

**BIODEGRADATION BEHAVIOR OF COCONUT HUSK (COIR) FIBER
REINFORCED STARCH BIO-COMPOSITE**

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ABSTRACT

In this work, a starch-based biopolymer was reinforced with coir fiber. The purpose of this research was to investigate a production of bio-composite based on different content percentage of coir fiber as a reinforcement material. Five compositions of the bio-composites were formulated containing 0%, 5%, 10%, 15% and 20% of coir fiber where 0% of fiber acts as the control sample. Coir fiber and starch were a good material for bio-composite production because there were readily biodegradable, renewable resource and low cost price. Characteristics of the bio-composite were evaluated by tensile strength, microstructure, chemical, physical and biodegradability properties. The evaluation of the mechanical properties gave a significant increase of the strength of the bio-composite with the percentage of coir fiber. The optimum value of mechanical strength was obtained at 15% fiber reinforcement and it also proved from Scanning Electron Microscope analysis where the structure of 15% fiber reinforcement showed a good interaction between coir fiber and matrix phase and coir fiber was seen well embedded in the matrix. Roughly, Fourier Transform Infrared Spectroscopy results showed the O-H peak become broader with the increment of the fiber reinforcement. The Differential Scanning Calorimetry result showed that the melting temperature was increase as the fiber composition in bio-composite was increased. In biodegradability test, it showed that when the fiber content was increase, the *Aspergillus Niger* was more difficult to growth and also difficult to degrade in soil. It was proven that the coir fiber reinforcement can improve the bio-composite's durability to degrade. Averagely, the best performance of bio-composite was at 15% of coir fiber with respect to chemical bonds, dispersion and agglomeration, heat resistance, biodegradability behavior, and mechanical properties

ABSTRAK

Dalam kajian ini, kanji berasaskan biopolimer diperkuatkan dengan sabut kelapa. Penyelidikan ini mengkaji pembuatan bio-komposit berdasarkan peratusan sabut kelapa sebagai penguat kanji berasaskan biopolimer. Lima komposisi bio-komposit dihasilkan yang mengandungi 0%, 5%, 10%, 15% dan 20% kandungan sabut kelapa yang mana 0% kandungan sabut kelapa adalah sampel kawalan. Sabut kelapa dan kanji merupakan bahan asas yang baik untuk penghasilan bio-komposit kerana mempunyai sifat biodegradasi, sumber yang banyak dan kos yang rendah. Ciri-ciri bio-komposit ditentukan melalui kekuatan ketegangan, struktur mikro, sifat fizikal, sifat kimia, dan sifat biodegradasinya. Bagi penilaian sifat mekanik, ia menunjukkan kekuatan bio-komposit meningkat selari dengan peningkatan jumlah sabut kelapa sebagai penguat bio-komposit. Kekuatan mekanikal tertinggi adalah pada kandungan 15% sabut kelapa dan ia juga dibuktikan melalui analisis Scanning Electron Microscope yang mana struktur bagi kandungan 15% sabut kelapa sebagai penguat menunjukkan interaksi yang baik antara sabut kelapa dengan matrik dan sabut kelapa juga tertanam dengan baik di dalam matrik. Secara kasarnya, data Fourier Transform Infrared Spectroscopy menunjukkan peak OH menjadi lebih memanjang seiring dengan kenaikan kandungan sabut kelapa sebagai penguat. Data Differential Scanning Calorimetry pula menunjukkan bahawa suhu lebur meningkat mengikut peningkatan kandungan sabut kelapa dalam bio-komposit. Dalam sifat biodegradasi bio-komposit, menunjukkan peningkatan kandungan sabut kelapa menjadikan pertumbuhan *Aspergillus Niger* lebih sukar dan ia juga susah untuk terurai di dalam tanah. Ini membuktikan bahawa sabut kelapa dapat meningkatkan sifat ketahanan bio-komposit daripada terurai. Rata-rata, prestasi terbaik bagi bio-komposit adalah pada 15% kandungan sabut kelapa berdasarkan ikatan kimia, taburan dan pengelompokan, daya tahan haba, sifat biodegradasi, dan sifat mekanik.

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LIST OF SYMBOLS

SEM	-	Scanning Electron Microscope
FTIR	-	Fourier Transform Infrared Spectroscopy
DSC	-	Differential Scanning Calorimetry
SDA	-	Sabaraud Dextrose Agar
UTS		Ultimate tensile strength
MW	-	Molecular weight
ΔH_c		Crystallization enthalpy
ΔH_m		Melting enthalpy
T _g		Glass transition temperature
T _c		Crystallization temperature
T _m		Melting temperature
cm	-	Centimeter
mm	-	Millimeter
N	-	Newton
°C	-	Degree Celsius
mg	-	Milligram
L	-	Liter
ml	-	Milliliters
h	-	Hours
min	-	Minutes
NaOH	-	Sodium hydroxide
H	-	Hydrogen
C	-	Carbon
O	-	Oxygen
%	-	Percentage

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CHAPTER 1

INTRODUCTION

1.1 Background of Study

In today's world, polymers also known as plastics where it plays a significant role in our daily life. Plastics have become one of the most widely used materials all over the world. Plastic materials have gained widespread use in the food, clothing, medical, shelter, transportation, construction and leisure industries due to its features of strength, lightweight, easily process able and energy efficient. Plastics clearly have become indispensable and important part to our life. 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 (Tharanathan, 2003). However, these plastics that are made of petroleum-based materials are non-biodegradable. Non-biodegradable is a word used to identify a product which is unable to break down into the nature. These synthetic plastics such as polyethylene and polypropylene are not degraded by microorganisms in the environment, which contributes to their long lifetime. This situation will cause pollution to the environment. Now, the world is concern about the non-biodegradable waste that becomes one of the factors of pollution.

Therefore, we have to find an alternative such as bio-composite that is more environmental friendly. Bio-composite is a material formed by a combination of natural fibers such as wood fibers (hardwood, wood dust and softwood) or non-wood fibers (e.g., wheat, husk, jute, sisal, coir and flax) with polymer matrices from both of the renewable and nonrenewable resources. Nowadays, starch is a material that has a potential use to produce bio-composite because of its lower cost, environmental friendly and natural abundance. Starch is the major carbohydrate in plant tubes and seed endosperm, where it is found as granules. The source of starch is corn, wheat, potato and rice. Starch is one of the least expensive biodegradable materials used for many non-food items such as paper making, cardboard, furniture and adhesives. More recently, starch has been used as the main polymer in thermoplastic compositions and has been processed into eating utensils and as raw material for film production. Converted to thermoplastic material, starch offers an interesting alternative for synthetic polymers where long-term durability is not needed and rapid degradation is an advantage (Flieger *et al.*, 2003).

Currently, many efforts have been made to identify the suitability of natural fibers as a reinforcing component for thermoplastic materials. They come out with further research in waste management and therefore will produce highly functional composite materials if used in combination with biodegradable polymers. Hence, a natural fiber likes jute, rice straw, sisal, flax, coconut husk, sugar cane husk, and oil palm husk are expected to act as reinforcing components in composite materials.

This research aim at developing a new formulation of bio-composite based on coconut husk (coir) fiber as a reinforcement of starch-based biopolymer. The quality at fiber–matrix interface and the intrinsic mechanical properties of coconut husk (coir) were measured. Besides, the aspect ratio of the reinforcement inside the polymer and the compatibility factor at fiber–matrix interface are determined by control of mixtures between fiber and starch.

1.2 Problem Statement

Nowadays, plastics clearly have become indispensable due to its wide array of applications particularly as packaging materials. Every day, a demand of plastic usage is always increase. However, almost plastics that we use now are made of petroleum-based materials and these plastics are not readily biodegradable. It's not environmental friendly and this situation will cause pollution to the environment. The bulk of plastics waste can spoil our ecological system. Other than that, the waste from coconut fruit (husk) can be recycled to produce something in the engineering field (composite). Hence, a possible solution to solve this problem is the prospect of biodegradable polymers like bio-composite. Thus, by applying waste material (coir), a least expensive and more environmental friendly composite can be produced. Bio-composite have many advantages to our life. Firstly, they are made from renewable resources. Renewable resources have gained much attention in the last decades due to the global increasing demand for alternatives to fossil resources. Secondly, they are biodegradable and able to break down into the nature after they become as wastes. Thus, we will reduce the amount of pollution caused by non-biodegradable polymers to the environment and we are also maximizing the use of renewable sources.

1.3 Research Objective

The main objectives of this study are:

1. To determine the effect of ratio between starch and coconut husk (coir) composition on the bio-composite strength.
2. To characterize the behavior of bio-composite in term of microstructure, chemical and physical analysis.
3. To analyze the biodegradation rate of bio-composite by microbial degradation using *Aspergillus Niger* and soil burial degradation test.

1.4 Scope of Research Work

The scopes of this research are as follow:

1. Production of bio-composite and tensile strength of bio-composite studies based on the effect of ratio between starch and coir fiber composition.
2. The characterization of the bio-composite using various analysis methods:
 - a. Scanning Electron Microscope (SEM)
 - b. Fourier Transform Infrared Spectroscopy (FTIR)
 - c. Differential Scanning Calorimetry (DSC)
 - d. Universal Testing Machine
3. The biodegradability test of bio-composite by microbial degradation using *Aspergillus Niger* and soil burial degradation test.

CHAPTER 2

LITERATURE REVIEW

2.1 Polymers

2.1.1 Introduction

Polymer is a long chain giant organic molecule that was made of repetitive units from many smaller molecules called monomers. Polymers consist of many repeating monomer units in long chains. A polymer was analogous to a necklace made from many small beads of monomers (Charles, 2003). Polymers were substances whose molecules have high molar mass. There were many types of polymers including synthetic and natural polymers. Synthetic polymer have been commercializes and produced in very large scale. The materials commonly called plastics. Plastics play a significant role in our daily lives. Their applications were nearly universal such as components in automobiles, home appliances, computer equipments, packages and even medical applications were areas where plastics clearly have become indispensable. Among of natural polymers were proteins, starch, cellulose, and latex. Besides, natural polymers include such things as tar and shellac, tortoise shell and horns, as well as tree saps that produce amber and latex. These polymers were processed with heat and pressure into useful articles like hair ornaments and jewelry. Natural polymers began to be chemically modified during the 1800s to produce many materials. The most famous of these were vulcanized rubber, gun cotton, and celluloid. The first semi-synthetic polymer produced was Bakelite in 1909 and was soon followed by the first synthetic

fiber, rayon, which was developed in 1911 (American Chemistry Council , 2007). Polymers affect every day of our life. These materials have so many varied characteristics and applications that their usefulness can only be measured by our imagination. Polymer was the materials of past, present, and future generations.

2.1.2 Types of Polymers

Popular usage of polymer was plastic that actually refers to a large class of natural and synthetic materials with a variety of properties. The word “polymer” was derived from the Greek *poly* and *meros*, meaning many and parts, respectively. Some scientists prefer to use the word “macromolecule” or large molecule, instead of polymer. Others maintain that naturally occurring polymers, or biopolymers, and synthetic polymers should be studied in different courses. Others name these large molecules simply “giant molecules”. However, the same principles apply to all polymers.

2.1.2.1 Synthetic Polymers

Another common name for many synthetic polymers was plastic which comes from the Greek word "plastikos", suitable for molding or shaping. The synthetic polymers include synthetic rubber, Bakelite, neoprene, nylon, PVC, polystyrene, polyethylene, polypropylene, PVB, silicone, and many more (Charles, 2003). Plastics have become one of the most widely used materials all over the world. Many materials in our daily use from packing, wrapping, and building materials include half of all polymers synthesized. Other uses include automotive, sport equipments, textiles, and infrastructure are made from polymers.

2.1.2.2 Natural Polymers

Natural polymer was a polymer resulting from raw materials found in nature such as shellac, amber, and natural rubber. A variety of other natural polymers exist, such as cellulose, which is the main constituent of wood and paper. Other Natural polymer was rubber (hydrocarbon base) and silicones (alternating silicon and oxygen) (Charles, 2003).

2.1.2.3 Biopolymers

Biopolymers were produced by living organisms. Biopolymers such as proteins and nucleic acids play crucial roles in biological processes. Cellulose was the most common biopolymer and about 33 percent of all plant matter was cellulose (the cellulose content of cotton is 90 percent and that of wood is 50 percent) (D. Lukkassen and A. Meidell, 2007). As illustrated in Figure 2.1 was the classification of biopolymers and their nomenclature.

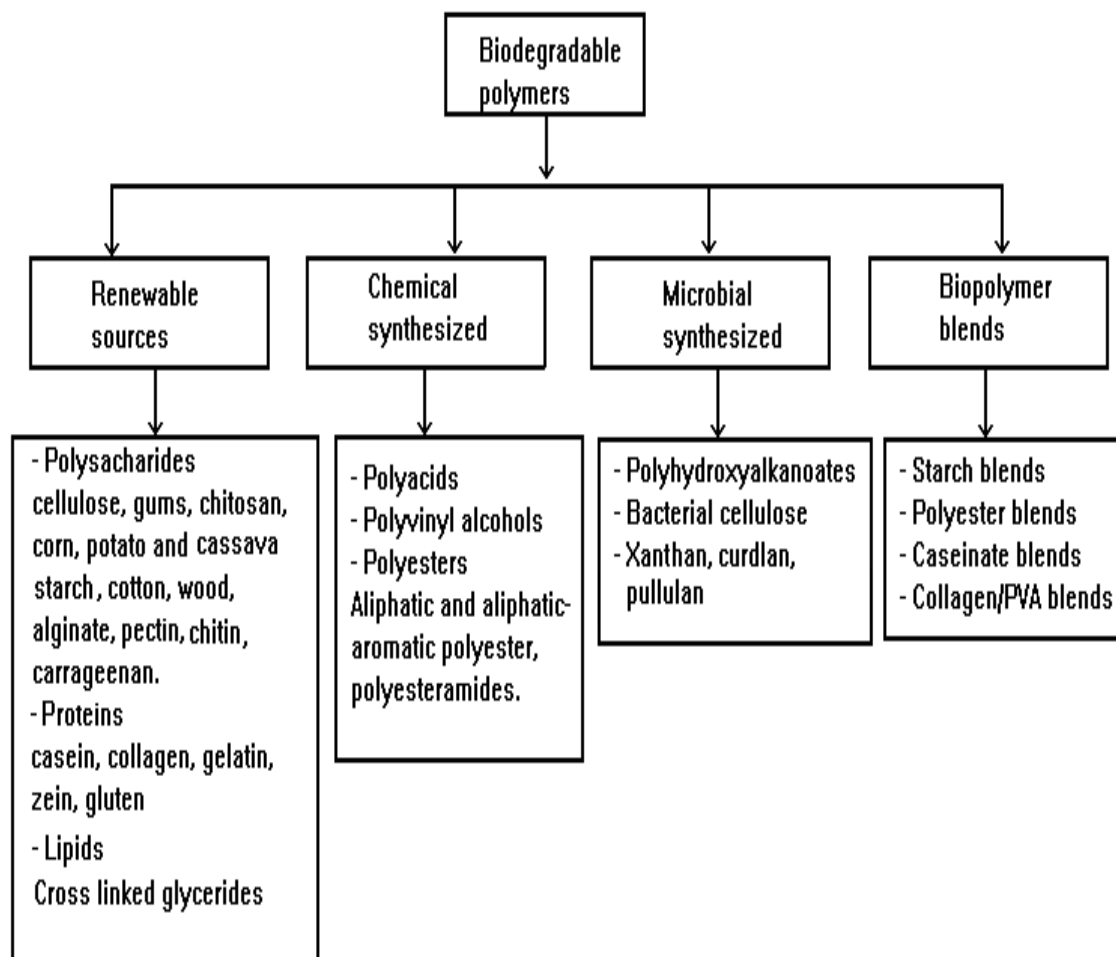


Figure 2.1 - Classification of biopolymers and their nomenclature (Kestur *et al.*, 2008)

2.1.3 Characteristic of Polymers

Polymers were divided into two broad categories: thermoplastics and thermosets. The majority of polymers were thermoplastic, meaning that once the polymer was formed it can be heated and reformed over and over again. This property allows for easy processing and recycling. In a thermosets polymer, it cannot be remelted by the application of heat. Once these polymers are formed, reheating will cause the

material to scorch. Every polymer has very distinct characteristics, but most polymers have the following general attributes (American Chemistry Council , 2007).

- Polymers can be very resistant to chemicals.
- Polymers can be both thermal and electrical insulators.
- Generally, polymers were very light in mass with varying degrees of strength.
- Polymers can be processed in various ways to produce thin fibers or very intricate parts.

2.2 Starch

2.2.1 Introduction

Starch was the cheapest and most abundant food biopolymer worldwide. Pure starch was a white, tasteless and odorless powder. It occurs in a variety of botanical sources including corn, potato, wheat and rice. It has many applications ranging from cereals, snacks and thickeners in the food industry. Other applications include packaging, paper and adhesives in the non-food industry. Among these, starch was a potentially useful material for biodegradable polymers because it was natural abundance and low cost. Starch was the major carbohydrate in plant tubes and seed endosperm, where it was found as granules. Each granule contains amylopectin molecules together with a larger number of smaller amylose molecules (N. Canigueral *et al.*, 2009).

For certain applications in the food and packaging industries, starch was extruded to achieve a desired product texture and quality. Some work has already been carried out and published on extrusion and extrusion expansion in general. However, there were many more challenges in the study of starch processing such as the compositional, structural and rheological complexities of starch systems, as well as inhomogenities encountered during processing. An understanding of the mechanisms

involved in the processing and expansion of starch is therefore required. Such insight would help optimize existing extrusion processes through better equipment design and applications, and develop new products with desired characteristics that would benefit both the consumers and the industry (Nowjee, 2004).

2.2.2 Composition and Structure

Starch was produced as granules in most plants cells and was referred to as native when in this particular granular state. Native starches from different botanical sources vary widely in structure and composition, but all granules consist of two major molecular components, amylose (20-30%) and amylopectin (70-80%), both of which were polymers of α -D-glucose units in the 4C_1 conformation. In amylose (Figure 2.2), these were linked $-(1 \rightarrow 4)-$, with the ring oxygen atoms all on the same side, whereas in amylopectin about one residue in every twenty is also linked $-(1 \rightarrow 6)-$ forming branch-points as shown in Figure 2.3. (Nowjee, 2004).

Several investigations have been carried out to establish the level of organization within the starch granules. Techniques used vary from X-ray diffraction experiments to atomic force microscopy (AFM) and transmission electron microscopy (TEM). In the native form of starch, amylose and amylopectin molecules were organized in granules as alternating semi-crystalline and amorphous layers that form growth rings as illustrated in Figure 2.4. The semi-crystalline layer consists of ordered regions composed of double helices formed by short amylopectin branches, most of which were further ordered into crystalline structures known as the crystalline lamellae. The amorphous regions of the semi-crystalline layers and the amorphous layers were composed of amylose and non-ordered amylopectin branches (Nowjee, 2004).

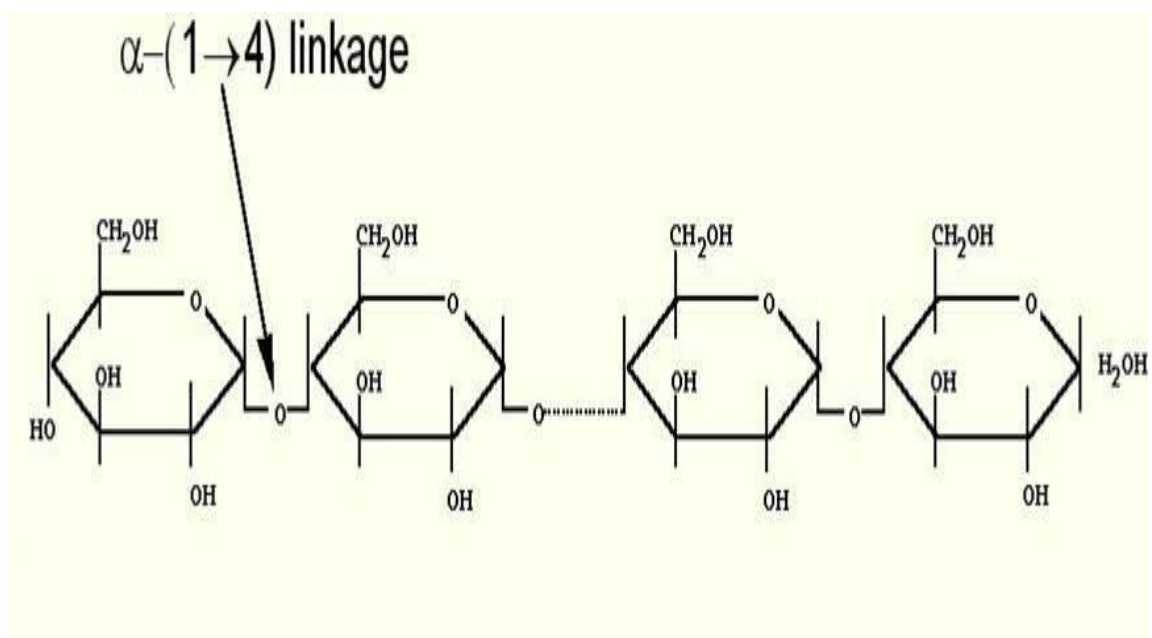


Figure 2.2 - Amylose molecule (Nowjee, 2004).

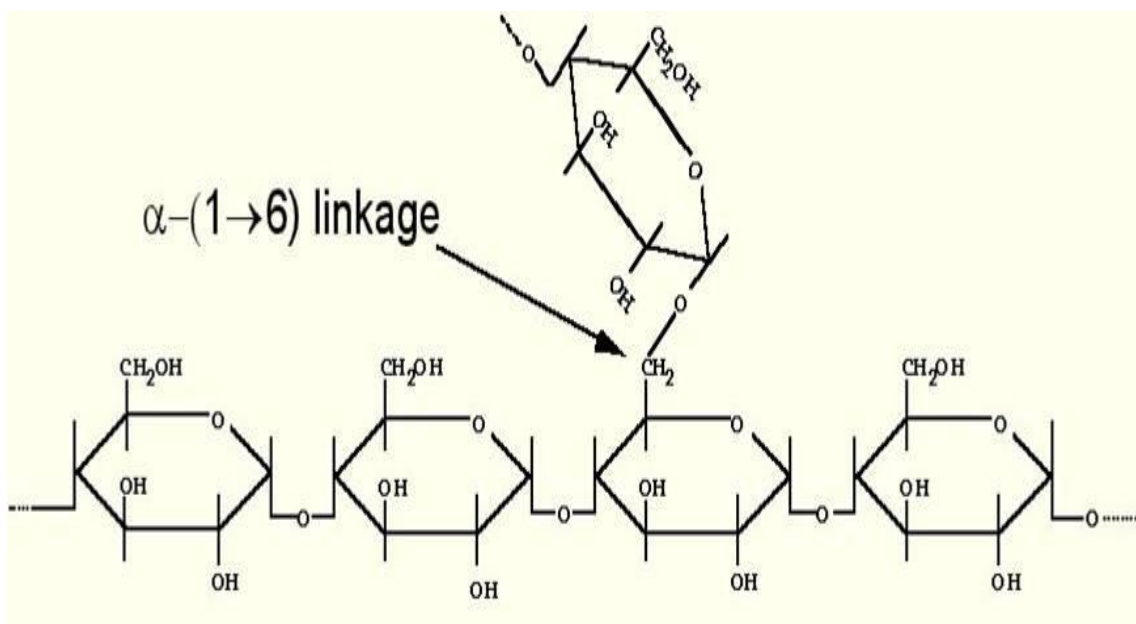


Figure 2.3 - Amylopectin molecule (Nowjee, 2004).

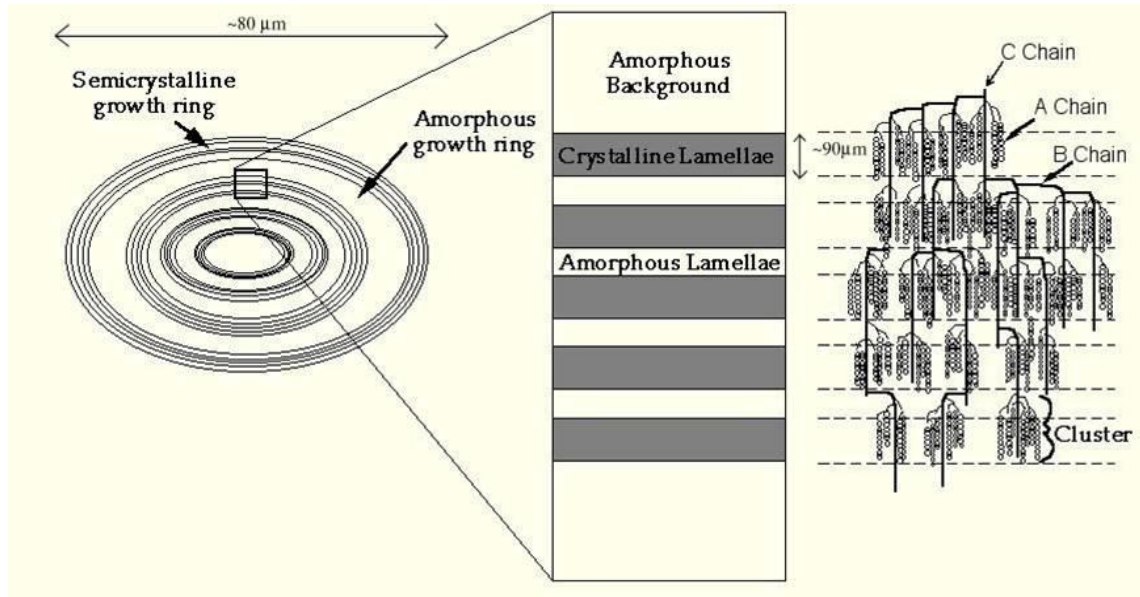


Figure 2.4: Schematic view of the structure of a starch granule, with alternating amorphous and semi-crystalline zones constituting the growth rings (Nowjee, 2004).

2.3 Fiber

2.3.1 Overview

Fibers were the principal constituents in a fiber-reinforced composite material. Fibers were the reinforcement and the main source of strength while the matrix glues all the fibers together in shape and transfers stresses between the reinforcing fibers. Fiber was a class of materials that were continuous filaments or were in discrete elongated pieces, similar to lengths of thread. They occupy the largest volume fraction in a composite laminate and share the major portion of the load acting on a composite structure. Proper selection of the fiber type, fiber volume fraction, fiber length, and fiber orientation was very important, since it influences the following characteristic of a composite laminate (Mallick, 2008). Figure 2.5 show the characteristic of a composite laminate. As illustrated in Figure 2.6 was the types of common fiber used in industry.

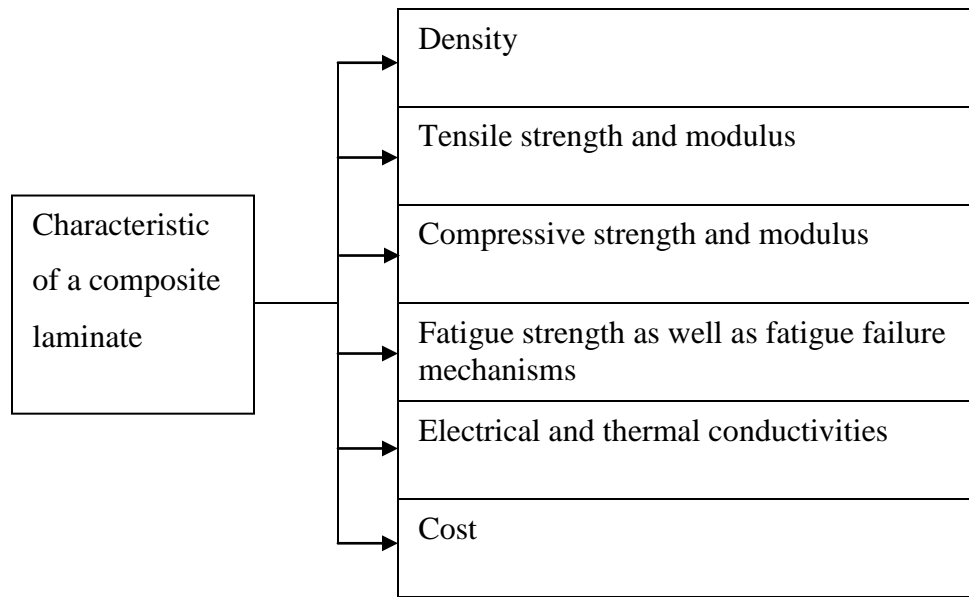


Figure 2.5: Characteristic of a composite laminate (Mallick, 2008)

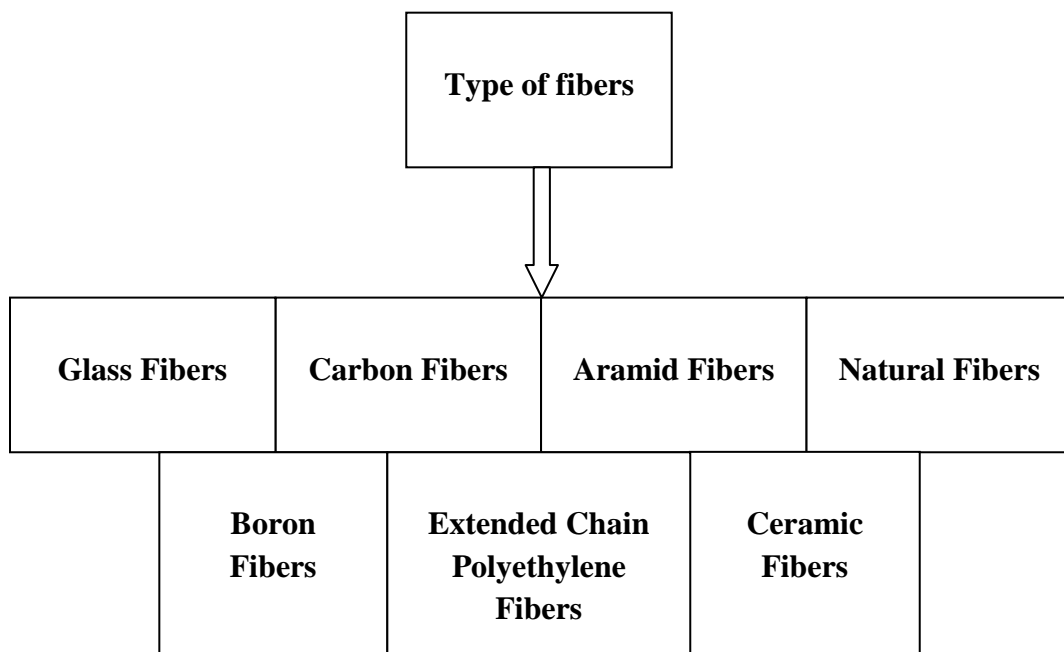


Figure 2.6: Type of fiber (Goulart *et al.*, 2000)

2.3.2 Natural fibers as Reinforcements in Bio-composite

2.3.2.1 Natural fiber classification

Natural fibers were subdivided based on their origins, whether they were derived from plants (Cellulose or Lignocelluloses), animals (Protein), or minerals. Natural fibers can be classified according to their origin. Plants fibers include bast (or stem or soft or sclerenchyma) fibers, leaf or hard fibers, seed, fruit, wood, cereal straw, and other grass fibers. Animal fibers include silk, wool and hair. While mineral fiber was include asbestos, fibrous brucite, and wollastonite (K. Mohanty *et al.*, 2005).

2.3.2.2 Coconut Husk or Coir

Coir fiber was obtained from the fibrous husk of the coconut from the coconut palm, which belongs to the palm family (Palmae). Coir fibers were obtained from the fibrous husk (mesocarp) encasing the fruit of the coconut palm, which was a by-product of the copra extraction process. The term coir was derived from kayar, a rope or cord, and kayaru, meaning to be twisted. Coconut palms were cultivated throughout tropical countries mostly for the high oil content of the endosperm (copra). The oil was widely used in both the food and nonfood industries (e.g. surfactant production). On average, from 100 coconuts 7.5 to 8.2 kg of coir fibers can be obtained (K. Mohanty *et al.*, 2005).

Coir fibers have high lignin but low cellulose content, as a result of which the fibers were resilient, strong, and highly durable. Coir was the one of the toughest plant fibers available. It does not pill and highly abrasion and rot (fungal and bacteria) resistant (Goulart *et al.*, 2000). Furthermore, coir was naturally insulating and sound absorbing, antistatic, and difficult to ignite. Due to the ability of coir fibers to tolerate water immersion for months without disintegrating, they find many applications as

horticulture and erosion control product (geotextiles). Recently, the academic and industrial R&D communities have begun seeking ways to develop new application for coir as reinforcement for polymers (K. Mohanty *et al.*, 2005).

2.3.3 Fiber Treatment and Modification

It was well known that the performance of composites depends on the properties of the individual components and their interfacial compatibility. For numerous applications plant fibers have to be prepared or modified with the following considerations in mind; homogenization of the properties of the fibers, degree of elementarization and degumming, degree of polymerization and crystallization, good adhesion between fiber and matrix, moisture repellence, and flame-retardant properties (George *et al.*, 2001).

These properties can be partly influenced by different fiber separation procedures but subsequent fiber treatment processes were more influential. Several noncellulose components have to be removed to assure the compatibility of the plant fibers to the surrounding polymer matrix. Alkalization, washing, or boiling of the plant fibers in 2 to 10% sodium, potassium, or lithium hydroxide solutions leads to the removal of unwanted fiber components, which dissolve during the process. Alkalization, depending on the concentration of the alkali and the process temperature, can significantly improve the fiber's mechanical and surface properties (K. Mohanty *et al.*, 2005).