MODIFICATION, CHARACTERIZATION AND UTILIZATION OF BANANA TRUNK FIBERS – *Musa acuminate x balbisiana Colla (ABB Group)*

by

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Starting Materials and Products

BTF	Unmodified banana trunk fibers
BTF-1	Sodium Hydroxide Treated Banana Trunk Fibers
BTF-2	Acytalated Banana Trunk Fibers
BTF-3	Peroxide Treated Banana Trunk Fibers
BTF-4	Stearic Acid Treated Banana Trunk Fibers
BTF-5	Sulphuric Acid Treated Banana Trunk Fibers
Cd(II)	Cadmium (II) ion
CGBTF	Castor oil Grafted Banana Trunk Fibers
Cu(II)	Copper (II) ion
DOC	Dissolved Organic Compound
FAGBTF	Fatty Acid Treated Banana Trunk Fibers
Fe(II)	Iron (II) ion
MDBTF	Mechanically Damaged Banana Trunk Fibers
MR	Methyl Red
PCP	1, 3 Cis – Poly chloroprene
PGBTF	Palm Oil Grafted Banana Trunk Fibers
PVA	Poly vinyl alcohol
PVAc	Poly vinyl acetate
PMMA	Poly methyl methacrylate
SAMDBTF	Stearic Acid Treated Mechanically Damaged Banana Trunk Fibers
SACCMDBTF	Stearic Acid & Calcium Carbonate Mechanically Damaged Banana Trunk Fibers

SGBTF	Stearic Acid Grafted Banana Trunk Fibers
OGBTF	Oleic Acid Grafted Banana Trunk Fibers
WOCS	Weathered Oil Contaminated Seawater
Zn(II)	Zink (II) ion

Symbols

cm	Centimeter
cm^{-1}	Wavenumber
DSC	Differential Scanning Calorimetry
DTG	Derivative Thermal Gravimetry
EDX	Energy Dispersive X-ray
FTIR	Fourier Transform Infra-Red
g	gram
hr	hour
KBr	Potassium Bromide
min	Minutes
mg	Miligram
mm	Milimeter
NaCl	Sodium Chloride
nm	Nanometer
SEM	Scanning Electron Microscope
SGVFF	Simple general valence force field
PED	Potential Energy Distribution
TG	Thermal Gravimetry
TGA	Thermogravimetry Analysis
ThBr	Thorium Bromide
%	Percentage
$[M^{2+}]$	Concentration of ions
°C	Celcius
q_e	Adsorption capacity
μg	Microgram

PENGUBAHSUAIAN, PENCIRIAN DAN PENGGUNAAN SERAT BATANG PISANG - *Musa acuminate x balbisiana Colla (ABB Group)*

Abstrak

Tesis ini terbahagi kepada dua bahagian. Bahagian pertama membincangkan penyingkiran melalui penjerapan (i) metil merah, suatu pewarna monoazo mutagenik, (ii) ion logam berat, Cd(II), Cu(II), Fe(II) dan Zn(II) dan (iii) minyak tertumpah daripada larutan akueus dengan menggunakan serat batang pisang (BTF) takterubahsuai dan terubahsuai. BTF yang diubahsuai dengan asid sulfurik and asid oleik menunjukkan penjerapan terbaik, masing masing, untuk metil merah dan minyak. Proses keseimbangan digambarkan dengan baik oleh model isoterma Freundlich dan kinetic pseudo tertib kedua. Bahagian kedua tesis ini membincang penghasilan komposit hijau. BTF diubahsuai secara mekanikal menggunakan pengisar berkelajuan tinggi dan serat yang dihasilkan (MDBTF) seterusnya diubahsuai dengan (i) asid stearat (SAMDBTF) dan (ii) kalsium karbonat yang diliputi dengan 5% (wt/wt) asid stearat (SACCMDBTF). Kajian penyerapan minyak telah dilakukan dan hasilnya menunjukkan bahawa SAMDBTF dan SACCMDBTF, masing-masing, menyerap 9.5 g/g dan 18.3 g/g minyak enjin. Kajian ini menunjukkan bahawa serat yang telah diubahsuai dapat menjerap minyak tumpahan dengan baik. Tesis ini juga membincangkan penyediaan adunan campuran pelbagai komposisi poli vinil alkohol (PVA) dan BTF yang diesterifikasi dengan asid lemak (FAGBTF). Ciri-ciri PVA/FAGBTF dikaji dengan analisis terma gravimetri (TGA), analsis pengimbasan kalorimetri pengkamiran (DSC), analsis imbasan mikroskop elektron (SEM), spectroskopi FT-IR dan ujian tarikan universal. Didapati bahawa peningkatan jumlah BTF dalam adunan memperbaiki sifat terma tetapi menurunkan tahap peratusan penyerapan air dibandingkan dengan ciri-ciri PVA asli.

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Abstract

There are two parts in this thesis. First part involves unmodified and modified banana trunk fibers (BTF) for the removal of (i) methyl red, a mutagenic monoazo dye, (ii) heavy metals ions, Cd (II), Cu (II), Fe (II) and Zn (II), and (iii) spilled oil in aqueous solution. Sulphuric acid treated BTF and oleic acid treated BTF exhibited the best sorption capacity for methyl red and spilled oil, respectively. The equilibrium processes of these studies were described well by the Freundlich isotherm and pseudo-second-order kinetic models. Secondly, this work discusses the production of green composites. BTF were mechanically damaged using a high speed blender and the resulting fibers (MDBTF) were treated with (i) stearic acid (SAMDBTF) and (ii) calcium carbonate coated with 5% (wt/wt) stearic acid (SACCMDBTF). Oil uptake study was conducted and the results reveal that SAMDBTF and SACCMDBTF sorbed 9.5 g/g and 18.3 g/g of used engine oil at equilibrium, respectively. This work reveals that the treated fibers were efficient sorbents for oil spill recovery. The preparation of blends of various compositions of poly vinyl alcohol (PVA) and fatty acid esterified BTF (FAGBTF) was also discussed in this work. The characteristics of PVA/FAGBTF films were evaluated by means of thermal gravimetric analysis (TGA), differential scanning calorimetry (DSC), scanning electron microscopy (SEM), FT-IR spectroscopy and tensile tests. It is found that the increased in the amount of BTF improved the thermal properties but decreased the percentage degree of swelling of blends as compared to the characteristics of pure PVA.

CHAPTER 1

INTRODUCTION

1.1 History of Fibers

The history of fibers is as old as human civilization. Traces of natural fibers have been located to ancient civilizations all over the world. Flax fibers which are generally considered as the oldest natural textile fiber were in use since 5000 BC. Previously the usage of fiber was limited to natural fibers such as flax, cotton, silk, wool and plant based fibers for different applications. Fibers are often divided into two categories; natural and synthetic fibers. In the year 1665, the English researcher Robert Hooke describe about physiological descriptions of minute bodies in his book which then lead to the production of synthetic fiber. Starting from then till now, man made fiber production has reached an estimate 24 million ton. Application of synthetic fibers vary; from home furnishings, medicine, aeronautics, energy, industry, and more. Currently, fiber engineers modify the nature of the natural fiber to improve its performance.

1.2 Types of Natural fiber

Plant, animal and mineral fibers fall into the natural fiber category. Examples of plant fibers are banana, cotton, jute, and pineapple fibers. Animal fibers include coir, wool and silk to name a few. Animal fiber is threadlike outgrowth of the skin and composed of keratin. The only mineral based natural fiber is asbestos. Plant and animal based fibers are biodegradable while asbestos is a non-biodegradable product. Synthetic fibers are derived from extensive research to improve natural fibers. In general, synthetic fibers are created by forcing, usually through extrusion process.

1.3 Properties of Natural and Synthetic Fibers

1.3.1 Natural Fibers

1.3.1.1 Physical Properties

Natural fibers can be classified several categories which are seed, bast, leaf and fruit qualities. In composite application, the commonly used fibers are bast and hard leaf fibers (Williams and Wool, 2000). Common bast fibers include hemp, jute, flax, ramie and kenaf and examples of leaf fibers are sisal and banana. Joseph *et al*., (2000) concluded that the strength characteristics of fiber depend on the properties of the individual constituents, the fibrillar structure and the lamellae matrix. Thus, the structural components of the fibers namely cellulose, hemicellulose and lignin have to be examined in order to obtain a better understanding of the mechanical properties and durability of the fibers. Furthermore, fiber strength, fiber fitness, the polymerization of the cellulose, cleanness or purity and homogeneity of the sample has to be studied to have a clear understanding of the physical properties of the fiber. Of late, the use of plant fibers as a reinforcing agent in fiber-reinforced composite to replace synthetic fibers is receiving attention due to its advantages such as high specific strength, cost, density, renewability, recyclability, abrasiveness and biodegradability properties (Li *et al*., 2007; Bledzki and Gassan, 1999; Edeerozey *et al*., 2007). Jähn *et al*. (2002) reported that plant fiber properties directly influence the physical parameters of the fiber-reinforced composites. Currently, cotton, kenaf, sisal, flax, palm oil, coir, arecanut and banana trunk fibers (BTF) acquire utmost significance, since reinforced plastics, strings, cords, cables, ropes, mats, brushes, hats, baskets and fancy articles such as bags are manufactured with those fibers (Mohan Rao and Mohana Rao, 2007). Furthermore, natural fibers are found to have high specific mechanical properties which are comparable to those of traditional reinforcements (Hanselka and Herrmann, 1995). Thus, the intrinsic properties of natural fibers can satisfy the requests of the global market (Herrmann and Hanselka, 1996) and gives less concern with environmental issues. This fulfils the needs of industries such as automobile, shipbuilding and building industries (Corrales *et al*., 2007). Thus natural fibers are deemed as potential substitute for non-renewable synthetic fibers (Gandini *et al*., 2001).

1.3.1.2 Chemical Properties

Natural fibers are complex in structure. They are generally lignocellulosic, consisting of helically wound cellulose micro fibrils in an amorphous matrix of lignin and hemicellulose (Wang 2004). **Table 1.1** shows the botanical composition of natural fibers. High cellulose content which improves the mechanical properties is a desirable property of a fiber to be used as reinforcement in polymer composites (Williams and Wool, 2000).

1.3.2 Properties of Synthetic Fibers

Synthetic fibers are created through a chemical process. Chemicals are forced into spinnerets, which have tiny holes where the synthetic fibers are created. Polyester is the most common synthetic fiber. Acetate, nylon, and spandex are several commonly used synthetic fibers as fabrics. Synthetic fibers are commonly used as fabrics as they pose a huge advantage over natural fibers due to its durable nature. The attractive forces between fibers play a large part in determining it’s properties.Due to its long chain, these interchain forces are amplified far beyond the attractions between conventional molecules. Different side groups on the fibers can lend to ionic bonding or hydrogen bonding between its own chains. These stronger forces typically result in higher tensile strength and higher crystalline melting points. In general, the physical properties of the fibers such as the tensile strength increase with the fiber length and the crosslinking of the chains.

1.4 Lignocellulosic Fiber

By nature, lignocellulosic fiber consists of helically wound cellulose microfibrils in a matrix of lignin and hemicellulose (Taj *et al*., 2007). Agricultural waste residues, such as rice straw, banana stalk, and palm ash fall in this category. Plant fibers are now being used to produce fuel, chemicals, enzymes, food (Khalil *et al*., 2006), automotive parts, in construction applications and biocomposites (Ton-That and Denault, 2007). Approximately 2×10^{11} tons of lignocellulosics are produced every year, compared with 1.5×10^{8} tons of synthetic polymers (Khalil *et al*., 2006). Plant waste fibers are available through out the year, abundant, renewable, and biodegradable. Since they cause environmental pollution when left on the plantation floor, economic utilization of these fibers will be beneficial.

1.4.1 Advantage and Limitation of Lignocellulosic Fibers

Lignocellulosic fiber wastes can be used to develop materials with high performances; as the National Academy of Science of USA puts it ‘‘hierarchical structures in biology as a guide for new materials technology’’ (Ganan *et al.*, 2007). This tendency is associated with particular advantages that combine low density with good mechanical properties, low cost and biodegradability. Natural fibres have attractive technical, economical and environmental advantages that include: renewable source of energy, low abrasion, medium thermal properties, high disposability, low energy consumption and low generation of neutral carbon dioxide (Bledzki *et al*., 1998). They can be ideal competitors for the non-renewable, expensive petroleum-based synthetic fibers in composite materials, particularly in the automotive industry (Bledzki *et al*., 1998) and including building sectors (Kedari *et al*., 2003).However, the applicability of these materials has been found to be limited due to leaching of organic substances such as lignin, tannin, pectin and cellulose into the solution. Furthermore, fibers are hydrophilic which causes fiber dispersion challenges and they are sensitive to humidity. Other challenges of natural fibers are such as fiber inconsistency, low impact strength and issue of stocking raw material for extended time period (Ton-That and Denault, 2007). To overcome such problems, chemical treatment on solid adsorbents has been used as a technique for improving physical and chemical properties of them and to increase their adsorption capacity (Noeline *et al*., 2005).

1.5 Cellulose

Cellulose is the most abundant organic chemical on the face of the earth. It is a glucan polymer of D-glucopyranose units, which are linked together by α-1,4-glucosidic bonds. The building block for cellulose is actually cellobiose, since the repeating unit in cellulose is a two-sugar unit. Cellulose molecules are randomly oriented and have a tendency to form intra- and intermolecular hydrogen bonds. As the packing density of cellulose increases, crystalline regions (65%) are formed. Lower packing density region is referred to as amorphous cellulose. The molecular chains pack in layers that are held together by weak Van der Waals forces. The layers consist of parallel chains of anhydroglucopyranose units and the chains are held together by intermolecular hydrogen bonds. There are also intramolecular hydrogen bonds between the atoms of adjacent glucose residues. There are several types of cellulose in wood: crystalline and noncrystalline and accessible and nonaccessible. Accessible and nonaccessible refer to the availability of the cellulose to water and microorganisms. The surfaces of crystalline cellulose are accessible but the rest of the crystalline cellulose is nonaccessible. Most of the noncrystalline cellulose is accessible but part of the noncrystalline cellulose is covered with both hemicelluloses and lignin that it becomes nonaccessible. Concepts of accessible and nonaccessible cellulose are very important in moisture sorption, pulping, chemical modification, extractions, and interactions with microorganisms (Rowell *et al.*, 2005).

1.6 Lignin

Lignins are amorphous and highly complex. Lignin is a polymer of aromatic phenylpropane units. The precursors of lignin biosynthesis are p-coumaryl alcohol, coniferyl alcohol and sinapyl alcohol. Softwood lignin has a methoxyl content of 15–16%; hardwood lignin has a methoxyl content of 21%. Lignin is produced through phenol derivatives and the three basic building blocks of guaiacyl, syringyl, and p-hydroxyphenyl moieties. Lignin is distributed throughout the secondary cell wall, with the highest concentration in the middle lamella. 70% of the lignin is located in the cell wall (Rowell *et al.*, 2005).

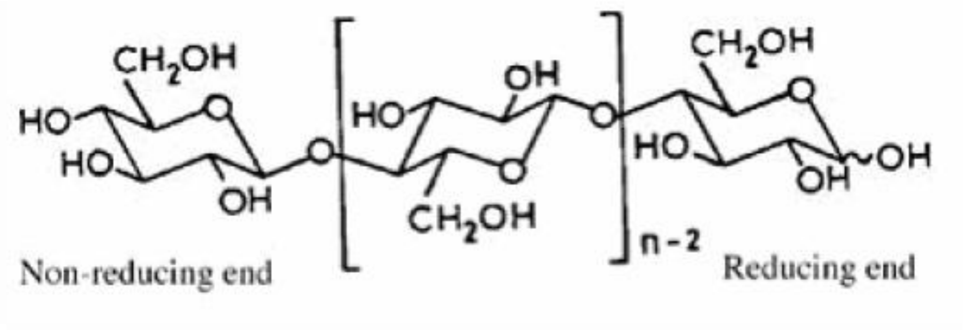
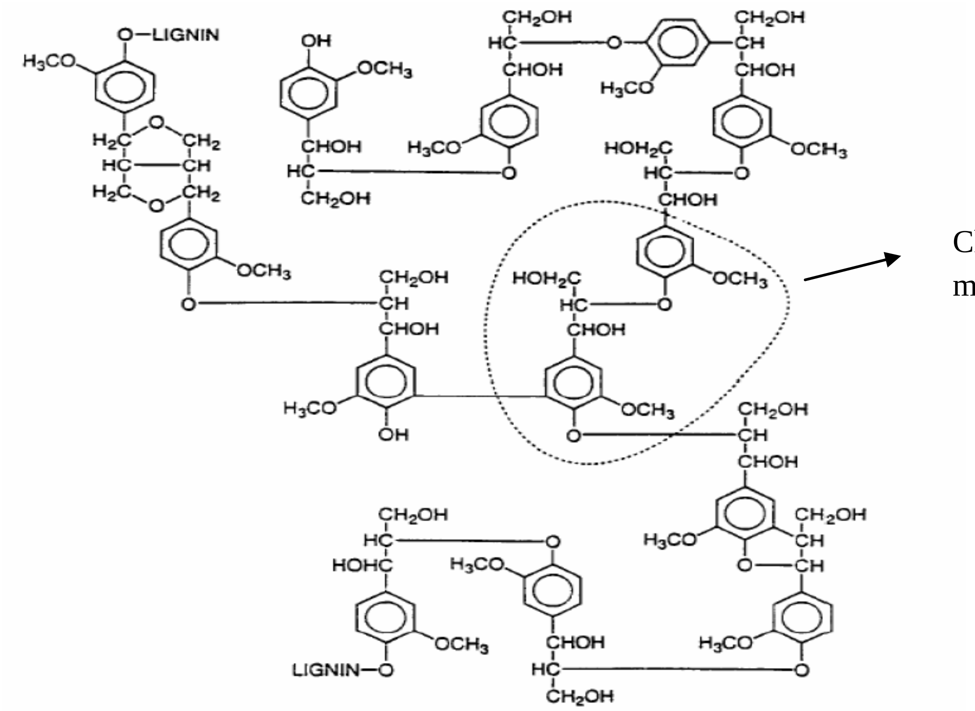


Figure 1.1: Structure of cellulose

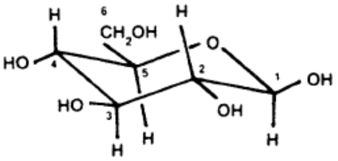


Chief
monomer unit

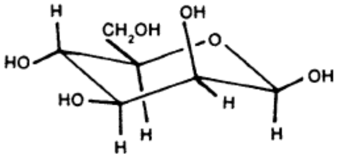
Figure 1.2: Structure of lignin

1.7 Hemicellulose

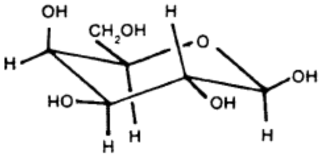
Hemicellulose is a branched heteropolymer. Hemicellulose has a random, amorphous structure with little strength and present along the cellulose. It is made up of monomers such as xylose, mannose, galactose, rhamnose, arabinose, mannuronic acid and galacturonic acid. Hemicelluloses contain large amount of D-pentose sugars and small amounts of L-sugars; consists of shorter chains of sugar units; embedded in the cell walls of plants and bind with pectin to cellulose to form a network of cross-linked fibres. Hemicelluloses include xylan, .glucuronoxylan, arabinoxylan, glucomannan, and xyloglucan.



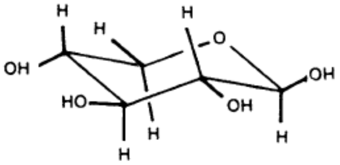
β-D-Glucose
β-D-Glucopyranose
β-D-Glup



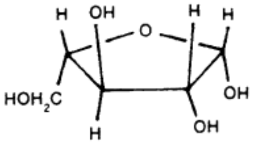
β-D-Mannose
β-D-Mannopyranose
β-D-Manp



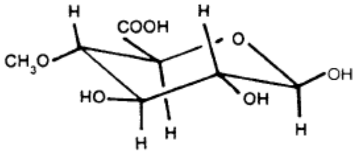
β-D-Galactose
β-D-Galactopyranose
β-D-Galp



β-D-Xylose
β-D-Xylopyranose
β-D-Xylp



α-L-Arabinose
α-L-Arabinofuranose
α-L-Araf



4-O-Methylgucuronic acid
4-O-Methylglucopyranosyluronic acid
4-O-Me-α-D-GlupA

Figure 1.3: Sugar monomer components of hemicellulose (Rowell *et al*., 2005)

1.8 Chemical Properties of Natural Fibers

Natural fibers are complex in structure. They are generally lignocellulosic, consisting of helically wound cellulose micro fibrils in an amorphous matrix of lignin and hemicellulose (Wang 2004). **Table 1.1** shows the botanical composition of natural fibers. High cellulose content which improves the mechanical properties is a desirable property of a fiber to be used as reinforcement in polymer composites (Williams and Wool, 2000).

1.9 Moisture Absorption Characteristics

The main disadvantage concerning the use of natural fibers is their susceptibility to moisture uptake (Sreekala *et al*., 1997a; Sreekala *et al*., 1997b; Selzer and Friedrich, 1995; Rana *et al*., 1997). High moisture sorption capacities of natural fibers adversely affect the adhesion with hydrophobic matrix (Bessadok *et al*., 2007; Satyanarayana *et al*., 2006). This results in material degradation, loss of strength and causes weaker adhesion in the fiber/matrix interface, thus compromising some of the mechanical properties of the manufactured composite. Thus, the fiber surface must be cleaned and chemically modified to decreases the rate of moisture absorption in a composite (Yuan *et al*., 2002).

Table 1.1 Chemical composition and structural parameters of natural fibers

Fibers	**% Cellulose**	**% Hemi-Cellulose**	**% Lignin**	**% Ash**	**% Extractives**	**Moisture Content**	**Reference**
Jute	61 -71.5	13.6- 20.4	12 – 13	-	-	12.6	Mohanty *et al*., 2000
Flax	71 -78.5	18.6 – 20.6	2.2	1.5	2.3	10.0	Mohanty *et al*., 2000
Hemp	70.2 -74.4	17.9 – 22.4	3.7-5.7	2.6	3.6	6.2	Mohanty *et al*., 2000
Banana Trunk	24.2 – 38.3	11.1 –18.9	13.8-16.4	8.6	4.5	9.7	Bilba *et al*., 2007
Sisal	67-78	10-14.2	8-11	-	-	11	Mohanty *et al*., 2000
Cotton	82.7	5.7	-	-	-	-	Mohanty *et al*., 2000
Coir	36 -43	36-43	41 – 45	-	-	8	Mohanty *et al*., 2000

1.10 Chemical Treatment of Natural Fibers

Chemical modifications of fibers are carried out to improve the physio-chemical properties of the fibers. Chemical treatment of the fibers can clean the fibers surface, chemically modify the surface, stop the moisture absorption process and increase the surface roughness (Edeerozey, *et al*., 2007). Modifications can be carried out on natural fibers as they possess hydroxyl groups from cellulose, hemi-cellulose and lignin. The hydroxyl groups possibly have interaction via the hydrogen bonding within the molecules and this reduces the activity towards the matrix. A chemical modification therefore activates these groups and also introduces new moieties that can effectively interlock with the matrix. Thus, various studies on the treatment of different types of plant fibers to improve their properties have been carried out. Many works have been reported with regards to the modification of natural fibers (Barkakty, 1976; Bisanda and Anshell, 1991). Among the popular treatments carried out by various researchers are alkali treatment (Ray *et al*., 2001; Bledzki *et al*., 2004; Aziz and Ansell, 2004; Mishra *et al*., 2001; Sreekala and Thomas, 2003), acetylation (Sreekala and Thomas, 2003; Mohanty *et al*., 2000), peroxide treatment (Joseph *et al*., 2002; Paul *et al*., 1997), graft co-polymerisation (Karmaker and Youngquist, 1996) silane treatment (George *et al*., 1998; Bataille *et al*., 1989) and benzoylation (Joseph *et al*., 1996; Nair *et al*., 1996).The different surface chemical modifications of natural fibers have achieved various levels of success in improving fiber strength, fiber fitness and fiber-matrix adhesion in natural fiber composites. Various fibers surface modifications were carried out to reduce the hydrophilicity and thereby decreasing the sorption characteristics of oil palm fibers (Sreekala *et al*., 2003). The data obtained from their study suggest that treatment reduces the water uptake at all temperatures. The decrease is due to its physical and chemical changes occurred to the fibers on modifications. Edeerozey, *et al*., 2007 was found that the alkalization treatment has improved the mechanical properties of the kenaf fibers significantly as compared to untreated kenaf fibers. Mercerization, acetylation, and resorcinol / hexamethylenetetramine (R/H) treatments have been applied to sisal fibers in order to improve their adhesion in composites materials (Martins *et al*., 2006). Treatment with acetic anhydride to bring acetylation of the cellulose fibers has also been proved to be an effective means of modifying cellulose fibers surface. Brief descriptions of several chemical modifications that were used in this study are discussed briefly in the following sections.

1.11 Surface Chemical Modifications of Natural Fibers

1.11.1 Mercerization

Mercerization is currently the most common chemical treatments of natural fibers when used to reinforce thermoplastics and thermosets. Mishra *et al*., 2001 and Ray *et al*., 2001 treated sisal, flax and jute fibers, respectively with 5% aqueous NaOH at room temperature. These researchers concluded that alkali treatment increased in amorphous cellulose and decreased crystalline cellulose. Alkaline treatment disrupts the hydrogen bonding in the network structure, hence increases fiber surface and roughness (Bilba *et al*., 2007) which results in better mechanical interlocking (Valadez-Gonzalez *et al*., 1999). This increases the specific surface area of the fibers, making the hydroxyl groups of cellulose macromolecules more easily accessible and increases the number of possible reaction sites (Valadez-Gonzalez *et al*., 1999). This treatment also removes a certain amount of lignin, hemicelluloses, wax and oils covering the external surface of the fiber cell wall (Pothan *et al*., 2006), luminium s cellulose and exposes the short length crystallites (Mohanty *et al.*, 2001). The following reaction (Scheme 1.1) takes place as a result of alkali treatment; addition of aqueous sodium hydroxide (NaOH) to fiber promotes the ionization of the hydroxyl group to the alkoxide (Agrawal *et al.*, 2000).

$$\text{Fiber- OH} + \text{NaOH} \longrightarrow \text{Fiber-Ona} + H_2O$$

Scheme 1.1

1.11.2 Acetylation

Acetylation of natural fibers is a common method causing plasticization of cellulosic fibers which introducing an acetyl functional group (CH_3COO–) into the fibers. However, the reactions produce acetic acid (CH_3COOH) as by-product and have to be removed from the lignocellulosic material before the fiber is used. Chemical modification with acetic anhydride (CH_3-C (=O)-O-C (=O)-CH_3) substitutes the hydroxyl groups of cellulose, hemi-cellulose and lignin with acetyl groups to be come hydrophobic (Hill *et al.*, 1998). The reaction of acetic anhydride with natural fiber is shown in Scheme 1.2. Acetylation increases the dimensional stability of composites and reduces the hygroscopic nature of natural fibers. Acetylation of sisal fibers was investigated Mishra *et al.* 2003. Sisal fibers were immersed in 5 % and 100 % NaOH (1 hour at). Next, the mercerized fibers were soaked in glacial acetic acid for 1 hour at 30°C, separated by decantation and then soaked in acetic anhydride containing one drops of concentrated H_2S0_4 for 5 minutes. Manikandan Nair *et al.* 2001 treated sisal fiber in 18% NaOH solution, then in glacial acetic acid (1 hour) and finally in acetic anhydride containing two drops of concentrated H_2SO_4 for 1 hour. The treated surface of sisal fiber was found to be very rough and had voids that would provide better mechanical interlocking with polymer matrix (Li *et al*., 2007).

$$\text{Fiber} - \text{OH} + CH_3\text{-C(=O)-O-C(=O)-}CH_3 \longrightarrow \text{Fiber-OCOC}H_3 + CH_3COOH$$

Scheme 1.2

1.11.3 Peroxide Treatment

Commonly, the chemicals used for peroxide treatment in natural fiber's surface modification are benzoyl peroxide (BP, $((C_6H_5CO)_2O_2)$ and dicumyl peroxide (DCP, $(C_6H_5C(CH_3)_2O)_2$). Peroxides have ROOR functional groups which contain the divalent ion O–O and tend to decompose easily to free radicals of the form RO. The RO groups then reacts with the hydrogen group of the matrix and cellulose fibers. Sreekala *et al*. (2003) treated oil palm fibers with BP or DCP in acetone for 30 minutes after alkali pre-treatment and concluded that thermal stability of fibers increased after treatment. The tensile strength of peroxide treated short sisal fibers reinforced polyethylene composites also increases (Joseph *et al*., 1996) and the hydrophobicity decreased (Paul *et al*., 1997) upon treatment.

1.11.4 Stearic Acid Treatment

Stearic acid ($CH_3(CH_2)_{16}COOH$) treated fibers in 1 % ethyl alcohol was investigated in Paul *et al*., 1997 and Zafeiropoulos *et al*. (2002). It was concluded that this treatment removed non-crystalline constituents of the fibers, hence alters the fiber surface morphology and topography. Zafeiropoulos (2002) also observed that treated flax fibers were found to be more crystalline than the untreated ones and stearation decreased the fiber surface free energy. Banerjee *et al*. (2006) used saw dust fibers, stearic acid, 2 drops of concentrated H_2SO_4 in 100 mL of n-hexane and refluxed this mixture in a Dean–Stark apparatus at 65 °C for 6 hour. The resulting material was found to be suitable for oil spill recovery.

1.11.5 Acid Treatment

Alvarez *et al*. (2004) treated saw dust fibers using 8N of sulphuric acid at 150 °C for 30 and 120 minutes. The results obtain shows that the sulphuric treated fibers are thermally more stable than the parent lignocellulosic material. Furthermore, upon carbonization at 900 °C, the acid treated saw dust yields a char with a well-ordered honeycomb structure. Activated carbon pretreated by heating with luminium acid and nitric acid at high temperature to evaluate their performance on hexavalent luminiu removal from aqueous phase. The obtained results indicated that the treated activated carbon is an efficient sorbent for the removal of (Cr (VI)). Hydrochloric acid treated date palm tree fiber (0.3, 0.9 and 1.6N for 1 hour at 100°C) resulted in deterioration in mechanical properties (Alawar *et al*., 2009).

1.12 Research of Banana Trunk Fibers

There are only limited reports on banana trunk fibers available in literature. Idicula *et al*. (2005) reported that user-friendly and cost-effective composite materials possessing appropriate stiffness and damping behaviour can be obtained by hybridising banana fibers. The role of fiber/matrix interactions of chemically modified banana fibre/polyester composites was studied by Pothan *et al*., 2006 and concluded that chemical modification improves the storage modulus of these composites. Sapuan *et al*. (2006) developed composite woven banana fibre reinforced epoxy composites for household utilities. Deepa *et al*. (2010) extracted cellulose nanofibers from banana fibers via a steam explosion technique. The nanofibers were found to enhance thermal properties as compared to the untreated fibers. Polylactic acid (PLA)/ banana fiber biocomposites were fabricated using melt compounding technique by Majhi et al (2010). The obtain results indicate that the improved interfacial adhesion in compatibilized and plasticized systems. Composites were prepared with maleic anhydride modified banana fibers in polypropylene by Chattopadhyay et al. (2010). The composite exhibited an increase in impact strength, flexural strength, flexural modulus and tensile strength. Banana fibers were treated with alkaline pulping and steam explosion to produce banana fibers and banana microfibrils by Ibrahim et al., 2010. The results obtained shows that the banana microfibrils has better compatibility and enhanced mechanical properties as compared to banana fibers. Hybrid composites of Polypropylene (PP) reinforced with intimately mixed short banana and glass fibers were fabricated using Haake twin screw extruder followed by compression molding with and without the presence maleic anhydride grafted polypropy-lene (MAPP) as a coupling agent by Samal *et al*. (2009). The composite exhibited an increase in tensile, flexural and impact strength and a decrease in water absorption rate. The use of banana fibers composites for automotive and transportation application were studies (Herrera-Estrada *et al*., 2008). The banana fiber/epoxy composites were found to yield a flexural strength of 34.99 Mpa and compressive strength of 122.11 Mpa. The alkaline pretreated composite exhibited an improved environmental exposure resistance. Joseph *et al*. (2008) treated banana fibers withsodium hydroxide, silanes, cyanoethylation, heat treatment, and latex treatment. The thermal stability of the treated fibers improved due to its physical and chemical changes induced by the treatments. Hameed *et al*. (2008) used banana stalk waste as a sorbent to sorb methylene blue. The Langmuir isotherm was demonstrated to provide the best correlation for the sorption of methylene blue with an adsorption capacity of 243.90 mg/g.

The author and his principle supervisor have also published several papers pertaining to banana trunk fibers (Mas Haris and Kathiresan, 2009; Kathiresan and Mas Haris, 2010a; Kathiresan and Mas Haris 2010b; Kathiresan *et al.*, 2010c, Mas Haris and Kathiresan, 2010).

1.13 Pollution

Pollution is defined by introduction of contaminant into the environment due to human activities or natural disasters. A major reason for pollution is industrialization. Rapid progress in industrial activities during recent years has led to the discharge of unprecedented amount of wastewater containing synthetic dyes, heavy metals and fossil fuels which pollutes our waters and consequently causes harm to human and other living organisms. A majority of the used dyes are azo reactive dyes (Leechart, *et al*, 2009). These are bright color dyes due to the presence of one or several azo (─N═N─) groups associated with substituted aromatic structures (Forgacs, *et al*., 2004). Effluents from textile, leather, food processing, cosmetics, paper and dye manufacturing industries are some examples of the sources of discharged azo dyes (Bhatnagar and Jain, 2005). These dyes or their breakdown products are toxic to living organisms (Chung *et al*., 1981). Furthermore, dyes in wastewater are difficult to remove because they are stable to light, heat and oxidizing agents. In short, they are not easily degradable (Jain and Sikarwar, 2008). Heavy metals are alsodeposited into our ecosystems (Sanyahumbi *et al*., 1998). These metals are not biodegradable and known to cause severe dysfunction of the kidney, reproductive system, liver, brain and central nervous system (Manahan, 2004). Fossil oil is beyond a doubt currently the most important raw material and energy source worldwide. There have been numerous occasions whereby oil was unintentionally introduced into the environment during production, transportation and refining process causing adverse effect on sea life and human economic activities (Boopathy 2000). Furthermore, oil spill may also cause strong odor to be felt miles away and the excessive growth of green algae may alter the color of sea and landscape (Annunciado *et al.*, 2005).

1.14 Waste Water Treatment

Several methods for removing heavy metal ions such as chemical precipitation, electro-deposition, ion exchange, reverse osmosis and adsorption have been used to treat wastewater

1.14.1 Chemical Precipitation

Selected chemicals are added to form coagulate particles and the particles are allowed to settle down. The treated water is then decanted to remove the contaminants and is appropriately disposed of or reused. This technique can be employed to remove heavy metals, fats, oils and greases, suspended solids phosphorus, fluoride, ferrocyanide and other inorganics compounds. The chemical precipitation is the most economical technique but is inefficient for dilute solution.

1.14.2 Electro-Deposition, Ion Exchange and Reverse Osmosis

Electro-deposition is a technique that deposits dissolved or suspended material by an electric field on an electrode. This technique is usually employed for the removal of heavy metal from water source (Dini, 1993). Ion exchange technique in waste water treatment is used for softening and nitrate removal. Ion exchange is a reversible chemical reaction wherein an ion from solution is exchanged for a similarly charged ion attached to an immobile solid particle. Reverse osmosis demonstrates a high heavy metal rejection, compared to ion exchange but operates on a continuous mode with low reagent consumption. However, these techniques have rather maintenance and operation costs and subject to fouling (Kathiresan and Mas Haris, 2010).

1.14.3 Adsorption

Adsorption techniques are generally categorized as physical adsorption, chemisorption or electrostatic adsorption. Weak molecular forces, namely Van der Waals forces, provide the driving force for physical adsorption. Chemical reaction on the other hand forms a chemical bond between the compound and the surface of the solid in chemisorption. Electrostatic adsorption involves the adsorption of ions through Coulombic forces, and is normally referred to as ion exchange. Adsorption techniques are generally considered as the preferred means for removing and purifying organic substances due to their high efficiency and ability to separate a wide range of chemical compounds (Imamura *et al*., 2002; Gemeay *et al*., 2002; Holzheu and Hoffmann; 2002; Ofomaja and Ho, 2006).This has prompted enormous research interest in the use of agricultural waste as starting materials because of their low-cost and widespread availability.

1.14.3.1 Adsorption of Dyes Using Natural Fibers

Some examples of agrowaste that have been used for the removal of dyes are palm kernel fibers (Ofomaja, 2008) coir pith (Namasivayam *et al*., 2001) sugarcane bagasse (Azahar. *Et al*., 2005), banana peel (Annadurai *et al*., 2002), peanut hull (Gong *et al*.,2005), rice bran and wheat bran (Xue, *et al*., 2008), rice husk (Lakshmi *et al*., 2009) tea waste (Tamez Uddin *et al*., 2009), waste apricot (Onal *et al*., 2007), rubber seed coat (Hameed and Daud, 2008) coconut shell (Singh *et al*., 2008), apple pomace and wheat straw (Robinson *et al*., 2002a), halzelnut shell (Dogan *et al*., 2009), plum kernel (Tseng, 2007), corncob and barley husk (Robinson *et al*., 2002b) , bamboo (Hameed *et al*., 2007), grass waste (Hameed, 2009), pumpkin seed hull (Hameed and El-Khaiary, 2008) and garlic peel (Hameed and Ahmed, 2009).

1.14.3.2 Adsorption of Heavy Metals Using Natural Fibers

Examples of low-cost fibers for the adsorption of heavy metals are pine bark (Al-Asheh and Duvnjak, 1998), cork and yohimbe bark (Villaescusa *et al*., 2000), spent grain (Low *et al*., 2000), peanut hull pellets (Johnson *et al*., 2002), rice milling by-products (Tarley and Arruda, 2004), grape stalk waste (Martinez *et al*., 2006) pectin rich fruit wastes (Schiewer and Patil, 2008), biowaste from fruit juice industry (Senthilkumaar *et al*., 2000). Agave Americana (Hamissa *et al*., 2010), banana trunk fibers (Kathiresan and Mas Haris, 2010), coconut fibers (Igwe *et al*., 2008), oil palm fibers (Isa *et al*., 2008), wheat barn (Farooq *et al*., 2010), coir (Cornad and Hansen, 2007; Shukla and Pai, 2005) and juniper fiber (Min *et al*., 2004)

1.14.3.3 Adsorption of Oil Using Natural Fibers

An efficient methods for combating oil spill as reported in literature is using fibers derived from kapok tree (*Ceiba pentandra*) as a natural hollow hydrophobic–oleophilic sorbent (Lim and Huang, 2007; Abdullah *et al*., 2010). Recently, barley straw waste was also used as an application on oil spill clean up (Husseien *et al*., 2009). Other low cost fibers used for oil spill recovery are wool fibers (Rajakovic *et al*., 2007), cotton grass (Suni *et al*., 2004), reed canary grass, flax and hemp fiber (Pasila, 2004), peat excavation (Suni *et al*., 2004) and saw dust (Banerjee, *et al*., 2006).

1.15 Composite

A composite is a multiphase material that is artificially made, as opposed to one that occurs or forms naturally. In addition, the constituent phases must be chemically dissimilar and separated by a distinct interface (Callister, 2006). Composites are obtained when two or more distinct constituents are combined. The distinct constituents are a reinforced phase of strong material, often fibrous in nature, embedded within a continuous matrix phase which is usually weaker and more compliant. This combination derives a new material with enhanced properties and is distinctively different from those of the individual components. Of late, many researchers are developing composite materials made from natural fiber and resins due to the increased of environmental awareness and interest in long term sustainability of material.

1.15.1 Biocomposite

Biocomposites are composite materials made from natural fiber and petroleum-derived non-biodegradable polymers such as polypropylene, polyethylene or biodegradable polymers such as polylactic acid (PLA) and polyhydroxyalkanoates (PHA). Biodegradable or synthetic polymers reinforced with natural fibers are a viable alternative to glass fiber composites. Biocomposites or biodegradable composites are designed in the hope to contribute to the reduction of environmental load placed upon by the disposal of used plastic product. These biocomposite plastic can be resolved into water and carbon dioxide through an enzymatic reaction by microorganisms. The polymer chains are enzymetically cleaved, and the portions that are small enough are transferred into cells and consumed (Kale *et al*., 2007). Biocomposite materials can lumin the requirements of various applications such as non-food packaging or short – lived application where long – lasting polymers are not entirely adequate. The need for biodegradable composites stimulated extensive research into the design of composites reinforced with natural fibers such as flax, jute, straw, wood fiber, rice husks, wheat, barley, oats, rye, sugar cane, bamboo cane, sisal, paper-mulberry, pineapple leaf fiber and papyrus (Taj *et al.*, 2007). Most of the initial efforts in this area have focused on the replacement of potentially hazardous chemical materials with alternative natural fibers that are often readily available as agricultural by-products (Savastano Jr. *et al.*, 2000) or industrial wastes (Savastano Jr. *et al.*, 2001), with little or no current economic value.

1.15.2 Green Composite

Green composite is defined as biocomposite produced from plant derived fiber and crop/bio-derived plastic and are likely to be more eco-friendly (John and Thomas, 2008). Netravali and Chabba (2003) reported that research effort is being harnessed in developing a new class of fully biodegradable green composites by combining fibers with biodegradable resins. There are several reports on the preparation of green composites (Corrales *et al*., 2007; Huang and Netravali, 2009; Shih *et al*., 2009; Liu and Erhan, 2008; Huang and Netravali, 2007; Gomes *et al*., 2007; Takagi and Asano, 2008; Asasutjarit *et al*., 2009). The major attractions about green composites are that they are environmentally friendly, fully degradable and sustainable. At the end of their life span, they can be easily disposed of or composted without harming the environment.

1.15.2.1 Preparation of Green Composites

Many researchers are attempting to producing green composite for various applications. Corrales *et al*. (2007) chemically modified jute fibers using jute fibers to produce green composite. The chemical modification was carried out in pyridine or dichloromethane solvents using oleoyl chloride as coupling agent. Jute fibers were vacuum dried at 90° C for 24 hr. Reactions were carried out in a dry nitrogen atmosphere by suspending the fibers in the dry solvent. The results obtained indicated that the chemical modification of cellulosic fibers with a fatty acid coupling agents improves the wetting and adhesion of the fiber surface. Biodegradable green composites made using bamboo micro/nano-fibrils and chemically modified soy protein resins were prepared by Huang and Netravali (2009). The objective of their study was to develop fully biodegradable and environment-friendly green composites possessing moderate strength and stiffness. Shih et al. (2009) prepared using biodegradable green composites reinforced by the fiber recycling from disposable chopsticks and poly lactic acid. The results obtained indicate an improvement on the mechanical or thermal properties of the reinforced composite. Fully environment-friendly, sustainable and biodegradable green composites were fabricated using modified soy protein concentrate resins and flax yarns and fabrics (Huang and Netravali, 2007). The produced composite produced improved mechanical properties. A review by Ashori (2008) concluded that wood–plastic composites are a promising green-composite for automotive industries applications due to their superior strength/weight and stiffness/weight ratios. These composites are normally produced by mixing plant fiber with polymer, or by adding wood fiber as filler in a polymer matrix. The mixed samples are pressed or molded under high pressure and temperature. Additives such as colorants, coupling agents, stabilizers, blowing agents, reinforcing agents, foaming agents and lubricants help tailor the end product to the target area of application (Ashori, 2008). Dobircau et al. (2009) prepared new composites materials, having waste cotton fiber as reinforcement in wheat flour. The mixture was extruded with a single-screw extrusion machine at 110 °C and at a rotating speed of 40 rpm. The obtained pellets are homogenized and were extruded again at the similar temperature but with higher rotating speed of 70 rpm to form the desired film. Finkenstadt et al. (2007) produced poly lactic acid green composites using oilseed co-products as fillers. The composites were compounded using a twin-screw extruder with a screw speed of 130 rpm and was the melt temperature was at 160 °C. Poly lactic acid was also used by to produce green composite using chicken feather as reinforcement material (Cheng *et al*., 2009). All samples were prepared by extrusion and injection molding methods. The chicken feather and poly lactic acid was palletized using twin-screw micro extruder with a temperature of 180 °C. The screw speed and the mixing duration were set to be 100 rpm and 10 min, respectively. A Thermo Hakke small scale injection molding machine was used to produce dumbbell-shape composite samples. Green composites and nano composites were prepared by Liu and Erhan (2008) using luminium soyabean oil. Epoxidized soyabean oil and 1, 1, 1-tris (*p*-hydroxyphenyl) ethane triglycidyl ether were mixed in the ratio of 1:0.33 (by weight) at 55 ◦C for 30 min. The mixture was prepared with Aerosil R805 and degassed for 15 min at 55 °C. Designated flax fiber was added to the mixture with mechanical stirring for 15 min. After the mixture cooled to room temperature, curing agent was added with mechanical stirring and then transferred into a designated mold. The flexural modulus and tensile modulus of the composites increased proportionally with the increase in 1, 1, 1-tris (*p*-hydroxyphenyl) ethane triglycidyl ether. Takagi and Asano (2008) prepared environmentally-friendly green composites which were fabricated from starch-based, dispersion-type biodegradable resin and cellulose nanofibers. The preparations of composites were carried out by mixing the biodegradable resin and cellulose. Upon removing excess water, the composites were dried in at 105 °C in air and vacuum. Nanofibers with loading of 70% (wt/wt) were added by hot pressing at a temperature of 140 °C and pressure between 10 – 50 Mpa. The results obtained showed an increase in flexural strength and modulus with increasing molding pressure. These enhanced mechanical properties are attributable partially to increased density. Moreover, the mixing treatment applied to a water solution of dispersion-type resin and low-speed stirrer treatment plays an important role in the strengthening of nanofiber-reinforced composites.

1.16 Industrial Polymers

To date, almost all industry uses polymers in one form or another. The transportation, electronics, construction, medical and aircraft industries rely on these materials to provide a higher quality product at lower cost than conventional materials, such as metals, wood, concrete or rubber. Industrial polymers structures are based on chemical elements such as carbon, oxygen, hydrogen, nitrogen, chlorine and sulfur. These elements are extracted from the air, water, gas, oil, coal, and even from living plants. Researchers past and present took these elements and combined them through various chemical reactions to create an almost unending series of combinations to produce the wide variety of polymers.

1.16.1 Poly Vinyl Alcohol

Poly vinyl alcohol (PVA) is a polymer of great interest in the commercial sector because of its various applications (Hassan and Peppas, 2000). PVA is a versatile polymer with many