BIODEGRADATION BEHAVIOR OF PALM OIL FIBER REINFORCED STARCH BIOCOMPOSITE

TUAN ROSMINA BINTI TUAN ISMAIL

UNIVERSITI MALAYSIA PAHANG

UNIVERSITI MALAYSIA PAHANG

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Signature	:	
Name of Supervisor	:	Ms. Zatul Iffah Bte Mohd Arshad
Date	:	

BIODEGRADATION BEHAVIOR OF PALM OIL FIBER REINFORCED STARCH BIOCOMPOSITE

TUAN ROSMINA BINTI TUAN ISMAIL KE06036

A thesis submitted in fulfillment of the requirements for the award of the degree of Bachelor of Chemical Engineering (Biotechnology)

Faculty of Chemical and Natural Resources Engineering Universiti Malaysia Pahang

MAY 2010

I declare that this thesis entitled "Biodegradation behavior of palm oil fiber reinforced starch biocomposite" is the result of my own research except as cited in references. The thesis has not been accepted for any degree and is not concurrently submitted in candidature of any other degree.

Signature	:
Name	: Tuan Rosmina Binti Tuan Ismail
Date	: 5 May 2010

Special dedication of this greatfull feeling to my:

Beloved father; Mr. Tuan Ismail bin Tuan Senik

Loving brothers and sisters; Tuan Mat, Ku Azam, Tuan Amri and Tuan Azwani

> **Supportive friend;** Mohd Razali

For their love, support and best wishes.

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ABSTRACT

The use of biodegradable materials from renewable resources had gained more attention in recent years. This research focus on the production of biocomposite from starch, glycerol and palm oil fiber whereby the fiber composition were 0%, 5%, 10%, 15% and 20%. The biocomposite produced was characterized in physical and chemical properties using Universal Testing Machine, Scanning Electron Microscope (SEM), Fourier Transform Infrared (FTIR), Differential Scanning Calorimetry (DSC), microbial degradation and soil burial degradation. This study revealed that the tensile strength increased by the addition of fiber until 10% but when the fiber compositions more than 10%, the tensile strength starts to decrease because of the poor adhesion between fiber and starch. On the thermal properties, the addition of fiber in the biocomposite improves the thermal stability as the melting point of the biocomposites increases when the fiber composition increased. For the biodegradability test, as the fiber composition increased, the longer time needed for the biocomposite to be degraded. Thus, the best performance of the biocomposites reinforced with fiber was at 10% fiber composition due to its higher mechanical testing and higher melting temperature. As a conclusion, the addition of fiber as a reinforcement in starch biocomposite can improve its mechanical strength, thermal properties and biodegradability whereby it had a various applications for biocomposites such as toys for children, furniture, flooring, and hardware for electronic products.

ABSTRAK

Penggunaan bahan biodegradasi dari sumber yang boleh diperbaharui telah mendapat lebih perhatian dalam beberapa tahun kebelakangan ini. Fokus kajian ini adalah menghasilkan biokomposit dari kanji, gliserol dan serat kelapa sawit di mana komposisi serat adalah 0%, 5%, 10%, 15% dan 20% dari serat kelapa sawit. Biokomposit yang terhasil dicirikan dalam sifat fizikal dan kimia dengan menggunakan mesin pengujian, mikroskopi pengimbasan elektron (SEM), spektroskopi inframerah transformasi Fourier (FTIR), kalorimeter pengimbasan perbezaan (DSC), biodegradasi menggunakan kaedah mikrobiologi dan kaedah timbus tanah. Kajian ini menunjukkan bahawa kekuatan ketegangan meningkat dengan penambahan serat sehingga 10% dari komposisi serat tetapi apabila komposisi serat melebihi 10%, kekuatan ketegangan menurun kerana percantuman yang lemah antara serat dan kanji. Bagi sifat haba, penambahan serat dalam biokomposit meningkatkan kestabilan haba dari biokomposit sebagaimana suhu degradasi dan takat lebur biokomposit meningkat apabila komposisi serat meningkat. Bagi ujian biodegradasi, apabila komposisi serat meningkat, masa yang diambil untuk biokomposit mendegradasi lebih lama. Dengan demikian, prestasi terbaik daripada biokomposit diperkuatkan dengan serat adalah pada komposisi serat 10% kerana ujian mekanikal dan takat lebur yang lebih tinggi. Sebagai kesimpulan, penambahan serat sebagai memperkuat biokomposit kanji dapat meningkatkan kekuatan mekanikal, sifat haba dan biodegradasi dimana ia mempunyai pelbagai aplikasi untuk biokomposit seperti mainan untuk kanak-kanak, perabot, lantai, dan peranti keras untuk produk elektronik.

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LIST OF NOMENCLATURES

${}^{\mathcal{C}}$	-	Celcius
ΔH_c	-	Crystallization enthalpy
ΔH_m	-	Heat of fusion
AFM	-	Atomic Force Microscopy
DSC	-	Differential Scanning Calorimetry
EFB	-	Empty Fruit Bunch
FTIR	-	Fourier Transform Infrared
h	-	Hour
NaOH	-	Sodium hydroxide
MPa	-	Mega Pascal
SDA	-	Sabouraud Dextrose Agar
SEM	-	Scanning Electron Microscope
TA	-	Thermal Analyzer
TGA	-	Thermogravimetry Analysis
T_c	-	Crystallinity temperature
T_g	-	Glass transition temperature
T_m	-	Melting temperature
W_0	-	Original weight
W_2	-	Final weight
W_{loss} %	-	Final percentage loss in weight

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CHAPTER 1

INTRODUCTION

1.1 Background of Study

Biocomposite is a material formed by a matrix of starch and reinforced by natural fibers that usually derived from plants or cellulose. It has a wide-range uses such as for drug and gene delivery, tissue engineering applications and cosmetic orthodontics. They often mimic the structures of the living materials involved in the process in addition to strengthen the properties of the matrix that was used but still providing biocompatibility, for example in creating scaffolds in bone tissue engineering (Coats *et al.*, 2008).

Natural fiber reinforced starch biocomposite have recently gained importance in various applications such as building materials and automotive components. The natural fibers offer advantages of large quantity, annual renewability, low cost, light weight, competitive specific mechanical properties, reduced energy consumption, and environmental friendliness. The natural fibers used to reinforce bio-plastics mainly include wood, cotton, flax, hemp, jute, sisal, and sugarcane fibers (Mohanty *et al.*, 2000).

Natural fibers also can be a renewable and cheaper substitute for synthetic fibers, such as glass and carbon and have numerous advantages, such as low cost, low density, high toughness, acceptable specific strength properties, ease of separation and biodegradability. However, the main drawback of natural fiber may be their hydrophilic nature, which decreases the compatibility with hydrophobic polymeric matrix. In these composite fields, therefore, most of the research has

focused on improving interfacial properties between the polymer matrices and natural fillers in order to enhance the physical and mechanical properties of the end products (Lee *et al.*, 2009).

The current biodegradable polymers may be divided into synthetic and natural polymers, where the latter are classified into those of plant and microbial origin. The degradation of such polymers includes the disintegration into their monomers. Therefore unstable and hydrolysable linkages are required, where chemical, biological or photochemical reactions can take place. A huge number of biodegradable polymers have been synthesized chemically or by microorganisms and plants (Cañigueral *et al.*, 2009).

Among these, starch is a potentially useful material for biodegradable plastics because of its natural abundance and low cost. Starch is the major carbohydrate in plant tubes and seed endosperm, where it is found as granules. Each granule contains amylopectin molecules together with a larger number of smaller amylose molecules. The largest source of starch is corn and the other commonly used sources are wheat, potato and rice (Satyanarayana *et al.*, 2008).

Awareness in using bio-plastics as a solution for environmental problems can help to lower down the burden caused by the plastics waste. Thus, less money will be allocated to the management of plastics waste and air pollution created from the burning of these non-degradable plastics.

In this research, we are fabricating biocomposite using starch and mix it with the natural fibers derived from palm oil fiber waste. Thus we can reduce the amount of pollution where we reuse waste produced from palm oil industries to create beneficial product such as biocomposite.

1.2 Problem Statement

Petrochemical based plastics such as polyolefin, polyesters and polyamides have been increasingly used as packaging materials because of their availability in large quantities at low cost and favorable functionality characteristics such as good tensile and tear strength, good barrier properties to oxygen and aroma compounds and heat seal ability (Goda et al., 2007). However, this will causing depletion source of petroleum. The majority of engineered plastic materials used today are made from synthetic polymers. The use of conventional petroleum-based polymer products creates many potential problems due to their non-renewable nature and ultimate disposal. As these conventional, the synthetic polymers are not easily degraded because of their high molecular mass and hydrophobic character, they may accumulate in the environment and represent a significant source of environmental pollution potentially harming wildlife (Silva et al, 2000). Moreover, plastic waste disposal becomes a huge eco-technological problem and one of the approaches to solve this problem is the development of bio-composites. Bio-composites are composites material composed of biodegradable matrix and biodegradable natural fiber as reinforcement (Ewa et al, 2007). The addition of fiber from any other sources can be added to increase the physical properties of bio-composites such as tensile strength and elongation to break. Thus, by applying waste material such as oil palm fruit fiber waste, a least expensive and more environmental friendly composite can be produced. Hence, oil palm fruit fiber waste is the one of the alternatives of fiber that can use in the fabrication of bio-composite as it is natural abundance waste and least expensive.

1.3 Objective of the Study

The main objectives of this research are to produce bio-composites reinforced by palm oil fiber from empty fruit bunch and study the characteristic of physical and chemical properties of the bio-composite produced.

1.4 Scope of the Study

In order to achieve the objective, there are few scopes of work that have been identified as follows:

- Study the effect of different percentage compositions of fiber that are 0%, 5%, 10%, 15% and 20%.
- To investigate the characteristics of the biocomposite by using Fourier Transform Infrared Spectroscopy, Differential Scanning Calorimetry, Scanning Electron Microscopy and Universal Testing Machine.
- iii. To determine the biodegradability behavior of biocomposite by microbial degradation using *Aspergillus niger* (A. Niger) and soil burial degradation test.

CHAPTER 2

LITERATURE REVIEW

2.1 Introduction

Fiber reinforced biocomposites have played a dominant role for a long time in a variety of applications for their high specific strength and modulus. Recently, there has been an increasing interest in the completely biodegradable composites reinforced with natural fibers (Mohanty *et al.*, 2000), because they are renewable, biodegradable and environmentally friendly, not withstanding their use in low-cost applications (Cao *et al.*, 2004). The advantages of natural fibers over traditional reinforcing materials have been due to their acceptable specific strength properties, low cost, low density, good thermal properties, enhanced energy recovery and biodegradability. Natural fibers such as jute, sisal, pineapple, abaca and coir (Gassan *et al.*, 1999) have been studied as a reinforcement and filler in composites.

Natural fiber-reinforced composites have attracted the attention of the research community mainly because they are turning out to be an alternative solution to the ever depleting petroleum sources (Goda *et al.*, 2007). The production of 100% natural fiber based materials as substitute for petroleum-based products was not an economical solution. A more viable solution would be to combine petroleum and bio-based resources to develop a cost-effective product with diverse applications. The application of natural fiber-reinforced composites has been extended to almost all fields.

Natural fibers were mainly composed of cellulose, lignin and hemicelluloses. In general there was 1–35% lignin content in fibers depending

mainly on fiber type. Ramie, hemp and flax fibers have very low lignin content (usually under 3%), whereas wheat straw and wood fibers possess high lignin content (respectively higher than 10% and 20%) (Kunanopparat *et al.*, 2007).

Natural fibers were hydrophilic in nature as they were derived from lignocellulose, which contain strongly polarized hydroxyl groups. These fibers, therefore, were inherently incompatible with hydrophobic thermoplastics, such as polyolefins. The major limitations of using these fibers as reinforcements in such matrices include poor interfacial adhesion between polar-hydrophilic fiber and nonpolarhydrophobic matrix, and difficulties in mixing due to poor wetting of the fiber with the matrix. This in turn would lead to composites with weak interface (Alvarez *et al.*, 2008).

Tserki *et al.* (2005) reported that there were many parameters which affect the performance of a natural fiber-reinforced composite. The degree and type of adhesion cannot be estimated quantitatively even though its importance was well recognized. Aspect ratio has a considerable effect on composite properties, hence it is important to conserve fiber length as much as possible during composite processing operations. Fiber aspect ratio must be in the range of 100–200 for optimum effectiveness. Fiber orientation has a significant effect on composite properties. During processing, the fibers tend to orient along the flow direction causing mechanical properties to vary in different directions.

Futhermore, optimization of interfacial adhesion between natural fibers and polymer matrices (thermoplastic and thermoset) has been the focus of a large amount of research conducted during the past two decades. This manuscript attempts to review the latest advancements in the field of chemical modification of natural fibers.

2.2 Starch

Starch or amylum was a polysaccharide carbohydrate consisting of a large number of glucose units joined together by glycosidic bonds. Starch is produced by all green plants as an energy store and was a major food source for humans. Pure starch was a white, tasteless and odorless powder that was insoluble in cold water or alcohol. It consists of two types of molecules: the linear and helical amylose and the branched amylopectin. Depending on the plant, starch generally contains 20 to 25% amylose and 75 to 80% amylopectin. Glycogen, the glucose store of animals, was a more branched version of amylopectin. Starch can be used as a thickening, stiffening or gluing agent when dissolved in warm water, giving wheatpaste.

Starch molecules arrange themselves in the plant in semi-crystalline granules. Each plant species has a unique starch granular size: rice starch was relatively small (about 2μ m), potato starch have larger granules (up to 100μ m). Although in absolute mass only about one quarter of the starch granules in plants consist of amylose, there were about 150 times more amylose molecules than amylopectin molecules. Amylose was a much smaller molecule than amylopectin.

2.2.1 Tapioca Starch

Tapioca was a starch extracted from the root of the plant species Manihot esculenta. This species, native to the Amazon but now cultivated worldwide. Tapioca was a staple food in some regions and was used worldwide as a thickening agent, principally in foods. Tapioca was gluten free, and nearly protein free. The commercial form of tapioca most familiar to many people was pearl tapioca.

Commercially prepared tapioca has many uses. The powder was commonly used as a thickener for soups and other liquid foods, and was also used as a binder in pharmaceutical tablets and natural paints. The flour was used to make tender breads, cakes, cookies, and other delicacies. Flakes were used to thicken the filling of pies made with fruits having high water content.

2.3 Natural Fibers as Reinforcements in Biocomposite

2.3.1 Introduction

Fibers were a class of hair-like materials that were continuous filaments or were in discrete elongated pieces, similar to pieces of thread. They can be spun into filaments, thread, or rope. They can be used as a component of composite materials. They can also be matted into sheets to make products such as paper or felt. Fibers were includes of three types: natural fiber, cellulose fiber and synthetic fiber. Natural fibers were made from plant, animal and mineral sources. Natural fibers can be classified according to their origin. There were two types of fiber that were vegetable fibers and animal fibers.

Vegetable fibers were generally comprised mainly of cellulose include cotton, jute, flax, ramie, sisal and hemp. Cellulose fibers serve in the manufacture of paper and cloth. This fiber can be further categorized into the following:

- i. Seed fiber: Fibers collected from seeds or seed cases. e.g. cotton and kapok.
- ii. Leaf fiber: Fibers collected from leaves. e.g. sisal and agave.
- iii. Bast fiber or skin fiber: Fibers were collected from the skin or bast surrounding the stem of their respective plant. These fibers have higher tensile strength than other fibers. Therefore, these fibers were used for durable yarn, fabric, packaging, and paper. Some examples were flax, jute, kenaf, industrial hemp, ramie, rattan, soybean fiber, and even vine fibers and banana fibers.
- iv. Fruit fiber: Fibers were collected from the fruit of the plant. e.g. coconut fiber.
- v. Stalk fiber: Fibers were actually the stalks of the plant. E.g. straws of wheat, rice, barley, and other crops including bamboo and grass. Tree wood was also such a fiber.

The most used vegetable fibers were cotton, flax and hemp, although sisal, jute, kenaf, bamboo and coconut were also widely used. Hemp fibers were mainly used for ropes and aerofoils because of their high suppleness and resistance within an aggressive environment. Hemp fibers were, for example, currently used as a seal within the heating and sanitary industries. These were the type of fiber which is:

- i. Animal fibers generally comprise proteins; examples include silk, wool, catgut, angora, mohair and alpaca.
- Animal hair (wool or hairs): Fiber or wool taken from animals or hairy mammals. e.g. sheep's wool, goat hair (cashmere, mohair), alpaca hair, horse hair, etc.
- iii. Silk fiber: Fiber collected from dried saliva of bugs or insects during the preparation of cocoons.
- iv. Avian fiber: Fibers from birds, e.g. feathers and feather fiber.

2.3.2 Palm Oil Fiber

Palm oil fiber was equivalent to coconut fiber at a competitive price. Palm oil fiber was non-hazardous biodegradable material extracted from palm oil's empty fruit bunch (EFB) through decortation process. The fibers were clean, noncarcinogenic, free from pesticides and soft parenchyma cells. Palm oil fiber was natural fiber extracted from palm oil vascular bundles in the empty fruit bunch. During the manufacturing process of palm oil fiber, EFB were shredded, separated, refined and dried. The manufacturing process does not involved chemical process or exposure. Hence the palm oil fiber was clean and non-toxic .Palm oil fiber was mainly used in the manufacturing of mattresses, sofa/car seat and etc.

Palm oil fibers were versatile and stable and can be processed into various dimensional grades to suit specific applications such as mattress cushion production, erosion control, soil stabilization/compaction, landscaping and horticulture, ceramic and brick manufacturing, thermoplastic filler, flat board manufacturing, paper production, acoustics control, livestock care, compost, fertilizer, animal feed, etc.



Figure 2.1: Palm oil fibers

2.4 Fiber Treatment and Modification

2.4.1 Alkali Treatment

Alkali treatment was a common method to clean and modify the fiber surface to lower surface tension and enhance interfacial adhesion between a natural fiber and a polymeric matrix (Liu *et al*, 2004). Natural fibers were light and renewable; they were low-cost and high-specific-strength resource. For those reasons, natural fiber composites have already been applied for fabricating some products such as furniture and architectural materials. Recently, they have gained widespread use in the automobile industry. In their application, synthetic resins, such as polypropylene and polyethylene, were commonly used as a matrix for natural fiber composites. However, those composites often display problems of fiber-matrix compatibility which results in decrease of mechanical properties. Therefore, in order to improve the interaction between fiber and matrix, surface treatments were necessary for modifying fibers' morphology. Treatments using alkaline solutions have been applied by several researches to improve mechanical properties and fiber-matrix adhesion of natural fiber reinforced plastics such as polypropylene/flax, epoxy/flax, and polyester/kenaf. During alkali treatment, the fibers' physical structure changes as a result of alkali were bleaching action which removes waxy materials and impurities. This action often leads to improvement of the interfacial bonding between fibers and matrix (Gomes et al., 2007).

2.5 Characterization

2.5.1 Scanning Electron Microscope (SEM)

The Scanning Electron Microscope (SEM), Figure 2.2 was a type of electron microscope that images the sample surface by scanning it with a high-energy beam of electrons in a raster scan pattern. The electrons interact with the atoms that make up the sample producing signals that contain information about the sample's surface topography, composition and other properties such as electrical conductivity.



Figure 2.2: Scanning Electron Microscope

According to the previous study, Rosa *et al.* (2009) reported the effects of the different treatments on the surface fiber and the adhesion between fibers and matrix were investigated by SEM. From the microphotographs, fibers contain impurities, wax, fatty substances and globular protrusions called "tyloses". The fiber surfaces were extremely heterogeneous, with smooth and rough portions in the same fiber. The role of the treatment was mainly to remove these impurities of the natural fiber, thus improving their wettability. Fiber washing was efficient in removing impurities. However, it did not remove the protrusions. Alkali treatment removed fatty-deposits and tyloses leading to the formation of a rough surface with pits, resulting in mechanical anchorage and reduced pullout and gaps between the fiber and matrix in

composite materials (Ma *et al.*, 2005). Moreover, alkali treatment resulted in a higher amount of cellulose exposed on the fiber surface, thereby increasing the number of possible reaction sites.

Cañigueral *et al.* (2009) also suggests that the intrinsic tensile properties of fibers were proportional to fiber cross-sectional area associated with more perfect circle shape and regular form of the cross-sections. The shape of fiber cross-section was observed by SEM microphotographs.

2.5.2 Fourier Transform Infrared Spectroscopy (FTIR)

FTIR stands for Fourier Transform InfraRed that showed in Figure 2.3 was the preferred method of infrared spectroscopy. In infrared spectroscopy, IR radiation was passed through a sample. Some of the infrared radiation was absorbed by the sample and some of it was passed through (transmitted). The resulting spectrum represents the molecular absorption and transmission, creating a molecular fingerprint of the sample. Like a fingerprint no two unique molecular structures produce the same infrared spectrum. This makes infrared spectroscopy useful for several types of analysis:

- i. It can identify unknown materials
- ii. It can determine the quality or consistency of a sample
- iii. It can determine the amount of components in a mixture

Alemdar *et al.* (2008) reported that FT-IR analysis of untreated and chemically treated fibers revealed the compositional changes in the fiber structures. The prominent peak at 1737 cm⁻¹ in the untreated fiber was attributed to either the acetyl and uronic ester groups of the hemicelluloses or the ester linkage of carboxylic group of the ferulic acids of lignin or hemicelluloses. Moreover, the treatment of fiber with NaOH should reduce their hydrophilic behavior and polarity, resulting in a poor fiber matrix interface adhesion (Cañigueral *et al.*, 2009). In addition, peaks at 3392 - 3425 cm⁻¹ and around 1639.35 cm⁻¹ were assigned to O-H stretching of hemicelluloses and C=O stretching of lignin in fiber and wave numbers at 1020 –

1052 cm⁻¹ showed the presence of C-O bonds and this absorption peaks become more prominent as fiber loading increases (Aizan *et al.*, 2008).



Figure 2.3: Fourier Transform Infrared (FTIR) Spectroscopy

2.5.3 Tensile Strength

Tensile strength was indicated by the maxima of a stress-strain curve and, in general, indicates when neckling will occur. As it was an intensive property, its value does not depend on the size of the test specimen. It was dependent on the preparation of the specimen and the temperature of the test environment and material.

A Universal Testing Machine, Figure 2.4 also known as a materials testing machine or materials test frame, was used to test the tensile stress and compressive stress of materials. It was named after the fact that it can perform many standard tensile and compression tests on materials, components, and structures.



Figure 2.4: Universal Testing Machine

According to Singh *et al.* (2007) reported that by embedding natural reinforcing fibers such as flax, hemp and ramie into a biopolymeric matrix made out of derivatives from cellulose, starch, polylactic acid, polycaprolactone for example, new fiber-reinforced materials called biocomposites have been created and were still currently being developed. Many studies have been carried out to investigate the suitability of natural fibers such as flax, jute, sisal, ramie, oil palm, cellulose as reinforcing components. Generally, the mechanical properties of the polymeric matrix were greatly improved.

Cañigueral *et al.* (2009) also reported that Young's modulus and tensile strength at break increase as fiber was added. Furthermore, the strain at break was reduced and seems to be stable as the fiber fraction increases, whatever the polymer matrix. Biocomposites have the same tensile behaviour as commonly used glass fiber composites. The maximal stress supported by the material increases and therefore, there was a transfer load mechanism from the polymer to the fiber. Furthermore, the strain at break seems to be constant and identical for all the composites. Consequently, one can conclude that the strain at break of the composites is dependent on the fibers (Bodros *et al.*, 2007).

2.5.4 Differential Scanning Calorimetry (DSC)

Differential Scanning Calorimetry (DSC), Figure 2.5 was a thermoanalytical technique in which the difference in the amount of heat required to increase the temperature of a sample and reference are measured as a function of temperature. Both the sample and reference were maintained at nearly the same temperature throughout the experiment. Generally, the temperature program for a DSC analysis is designed such that the sample holder temperature increases linearly as a function of time. The reference sample should have a well-defined heat capacity over the range of temperatures to be scanned. The main application of DSC was in studying phase transitions, such as melting, glass transitions, or exothermic decompositions. These transitions involve energy changes or heat capacity changes that can be detected by DSC with great sensitivity.



Figure 2.5: Differential Scanning Calorimetry

According to previous study, Rosa *et al.* (2009) reported the thermal behavior of composites reinforced with treated fibers shows that the changes occurring in the fibers due to the treatments led to a positive effect on the thermal degradation behavior of the composites, as indicated by the higher thermal stability of the treated composites. Lovino *et al.* (2008) found that the starch exhibits glass transition T_g at

about 69.1°C, indicating the amorphous nature of plasticized starch matrix. The T_g of starch was experimentally inaccessible due to the thermal degradation of starch polymers at elevated temperatures. According to literature, the T_g of dry amylose and amylopectin has been previously estimated to be at 227° C. Thus, by incorporating natural fiber fillers into starch matrix, the T_g transition for starch rich phase shifts to higher temperature (Ewa *et al.*, 2007). Moreover, the increase in glass transition temperature, T_g also dependence of the content of fiber fillers might be attributed to the occurrence of intermolecular interactions occurred between starch and stiff crystallites, which reduces the flexibility of molecular chains of starch.

Bodros *et al.* (2007) found that an increase in temperature can create irreversible damage and leads to porosity which reduces the mechanical properties of the fibers. When processed, exposing natural fibers to temperatures over 200°C for a few minutes can lead to severe fiber degradation and a loss of integrity and the tensile strength of fibers was reduced by 40% after storing the fibres at 200°C for 1 h. As for the standard composite, the crystallization temperature trend was not regular since it increased in the initial step and then decreased; the melting temperature, ΔH_m , and ΔH_c values decreased during degradation (Lovino *et al.*, 2008).

CHAPTER 3

METHODOLOGY

3.1 Introduction

This chapter presents the detail procedure for the production of biocomposite reinforced with palm oil fibers. Then, this chapter will describe how to analyze the biocomposite by using Scanning Electron Microscopy, Fourier Transform Infrared Microscopy and Differential Scanning Calorimetry. The procedure of biodegradation test by using fungal growth and using soil also discussed in this chapter.

3.2 Materials

The raw material used in this research was palm oil fibers which were collected from Palm Oil Industry at Lepar Hilir Pahang. Five compositions of the biocomposites were formulated containing 0%, 5%, 10%, 15% and 20% of palm fiber. Tapioca starch (Bunga Merah Brand) used was obtained from local supermarket. Glycerol was purchased from Systerm. Fungi *Aspergillus niger* (*A. Niger*) used for the microbiological degradation was purchased from The Department of Microbiology, MARDI, Serdang, Malaysia and soils for the soil burial test were taken from the field nearby the university campus.

3.3 Palm Oil Fiber Treatments

Fiber treatment was used to remove the lignin and hemicelluloses content in the fiber to improve the strength of the fiber. The chemical solution used for the treatment was sodium hydroxide solution. Firstly, all fibers were pre-washed with large amount of distilled water and dried at 60°C. The treatment process consisted of immersing palm oil fibers in 1% (w/v) sodium hydroxide aqueous solution (4L) for 2 hour at ambient temperature followed by washing with distilled water several times to removed any absorbed alkali (Rosa *et al.*, 2009) and dried for 24 hour at temperature 60°C. Then, the dried fibers were grinded using grinder/crusher machine to form fine particles.

3.4 Sample Preparations

The experiment started with starch and glycerol were mixed prior with addition of water. Solutions containing starch and glycerol were stirred at 80°C for 30 minutes until the solution well mixed. After 30 minutes, the fibers were immersed into the solutions and blended until the solution mix properly. Then, the blended sample were pressed to form a film and dried in oven for 24 hours at 60°C. The film samples then were cut into uniforms shaped specimens for analysis. Figure 3.1 shows the process flow of the biocomposites preparations.



Figure 3.1: Process flow of biocomposites preparations

3.5 Scanning Electron Microscope (SEM)

SEM was a type of electron microscope that images the sample surface by scanning it with a high-energy beam of electrons in a raster scan pattern. SEM was used to view the fiber orientation in reinforced bio-composites by using EDX Spectrometer EVO 50. The samples were mounted on aluminum stabs and the acceleration voltage used was 8 kV.
3.6 Fourier Transform Infrared (FTIR) Spectroscopy

FTIR used to identifies the chemical bonds in a molecule by producing an infrared absorption spectrum. The FTIR generates an infrared spectral scan of samples that absorb infrared light. Samples were characterized for its active group by using FTIR Nicolet Avatar 370 DTDS at a constant spectral resolution of range 500 cm⁻¹ until 4000 cm⁻¹.

3.7 Tensile Test

Tensile test was mechanical tests that be perform on material. Tensile tests were simple, relatively inexpensive, and fully standardized. Tensile strength was determined under ambient temperature using Shimadzu Universal Testing Machine. The testing conditions used were cross head speed of 5mm/min and load cell of 5 kN. Dumbbell samples (1.6 mm thick) were tested with a gauge length of 154 mm. The reported values are the average of 5 measurements.

3.8 Differential Scanning Calorimetry (DSC)

DSC was a thermoanalytical technique in which the difference in the amount of heat required to increase the temperature of a sample. DSC used to identify the melting temperature of the bio-composite by using DSC Q1000 Series (TA Instrument). The samples were heated from 30°C to 300°C at heating rate of 10°C/min and a nitrogen gas flow rate of 60mL/min. The derivative of DSC curves was obtained using Thermal Analyzer (TA) analysis software.

3.9 Biodegradability Test

3.9.1 Microbial Degradation using Aspergillus niger

In this method, samples films was inoculated with *Aspergillus niger* (*A. Niger*) on a medium and incubated at surrounding temperature $(25^{\circ}C - 37^{\circ}C)$ for 21 days. Samples were faced on the surface of Sabouraud Dextrose Agar (SDA) in a petri dish containing no additional carbon source. Before faced the samples, agar surfaced were cultivated with *A. Niger*. The samples were observed for the growth of colony for 21 days.

3.9.2 Soil Burial Degradation

Biodegradability of the samples was studied by evaluating weight loss of biocomposites over time in a soil environment. Samples of 2.5 x 2.5 cm were weighted precisely and buried into soil in a plastic vase. The samples were buried into a depth of 10 cm. The buried samples were dug out at a different periods of time (7, 14 and 21 days), washed with distilled water then dried in oven at 60°C for 24 hour. The percentage loss in weight was calculated by using the equation:

$$W_{\text{loss}} \% = [1 - (W_2/W_0)] \ge 100\%$$

where W_{loss} % was the final percentage loss in weight, W_2 was the final weight after different days of burial in the soil and W_0 was the original weight (Kuo *et al.*, 2006).

CHAPTER 4

RESULT AND DISCUSSION

4.1 Introduction

This chapter provides a detail description about the results obtain from the analysis. This was followed by the effect of different fiber compositions and starch content in the biocomposite towards the biodegradability, thermal properties and tensile strength.

4.2 Scanning Electron Microscope (SEM)

The effects of the different fiber compositions on the surface fiber and the adhesion between fibers and starch were investigated by SEM. Figure 4.1 illustrate a uniform matrix at 100% starch. The surface of the bio-composites was smooth. For 5% and 20% fiber compositions in Figure 4.2 and Figure 4.4, it can be clearly seen that fiber were pulled out from the starch matrix. These findings suggest that the interaction between matrix and filler was very weak, resulting in less interfacial adhesion. These features are typical of incompatible polymer composites (Seung *et al.*, 2009).

As for the 10% fiber compositions in Figure 4.3 showed that fiber appeared to be coated with starch matrix. Although interactions of fiber-matrix interface was poor but the fiber orientation together with the agglomeration hinder the pull out process making the fibers to be well encapsulated by the starch. The ease of fiber

pull out seems smooth supporting the weak adhesion (Aizan *et al.*, 2008). This improved interfacial adhesion may be due to chemical reaction between the hydroxyl groups of starch and fiber under kneading conditions of higher temperatures and pressure (Seung *et al.*, 2009).

Furthermore, the treatment of fiber helps to reduce the biocomposite hydrophilic behavior and polarity that resulting in a poor fiber-matrix interface adhesion (Cañigueral *et al.*, 2009). Thus, the best performance in microstructure was at 10% fiber compositions because of its strong adhesion between fiber and starch.



Figure 4.1: Microstructure of 0% oil palm fiber compositions with starch



Figure 4.2: Microstructure of 5% oil palm fiber compositions with starch



Figure 4.3: Microstructure of 10% oil palm fiber compositions with starch



Figure 4.4: Microstructure of 20% oil palm fiber compositions with starch

4.3 Fourier Transform Infrared (FTIR) Spectroscopy



Figure 4.5: FTIR spectra for 0%, 5%, 10%, 15% and 20% of fiber compositions

FTIR spectra of bio-composites were presented in Figure 4.5. The spectrum in the region of 3500 to 3000 cm⁻¹ was the O-H stretching where shows the presence of starch and water in the bio-composite. O-H peak become broader as fiber loading was increased which relates to the higher water content. This compounded biocomposite was a polar compound that requires drying process prior to product manufacturing (Aizan et al., 2008). For wave numbers between 1015 to 1075 cm⁻¹ there was a presence C-O bond of polysaccharides as the present of fiber in the biocomposites (Cañigueral et al., 2009). The absorption peaks become more prominent as fiber loading increases. Furthermore, a phenolic hydroxyl group band was observed in this region of 1370 to 1400 cm⁻¹ (Guo *et al.*, 2008). These features were recognized as the common functional groups associated with the structure of lignin. The increased in the peak at around 1200cm⁻¹ suggest and increase contribution from O-H groups. These results indicated that the linkages within the structure of lignin or hemicelluloses complex are probably cleaved by 1% sodium hydroxide treatment (Guo et al., 2008). Hemicellulose, was composed of a mixture of different sugars and other various substituents which are water or base soluble because it was easy to hydrolysis. Lignin has low oxygen to carbon ratio and the structure of lignin was similar to a highly unsaturated or aromatic polymer. Part of lignin was soluble in alkali solution (Liu et al., 2004). Therefore, it was possible that a part of hemicellulose and lignin was dissolved during alkali treatment. The alkali treatment also changes the chemical properties of the lignin, which become more hydrophilic due to the increase in carbonyl and hydroxyl groups.

In addition, palm oil fiber was a natural lignocellulose fiber. Each unit of palm oil fibers mainly consists of crystalline cellulose surrounded and cemented together with hemicellulose and lignin. These ultimate cells extend longitudinally overlapping each other and form the cellular reaction between fiber and sodium hydroxide.

Fiber
$$-OH + NaOH \longrightarrow Fiber - O^{-}Na^{+} + H_2O$$

The NaOH reacts with hydroxyl groups of the cementing material hemicellulose, and it brings on the destruction of the cellular structure and thereby the fibres split into filaments. This phenomenon was termed as fibrillation, which breaks the untreated fiber bundle down into smaller ones by the dissolution of the hemicellulose. The fibrillation increases effective surface area available for contact with the matrix (Cao *et al.*, 2004) and hence the interfacial adhesion was improved.

4.4 Tensile Test

Tensile test was mechanical tests that be perform on the samples. Five samples from the each biocomposite at ratio 0%, 5%, 10%, 15%, and 20% of reinforcing fiber were used and three of the best samples data from each ratio of biocomposite were collected and shown in Figure 4.6, 4.7, 4.8, 4.9 and 4.10. Data collected were presented in a stress-strain diagram.

Figure 4.6 shows the graph of stress over strain for three samples of 0% fiber (control sample) and the average maximum stress was 0.13514 N/mm^2 and the maximum force obtained for starch was 2.20034 N.

Figure 4.7 and Figure 4.8 shows the stress-strain diagram for 5% and 10% fiber compositions and the average maximum stress for the samples were 0.40463 and 0.46265 N/mm² which was higher compared with biocomposite without fiber. The average maximum force for 5% and 10% fiber compositions were 5.76602 and 6.24577 N. Based from the results, as the fiber compositions increased, the maximum force also increased. In general, the mechanical properties of the starch-based biocomposite improved with the amount of fiber reinforcement (Cañigueral *et al.*, 2009). As expected, the strength of the bio-composites showed a lineal dependence with the amount of reinforcement with respect to the non-reinforced starch-based biocomposite. It was proven that the addition of fiber improve the mechanical properties. Interfacial adhesion between fibers was also increased by treatment of fibers with alkali. Moreover, alkali treatment reduces fiber diameter and thereby increases the aspect ratio, improving mechanical characteristics of the composites (Rosa *et al.*, 2009).

Then, the results for 15% and 20% fiber compositions showed in the Figure 4.9 and Figure 4.10. The average maximum stresses were 0.33325 and 0.30840

 N/mm^2 . The maximum force was determined at 5.20786 and 4.93447 N. The results showed that the maximum stress and maximum force were decreased after 10% compositions .This may be due to poor interfacial adhesion between the starch matrix and fiber filler. This was a general phenomenon in incompatible composites with different characteristics, such as hydrophobicity of the polymer matrix and hydrophilicity of the fiber (Seung *et al.*, 2009).



Figure 4.6: Stress-strain diagram for 100% starch compositions



Figure 4.7: Stress-strain diagram for 5% fiber composition



Figure 4.8: Stress-strain diagram for 10% fiber compositions



Figure 4.9: Stress-strain for 15% fiber compositions



Figure 4.10: Stress-strain diagram for 20% fiber compositions



Figure 4.11: Graph of tensile strength versus fiber composition

Figure 4.11 shows the graph of tensile strength versus percent fiber compositions in unit Mega Pascal (MPa). When fiber was loaded into the starch biocomposite, an increment in tensile strength occurs due to the fiber reinforcing effect. At 0% fiber, the tensile strength was the lowest but after addition of fiber, the tensile strength were increased. However, after 10% fiber compositions, the tensile

strength decreases. This decrement was because of the poor adhesion between fiber and starch thus fiber-fiber interaction was preferred by the system. The decrement in tensile strength might be due to the poor interaction between fiber and starch phases, causing internal voids in between starch-fiber or weak points inside the specimens or because of fiber agglomerations which cause the dispersion in starch biocomposite. The agglomeration of the fibers may be due to the relatively weak interaction between fibers and matrix, compared to the strong fiber-fiber interaction caused by hydrogen bonding (Aizan *et al.*, 2008). Therefore, fibers have tendency to agglomerate among themselves into fiber bundles. This also may be due to the insufficient filling of the starch matrix into the surrounding fibres (Lee *et al.*, 2008) during composite processing at over 10 wt% loading. Thus, the tensile strength shows the best performance at 10% fiber composition.

4.5 Differential Scanning Calorimetry (DSC)

DSC was used to identify the melting behavior of the biocomposite. The results of DSC analysis were summarized in Table 4.1. The result shows that the melting point for starch was 229.48°C as the biocomposite with fiber compositions start to melt at a higher temperature. In this process, the melting point of starch decrease due to the mechanical and heat energy (Satyanarayana et al., 2009). Figure 4.12, 4.13, 4.14, 4.15, and 4.16 shows the DSC curve for the bio-composites with the different fiber composition. From the graph, it can be seen that the melting point increase when the fiber compositions increased. It was because the fiber has higher melting point compare to starch. By incorporating the fiber into the starch matrix, the transition for melting temperature of starch shifts to higher temperature (Ewa et al., 2007). This trend may be explained by intermolecular interactions between the hydroxyl groups of the starch and the carbonyl groups of the fibers. These hydrogen bonds would probably reduce the biocomposite mobility and then increase melting temperature values (Avérous *et al.*, 2009). However, the heat of fusion (ΔH_m) in the composites was decreased by addition of fiber. This may be also attributed to the strong interfacial interaction between starch matrix and fiber, confining polymer chain orientation (Lee et al., 2009).

From the results, pure starch had a crystallization temperature (T_c) of 145°C. In starch biocomposite, the T_c shifted to high temperature by adding fiber. But, the decreasing in T_c could be considered to be due to less nucleation effect of the fiber. In particular, the linkage between starch matrix and fiber produced might further enhance nucleation of biocomposite matrix (Lee *et al.*, 2009). However, crystallization enthalpy (ΔH_c) was decreased by increasing fiber content. The molecular motion of the polymer matrix could be restricted by the addition of fiber, resulting in a decrease of crystallization enthalpy (Mohanty *et al.*, 2005).

The best performance of the biocomposite for thermal properties was at 20% fiber composition as it shows the higher melting point. Thus, the addition of fiber improves the thermal properties of the biocomposites.

Fiber Compositions (%)	Melting Temperature, T _m (°C)
0	229.48
5	229.49
10	237.84
15	245.15
20	251.21

Table 4.1: Results of DSC analysis for biocomposites



Figure 4.12: DSC curve of 0% palm oil fiber in starch biocomposite



Figure 4.13: DSC curve of 5% palm oil fiber reinforced starch biocomposite



Figure 4.14: DSC curve of 10% palm oil fiber reinforced starch biocomposite



Figure 4.15: DSC curve of 15% palm oil fiber reinforced starch biocomposite



Figure 4.16: DSC curve of 20% palm oil fiber reinforced starch biocomposite

4.6 Biodegradability Test

4.6.1 Microbial Degradation using Aspergillus niger

Table 4.2 shows the rating of fungal growth for the starch biocomposite with the addition of fiber. From the results, after 21 days, most of the specimen surface was covered by the fungi growth for 0% fiber or 100% starch content. The granular starch present on the surface of biocomposite was attacked by the fungi. Then, as the fiber compositions increased, the fungi growth decreases. This indicated that the growth of *A. Niger* colony increases as the fiber compositions is increased. This weakens the starch matrix and increases the the surface volume ratio, hydrophilic and permeability of the bio-composite (Aizan *et al.*, 2009).

Materials	Rating* of fungal growth
Starch	3
Fiber 5%	2
Fiber 10%	2
Fiber 15%	1
Fiber 20%	1

Table 4.2: Assessment of fungi growth for each formulations of starch/fiber

*Rating: 1-growth covering less than 25% of specimen surface, 2-growth covering almost 50% of specimen surface, 3-growth covering more than 50% of the specimen surface



Figure 4.17: Evidence of fungi growth (*A. Niger*) on surface of biocomposite in petri dish

Figure 4.17 shows the evidence of the fungal growth in the petri dish from the observation after 21 days. It can be seen that almost the entire specimen surface covered by the fungal growth but as the fiber was added into the biocomposite, the growth of fungal was decreased. For 5% and 10%, the growth of fungal covering the surface of the sample was more than 50% of the surface. It was because the fiber addition generally decreases the degradation rate of composites and alkaline treatment of fibers produces a slightly higher degradation rate than pure starch matrix (Behjat *et al.*, 2009). In addition, the composites biodegradability also enhanced with increasing starch content because the starch was easily attacked by microorganisms. Thus, from this evidence we can conclude that the additions of fiber in the biocomposite can make the time retention was longer for the biocomposite to degrade and the best performance for biodegradability test was at 20% fiber compositions which show the least growth of fungal.

4.6.2 Soil Burial Degradation

The biodegradability of the biocomposites was determined by evaluating the weight loss over the time in a soil environment. Table 4.4 displays the results of percentage weight loss evaluated in a soil environment for every 7 days interval until 21 days.

	Buried Time			
Samples	7 Days	14 Days	21 Days	
0% fiber	32	68	87	
5% fiber	28	47	73	
10% fiber	20	43	65	
15% fiber	18	38	61	
20% fiber	17	35	59	

Table 4.3 :	The weight loss percen	tage (W_{loss} %):	in biodegradability	examination for
	21 days			



Figure 4.18: Graph of weight loss of biocomposite versus time

In Figure 4.18, the weight loss curves show a gradual increase for biodegradation studies of biocomposites. The higher weight loss of biocomposites can be seen at 100% starch compositions. Then, as the fiber compositions increased to 5%, the weight loss percentage was decreased followed by the addition of 10% and 15% fiber compositions, the percentage of weight loss also decreased. It was because exposure to a soil environment also caused deterioration in tensile strength and elongation at breaks for biocomposites. In the soil, water diffused into the biocomposite samples, causing swelling and enhances the biodegradation (Kuo et al., 2006). After 21 days, the weight loss percentage in the biocomposite is low when the compositions of fiber are increased. It shows that the time retention to degrade for the biocomposite with fiber will become longer compared to bicomposite with starch compositions only. Moreover, microorganisms present in soil are relatively active to hemicelluloses at suitable humidity and temperature conditions (Alvarez et al., 2008). The minimum percentage of weight loss was determined at 20% fiber. This was happened because the fiber was resilient, strong, and highly durable and it's more difficult to degrade. So, it was proven that the fiber reinforcement can improve the bio-composite's durability to degrade. Thus, the best performance of biocomposite in biodegradability was at 20% fiber composition.

CHAPTER 5

CONCLUSION AND RECOMMENDATION

5.1 Conclusion

The use of biodegradable materials from renewable resources had gained more attention in recent years. In this study, palm oil fruit fibers were used as reinforcement in biodegradable composites of starch. The addition of natural fiber in starch can produce bio-composite that demonstrate high strength in mechanical testing. It can be seen at 10% fiber loading, the tensile strength was in highest value but after 10% fiber loading, the tensile strength decrease. These improvements were due to the enhanced interfacial adhesion between the starch matrix and fiber.

In thermal properties, the melting temperature also increased as the fiber composition increased. It was because the fiber has higher melting point compare to starch. By incorporating the fiber into the starch matrix, the transition for melting temperature of starch shifts to higher temperature. This may be also attributed to the strong interfacial interaction between starch matrix and fiber, confining polymer chain orientation.

Furthermore, in biodegradability test, it can be seen that when the fiber compositions in the biocomposite increased, the longer time needed for the biocomposite to be degraded. This was happened because the fiber was resilient, strong, and highly durable and it's more difficult to degrade. So, the present of fiber can delay the time retention for the biocomposite to degrade. As a conclusion, the optimum fiber loading for best performance was at 10% fiber compositions with respect to higher mechanical testing from tensile test and strong adhesion between fiber and starch matrix from SEM analysis. So, it was proven that starch biocomposite can be reinforced by natural fiber. Advantages of natural fibers over other reinforcing materials like oil palm fiber are their low cost, acceptable specific strength properties and biodegradability. In additional, the biocomposite can be used in various applications for bio-composites such as toys for children, furniture, flooring, hardware for electronic products, especially in one-way disposable products.

5.2 **Recommendation**

For recommendations, we can use other natural fibers instead of oil palm fiber such as coir, wood, cotton, flax, hemp, jute, sisal, and sugarcane fibers to vary the raw material used.

Then, we can add other analysis such as Atomic Force Microscopy (AFM) and Thermogravimetry Analysis (TGA). AFM is used to check the roughness of the surface of biocomposite and check the structure of the biocomposite to investigate the weakness of the biocomposite. Then, TGA is used to investigate the thermal properties of the biocomposites. From this analysis, the weight loss of the biocomposites can be determined.

For microbial degradation using *Aspegillus niger*, the petri dish with the sample should be weighted before incubated. After 21 days, the petri dish must be reweighted again to determine the weight loss of the samples.

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APPENDIX A

MATERIALS

APPENDIX A

MATERIALS



Figure A1: Tapioca Starch



Figure A2: Palm oil fiber



Figure A3: Glycerol



Figure A4: Distilled water

APPENDIX B

PREPARATIONS

APPENDIX B

PREPARATIONS



Figure B1: Starch, glycerol and distilled water were stirred together



Figure B2: Addition of palm oil fiber into the solution



Figure B3: Solution was poured into the glass plate for casting method

APPENDIX C

RESULTS OF ANALYSIS

FTIR RESULTS ANALYSIS





Figure C1: FTIR spectra for 0% fiber compositions





Figure C2: FTIR spectra for 5% fiber compositions





Figure C3: FTIR spectra for 10% fiber compositions





Figure C4: FTIR spectra for 15% fiber compositions




Figure C5: FTIR spectra for 20% fiber compositions

TENSILE TEST ANALYSIS

Key Word			Product N	lame			
Test File Name	0% fiber-s	0% fiber-starch.xtak Method File Name			tensile- zatul student.xm		
Report Date	2006/12/03	2006/12/03		Test Date		2006/11/30	
Test Mode	Single	Single		Test Type		Tensile	
Speed	5mm/min	5mm/min		Shape		Plate	
No of Batches:	1	1		Qty/Batch:		5	
Name	Elastic	Max_	Force	Max_Stress		Max_Strain	
Parameters	Force 10 - 20 N	Calc. a	at Entire A	Calc. at Entire A		Calc. at Entire A	
Unit	N/mm2	1	N I	N/mm2		%	
1_1	-,-	2.10)444	0.12990		2.75130	
1_3		2.23796		0.13713		3.20772	
1_4		2.25862		0.13840		3.18893	
Average	-,-	2.20034		0.13514		3.04932	
Standard Devi	-,-	0.08369		0.00459		0.25826	
Maximum	-,-	2.25862		0.13840		3.20772	
Minimum		2.10444		0.12990		2.75130	
Name	Break_Stress	EASL1	Stroke	EASL1_Stra	ain		
Parameters	Sensitivity: 10	Force 1 N		Force 1 N			
Unit	N/mm2	mm		%			
1_1	-,-	1.39285		1.21117			
1_3	-,-	1.50082		1.30506			
1_4	-,-	1.53022		1.33062			
Average	-,-	1.47463		1.28228			
Standard Devi		0.07233		0.06290			
Maximum	-,-	1.53022		1.33062			



Key Word			Product Name				
Test File Name	est File Name 5% palm oil fiber-stard		Method File Name		tensile- zatul student.xm		
Report Date 2006/12/01		Test Date		2006/12/01			
Test Mode Single			Test Type		Tensile		
Speed	5mm/min	5mm/min		Shape		Plate	
No of Batches:	1	1		Qty/Batch:		5	
Name	Elastic	Max_	Force	Max_Stress		Max_Strain	
Parameters	Force 10 - 20 N	Calc. a	at Entire A	Calc. at Entire A		Calc. at Entire A	
Unit	N/mm2	1	N	N/mm2		%	
1_1	-,-	6.53823		0.45882		6.12158	
1_2	-,-	5.80629		0.40746		6.10056	
1_5		4.95354		0.34762		5.97520	
Average		5.76602		0.40463		6.06578	
Standard Devi	-,-	0.79311		0.05565		0.07915	
Maximum		6.53823		0.45882		6.12158	
Minimum	-,-	4.9	4.95354			5.97520	
Name	Break Stress	EASL1	Stroke	EASL1 Stra	ain		
Parameters	Sensitivity: 10	Force 1 N		Force 1 N	1		
Unit	N/mm2	mm		%			
1_1	-,-	0.90141		0.78384			
1_2	-,-	0.94389		0.82078			
1_5	-,-	1.10779		0.96330			
Average		0.98436		0.85597			
Standard Devi	-,-	0.10898		0.09476			
Maximum	-,-	1.10	0779	0.96330			
Minimum	-,-	0.90141		0.78384			



Key Word		Product Name					
Test File Name	10% palm oil fi	10% palm oil fiber-starct Method File Nam			tensile- zatul student.xm		
Report Date	2006/12/01	2006/12/01 Test D		Date		2006/12/01	
Test Mode	Single		Test Type		Tensile		
Speed	5mm/min	5mm/min		Shape		Plate	
No of Batches:	1	1		Qty/Batch:		5	
Name	Elastic	Max_	Force	Max_Stress		Max_Strain	
Parameters	Force 10 - 20 N	Calc. a	at Entire A	Calc. at Entire		Calc. at Entire A	
Unit	N/mm2	1	V	N/mm2		%	
1_1	-,-	6.04	4788	0.44799		5.82589	
1_3		6.42	2061	0.47560		6.17803	
1_4	-,-	6.26882		0.46436		4.27087	
Average		6.24577		0.46265		5.42493	
Standard Devi	-,-	0.18743		0.01388		1.01484	
Maximum		6.42061		0.47560		6.17803	
Minimum	-,-	6.04788		0.44799		4.27087	
Name	Break Stress	EASL1	Stroke	EASL1 Stra	ain		
Parameters	Sensitivity: 10	Force 1 N		Force 1 N			
Unit	N/mm2	mm		%			
1_1		1.10006		0.95657			
1_3	-,-	1.04725		0.91065			
1_4		0.70681		0.61461			
Average		0.95137		0.82728			
Standard Devi		0.21344		0.18560			
Maximum	-,-	1.10006		0.95657			
Minimum	-,-	0.70681		0.61461			



Key Word		Product Name					
Test File Name	e Name 15% palm oil fiber			ile Name	tensil	le- zatul student.xm	
Report Date	2006/12/01	Tes	Test Date		2006/11/30		
Test Mode Single		Test Type		e Te		Fensile	
Speed	5mm/min	Sha	Shape		Plate		
No of Batches:	1	Qty	Qty/Batch:		5		
Name	Elastic	Max_For	e	Max_Stress		Max_Strain	
Parameters	Force 10 - 20 N	Calc. at En	tire A	Calc. at Entire A		Calc. at Entire A	
Unit	N/mm2	N		N/mm2		%	
1_1	-,-	6.30538	}	0.36468		3.86342	
1_3		4.66585	5	0.33685		3.83460	
1_5	-,-	4.65234		0.29823		2.59899	
Average	-,-	5.20786		0.33325		3.43234	
Standard Devi	-,-	0.95051		0.03337		0.72184	
Maximum		6.30538		0.36468		3.86342	
Minimum	-,-	4.65234		0.29823		2.59899	
Name	Break Stress	EASL1 Str	oke	FASL1 Str	ain		
Parameters	Sensitivity: 10	Force 1 N		Force 1 N			
Unit	N/mm2	mm		%			
1_1	-,-	0.63611		0.55314			
1_3	-,-	0.98514		0.85665			
1_5	-,-	0.64775		0.56326			
Average	-,-	0.75633		0.65768			
Standard Devi	-,-	0.19824		0.17238			
Maximum	-,-	0.98514	Ļ	0.85665			
Minimum	-,-	0.63611		0.55314			



Key Word		Product Name	
Test File Name	20% palm oil fiber-starcl	Method File Name	tensile- zatul student.xm
Report Date	2006/12/01	Test Date	2006/12/01
Test Mode	Single	Test Type	Tensile
Speed	5mm/min	Shape	Plate
No of Batches:	1	Qty/Batch:	5

Name	Elastic	Max_Force	Max_Stress	Max_Strain
Parameters	Force 10 - 20 N	Calc. at Entire A	Calc. at Entire A	Calc. at Entire A
Unit	N/mm2	N	N/mm2	%
1_1	-,-	5.99464	0.37466	3.73239
1_2	-,-	4.43935	0.27746	3.42723
1_4		4.36942	0.27309	2.37794
Average	-,-	4.93447	0.30840	3.17919
Standard Devi	-,-	0.91880	0.05742	0.71048
Maximum		5.99464	0.37466	3.73239
Minimum		4.36942	0.27309	2.37794
Name	Break_Stress	EASL1_Stroke	EASL1_Strain	
Parameters	Sensitivity: 10	Force 1 N	Force 1 N	
Unit	N/mm2	mm	%	
1_1		0.62202	0.54088	
1_2		0.71231	0.61940	
		0.40.24.5	0.10001	



