

**CHARACTERIZATION OF OIL PALM SHELL
NANO FILLER IN HYBRID KENAF/COCONUT
FIBRES REINFORCED POLYESTER
COMPOSITES**

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**UNIVERSITI SAINS MALAYSIA
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By

WAN NORNA DIRAH BINTI WAN OTHMAN

**Thesis submitted in fulfillment of the requirements
for the degree of Masters of Sciences**

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LIST OF ABBREVIATIONS

ASTM	American Society for Testing and Materials
CC	Calcium carbonate
CMC	Ceramic Matrix Composites
CNT	Carbon Nanotubes
EFB	Empty Fruit Bunches
EDX	Energy Dispersion of X-Ray
FT-IR	Fourier Transform Infrared Spectroscopy
GPa	Giga Pascal
MEKP	Methyl Ethyl Ketone Peroxide
MMC	Metal Matrix Composites
MMT	Montmorillonite
MPa	Mega Pascal
OPF	Oil Palm Fronds
OPS	Oil Palm Shell
OPT	Oil Palm Trunks
PHMS	polyhydromethylsiloxane
SEM	Scanning Electron Microscope
TEM	Transmission Electron Microscopy
TGA	Thermogravimetric Analysis
XRD	X-Ray Diffraction

LIST OF SYMBOLS

%	Percentage
°C	Degree Celsius
cm	Centimeter
d	Thickness
g	Gram
J	Joule
kg	Kilogram
M	Mega
m	Meter
m	Mass
mg	Miligram
mm	Milimeter
nm	Nanometer
T _g	Glass Transition Temperature
V	Volume
V _f	Volume Fraction
W _f	Weight Fraction
ρ	Density
ρ _c	Composite Density
ρ _f	Filler Density

LIST OF PUBLICATIONS

APPENDIX A		Pages
1.1	WAN NADIRAH, W. O., JAWAID, M., ABEER A. AL MASRI, ABDUL KHALIL, H. P. S., SUHAILY S. S. 2011. Cell wall morphology, chemical and thermal analysis of cultivated pineapple leaf fibres for industrial applications. <i>Journal of Polymers and The Environment.</i> 20, 404 – 411.	102
1.2	NAZRUL ISLAM, MD., DUNGANI, R., ABDUL KHALIL, H.P.S., SITI ALWANI, M., WAN NADIRAH, W.O., FIZREE, H.M. 2013. Natural studies of oil palm trunk lumber (OPTL) green polymer composites enhanced with oil palm shell (OPS) nanoparticles. <i>SpringerPlus.</i> 2, 592.	103
1.3	WAN NADIRAH, W. O., ABDUL KHALIL, H. P. S., JAWAID, M., NURULHUDA, A. M. (2012). The effect of physical and mechanical properties on hybrid empty fruit bunches fibres/bamboo strips reinforced polyester composites. Paper presented at 8 th Asian-Australian Conference on Composite Materials (ACCM8), Kuala Lumpur, Malaysia.	104

**PENCIRIAN PENGISI NANO TEMPURUNG KELAPA SAWIT DI DALAM
HIBRID GENTIAN KENAF/KELAPA YANG DIPERKUAT KOMPOSIT
POLIESTER**

ABSTRAK

Dalam kajian ini, komposit poliester yang diperkuat gentian kenaf/kelapa hibrid telah dibangunkan dan ditingkatkan lagi dengan kemasukan pengisi nano tempurung kelapa sawit (OPS). Penukaran OPS mentah kepada partikel nano OPS adalah melalui proses pengisaran diikuti dengan proses pengilangan bola bertenaga tinggi selama 30jam. Pengisi nano OPS yang telah dihasilkan dinilai berdasarkan ciri-ciri seperti morfologi permukaan, unsur-unsur komposisi dan kumpulan berfungsi dengan menggunakan Pengimbas Mikroskopi Elektron dilengkapi Tenaga Sebaran Sinar-X (SEM-EDX) dan Spektroskopi Inframerah Transformasi Fourier (FT-IR). Imej dari SEM-EDX menunjukkan bahawa pengisi nano OPS yang dihasilkan mempunyai struktur bentuk hancur dan tidak teratur bersama-sama dengan kehadiran karbon dan oksigen sebagai komposisi utama. Selain itu, Penghantar Mikroskopi Elektron (TEM), Analisis Belauan Sinar-X (XRD) dan Analisis Termogravimetri (TGA) telah digunakan untuk mencirikan saiz zarah, indeks penghaburan dan sifat haba pengisi nano OPS. Mikrograf TEM menunjukkan bahawa saiz pengisi nano OPS adalah diantara 10 hingga 30 nm dimana ini menunjukkan sifat nano mereka. Analisis yang dijalankan oleh XRD menunjukkan pengisi nano OPS mempunyai darjah indeks penghaburan yang rendah iaitu sebanyak 34.56%. Kesan penambahan pengisi nano OPS yang berbeza (0, 1, 3 dan 5 berat peratus) ke dalam hibrid gentian kenaf / kelapa yang diperkuat poliester nanokomposit telah dikaji. Hibrid nano komposit ini telah

disediakan dengan menggunakan kaedah ‘hand lay-up’ dan dibiarkan matang pada suhu bilik selama 24 jam. Sifat hibrid nanokomposit seperti fizikal, mekanikal, morfologi dan sifat haba telah dikaji. Dapat diperhatikan bahawa kemasukan pengisi nano OPS ke dalam hibrid nanokomposit telah meningkatkan sifat-sifat fizikal seperti ketumpatan, kandungan ruang kosong dan penyerapan air seiring dengan peningkatan pengisi muatan. Untuk sifat-sifat mekanik, kekuatan tegangan, lenturan dan hentaman serta modulus tegangan dan lenturan hibrid nanokomposit bertambah apabila pengisi muatan bertambah sehingga 3% dan kemudian menurun pada pengisi muatan 5%. Walaubagaimanapun, aliran bertentangan dipamerkan oleh pemanjangan pada takat putus dimana penambahan pengisi muatan mengurangkan pemanjangan pada takat putus. Sifat-sifat kestabilan terma bagi hibrid nanokomposit mempamerkan aliran yang sama dengan sifat-sifat mekanikal. Pada morfologi struktur hentaman, 3% muatan pengisi untuk hibrid nanokomposit menunjukkan ruang kosong minimum yang lebih kecil, dan permukaan matriks dibentuk dengan baik dan kehadiran gentian patah dan bukannya gentian tarik keluar yang menunjukkan bahawa tekanan telah berjaya diserap oleh gentian. Oleh itu, penambahan sebanyak 3% muatan pengisi nano OPS dipercayai adalah muatan optimum untuk hibrid nanokomposit dan menunjukkan ciri-ciri yang luar biasa berbanding dengan muatan pengisi nano yang lain.

CHARACTERIZATION OF OIL PALM SHELL NANO FILLER IN HYBRID KENAF/COCONUT FIBRES REINFORCED POLYESTER COMPOSITES

ABSTRACT

In this research, hybrid kenaf/coconut fibres reinforced polyester composites were developed and enhanced with nano filler Oil Palm Shell (OPS). The raw OPS was subjected to grinding followed by high-energy ball milling for 30 hours to become OPS nano particles. Characterization of OPS nano filler such as surface morphologies, elemental composition and functional groups were evaluated by using Scanning Electron Microscopy equipped with Energy Dispersive X-ray Analysis (SEM with EDX), Fourier Transform Infrared Spectroscopy (FT-IR) respectively. The SEM-EDX images revealed that OPS nano filler produced consisted of crushed and irregular shape structures with presence of carbon and oxygen as a major elements composition. Besides, Transmission Electron Microscope (TEM), X-ray Diffraction (XRD) and Thermogravimetric Analysis (TGA) were used to characterize particle size, crystallinity index and thermal properties of OPS nano filler respectively. The TEM micrograph revealed that OPS particles size ranged between 10 to 30 nm indicates their nanometric nature. Percentage crystallinity of OPS nano filler by XRD analysis comes out to be 34.56% show its lower degree of crystallinity index. Effect of different filler loading (0, 1, 3 and 5 wt %) of OPS nano filler into hybrid kenaf/coconut fiber reinforced polyester nanocomposites were studied. These hybrid nanocomposites were prepared by using hand lay-up techniques and left cured at room temperature for 24 hours. The physical, mechanical, morphology and thermal properties of hybrid nanocomposites were studied. It was observed that the

incorporation OPS nano filler into hybrid nanocomposites increased physical properties such as the density, void content and water absorption of composites as filler loading increases. As for mechanical properties, the strength of tensile, flexural and impact as well as modulus of tensile and flexural of hybrid nanocomposites increases as the filler loading increases up to 3% filler loading and then decrease. However, the opposite trends exhibited by elongation at break properties where increments of filler loading reduce the elongation at break. The thermal stability properties of hybrid nanocomposites exhibit the similar trends with mechanical properties. On morphological of impart structures, 3% filler loading of hybrid nanocomposites showed smaller minimum voids, well skin matrix formed and presence of fibre fracture instead of fibre pull out which indicated that the stress was successfully absorbed by the fibre. Therefore, addition of 3% OPS filler loading were believed to be optimum for hybrid nanocomposites and shows remarkable properties compared to other filler loading.

CHAPTER ONE

INTRODUCTION

1.1 Overview

Rapidly growing economical and population growth from day to day life has lead to the unstable ecological and environmental sustainability such as wood scarcity, depletion of fossils and environmental degradation. Taking into consideration of this context, bio-based materials in general and agricultural waste in particular appear as a subject of interest nowadays. Utilization of these materials not only reduces the numerous environmental issues but also contributes the major effect in respect of cost, technological and scientific point of view as well.

Natural plant fibres have good qualities in terms of low density, low cost, specific strength, economical viability, environmental friendly, biodegradability, renewability and enhanced energy recovery (Saheb & Jog, 1999). For this reasons, natural fibres have gained interest that make them suitable for fabrication of composites applications. The common natural fibres include hemp, jute, flex, coconut, bamboo, kenaf, sisal and cotton. Within that mixed portfolio, kenaf and coconut fibres are one of the important natural resources which can be principal sources for fibres and other products (Wambua et al., 2003).

Kenaf (*Hibiscus Cannabinus L.*) is a herbaceous fast growing annual crop spring that can grows to a height of 4-5 m in about 4-5 months growing season. Currently, more countries pay more attention to kenaf research and its cultivation whereby over than 20 countries mainly China, India, Thailand and Vietnam has commercialize kenaf cultivation (IJSG, 2012). In 2010, India has been recognized as

top producer of kenaf with 140 000 MT, followed by China (75 000MT) and Thailand (18 000MT) (IJSG, 2012). It is commonly known as ‘the future crop’ on account to its excellent cellulose fibres sources. In addition, kenaf fibre is one of the best reinforcement choices in biocomposites fields (Intan Syafinaz et al., 2012; Norlin et al., 2009).

Coconut fibre (*Cocos Nucifera*) or best known as Coir fibre is obtained from the husk (mesocarp) of coconut. Coconut fibres are produced annually around the world especially in Indonesia, India and Sri Lanka. Indonesia lead the coconut production with about 1800000 MT production in 2012 (FAO, 2012). In general, coconut fibres have excellent sound and temperature insulation, not influenced by moisture and dampness and so on. Hence, it is attractive for certain technical purpose such as wall panel lings and floor coverings especially in auditoria. In addition, coconut fibres as reinforcement in polymer composites have potential for further utilization (Onuegbu et al., 2013; Santafé Júnior et al., 2010).

In Malaysia, palm oil production has given rise for several years from 6.1 million tones in 1990 to 16.9 million tones in 2010 and to 18.9 million tones in 2011. The production is reached 19.2 million tones in 2013 (MPOB, 2012). Oil palm shell (OPS) is an agricultural waste produced after oil extraction from fresh fruit bunch (FFB) of oil palm. The wide production of oil palm in Malaysia years by years had indicated tones of waste including OPS have been created each year. Normally, this waste is disposed through incineration and sometimes just left on the ground without proper management which in turn gives rise to the major pollutions and disposal problems. This abundant residue available has provided a golden opportunity for scientist to convert the waste into wealth by producing an alternative raw material for new applications and value added products.

The field of nanotechnology is one of the most popular scopes for current research and development. Nano materials are main component in manufacturing polymer nanocomposite whereby terms ‘nano’ refer to the smallest length below than 100 nm (Koo, 2006). Having the uniques characteristics, nanomaterials have offered superior properties to its final product produced. Polymer nanocomposites have been proposed for numerous applications range from automotive applications to advanced optoelectronic device.

Hybrid concept is a system in which presence of two or more reinforcement and filled materials in a single matrix. The behavior of hybrid composites is a weighed sum of the individual components whereby there is a more beneficial balance between the natural advantages and disadvantages. Through hybridization, advantages of one fibres could complement what are lacking in other fibres and vice versa (John et al., 2009).

Combination of different kinds of material properties attributed to increment in demand for unique materials whereby lead to the idea of new composites. The idea of combining filler-resin as material of matrix with reinforcing fibre in new three-phased composites reinforcement is still infancy but shows high potential to be very successful (Njuguna et al., 2008). This new family of composites materials exhibit remarkable improvement especially in mechanical and thermal resistance in comparison with existing composites performances. Consequently, a new material that full filled desired properties, meet the industrial requirement and at the same time maintaining the ecological and environmental sustainability can be successfully produced.

1.2 Problem Statement

Natural fibres are seen as promising raw material to be used as reinforcement in polymer composites due to its low density, recyclability and biodegradability. For this reason, natural fibres reinforced polymer composites have been gained important focus of extensive research especially in automotive, building and construction applications. In spite its numerous advantages, certain drawback were reported such as lacks of good interfacial adhesion, low modulus elasticity, low melting point and so on therefore make the use of natural fibres reinforced polymer composites less attractive.

To be realized with this scenario, nano particles have been identified as a new tool for enhancement in natural fibre reinforced polymer composites properties. To the best of our knowledges, there are many published research work in nano area, but very limited research has been conducted on using oil palm shell (OPS) as nano filler in biocomposites. Apart from that, this is a novelty research whereby agriculture waste is converted into nanomaterials despite various commercialized man made nano materials in nano technological fields.

To fill up this gap, this research therefore is aiming to produce OPS as bio nanomaterials and incorporate into kenaf/coconut fibre reinforced polyester nanocomposites. The incorporation of nano filler in hybrid composites is expected to enhance the strength and properties of hybrid nanocomposites produced. Hence, the hybrid nanocomposite can used for advanced applications and eventually reduce world's abundant agricultural waste problems.

1.3 Objectives of The Study

The objectives of this present study are:

- To characterize fundamental properties of nano oil palm shell (OPS).
- To develop hybrid kenaf/coconut fibre reinforced polyester composites enhanced with OPS nano filler.
- To study effect of OPS nano filler loading percentage on physical, mechanical, thermal and morphology of the hybrid kenaf/coconut fibre reinforced polyester composites.

1.4 Organization of Thesis

This thesis has been organized into five different chapters.

Chapter 1 – Focused on background, problem statement and objectives of present study.

Chapter 2 – Focused on literature review of present study. Overview of composites and its classification, reinforcement's raw materials, oil palm shell (OPS), nanomaterial, polymer nanocomposites, and hybrid composites are reported.

Chapter 3 – Explains about materials and methodology nanomaterial's production, manufacturing and characterization of hybrid nanocomposites.

Chapter 4 – Provide the results and discussion of characterization of nano-structured OPS and nano-structured OPS in hybrid kenaf/coconut fibre reinforced polyester nanocomposites.

Chapter 5 – Summarizes the overall conclusions and recommendation for future research of this study.

CHAPTER TWO

LITERATURE REVIEW

2.1 Composites

2.1.1 Definition

Nowadays, it shows that composites are one of the choice of materials for various engineering applications (Mazumdar, 2002). A composite material is produced by combination of two or more materials clearly separated one from another in order to create a special combination of properties. Typically, most composite is formed by two constituent materials which are reinforcement components and matrix or binder. In composite concept, matrix/binder functioned as load transfer while reinforcing element functioned as load carrier.

2.1.2 Classification

Classification systems of composite materials are classified based on matrix material or reinforcing material structure. There are three types of categories under matrix material systems which are known as Metal Matrix Composites (MMC), Ceramic Matrix Composites (CMC) and Polymer Matrix Composites (PMC) (Pandey, 2004). In reinforcing material structure, it is categorized as particulate reinforced composites, fibre reinforced composites and laminar composites (Pandey, 2004).

Figure 2.1 illustrates the classification of composites system.

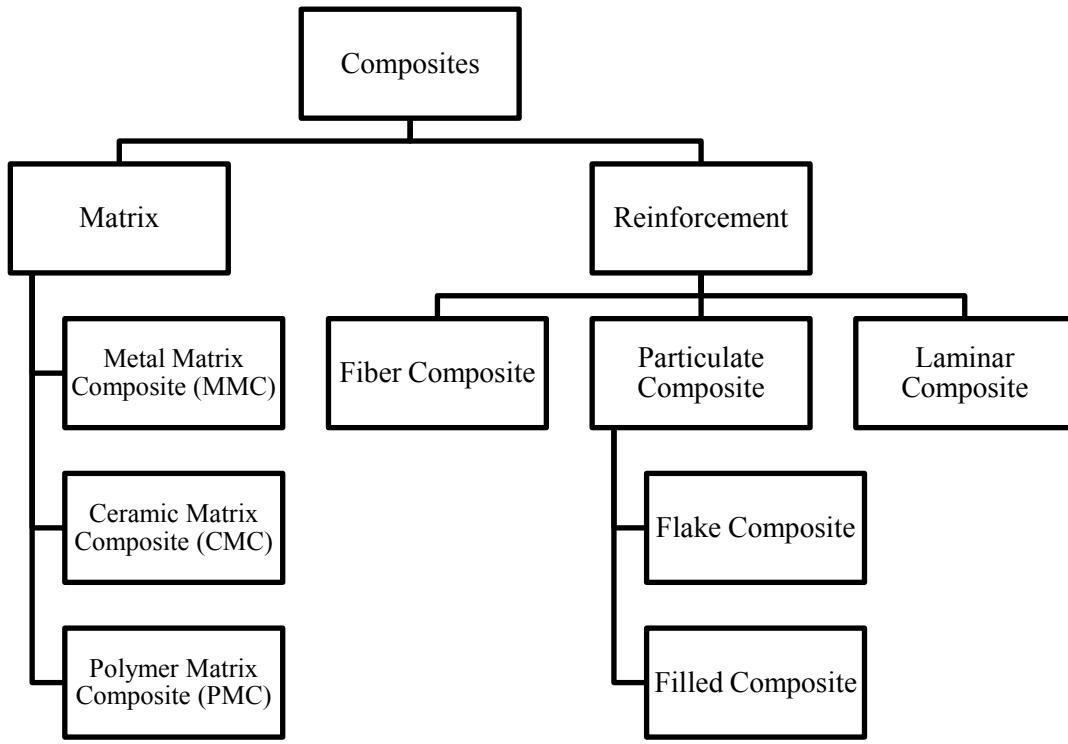


Figure 2.1: Classification of composites system (Pandey, 2004)

2.1.3 Applications

In several industries, composite materials have become the primary materials of choice. Broadly speaking, the composites applications can be categorized into the differ industry types such as automotive, aerospace, marine, construction, corrosion-resistant equipment, consumer products, appliance/business equipment and others. In 2000, transportation industry used 1.3 billion pounds of composites thereby make it one of the largest user of composite materials (Mazumdar, 2002).

2.2 Matrix

The matrix material act as important parts to obtain advanced properties of composite construction. The matrix material surrounds and supports the reinforcing materials by sustaining their relative position (Cleveland, 2008). The main function of the matrix is to transfer the load to the reinforcement elements. Besides, it also gives protection to reinforcing elements against chemical attack and mechanical damage (wear) (Akovali, 2001). The matrix is divided into two types which are thermoplastic and thermoset resins. Thermoplastic is a resin that will soften when heated and harden when cooled. Meanwhile, thermoset resin which initially is a liquid will be cured by either application of heat or catalyst (Osman et al., 2012). Solid thermoset resin cannot be converted back to their original liquid form once cured.

2.2.1 Thermoset-based Matrix

Thermoset-based matrix is the most widely used resins in composites. Typically, thermoset resin initially is a low molecular-weight liquid chemical with very low viscosities (Schwartz, 1997). It contain polymer that undergo chemical reaction thereby cross-link the polymer chain together during the curing process. Thus, the entire matrix is connecting together in a three dimensional cross-linked structure (Gibbons, 1988). As a result, it have high tendency to enhance its properties such as high dimensional stability, high temperature resistance and good resistance to solvents, chemicals as well as structural integrity. The purpose of cross linking process is to eliminate the risk of the product re-melting when heat is applied. It is irreversible, and cannot be remolded, recycled or reshaped. Thermoset resins include polyester, epoxy, vinyl esters, polyimide and polyurethane (Pandey, 2004).

2.2.1.1 Unsaturated Polyester Resin

Unsaturated polyester resin is a heterochain macromolecules that have carboxylate ester groups as a primary element of their polymer backbones (Dholakiya, 2012). This resin is the condensation products of unsaturated acids or anhydrides and diols with/without diacids. Presence of unsaturated in this polymer prepares a site for following cross-linking (Boening, 1964; Parkyn et al., 1967). Figure 2.2 presents idealized chemical structure of a typical unsaturated polyester resin (Ray & Rout, 2005).

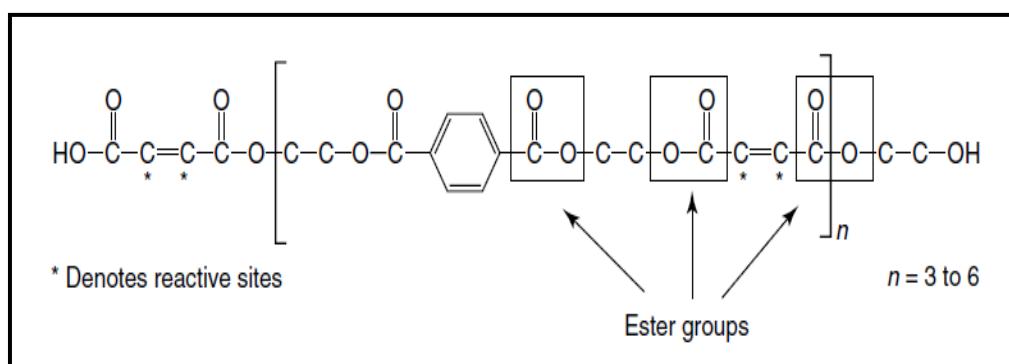


Figure 2.2: Idealized chemical structure of a typical unsaturated polyester resin (Ray & Rout, 2005)

The cross linking reaction of polyester resin is a free radical chain growth copolymerization (Ruban et al., 2014). Cross linking action of this resin has five different steps known as induction, microgel formation, transition with or without phase separation, macro gelation and post gelation. Cross linking position for network development prepared by the polyester oligomer while linear chain extension provided by the styrene monomer (Liqun & James, 2005). Normally, styrene mixed with polyester to form a reactive resin solution. In this case, in order the resin can be processed, the styrene plays role as a both cross linking agent and a viscosity reducer

(Dholakiya, 2012). However, styrene monomer contributes to a problem of environmental and occupational health causes by evaporation and emissions, which occur during the resin processing.

Polyester resin can be further classified into three categories which are Ortho-phthalic polyesters, Iso-phthalic polyesters and Tere-phthalic polyesters. Ortho-phthalic polyesters is the typically used standard economic resin and it generate highly rigid products meanwhile Iso-phthalic polyesters is the preferable material in the marine industry where advanced water resistance is required (Ray & Rout, 2005). Tere-phthalic polyesters are considered a specialty resin currently made in small volumes.

This resin has low cost, high strength, high durable structure and ease processing, despite its brittleness characteristics. For this reasons, polyester resin known as one of the primary material candidates used in wide range of industrial applications especially in advanced structural composites. According to Mark et al. (1988), approximately 75% of the entire thermoset composites, marked in tonnage represented by polyester resin.

2.3 Reinforcements

Reinforcements are discrete inclusions used to improve the structural characteristics of a material. The strength and stiffness is provided by the reinforcement components (Gibbons, 1988). In most cases, the reinforcement is harder, stronger and stiffer than the capability of matrix to change failure mechanism to the beneficial of the composite. As illustrated in Figure 2.1, reinforcement

components are usually fibre, particulate or laminate. Particulate reinforcements have approximately equaled in all directions of dimensions. They might be in form of spherical, platelets or any other regular or irregular geometry. In contrast, the length of fibre reinforcement is much greater than its diameter. Length-to-diameter (lld) ratio is defined as *Aspect ratio* can be varying in different form of fibres. They can be categorized in continuous or discontinuous form. Continuous fibres have long aspect ratio with preferred orientation while discontinuous fibres have short aspect ratio with random orientation. Examples of continuous fibre reinforcement are unidirectional, woven cloth and helical winding while examples of discontinuous reinforcements are chopped fibres and random mat (Campbell, 2010).

2.3.1 Natural Fibres

2.3.1.1 Source, Classification, and Applications

Recently, natural fibres have received attention from the generated research and development community. Natural fibres are distinguished according to their origins, obtained from plants, animals or minerals. All plant fibres are composed of cellulose while animal fibres consist of protein (hair, silk and wool). Plant fibres consist of bast (or stem or soft sclerenchyma) fibres, leaf or hard fibres. Figure 2.3 shows classification of natural fibres (Jawaid & Abdul Khalil, 2011).

Currently, abundant of research material is being generated on the potential of lignocelluloses based fibres as reinforcement for plastics. Technically speaking, almost any agricultural fibre can be used to manufacture composite panels (English et al., 1997). The utilization of such materials in composites has enlarged in respect to their relative cheapness, their ability to recycle, abundant, non abrasive, non

hazardous and for the fact that they can compete well in terms of strength per weight of material (John & Thomas, 2008). Taking into consideration of arising environmental awareness, natural fibres is one the most promising material in advanced composites engineering applications.

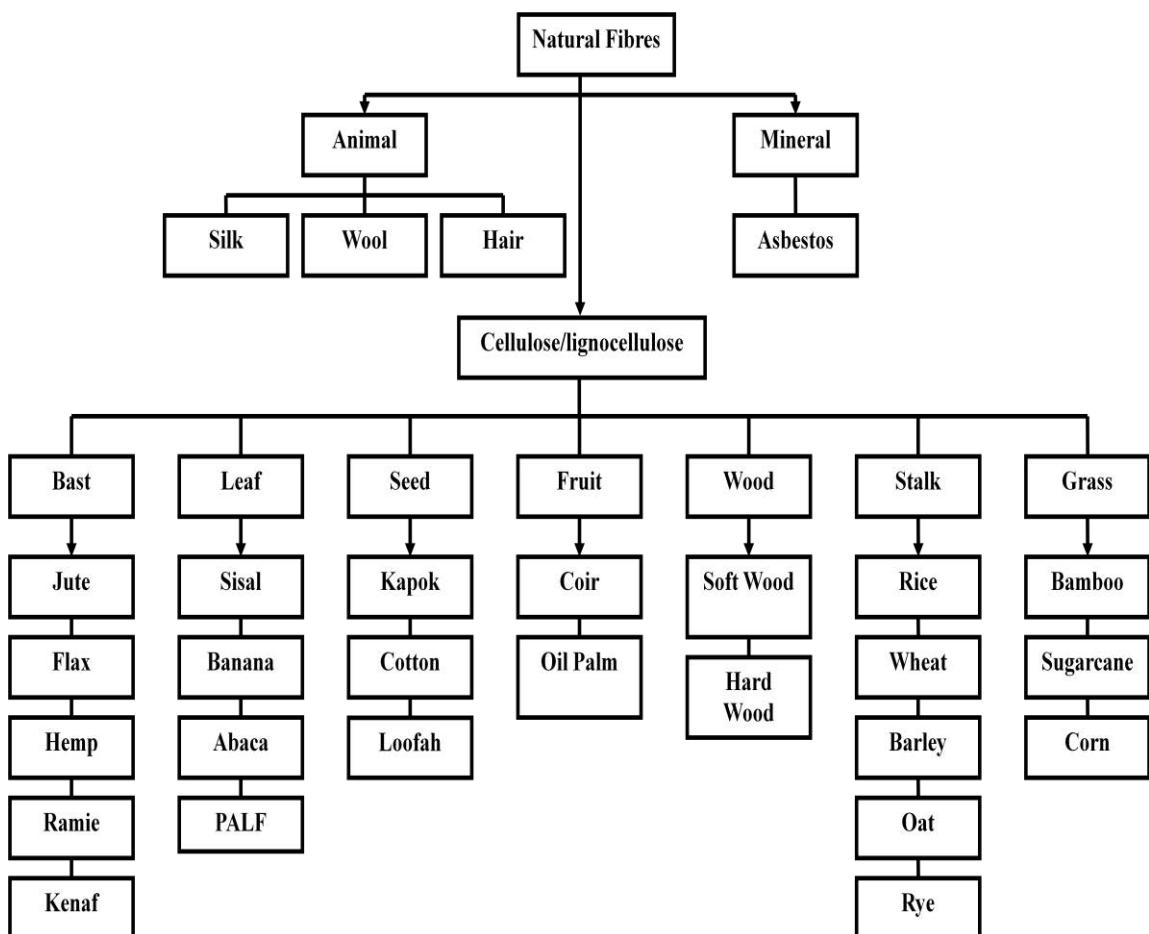


Figure 2.3: Classification of natural fibres (Jawaid & Abdul Khalil, 2011)

2.3.1.2 Chemical Composition of Natural Fibres

The chemical composition of natural fibre is depending on the particular needs of the plants. Any lignocellulosic source consists of three main constituent which are; cellulose, hemicelluloses and lignin and also lesser amounts of extractives, protein,

starch and inorganics. According to Reddy and Yang (2005) the proportion of these components in a fibre depends on the age source of the fibre and the extraction conditions used to obtain fibres. Chemical composition varies from plant to plant and within parts of the same plant. Within plants, chemical composition also differs due to different geographic locations, ages, climates and soil conditions (Rowell & Han, 2004).

2.3.1.3 Physical and Mechanical Properties of Natural Fibres

Fibre strength is an essential key to select fibre that is specific for certain applications. Consideration of specific modulus of natural fibres indicates comparable values to or even better than glass fibres. Natural fibres show improvement of elongation at break which in turn produced superior composite damage tolerance.

Properties of final product manufactured influenced by physical properties of natural fibre such as density, contact angle and fibre length. Besides, the nature of cellulose, hemicelluloses, lignin and crystallinity of natural fibres also contribute to performance of final product produced (Jawaid, 2011).

2.3.2 Kenaf

2.3.2.1 History, Distribution, Properties and Applications

Kenaf (*Hibiscus cannabinus L.*) is an herbaceous annual spring crop belongs to the Malvaceae family and section Furcaria. The genus of Hibiscus is widespread including some 400 species. Kenaf is closely related to cotton, okra, hollyhock and roselle. It is a fast growing plant that can grow to a height of 4-5m in about 4-5 months growing season with the kenaf stalk diameter of 25-35mm (Li & Mai, 2006).

Kenaf is cultivated for probably as early as long (4000 BC) (Roseberg, 1996) and its origin is from Africa. Although kenaf originated from Africa, its production in Africa is very low. In 2010, the total production in Africa was just 3% of the world production. Currently, more countries pay more attention to kenaf research and its cultivation. Over than 20 countries mainly in China, India, Thailand and Vietnam had commercialized kenaf cultivation. In 2010, India has been recognized as a top producer of kenaf with 140 000 MT, followed by China (75 000 MT) and Thailand (18 000 MT) (IJSG, 2012). In Malaysia, it was first introduced in the early 1970s and was highlighted in the late 1990s as an alternative and cheaper source of materials choices. Kenaf also planted in Malaysia as alternative crop to tobacco in medium and long term. Figure 2.4 shows kenaf tree.



Figure 2.4: kenaf tree (Blog, 2014)

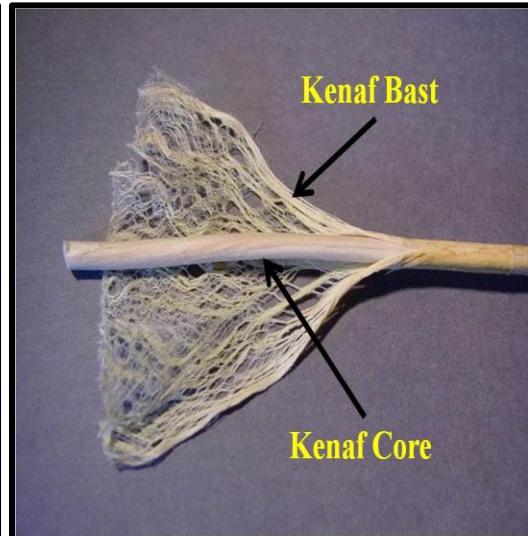


Figure 2.5: Kenaf bast and core stem (Answers.com, 2014)

In this context, kenaf is harvested for its stalk/stems. Generally, kenaf stem are round with thorns ranging from tiny to large depending on it's variety. The stem colour varies from pure green to deep burgundy. Kenaf is consisted of two different fibres which are bast and core. Figure 2.5 shows structure of kenaf bast and core stem.

Kenaf bast and core have different aspect in terms of fibre properties and chemical compositions. Kenaf bast fibres have long fibres with more cellulose content while kenaf core fibres have short fibres with more lignin content (Clark et al., 1971). Lower quality paper can be made from the short core fibres while high quality paper can be made from the long bast fibres of the kenaf (Alexopoulou et al., 2013). Table 2.1 exhibits the general chemical composition of kenaf core and bast fibres.

Table 2.1: Chemical composition of kenaf core and bast fibre

Chemical Composition	Kenaf Core (%)	Kenaf Bast (%)
Holocellulose	87.2	86.8
α – Cellulose	49.0	55.0
Lignin	19.2	14.7

Abdul Khalil et al. (2010a); Tsoumis (1991)

Kenaf is commonly known as ‘the future crop’ received interest from researchers due to its excellent cellulose fibres sources. Table 2.2 represents properties of kenaf fibres. Besides, it also exhibit low density, non-abrasiveness during processing, biodegradability, and high specific mechanical properties. For this reason, kenaf is considered as promising material of choice that can be utilized in numerous industrial applications for instances paper and pulp, insulation mats, fabrics, textiles, biocomposites, absorption materials, automotive, construction and housing industry (Alexopoulou et al., 2013). In automotive industrial applications, the 1996 sold abroad Ford Mondeo has interior automobile panels made of kenaf fiber (Ridzwan, 2007).

Table 2.2: Properties of kenaf fibres

Properties	Kenaf Fibres
Density (g/cm3)	1.2
Tensile Strength (MPa)	930
Young's Modulus (GPa)	53
Elongation at Break (%)	1.6
Cellulose Content (%)	45-57
Hemicellulose content (%)	21.5
Lignin Content (%)	8-13

(Abdul Khalil et al., 2010a); Rouison et al. (2004)

2.3.2.2 Kenaf Fibre as Reinforcement for Polymeric Composites

Kenaf fibre has highly potential to be utilized as reinforcing materials in biocomposite field. Chin and Yousif (2009) has made tribo-composite by using epoxy resin and kenaf fibres for bearing applications. Shibata et al. (2006) studied on effects of layering number and weight fraction focused on kenaf fibres for flexural modulus. The results indicated that the increasing number of kenaf layers contributed to the increment of flexural modulus. Other researcher evaluated several different natural fibres (kenaf, coir, sisal, hemp) reinforced with polypropylene in comparison with glass fibre (Wambua et al., 2003). The results revealed that among fibres used, kenaf fibre-polypropylene composite has higher ultimate strength and tensile modulus. However, the composite shows low impact strength compared with glass fibre composite. Meanwhile, Ishak et al. (2009) have made comparison between kenaf bast and core fibre based polyester resin with different fibres content tested for mechanical properties. The results come out as kenaf bast fibre exhibit better mechanical performances compared with kenaf core fibre composite. However, both types of fibres composites shows reduction for elongation at break as fibre content increased.

2.3.3 Coconut

2.3.3.1 History, Distribution, Properties and Applications

Coconut (*Cocos nucifera*) tree is one of nature's greatest gifts to mankind where practically all parts of the plant are useful in one way or another (Singh et al., 2008). Generally the coconut is used for cooking. However, the fruit shell is discarded as a waste after extraction of the copra and/or of the liquid endosperm that fills the inner part of the fruits (Monteiro et al., 2008). Due to environment concern, this waste has undergone certain transformation process to be extracted as coconut fibres that catches growing attention nowadays. Coconut fibre or best known as Coir fibre is obtained from the husk (mesocarp) of coconut. Figure 2.6 illustrates components of coconut fruits. It is one of abundantly available lignocellulosic fibres in tropical and sub-tropical areas and is a crucial item in the economy of these areas. Coconut is cultivated in 93 countries in the world. As for coconut fibres, it is generated annually worldwide approximately 500 000 tonnes of coconut fibres especially in India and Sri Lanka. In 2012, Indonesia leads the coconut production with about 1800000 MT productions followed by Phillipines, India and Brazil (FAO, 2012).

Coconut fibres can be differentiated into two types which are brown fibres and white fibres. Brown fibres are extracted from matured coconut (11-12 months old) which turn out as thick, strong and have high abrasion resistance fibres. Whereas, white fibres are extracted from immature coconuts which comes out as smoother and finer yet weaker in strength of fibres. Brown fibres are mostly utilized in most engineering applications (Ali, 2010).

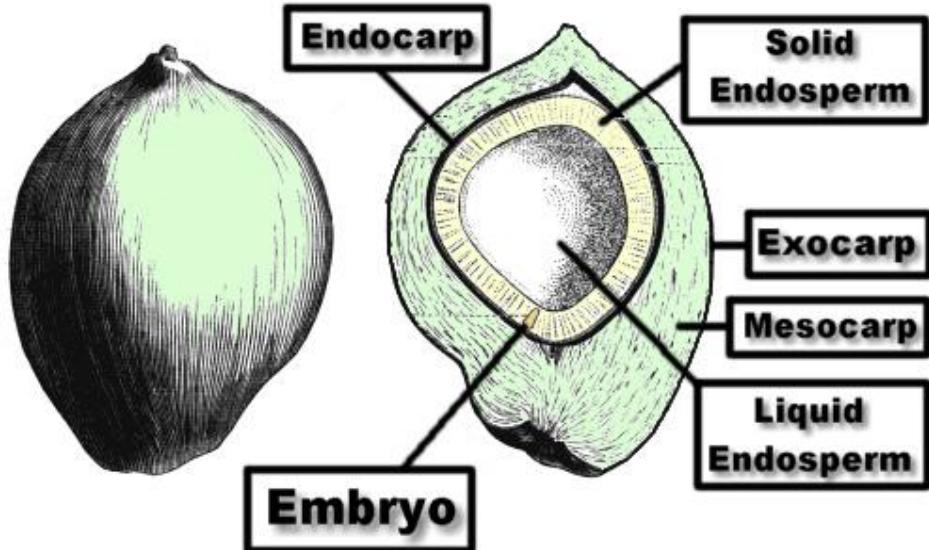


Figure 2.6: Components of coconut fruits (Design, 2013)

Coconut fibre is the long fibres (15-35cm) from the husk of the mature coconut. Table 2.3 represents properties of coconut fibres. Coconut fibre is very strong and flexible due to its high crystalline alpha cellulose and lignin content. Strength, elasticity, elongation and torsion rigidity are some crucial mechanical properties that can defined advantages of coconut fibres bundle. Apart from that, length and fineness are the important physical properties which also determine the spinnability and commercial utility of coconut fibre bundles (Ratnayake, 1996). It is also highly resistant to termite, fungi and rod, excellent temperature and sound insulation, unaffected by moisture and dampness, tough and highly durable (Ali, 2010).

From application point of view, coconut fibres are attractive for certain technical purpose such as wall panel lings and floor coverings especially in auditoria (Jayasekara & Amarasinghe, 2010). Besides, it is also utilize in coconut fibre reinforces composites, lightweight aggregate, geo-textile and cement board (Verma et

al., 2013). Meanwhile, coconut fibre-polyester composites were applied as materials in as helmets, roofing and post-boxes productions (Satyanarayana et al., 1986).

Table 2.3: Properties of coconut fibres

Properties	Value
Density (g/cm ³)	1.15-1.46
Tensile Strength (MPa)	131-220
Young's Modulus (GPa)	4-6
Elongation at Break (%)	15-40
Cellulose Content (%)	32-43
Hemicellulose content (%)	0.15-0.25
Lignin Content (%)	40-45

Bismarck et al. (2005); Rouison et al. (2004)

2.3.3.2 Coconut Fibre as Reinforcement for Polymeric Composites

Many aspects of the utilization of coconut fibres as reinforcement in polymer-matrix composites are explained in the previous studies. Aireddy and Mishar (2011) investigated coconut dust reinforced epoxy matrix composites of different compositions specifically for abrasive wear resistance. It was observed that the abrasive wear resistance increased with increasing of coconut dust concentration. Viability of coconut fibre reinforced composites in sound absorption panel had been studied by Mahzan et al. (2010). The result obtained showed good acoustic properties of the composites produced. It is interesting to note that the potential of the coconut fibre reinforced composites in sound absorption panel was highlighted in this study.

Meanwhile, Bujang et al. (2007) investigated the dynamic characteristics of coconut fibre reinforced composites. The tensile test was carried out shows that tensile modulus changes with the fibre content. Rosa et al. (2009) studied effect

treatment on coconut fibres biocomposites and tested its performances on tensile and thermal tests. In addition, Harish et al. (2009) studied the mechanical properties of coconut fibre composites. The fractured surface of specimens was viewed using SEM for a qualitative evaluation of the interfacial characteristics of coconut/epoxy and used glass fiber epoxy as a comparison. The author has concluded that coconut fibre is a highly potential reinforcement material for manufacturing low load bearing thermoset composites. Other researcher has also studied the structural characteristics and mechanical performance of coconut fibre reinforced polyester composites (Almeida et al., 2008).

2.4 Oil Palm Shell (OPS) Filler as Enhancement

Oil palm belongs to the species *Elaeis guineensis* under the family Palmacea. It is believed originated in the tropical forests of West Africa. It is produced about 27 million acres in 42 countries worldwide including Malaysia, Indonesia and Thailand. Malaysia is emerged as world's leading producer and exporter of the oil palm accounting for approximately 60% of the world's oil and fat production. With total area plantation about 6 million, oil palm industry in Malaysia can produce over 11.9 million tons of oil and 100 million tons of biomass (Abdul Khalil et al., 2010b). Over the past four decades, oil palm has made remarkable and continuous development in the world market. It is projected the average annual production in Malaysia will achieve 15.4 MT in the period 2016-2020 (Teoh, 2000).

Oil palm tree has an economic life span about 25 years which in turn contributes to a high amount of agricultural wastes. Waste which is produced from oil palm industries includes oil palm trunk (OPT), oil palm frond (OPF), oil palm shell

(OPS), empty fruit bunch (EFB), fresh fruit fibre (PFF) and palm oil mill effluent (POME).

2.4.1 Oil Palm Shell

2.4.1.1 History, Production and Compositions

Oil palm shell (OPS) is an agricultural solid residue produced after the processing and oil extraction of fresh fruit bunch (FFB) takes place. In general, the FFB contains about 5.5% shell (Ma et al., 1999). Consequently, over 4 million tonnes of oil palm shell solid waste is generated annually. This waste is normally discarded through incineration and at times, the shell is left to rot in huge mounds as shown in Fig. 2.7 (Teo et al., 2006). Therefore exploitation of this waste material is urgently needed to preserve the natural resources and sustain the environmental stability. The OPS can be described has semicircular in shape but with greater tendency to be flat (Sangwichien et al., 2010). It is reported that OPS has high density, relatively high carbon content and low ash content. Table 2.4 presents composition analysis in OPS.



Figure 2.7: Waste oil palm shell (OPS) left at oil palm mill (Teo et al., 2006)

Table 2.4 Analysis of oil palm shell (OPS)

Composition Analysis (wt%)	
Carbon	54.7
Hydrogen	7.49
Nitrogen	2.03
Oxygen	35.6
Moisture	4.7
Ash	7.2
Volatile	74.3

Arami-Niya et al. (2010)

2.4.1.2 Application of Oil Palm Shell (OPS)

2.4.1.2.1 Tar

The oil palm shell has been studied as a suitable material for surfacing roads. In general, fibre and shell are used as fuel in boilers. This boiler usually will produce slag, caused by the burning of biomass residues. The use of boiler slag after sieving it properly to remove ash could be used as biomass tar for surfacing roads. The idea of using OPS is proved to be cheap yet practical way using waste to its utmost potentials.

2.4.1.2.2 Carbon and Activated Carbon

Oil palm shell is a promising raw material in manufacturing carbon and activated carbon which had been thoroughly discussed by Guo and Lua (2002); Lim et al. (2010); (Nur et al., 2012). Palm shell is suitable for adsorption due to its ability to be modified, thus becoming high porosity carbon. Apart from that, short time of palm shell activation can be achieved due to less fibrous structure since it contained high lignin content and low cellulose content (Daud & Ali, 2004). In another interesting research, the development of microporosity for the oil palm shell activated carbon would lead to potential applications in gas-phase adsorption for the removal of air pollutants. The oil palm shell activated carbon could absorb gaseous pollutant SO₂

effectively at a capacity which was slightly higher in comparison with commercial product (Microcrab) (Lua & Guo, 2001).

2.4.1.2.3 Concrete

Since it is light and naturally sized, the OPS is an ideal material for replacing aggregates in lightweight concrete construction. On account of being hard and of organic origin, they will less contaminate or leach to produce toxic substances once they are bound in concrete matrix. Therefore, OPS concrete can potentially be utilized in lightweight concrete applications that needed low to moderate strength such as pavements and infill panel for floorings and walls (Basri et al., 1999). In addition, owing to the hard characteristics, oil palm shell as coarse aggregate is a promising materials to replace conventional coarse aggregates traditionally used for concrete production (Mohd et al., 2008).

2.4.1.2.4 Bioenergy

Oil palm shell is one of the biomass groups that can be used for conversion to bio-oil by using pyrolysis process. Islam et al. (1999) reported that pyrolytic oil which is produced from pyrolysed palm shell contains chemical of 6.9% acetic acid, 28.3% phenols plus other phenol and methyl derivatives. Further development by Ani et al. (2004) on the phenols derived from the pyrolysis oil of palm shell residues had resulted in the production of palm shell-based adhesives which is comparable to the petroleum-based adhesive.

2.5 Hybrid Composites

2.5.1 Introduction

The word “hybrid” is originated from Greek-Latin and can be found in various scientific areas. In the case of polymer composites, hybrid composites are these systems in which one type of reinforcing material is incorporated in a mixture of different matrices (blends) (Thwe & Liao, 2003), or two or more reinforcing and filling materials are present in a single matrix (Fu et al., 2002) or both approaches are combined. The hybridization of several different kinds of fibres into a single matrix has lead to the advancement of hybrid biocomposites. The behavior of hybrid composites is a weighed sum of the individual components in which there is a more beneficial balance between the natural advantages and disadvantages. Through hybridization, advantages of one fibres could complement what are lacking in other fibres and vice versa. Behaviour of hybrid composites is interesting approach to design proper material that has balance in cost, superior strength performance and environmental friendly as well in order to suit certain requirements for numerous innovative applications (Thwe & Liao, 2003).

The performances of hybrid composites primarily rely upon the fibre content, length of individual fibres, orientation, extent of intermingling of fibres, fibre to matrix bonding and arrangement of both the fibres. The term hybrid effect has been related to the occurrences of an clear synergistic development in the performances of a composite consist of two or more types of fibre (Jones, 1994). In order to select components in hybrid composites, certain factors that must be considered for examples purpose hybridization and requirement subjected on the material or construction being designed (John et al., 2009). There are several categories of hybrid composites characterized such as: interplay, sandwich hybrids, interply and intimately