5-2004

Processing and optimization of natural fiber reinforced composites using date palm fibers

Khalifa Musabbeh Suwaidan Hamad Al-Kaabi

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Abstract

During the last twenty years, ecological concerns have resulted in a renewed interest in natural materials. Composite materials usually depend on using man-made fibers as the reinforcing element. The possibility of using natural fibers instead of synthetic fibers is gaining acceptance among users. This work aims to investigate the possibility of extracting natural fibers from date palm trees to be used as reinforcement with polymeric materials. This objective was achieved by characterizing fibers through different chemical, physical and mechanical tests and using different treatment methods in order to modify the fiber properties. Date Palm Fibers (DPF) exhibit a cylindrical shape and are composed of a collection of hollow fibers surrounded by relatively thick layers. The density of raw DPF was found to be about 0.92 g/cm³, and its chemical composition is comparable to that of other common natural fibers and consists of approximately 46 wt.% cellulose, 20 wt.% lignin and 18 wt.% hemicelluloses. The diameter of DPF ranges from 100 to 1000 μm. However, thermal analysis by using thermogravimetric analyzer (TGA) under air atmosphere and heating rate 10⁰C/min⁻¹ shows that thermal degradation of DPF starts at 250°C. The tensile strength of DPF ranges from 170 to 275 MPa and the modulus of elasticity ranges from 5 to 12 GPa with an elongation to break of 5-10%. Surface modification of raw DPF affects its surface morphology, density, thermal stability, chemical composition and maximum tensile strength. The fibers were used as reinforcement for a polymeric matrix (polyester resin) and the resulting composites were subjected to different kinds of mechanical and physical testing, which included flexural, impact testing and water absorption. Also, composite specimens containing randomly oriented glass fibers were used for benchmarking. Higher flexural properties (flexural strength and modulus) were achieved for critical length of 2 cm and weight fraction of 9 wt.%. The mechanical properties of specimens prepared using critical length and critical
weight fraction of fibers from different treatment processes have shown variation in the mechanical properties. Also, surface modification of fibers affects the mechanical properties of DPF/polyester composites and specimens prepared with alkali treated DPF yields the highest flexural strength about 70 MPa among other treatment methods.

Furthermore, impact strength (toughness of composites) shows different behavior when using fibers from different treatment methods. Composites prepared with fibers treated with a soda solution giving the highest impact strength about 12 KJm\(^{-2}\). Moisture absorption of DPF/polyester composites was low and did not reach 2% with all types of fibers used but it was higher than moisture absorption of glass/polyester composites which was about 0.7\%.
Acknowledgments

All praise and thanks is due to (GOD), the One, the only and the Indivisible Creator and Sustainer of the World. To Him we belong and to Him we will return. I wish to thank Him for all that He has given us with, although He can never be praised or thanked enough.

The author would like to thank his advisors, Dr. Abdallah Al-Khanbashi and Dr. Adel Hammami for their continuing support and excellent mentorship. Also, the author would like to thank his supervisors and co-workers for their help, encouragement and cooperation during the work period. The author would like to thank his family for their patience and support. The author would like to thank Dr. Galeb Al-Hadrami and Mr. Adel El-Awad from the College of Food Systems for the valuable discussions and the help provided in the preparation of the DPF. The author is also grateful to Dr. Mahmoud Allawy Mohsin from the College of Science for the valuable discussions. Also, the help provided by Dr. Mike Jacobsen, Mr. Stephen Aston and Mr. Steven Raynor in revising the language of the manuscript is appreciated. Eng. Abdulsatar Nooraldeen (Mechanical Engineering Department) United Arab Emirates University is appreciated for help in experimental work. The author would like to thank the librarians of Zayed central library for their help and support in providing articles needed for the literature review of this research. Also, the author wishes to thank Al-Ain Date Factory for providing date palm fibers for this study and the Department of Information of the Dubai Government for providing media about traditional methods of making ropes from date palm fibers. The Central Laboratory Unit, United Arab Emirates especially Mr. Essam Shabana are thanked for their help in SEM examinations and thermogravimetric analysis. Finally, I would like to thank my colleagues in the MSc program, especially, Aladdin Abu-Assi and Ibrahim AL-Saeedi for their endless help, encouragement and support.
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Chapter 1

INTRODUCTION
1. Introduction

1.1 Problem Statement

Since the early 1960s, there has been an ever-increasing demand for newer, stronger, stiffer, and yet lower-weight materials in many fields. High demands on materials for better overall performance has led to extensive research and development efforts in the field of composite materials. Composite materials are widely used in current design practices of many of today's engineering disciplines. The use of composite materials in the aircraft, space, automobile and marine engineering industries is to be noted in particular and it is envisaged that these materials will have a still wider role to play in the future as more is known of their capabilities through continued applied research and development. Tracing the history of composite materials is like taking a fascinating look at the very development of civilized life itself. Composite materials are found in many different applications throughout the ages. Examples include mixed, crushed rock in pottery clay to alleviate shrinkage, riverboats reinforced with bitumen or pitch, straw reinforced building products and the Chinese composite bow [1].

A key factor in selecting a composite material is to eliminate or overcome some property deficiency associated with a certain material by mixing it with other materials, thus forming a new combination with the desired properties. A classical example is polymer/glass fiber composite. This light weight composite combines the strength of small-diameter glass fibers with the ductility of a polymer matrix to provide a product superior to either component alone [2]. The properties of the new material are superior in strength and modulus to many traditional engineering materials such as metals [1]. For these reasons, research and development has greatly increased in the area of composite materials.
Composite materials that exist today can be classified into three main groups. These include ceramic matrix composites (CMCs), metal matrix composites (MMCs) and polymer matrix composites (PMCs) [3]. In this discussion, attention is paid to the latter class of materials (PMCs).

Polymers have made a major impact in the use of modern engineering technology in everyday life. Most people know them as plastics. Nevertheless, the biggest drawback in polymers is a low ultimate strength in comparison with other materials. This has led to the reinforcement of polymers with other materials, typically in the form of fibers, to develop materials that have high specific strength, that is to say, one that is strong and light at the same time. Fibers used to reinforce plastics can be continuous or random short fibers. Different types of fibers, for example glass, Kevlar, carbon, boron and natural fibers such as flax, are used to reinforce plastics.

Continuous-fiber composites offer the best mechanical properties when compared with other fiber-reinforced polymers [2, 3]. They are primarily reinforced with high performance fibers such as carbon, Kevlar or glass. These composites are used in special applications like aircraft components and military vehicles. However, the production of these composites is complex and consumes a lot of time and money [3].

On the other hand, short-fiber composites have a reinforced matrix with chopped fibers, such as glass, graphite and cellulose fibers. These types of composites are used for applications that do not require high strength or stiffness such as automotive components, furniture, building materials, sporting goods and packing applications. Short fiber composites have an advantage over continuous-fiber composites in that they are easily processed. Similar manufacturing techniques used in the polymer industry can be used to produce short-fiber composites. For example, injection molding can be used with a thermoplastic matrix. In the case of thermoset manufacturing, techniques such as hot press
or hand lay up can be used. Such a diversity of techniques used to produce short-fiber composites make mass production and the development of complex shapes possible [3].

As mentioned before, different types of fibers are used as reinforcement for composites. Glass fibers, carbon fibers, and Kevlar fibers are common types. The expensive nature of carbon and Kevlar fibers limits their use to special applications, such as uses in the military and aerospace industries. On the other hand, the low price of glass fibers make it the best option for low or moderate performance applications. Glass fiber composites using continuous, random chopped or woven fibers are found in many applications, such as construction, marine boats, water tanks, pipes, and car door panels. Short glass fiber reinforced polymers are found to be the best solution for interior and non-structural components. The main disadvantages of glass fibers are their high density, abrasive nature and the difficulty of recycling them [4].

There has been a rapid growth in the volume and number of applications of composite materials. However, the disposal of these materials at the end of their intended life is becoming a critical and expensive consideration. The recycling or reuse of composites is often not easily achieved because of the different natures of the various materials forming the composite. Most composites currently end their life in landfills or are incinerated. One particular problem associated with this is the decreasing availability of landfills. For example, the number of landfills in the US dropped from 8000 to 2314 between 1988 and 1998 [5].

Most composites currently available are made from polymeric resins and synthetic fibers based on petroleum, which is a non-renewable resource and subject to price fluctuation. In addition, growing global environmental awareness is causing consumers, manufacturers and governments to find solutions to these problems, or shift from using non-environmental and unsustainable materials to using renewable and biodegradable
materi al. An ex ample of this new trend can be ill ustrated by the action of some
governments that apply ‘take back’ laws, forcing manufacturers to take back their
packa ging and products after their intended use. Another example consists in trying to
ensure that every manufactured component is either recyclable or biodegradable. This is
the reason for attempting to substitute glass fiber with natural fibers, especially in the the
automotive industry with its high mass production. Natural fibers have many advantages
such as low cost, low weight and the fact that they consume CO₂ instead of emitting it.

As mentioned before, natural fibers have been used for a long time in various
applications. Natural fibers were typically used in textiles, ropes, canvas and paper and are
still used in the same way today. As early as 1908, the first natural-fiber reinforced
polymeric material, consisting of phenol formaldehyde resins reinforced with cotton fiber,
was used for electrical insulation [6]. Low costs and the increasing performance of plastics
has led to a decrease in the use of natural fibers. The increase in the price of materials such
as fibers and resins, the high demand for composite materials, environmental regulations
concerning the use of natural resources and recycling, and the low weight of natural fibers
have led to a renewed interest in natural fibers over the last two decades. Natural fibers
may be plant, animal or mineral in origin. Figure 1.1 displays the types of plants that
produce natural fibers and the fibers source.

Natural fibers offer many advantages over their inorganic counterparts such as talc
and glass fiber, that make them more attractive as fillers and reinforcements within a
polymeric matrix. We mentioned some advantages, such as low cost, the fact that they are
derived from renewable resources and their low density. In addition, they are nonabrasive
for tooling; they cause no skin irritations and are known for their low energy consumption.

Despite these advantages, natural fibers exhibit some undesirable characteristics.
High moisture absorption, low thermal resistance and highly anisotropic properties are the
Figure 1.1 Classification of plant fibers from different plants according to the parts which produce fibers [4].
main disadvantages associated with natural fibers. The low thermal stability of natural fibers, about 200°C, is considered to be one of the major limitations when using them with all types of thermoplastic polymers. Only those thermoplastic polymers whose processing temperature does not exceed 200°C are usable for natural fiber reinforced composites. These are mostly polyolefins, like polyethylene and polypropylene. Technical thermoplastics such as polyamides and polycarbonates cannot be used with natural fibers due to processing temperature of more than 200°C [7]. High anisotropic characteristics in the mechanical and physical properties are considered to be a problem in predicting and designing composite parts. This problem becomes more critical when we know that the properties of fibers are affected by some parameters not easily controlled such as chemical composition and the circumstance of growth (climate and rainfall). Also, there are other disadvantages such as local and seasonal quality variations, low compressive strength and poor compatibility with hydrophobic matrices [7,8].

The date palm tree (Fig.1.2-a), a member of the palm tree family (phoenix dactylifera), is normally found in the Middle East, North Africa, the Canary Islands, Pakistan, India, and in the United States (California). There are more than 100 million date palm trees in the world and each tree can grow for more than 100 years [6]. There are about 40 million date palm trees just in the United Arab Emirates, which is considered as one of the largest growers of date palms in the world. The palm tree stem is covered with a mesh made of single fibers. These fibers create a natural woven mat of crossed fibers of different diameters. Traditionally the mat is removed from the trees and cleaned to provide a bundles of fibers used in making ropes and baskets in many parts of the world (Fig.1.2-b).

However, these applications account for a small percentage of the total potential world production. The possibility of finding use for DPF in composite manufacturing will
Figure 1.2 Photograph of date palm tree (a), and date palm fiber after retting from the date palm trees (b).
help open new market for what normally considered waste or used in low value products.

1.2 Research Objectives

The objective of this study is to characterize the Date Palm Fiber (DPF) as a reinforcement for a polymeric matrix. The characterization will include a study of the properties of raw DPF in order to be compared with other common used natural fibers, which are studied in the literature. Raw DPF will go under different surface modification methods such as detergent solution, alkali treatment and bleaching with dioxin solution. Then properties and surface morphology of resulting DPF are investigated. DPF with different length and weight fraction were used with polyester matrix in order to produce composite specimens and evaluate the fiber critical length and weight fraction for this system. Composite specimens with critical length and weight fraction were prepared from fibers subjected to different treatment methods in order to study the effect of treatment processes on their flexural properties. Also, impact testing was used to study the effect of fiber content and treatment methods on toughness of composites specimens. Finally, the moisture absorption of composites containing DPF were determined and compared with glass fibers composites. An introduction to a detailed literature review on the history, development, characterisation of natural fibers, surface modification and its use as a reinforcement with different polymeric matrix is presented in Chapter 2. Chapter 3 will be dedicated to materials and experimental techniques. The raw DPF properties and the effect of different treatment methods are discussed in Chapter 4, in addition to the main processing parameters. Finally, the conclusion and some recommendations for future work presented in Chapter 5.
Chapter 2
LITERATURE REVIEW
2. Literature Review

2.1 Natural Fiber Reinforced Composites: History and Development

During the last 20 years, research involving natural fibers has experienced a phenomenal growth. This research has focused mainly on replacing glass fibers in composite materials. The use of natural fibers was known from a long time ago. There are indications that natural fibers were first utilized at least some 3,000 years ago in ancient Egypt in the mixing of straw and clay to build walls [1, 9]. This method was used until recently in some Middle Eastern countries and may still be in use in some countries such as Yemen. Natural fibers are also used to make ship sails, ropes and clothes, to name a few additional examples [1, 10]. According to some books of antiquity, such as Genesis and Exodus in the Bible, the first reinforced polymeric based materials were used by the people of Babylonia around 4000-2000 BC. Additionally, around 3000 BC, there are many indications that in Egypt and Mesopotamia some types of river boats were constructed from bundles of papyrus reed embedded in a matrix of bitumen [1] as shown in Figure 2.1. Furthermore, it is known that the Assyrians constructed pontoon bridges using floats made from basket-work boats impregnated with a waterproofing of bitumen, earlier than 100 BC. In the Near East, these boats were used and known as gufas. In Western Asia and China at the same time, a composite bow made of wood and horn lamellae was developed. It was small and powerful and was used for firing from horseback.

Bledzki and Gassan [6] report in their review that the first use of natural fibers to reinforce plastic and to produce composite parts was in 1896, when aeroplane seats and fuel-tanks were made of natural fibers with a small content of polymeric binders.
Figure 2.1 Reconstruction comparing Egyptian and Mesopotamian boats of about 3000 BC [1].
In 1908, composite materials made with paper or cotton to reinforce phenolics and extending to melamine were reported in the literature [6, 11].

Due to the increasing performance of technical and standard plastics with a decrease in price, the use of natural fibers was limited to simple applications. In the last few years, environmental concerns such as the need to preserve natural resources and produce goods that can be recycled or that biodegrade, together with the uncertainty of the petroleum supply and price, have accelerated the search for substitutes for materials derived from hydrocarbons [6, 11].

Lignocelluloses materials are widely distributed in the biosphere in the form of trees (wood), plants and crops. Lignocelluloses materials are the most abundant renewable biomaterial of photosynthesis on earth. In terms of mass units, the net primary production per year is estimated to be about $2 \times 10^{11}$ tons as compared with that of synthetic polymers which is about $1.5 \times 10^8$ tons [11].

Some regions in the world, such as India, have continued using natural fibers in many different applications. The use of natural fibers in India has ranged from pipes, pultruded profiles, and panels with polyester matrices. In fact, India is one of the world’s largest producers of jute and coir fibers, with production of jute fiber alone reaching approximately 1.64 million tonnes in 1999-2000 [6, 9].

Around the world there are many research centers, programs and projects focusing on the use of natural fibers as a polymeric matrix reinforcement. One example of these projects is the University of Delaware’s ACRES program (Affordable Composites from Renewable Resources), which received a $11m grant from the U.S. Department of Energy to develop all-natural composites [9]. The technology roadmap for plant/crop-based renewable resources 2020 aims use plant-derived materials to meet 10% of the chemical feedstock demand by 2020 and 50% by 2050 [12]. The Department of Science and
Technology, Government of India, has launched the Advanced Composites Mission program, which aims to increase the use of natural fibers in building materials. Additionally, there are some automotive companies that are developing natural fiber composites. Daimler-Chrysler, for example, has a global natural fiber initiative program that benefits third world nations by developing products made from indigenous agricultural materials [9].

Several conferences research on lignocelluloses composites are held annually to discuss the latest investigations and development in the manufacturing of natural fibers. The Risø International Symposium on Materials Science and International Conferences on Wood-Fiber-Plastic Composites in Madison, WI, USA are two examples of such conferences [13].

In recent years, natural fibers have been used as reinforcements is focused mainly in the automotive and packing industries [6]. It was found that natural fibers could be used in reinforcing plastic and successfully replace glass fibers in low to moderate strength applications, such as door panels and car roofs [9]. This reduces vehicle weight and gives automotive manufacturers other materials to choose from beyond steel and aluminum [3].

In Germany, car manufacturers are aiming to make every component of their vehicles either recyclable or biodegradable, and this will increase the need for natural fibers. Car manufacturers are searching for low cost and low weight parts to be used in interior body components, such as front door liners, rear door liners, boot liners, parcel shelves, seat backs, sunroof interior shields and headrests [9]. The applications of natural fibers in the current Mercedes-Benz E-Class are shown in Figure 2.2. The high volume production in automotive industries and the need for low cost materials make natural fibers the best replacement for synthetic fibers. The mass of natural fibers used in automotive companies differs from one company to another. For example, Mercedes Benz uses
Figure 2.2 Plant fiber applications in the current Mercedes-Benz E-Class [4].
around 22 kg for about 30 components, whereas the BMW 3 series uses around 12 kg [14].

In a 1999 study carried out by UK government agency, the MAFF (Ministry of Agriculture Fisheries and Food) Materials Division, it was found that up to 20 kg of natural fibers could be used in each of the 53 million vehicles being produced globally each year [15].

Although natural fiber applications in the automobiles are growing rapidly, the current major market for natural fibers is in building applications. Types of these products are signs, picnic tables, decks, and landscape timbers. Figure 2.3 gives an example of a plant fiber application, the use of polyester/flex composites in the construction of a catamaran hull.
Figure 2.3 Catamaran boat hull made from polyester/flax composites [9].
2.2 Plant Fibers Types, Production and Consumption

Natural fibers are classified according to their source: animal, mineral or plant. Wool and hair are examples of animal fibers. Glass and asbestos fibers are types of mineral fibers. This project focuses on the third type, natural fibers that are derived from plants. Plant fibers are termed cellulosic or lignocellulosic fibers in some references in according to their main chemical composition. Prabh [14] reports that natural fibers are reinforced materials which contain microfibrils cellulose in an amorphous matrix composed of hemicelluloses and lignin. Add to these, plant fibers are classified according to which part of the plant they are extracted from. Table 2.1 shows a classification of fibers from different plant parts and some of their properties.

One of the biggest problems in using natural fibers in composites is the variation of fiber properties from one growing season to the next [16]. This is due to the effect on fiber chemical composition, and thus fiber properties, from the growth circumstance, such as climate conditions.

Even so, Prabhu [14] reported that the world production of natural fibers was around 25 million tons annually and only 15% of this production goes into the manufacture of composites. The availability of natural fibers seems to be exceeding the demand. The world production of commercially types of fibers is shown in Table 2.2. This table shows that wood fiber is the most abundant among all fibers. Europe is considered as the biggest world consumer of natural fibers and the most commercialized fiber is flax which is suitable for the European environment. As a result, the use of natural fibers in composites is much higher in Europe than in North America. This could be attributed to many reasons such as the increased concern for the environment, shortage of landfills, and high cost of waste management for European countries such as Germany, Belgium and the Netherlands [11]. Table 2.3 shows the dramatic increase in the use of natural fibers in Germany and the
Table 2.1 Types of fibers from different parts of plants and some properties [11,17].

<table>
<thead>
<tr>
<th>Parts of plant</th>
<th>Types of plants</th>
<th>Notes</th>
</tr>
</thead>
<tbody>
<tr>
<td>Leaf fibers</td>
<td>Sisal, abaca, esparto, henequen and pineapple leaf fiber.</td>
<td>They are called hard fibers.</td>
</tr>
<tr>
<td>Bast fibers</td>
<td>Jute, flax, hemp and kenaf.</td>
<td>They come as bundles from the inner bark (of the stems of dicotyledonous plants).</td>
</tr>
<tr>
<td>Seed and fruit hairs</td>
<td>Cotton and coconut (coir).</td>
<td>Fibers come from seed-hairs.</td>
</tr>
<tr>
<td>Straw fibers</td>
<td>Corn, wheat, and rice straws.</td>
<td></td>
</tr>
<tr>
<td>Grass and reed fibers</td>
<td>Bamboo and sugar cane, switch grass, elephant grass etc.</td>
<td>This fibers come from the stems of monocotyledonous plants.</td>
</tr>
<tr>
<td>Wood fibers</td>
<td>Yellow paper, spruce, soft and hard woods.</td>
<td>They are comes from xylem of angiosperm as hard wood or from gymnosperm as soft wood.</td>
</tr>
<tr>
<td>Fiber source</td>
<td>World production ($10^3$/tonnes)</td>
<td></td>
</tr>
<tr>
<td>--------------</td>
<td>----------------------------------</td>
<td></td>
</tr>
<tr>
<td>Wood</td>
<td>1,750,000</td>
<td></td>
</tr>
<tr>
<td>Bamboo</td>
<td>10,000</td>
<td></td>
</tr>
<tr>
<td>Cotton lint</td>
<td>18,450</td>
<td></td>
</tr>
<tr>
<td>Jute</td>
<td>2,300</td>
<td></td>
</tr>
<tr>
<td>Kenaf</td>
<td>970</td>
<td></td>
</tr>
<tr>
<td>Flax</td>
<td>830</td>
<td></td>
</tr>
<tr>
<td>Sisal</td>
<td>378</td>
<td></td>
</tr>
<tr>
<td>Roselle</td>
<td>250</td>
<td></td>
</tr>
<tr>
<td>Hemp</td>
<td>214</td>
<td></td>
</tr>
<tr>
<td>Coir</td>
<td>100</td>
<td></td>
</tr>
<tr>
<td>Ramie</td>
<td>100</td>
<td></td>
</tr>
<tr>
<td>Abaca</td>
<td>70</td>
<td></td>
</tr>
<tr>
<td>Sun hemp</td>
<td>70</td>
<td></td>
</tr>
</tbody>
</table>
Table 2.3 European use of natural fibers (tonnes) [9].

<table>
<thead>
<tr>
<th>Fiber</th>
<th>1996</th>
<th>1999</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Germany</td>
<td>Rest of Europe</td>
</tr>
<tr>
<td>Flax</td>
<td>1800</td>
<td>Na</td>
</tr>
<tr>
<td>Jute</td>
<td>1800</td>
<td>300</td>
</tr>
<tr>
<td>Hemp</td>
<td>Na</td>
<td>Na</td>
</tr>
<tr>
<td>Kenaf</td>
<td>400</td>
<td>Na</td>
</tr>
<tr>
<td>Sisal</td>
<td>Na</td>
<td>Na</td>
</tr>
<tr>
<td>Totals</td>
<td>4000</td>
<td>300</td>
</tr>
</tbody>
</table>
rest of European countries between 1996 and 1999. By comparing the production of flax fiber in Table 2.2 and to the total European consumption shown in Table 2.3, only 2% of the total production is consumed in Europe. Also, it is noted from Table 2.3 that the use of flax fibers in Germany increased from 1996 to 1999 by about 511%. Furthermore, when considering all natural fibers in Germany and the rest of the European countries from 1996 to 1999, the increase ranged from 287% to 2200%, respectively.

As previously mentioned, the price of natural fibers is lower than that of glass fibers. However, the price differs from one type of natural fiber to another. Comparison in price between glass fibers and some common natural fibers ($/Kg) is shown in Table 2.4. Mohanty et al [11] reported that a substitution of glass fibers by natural fibers is not easily realized from an economic point of view. This is because having a continuous supply of glass fibers having constant properties at a higher price is better than using low cost natural fibers with a fluctuation in price and properties. Furthermore, it was found that the cost of natural fibers varied greatly depending on the place of origin and the economy of that source.
Table 2.4 Price comparisons between glass fibers and some natural fibers (1993) [11].

<table>
<thead>
<tr>
<th>Fiber</th>
<th>Price in comparison to glass fiber (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Glass</td>
<td>100</td>
</tr>
<tr>
<td>Jute</td>
<td>18</td>
</tr>
<tr>
<td>Flax</td>
<td>130</td>
</tr>
<tr>
<td>Sișal</td>
<td>21</td>
</tr>
<tr>
<td>Banana</td>
<td>40</td>
</tr>
<tr>
<td>Coir</td>
<td>17</td>
</tr>
</tbody>
</table>
2.3 Chemical, Structural, Physical and Mechanical Properties of Natural Fibers

It is important to know the chemical, structural, physical and mechanical properties of fibers in order to predict their contribution as a reinforcement in the design of composite materials. For example, in order to find the volume fraction of reinforcement in composites, the density of fibers must be known. Factors affecting the chemical composition of natural fibers also affect the fiber's structure and physical properties. The following sections will include a discussion about the main polysaccharides composed of natural fibers, structural of natural fibers and physical and mechanical properties of natural fibers.

2.3.1 Chemical composition of natural fibers

The chemical composition of plant fibers contains many different natural polymer elements. Cellulose, hemi-cellulose and lignin are the main components of plant fibers and represent more than 70% of their chemical composition. Only cotton doesn’t contain lignin in its chemical composition and has the highest percentage of cellulose. The other components are pectin, wax and moisture content [6]. The chemical composition of some common plant fibers from different references is shown in Table 2.5.

Baley [18] mentions that there are many factors that can affect the chemical composition of plant fibers, and thus their properties. These variations can be attributed to many factors, such as plant species and variety, agricultural variables such as soil quality, weathering conditions, plant maturity, quality of the retting process and the measurement conditions. Chemical composition analyses for flax fibers taken from different authors are
Table 2.5 Chemical composition of some natural fibers [6, 11].

<table>
<thead>
<tr>
<th>Type of fiber</th>
<th>Cellulose Wt.-%</th>
<th>Lignin Wt.-%</th>
<th>Hemicellulose Wt.-%</th>
<th>Pectin Wt.-%</th>
<th>Wax Wt.-%</th>
<th>Moisture content Wt.-%</th>
</tr>
</thead>
<tbody>
<tr>
<td><strong>Bast</strong></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Jute</td>
<td>61-71.5</td>
<td>12-13</td>
<td>13.6-20.4</td>
<td>0.2</td>
<td>0.5</td>
<td>12.6</td>
</tr>
<tr>
<td>Flax</td>
<td>71</td>
<td>2.2</td>
<td>18.6-20.6</td>
<td>2.3</td>
<td>1.7</td>
<td>11.0</td>
</tr>
<tr>
<td>Hemp</td>
<td>70.2-74.4</td>
<td>3.7-5.7</td>
<td>7.9-2.4</td>
<td>0.9</td>
<td>0.8</td>
<td>10.8</td>
</tr>
<tr>
<td>Ramie</td>
<td>68.6-76.2</td>
<td>0.6-0.7</td>
<td>13.1-16.7</td>
<td>1.9</td>
<td>0.3</td>
<td>8.0</td>
</tr>
<tr>
<td>Kenaf</td>
<td>31-39</td>
<td>15-19</td>
<td>21.5</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td><strong>Leaf</strong></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Sisal</td>
<td>67-78</td>
<td>8.0-11.0</td>
<td>10.0-14.2</td>
<td>10.0</td>
<td>2.0</td>
<td>11.0</td>
</tr>
<tr>
<td>PALF</td>
<td>70-82</td>
<td>5-12</td>
<td></td>
<td></td>
<td></td>
<td>11.8</td>
</tr>
<tr>
<td>Henequen</td>
<td>77.6</td>
<td>13.1</td>
<td>4-8</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td><strong>Seed</strong></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Cotton</td>
<td>82.7</td>
<td></td>
<td>5.7</td>
<td></td>
<td>0.6</td>
<td></td>
</tr>
<tr>
<td><strong>Fruit</strong></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Coir</td>
<td>36-43</td>
<td>41-54</td>
<td>0.15-0.25</td>
<td>3-4</td>
<td></td>
<td>8.0</td>
</tr>
</tbody>
</table>
shown in Table 2.6. A characteristic of this table is the variation in the chemical composition between one author and another, which is due to previously mentioned reasons. Next part will discusses the main polysaccharides composed natural fibers.

a. Cellulose

The term “cellulose” was first used by Anselme Payen in 1838, when he discovered that when plant tissue and cotton linters were purified with an acid-ammonia treatment and extracted in water, a constant fibrous material was formed [10]. Cellulose considered to be a type of polysaccharide containing many linked monosaccharides [19]. Cellulose fibers can be man-made (regenerated) or natural (native) and are widely distributed in nature as one of the principal constituents of cotton, kapok, flax, hemp, jute, ramie and wood.

The chemical structure of cellulose is the same for different types of natural fibers, whereas the degree of polymerization (DP) varies [19]. This difference affects the mechanical properties and it is reported that natural fibers derived from the bast have the highest degree of polymerization (DP) [11].

Cellulose is an unbranched polymer consisting of D-anhydroglucopyranose units (known as glucose for convenience) joined by 1,4,β-glycosidic bonds [6, 19]. The cellulose consists of linear chains of cellobiose units that may be detected using X-ray examination [19]. Figure 2.4 shows the main unit of cellulose macromolecule, which contains three alcohol hydroxyls (-OH) forming hydrogen bonds inside the macromolecule itself and attract moisture through hydrogen bonding. This gives the a hydrophilic nature of the fibers, thus allowing the moisture content to reach between 8 and 12.6% [20]. Cellulose is also highly crystalline and may contain as much as 80% crystalline regions [21]. The determination of cellulose content in fibers could be done either by using the Van Soest method [22] or the method described in Callum et el work [23].
Table 2.6 Different chemical compositions of flax fibers from different references [18].

<table>
<thead>
<tr>
<th>Cellulose (%)</th>
<th>Hemicellulose (%)</th>
<th>Pectin (%)</th>
<th>Lignin (%)</th>
<th>Wax (%)</th>
<th>Water (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>64.1</td>
<td>16.7</td>
<td>1.8</td>
<td>2.0</td>
<td>1.5</td>
<td>10.0</td>
</tr>
<tr>
<td>67</td>
<td>11</td>
<td>–</td>
<td>2.0</td>
<td>–</td>
<td>–</td>
</tr>
<tr>
<td>73.8</td>
<td>13.7</td>
<td>–</td>
<td>2.9</td>
<td>–</td>
<td>7.9</td>
</tr>
</tbody>
</table>
Figure 2.4 Partial structure of a cellulose molecule shows the β-linkages of each glucose unit [19].
b. Hemicelluloses

In addition to cellulose, other types of polysaccharides called "hemicelluloses," are present in plant fibers. There is an agreement that hemicellulose is not a form of cellulose [6, 21]. It comprises a group of polysaccharides that remain associated with the cellulose after the lignin has been removed [10].

Hemicellulose differs from cellulose in three important respects. In the first place, it contains several different sugar units whereas cellulose contains only 1,4-β-D-anhydroglucopyranose units. Second, hemicelluloses exhibit a considerable degree of chain branching, whereas cellulose is a strictly linear polymer. Third, the degree of polymerization of native cellulose is ten to one hundred times higher than that of hemicellulose [6]. Moreover, hemicellulose tends to differ from one plant to another in its constituents. An example structure of hemicelluloses is shown in Figure 2.5.

c. Lignin

Lignin is a complex hydrocarbon polymer with both aliphatic and aromatic constituents [23]. During the synthesis of plant cell walls, polysaccharides such as cellulose and hemicellulose are laid down first, and lignin fills the spaces between the polysaccharide fibers, which cements them together [11]. The exact structural formula of lignin in natural fiber has not yet been established; however, most of the functional groups and units which make up the molecule have been identified [11]. Figure 2.6 shows one of the probable molecular structures of lignin. Lignin monomer units have various ring-substituted phenyl-propanes which are linked together in ways not well understood [6, 10]. Bledzk [20] reports that lignin is considered to be one of the most important elements contributing to natural fibers. Mohanty et al [11] mentioned that the high carbon and low hydrogen content of lignin suggests that lignin is highly unsaturated or aromatic in
Figure 2.5 The structure of hemicellulose [6].
Figure 2.6 Possible structure of lignin [7].
character and that the main difficulty in lignin chemistry is that no method has so far been established by which it is possible to isolate the lignin in its native state from the fiber. They go on to mention that by filling the space between cellulose and hemicellulose, lignin protects the fiber from chemical and physical effects.

d. Pectin and Waxes

Pectin is a heteropolysaccharide and it is soluble in water only after a partial neutralization with alkali or ammonium hydroxide [6, 10]. The Waxes consist of various alcohols and form a small percentage (less than 2 Wt.%) of the structure [10]. Due to the existence of alcohols, waxes are insoluble in water or acids but can be extracted with organic solutions [6]. Dewaxing of the natural fibers surface is a very well known process. Many authors have used this process as the main treatment method in the surface modification of fibers or as an earlier step of a treatment process [24, 25].

2.3.2 Structural of natural fibers

Beldzki and Gassan [6] report that all plant-based natural fibers consist of single fibers of various cells. Plant fibers are threadlike (microfibrils) connected by lignin and hemicellulose. The microfibrils are crystalline and based on cellulose. Whereas lignin and hemicellulose are described as amorphous regions [6, 11]. X-ray diffraction shows that the cellulose in these fibers has a higher degree of order and crystallinity [26]. A summary of some international research projects on microfibrils angle measurements is found in Eichhorn et al at [10]. Another important study and a comprehensive discussion of plant fiber structure is presented by Herman et al at [26]. Figure 2.7 shows schematic representation of a cotton fiber.
Figure 2.7 Schematic representation of a cotton fiber [26].
The deposition of cellulose never fills the cell completely and leaves a center canal called the micro void (lumen) [6]. There are three secondary layers in the fibers which stick together as a composite, with one as a primary. Figure 2.8 shows these different layers: S₁ (outer layer), S₂ (middle layer), and S₃ (inner layer). It can be noted that S₂ is the thickest of the layers. Furthermore, these cell walls differ in their composition (amount of cellulose and lignin/hemicellulose) and in the orientation of the cellulose microfibrils [6].

This orientation is an important factor in the mechanical properties of natural fibers and is called the spiral angle. Table 2.7 shows values for the spiral angle for some common natural fibers. Spiral angle is defined as the angle between microfibrils cellulose and the fiber axis. It is different from one plant to another. Usually a decrease in the spiral angle will enhance the mechanical properties of the fibers [27].

Another important feature of natural fiber, which prevents it from attaining the high crystal modulus of cellulose, is the presence of defects. These defects not only reduce the modulus but also cause anisotropy in the resulting composite material [10].

As is shown in Figure 2.8, natural fibers consist of cellulose microfibrils, with diameters in the order of a couple of nanometers, and bonded together by an organic matrix to form fiber bundles of different diameters. If the fiber opening process is taken further down to the microstructure level, the microfibrils obtained can be used as a reinforcement element in nano-composites [28].

2.3.3 Physical and mechanical properties of natural fibers

Density and diameter are two important parameters when fibers are used as reinforcement. Tensile strength and modulus, which are critical factors in characterizing fibers, depend on these parameters. Natural fibers exhibit considerable variation in diameter along the length of individual filaments. Composite properties are greatly

34
Figure 2.8 Constitution of a natural fiber cell [18].
Table 2.7 Natural fibers and spiral angle [6].

<table>
<thead>
<tr>
<th>Fibers</th>
<th>Spiral angle (°)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Jute</td>
<td>8</td>
</tr>
<tr>
<td>Flax</td>
<td>10</td>
</tr>
<tr>
<td>Hemp</td>
<td>6.2</td>
</tr>
<tr>
<td>Ramie</td>
<td>7.5</td>
</tr>
<tr>
<td>Sisal</td>
<td>20</td>
</tr>
<tr>
<td>Coir</td>
<td>45</td>
</tr>
</tbody>
</table>
affected by fiber properties. Thus, natural fiber properties are highly variable and it is difficult to obtain the same mechanical properties after repeated testing.

There is a strong relationship between the fiber internal structure, chemical composition and properties such as density, ultimate tensile strength and initial modulus [11]. The density of natural fibers is lower than that of glass fiber. This improves the specific properties of the natural fibers when compared to glass fiber, especially stiffness. Natural fibers are about 50% lighter than glass fibers. Table 2.8 displays the properties of various fibers, including density, diameter, tensile strength, Young’s modulus and an elongation to break. Mohanty et al [11] in their review report that chemical composition, diameter, microfibrillar angle and growing conditions affect the mechanical properties of the fiber. Table 2.8 shows that in general flax fibers have lower diameter and higher mechanical properties (tensile strength and Young’s modulus), whereas coir fibers have a larger diameter and lower mechanical properties. As previously mentioned, a lower spiral angle is preferred to achieve higher strength and modulus. Flax, jute, hemp and ramie fibers have low spiral angles (Table 2.7) and better mechanical properties. On the other hand, coir and sisal have larger spiral angles and worse mechanical properties.

The relationship between fiber diameter and Young’s modulus has also been analyzed. Specifically, Bally and Roberts [18,29] report the relationship between flax fiber diameter and Young’s modulus. These two studies agree that fibers with high diameter has low modulus, as shown in Figure 2.9.

Mohanty et al [11] report that its not possible to exactly correlate fiber strength with cellulose content and microfibrillar angle because of the complex structure of natural fibers and since the filament and individual fiber properties can vary widely depending on the source, age, separating technique, moisture content, speed of testing and history of fiber [11].
Table 2.8 Comparative properties of some natural fibers and conventional man-made fibers [6, 10, 11, 18, 21, 30].

<table>
<thead>
<tr>
<th>Properties</th>
<th>Density (g/cm³)</th>
<th>Diameter (µm)</th>
<th>Tensile strength (MPa)</th>
<th>Young’s modulus (GPa)</th>
<th>Elongation at break (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Jute</td>
<td>1.3-1.45</td>
<td>25-200</td>
<td>393-773</td>
<td>13-26.5</td>
<td>1.16-1.5</td>
</tr>
<tr>
<td>Flax</td>
<td>1.4-1.50</td>
<td>10-40</td>
<td>600-2000</td>
<td>12-85</td>
<td>1-4</td>
</tr>
<tr>
<td>Hemp</td>
<td>1.48</td>
<td>-</td>
<td>690</td>
<td>-</td>
<td>1.6</td>
</tr>
<tr>
<td>Ramie</td>
<td>1.50</td>
<td>-</td>
<td>400-938</td>
<td>61.4-128</td>
<td>1.2-3.8</td>
</tr>
<tr>
<td>Sisal</td>
<td>1.45-1.50</td>
<td>50-200</td>
<td>468-640</td>
<td>9.4-22.0</td>
<td>3-7</td>
</tr>
<tr>
<td>PALF</td>
<td>1.44</td>
<td>20-80</td>
<td>413-1627</td>
<td>34.5-82.51</td>
<td>1.6</td>
</tr>
<tr>
<td>Cotton</td>
<td>1.5-1.6</td>
<td>-</td>
<td>287-800</td>
<td>5.5-12.6</td>
<td>7.0-8.0</td>
</tr>
<tr>
<td>Coir</td>
<td>1.15</td>
<td>100-450</td>
<td>131-175</td>
<td>4-6</td>
<td>15-40</td>
</tr>
<tr>
<td>Banana</td>
<td>1.35</td>
<td>80-250</td>
<td>529-754</td>
<td>7.7-20.8</td>
<td>-</td>
</tr>
<tr>
<td>E-glass</td>
<td>2.5</td>
<td>-</td>
<td>2000-3500</td>
<td>70</td>
<td>2.5</td>
</tr>
<tr>
<td>S-glass</td>
<td>2.5</td>
<td>-</td>
<td>4570</td>
<td>86</td>
<td>2.8</td>
</tr>
<tr>
<td>Aramid</td>
<td>1.4</td>
<td>-</td>
<td>3000-3150</td>
<td>63-67</td>
<td>3.3-3.7</td>
</tr>
<tr>
<td>Carbon</td>
<td>1.7</td>
<td>-</td>
<td>4000</td>
<td>230-240</td>
<td>1.4-1.8</td>
</tr>
</tbody>
</table>
Figure 2.9 Flax fiber modulus as a function of fiber diameter [29].
2.3.4 Degradation of natural fibers

There are many possible factors that degrade natural fibers. These degradations may be biological, thermal, photochemical, chemical or mechanical in origin. In order to produce cellulose fiber-based composites with a long service life, the degradation processes caused by nature need to be retarded. One way of preventing or slowing down the natural degradation process is by modifying the cell wall chemistry which is responsible for many of the fiber's properties [21]. Table 2.9 shows how the components of lignocellulosics fiber interact in various ways.

a. Biological degradation

This property has both an advantage and a disadvantage. Biological degradation is desirable at the end of a material’s life. However, degradation during use or storage of the material cannot be accepted. Biodegradation of high molecular weight cellulose leads to weaker fibers [11]. Natural fibers degrade biologically because organisms such as fungi, bacteria, insects and termites attack the carbohydrate polymers (mainly the hemicelluloses) in the cell wall and have specific enzyme systems to hydrolyze the polymers to digestible units. Some studies report the use of acetylation to improve the resistance of wood and to allow fibers to be less susceptible to biological decay [23].

b. Moisture absorption

Natural fibers are known to be hydrophilic in nature and absorb moisture readily. Absorption of water by cellulose is an important process due to the existence of hydroxyl groups in the polymers forming the cell wall of fibers (see Section 2.3.1-a). Water bonded to the OH groups in the amorphous regions (hemicellulose) and on the surfaces of the crystalline regions is less strongly bonded (crystalline cellulose) [26]. Moisture absorption
Table 2.9 Cell wall polymers responsible for the properties of lignocellulosics in the order of importance [11].

<table>
<thead>
<tr>
<th>Biological degradation</th>
</tr>
</thead>
<tbody>
<tr>
<td>- Hemicellulose</td>
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<tr>
<td>- Accessible cellulose</td>
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<td>- Non-crystalline cellulose</td>
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<tr>
<td>- Crystalline cellulose</td>
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<tr>
<td>- Lignin</td>
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<tr>
<th>Moisture absorption</th>
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<tbody>
<tr>
<td>- Hemicellulose</td>
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<td>- Accessible cellulose</td>
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<td>- Lignin</td>
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<td>- Crystalline cellulose</td>
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<th>Ultraviolet Degradation</th>
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<tr>
<td>- Lignin</td>
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<tr>
<td>- Hemicellulose</td>
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<tr>
<td>- Accessible cellulose</td>
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<td>- Non-crystalline cellulose</td>
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<td>- Crystalline cellulose</td>
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<table>
<thead>
<tr>
<th>Thermal degradation</th>
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</thead>
<tbody>
<tr>
<td>- Hemicellulose</td>
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<tr>
<td>- Cellulose</td>
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<tr>
<td>- Lignin</td>
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<table>
<thead>
<tr>
<th>Strength</th>
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<tbody>
<tr>
<td>- Crystalline cellulose</td>
</tr>
<tr>
<td>- Matrix (Non-crystalline</td>
</tr>
<tr>
<td>cellulose + Hemicellulose</td>
</tr>
<tr>
<td>+ Lignin)</td>
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<tr>
<td>- Hemicellulose</td>
</tr>
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</table>
is classified as one among the main limitations of using natural fibers. From Table 2.10 we see that hemicelluloses play a major part on water absorption from all other main polysaccharides that compose natural fibers. Surface modification processes such as acetylation are used to decrease the moisture absorption of fibers.

c. Thermal degradation

At temperatures around 200°C, thermal degradation of natural fibers begins. Curing temperatures for thermoset polymers are below this temperature, whereas the melting points of thermoplastic polymers lie below and above this point. Thus, the processing temperature of natural fiber reinforced polymeric materials needs to be below 200°C in order to avoid the possibility of fiber degradation. Studies of thermal degradation are important to understand the thermal effect in the loss of the desirable fiber properties. The problem of thermal stability is considered the main limitation in using natural fibers with all types of thermoplastics, and limits their use to only low melting polymers [7]. Thermogravimetric analysis is used to examine fibers thermal stability, and a decrease in weight at temperatures less then 100°C is usually attributed to the loss of absorbed water.
2.4 Surface Modification of Natural Fibers

The literature on cellulose surface modification is extensive and this overview covers only the most common methods used in composite applications. The properties of composites depend on the matrix and fibers and on their interfacial compatibility, whether the fibers are natural or synthetic. The surface of synthetic fibers is usually modified using various types of processes in order to improve the fiber surface wettability with the matrix and to create a strong bond at the fiber-matrix interface which, in turn, provides an effective stress transfer between the matrix and the reinforcement fibers [1, 3]. When it comes to using natural fiber reinforcement in composite materials, many problems occur at this interface due to imperfect bonding [13]. Therefore, surface modification of the fibers by chemical treatments is one of the largest area of research. Several researchers are currently investigating how to improve compatibility and interfacial bond strength. Also, this modification of the fibers can both increase or decrease the strength of the fibers, and thus an understanding of what occurs structurally is of great importance [10].

A high degree of adhesion between fibers and matrix is desired in order to obtain the best mechanical properties. A poor interfacial interaction leads to internal strains, porosity, environmental degradation, moisture absorption and poor mechanical properties [20]. Adhesion and interlocking between composite components can be improved by a high degree of compatibility, an ability of the resin to wet fibers and a fiber surface clear of impurities [3, 24, 25, 29]. On the other hand, the absorption of moisture by untreated fibers, poor wettability and insufficient adhesion between polymer matrix and fibers lead to de-bonding over time.

Wetting is an important factor affecting strong interfacial adhesion. There are many parameters that affect the wettability of fibers, such as the viscosity of the polymers
and the surface tension of both materials [3]. A low polymer surface tension is better and this affects the choice of the matrix.

As previously mentioned, the modification of the fiber and/or the polymer matrix in order to obtain the optimum fiber-matrix properties is a current key area of research. Up to now, most research has focused on the interfacial bond strength within the polymer matrix in order to improve the properties of natural fiber composites [7, 13, 20, 21, 29]. There are some limitations to the modification processes, due to the thermal limitation of natural fibers (Section 2.3.4-b). If cellulose fibers are damaged thermally, they will change into other compounds, which affects the composite’s properties. Additionally, interaction between natural fibers and strong concentrations of chemical solutions used (i.e., soda or acid) may degrade the fibers chemically. Therefore, understanding the fibers’ limitations during surface treatment is very important in order to avoid degradation of the fiber’s properties.

The surface of natural fibers is influenced by the fiber morphology (composition of fibers), chemicals used in treatment and processing conditions. The use of different kinds of physical (example, corona discharge and plasma) and chemical surface treatment methods (i.e. alkali, acetylation, bleaching and coupling agents such as silanes) leads to changes in the surface structure of the fibers, as well as to changes in the surface energy which can be determined by using contact angle analysis [6]. The polar and hydrophilic nature of natural fibers (cellulose is considered a strong polar polymer) have a compatibility problem with non-polar hydrophobic polymers [7]. This problem and others such as impurities, waxy substances and the presence of free water and hydroxyl groups on fiber surface, makes treatment essential. Next two sections will discuses some physical and chemical treatment methods used with natural fibers.
2.4.1 Physical treatment methods

Natural fibers can be modified by physical methods such as stretching, calendaring, thermotreatment and the production of hybrid yarns. These types of treatment processes do not change the chemical composition of fibers, but do affect fiber structure and its surface properties [20]. This means that physical treatment changes the fiber matrix bonding and, consequently, the composites mechanical properties. Electric discharge (corona, cold plasma) is another type of physical treatment. Corona treatment is one of the most interesting techniques used for surface oxidation activation. This process changes the surface energy of the cellulose fibers [6]. Gassan and Voyleks [31] use corona discharged and ultraviolet (UV) treatment to improve the mechanical properties of composites fabricated from jute fiber reinforced epoxy. They found that individual jute fibers treated with corona discharge exhibit a significant increase in the polar component of the free surface energy with an increase in corona energy. The UV treatment of the jute yarn led to a higher polarity (up to a 200% increase) with increased treatment time. Treatment distance has a significant effect on fiber strength. Improvement of a composite's mechanical properties needs to be balanced between increased polarity of the fiber's surface and a decrease in fiber strength. By using an optimum treatment condition, an increase of a composite's flexural strength of about 30% has been achieved.

This research focused on the effect of chemical treatment methods, a review of such methods is described in the following section.

2.4.2 Chemical treatment methods

Alkali and acetylation treatment processes are cost effective and have been widely used to improve natural fiber surface properties, and thus, composite properties. Besides these, many others chemical modification methods designed to enhance the interfacial
adhesion between fibers and matrix have been researched. The following two sections review the use of alkali and acetylation treatment methods.

a. Alkali treatment of natural fibers

The importance of alkali treatment methods can be attributed to a very effective surface modification of the natural fibers at a low cost. Alkali treatment is known to extract residual lignin, hemicellulose and other impurities and promote fiber fibrillation. The treatment is also known to enhance the reactivity of cellulose towards various chemicals by means of restricted swelling of crystalline regions and possible cellulose structural rearrangement [13]. Furthermore, it is known to decrease the spiral angle of cellulose microfibrils, which increases fiber strength [6].

Eichhorn et al [10] stated in their review that the alkalinization process affects natural fibers and has particular importance for fiber-matrix adhesion by the creation of high fiber surface area, which is required for the optimization of fiber resin reinforcement. They concluded that the modification of cellulose fibers, therefore, develops by changes in the morphology and increases in the hydroxyl groups. These changes effectively result in improved surface tension, wettability, swelling, adhesion and compatibility with polymeric materials. However, high concentrations of caustic soda result in a decrease of fiber tensile strength due to notched grooves at the plant fiber surface.

Cellulose and other natural fiber components have varying reactions to a soda solution. Hemicellulose is considered to be very sensitive to soda solution, whereas lignin and cellulose are only slightly affected. Some studies mention the removal of lignin and hemicellulose from plant fibers, which has the effect of changing the mechanical properties of the fibers. Removing hemicellulose leads to the internal region being less dense and less rigid. This gives the fibers the ability to rearrange themselves along the direction of tensile deformation and this increases their strength. With soda treatment, lignin is
gradually removed, and the middle lamella becomes more plastic as well as homogeneous due to the gradual elimination of microvoids [6]. Figure 2.10 shows the improvement in the ultimate tensile strength of coir fibers a function of soaking time in 5% soda solution.

In another investigation carried out by Rout et al [25], coir fibers were treated with a 10% alkali solution for 1 hr at 30°C. This resulted in about 58% improvement in the ultimate tensile strength when compared with untreated coir fibers. This result can be attributed to an increase in the degree of crystallinity. Many other authors report an increase in crystallinity in coir and flax fibers through alkaline treatment. This increase in the crystallinity can be attributed to the removal of cementing materials (lignin and hemicellulose) and better packing of the cellulose chains [10]. Alkali treatment also leads to a decrease in spiral angle by increasing the molecular orientation. This modification in molecular orientation leads to higher Young’s modulus.

As previously mentioned, one of the main problems with using natural fibers as a reinforcement in composite materials is imperfect bonding, which leads to void formation and improper adhesion between fibers and matrix. Alkali treatment could increase the quality of the composite due to possible improvement in fiber-matrix adhesion and compatibility. Some studies have confirmed this improvement by carrying out pull-out test on fibers before treatment and after treatment [32].

Mohanty et al [7] investigate alkali treatment with kenaf and henequen fibers which used as a reinforcement for polypropylene. These natural fibers are hydrophilic and thus need suitable surface modification to achieve better compatibility with a hydrophobic polymer matrix in order to improve its properties. They found that using an alkali treatment for fibers with polyolefins was very efficient in improving fiber-matrix adhesion.

In the literature Scanning Electron Microscopy has been used to record the effect of alkali treatment on the fiber surface and to show fiber fibrillation and cleaning of
Figure 2.10 Effect of soaking time on ultimate tensile strength (UTS) of coir fibers soaked in 5% aqueous solution of NaOH [6].
the fiber surface. Figure 2.11 shows effect of a NaOH solution on flax fibers. The alkalization treatment affected surface topography. Alkali treatment also affect the thermal stability of the natural fibers. It is reported in more than one work that soda treatment increased the thermal stability of various types of natural fibers [7, 32]. The concentration of caustic soda for alkalization should be carefully selected. Fibers treated with high NaOH concentrations have reduced thermal resistance and tensile strength. As an example, the effect of soda concentration on the thermal resistance of sisal fibers is shown in Figure 2.12 [32].

The influence of alkali treatments on cellulose fiber composites has been widely researched. As previously mentioned, an improvement in fiber matrix adhesion after fiber treatment enhances the composites properties. Also, the different concentrations of alkali solutions result in different effects on mechanical properties of composites. Rout et al [25] report that alkali treated coir fiber used in polyester composites displayed different effects depending on treatment method. Composites prepared with fiber 2% alkali treated displayed better tensile strength (26.8MPa) whereas composites prepared with fiber 5% alkali displayed better flexural strength (60.4MPa) and impact strength (634.6J/m).

We can summarize the effects of alkali treatment process on natural fibers as follows [6, 7, 10, 18, 20, 33]:

- Extract hemicellulose, lower molecular weight substance and residual lignin.
- Enhance the reactivity of cellulose fibers for various chemicals by restricting swelling of crystalline regions.
- Improve the arrangements and orientation of cellulose molecules.
- Decrease the spiral angle of microfibrilles within the fiber axis.
- Increase fiber tensile strength and elastic modulus.
- Increase the crystalline regions of the fiber by removal of the cementing materials.
Figure 2.11 ESEM micrographs of (a) untreated flax fibers; and (b) 5% alkali-treated flax fibers [34].
Figure 2.12 The TGA curve of sisal samples [32].
- Increase the fiber surface roughness and the formation of fibrillar on the fiber topography.
- The concentration of the NaOH solution, treatment times and temperature of the treatment play a vital role in achieving the best performance of the fiber.

b. Acetylation of natural fibers

Absorption of moisture by natural fibers is one of their most serious disadvantages. This high moisture absorption can be attributed to the presence of hydroxyl groups in cellulose in the cell-wall polymers (see Section 2.3.4-b). Solutions for this problem can be found in the acetyl group of organic combinations, which contains OH-, SN-, or NH2 groups. This normally happens by combining acetic acid and acetyl chloride, and then heating in the presence of a solvent such as benzol [6].

The affect of chemical modification using acetic anhydride at 120°C on the tensile properties and biodeterioration of coir, oil palm, flax and jute fibers has been investigated [23]. The authors concluded that improvements in mechanical properties and, in particular, resistance to microbial degradation can be obtained [23].

A recent investigation by Abdul Khalil et al [35] concludes that the effect on mechanical properties after soil burial tests depends on the fiber treatment process used. Unmodified fibers display a greater decrease in properties over time. Acetylated fibers and silane treatment did not show any decrease in properties during the tests. Different types of acetylation treatment processes carried out by Rana et al [36] who reported that acetylation decreased moisture absorption due to the formation of cover gaps between the fibrils.
2.5 Matrix Used with Natural Fibers Composites

Natural fibers have been used as a reinforcement with thermosets and thermoplastics. They have been also used with biodegradable polymers. Composites produce by natural fibers and biodegradable polymers are environmentally friendly and also called green composites [5, 25, 21, 24]. These composites are made by combining natural fibers with biodegradable plastics. Thermosets resin such as epoxy, polyester and vinyl ester are used with natural fibers to produce low strength materials. They have good dimensional stability, are easily manufactured and limit moisture absorption. The major drawback of thermosets is the fact that they cannot be recycled or reshaped after being cured. Thermoplastics are limited by their melting point as was previously mentioned. Thermoplastics are more attractive due to their recyclability, and manufacturing processes which allow for injection molding or compression, which in turn, allows for high volume production. The following sections illustrate a recent developments in the use of natural fibers with thermosets, thermoplastics and biodegradable polymers.

2.5.1 Natural fiber reinforced thermosets

A lot of research has recently investigated the reinforcement of thermosets, such as epoxies, polyesters and vinyl esters, with various types of plant fibers. Epoxy/cotton for electrical insulation is the oldest application of natural fiber reinforced composites. Using a matrix made of thermoset resins eliminates one of the main problems encountered with natural fibers, their high moisture absorption [6].

There is a lot of research concerning the use of polyester as a matrix with natural fibers in order to improve its mechanical properties. Additionally, the low cost of polyester when combined with cheap fibers leads to low cost composites. There are some
other beneficial parameters such as low viscosity, short curing time, low processing temperature and good thermal resistance [3]. All this enables to a simple manufacturing process such as resin transfer molding (RTM).

Albuquerque et al [37] investigated the physical and mechanical properties of uniaxially oriented jute reinforced polyester composites. They found that fibers treated with an alkali solution at 100°C is better than fibers treated with the same alkali solution but at room temperature in terms of the composites mechanical properties. Also, composite properties and water absorption increase with an increase in fiber content, where impact strength of composites with 30% fiber weight fraction found to be about 350% greater than that of pure polyester resin.

Devi et al [38] found a good improvement in the strength of polyester reinforced by pineapple leaf. The addition of pineapple leaf fibers changed the behavior of polyester from being brittle to more ductile. Table 2.10 shows the properties of polyester reinforced by three different types of natural fibers: straw, sisal, and pineapple.

Rout et al [25] used coir fiber as a reinforcement for polyester resin after exposing the fibers to various surface modifications, such as alkali treatment, bleaching and vinyl grafting. This study discovered a poor adhesion between coir fibers and the matrix, and that surface modification of the coir fibers improved the adhesion. Additionally, composite properties differed depending on the treatment process.

Joseph et al [39] reported the use of banana fibers with phenol formaldehyde and compared them to the use of glass fibers. This study showed that interfacial shear strength values for banana fiber were higher than those of glass fiber. This result was achieved through applying a single fiber pull-out test. Also, using banana fibers with phenol formaldehyde change their brittle nature to be more ductile behavior. In addition, the tensile, flexural and impact properties of the composite were dependent on fiber length and
Table 2.10 Properties of different natural fiber/polyester composites prepared by hand lay-up methods (Fiber Content 30 wt.%) [38].

<table>
<thead>
<tr>
<th>Property/Composite</th>
<th>Straw/reinforced Polyester</th>
<th>Sisal/Polyester</th>
<th>PALF/Polyester</th>
</tr>
</thead>
<tbody>
<tr>
<td>Tensile strength (MPa)</td>
<td>-</td>
<td>28</td>
<td>52.7</td>
</tr>
<tr>
<td>Flexural strength (MPa)</td>
<td>47</td>
<td>53</td>
<td>80.2</td>
</tr>
<tr>
<td>Impact strength (unnotched),(KJ/m^2)</td>
<td>2.6</td>
<td>11</td>
<td>24.2</td>
</tr>
</tbody>
</table>
increased with fiber content. Finally, the specific properties of banana composites were compared with synthetic fibers like glass.

Mishra et al [30] studied the mechanical properties of hybrid composites resulting from a combination of bio-fiber (pineapple leaf fiber/sisal fiber) and glass fibers in polyester resin. The addition of relatively small amounts of glass fibers to natural fiber composites enhanced the mechanical properties of the resulting composites.

In general for a given thermoset matrix, surface modification and fiber content have a major effect on composite properties. This can be attributed to improvement in adhesion and wetting between fibers and matrix. Water absorption reduce when natural fibers used with thermoset matrix and influence by fiber surface modification methods. Using a coupling agent such as A N-grafted or silane improved the mechanical properties and decreased moisture absorption by fibers due to the improved bonding and compatibility of fibers and matrix.

2.5.2 Natural fiber reinforced thermoplastics

As previously mentioned, thermoplastics are more environmentally friendly due to their ability to be recycled. However, there are some problems encountered when using natural fibers with thermoplastics. Thermoplastics are hydrophobic and this leads to a difficult dispersion of fibers in a non-polar matrix where natural fiber is a hydrophilic nature. Thus, natural fibers are not compatible and don’t wet with many thermoplastic matrices due to the difference in polarity. This leads to voids or porosity, and thus, weak fiber-matrix interface and poor overall mechanical properties [21].

Some solutions to these problems have been found. The most extensively used methods involve the use of coupling or compatibilization agents, and surface treatments of the fibers which introduce a bridge of chemical bonds between the two components.
Furthermore, the lower thermal stability of natural fibers, which cannot exceed 230°C (Section 2.3.4-c), reduces the types of thermoplastic that can be used with natural fibers. These are usually polyolefins, like polyethylene and polypropylene. Other thermoplastics like polycarbonates cannot be used since their processing temperature is more than 230°C [6, 7].

Many researchers have investigated the use of polypropylene and polyethylene with different types of natural fibers. Mohanty et al [7] studied using bast fiber (Kenaf) and leaf fiber (henequen) as a reinforcement for polypropylene through a powder impregnation processing technique. They conclude that powder impregnation processing is a novel and environmentally benign technique in which fiber damage can be eliminated. Also, bast fiber based biocomposites show the best flexural properties, whereas leaf fibers have the best impact properties.

Zafeiropoulos et al [40] reported on using flax fibers as a reinforcement with polypropylene following different types of surface treatment to improve the interface adhesion compatibility between matrix and fibers.

Singleton et al [41] study use of recycled high-density polyethylene from recycled bottles with flax fibers. According to these results, similar mechanical properties were obtained using either flax fiber/PP or flax fiber/recycled HDPE. Consequently, using recycled HDPE as a matrix has no negative effects upon tensile strength, stiffness, and toughness. This work allows the production of very cheap composites with a substantial reduction of waste by means of recycling.

Mankandan et al [42] investigated the tensile properties of short sisal fiber as a reinforcement for polystyrene matrix. Raw and benzyolated sisal fiber were used in this study. Fibers from the benzyolation process showed better adhesion than raw sisal fibers and gave higher tensile properties.
Common methods for manufacturing natural fiber reinforced thermoplastic composites such as injection molding and extrusion, tend to degrade the fibers during processing. Krishnan [43] developed a simple manufacturing technique for sisal fiber-reinforced polypropylene composites to minimize fiber degradation. In this work, sisal fiber mats were formed by sprinkling the fiber down a drop feed tower, which avoids much of the processing degradation found with another methods. Then, sheets of composites were manufactured by using vacuum forming or hot-pressing.

Bhattacharyya et al [44] reported about thermoforming sheets of wood fiber polypropylene composites. This process is used to produce pre-consolidated wood fiber thermoplastic composites sheets in 3-dimensional shapes for product manufacturing. Different types of thermoforming processes such as die-match forming, air pressure forming and deep drawing were applied to an unmodified wood fiber-polypropylene composite.

Wood flour, a low cost reinforcing filler, was used with high density polyethylene (HDPE). Different fiber treatment processes were applied in order to improve the bonding between the fiber and polymers. Alkoxy-silane, was used as a coupling agent for wood flour and a significant improvement in mechanical properties was achieved [45].

Bledzki et al studied the effect of wood fiber geometry and coupling agent when wood was used as a reinforcement for polypropylene composites. In this study, hard wood fiber, soft wood fiber, long wood fiber and wood chips were used. It was found that composites display different mechanical properties depending upon fiber geometry. Specifically, wood chip/PP showed better tensile and flexural properties in comparison to other wood fiber/PP composites. Also, the addition of 5% maleic anhydride-polypropylene copolymer (MAH-PP) maximized the composites' properties [46].
2.5.3 Natural fibers reinforced biodegradable polymers

New advances in genetic engineering, composite science and natural fibers development offer significant opportunities for new, improved materials from renewable resources that can biodegrade or be recycled, thus enhancing global sustainability. The concept of sustainable bio-based product is explained in Figure 2.13.

A bio-based product derived from renewable resources has recycling capability and triggered biodegradability, which means that it is stable during its lifetime of use but biodegrades after disposal in composting conditions. Combined commercial viability and environmental acceptability define a "sustainable" bio-based product [7]. The fatty acids found in plant oils have been found to be linear, branched or cross-linked polymers. These materials are called the biodegradable polymers, and they are a good solution for the environmental concerns. High oil prices and concerns over the dwindling availability of landfill sites have revived interest in biodegradable materials today.

A biodegradable polymer offers a possible solution to waste-disposal problems found with traditional petroleum-derived plastics. Biodegradable polymers degrade through the action of enzymes and/or chemical decomposition associated with living organisms (bacteria, fungi) and their secretion products. Examples of biopolymers used as a matrix are biopol, polyhydroxybutyrate (PHB), poly-β-hydroxybutyrate-co-α-lactate (PHBV), cellulose acetate (CA), cellulose acetate butyrate (CAB), bioceta, mater bi and sconacell. Some mechanical properties of flax fiber reinforced biodegradable polymers are presented in Table 2.11. A detailed overview of biopolymers is presented by Mohentay et al in two different articles [11, 33].

Also, the mechanical properties, such as tensile strength and Young's modulus, of biodegradable composites are similar to those of other composites and are highly
Figure 2.13 Concept of “sustainable” bio-based product [17].
Table 2-11 Mechanical properties of flax-fiber reinforced biodegradable polymers (BDP) with fibers 50 vol%, linen woven [6].

<table>
<thead>
<tr>
<th>Fibers-BDP</th>
<th>Tensile strength (MPa)</th>
<th>Young's modulus (MPa)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Flax-Bioceta</td>
<td>65.7</td>
<td>1400</td>
</tr>
<tr>
<td>Flax-Sconacell A</td>
<td>106.9</td>
<td>8180</td>
</tr>
<tr>
<td>Flax-Mater Bi</td>
<td>124.3</td>
<td>10580</td>
</tr>
</tbody>
</table>
influenced by the particular matrix and the adhesion between fiber and matrix [6, 17].

Mohanty et al [47] studied the effect of process engineering on the performance of composites fabricated from chopped hemp fiber and cellulose ester biodegradable plastic through two different processes. They used powder impregnation through compression molding as the first process, and extrusion followed by injection molding as the second process. The two processes were successful in producing biocomposites and the second process gave better strength compared with the first process. They attributed this result to improved shear forces between the fiber and the matrix resulting from good mixing.

Dweib et al [48] studied natural composite materials derived from soy oil based resin and flax fibers, cellulose and recycled paper, which gave a good mechanical behavior. Flax fibers increased the flexural modulus of the composite 5 times compared with neat resin. Reinforced biopol matrix with jute fibers using different types of surface modification, resulted in an improvement in mechanical properties of biopol composites compared with neat biopol [49].

Using flax fibers to reinforce polylactic acid (PLA) resulted in a composite with better strength than polypropylene/flax fiber composites, which are used today in many automotive panels [50]. When using biodegradable polymers, some problems were encountered, such as cost, moisture absorption and degradation during storage [17]. The high cost of biopolymers compared with traditional plastics is not due to the cost of the raw materials for biopolymer synthesis, but rather to the low volume of production. Also, the use of biodegradable polymers is related to a stable product properties during storage and usage but then degrades after disposal. The design of such materials needs a clear understanding of the factors influencing their properties and performance, as well as factors affecting biodegradability, so that an appropriate trade-off can be made.
Chapter 3

MATERIALS AND EXPERIMENTAL TECHNIQUES
3. Materials and Experimental Techniques

3.1 Materials Used

Raw samples of the mesh (natural mat) surrounding the date palm trees stem were supplied kindly by Al-Ain Date Factory, Al-Ain, UAE. These samples were removed from date palm stems and preserved in polyethylene bags. General-purpose polyester resin (8120TEC) was obtained from Qualipoly Chemical Corporation, Taiwan through a local supplier and was used as the matrix. Table 3.1 shows the resin’s specifications. Polyester resin density was determined by a picnometer. The resin’s density was found to be 1.125g/cm³. Sodium hydroxide and dioxin were supplied by PDH Middle East L.L.C. Dubai, UAE and were used without any purification.

3.2 Fiber Preparation Methods

The natural mat consists of single fibers crossing each other. The natural fibers were contaminated with a large amount of sand and dust, due to their exposure to the natural environment. The samples were washed with tap water and manually dismantled into bundles of virgin fibers. The bundled fibers were dried at room temperature before being cut to the desired length using a sharp blade. Fiber diameter was checked and fibers with diameters of up to about 0.5 mm were selected to be used in this study. These fibers were then dried in a vacuum oven at 60°C for about 24 hr. These fibers are termed raw fibers and are referred to by the symbol (A). In this study fibers were used raw (A), as described above, or after being subjected to different treatment methods.
Table 3.1 Polyester resin (8120TEC) specification according to manufacturer supplied data sheet.

<table>
<thead>
<tr>
<th>Property</th>
<th>Value</th>
</tr>
</thead>
<tbody>
<tr>
<td>Percent Styrene, %</td>
<td>37-42</td>
</tr>
<tr>
<td>Viscosity, Brookfield No. 3/60 rpm, 25 °C</td>
<td>400-500</td>
</tr>
<tr>
<td>Acid Value, mgK OH/g</td>
<td>15-25</td>
</tr>
<tr>
<td>Thixotropix Index</td>
<td>1.4-2.2</td>
</tr>
<tr>
<td>Gel Time, min, 25 °C, 1% MEKPO(50%)</td>
<td>25-30</td>
</tr>
</tbody>
</table>
Three different treatment methods were used. In the first treatment method, fibers were soaked in a commercial detergent at 100°C for 2 hr. Fibers obtained from this process are called detergent fibers and referred to by the symbol (B). The second treatment method consisted of soaking fibers in 5% NaOH solution at 100°C for 1, 2 and 3 hr and these fibers are labeled (C). The last treatment method involved bleaching the fibers obtained from the second method with a dioxin solution for 24 hr. These fibers are referred to dioxin bleaching fibers and are referred to by symbol (D). Table 3.2 summarizes the treatment methods used. In all treatment methods, fibers were washed with tap water and dried in a vacuum oven at 60°C for 24 hr. Finally, fibers were preserved in airtight polyethylene bags to reduce moisture absorption until they were used.

3.3 Composites Fabrication

To fabricate composite specimens, an aluminium mould was used. The cavity of the mould replicated the specimen's dimensions. Each mould provided only a single specimen. A mould-releasing agent was applied to the surface. A general-purpose polyester resin was mixed with 1 wt.% methyl ethyl ketone peroxide (MEKPO) as the catalyst. The resin mixture was degassed in a vacuum desiccator to remove entrapped air and then poured on the randomly placed fibers in the mold. When the fiber was completely wet by the resin, the mould was closed using a Teflon sheet and pressed. Molds were left to cure at room temperature for 24 hr. Finally, the composite was placed in an oven at 100°C for 2 hr in order to post cure the resin. This method of fabrication was used for all specimens manufactured either with glass or date palm fibers.
Table 3.2 Symbols and fiber treatment methods.

<table>
<thead>
<tr>
<th>Treatment method</th>
<th>Symbols</th>
</tr>
</thead>
<tbody>
<tr>
<td>Raw fibers</td>
<td>A</td>
</tr>
<tr>
<td>Detergent fibers</td>
<td>B</td>
</tr>
<tr>
<td>NaOH (5%) + Water</td>
<td>C</td>
</tr>
<tr>
<td>NaOH (5%) + bleaching with dioxin</td>
<td>D</td>
</tr>
</tbody>
</table>
3.4 Chemical and Physical Analysis

This section will describe the experimental techniques used to find the chemical composition of DPF, fiber density, thermal analysis, morphology analysis and water absorption of composites.

3.4.1 Chemical analysis of date palm fiber

The Van Soest method was used to determine the chemical composition of natural fibers [22]. This method depends on the use of two different solutions, the neutral detergent fibers (NDF) and acid detergent fibers (ADF). First the neutral detergent solution is used to dissolve the easily digested pectins and plant cell contents (proteins, sugars and lipids), leaving a fibrous residue that is primarily cell wall components of plants (cellulose, hemicellulose and lignin). The treated fibrous residue when used with the acid detergent fiber solution will digest hemicellulose. Therefore, the difference between ADF and NDF is hemicelluloses. The residue of the ADF was treated with 72% H₂SO₄ at 25°C for 4 hr in order to digest the lignin. The residual substance will be cellulose and indigestible minerals. The residual substance was brought to ignition at 500°C and the residue was indigestible minerals.

3.4.2 Density measurement

Fiber density was determined using a classical volumetric method (mass/volume) [18, 47]. An average of 6 measurements was used to determine fiber density. Ethanol (density 0.789 g/cm³, and vapor pressure 7.87 KPa @ 25°C) was used as a solvent. As ethanol evaporates at room temperature, the solvent was cooled to 0°C during density measurements in order to reduce evaporation.
3.4.3 Thermal analysis

The thermal stability of DPF was characterized using a thermogravimetric analyzer (TGA). The instrument used was the TA instrument GA 2950. The TGA analyzer has a balance sensitivity of 0.1 μgm and an accuracy better than 0.1% with a temperature range from ambient temperature to 1000°C. About 10 mg of DPF was exposed to air atmosphere with a heating rate of 10°C min⁻¹ from room temperature to 400°C. This method was followed to evaluate the thermal stability of raw and treated date palm fibers.

3.4.4 Morphology analysis

Microscopic examination was carried out using a Jeol Model JSM-5600A scanning electron microscope (SEM). Several samples were examined to ascertain the observed phenomena. All specimens were sputtered with approximately 10 nm layer of gold prior to SEM observations. The SEM was used to study fiber morphology, fiber cross-section structure and the fracture surface of the composites samples. Samples were mounted on aluminium holders using double-sided electrically conducting carbon adhesive tabs prior to the analysis.

3.4.5 Water absorption

The water absorption behavior of untreated and surface modified DPF/polyester composites as well as that of glass/polyester composites was recorded according to the ASTM 570 standard. Three specimens with dimensions of 76.2×23.4×3.2 mm and an analytical balance capable of reading 0.001g were used in this experiment.

Two standard procedures were used in order to detect the effect of fiber weight fraction and effect of surface modification on the water absorption of composites. In the first procedure, composite samples were dried by heating in an oven at 70°C for 24 hr,
weighed \((W_1)\) and then soaked in deionised water at room temperature for 24 hr, before removal and drying with cotton and weighing. This is referred to as weight of the wet sample \((W_2)\). Finally, samples were put in the oven at 70°C for 24 hr and the weight \((W_3)\) was recorded. Water absorption was calculated:

\[
\% \text{ Water absorption} = \frac{W_2 - W_3}{W_3} \times 100
\]  

(3.1)

The second procedure involved placing dried specimens in boiling distilled water for \(120 \pm 4\) min. Then specimens were removed from the water and cooled at room temperature in distilled water. Finally, all surface water was removed with a dry cloth, and the specimens were weighed.

### 3.5 Mechanical Properties

This section will include descriptions of experimental techniques used to find the mechanical properties of single DPF and composites. The tensile test was applied to find the elastic properties of single fibers. Whereas, flexural (bending) properties and Izod impact test are carried out on composites.

#### 3.5.1 Single fiber tensile test

Single fibers were carefully separated manually from the fiber bundles. The mechanical properties (tensile strength, young’s modulus and an elongation to break) of a single DPF were determined according to ASTM D-3379-75. Specimens were prepared by gluing both fiber ends (about 15 mm) on a stiff galvanic cardboard 50 x 15 x 1 mm with TML strain gauge adhesive obtained from Tokyo Sokki Kenkyujo CO., Ltd, Japan. A gauge length of 10 mm and a crosshead speed of 1 mm/min were employed. Fiber diameter averages were measured using a digital caliper by taking three different readings.
of the diameter at three different points along the gauge length. Tensile testing of DPF was carried out using a universal tensile testing machine MTS (20/MH) model equipped with a 5 KN standard load cell. The system is equipped with universal testing software (TestWorks). The temperature and relative humidity for all experiments were about (23 ± 2 °C, 65 ± 5%), respectively.

3.5.2 Flexural (bending) test

The three point flexural properties were determined using a universal tensile testing machine MTS (20/MH) model according to ASTM D-790-03. The load displacement curves were obtained and the flexural strength and modulus of composites fabricated from treated and untreated DPF were calculated. The cross-head speed used was 1.6 mm/min and the dimensions of the specimen were 80 x 16 x 4 mm. A span-to-depth (thickness) ratio of not more than 16 was applied. The support span for the flexural tests was 7 mm and the diameter of the loading tip was 10 mm. The temperature and relative humidity for all experiments were about 23 ± 2 °C, 65 ± 5%, respectively. The load deflection curve was recorded until failure start in the specimens. The flexural strength (σf) was calculated using:

\[
\sigma_f = \frac{3PL}{2bd^2}
\]  

(3.2)

where \(P\) is the force (N), \(L\) is the distance between the support span (mm), \(b\) is the specimen width (mm), and \(d\) is the specimen thickness (mm). The flexural modulus (\(E_f\)) was calculated using:

\[
E_f = \frac{L^3m}{4bd^3}
\]  

(3.3)

where \(m\) is the slope of the initial straight-line portion of the load-deflection curve.
3.5.3 Izod impact test

Izod impact tests on un-notched specimens were performed using an impactometer (Avery-Denison) according to ASTM-D256-02. The machine operated with a 5 J pendulum and the striking velocity was 3.46 meters/second. Specimens of neat resin, DPF composites and glass fiber composites were used. All the results are taken as the average of 6 samples for each test. The impact strength was calculated using a test method explained in [51]:

\[
\text{Impact strength} = \left( \frac{A}{xy} \right) \times 10^3
\]

(3.4)

where A is the impact energy (J), x is the specimen width (mm), and y is the specimen thickness (mm). In this experiment width and thickness were 16 mm and 4 mm, respectively.
Chapter 4

RESULTS AND DISCUSSION
4. Results and Discussion

This chapter is divided into three main parts. In the first part, a general characterizations of raw DPF is presented. The physical, chemical and mechanical properties of raw DPF are presented and compared to common natural fibers. The second part is dedicated to the effect of different treatment processes applied on the fiber. The effects of the treatment methods on the chemical, physical and mechanical properties are discussed. Finally, various physical and mechanical properties, such as flexural strength and modulus, impact strength and moisture absorption of composite specimens manufactured using treated and untreated DPF reinforced with polyester resin, are analysed and discussed.

4.1 Characterization of Raw DPF

4.1.1 Structure and surface morphology

The date palm fiber is cylindrical in shape, as shown in (Figure 4.1-a). The SEM micrograph of a raw fiber shows the surface containing a large numbers of uncompleted grown fibers (expected to be residual lignin) and artificial impurities (sand and dust). A cross section of single DPF (Figure 4.1-b) reveals large numbers of hollow single fibers collected and bonded by primary layers.

The fiber is a collection of multicellular fibers each containing a central void (lumen). The multicellular fibers (individual fibers) are about 2 to 5 μm in diameter. The structure and shape of DPF are similar to those of coir fibers [52]. Both DPF and coir fiber are cylindrical in shape and each fiber consists of multicellular fibers. According to
Figure 4.1 SEM micrographs of raw DPF showing its cylindrical shape (a) and the multicellular fiber (b) in a cross-sectional view of the fiber.
morphological studies performed on coir fiber, the outer layer is expected to be lignin and removal of this surface layer is expected to result in a better and more stable bond between fibers and the matrix [25, 52].

Accordingly, surface modification seems to be essential for purifying and cleaning the surface of the fibers from a large amount of impurities, which may be responsible for poor adhesion between the fiber and the matrix.

4.1.2 Density

Results of density measurements show that the density of raw DPF is about \( (0.917 \pm 0.127 \text{ g/cm}^3) \). This value is in the range of known values for natural fiber density, \( (0.9-1.5 \text{ g/cm}^3) \) [5]. DPFs have a density lower by about 60% than that of glass fibers \( (2.5 \text{ g/cm}^3) \). Compared to other known natural fibers (Table 2.9), coir fiber is the closest in term of density \( (1.15 \text{ g/cm}^3) \). The lower density of raw DPF when compared to other natural fibers can be attributed to large fiber diameter, which results in an increase in the diameter of the central void (lumen). Attaining a reliable value for density is difficult due to the porous nature of the fibers. Baley [18] reports similar problem with flax fibers.

4.1.3 Chemical composition

The chemical compositions of the main natural polymers contributing to the DPF are shown in Table 4.1. Many authors have reported that the cellulose content, in addition to other parameters such as the industrial process, the degree of crystallinity, the spiral angle of fibrils, the degree of polymerization, the porosity content, and the size of the center void (lumen), has an important influence on the mechanical properties of natural fibers [6, 18]. Actually, natural fiber can be considered as a fibrous composite material [11]. By comparing the chemical composition of DPF in Table 4.1 with that of the common types of natural fibers in Table 2.6, it is clear that the high amount of cellulose
Table 4.1 Chemical composition of raw DPF.

<table>
<thead>
<tr>
<th>Element</th>
<th>Cellulose Wt.%</th>
<th>Lignin Wt.%</th>
<th>Hemicelluloses Wt.%</th>
<th>Moisture content Wt.%</th>
<th>Others Wt.%</th>
</tr>
</thead>
<tbody>
<tr>
<td>Composition</td>
<td>46</td>
<td>20</td>
<td>18</td>
<td>5</td>
<td>11%</td>
</tr>
</tbody>
</table>
(46%) in DPF is a good indication of the strength of the fiber. Coir fibers show the lowest
tensile strength among all the natural fibers characterized, and this is attributed to their low
cellulose content and high microfibrils angle [53]. On the other hand, flax fibers, which
have the highest tensile strength, have at the same time the highest cellulose content of
natural fibers. Mohenty et al [33] report that due to the very complex structure of natural
fibers and the unclear nature of their chemical composition, it is not easy to exactly
correlate fiber strength with cellulose content. DPF has 18 wt.% hemicelluloses and 20
wt.% lignin. The amount of hemicelluloses is similar to that in common natural fibers.
However, the percentage of lignin is generally higher than that of common fibers and
lower than that of coir fibers (45%). Mohenty et al [11, 33] report that the lignin content of
the fibers influences their structure, properties and morphology. Finally, we should keep in
mind that the chemical analysis of DPF could be affected by the plant’s origin and
agricultural parameters, such as soil quality, weathering conditions and the process of
retting [18].

4.1.4 Thermal stability

Natural and synthetic polymers are subject to degradation of their mechanical
properties under the influence of increased temperatures. Thermal stability is considered
one of the limiting factors in the use of natural fibers. It is well known that the different
polymer processing techniques are carried out at relatively high temperatures. These fibers
when used as reinforcement, will be subjected to a thermal gradient (peak exotherm of the
thermoset resin or the melting temperature of the thermoplastic resin). For all these
reasons, it is important to understand the thermal degradation of these fibers. Figure 4.2
shows the results of a thermal analysis of raw DPF between 25-390°C using a
thermogravimetric instrument.
Figure 4.2 Thermogravimetric analysis of raw DPF, about 10mg sample heated at 10 °Cmin⁻¹ in air atmosphere.
Figure 4.2 shows a slight decrease in weight as the temperature increases to 100°C, and this may be attributed to the fiber moisture content. The weight loss shows a steady state behavior up to a temperature of around 250°C, at which thermal degradation starts. These results are comparable to those obtained with other types of natural fibers such as henequen, kenaf and sisal [7, 42]. By analyzing the thermal performance of these fibers, it is obvious that processing and treatment temperatures should not exceed the degradation temperature of 250°C. It is well known that the exothermic reactions of polyester and vinyl ester are well below this temperature and can therefore be used as a matrix with DPF. Also, Polyethylene (PE), and Polypropylene (PP) have lower melting points than the degradation temperature of DPF and can also be used as a matrix when DPF is used as a reinforcement.

4.1.5 Elastic properties

Tensile tests on single DPF samples which have a nominal length of $L_o=10$ mm were carried out at a speed $(V)$ of 1 mm/min. The average mechanical properties of 30 single raw DPFs are presented in Table 4.2. Typical values for the raw DPF tensile strength were between 170 and 275 MPa, whereas values for the Young’s modulus were between 5 and 12 GPa. The elongation to break was between 5 and 10%. No previous published data that can be used for benchmarking were available.

When comparing the average diameter and mechanical properties of a single DPF with other common natural fibers (Table 2.8), the following can be observed. The tensile strength of DPF is comparable to that of coir fibers. The DPF tensile modulus was lower than that of all other natural fibers except for that of coir fiber, which was the lowest. It is worth mentioning that these values of tensile strength and Young's modulus are sensitive to many parameters, including the species and the variety of the plant, agricultural variables such as soil quality, weathering conditions, the level of plant maturity and the
Table 4.2 Average mechanical properties of single DPF.

<table>
<thead>
<tr>
<th>Diameter (μm)</th>
<th>Tensile strength (MPa)</th>
<th>Young’s modulus (GPa)</th>
<th>Elongation to break (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>100-1000</td>
<td>170-275</td>
<td>5-12</td>
<td>5-10</td>
</tr>
</tbody>
</table>
quality of the retting process, to cite but a few. A good example of this scattering in the properties is given by reported values for PALF fibers, when comparing the Young’s modulus given in Table 2.9 with results in other works [38], which report lower values. DPFs have a relatively large diameter which varies along its length. Typical values for the diameter of DPF are from 100-1000μm, which are higher than those for other fibers.

Figure 4.3 shows a typical stress-strain curve. DPF is similar to other types of natural fibers in that it behaves like a viscoelastic material when subjected to tension. The stress-strain curve for date palm fiber is characterized by an initial linear region followed by a curvature indicating the increased rate of strain produced with increasing stress. The variations in properties are observed in the DPF as another types of natural fibers, stress-strain curves are a good indication of the difficult in predicting the internal structure of date palm fibers. According to the tensile tests performed on a single DPF, it appears that greater fiber diameters will result in lower values for the Young’s modulus. This can be attributed to an increase in the area of the lumen (micro center void) in the fibers (Figure 4.4). A similar phenomenon was reported by Mohanty and others for different types of natural fibers, such as flax and abaca [11, 18, 29, 54].

The outer surface of fractured specimens was analyzed after tensile testing (Figure 4.5). SEM micrographs show the outer layer covering multicellular fibers. Each one of these multicellular fibers contains microvoids (center lumen). As anticipated, the outer layer is composed essentially of lignin, which is reported to act as a protective shield for the fiber against physical and chemical attack [11]. Modifying this shield will probably affect the mechanical properties of composites produced from DPF. In higher magnification of SEM micrographs, we can see small broken filaments pulled out from hollow fibers.
Figure 4.3 A typical stress-strain curve of a single date palm fibers.
Figure 4.4 Young's modulus of DPF as a function of fiber diameter.
Figure 4.5 SEM micrographs show the break area of tip DPF after a tensile test. A number of multicellular or hollow fibers appear in the direction of the axis of the fiber (a) and hollow fibers having a center void (lumen) on it is center and small filament pull out (b).
4.2 Effect of the Treatment Process on DPF Properties

The effect of the three different treatment methods on fiber surface morphology, density, chemical composition, thermal stability and ultimate tensile strength were investigated. Each method of fiber surface modification shows different effects on fiber properties. This section presents the results of different treatment processes on fiber surface morphology, density, chemical composition, thermal stability and ultimate tensile strength.

4.2.1 Effect on DPF surface morphology

While the surface of raw fibers shows a large amount of artificial and natural impurities (Figure 4.1-a), detergent tends to decrease these impurities by cleaning the fiber surface. Figure 4.6 shows the DPF surface after being treated with a commercial detergent. This method of treatment results in an improvement of the fiber surface by removing artificial impurities such as dust and sand. However, uncompleted grown fibers are still contaminating the surface. This can be attributed to a weak sensitivity of the detergent solution during fiber treatment.

Mercerization is well known as a simple treatment process used to improve the luster of textile fibers such as cotton. In addition, it is used to modify a natural fiber surface prior to use as a reinforcement. Many researchers have study the effect of alkali treatment on the microstructure, surface topography and tensile strength of different types of natural fibers (See Section 2.4.2-a) [7, 13, 24, 25, 53]. In this study, raw DPF were treated using a soda solution (5% NaOH) at 100°C followed by washing with tap water and drying in an oven at 60°C. The improvement in fiber surface morphology after applying the soda treatment can be seen in Figure 4.7.
Figure 4.6 SEM micrograph of a DPF treated with a commercial detergent. Notice the reduction in small particles (artificial) on the fiber’s surface (a). However, the fiber surface contains small uncompleted grown fibers (b).
Figure 4.7 SEM micrograph of DPF treated using 5% NaOH solution at 100°C. Fiber surface is clean from artificial and natural impurities (a). Improvement in the fiber surface by the appearance of fiber fibrillation on the surface (b).
The soda treatment process has a clear effect on the DPF surface. SEM micrographs show that using the soda treatment cleans the fiber surface from a large amount of impurities and causes fibrillation. This result was expected and similar results with other types of natural fibers were reported (see Section 2.4.2-a).

The effect of exposing alkali treated fibers to dioxin (as a bleaching solution) at room temperature for 24 hr is shown in Figure 4.8. As a result of using dioxin as a bleaching solution, a large number of regularly distributed holes appear on the fiber surface. It is anticipated that these holes were caused by the reaction between the dioxin and the outer layer of fibers, which is expected to be made up of lignin since lignin is less sensitive to soda solution than hemicelluloses [6]. Mohanty et al [33], report that in the case of surface modification of natural fibers, lignin has two different linkages. One linkage shows sensitivity towards alkali whereas the other linkage is alkali resistant. When compared with fibers treated with the soda solution, there are no holes or pitting on the fiber surface, which is an indication that such a leach-out hardly occurs. On the other hand, Rout et al [52] used a NaOH solution (5%) at 30°C for 1 hr with coir fibers. They recorded a large number of regularly spaced holes or pits on the coir surface and they concluded that these holes most probably originate from the removal of fatty deposit (tyloses) on the surface. In our case, treatment with the same concentration of NaOH solution for a longer time and at a higher temperature did not produce such holes on the fiber surface.

Accordingly, it can be concluded that DPF has a harder surface and is more stable when exposed to soda solution than coir fibers. In other words, DPF has a greater amount of alkali resistant lignin linkage. In addition, less formation of holes after soda treatment could be attributed to the low contamination of fatty deposit on the surface of the DPF.
Figure 4.8 SEM micrograph of DPF treated with 5% NaOH and bleached with dioxin for 24 hr. Clear attack of the outer fiber layer by the dioxin solution and formation of a large number of pits and regularly placed holes on the surface (a), holes on the fiber surface are along the fiber axis and have different sizes (b).
Date trees are exposed to very hot and dry weather for a long time and date palm fibers act as a protective shield for the tree trunk. All this is expected to yield a hard surface and a lower amount of fatty and waxy deposit. In the case of the dioxin solution, appearance of large numbers of holes can be attributed to the effectiveness of the interaction between the treatment solution and the outer layer of the date palm fibers (excepted to be lignin). Usually, a large number of holes is desired in order to increase the adhesion between the fibers and the matrix by creating a strong interlock between them. However, such a large number could also tend to decrease mechanical properties of single fibers.

4.2.2 Effect on DPF densities

Knowing the density of the reinforcement is important in order to estimate the theoretical density of composites that use those fibers. Theoretical calculations for mechanical properties and the formation of voids involve using volume fraction, which depends on the density of fibers. Beldzki and Gassan [6] report that soda treatment affects the center voids and contributes to the gradual elimination of microvoids, which may result in an increase in fiber density. In our case, the central hollow region is large due to the relatively high diameter of DPF, and elimination of microvoids is expected to result in a noticeable change in fiber density.

Different treatment processes applied to DPF lead to different levels of density, and the results are recorded in Table 4.3. Chemical treatment was found to increase fiber density. The reported values can be attributed to the ability of each chemical solution to extract soluble products (expected to be low molecular weight). The amount of extracted soluble components will depend obviously in the treatment method. In addition, the treatment process at high temperature is expected to affect fiber structure by making fiber
Table 4.3 Density measurements for date palm fibers showing the effect of treatment methods.

<table>
<thead>
<tr>
<th>Fibers</th>
<th>Density (g/cm$^3$)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Raw DPF (A)</td>
<td>0.9170</td>
</tr>
<tr>
<td>Detergent treatment (B)</td>
<td>0.9624</td>
</tr>
<tr>
<td>Soda treatment (C)</td>
<td>1.3424</td>
</tr>
<tr>
<td>Dioxin bleached (D)</td>
<td>1.3989</td>
</tr>
</tbody>
</table>
structure more compact (reducing lumen), which will decrease fiber volume and increase fiber density. For detergent treated fibers, there was a slight increase in the density by about 5% compared with raw fibers. This small variation could be attributed to the inability of the detergent solution to extract all soluble products or remove natural polymers of lower molecular weight. Evidence for this conclusion can be provided by the failure of this treatment method to remove residual fibers contaminating the fiber surface (Fig. 4.6). For the soda and dioxin treatment processes, an increase in the density of about 33% was reported. Relatively large increase can be attributed to the ability of the treatment solution to remove a large amount of low molecular weight polymers and substances with amorphous nature, with a consequent increase in fiber density. Sydenstricker et al [32] report that the density of sisal fibers decreases with chemical treatment, which is the opposite of our findings with respect to DPF.

4.2.3 Effect on DPF chemical composition

Treated DPF fibers were subjected to chemical analysis using the Van Soest method [22]. Table 4.4 shows the chemical compositions of DPF obtained by different treatment processes. All treatment processes show an increase in cellulose content. Also, it is observed that the lignin content decreased with treatment. The decrease in lignin was more obvious in dioxin treatment were lignin content dropped from about 20% for raw fibers to about 9%. Hemicelluloses content increased for detergent and soda treatment, but decreased slightly for dioxin. It is worth mentioning that the fiber itself is composed of other complex polymers (polysaccharides), which are also expected to show different reactivity towards the various treatment solutions.
Table 4.4 The effect of treatment methods on DPF chemical composition.

<table>
<thead>
<tr>
<th>Treatment process</th>
<th>Cellulose Wt.%</th>
<th>Lignin Wt.%</th>
<th>Hemicellulose Wt.%</th>
</tr>
</thead>
<tbody>
<tr>
<td>Raw</td>
<td>45.78</td>
<td>20.57</td>
<td>17.84</td>
</tr>
<tr>
<td>Detergent treatment</td>
<td>54.63</td>
<td>15.30</td>
<td>23.38</td>
</tr>
<tr>
<td>Soda treatment</td>
<td>54.91</td>
<td>12.12</td>
<td>21.19</td>
</tr>
<tr>
<td>Soda treatment + Dioxin bleaching</td>
<td>56.90</td>
<td>8.97</td>
<td>15.81</td>
</tr>
</tbody>
</table>
It is reported in many studies that the complex structure and chemical composition of natural fibers prevent an easy investigation into the effect of treatment processes, or the way the mechanical properties relate to the chemical compositions of the fibers [33].

4.2.4 Effect on DPF thermal stability

As mentioned before, the low thermal stability of natural fibers is one of the disadvantages associated with their use. This problem appears more clearly when using natural fibers as a reinforcement with thermoplastic polymers than with thermosets. In a previous section, it was established that raw DPF shows stability up to 250°C, above which degradation starts. As natural fibers are composed of different natural polymers having different thermal stability (Table 2-10), it is expected that different treatment processes will affect the thermal behavior of natural fibers. Mohanty et al [11] report that hemicellulose has the lowest thermal stability, followed by cellulose, whereas lignin shows the highest thermal resistance (see Table 2.10).

The effects of the treatment processes on thermal stability of DPF are shown in Figure 4.9. In general, the onset of degradation for all treatment methods is practically similar, about 250°C. At this temperature the degradation starts, however above this temperature the progress of degradation (weight loss) is dependent on the treatment method. Figure 4.9 shows different rates of weight loss, dependent on fiber treatment. Soda treated fibers show the most resistance to thermal degradation, where detergent treated fibers have the lowest resistance. The resistance of soda treated fibers could be attributed to the decrease in the hemicelluloses content and the removal of components that degrade at lower temperatures due to the reaction with the soda solution. Similar improvement in the thermal stability with alkali treatment was observed for other natural fibers [7, 32].
Figure 4.9 Thermogravimetric curves of: (A) Raw; (B) Detergent treated; (C) Soda solution (5%); and (D) Dioxin bleaching DPF.
Manikandan et al [42] report that treated sisal fibers with a 18% NaOH solution exhibit a higher thermal stability than untreated fiber. This enhancement is attributed to the substitution of bulky phenyl groups in the fiber that restrict segmental mobility, thereby increasing the stiffness of the cellulose backbone.

Detergent treated fibers show the lowest thermal stability of them all. This result can be attributed to the higher amount of amorphous and low molecular weight polymers found in detergent fibers [11]. On the other hand, the lower thermal stability of dioxin fibers compared with raw fibers can be attributed to the elimination of lignin, which is reported to be highly thermal resistant (Table 2-9) due to the presence of bulky phenyl groups in its chemical structure (Fig 2.6) [7]. The lower thermal stability of DPF treated with dioxin can be considered as evidence for the high content of lignin in the outer layer, and the reaction between dioxin and these layers results in a large number of holes on this type of surfaces [11].

4.2.5 Effect on DPF tensile strength

Alkali treatment processes have a clear effect on the tensile strength of fibers, as reported by many investigators studying different types of fibers (see Section 2.4.2-a). Bledzki and Gassan [6] in their review report that the removal of lignin and hemicelluloses affects the tensile characteristics of the fibers. When the hemicellulose is removed, the interfibrillar region is likely to be less dense and less rigid, thereby making the fibrils more capable of rearranging themselves along the direction of tensile deformation. Eichhorn et al [10] report that the modification of the fibers can both increase or decrease their strength, thus an understanding of what occurs structurally is of paramount importance. Mohenty et al [33] report that the concentration of the NaOH solution, treatment time and
temperature play a vital role in achieving the optimum efficiency of the fiber, as far as the adhesive characteristics of the fibers are concerned.

Different soaking times (1, 2, 3 hours) were used with a 5% soda solution at 100°C to find the effect of treatment time on the type of tensile strength of DPF, and the results are shown in Figure 4.10. It is clear that, regardless of the treatment, treated fibers have higher tensile strength than raw fibers. With an increase in the treatment time to two hours, the strength increased and reached its maximum value. Three hours of treatment resulted in lower tensile strength, but this was still higher than that of raw fibers. The improvement in fiber tensile strength could be attributed to the removal of amorphous materials, an increase in the cellulose content and crystalline regions and a decrease in lumen. The reduction in the tensile strength after three hours may result from the strong action of the soda solution. The treatment of the DPF for two hours with a 5% alkali solution yields about 496 MPa which leads to about 45% increase then raw DPF in tensile strength values.

The effect of different treatment processes on DPF tensile strength was investigated and the results are shown in Figure 4.11. There is about 12% reduction in the maximum tensile strength of fibers treated using detergent solution 241 MPa, when compared with raw fibers. This reduction in the tensile strength might be a result of the inability of the detergent solution to affect the structure of fibers and eliminate amorphous regions [11, 33]. The improvement in the maximum tensile strength of DPF treated with a soda solution and dioxin as a bleaching agent were about 44% and 25% respectively. Treatment with the soda solution gives high tensile strength. This might be linked to the increase in the degree of arrangement of the cellulose and lignin structure present in the fiber [33]. The relatively lower increase in the maximum tensile strength for dioxin-bleached fibers can be attributed to the formation of a large number of pits and holes on the fiber surface. Rout et al [55]
Figure 4.10 Effect of treatment time on ultimate tensile strength (UTS) of DPFs that were treated in 5% NaOH solution at 100°C.
Figure 4.11 Effect of different treatment processes on ultimate tensile strength (UTS) of DPF; (A) raw, (B) detergent, (C) soda, (D) dioxin bleached fibers.
found that treating coir fibers with a 10% alkali solution results in an increase of more than 55% in maximum stress. However, treatment with a 5% alkali solution results in a reduction in the maximum tensile strength when compared to raw coir fibers. They attributed this reduction in strength at 5% to the formation of holes on the fiber's surface and the improvement at 10% to the thickening of the cell walls. Sydenstricker et al. [32] reported the effect of different concentrations of the soda solution on the tensile strength of sisal fibers. Results show an increase in the tensile strength of NaOH-treated sisal samples, which suggests that the alkali treatment extracts the amorphous portions (lignin and hemicellulose) of the fibers.

4.3 DPF/Polyester Composites Properties

Treated and untreated fibers were used with polyester resin to produce composite specimens. These specimens were subjected to mechanical testing (flexural test and Izod impact test) to evaluate the effect of DPF on the properties of the composites. The first part of this section will address the critical length and weight fraction of DPF in a polyester matrix. Then using these two parameters, the effect of different treatment processes will be investigated through flexural bending and Izod impact tests. Also, specimens from randomly oriented glass fibers with the same weight fraction will be used for benchmarking. Finally, the moisture absorption of DPF/polyester composites will be investigated and compared with glass fiber/polyester composite specimens.

4.3.1 Flexural properties

By the application of flexural force, the upper and lower surface of the specimen under a three point bending load is subjected to compression and tension at the same time.
Thus there are two failure modes in the materials, bending and shear failure [3]. The specimen fails when bending or shear stress reaches the corresponding critical value.

**a. Effect of fiber length on flexural properties**

The properties of the composites are strongly influenced by fiber length. The fiber ends play an important role in the fracture of short fiber composites. In order to achieve the maximum level of stress in the fiber, the fiber length \( l_f \) must be at least equal to the critical fiber length \( l_c \), the minimum length of fiber required for the stress to reach the fracture stress of the fiber. Thus it is very important to optimize the fiber length for a particular matrix/fiber system so that the maximum properties can be achieved. Figure 4.12 shows the effect of fiber length on flexural strength and modulus of DPF/polyester composites. The flexural strength of the composites containing 2 cm-long fibers was 26% higher than that of composites containing fibers 0.5 cm long about 57 MPa. Also, composites containing 1 cm and 3 cm long fibers show lower flexural strength than 2 cm long fibers. The flexural modulus of the composites containing 2 cm-long fibers was about 17% higher than that of composites containing fibers 0.5 cm long. The decrease recorded for composites having a fiber length of 3 cm can be attributed to the fiber entanglements formed at higher lengths [3]. The best flexural properties of the composites were obtained with a fiber length of 2 cm.

The optimum fiber length (critical length) is a system parameter. It depends on the type of fiber and matrix used. Deve et al [38] report that the optimum length of the fiber required to obtain PALF/polyester composites of maximum flexural properties was found to be 3 cm. Seena et al [39] report that the best flexural properties of the banana/phenol formaldehyde composite are dependent on fiber length and the optimum length of banana fibers to obtain the maximum properties is 3 cm.
Figure 4.12 The variation in flexural strength (a) and flexural modulus (b) with fiber length for DPF/Polyester composites.
b. Effect of fiber weight fraction on flexural properties

The variation in flexural strength and flexural modulus values with fiber weight fraction in DPF/polyester composites is shown in Figure 4.13. The effect of weight fraction in the range from 6 wt.% to 10 wt.% of fibers content was studied. A gradual increase in the flexural strength values of the DPF/polyester composites was observed as fiber fraction increases from 6 wt.% to 9 wt.% However at 10% weight fraction, the flexural strength decreases. The maximum flexural strength of DPF/polyester composites is obtained with a fiber weight fraction of 9%. The lower flexural strength with a fiber weight fraction lower than 9% is probably a result of lower density of the composite when compared with the density of the neat resin, or it could be attributed to the inability of the fiber to carry the load [3, 11, 13, 33]. However, at higher fiber weight fractions, fiber-fiber contact occurs, which results in non-efficient bonding and a lower stress transfer between fibers and the matrix. At the same time the flexural modulus shows greater improvement at 9% weight fraction, in the same range studied (6-10%). Accordingly, using a fiber weight fraction of 9% will give the highest flexural properties.

The optimum fiber fraction depends on the fiber and composite types. J. Rout et al [53] reported that the best mechanical properties of coir/polyester composites were obtained by composites containing 17-25wt.% of fiber, C.A.S. Hill and Abdul Khalil [51] found the best flexural strength and modulus for coir/polyester composites were found at 45%. Deve et al [38] report that the best flexural properties for PALF/polyester composites are observed in composites with 30% fiber content. Seena et al [30] report that the best flexural properties of the banana/phenol formaldehyde composite are found at 45 wt.% from banana fibers. On the other hand, studies of unidirectional composites formed of sisal/epoxy show that there is a linear relationship between fiber content and mechanical proprieties [56]. It should be remembered that processing parameters such as the viscosity
Figure 4.13 Variation in flexural strength (a) and flexural modulus (b) with fiber content of DPF/polyester composites.
of the resin, the degree of curing, resin flow, gel time and the formations of voids have a strong effect on composites; and the difference in ability in treating these parameters from one manufacturing process to another will account for the large differences in the mechanical properties of the specimens [3].

c. Effect of surface modification on flexural properties

The flexural strength and modulus of DPF/polyester composites subjected to different types of surface modification and using 9 wt.% and 2 cm fiber length are shown in Figure 4.14. Composites prepared from alkali treated DPF have flexural strength of 70 MPa. This is about 24% increase over the flexural strength of raw DPF/polyester composites. The enhancement of the flexural strength in these composites can be attributed to the improvement of the wetting of alkali treated fibers, which occurs as a result of the removal of artificial and natural impurities from the fiber surface and the formation of fiber fibrillation due to the reaction with the soda solution. A strong interfacial adhesion takes place between the fibers and the matrix, which improves the mechanical properties of the composites through an efficient load transfer between the fibers and the matrix. Composites prepared from DPF treated either by using detergent solution about 60 MPa flexural strength or bleached in dioxin solution about 61 MPa flexural strength show better flexural strength than composites prepared by using raw DPF but lower strength than alkali treated fibers composites. The lower flexural strength of raw DPF/polyester composites can be attributed to a large amount of impurities on the fibers surface (Figure 4.1-a), which will lead to poor bonding between untreated fibers and polyester resin and so on lower flexural strength. Also, the lower flexural strength of composites prepared from DPF treated with detergent can be attributed to the inability of the detergent solution to clean the fiber surface of all artificial and natural impurities.
Figure 4.14 Flexural strength (a) and modulus (b) for neat resin, different treatment processes DPF/polyester composites and glass/polyester composites.
(Figure 4.6), which reduces adhesion at the interface between the fibers and the matrix, thus reducing stress transfer between the fiber and the matrix.

The flexural strength of composites made using bleached fibers about 61 MPa was lower than that of specimens prepared with alkali treated fibers (-12%). This reduction could be linked to the high number of holes and pitting (Figure 4.7) on the fiber surface, which cause a decrease in fiber strength. The flexural strength of composites prepared with DPF alkali treated fibers was lower than that of glass fiber composites (-35%). The high flexural strength of glass/polyester composites about 105 MPa can be attributed to good adhesion between the fibers and the matrix, and this can be attributed to the surface treatment of glass fibers to improve adhesion.

The flexural modulus of the different types of surface modified DPF reinforced polyester exceeds that of the neat resin (about 3.9 GPa). Raw and dioxin bleached DPF show a higher modulus (about 7 GPa) than detergent and alkali fibers about 6 GPa. The higher modulus of raw fibers could be due to the large amount of sand contaminating the date palm fibers, which works as a filler [3]. However, composites made with DPF bleached with dioxin have higher flexural modulus due to the large number of holes, which can be increase interlocking between the fiber and the matrix. In general, improvement in the flexural modulus by more than about 3 GPa was achieved by incorporating DPF in a polyester matrix, and this suggests creating high modulus specimens by using cheap, low weight and eco-friendly reinforcement.

d. Analysis of fractured surfaces of DPF/polyester composites using SEM

Fractured surfaces were analyzed using scanning electron microscopy to investigate the different failure mechanisms observed such as fiber pull-out and matrix de-bonding.
Fractured surfaces of composites prepared by raw fibers and fibers that were subjected to three different treatment processes are shown in Figures 4.15-4.18. A first feature that is common to all types of surfaces observed is the non-uniform distribution of fibers inside the matrix. Accordingly, a better performance of these types of composites could be obtained through a more uniform distribution of fibers, which will lead to better fiber surface wetting and better load distribution. Furthermore for all the observed fracture surfaces, fiber pull-out and matrix de-bonding seem to be the dominant fracture mechanisms, which is an indication of poor adhesion between the fiber and the matrix. One important observation is the relative smoothness of the fiber surface in the case of raw and detergent treated fibers as shown in Figures 4.15 and 4.16, whereas more roughness is observed in case of soda and dioxin fibers as shown in Figures 4.17 and 4.18 (the arrow pointing to different texture of fiber surface). Although adhesion seems to be improved through the different types of treatment used as shown in the respective images, it appears also that a problem of compatibility may arise from the bleaching agent used, which also affects the coupling between the fiber and the matrix as shown in Figure 4.18. This can be explain by relatively larger microvoids and matrix cracking in case of composites made by dioxin bleached fibers.

Accordingly, it is important to select the proper coupling agent in order to increase the compatibility between the fiber and the matrix so as to improve the mechanical properties of the composites. This problem of fiber surface compatibility is common to all types of natural fibers. For instance, Rout et al [25] report that using a coupling agent, it is possible to enhance the adhesion between coir fiber and a polyester matrix, which initially have poor adhesion when fibers are used without a coupling agent. Hill and Abdul Khalil [51] found poor fiber/matrix (oil palm/polyester) bonding using unmodified fibers.
Figure 4.15 SEM micrograph of fracture surface of raw DPF/polyester composites under flexural failure.
Figure 4.16 SEM micrograph of fracture surface of detergent treated DPF/polyester composites under flexural failure.
Figure 4.17 SEM micrograph of fracture surface of soda treated DPF/polyester composites under flexural failure.
Figure 4.18 SEM micrograph of fracture surface of dioxin bleached DPF/polyester composites under flexural failure.
However, when using a silane as treatment for the reinforcement, compatibility was improved and the mechanical properties increased. Finally, it is important to mention that with SEM micrographs it was observed that raw and detergent treated DPF (Figure 4.15 and Figure 4.16) show fibers with a smoother surface than DPF treated with soda and dioxin. It is anticipated that a smooth surface will facilitate extraction of the fiber from the matrix. On the other hand, soda and dioxin treated fibers (See Figure 4.17 and Figure 4.18) show rougher surfaces, which are expected to increase the amount of energy required for extraction and pulling out of fibers from the matrix.

4.3.2 Impact properties

Impact strength is defined as the ability of a material to resist fracture under stress applied at high speed. The impact properties of composite materials are directly related to their overall toughness. The impact performance of fiber-reinforced composites depends on many factors, including the nature of the constituent, the fiber/matrix interface, the construction and geometry of the composite and test conditions. The impact failure of a composite is affected by factors like matrix toughness and fiber pullout, which is found to be an important energy dissipation mechanism in fiber reinforced composites. The applied load transferred by shear to the fibers may exceed the fiber/matrix interfacial bond strength, in which case de-bonding occurs. When the stress level exceeds the fiber strength, fiber fracture occurs. The fractured fibers may be pulled out of the matrix, which involves energy dissipation [57].

In most fiber-filled composites, a significant part of the energy absorption during impact takes place during the fiber pullout process. It is known that the energy involved and the toughness are greatest when the length of the fibers is equal to the critical length ($l_c$). The relationship between fiber content and impact strength exhibits a commonly
observed relationship, in that at low fiber loading a slight increase is seen and there is an approximately linear increase thereafter.

**a. Effect of fibers weight fraction on impact strength**

In this study, the effect of different fiber weight fractions using (Ie) from soda treated fibers on the impact strength was examined and shown in Figure 4.19. The impact strength of neat resin is about 2 KJm$^{-2}$. By increasing the fiber weight fraction from 0 to 3 wt.%, the impact strength is slightly increased (by about +13%). With further increases in the weight fraction to 6% and 9%, the increase in impact strength was about 105% and 490%, respectively. According to our findings, there is a linear relationship between the impact strength and the fiber weight fraction. This is in agreement with other studies [37, 54].

**b. Effect of the treatment process on impact strength**

The effect of various surface treatments of date palm fibers on the impact strength of DPF/polyester composites containing 9 wt.% and a length of 2 cm is shown in Figure 4.20. Alkali treatment of DPF results in a higher impact strength of the composite about 12 KJm$^{-2}$ than other treatment processes used. Low impact strength in the case of raw and detergent treated fibers can be attributed to poor adhesion between fibers and the matrix, which will decrease the energy needed for fiber pullout. It is worth mentioning that this feature was also found previously with the flexural test. DPF bleaching with a dioxin solution shows comparable results with alkali treated fibers. This result can be attributed to interlocking between fibers and the matrix due to holes on the dioxin treated fiber surface. On the other hand, glass fiber/polyester composites show higher impact strength than DPF/polyester composites. The impact strength of a composite is affected by many
Figure 4.19 The variation in impact strength with fiber weight fraction.
Figure 4.20 The impact strength of different treatment processes and random glass fibers/polyester composites.
factors, such as the toughness properties of the fibers, the nature of the interfacial region between fibers and matrix, and the frictional work needed to pull out the fibers from the matrix [3, 38, 57]. The high toughness of glass fiber/polyester composites is attributed to the high energy needed to pull out fibers from the matrix. This is a major contributor to the observed toughness of the composites and can be attributed mainly to the regular cross section of glass fibers and the surface treatment of glass fibers, which has been highly optimized. These characteristics are absent from natural fibers and consequently any enhancement in the toughness of these natural fibers must be attributed to the nature of the fiber matrix bond or the inherent toughness of the fibers themselves [51]. It is known that impact strength is very sensitive to adhesion and very strong interfaces usually have a detrimental effect on impact properties. In addition, toughness enhancements may also occur because of the presence of plant fibers, because they are composites themselves and are capable of displaying additional energy absorbing mechanisms such as defibrillation.

Natural fibers are composed of amorphous and crystalline regions and different treatment processes, such as alkali treatment will increase crystalline regions, which is expected to decrease the toughness of the fiber itself. However, this will be a minor effect when compared to the adhesion between the fiber and matrix and the energy needed to pull out the fiber from the matrix.

4.2.3 Water absorption

Using thermoset polymers as a matrix has the possibility to eliminate one of the main problems encountered with natural fibers, which is the high degree of moisture absorption. In this section, the effect of fiber weight fraction on water absorption is presented first by using two different weight fractions. Then, the effect of different
treatment processes will shown and compared with composites prepared with the same weight fraction from glass fibers. In both cases the fiber length is \( l_e \).

It has been reported that increasing the fiber content will increase water absorption [37]. In our case, we used two different weight fraction (4 wt.% and 9 wt.%) of raw DPF, and investigated the effect of fiber weight fraction on moisture absorption by immersing specimens in deionised water bath for 24 hr at room temperature. The results show agreement with previous findings, where 4 wt. % results in lower water absorption about 0.2% than 9 wt.%, which results in 0.5%. We can conclude from this experiment that high weight fraction results in a higher degree of water absorption compared with a lower weight fraction.

A weight fraction of 9 wt.% of 2 cm fiber length was used in studying the effect of treatment processes on water absorption. Also, specimens of randomly oriented glass fibers with the same weight fraction were used to study the water absorption. We used an accelerated water absorption procedure, where samples are first dried in an oven at 60°C for 24 hr before boiling with distilled water for 2 hr. The samples were cooled, water was gently removed from the surface by using cotton and the weight was recorded. Table 4.5 shows the water absorption behavior of DPF/polyester composites resulting from different treatment processes, as well as that of glass/polyester composites.

The water absorption of glass reinforced polyester composites is less than that of DPF/polyester composites. This result is expected due to the resistance of glass fibers to moisture absorption and the strong adhesion between glass fibers and the matrix, which will prevent water impregnation. Another important observation is the low water absorption of DPF/polyester composites, which doesn’t exceed 2%.
<table>
<thead>
<tr>
<th>Composite samples</th>
<th>Water absorption (%) (2h, 100°C)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Raw DPF/polyester</td>
<td>1.66</td>
</tr>
<tr>
<td>Detergent DPF/polyester</td>
<td>1.56</td>
</tr>
<tr>
<td>Soda treatment DPF/polyester</td>
<td>1.62</td>
</tr>
<tr>
<td>Dioxin bleaching DPF/polyester</td>
<td>1.74</td>
</tr>
<tr>
<td>Glass fiber/polyester</td>
<td>0.74</td>
</tr>
</tbody>
</table>
Chapter 5

CONCLUSIONS

&

RECOMMENDATIONS
5. **Conclusions and Recommendation**

5.1 **Conclusions**

There is a rapid growth in the use of renewable materials as a replacement for synthetic materials as environmental regulations become more stringent. Research and development dealing with the use of natural fibers is increasing. In addition, the development of eco-friendly biodegradable composites depends on using renewable resources (bio-fiber and natural resin) that are competitive with synthetic ones.

The ability to use natural fibers in industrial applications depends on finding an effective way of extracting and characterizing these materials in order to yield a strong bond between the matrix and the fiber. Generally, natural fiber is a promising reinforcement for use in composites because of its low cost, low density, good specific strength and modulus, lack of health risk, high availability in some countries and renewability.

The objective of this study was to investigate the potential of using date palm fibers as a reinforcement for polymeric matrix composites. This objective was fulfilled and the following points and comments can be made:

1. Natural fibers derived from date palm trees have a good potential as cheap, low weight, renewable, environmentally friendly and biodegradable reinforcement for polymeric matrix to produce composites for use in moderate load applications. This potential is based on high volume production and the low price of DPF. Added to this, growing global environmental awareness and societal concern, the high rate of depletion of
petroleum resources and new environmental regulations encourage the increased use of natural fibers.

2. Raw DPF has a cylindrical shape with low density (0.9 g/cm³), good thermal resistance up to 240°C and half of its main chemical composition consists of cellulose (46 wt.%).

3. The diameter of DPF ranges from 100 to 1000 µm and a large diameter should be discarded during the selection process and the separation process should be carried out gently to avoid breaking or damaging the fibers.

4. Raw DPF exhibits a range of tensile strength (170-275 MPa), Young’s modulus (5-12 GPa) and an elongation to break (5-10 %) values and, like other types of natural fibers, DPF shows a wide variation of properties. Also, stress-strain curve of raw DPF is similar to other types of natural fibers in that it behaves like a viscoelastic material when subjected to tension.

5. Surface modification processes not only affect the fiber surface but also the chemical composition, physical and mechanical properties of the fibers. An in-depth understanding of the effect of surface modification on DPF is difficult due to its highly complex internal structure, and because the difficult to understand the role of polysaccharides present in natural fibers.

6. Mechanical properties (flexural properties and impact strength) of DPF/polyester composites were found to be influenced by fiber type and fiber content. The mechanical properties of composites prepared using soda treatment DPF/polyester are higher than those of neat resin. In addition, the best mechanical properties were achieved with a fiber length of 2 cm and a weight fraction of 9 wt.%. 

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7. Raw date palm fibers can be used unmodified in many composites where cost or lower weight with good stiffness about 7 GPa are of greater importance than high strength.

8. Different surface modification processes applied to date palm fibers resulted in a variation in the fibers properties and variation in surface topography.

9. Scanning electron microscopy for fracture surfaces confirmed that a poor interface between the fiber and the matrix is responsible for the low mechanical properties (flexural strength) of DPF/polyester composites. Improving the adhesion might led to improvement in mechanical properties.

10. Water absorption was relatively low; it increased with increase in the weight fraction of the date palm fibers; and it was slightly affected by surface modification of the fibers.

5.2 Recommendations

The use of natural fibers in reinforced composites is still at an early stage when compared with the use of synthetic fibers. DPF can be used as a reinforcement with a polymeric matrix, but there are many points that should be investigated. The following is a summary of research items that may enhance our understanding of these fibers in order for them to be used efficiently.

1- Study the effect of different environments where date palm trees are grown on the fibers extracted from them. This effect could be clearly understood after studying the chemical, physical and mechanical properties of DPF.

2- Explore the potential of using date palm fibers in woven form, which can be readily impregnated with plastic, in order that these fibers may find a better utilization,
particularly as fillers or reinforcement in relatively low cost applications (where high strength or high modulus is not required).

3- Investigate the internal microstructure of DPF, especially the spiral angle and the degree of crystallinity, by using X-ray diffraction techniques.

4- DPF as a hollow tubular (cellular fibers) structure is expected to yield good reinforcement with high damping properties, and have the capacity to reduce the transmission of vibration by mechanical distribution to a structure. For that reasons, DPF may provide better insulation when compared to glass fibers against noise and heat (low thermal conductivity) in applications such as automotive door/ceiling panels and panels separating the engine and the passenger compartment. It is worth mentioning that one of the most important functions of DPF is to protect the date palm stem from the surrounding high temperature environment.

5- Study the effect of other surface modification processes on DPF. This target could be attained by using different physical and chemical treatment methods, presently used with other common natural fibers in literature.

6- Use different types of polymeric matrix, especially thermoplastics, with date palm fiber and investigate the opportunity for using DPF as a reinforcement with biodegradable polymers.

7- Investigate the possibility of separating the hollow fibers composing date palm fiber, which will produce fibers with a lower diameter and a higher fiber aspect ratio.
References


40. N.E. Zafeiropoulos, C.A. Baillie and J.M. Hodgkinson, "Engineering and characterisation of the interface in flax fiber/polypropylene composite


قوة الاتصال بين المواد البلاستيكية والألباني. وأظهرت الدراسة جليل الأثر للمعالجة على خواص الألياف وعلي السطح الخارجي للألياف وكذلك الاختلاف في مدى التأثير لكل طريقة عن الأخرى.

تم استخدام ألياف النخيل كمواد مدعمة لمادة بلاستيكية (بوليوستير) وذلك بهدف إنتاج مواد مركبة وإخضاعها إلى بعض الاختبارات الميكانيكية: مثل الثني وقوة تحمل الصدمات وتم التعرف إلى مقاومة الثني ومقاومة الصدم للمادة المنتجة. وأثبتت الاختبارات ارتفاع المواصفات الميكانيكية للمواد البلاستيكية بعد تقويتها بألياف النخيل مثل الصلابة وزيادة المقدرة على امتصاص الصدمات، وكذلك أظهرت الدراسة أن الألياف المعالجة بملحول الصودا كانت مواصفاتها الميكانيكية أعلى من الألياف المعالجة بالمحاليل الأخرى، وذلك عل

بقوة الاتصال بين الألياف والمادة البلاستيكية.

لقد أثبتت هذه الدراسة إمكانية استخدام الألياف المستخرجة من النخيل كمواد مدعمة ومقوية للمواد البلاستيكية وكذلك كبدائل للألياف الصناعية.

إن النتائج المهمة لهذه الدراسة التوصل إلى إيجاد مادة خام محلية رخيصة التكلفة وتتجددها وصديقة للبيئة وقابلة للتدوير أو التحلل ولها القدرة على استبدال مواد خام صناعية عالية التكلفة وغير قابلة للتحلل. وكذلك أوجد فرصة لاستخدام الكميات الكبيرة المنتجة من ألياف النخيل المحلية.
الملخص

خلال السنوات الأخيرة زاد الاهتمام العالمي ببحث وتطوير المواد المستخرجة من المصادر الطبيعية والمجيدة للمحافظة على المواد الطبيعية والتنغشات المتنوعة في أسعار وأنتاج المواد الكربونية والتغييرات البيئية إلى جانب انخفاض أمان وفراد النفايات الصناعية وزراعة تكثيف التخلص من النفايات، وتداعي الوعي البيئي في المجتمعات. من عام 1960 زاد الطلب على المواد القوية والخفيفة في مختلف المجالات وأصبحت المواد المرنة أكثر انتشارًا مما تتميز به من مراوات مقاومة الظروف المناخية والمصالح الكيميائية ومقاومة تمات معاً من الظروف الصناعية مثل (الألّياف الزجاجية والكرسيوية). ولذا فكرت فكرة استبدال هذه المواد بأخرى مستخرجة من النباتات قدوة كبيرة وذلك للاستخدام العلوي والازناء النباتية، واستعداد المواد المستخرجة من الألياف النباتية للإثارة التجارية، كما تخدم المواد المرنة بألّياف النباتات في العديد من الصناعات. وخلال السنوات الأخيرة استكشاف العديد من شركات السيارات العالمية مثل (سيبز بنز وكريسلر) استخدم المواد المرنة المعتادة على الألياف النباتية في العديد من الأجزاء الداخلية للسيارات.

تحقق الهدف من هذه الدراسة وذلك بعد التعرف على الخواص الميكانيكية والفيزيائية إضافة إلى التركيب الكيميائي والمقاومة الجزيئية لألياف النخيل، ومقارنتها بالخواص الطبيعية أخرى مستخدمة في بعض البلدان حول العالم. وظهرت الصور الميكروسكوبية أن الألياف كثيفة على شكل أسطوانة وتببل على مقدارها ما يقارب (0.02 -0.04 ميليمتر) ويتكون تركيبها الكيميائي الأساسي من ما يقارب 42% من السيليكون والألمنيوم و18% من السيليكون. وظهر أن الألياف مقاومة للحرارة إلى ما يقارب 450 درجة مئوية. ثم تم ملاحظة الألياف باستخدام بعض المحاليل الكيميائية مثل المواد الصناعية وحول الكربون وحلول اللوكس، وذلك بظهور خواص الألياف وجعلها أكثر مرونة وقوية وتحسين السطح الخارجي للألياف وذلك لزيادة
أعضاء لجنة الإشراف على الرسالة

المشرف

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دراسة إمكانية استخدام ألياف النخيل كمواد مدعمة للمواد البلاستيكية

رسالة مقدمة من الطالب

خليفة مصبح سويدان حمد الكعبي

إلى

جامعة الإمارات العربية المتحدة

استكمالاً لمتطلبات الحصول على درجة الماجستير في علوم وهندسة المواد

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