunch reinforced thermoplastic

1.4 Research Scope

- I. For alkali treatment of fibre, NaOH will be used for oil palm empty fruit bunch to improve the interfacial bonding, increase fibre surface charge and increasing the Young's Modulus (YM). Alkali treatment also will decrease the residual lignin content that cause improving composite strength.
- II. To study about the effectiveness using plasticizer with MAPP between oil palm empty fruit bunch fibre. Plasticizer work by embedding themselves between the chain of polymers, spacing them apart (increasing the free volume) and thus significantly lowering the glass transition temperature for the plastic and making it softer.

1.5 Rational And Significance

- I. Reduced environmental pollution
- II. Environmental friendly product will produced
- III. Create a new composite from natural resources of oil palm EFB
- IV. Improve the mechanical performance

CHAPTER 2

LITERATURE REVIEW

2.1 Bio-composite

By definition, bio-composite are composite materials made from natural fibres and petroleum-derived non-biodegradable polymers like polypropylene, polyethylene and epoxies or biopolymer and synthetic fibres such as glass and carbon also come under bio-composites. Bio-composites derived from plant-derived fibre (natural/bio-fibre) and crop/bio-derived plastics (biopolymer/bio-plastic) are likely to be more eco-friendly, and such bio-composites are sometimes termed “green composites”. (Mohanty *et al.*, 2005)

Bio-composites consist of reinforcing bio-fibre and matrix polymer systems. Since the bio-fibre is biodegradable and traditional thermoplastics (like polypropylene) and thermosets (like unsaturated polyester) are non-biodegradable, bio-composites from such fibre-reinforced polymers are classified as the “partly eco-friendly” types. If the matrix resin /polymer comes from a renewable resource-based bio-plastic, the bio-fibre reinforced biopolymer composites would come under the “eco-friendly” type (refer figure 2.1). Two or more bio-fibres in combination with a polymer matrix result in “hybrid” bio-composites.

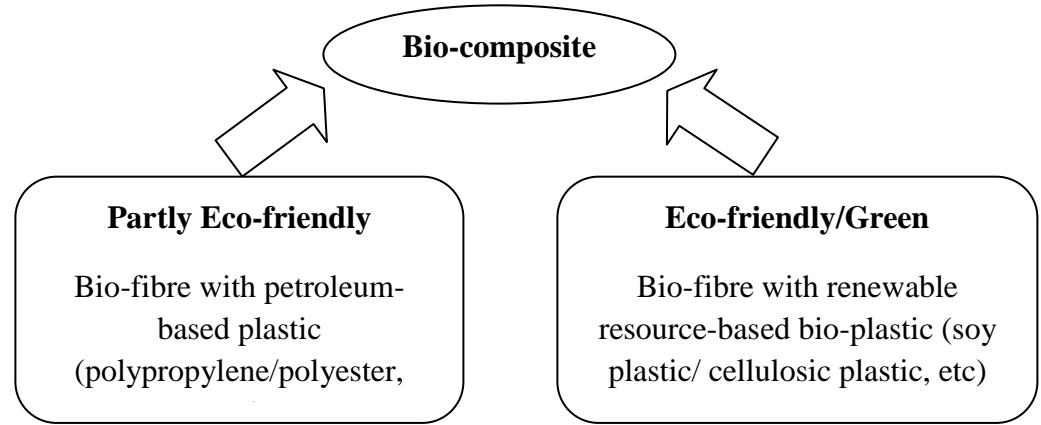


Figure 2.1: Classification of bio-composites

For this study, the bio-composites used for external reinforcement are from the “partly eco-friendly” type. The type of bio-composite used is oil palm empty fruit bunch fibre reinforced polymer thermoset composite.

2.1 Oil Palm Empty Fruit Bunch (OPEFB) Fibre

Oil palm (*Elais guineensis*) is widely planted commercially due to its oil producing fruit. Its two main products are palm oil and palm kernel oil. However, in the process of obtaining these products, it will leave various agricultural waste such as oil palm frond (OPF), trunks, mesocarp, empty fruit bunch (EFB), palm oil mill effluent, palm kernel cake and palm press.

The main components of oil palm fibre are cellulose and lignin (Bismarck *et al.*, 2005). The oil palm fibres are very porous and their diameters greatly vary. This categories oil palm fibre as lignocellulosic materials. These entire factors affect the mechanical properties of the fibre. Generally, lignocellulosic materials are lightweight and very ductile.

A fruit bunch is where the palm fruit is embedded. An empty fruit bunch is obtained when the oil is extracted from the palm fruit leaving a bunch of fibres. According to SIRIM Berhad, Malaysia 23% of empty fruit bunch (EFB) is obtained per ton of fresh fruit bunch (FFB) processed in an oil palm mill. Therefore, EFB is being sold commercially to produce mattresses, car seats, insulation, composite panel products and particle boards.

Table 2.1 Comparison of properties between different oil palm fibres

Properties	EFB	OPF	Oil Palm Trunk
Fibre length (mm)	0.67	1.03	1.37
Width of fibre (μm)	12.50	15.10	20.50
Width of lumen (μm)	7.90	8.20	17.60
Runkel ratio	0.59	0.84	0.26
Area of fibre (μm^2)	75.60	126.20	86.70

2.2 Extrusion Compounding Process

Extrusion is an important process which is used to manufacture products of good dimensional uniformity and quality at economic rates. It basically involves continuously melting and mixing of polymer, shaping a fluid polymer through the orifice of a suitable tool (die), and subsequently solidifying into a product (Strong, 2006). In the case of thermoplastics, the feed material in powder or pellet form, is heated to a fluid state and pumped into the die through a screw extruder and then while exiting from die, solidified by cooling.

In order for emerging extrudates to maintain their shape until they solidify, extrusion grades tend to have relatively high molecular weight associated with high viscosity and melt strength (Charrier, 1991). (Oladipo *et al.*, 1999) investigated aspen

wood fibre/HDPE composite and the components were feed at pre-determined mass flow rates, based on the desired wood fibre mass fraction in the composite, into an extruder having a 28 mm corotating twin screws operating at temperature of 150°C and a screw rotational speed of 100 rpm. This temperature ensured that while the polymer was fully melted (melting point is 120-130°C), the wood fibres were not burned. Short flax fibre-reinforced compounds can be made by mixing flax fibres with a thermoplastic matrix at elevated temperature in an extruder or a kneader (Haeiette *et al.*, 2005). It was reported that keratin feather fibre and HDPE are compounded best at 205°C at 75 rpm in a Brabendar mixing head (Barone *at al.*, 2005). (Siatong, 2006) investigated the extrusion parameters, their effect on bio-composite properties and determined optimum extrusion parameters (temperature = 75-118.1-128.1-138.1-148.1°C and screw speed = 125.56 rpm). It was also reported temperature and screw speed affect the fibre degradation.

2.3 Injection Molding

Injection molding is the most widely used polymeric fabrication process. A large force must be used to inject polymer into the hollow mould cavity. More melt must be packed into the mould during solidification to avoid shrinkage in the mould. Identical parts are produced through a cycle process involving the melting of pellet or powder resin followed by the injection of the polymer melt into the hollow mould cavity under higher pressure. Injection molding can be used to form a wide variety of products, for both thermosets and plastics. Complexity is virtually unlimited, sizes can vary and excellent tolerance is also possible. With respect to reinforced composites, only randomly oriented short fibres can be used in this process, the orientation of the fibres in the composite are determined by the flow of the composite melt during filling of the mould cavity. The incorporation of short fibres in an injection molded polymer increases the strength, modulus, impact, creep and heat resistance while decreasing the failure strain. Extrusion is used prior to injection molding when mixing of a thermoplastic, filler and additive is required. Pellets of the compounded mix are then fed into the hoper are

melted, the liquid melt is then injected into the closed mould cavity with a force. The melt is then allowed to cool and solidify before the mould is opened and the final product removed (Dalour, 2007).

2.4 Polypropylene

Polypropylene (PP) is a thermoplastic polymer, can be made by addition polymerization process. PP has a melting point of about 165°C, glass transition temperature (T_g) about -100°C. Most commercial PP has an intermediate level of crystallinity, between 40-60%. Depending on their crystallinity, density range from 0.85 g/cm³ to 0.95 g/cm³. Its Young's modulus is also intermediate. Although, it is less tough than high density polyethylene (HDPE) and less flexible than low density polyethylene (LDPE), it is much more brittle than HDPE. This allows PP to be used as a replacement for engineering plastics, such as acrylonitrile butadiene styrene (ABS). PP can be made translucent when uncolored but not completely transparent as polystyrene or acrylic. PP has very good resistance to fatigue (Dalour, 2007). There is a wide variety of application of PP including food packaging, plastic parts and reusable containers of various types, thermal pants and shirts made for the military, laboratory equipment, loudspeakers, automotive components and polymer banknotes (Dalour, 2007).

2.6 Plasticizer

As defined by ASTM, a plasticizer is a substance incorporated into a plastic or elastomer to increase its flexibility, workability or distensibility. In its simplest concept, it is a high-boiling organic solvent which, when added to a rigid substance, imparts flexibility. Plasticizers include a large variety of organic liquids e.g., petroleum fractions, coal tar distillates, animal fats, plant extracts, etc., and reacted products made of those materials. Ester plasticizers, the subject of this paper, are the latter. Elastomer and plastic polymers may be tough, dry, or rigid materials which, for many applications, have a need for plasticizers. A plasticizer, among other contributions, will reduce the

melt viscosity, lower the temperature of a second order glass transition, or lower the elastic modulus of a polymer.

2.6.1 Function of Plasticizer

Plasticizers are polymer modifiers as are all the other ingredients included for the formation of an elastomer compound. Plasticizers may be thought of according to their function in a compound or by their type. Some of those classifications might be Internal, External, Chemical, Physical, Esters, Oils, Primary, Secondary, etc. Internal plasticizers include flexible monomers (soft segments) incorporated regularly or irregularly between inflexible monomers (hard segments) of a polymer chain. Flexible polymers may be added to rigid polymers, e. g. Nitrile rubber to PVC, or grafted as side chains that reduce crystallinity and glass transition through reduction of intermolecular forces. External plasticizers are materials that interact physically with the elastomer, but are not chemically reacted with the polymer (Darby *et al.*, 1968). Solvent and non-solvent are two distinct types of external plasticizers. Common esters and polymeric polyesters are both External and Physical plasticizers. Physical plasticizers may have some weak attraction to the polymer such as through hydrogen bonding or Van der Waals forces but, as with External plasticizers, do not chemically react with the elastomer. An exception to this can occur under the right conditions provided one of the reactants used to make the plasticizer, after the esterification reaction, retained a reactive group. A potential problem arises there, however, as materials reacted with the polymer molecules will make the polymer molecule larger, thus, less flexible. Chemical plasticizers attack, thereby reducing the molecular weight of the elastomer chain.

Under suitable conditions, esters are solvents for amorphous polymers, i.e. the polymer would eventually dissolve in plasticizer. With crystalline or semi-crystalline polymer, some plasticizers may enter the crystalline (ordered) and the amorphous (disordered) regions. Primary and secondary plasticizers are terms related to compatibility with polymers (compatibility is discussed later). Primary plasticizers enter

the polymer systems first. Plasticizers entering the crystalline regions of crystalline or semi-crystalline polymers are referred to as primary. If the amorphous regions of those polymers are penetrated, the plasticizer may be considered a solvent type. Secondaries are plasticizers that would not penetrate the original polymer system and are used as diluents for primary plasticizers. External plasticization permits the greatest latitude in formulating for specific compound properties, and may be the least expensive route for that need. In the instance of PVC, esters are quite compatible as primaries (Nakomura *et al.*, 1975). Petroleum oils are not useful as primaries. However, by incorporating an ester as a primary the desired properties of softness, low temperature, processing may be achieved. Then a limited amount of petroleum oil may be incorporated, and frequently is, strictly to reduce compound cost. The petroleum oil is compatible with the primary plasticizer and thus stays in the PVC compound. On the elastomer side, esters are essentially incompatible with EPDM polymers. However, by incorporating petroleum oil with EPDM, esters may be included in those recipes successfully. That purpose is to achieve properties the petroleum oils will not provide to that elastomer, such as original compound low temperature, which accompanies high green strength EPDM (Nakomura *et al.*, 1975).

Plasticizers have two distinctly different uses. Table 2.2 shows their different functions and in general the affects as 1) process aids and 2) property modifiers.

Table 2.2: Physical function of plasticizers (depending upon choice of material)

1) As processing aid	<ul style="list-style-type: none"> ▪ Lower the processing temperature ▪ Increase lubricity ▪ Reduce mold sticking ▪ Reduce mixer sticking ▪ Improve flow out ▪ Improve wetting ▪ Reduce nerve ▪ Increase tackiness
----------------------	--

2) As elastomer property modifiers	<ul style="list-style-type: none"> ▪ Soften the polymer ▪ Lower the modulus ▪ Lower the tensile strength ▪ Increase elongation ▪ Increase flexibility ▪ Lower glass transition ▪ Increase tear length ▪ Increase the temperature ▪ Range of usefulness ▪ Increase cohesion ▪ Modify frictional character ▪ Improve surface appearance ▪ Decrease static charge
------------------------------------	---

If want a process aid to reduce the temperature of processing without affecting the softening temperature of the final product. The softening temperature will be the upper limit of usefulness of that final product. A plasticizer as a final property modifier will ideally lower the glass transition (T_g) temperature or softening temperature without lowering the flow temperature. This represents a broadening of the temperature range of usefulness desired from the plasticizer. The softening temperature will now be the lower limit of usefulness of the finished product. As an example, apply that reasoning to rubber tires used in arctic regions. Plasticizer added to the recipe lowers the compound T_g providing the compound with improved low temperature for the cold climate. This allows the tire to have a colder softening temperature, but does not alter the flow temperature of the compound (Hall Company, 2000).

2.6.2 Compatability of Plasticizer

If plasticizer-polymer compatibility are correct, the two materials will form a homogeneous mixture during processing and once cured, the plasticizer will remain in the compound upon cooling and resting at low temperature. From a practical standpoint, it is only necessary that the compatibility be observed at a plasticizer quantity suitable to produce the desired effect. To achieve a high degree of plasticizer compatibility, it is generally necessary that the plasticizer and polymer have approximately the same polarity. Previously, plasticizers were described as solvents of moderately high molecular weight and low volatility. Their ability to achieve and maintain compatibility with the polymer depends on the same factors that govern the behavior of simpler organic solvents and solutes. The thermodynamic basis for such interactions is expressed by Hildebrand solubility parameters, defined as the square root of cohesive energy density. Plasticizer compatibility with an amorphous polymer (or the amorphous phase of a partially crystalline polymer), δ , normally requires values that do not differ by more than ± 1.5 (cal./cc) (Doolittle et al.,1954). Solubility parameters for both polymers and plasticizers are conveniently calculated by the additive method of Small, who derived individual parameters for various atoms and groups in the molecules. Compilations of molar attraction constants, commonly known as Small's constants, are given in many handbooks. Figure 2.2 below show the lists polymers and plasticizers from high to low polarity.

POLYMER	<div style="border: 1px solid black; padding: 5px; display: inline-block;"> H I G H </div>		PLASTICIZER CLASS
Nylon 6/6			Aromatic sulfonamide
Nylon 6			Aromatic Phosphate Esters
Cellulose Acetate			Alkyl Phosphate Esters
Polyurethane			Dialkylether Aromatic Esters
Nitrocellulose			Polymeric Plasticizers
Epoxy			Dialkylether Diesters
Polycarbonate			Polyglycol Diesters
Acrylic (PMMA)			
Polyvinyl Acetate			Tricarboxylic Esters
Acrylate Elastomers			
Polyvinyl Butyral			Polyester Resins
Epichlorohydrin			
Chlorosulfonated Polyethylene			Aromatic Diesters
Polyvinyl Chloride			Aromatic Triesters (Trimellitates)
Cellulose Acetate Butyrate			
Polystyrene			
Polychloroprene			Aliphatic Diesters
Chlorinated Polyethylene			
Highly Saturated Nitrile			Epoxidized Esters
SBR			
Polybutadiene			Chlorinated Hydrocarbons
Natural Rubber			Aromatic Oils
Halogenated Butyl			Alkylether Monoesters
EPDM			Naphthenic Oils
EPR			
Butyl			Alkyl Monoesters
Fluorinated Polymers			
Silicone			Paraffinic Oils
		<div style="border: 1px solid black; padding: 5px; display: inline-block;"> L O W </div>	
			Silicone Oils

Figure 2.2: Lists polymers and plasticizers from high to low polarity.

CHAPTER 3

METHODOLOGY

3.1 Introduction

This study is to determine the effect using different plasticizer and without plasticizers on the chemical and mechanical properties of OPEFB fibre reinforced with PP composite. Initially composite were produced with 40wt% fibre content, MAPP used was 1:10 fibre as compatibilizer/coupling agent, 10wt% MgOH as flame retardant, 3wt% plasticizer and the remain from total composite is PP. Composite was produced have several step to follow. Firstly, production of untreated fibre and followed by alkaline peroxide treatment using NaOH called as alkaline treatment. After that, fibre was treated using H₂O₂ as bleaching process and the last treatment is neutralizing process using acetic acid, finally treated fibre was produced. The next process is compounding process that is all composition was mixed together using plastic mixer to make the composition homogenously mixed. The final process is to prepare sample through the molding process using hot press. This process is to make the sample in shape using the standard mold. The mechanical and chemical properties were assessed using fourier transform infrared (FTIR) spectroscopy, melt flow index (MFI) and density using gas pycnometer.

3.2 Materials

In order to produce composite from plastic (PP) reinforced with oil palm empty fruit bunch fibre (OPEFB), the main raw material needed are virgin polypropylene (PP) and oil palm empty fruit bunch (OPEFB). Oil palm empty fruit bunch (OPEFB) was supplied by Felda Palm Industries Berhad, Felda Lepar Hilir 3 and virgin polypropylene (PP) with density 0.9g/cm^3 and melt flow index (MFI) 40 was supplied by MTBE (M) Berhad. Besides that, Maleic anhydride grafted polypropylene (MAPP) as a compatibilizer, Magnesium hydroxide (MgOH) as a flame retardant and three different plasticizers are sorbitol, glycerol and polyethylene glycol was used as an additive to the composite. For the alkaline peroxide treatment the chemical used were sodium hydroxide (NaOH), hydrogen peroxide (H_2O_2) and acetic acid ($\text{C}_2\text{H}_4\text{O}_2$).

3.3 Experimental Procedures

3.3.1 Production of Untreated Fibre

Preparation of untreated fibre from oil palm empty fruit bunch agro waste was prepared in two stages, there is physical treatment for first stage and second stage is chemical treatment. For the first stage, OPEFB fibre was dried minimum for three days under ultra violet light or sun light. After that, OPEFB fibre was cut using hammer mill or crusher machine and it was passed through $500\mu\text{m}$ sieve to obtain uniform size of OPEFB fibre. Besides that, sieve process is used to separate between fibre and ash. Next untreated OPEFB fibre was soaked with hot water at 70°C at least for 4 hour to 6 hour to removes the impurities and large particles at the surface of untreated fibre. Then untreated OPEFB fibre was dried at 70°C in the oven for 1 day. The flow chart of production of untreated OPEFB fibre is illustrated on figure 3.1.

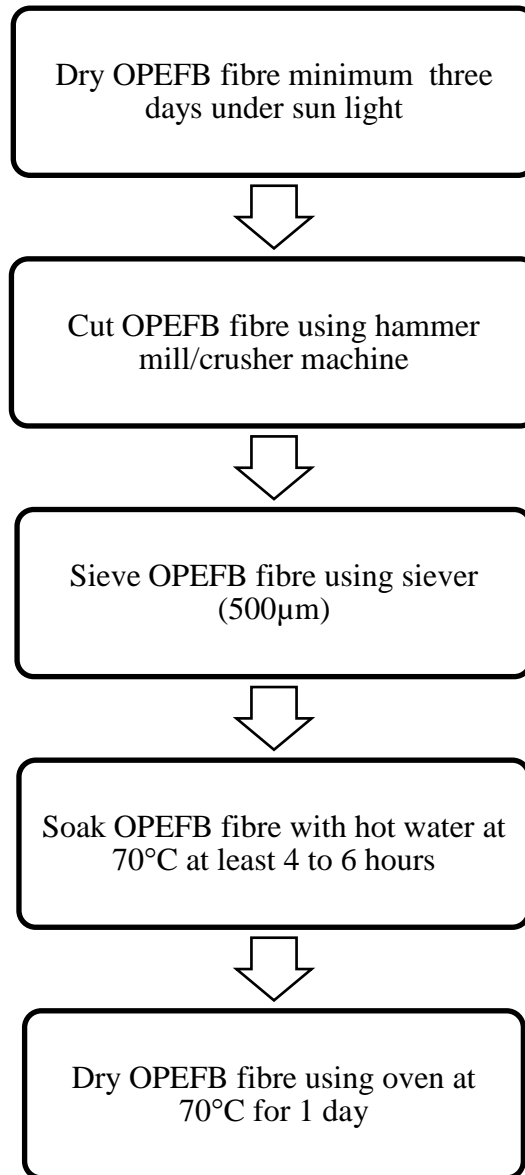


Figure 3.1: Production of untreated OPEFB fibers

3.3.2 Production of Treated Fibre

Preparation of treated fibres involved chemical treatment to fibre that is second stages in production of OPEFB fibre. It is also called alkaline peroxide treatment. In this treatment, fracture of fibres was modified follow several treatment. The treatments are alkaline treatment using NaOH, bleaching process with H₂O₂ and neutralizing process using acetic acid.

For alkaline treatment, fibers were treated with NaOH by placing about 120g fibres in 2000mL conical flask of distilled water containing 15wt% NaOH. After that, fibres were soaked in water bath at temperature 75°C about 4 hours. Then, the fibers were taken out from water bath after 4 hours before it was washed using distilled water repeatedly and then the fibers was dried in the oven for 1 day.

The next treatment is bleaching process. 120g dry fibers were filled in 2000mL conical flask containing of distilled water with 2% concentration H₂O₂ solution. Next, fibers was soaked in water bath at temperature 45°C with agitation speed 40 rpm for 8 hours before it was washed using distilled water repeatedly and then the fibers was dried in oven at temperature 70°C for 1 day.

Neutralizing process is the process to neutralize the fibers after going through the treatment above. Treated fibers were neutralized by using 10% (v/v) acetic acid for 30 minutes in water bath at temperature 50°C. After 30 minutes, the fibers was taken out from water bath and it was washed using distilled water repeatedly until free from acid and the pH was checked.

Lastly, treated fibers were dried in oven at temperature 70°C for 48 hours. After 48 hours OPEFB fibers was produced and stand by to the next process. The flow chart of production of treated OPEFB fibers is illustrated on figure 3.2.

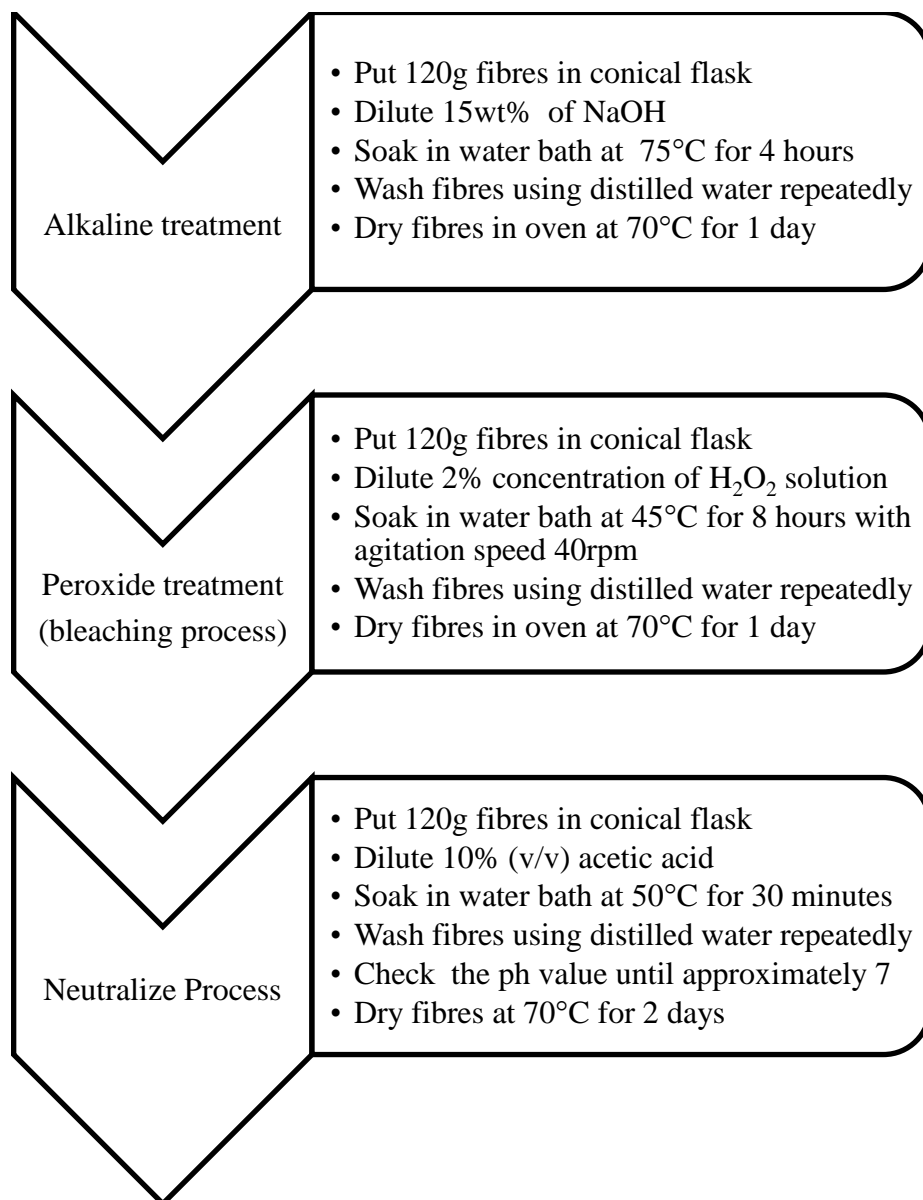


Figure 3.2: Production of treated fibers



a) Alkaline treatment in water bath



b) Taken out from water bath after treated using H_2O_2

Figure 3.3: Production of treated fibers

3.3.3 Extrusion Process

Composites were fabricated using plastic mixer (Figure 3.4 (a) and (b)) at FRIM (Forest Research Institute Malaysia) at temperature $180^{\circ}C$ to $220^{\circ}C$ with speed level 15rpm. This equipment is used to mix the composition of composite homogeneously. The formulation composition were mixed into the plastic mixture are 30wt% fibers content, 10wt% of flame retardant, 3wt% plasticizer, MAPP content is 1:10 fibers content and the remaining of composition of composite is virgin PP. All the composition was mixed in internal mixer, followed by palletized fibers into uniform length less than 5mm using crusher machine. Figure 3.3 was showed the formulation of composite for the extrusion process.

Fibers (30wt %) + MAPP (1:10 fibers) + Flame Retardant, FR (10wt %) + Plasticizers (3wt %) + PP

Figure 3.3: Formulation of composite

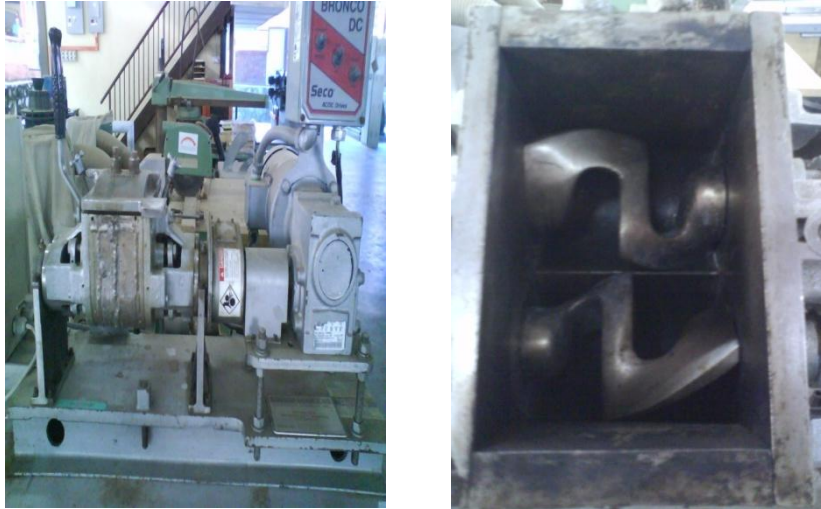


Figure 3.4: Equipment (a) and (b) used for extrusion process

3.3.4 Compression Molding

Compression molding using hot press is a method of molding in which the molding material, generally preheated, is first placed in an open, heated mold cavity. The mold is closed with a top force; pressure is applied to force the material into contact with all mold areas, while heat and pressure are maintained until the molding material has cured. The compression molding starts with allotted amount of materials that has been crashed was inserted into a mold. Afterward, the mold was put into the hot press and the mold was heated at temperature 180°C to 190°C for 5minutes to 10minutes. After that, mold was taken out to cold the mold before sample that has been molded was removed. Figure 3.5 showed compression molding process.

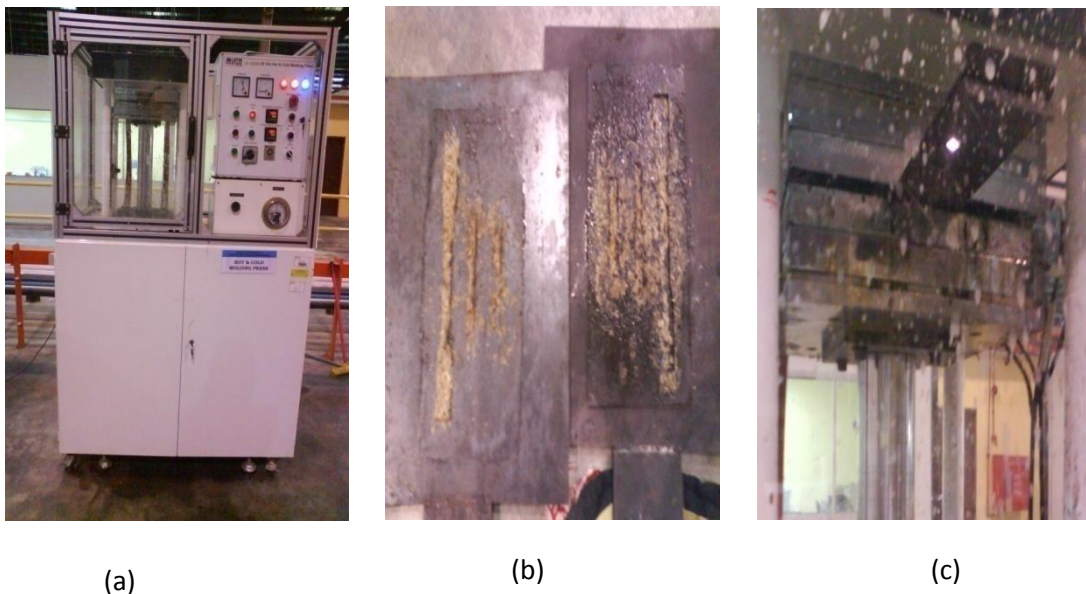


Figure 3.5: Compression molding process; (a) hot press equipment, (b) mold used and (c) hydraulic press

3.3.5 Fourier Transform Infrared (FTIR) Spectroscopy Analysis

Fourier Transform Infrared Spectroscopy (FTIR) can provide very useful information about functional group. The technique can be used to analyze organic materials and some of inorganic materials. The FTIR technique is to measure the absorption of various infrared radiations by the target material, to produce an IR spectrum that can be used to identify functional groups and molecular structure in the sample. FTIR can analyze sample in solid and liquid phase. In this analysis, two samples were prepared. The samples are untreated and treated OPEFB fibre and composite with and without plasticizer. For the solid phase sample, plate was used as a medium to put sample and Potassium Bromide (KBr). KBr was used to support the analysis and as a background to collect data for the samples. Samples were put on the plate and then plate was pushed into the FTIR and the data was collected in range 120seconds and the graph was appeared with trend and wavelength values. Figure 3.6 showed the FTIR equipment.



Figure 3.6: FTIR equipment

3.3.6 Melt Flow Index (MFI) Analysis

Melt Flow Index is the output rate (flow) in grammas that occurs in 10 minutes through a standard die of 2.0955 ± 0.0051 mm diameter and 8.000 ± 0.025 mm in length when a fixed pressure was applied to the melt via a piston and a load of total mass of 2.16 kg at a temperature of 230°C melt time used was 20 seconds. The cutting number was 3 and cut time was 15 seconds. Three samples were prepared which is three different types of plasticizers was tested. Those are sorbitol, glycerol and polyethylene glycol. From this test the data of Melt Flow Rate (MFR) was collected. Figure 3.7 was showed the MFI process.

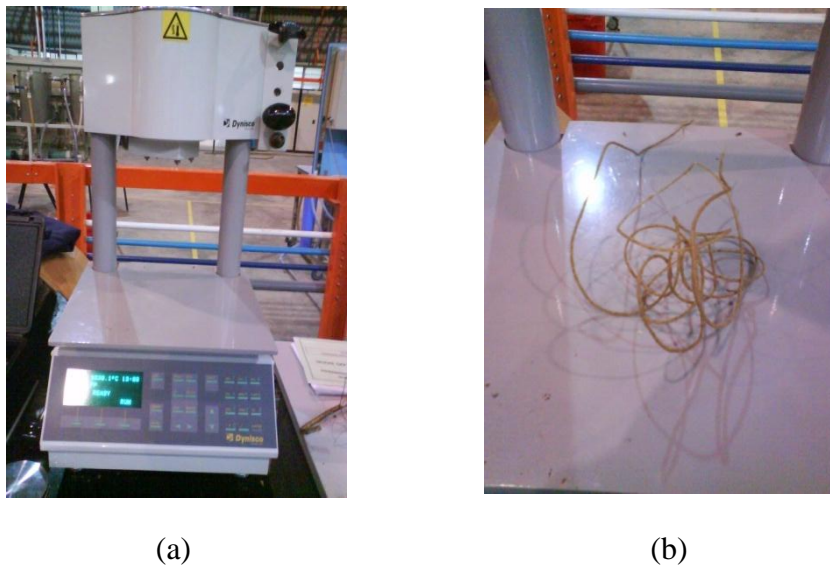


Figure 3.7: MFI process; (a) MFI equipment and (b) sample after MFI process

3.3.7 Density Analysis

Density analysis has been done using Micromeritics Gas Pycnometer. It is being used to obtain materials density measurements. These measurements are necessary in the manufacture of many of today's products. Simply defined as mass divided by volume (g/m^3), density measurements guide the formulation process and influence the overall quality of manufactured products. The equipment was used with weight the container without sample. Next, the sample weight was more than $\frac{3}{4}$ from weight container. And then key in the mass of sample in the software and then the equipment was run. This equipment is used helium gas as transporter in analysis of density.

CHAPTER 4

RESULT AND DISCUSSION

4.1 Introduction

This chapter discussed the overall result obtained in this research. As mentioned in Chapter 1 there are one parameter was studied in this research that is mechanical performance of composite with three different types of plasticizers as a additive in the composite. From this research, several testing have been done to find the mechanical performances of the composites. The testing are FTIR analysis, MFI analysis and Density analysis using Gas Pycnometer. Further explanation is provided in the following section.

4.2 Fourier Transform Infrared Spectroscopy (FTIR)

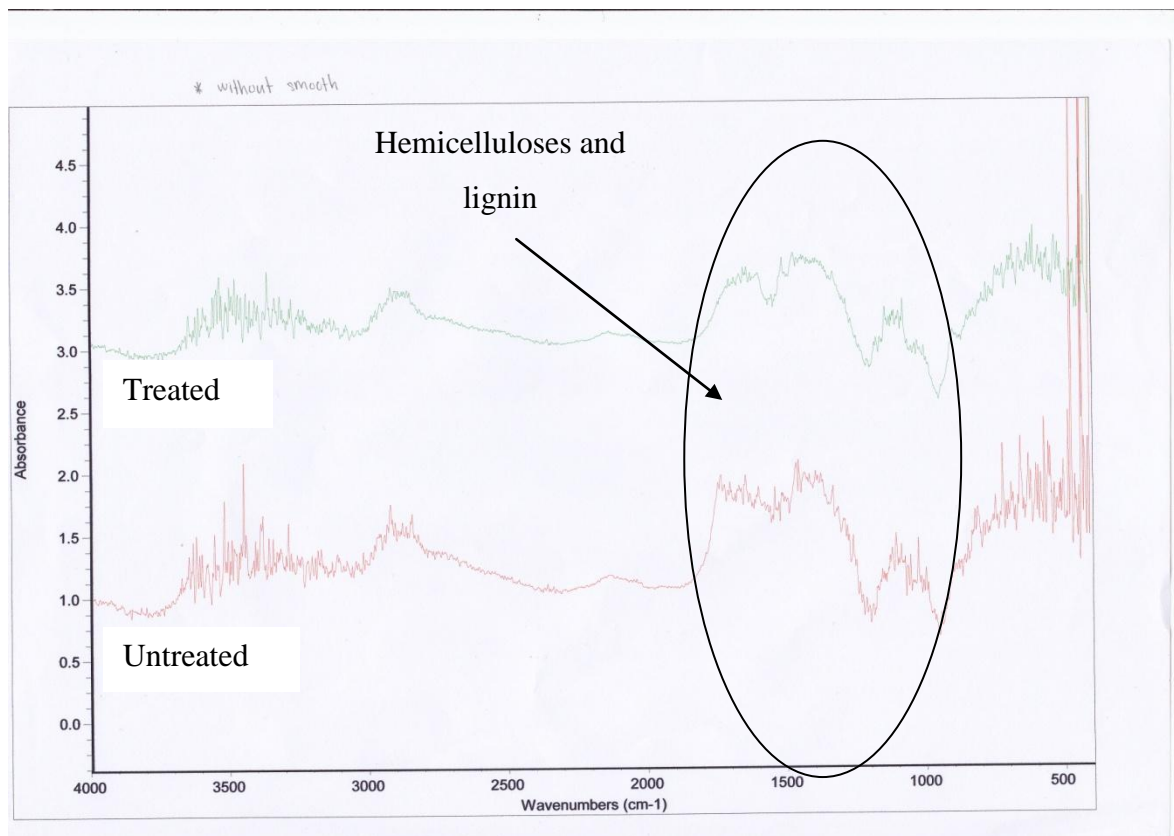


Figure 4.1: The FT-IR spectra of untreated fibers and treated fibers

The FTIR technique is to measure the absorption of various infrared radiations by the target material, to produce an IR spectrum that can be used to identify functional groups and molecular structure in the sample. From figure 4.1 the trend wavelength and functional that has been determined of graph was summary in table 4.1. Based on the table at wave length 1654.65 cm^{-1} - 1256.43 cm^{-1} the peak of functional group of lignin and hemicelluloses was detected at untreated fibers. And at wave length 1647.11 cm^{-1} the peak of lignin and hemicelluloses was detected at treated fiber. From the figure 4.1 the certain peak at untreated fibers wave length was disappeared after fibers was treated. It was showed that alkaline treatment was removed the lignin and hemicelluloses and it

was reduced the water adsorption of the fiber. Alkaline treatment was modified the structure of fibers from hydrophilic to hydrophobic.

Table 4.1: Functional group of FTIR

Functional Group	Spectra value (cm ⁻¹)			
	Untreated OPEFB fibre	Treated OPEFB fibre	Treated OPEFB fibre + MAPP	Treated OPEFB fibre + MAPP + FR
Hydroxyl (O-H)- phenolic, aliphatic Broad band	3382.74	3428.92	3408.02	3540.11 3405.56
Aromatic methoxyl (C-H), C=C, triple bond	2909.20 1130.37	2932.31 1137.02	2887.66 1133.09	2983.22 1034.12
Carbonyl (-C=O), Stretching C=C, triple bond, C-H.	1523.11 1498.00 1321.37 1130.75	1461.88 1335.17 1132.71	1477.65 1395.02 1210.11 1130.08	1496.46 1356.44 1188.33
Carboxyl-Carbonyl (O=C-OH), lignin, hemicellulose,	1654.65 1256.43	1647.11	1605.09	1589.06
Ester (O=C-O)	1740.24	N/A	N/A	N/A
MAPP(O=C-O) (C=C)	N/A	N/A	3378.45 1607.87	3385.45 1599.87
Metal (FR)	N/A	N/A	N/A	943.34 476.54

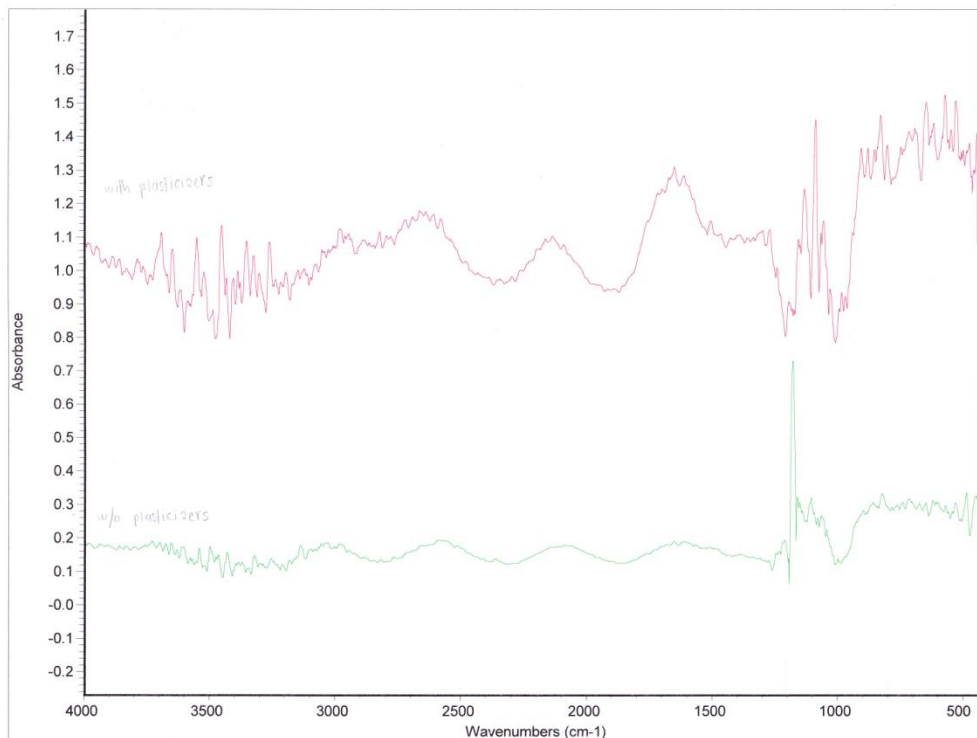


Figure 4.2: The FT-IR spectra of composite without plasticizer and with plasticizer

To investigate the polymer–fibre interactions, binary and ternary systems containing OPEFB fibre, MAPP, PP and flame retardant (FR) at various cellulose content, were analysed by FT-IR spectroscopy. The composite's spectra are shown in figure 4.2. The occurrence of interactions between maleic anhydride and hydroxyl groups of cellulose was carefully investigated in the frequency ranges 3378.45cm^{-1} – 1607.87cm^{-1} and 3408.02cm^{-1} . With MAPP the interaction between fibers and PP was improved. MAPP as additive to build cross linking between fibers and PP. Flame retardant was detected at wave length range 943.34cm^{-1} - 476.54cm^{-1} .

4.3 Melt Flow Index (MFI)

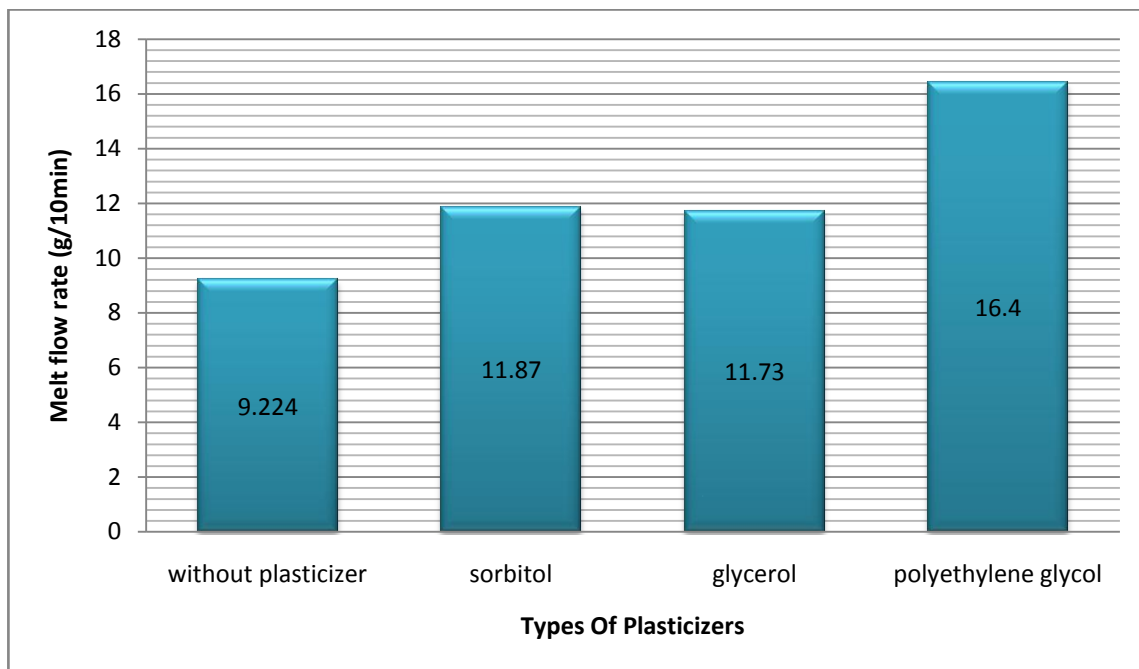


Figure 4.3: Melt Flow Index analysis

Melt Flow Index is an assessment of average molecular mass and is an inverse measure of the melt viscosity; in other words, the higher a MFI, the more polymer flows under test conditions. Knowing the MFI of a polymer is vital to anticipating and controlling its processing. Generally, higher MFI polymers are used in injection molding, and lower MFI polymers are used with blow molding or extrusion processes. Many factors affect polymers' flow properties. Molecular weight distribution, the presence of co-monomers, the degree of chain branching and crystallinity influence a polymer's MFI as well as heat transfer in polymer processing. From figure 4.3 was showed polyethylene glycol have highest melt flow index 16.4g/10min followed by sorbitol 11.87g/10min, glycerol 11.37g/10min and the lowest melt flow index is composite without plasticizers which is MFI is 9,224g/10min. Based on this result it showed composite with plasticizers was gave high melt flow index compared to without plasticizers. When composite have higher MFI the process to produce the composite

easier and when the materials through the extrusion process the composite more easy to extrude.

4.4 Density

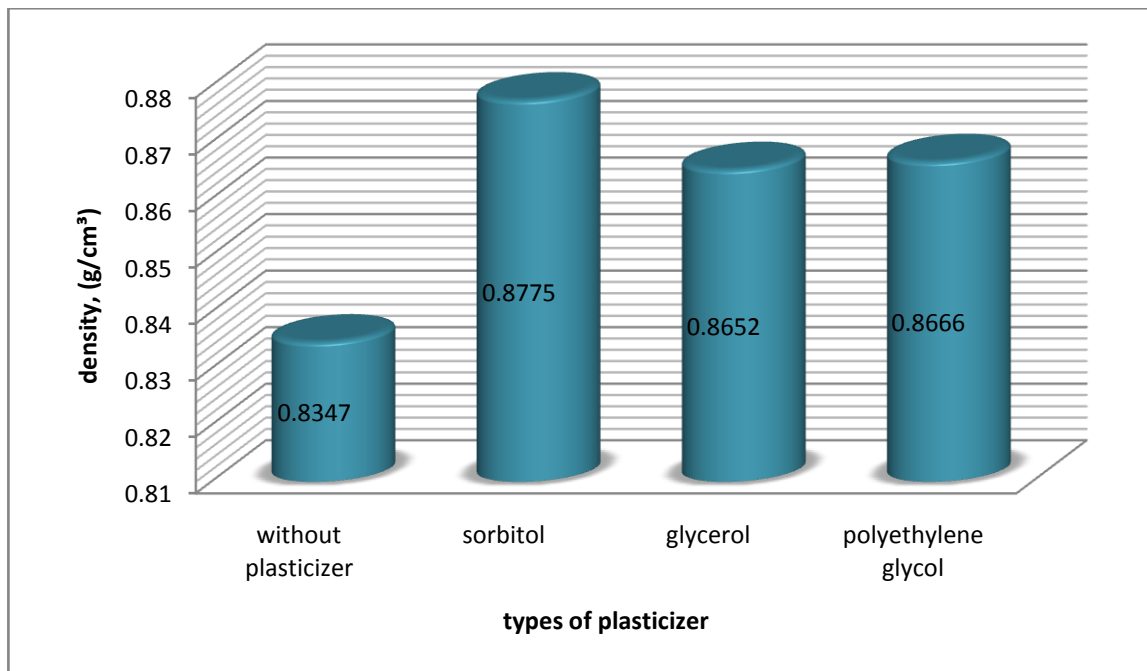


Figure 4.4: Density analysis using Gas Pycnometer

The density of the EFB filler with plasticizers found were 0.8775g/cm³ for sorbitol, 0.8666 g/cm³ for polyethylene glycol, 0.8652 g/cm³ for glycerol and 0.8347 g/cm³ for composite without plasticizer. These values were within the known range of densities reported by other researchers (Titow, 1991; Sreekala and Thomas, 2003). The results showed that the density of OPEFB with plasticizers was slightly higher than composite without plasticizers. The lowering of experimental densities was attributed to the presence of voids within the filled composites. The voids probably occurred in the matrix, at the fillermatrix interface, or within the filler bundles. The presence of voids in natural fibrefilled polymer composites has also been noted by other researchers who

found that the void content increased as the fibre content increased (Hill *et al.*, 2000; Klaric *et al.*,2000).

CHAPTER 5

CONCLUSION AND RECOMMENDATION

5.1 Conclusion

The control of properties of natural fibres reinforced polymer composites is strictly depending on the possibility of modifying the interfacial adhesion, through the improvement of the fibre–matrix interactions. In this article the effect of the addition of compatibilizers containing reactive and/or groups (OPEFB fibre, MAPP, FR, PP, FR), on the mechanical characteristics has been examined for composites containing cellulose and oat. For all compatibilized and plasticizers systems an enhanced fibre dispersion and interfacial adhesion has been observed, supporting the occurrence of effective interactions between the functional groups on the copolymer chains and the polar groups of the fibres, as demonstrated by FT-IR analysis. Besides that, the density and melt flow index of composites with different plasticizer was determined and from the all test that have been done it showed that polyethylene glycol is the good plasticizers compared to sorbitol and glycerol.

5.2 Recommendation

In this research have error from the data collected. This error occurred because several factor and constrain that cannot avoided. One of constrain is equipment constrain that effect the accuracy of the data had been collected. This research can be improve with using advanced equipment like extruder to replace plastic mixer and injection molding to replace compression molding. To improve the mechanical performance of composite addition of testing can be doing such as SEM to know the morphology structure of composite and natural fibers, TGA to know the glass transition temperature, TGA and tensile strength testing to know the mechanical performances of composites.

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APPENDIX A

FTIR ANALYSIS

APPENDIX A-1

1. Clean the KBr plate with acetone
2. Put KBr and grease into the plate

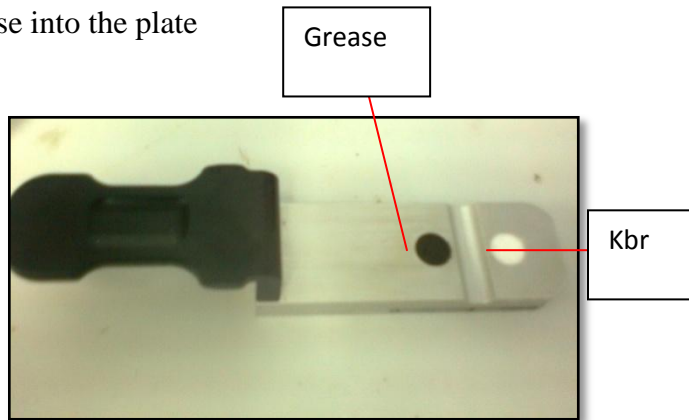


Figure B1: Kbr plate

3. Run with FTIR

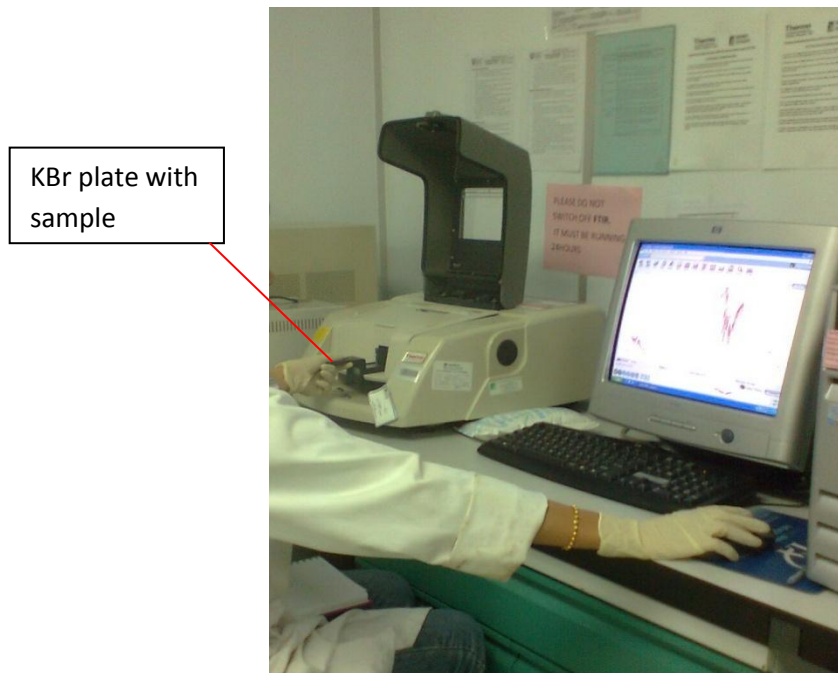


Figure B2: Fourier Transform Infrared Spectroscopy (FTIR)

4. Results from FTIR analysis were printed out
*refer attachment

APPENDIX B

DENSITY ANALYSIS