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Composites reinforced with plant fibers for biomedical applications

Trabajo de titulación presentado como requisito para la obtención del título de Ingeniera Biomédica

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Abstract

Natural fibers have acquired great interest in recent years for reinforcing composites with the aim of substitute the dependence of synthetic and glass fibers. In Ecuador, there is a potential field of natural fiber production because of its biological wealth that has positioned it within the richest countries of biodiversity in the world. From this situation arose the idea of a study with the objective to incorporate plant fibers in a polymer matrix to develop a composite, enhancing the mechanical and physical properties of their constituents, for the future use in biomedical application. Various biomedical applications, such as drug delivery systems, scaffolds for tissue regeneration, and injectable biomaterials, can be provided with the use of composites, with appropriate selection of their components and microstructure. In conclusion, aspects such as the physical and mechanical characterization of fibers and composites must be approached to approve the use of these composites in the biomedical field. Also, will be necessary to standardize the tests for plant fibers and reinforced composites with the fibers in study for future research.

Keywords: fibers, composites, biodiversity, biomedical, biomaterials.

Resumen

Las fibras naturales han adquirido un gran interés en los últimos años por reforzar los compuestos con el objetivo de sustituir la dependencia de las fibras sintéticas y de vidrio. En Ecuador, existe un campo potencial de producción de fibra natural debido a su riqueza biológica que lo ha posicionado dentro de los países más ricos en biodiversidad del mundo. De esta situación surgió la idea de un estudio con el objetivo de incorporar fibras vegetales en una matriz polimérica para desarrollar un compuesto, mejorando las propiedades mecánicas y físicas de sus constituyentes, para su uso futuro en aplicaciones biomédicas. Se pueden proporcionar diversas aplicaciones biomédicas, como sistemas de administración de fármacos, andamios para la regeneración de tejidos y biomateriales inyectables, con el uso de materiales compuestos, con una selección adecuada de sus componentes y microestructura. En conclusión, deben abordarse aspectos como la caracterización física y mecánica de fibras y compuestos para aprobar el uso de estos compuestos en el campo biomédico. Además, será necesario estandarizar las pruebas para fibras vegetales y compuestos reforzados con las fibras en estudio para futuras investigaciones

Palabras clave: fibras, compuestos, biodiversidad, biomedico, biomateriales.

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Chapter 1

Introduction

Up to now synthetic fibers such as glass, carbon and aramid fibers take globally more than 95% of the market for reinforcement fibers in the composites industry. Currently, these composites have been shown to satisfy the required mechanical properties and an established manufacturing process have supported the insertion of this type of fibers within the production of compounds (Zhu et al., 2013). However, these composites have some disadvantages such as their high fiber density and lead to some environmental problems (Cheung et al., 2009).

The growing idea of using products with lower environmental impact makes the use of natural fibers as reinforcing agents in compounds a good alternative to replace the synthetic fibers commonly used. Natural fibers in a simple definition are those fibers that are not synthetic or man-made, which come from the nature and are fully biodegradable in nature, are subdivided based on their origins, coming from plants (e.g. jute and sisal), animals (e.g. wool and silk) or minerals (e.g. asbestos) (Puttegowda et al., 2018). They are low-density materials giving place to lightweight composites with definite characteristics, generally considered as biodegradable and non-toxic (Mohanty et al., 2000). Likewise, natural fiber composite materials are limited in component applications for biomedical industry due to their relatively low mechanical properties compared to synthetic fibers and their poor interaction between fiber and matrix. These properties are being improved with the use of surface treatments, additives or coatings to acquire greater mechanical characteristics and adhesion of the fiber with the matrix (Mohammed et al., 2015).

Recently several composites of biomaterials have been examined and tested for medical applications, such as drug delivery systems, wound dressing, scaffolds for tissue regeneration and injectable biomaterials, which can be supplied with an appropriate choice of its constituents. In general, natural fibers have similar mechanical properties, as does biocompatibility with human tissues so that there are mostly no negative consequences on host tissue, which is required for any material that is used in biomedical applications. Commonly, compounds with natural fibers are applied for hard tissue applications, for example in the application of dental treatments. Dental treatments range from filling cavities (also called “dental decay”) to replacing fractured or decayed teeth, procedures where large varieties

of materials are used, such as cavity lining, cavity filling, ligated, the endodontic and periodontal treatment of teeth, etc. The choice of material depends on its ability to resemble the physical, mechanical and aesthetic properties of the natural tooth structure (Namvar et al., 2014).

Countries like Ecuador, because of its biological affluence, has positioned it within the richest countries of biodiversity in the world. It is residence to 10% of all plants species on the world and until date, it has applied only 0.1% in the manufacture of fibers (Ramalingam et al., 2016). Due to the advantages in have many natural resources, Ecuador have the chance to give high accessibility and variety of plant fibers for apply as reinforcing material in composites.

In the present study, some types of natural fibers were used as reinforcing materials, since they are abundant in nature and have minimal effect on the environment because of their biodegradable properties. Natural fiber-reinforced composites were manufactured with polyester resin like a matrix using resin transfer molding (RTM) process. The effect of natural fibers as a reinforcement on the mechanical and physical properties of polyester composite was investigated. Finally, tensile properties of natural fiber-reinforced composites were determined as a function of fiber loading and fiber extraction.

1.1 Problem Statement

Ecological problems in recent decades have induced the necessity of researchers to search novel options as agricultural and other sustainable resources, which could substitute the conventional fiber reinforced composites (FRCs). These composite materials consisted of high strength fibers and low strength polymeric matrix, now have dominated the aerospace, leisure, automotive, construction and sporting industries. Unfortunately, these fibers have serious deficiencies such as these fibers are non-renewable, non-recyclable and non-biodegradable (Sanjay et al., 2016).

In such concern, manufacturing high performance engineering materials from sustainable resources is one ambitious interest currently being pursued by researchers and entrepreneurs across the world. The use of renewable resources such as plant and animal fibers in polymer composites has been becoming an emerging area of interest for designing and manufacturing eco-friendly composites (Belgacem and Gandini, 2005). The primary reason for the development of composites from natural fiber is flexibility of type and the opportunity to acquire bio-composites possessing a broad variety of mechanical and biological characteristics.

In tropical countries like Ecuador, natural plants are accessible in a great proportion. The fibers with higher demand are the fibers of cabuya, cotton and abaca; however, it is considered that there is a large variety of fibers (Reddy and Yang, 2007). Other constituents abundant in fibers that have not been enough used are agro waste from banana, sugar cane bagasse, rice husk and coconut shell, these waste products have the benefit of being new materials that can be used like reinforcement due for their higher accessibility. Therefore, the employ of natural fibers has become a crucial approach to solve resource shortage and environmental contamination problems (Dungani et al., 2016).

Hence, from this situation arose the hypothesis that the use of plant fibers from biodiversity in polymer composites could enhance the mechanical and physical properties of both materials for the future use in biomedical applications.

1.2 General and Specific Objectives

1.2.1 General Objective

This study aims to develop and characterize a novel composite system made of biocompatible polymeric matrix with plant fibers for biomedical applications at a low cost.

1.2.2 Specific Objectives

1. Extract plant fibers with a method that has a minimal impact about the mechanical properties of the material.

2. Chemical and physical characterization of the plant fibers.

3. Prepare plant fiber-resin composite and test the mechanical properties.

Chapter 2

Natural fibers

The present chapter study the natural fibers, its classification and properties by discussing the chemical, physical and mechanical composition that these fibers possess. Additionally, the advantages and disadvantages in comparison with synthetic fibers. Finally, the applications of natural fibers as reinforcements in composite materials are introduced.

2.1 Definition

Natural fibers in a clear definition are those fibers that are not artificial, which come from the nature and are completely decomposable in environment (Chandramohan et al., 2011). These fibers are mostly classified into three varieties based on their origins in plant, mineral and animal fibers (Namvar et al., 2014). Natural fibers can be acquired from plant fibers such as sisal, hemp, bamboo, flax, kenaf, pineapple and banana as well as from animal sources such as silk and chicken feather fibers (Rohit and Dixit, 2016). Figure 2.1 shows the schematic representation of natural fiber classification.

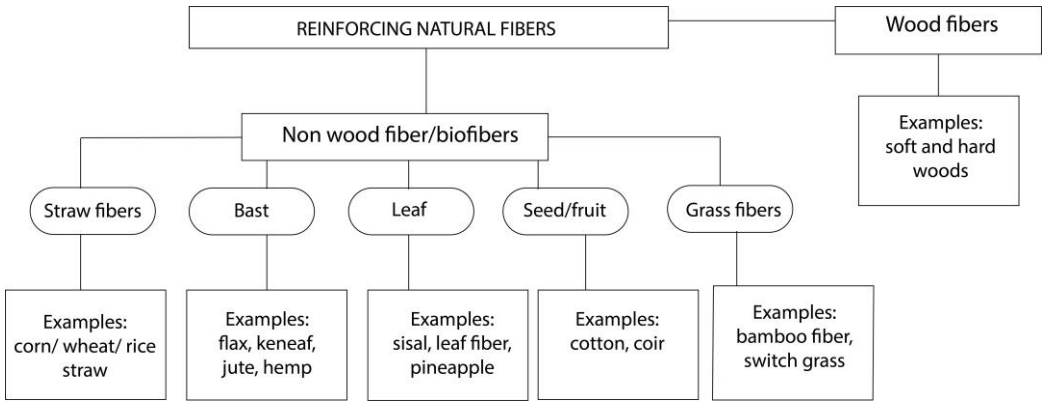


Figure 2.1: Schematic representation of natural fibers

2.2 Vegetable fibers

Vegetable fibers are composed mainly of cellulose, can be divided into six main groups based on the part of the plant from which they are collected (Fahim and Chand, 2008), such as:

1. Bast fibers: Fibers obtained from the skin or bast surrounding the stem of their specific plant. These fibers have higher tensile strength in comparison with other fibers. Some examples are flax, hemp, jute, kenaf and ramie.
2. Leaf fibers: Fibers collected from leaves. E.g. abaca, pineapple and sisal.
3. Seed fibers: Fibers collected from seed or seed cases. E.g. coir, cotton, and kapok.
4. Straw fibers. E.g. corn, rice, and wheat.
5. Grass fibers. E.g. bagasse and bamboo.
6. Wood fibers. E.g. softwood and hardwood.

2.3 Animal fibers

Animal fibers commonly composed of proteins, e.g. silk, wool, hair, feathers, are the second most important source of natural fiber in terms of accessibility after vegetable fibers for reinforcement in composites. There are various sources of each kind of animal fibers, e.g. wool or hairs are acquired from sheep, alpaca, angora, bison, cashmere, muskox and so forth (Lalit et al., 2018). Silk fibers are obtained from dried saliva of bugs or insects in the course of preparation of cocoons, examples involve silk from silk worms; and lastly avian fiber collected from birds, e.g. feathers and feather fiber (Chandramohan et al., 2011).

Researchers have examined the animal fibers potential as reinforcement in composites. Generally, animal fibers are more expensive and less available than plant fibers, the chemical composition and the mechanical characteristics of these fibers are critical to be applied as reinforcement (Madsen and Gamstedt, 2013).

2.4 Mineral fibers

Mineral fibers are naturally fibers obtained from minerals (consider synthetic fibers). These can be classified into the following categories (Pandey et al., 2012):

1. Asbestos: The only naturally existing mineral fiber. Variations are serpentine (chrysotile) and amphiboles (amosite, crocidolite, tremolite, actinolite, and anthophyllite).
2. Ceramic fibers: Glass fibers (Glass wool and Quartz), aluminum oxide, silicon carbide, and boron carbide
3. Metal fibers: Aluminum fibers

2.5 Chemical composition

The chemical composition of natural fibers differ in terms of fiber types. Most plant fibers, except for cotton, are composed mostly of cellulose, lignin, hemicellulose or similar compounds. The properties of each constituents provide the entire properties of the fiber (Pereira et al., 2015).

Cellulose is the main constituent of vegetal fibers, the strongest and stiffest part of the fiber, which plays a fundamental role in retaining the build of plant cell walls. Therefore, it is fundamental the selection of natural fibers that comprised a high percentage of cellulose in their chemical composition, with rates between 40% and 95% (Fan and Fu, 2016).

Hemicellulose is found around the cellulose fibers and is crystallized with they, it is responsible for the degradation, microabsorption and thermal degradation of the fiber (Poletto et al., 2014). Lignin is the phenolic compound, which is believed to support the structure of the plant, and it is resistant to microbial degradation. Pectin is a collective name for heteropolysaccharides and they provide flexibility to plants. The wax content make up the last part of fibers and they consist of different types of alcohols, plays a crucial role on processing composites as it influences wettability when the matrix is introduced, furthermore influencing interfacial fiber-matrix adhesion (Khoathane, 2005).

The quantity of each of these compounds vary from plant to plant, due to age and species. These basic compounds in part establish the physical and mechanical properties of the fibers, so requires a comprehensive comparison. (Council et al., 1994). Chemical composition of some selected natural fibers is reported in Table 2.1.

Fiber	Cellulose [wt%]	Lignin [wt%]	Hemi-cellulose [wt%]	Pectin [wt%]	Wax [wt%]	Moisture [wt%]	Ash [wt%]	Micro-fibrillar angle [°]
Jute	61-71.5	12-13	17.9-22.4	0.2	0.5	12.5-13.7	0.5-2	8
Flax	64.1-71.9	2-2.2	64.1-71.9	1.8-2.3	1.7	8-1.2	-	5-10
Sisal	78	8	10	-	2	11	1	-
Coir	37	42	-	-	-	11.36	-	30.45
Banana	83	5	-	-	-	10.71	-	11-12
Kenaf	45-57	21.5	8-13	0.6	0.8	6.2-12	2-5	2-6.2
Hemp	70.2-74.4	3.7-5.7	17.9-22.4	0.9	0.8	6.2-12	0.8	2-6.2

Table 2.1: Chemical composition of selected natural fibers (Malkapuram et al., 2009).

2.6 Physical and mechanical properties

The mechanical and physical properties of natural fibers vary considerably depending on their chemical and physical composition such as the structure of fibers, cellulose content, angle of fibrils, cross section, fiber type, growth conditions and processing methods (Asim et al., 2015). Table 2.2 summarizes important physical and mechanical properties of various natural and synthetic fibers, which have been adjusted from various bibliographical sources.

Fiber	Density (g/cm^3)	Elongation (%)	Tensile Strength (MPa)	Young's modulus (GPa)
Jute	1.3	1.5-1.8	393-773	26.5
Flax	1.5	2.7-3.2	345-1035	27.6
Sisal	1.5	2.0-2.5	511-635	9.4-22.0
Coir	1.2	30.0	175	4.0-6.0
E-glass	2.5	2.5	2000-3500	70.0
S-glass	2.5	2.8	4570	86.0
Carbon (standard)	1.4	1.4-1.8	4000	230-240

Table 2.2: Mechanical properties of natural fibers as compared to conventional reinforcing fibers (Bledzki and Gassan, 1999), (Islam et al., 2008).

Since these promising characteristics of natural fibers, arises the idea a potential application as a reinforcing component. The formation of hard and strong composite materials needs reinforcing fibers with critical qualities like high strength and low elongation, so these two features must be taken into consideration (Mohammed et al., 2015).

2.7 Advantages and limitations

Clearly, synthetic fibers provide properties more stable than natural ones, nevertheless, the biodegradable reinforcement has several environmental and cost benefits such as low weight, low cost, and it is environmentally friendly (Thakur et al., 2014). Cost benefits may also be realized by the use of plant fibers as a replacement for glass. The over production of certain agricultural commodities has resulted in great interest in the production of crops. For example, tropical fibers are produced in millions of tons per annum; so new applications are urgently required for these materials (Sanjay et al., 2016). For these reasons, natural fibers have recently attracted the attention of researchers.

Although natural fibers and their composites may bring various benefits (unlike traditional sources of reinforcement), these have many disadvantages. These have weak wettability, incompatibility with several polymeric matrices and high moisture absorption (Faruk and Sain, 2014). The big drawback of natural fibers, as compared to synthetic fibers, is that they do not possess the equal uniformity in composition and consequently in quality. This difference is given for some reasons, such as climate, harvest diversity and manufacturing technique applied for fibers (Fan and Fu, 2016).

2.8 Applications of natural fiber reinforced composites

The wide applications of natural fiber composites are increasing broadly throughout the world in numerous applications including automobile, building, sports, aerospace, and others industries (Sanjay et al., 2016).

This growth in the use of natural fibers could be assigned to their great achievement in mechanical properties and excellent physic-chemical properties, doing them good options for biomedical applications (Mohammed et al., 2015). The different variety of products now being developed using natural fibers is offering an opportunity to a new era of bio-composites.

Chapter 3

Natural fibers bio-composites

3.1 Definition

The word “composite” is attributed to materials that comprise an association of two or more different constituents, a strong load carrying material (known as reinforcement) embedded in weaker material (known as matrix) , having an evident interface separating them (Boeree et al., 1993). Reinforcement gives strength and rigidity, supporting the structural load. In contrast, the enveloping matrix is the composite body, the major function in a fiber-reinforced composite is to transfer stress between the fibers, surface resistance, environmental consistency and durability (Fan and Fu, 2016). Considerably, constituents of the composites maintain their specific physical and chemical properties; but together they provide a mixture of qualities which constituents separately would be incapable of generate (Tandon et al., 2013).

On the other hand, bio-composite materials are determined as composite materials in which one or more of the constituents is acquired from natural resources. Anyway, the properties and achievement of products done with natural fiber composites depend on the manufacturing techniques, properties of their individual components, as well as their interfacial bonding between polymer and fiber (Masuelli, 2013).

3.2 Polymer matrices

The principal function of the matrix, specifically in composite materials, are to give support for the reinforcement fibers. Furthermore, the matrix provides to material its shape, appearance, surface and protects the fibers before foreign agents (Bagherpour, 2012). Correct matrix selection is limited by the temperature at which natural fibers degrade. Most of the natural fibers used for reinforcement in natural fiber composite are thermally unstable above 200 ˚C, although under some circumstances it is possible for them to be processed at higher temperature for a short period. The most frequently used matrices are polymeric, metallic and ceramic; nevertheless, the most usual

modern composites are composed of a polymer matrix.

A large number of polymers are widely used because give some benefits such as low density, corrosion resistance, low manufacturing costs, large types of shapes (solids, fibers, fabrics, films, and gels), high rigidity and biodegradation (Rathod et al., 2017). Polymers can be classified into thermoplastic and thermosetting, although these terms seem similar, they possess notable differences in their properties and applications (Schneider, 2007). Understanding the main differences between these polymer types can help to make better elections to produce composites.

3.2.1 Thermosetting polymers

Thermoset polymers are hard and stiff cross-linked materials that do not melt or deforming under the influence of heat, light and chemical agents. Because its molecular structure forms a three-dimensional network of bonds (cross-linking), passing from a fluid and soluble material to another stronger and insoluble material (Ebewele, 2000). This gives the material significantly improving in the material's mechanical properties and chemical resistance, which make it a good option to be used as a matrix of composite materials.

Frequently, the costs of raw materials for producing thermoset are lower as compared to thermoplastic and possess high strength to support high stress or load compared with thermoplastics (Shubhra et al., 2013). In addition, thermoset is often easy for wetting the reinforcing fibers and forming final composites products. Some examples of this type of material are unsaturated polyester, vinyl ester, epoxies, polyamides and phenolic epoxy.

3.2.2 Thermoplastic polymers

The thermoplastic polymers are composed of linear chains of molecules with strong intramolecular bonds, but weak intermolecular bonds. This type of plastic have the particular characteristic that once polymerized they can soften or melt at a certain temperature to change its shape (Henry, 2014). Unfortunately, the development of thermoplastic natural-fiber composites is constrained because its processing is not easy to control when they are crystalline or semi-crystalline polymers, they have a short life for fatigue and are highly dependent on temperature changes (Hayashi et al., 2017). Among the thermoplastic polymers, the most used in composite materials are polyethylene, polypropylene (PP), nylon and polycarbonate, among others.

3.3 Reinforcement Architecture

Generally, materials are harder and stiffer in a fibrous shape than in any other shape. For this reason, in this study we take into consideration composites, in which the reinforcement is formed by fibers of high strength (Mazumdar, 2001). The reinforcement architecture should be adapted to the ease in the fabrication process and to perform the design conditions. Fibers are reinforced in a matrix in different ways, whether continuous or discontinuous fibers (Campbell Jr, 2003).

The fabric architecture is categorized into three types specifically nonwoven, woven and knitted (Fig 3.1). The engineered architecture has an important effect on the final physical and mechanical properties of fabric, as well as the resulting composites (Aly, 2017).

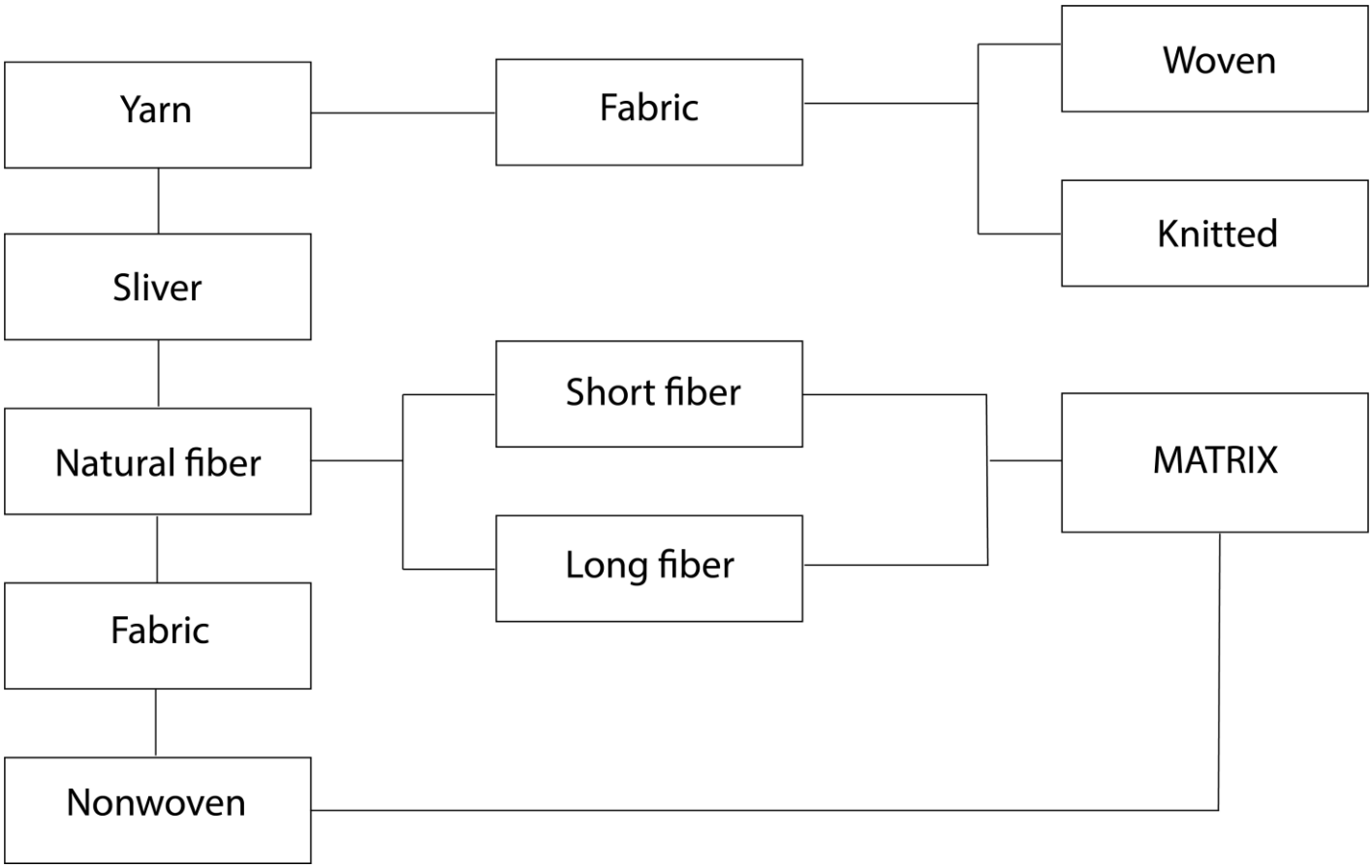


Figure 3.1: Schematic representation of natural fibers

Nonwoven fabrics are constructed based on short or long fibers, created by entangling fibers mechanically, thermally, or chemically. Several products such as filters, tissue paper, surgical drapes, and many other consumer products are nonwoven fabrics (Kalebek and Babaarslan, 2016). Nonwoven fabric has good tensile strength in fiber direction although poor strength perpendicularly to the fiber. Nonwoven fabric is additionally applied as reinforcement in composites where the stress is applied in one fiber direction (Gowayed, 2013).

Woven fabrics are generated from threads by weaving (threads interlacing) on loom. In contrast with nonwovens, woven fabric has good strength in both the directions, as fibers are oriented in 0 and 90 $^{\circ}$ (Dubrovski, 2010). Lastly,

knitted fabrics are generated from threads by knitting (threads interloping), these fabrics have higher elastic quality but lower dimensional constancy than woven fabric (Sitotaw, 2018).

3.4 Manufacture process

The primary reason to a correct selection of the manufacturing process for natural-fiber composite is that the selected process has a direct effect on the desired properties of composite structures, such as cost and ease of processing. Some significant considerations should be taken account: conservation of mechanical properties of the fibers by reducing abrasion and thermal degradation, attain a high degree of fiber dispersion controlling fiber orientation and ensure great wettability (Ahmad et al., 2015).

Composite materials admit a flexible design, considering that their organization and properties can be optimized and adapted to certain applications, so manufacturing techniques purposed and applied to fabricate natural fiber-reinforced composites are in accordance with current techniques for manufacture traditional composite materials. Despite that, methods of manufacture differ in conformity to physical and chemical properties of the matrices and reinforcing fibers (Dopko, 2018). For example, thermoset and thermoplastic polymers are different, so distinct techniques are applied for the manufacture of composites.

For instance, compression molding, extrusion, hand lay-up, injection molding, resin transfer molding and sheet molding compound could be applied for short natural fibers. Until now, the most frequent methods used for natural fiber composites are extrusion, injection molding (IM) and compression molding (Council et al., 1994). In this study, the focus was on the manufacturing of thermoset polymer composites, in particular some thermoset matrix process would be take into account as this polymer type has governed the manufacturing of composites.

3.5 Application of natural fiber composites in Biomedical Field

Majority of human tissues such as bones, tendons, skin, ligaments and teeth are composites, principally formed by of single constituents whose quantity, dispersion, morphology and properties establish the ultimate behavior of the resulting tissue or organ (Jenkins and de Carvalho, 1977). Composites can be applied to produce prostheses capable to imitate biological tissues, their mechanical behavior and recover the mechanical operations of the injured tissue (Chen and Liu, 2016).

New developments in natural fiber- reinforced composites have improve their use in biomedical applications and provide major chances for better materials from sustainable resources (Song et al., 2018). For example, silkworm silk fiber is one of the animal-based fibers, which is a possible option for structural composites and can be applied for some medical applications such as wound sutures and biomedical scaffolds. Several materials used as matrix in biomedical applications may be classified into metals, ceramics, polymers and composites fabricated from various

combinations of these materials (Li et al., 2015).

Natural fiber composites can be applied for bone and tissues restore and rehabilitation. Commonly, tissues are classified into hard and soft tissues. Bone and tooth are examples of hard tissues, and skin, blood vessels, cartilage and ligaments are some examples of soft tissues. As the name indicate generally, the hard tissues are stiffer (with higher elastic modulus) and stronger (with higher tensile strength) compared with soft tissues (Baino et al., 2015). Tables 3.1 and 3.2 shown the mechanical properties of hard and soft tissues, respectively.

Hard tissue	Modulus(GPa)	Tensile Strength (MPa)
Cortical bone (longitudinal direction)	17.7	133
Cortical bone (transverse direction)	12.8	52
Cancellous bone	0.4	7.4
Enamel	84.3	10
Dentine	11.0	39.3

Table 3.1: Mechanical properties of hard tissues (Black and Hastings, 2013).

Soft tissue	Modulus(GPa)	Tensile Strength (MPa)
Articular cartilage	10.5	27.5
Fibrocartilage	159.1	10.4
Ligament	303.0	29.5
Tendon	401.5	46.5
Skin	0.1-0.2	7.6
Arterial tissue(longitudinal direction)	-	0.1
Arterial tissue(transverse direction)	-	1.1
Intraocular lens	5.6	2.3

Table 3.2: Mechanical properties of soft tissues (Black and Hastings, 2013).

Usually, natural fibers have similar mechanical properties as well as biocompatibility to human tissues, such that commonly there is no negative effect on host tissue, which is necessary for materials to be applied in biomedical applications (Sheikh et al., 2015). With a correct comprehension of the function and interaction of implants with

the human body, it is evident until now that for better success, the implants need to be structurally compatible with the host tissues (Namvar et al., 2014). The biological environment also be dependent according to the patient's conditions and activities. Synthetic materials present their own difficulties, for this reason, inventions in the design and manufacture processes of natural fiber-reinforced composite are growing the opportunity of producing implants with better efficiency (Gittens et al., 2014).

Bio-composites can be employed in biomedical applications treating problems such as price and pollution. At present, fiber-reinforced composites are widely used materials in orthopedics, and the majority of today's upper and lower limb prostheses are fabricate from composites within a polymer matrix (Dorozhkin, 2011). Fig.3.2 shows some parts, which can be fabricated using fiber-based composites.

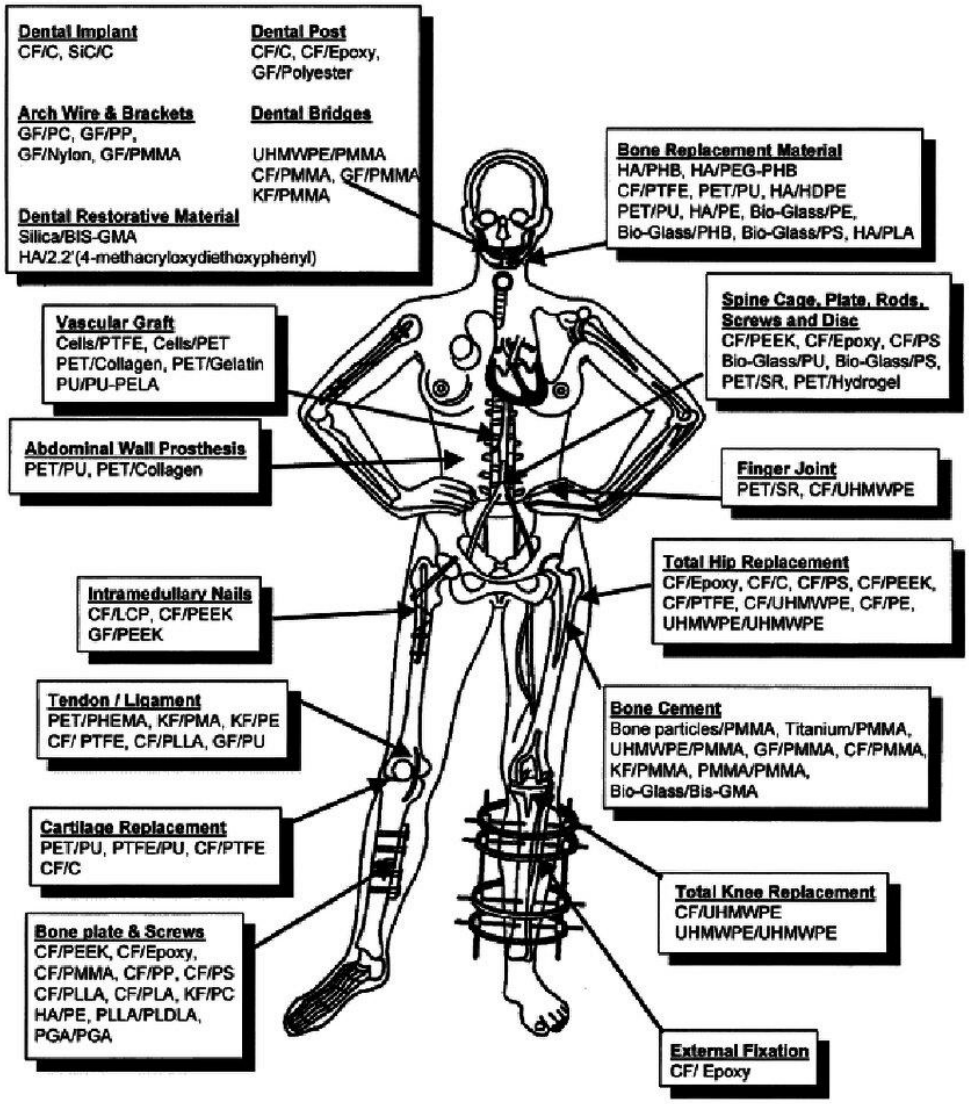


Figure 3.2: Various applications of different polymer composite biomaterials. (Ramakrishna et al., 2001))

3.6 Biocompatibility

In general, the development of biomaterials begins with those materials such as metals and ceramics, which are inserted permanently inside the host body without producing some negative reaction and interaction with surrounding tissues. However, the request of biocompatible and biodegradable materials has growing considerably since the last decade (Prasad et al., 2017).

Biocompatible materials are defined as biomaterials, and the biocompatibility is a descriptive term, which indicates the capacity of a material to operate with a correct host response, in a specific application (Ramakrishna, 2004). Several difficulties must be take account in conformity with the biological and host response to design biomedical composites and predicting their achievement. It is important to take into account that as the number of constituent materials in the composite increases, so may the variations in the host response (Chen and Liu, 2016).

3.7 Selection of biomaterials for Biomedical Applications

Clinical experience certainly suggests that not all materials commonly used in engineering are adequate for biomedical applications. For this reason, material selection is one of the most significant and crucial steps in the structural or mechanical design of composites. In this instance, it is associated to a correct selection of a reinforcement and a matrix. If the material selection is not done appropriately, the design be able to demonstrate weak performance (Sapuan, 2017). Therefore, several factors such as strength, stiffness, cost and manufacturability needed to be take into account prior the appropriate selection of natural fiber composites for biomedical applications (Khan et al., 2018).

For example, a biomaterial used as implant contains determine characteristics with the purpose to allow long-term application in the body without rejection (Wang, 2013). Considering these critical items, comparisons were done among natural fiber composites and synthetic fiber composites, for an appropriate materials selection and manufacturing process for composites (Noryani et al., 2018). Depending on the types of natural fiber, type of matrix, the proportion of fiber-matrix and the manufacturing process, the properties of fiber-reinforced composites can be adapted to obtain the requested final product (Mohammed et al., 2015).

To obtain great performance composites, coir and cabuya fibers were chosen as potential options due to their proper mechanical properties and because these fibers can be incorporated in polymers in different ways for reaching requested properties and texture, to form a biodegradable composite. With respect to the polymer that could serves as matrix, thermoplastic polymers clearly predominate in composite applications. The main reason for domination is that they are processed more easily due to viscosity that these type of polymer possess (Matthews and Rawlings, 1999). So, for the development of composite material reinforced with natural fibers predominates the idea of use thermosetting matrix, in the case of this study, polyester resin was selected as matrix to manufacture natural fiber-reinforced composites (Fig 3.3).

(a) Coir fiber (b) Cabuya fiber

(c) Polyester resin

Figure 3.3: Selected materials in this study

Chapter 4

Methodology

The chapter provides experimental methodologies that were used to obtain coconut and cabuya fibers and characterize them in physical and mechanical terms. The procedures were governed as far as possible by certified technical standards and were carried out at Yachay Tech University and Universidad de las Fuerzas Armadas (ESPE) laboratories. First, it is explained the procedure that was used to extract the cellulose fibers without affecting the mechanical properties of them .This procedure was carried out at Yachay Tech University.

Subsequently, the characterization of the fibers extracted at Universidad de las Fuerzas Armadas (ESPE) was made. Manufacturing of composites was carried out at FabLab- Innopolis with a Resin Transfer Molding resin transfer molding (RTM) process. Lastly, tensile test were carried out on the material extracted and in the composites obtained to determine their mechanical properties, this step was carried out at Universidad de las Fuerzas Armadas (ESPE).

4.1 Materials

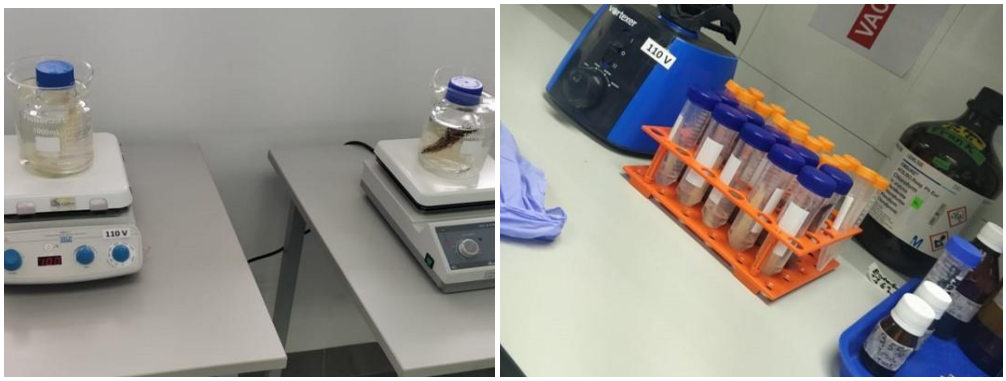
Natural fibers used in this study were coconut and cabuya fibers. Coconut fibers extracted from residual mature coconut shells and cabuya fibers were purchased from the local markets in Ibarra, Imbabura, Ecuador.

A commercially available polyester resin was used as the matrix phase for the composite and the curing agents methyl ethyl ketone peroxide methyl ethyl ketone peroxide (MEKP), catalyst cobalt naphthenate (12%) used were of commercial grade, these components were obtained from Pintulac in Ibarra, Imbabura, Ecuador.

4.2 Cellulose extraction

Fibers are strongly linked to the other components of the plant and the process of separating them must be done with great care, since the least damage is sought possible. Fiber extraction methods are diverse and involve a great influence on the fine structure of the resultant fibers and consequently on the chemical composition (cellulose, lignin, and hemicellulose contents), physical properties (density, fineness, crystallinity) as well as mechanical properties of the obtained fibers.

In this study, to extract cellulose fibers a chemical extraction have been performed, the extraction was done through a mixed process, which combine solvents, acids (hydrochloric acid (HCl)), bases (sodium hydroxide (NaOH)) and bleaching treatment process to obtain ultimate cellulose fibers (Fig 4.1).



(a) (b)

Figure 4.1: Cellulose extraction at Yachay Tech University laboratories

4.3 Characterization of fibers

4.3.1 Fourier Transform Infrared Spectroscopy Analysis

The chemical characterization of the fibers was carried out using the Fourier Transform Infrared Spectroscopy (FTIR) technique.

Finely chopped cellulosic fibers were examined by using Spectrum Spotlight 200 FT-IR instrument (Perkin Elmer, USA). First, a spectrum of the gold-plated sample holder was acquired as background, and then the spectra of the samples were recorded. The Fourier-transform infrared spectroscopy (FTIR) spectra of scans were collected for wave number ranging from 4000 to 500 cm^{-1} with a total number of scans of 36 and a wavelength resolution of 4 cm^{-1}.

4.3.2 Scanning Electron Microscopy Analysis

Scanning electron microscopy Scanning electron microscopy (SEM) is an electronic microscope technique able to produce high-resolution images of surfaces of different materials, is widely applied to image and examine bulk specimens.

The surface morphology and structure of coconut and cabuya fiber samples were examined using a MIRA 3 (TESCAN, CZ) field emission scanning electron microscope (FEG- SEM); previously a thin layer of gold coated all samples. The micrographs were taken with a voltage of 15 kV.

4.3.3 X-ray Diffraction Analysis

X-ray diffraction X-ray diffraction (XRD) has been a powerful tool for the determination of fiber crystallinity. X-ray diffraction XRD patterns were collected on an EMPYREAN diffractometer (PANalytical, NL) in a Bragg-Brentano configuration at 40 kV and 45 A and monochromatic X Rays of Cu K-α wavelength (λ = 1.541 $\dot{A}$) using a Ni filter.

4.4 Mechanical testing of fibers

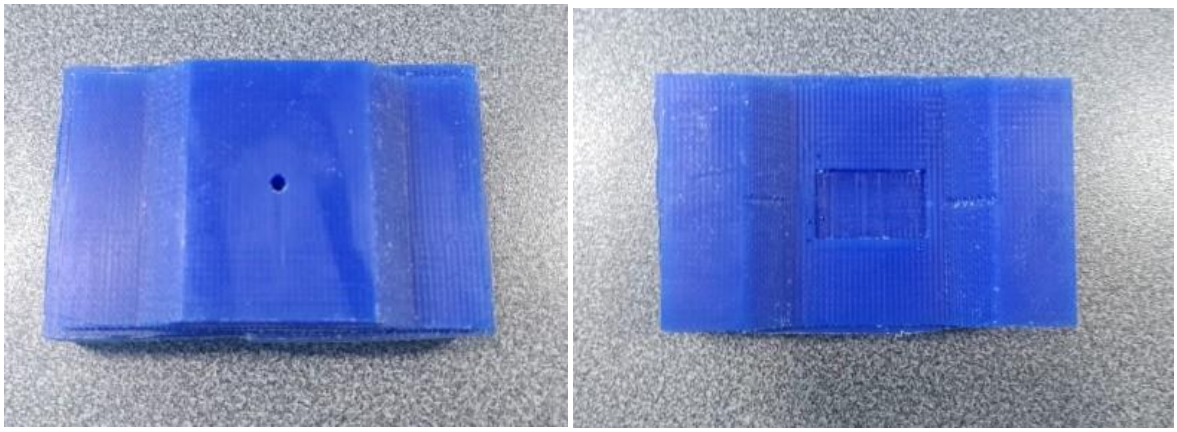
Some of the fibers obtained in the extraction process were tested under tension to find their mechanical properties, specifically the ultimate tensile strength (MPa), modulus of elasticity in tension (E) and elongation to break (%). The tests were determined according to ASTM D-3379-75 standard and were performed on a rheometer test machine (Discovery Series Hybrid Rheometer (DHR)) at a crosshead speed of 10 mm/min, operated at room temperature (25°C).

4.5 Manufacturing Process of Natural Fiber-Reinforced Composites

There are two main goals during composite preparation: to create a homogeneous dispersion of fibers ensuring uniform properties throughout the composite, and to encourage interaction between the matrix and fibers. Composites from natural fibers can be fabricated by several methods such as compression molding, vacuum bag molding, resin transfer molding, etc. Proper selection and control of processing parameters such as temperature, pressure, time, etc., are a mandatory requirement to manufacture good composites.

In this study, composites were manufactured using the resin transfer molding RTM processing technique, this process was selected as the most suitable because of the advantages listed in Table A.1 in the appendices. In RTM process; liquid thermoset resin is injected into a mold containing a fiber preform. The matrix used to fabricate the fiber specimen was a commercially available polyester resin. The main controlling parameters with this process are temperature, injection time, injection pressure, resin viscosity, preform architecture and mold configuration.

The two components for mixing, polyester resin and natural fibers, were dried under vacuum in an oven at 40 °C for 48 h, before molding. In addition, the resin was preheated and degassed at 35 °C for 20 min. The mold was designed in Onshape program and its general dimensions were 80 x 50 x 10 mm (Fig 4.2).



(a) (b)

Figure 4.2: Final mold for RTM process

The dimensions were chosen in order to obtain a unidirectional flow of the fibers. First, the wax mold was polished and then a releasing agent was applied on the mold surfaces. One percentage cobalt napthenate and MEKP was mixed in polyester resin. The dry fiber mats were placed into the mold cavity, the mold was closed, and then polyester resin was injected. The mold temperature was 30 °C at the beginning of the resin injection that was performed a varied pressure during the injection depending on the fiber content, a pressure of two bar was used for higher fiber content and 1 bar for lower fiber content. Finally, the laminates were demolded and the composites were cured according to the manufacture instructions. The composites was cured (at room temperature 25 °C) under these conditions for 24 h before testing. The final dimensions of the samples were 2 x 1 x 3 mm.

Composites were manufactured using several fiber contents (wt.%), to identify the best volume fraction in relation to the mechanical properties. The composites manufactured with varying wt. % of fibers is shown in Table 4.1.

Composite	Fiber type	Fiber content wt. %
A	Cabuya	1
B	Cabuya	5
C	Cabuya	10
D	Cabuya	25
E	Coir	1
F	Coir	5
G	Coir	10
H	Coir	25

Table 4.1: Composition of polyester resin and natural fibers composites

4.6 Mechanical Testing of Composites

After fabrication, test specimens were subjected to tensile testing to find their mechanical properties. The dimensions of the composite specimens were 2 x 1 x 3 mm for tensile testing. Tensile properties were determined based on ASTM D3039 standard using a rheometer test machine (Discovery Series Hybrid Rheometer (DHR)), at a crosshead speed of 10 mm/min, operated at room temperature (25˚C).

Chapter 5

Results & Discussion

5.1 Characterization of fibers

5.1.1 Fourier Transform Infrared Spectroscopy Analysis

Observations of FTIR spectrums were performed in order to identify characteristic peaks related to the functional groups in the fibers, such as cellulose content and residual molecules of cellular wall components of plant fibers (lignin, pectin and other inorganic/organic compounds).

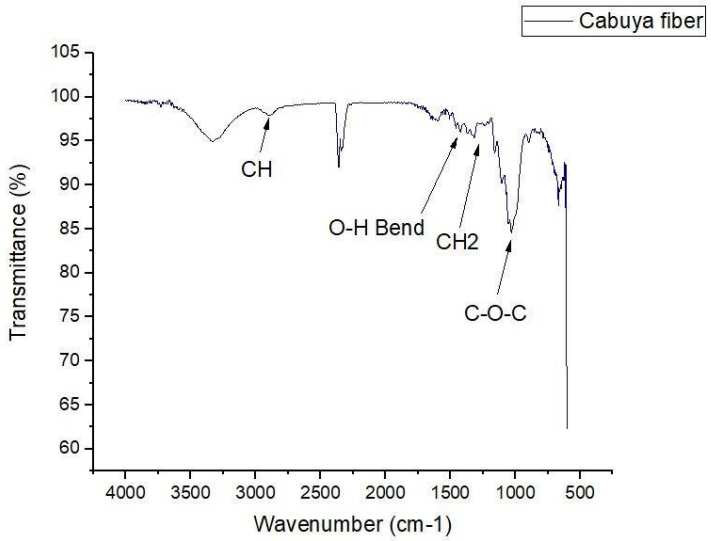


Figure 5.1: Fourier transform infrared spectroscopy (FTIR) spectra of cabuya fiber

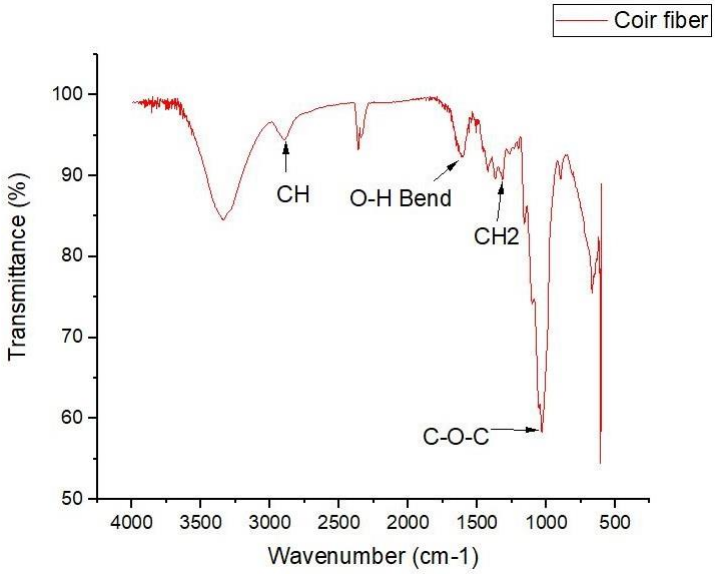


Figure 5.2: Fourier transform infrared spectroscopy (FTIR) spectra of coir fiber

Fig 5.1 and 5.2 shown the Fourier-transform infrared spectroscopy (FTIR) spectra of cabuya and coir fiber, respectively. It can be seen from these two spectra that, no considerable change occurs either in position of appearance/disappearance of the peaks. However, there was a change in intensity of some of the peaks, such as 1.250 cm^{-1}, 1.332 cm^{-1}, 1.515 cm^{-1} observed. The broad and intense peak at 3.340 cm^{-1}, suggesting OH stretching vibrations for cellulose and lignin in coir fiber.

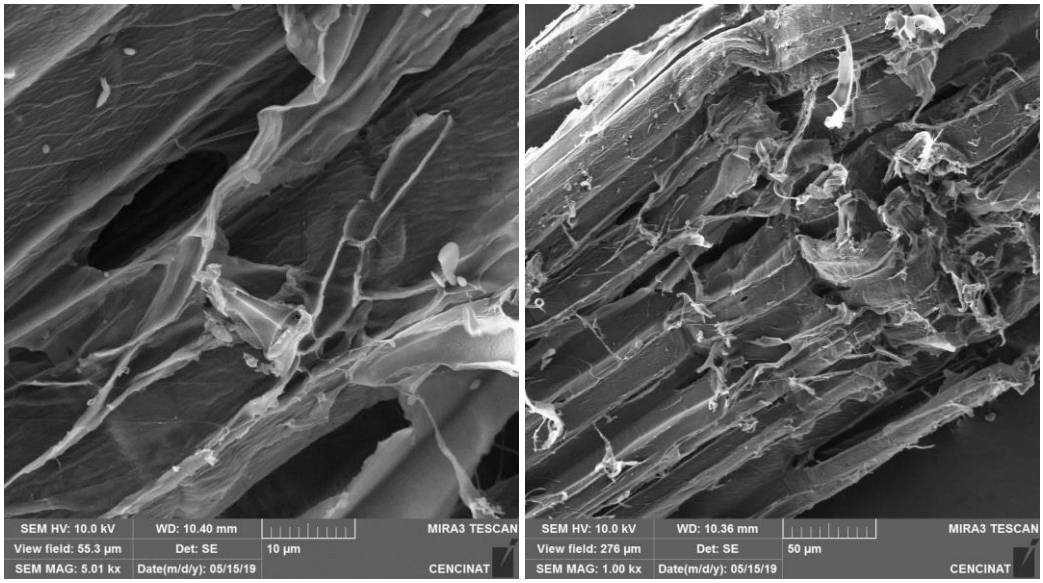
The bands at 2.893 and 1.434 cm^{-1} are attributed to the stretching of the groups C-H and CH2, respectively. On the other hand, the peak of 1.644 cm^{-1} represents the confirmation of the stretches of the O-H groups. The bands at 1.304, 1.158, 1.052, and 1.026 cm^{-1} correspond to C-C bonds, a stretch at the bridge of the glycosidic bond of cellulose (C-O-C) and C-O stretches at the positions C-6 and C-3 of cellulose, respectively. No change is observed in $\boldsymbol{\beta}$-glucosidic linkage peak of cellulose at 897 cm^{-1} in the two fibers. The C-O-C stretching absorption peak of cellulose at 1.158 cm^{-1} is more prominent in cabuya fiber than in coir fiber.

5.1.2 Scanning Electron Microscopy Analysis

Scanning electron micrographs obtained from fibers were used for a qualitative evaluation of the fiber morphology. The morphology of the cabuya and coir fibers at two different scales (1000 and 5000) are shown in Figure 5.3 and Figure 5.4, respectively. Scanning electron microscopy (SEM) observations showed that coir fiber presents a tube-like microstructure while the cabuya fiber possess a flake-like structure. In addition, observation of the scanning

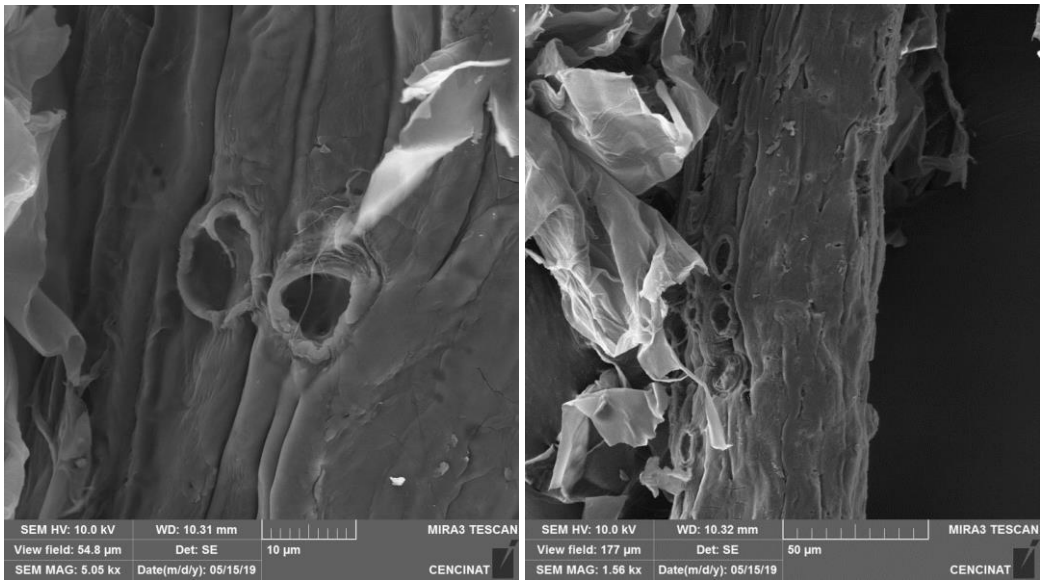
electron micrographs of coir fiber (Fig 5.4) reveals cracks, voids and parallel ridges. In the scanning electron micrographs of cabuya fiber (Fig 5.3) non uniformity seen is probably caused by heterogeneous distribution of impurities.

Therefore, scanning electron microscopy (SEM) observations reveal that each cellulose fiber presents a unique morphology, porosity, and size. Fibrillation is observed in the extracted fibers and may be due to the leaching out of the waxy substances. In addition, pores became visibly clear and it this may be due also to leaching out of waxy substances and impurities from the surface of fiber.



(a) (b)

Figure 5.3: SEM analysis of cabuya fiber



(a) (b)

Figure 5.4: SEM analysis of coir fiber

5.1.3 X-ray Diffraction Analysis

X-ray crystallography was used to investigate the crystallinity of the samples after the extraction method. The diffractograms of cabuya fiber and coir fiber are shown in Fig. 5.5 and Fig. 5.6, respectively. The crystallinity index was identified for each cellulose samples following the method described by Segal et al. using the following equation:

$$CrI(\%) = \frac{I_{002} - I_{am}}{I_{002}} \times 100 \tag{5.1}$$

where, I_{002} is the maximum intensity of the 002 lattice diffraction peak and I_{am} is the intensity shown by the amorphous part of the cellulose samples. The data obtained are summarized in Table 5.1.

Fiber	Angle I002	Intensity I002	Angle Iam	Intensity Iam	Crystallinity index (CrI)
Cabuya	22.60	6341.06	15.73	3046.70	51.95 %
Coir	22.25	2644.47	15.99	1519.64	42.53 %

Table 5.1: Angles, intensity and crystallinity index of fibers

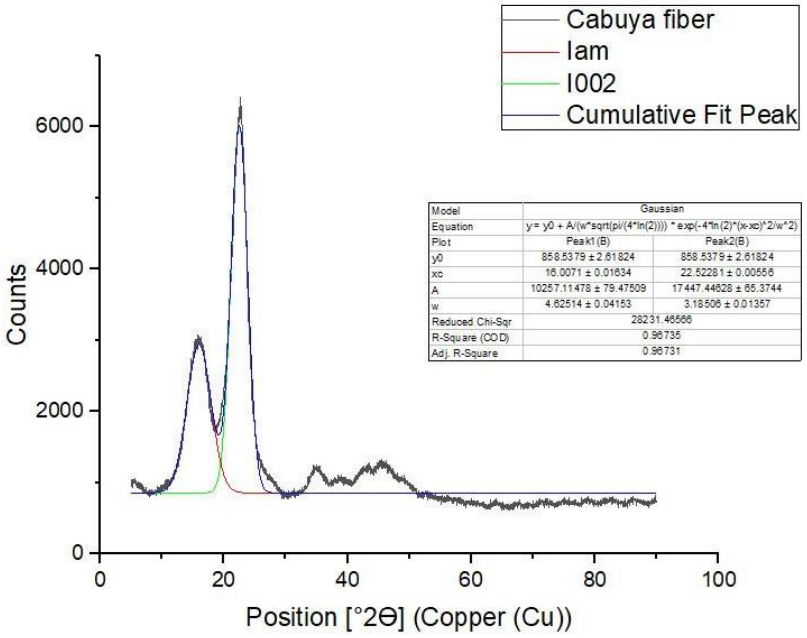


Figure 5.5: XRD patterns of cabuya fiber

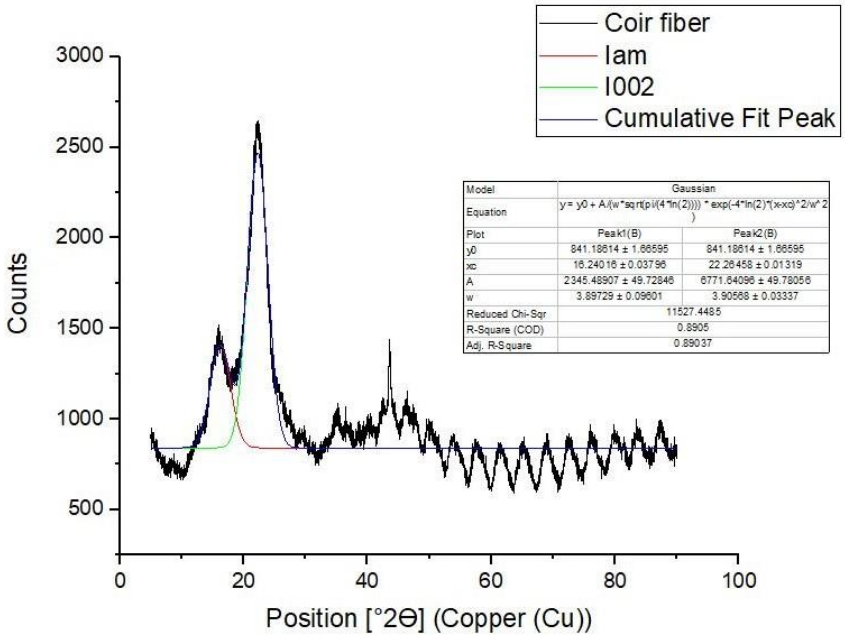


Figure 5.6: XRD patterns of coir fiber

The peaks observed for graphs displayed in Fig. 5.5 and 5.6 are quite similar and correspond to a degree of crystallinity of 51.95 % and 42.53%, respectively. The major peaks around $2\boldsymbol{\theta}$ = 20 - 25 ° are attributed to cellulose crystalline structure, while the peaks around $2\boldsymbol{\theta}$ = 15 - 18 °, represents the samples amorphous region.

The diffractogram peaks (mainly peak 002) for the cabuya fiber intensified considerably and got sharpened than coir fiber, indicating an enhancement of crystalline perfection due to removal of non-crystalline materials like lignin, hemicellulose, pectin and other inorganic/organic compounds by the extraction method.

5.2 Mechanical properties of fibers

The mechanical response of the fibers is susceptible to the speed of the test and to the environmental characteristics of the enclosure. Therefore, the tests are carried out in a laboratory that maintains stable temperature and humidity, as well as the machine that applies the load at a stable and relatively fast speed. The assembly of the fibers in the machine must be very careful, since a bad alignment between the components causes a non-uniform stress distribution and unexpected breakage of the fiber. Other effects such as torsion or bad fixation of the fiber generate erroneous readings in the results (these tests must be discarded to have reliable results).

The fibers were subjected to tensile tests to determine their strength and rigidity through a stress-strain curve. Stress-strain curves for cabuya and coir fibers are shown in Fig 5.7 and Fig 5.8, respectively. The tensile properties of the two fibers are summarized in Table 5.2. The ultimate tensile strength and the strain percentage at break of the cabuya fiber are about 63666.4 Pa and 9.64246%, respectively; whereas for coir fiber, the measured results are about 10686.1 Pa and 1.11969 %, respectively. The elastic modulus of cabuya and coir fiber are 4.67998 GPa and 0.107291 GPa, respectively. Tensile strength and modulus of cabuya fiber is higher than coir fiber. In addition, elongation at break is slightly greater than coir. Therefore, cabuya fiber exhibits stiffer characteristics as compared to coir fiber.

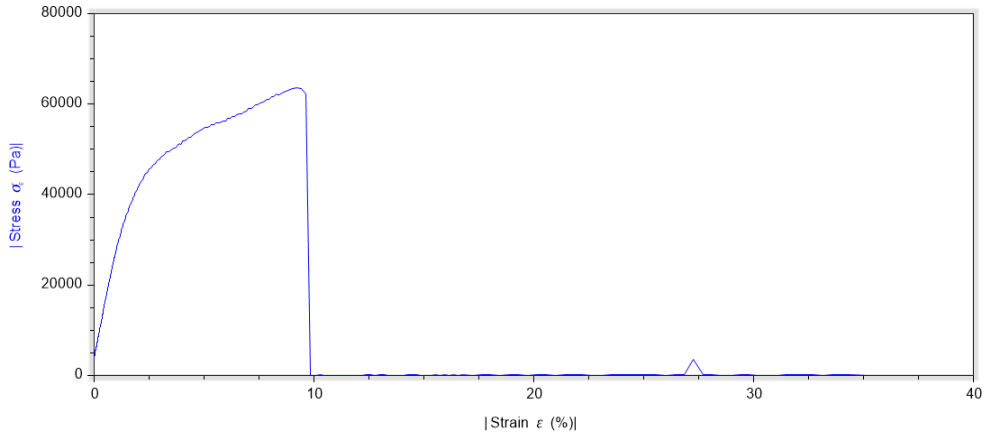


Figure 5.7: Stress-strain curve of cabuya fiber

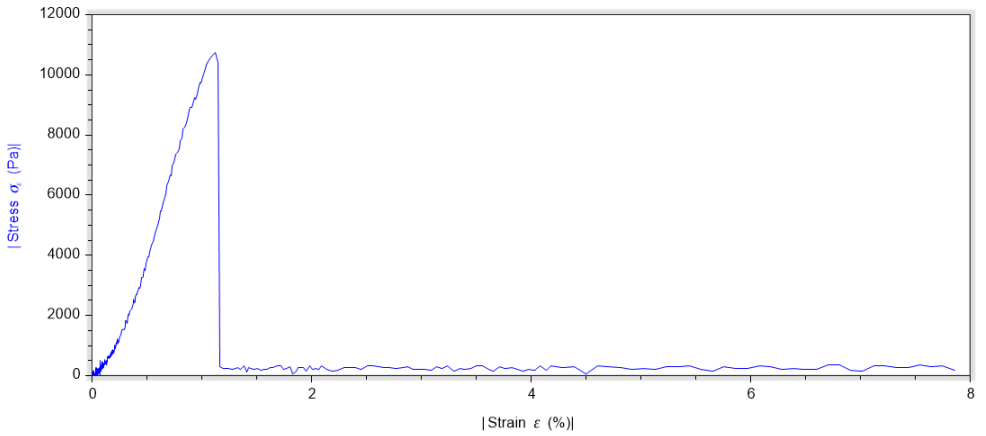


Figure 5.8: Stress-strain curve of coir fiber

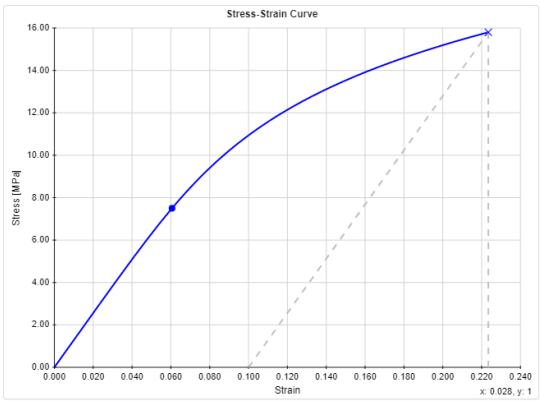
Samples	Ultimate tensile strength (Pa)	Yield strength (Pa)	Elastic Modulus (GPa)	Strain percentage at break (%)
Cabuya fiber	63666.4	44225.2	4.67998	9.64246
Coir fiber	10686.1	6030.52	0.107291	1.11969

Table 5.2: Tensile properties of cabuya and coir fibers

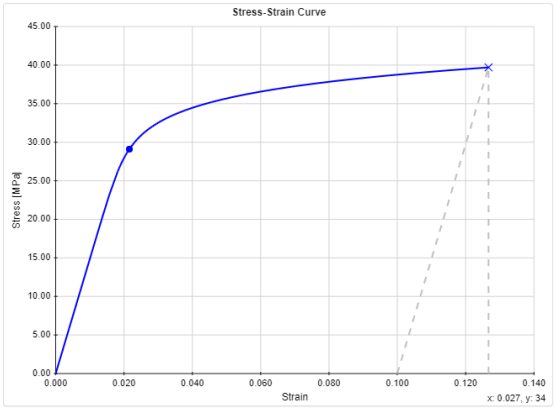
5.3 Manufacturing Process of Natural Fiber-Reinforced Composites

5.4 Mechanical Testing of Composites

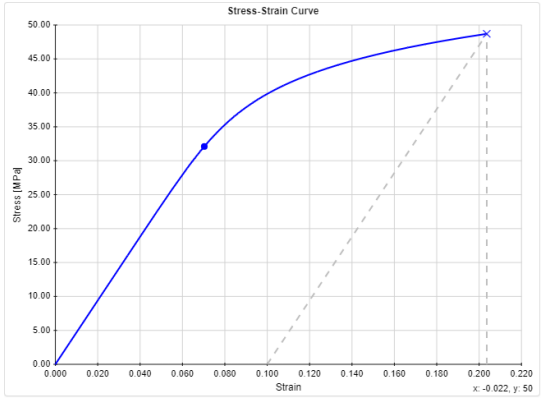
The composites were subjected to tensile tests to determine their strength and rigidity through a stress-strain curve. The stress-strain curve of the composites as a function of fiber loading for cabuya and coir fiber are given in Fig 5.9 and Fig 5.10, respectively. The ultimate tensile strength, elastic modulus, and yield strength of fiber-reinforced composites are shown in Table 5.3.



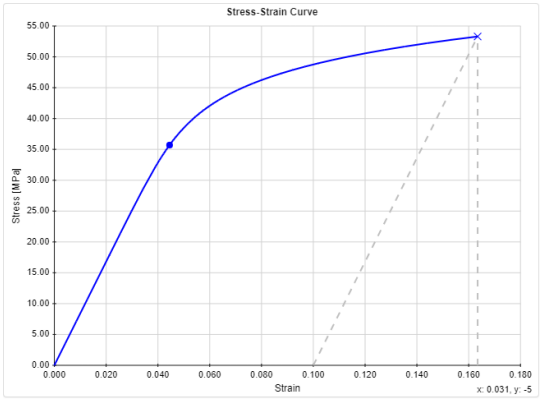
(a) 1 wt.%



(b) 5 wt.%

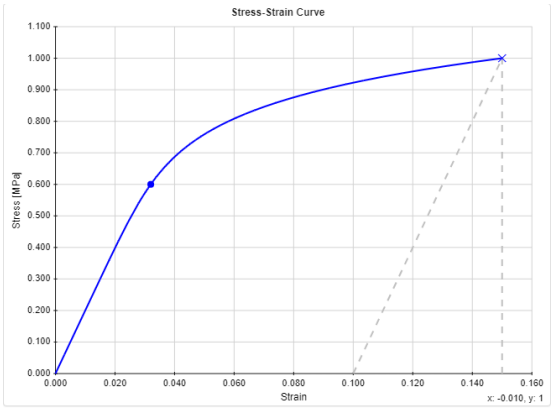


(c) 10 wt.%

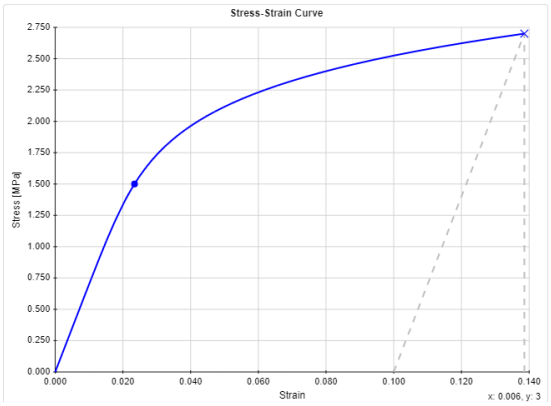


(d) 25 wt.%

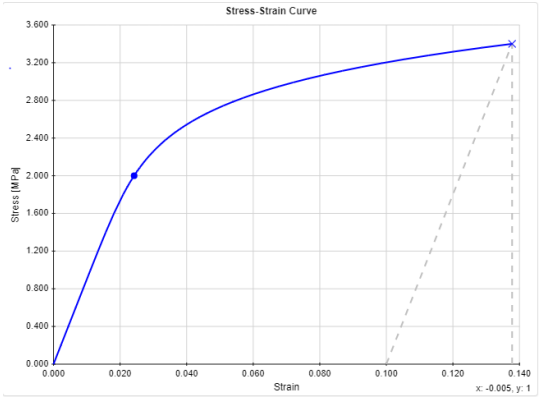
Figure 5.9: Stress-strain curves of cabuya fiber-reinforced composites at different fiber contents



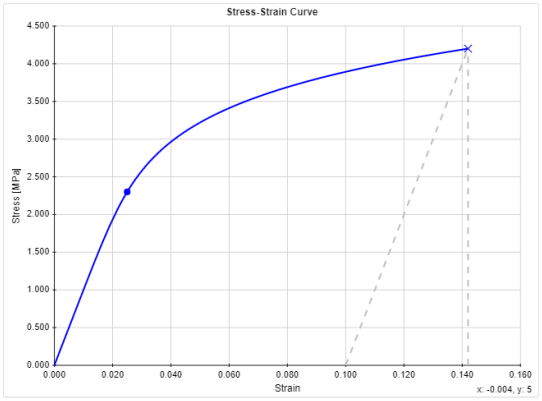
(a) 1 wt.%



(b) 5 wt.%



(c) 10 wt.%



(d) 25 wt.%

Figure 5.10: Stress-strain curves of coir fiber-reinforced composites at different fiber contents

Composites	**Fiber content (wt. %)**	**Ultimate tensile strength (MPa)**	**Yield strength (MPa)**	**Elastic Modulus (MPa)**
Cabuya fiber/ polyester resin	1	15.8	7.5	128.3
	5	39.7	29.1	1486.6
	10	49.7	33.2	450
	25	53.3	35.7	840
Coir fiber/ polyester resin	1	1	0.68	20.5
	5	2.7	1.5	70.1
	10	3.4	2	90.1
	25	4.2	2.3	100

Table 5.3: Tensile properties of composites at different fiber content

As shown in Table 5.3, tensile properties of natural fiber-reinforced polyester composites were evaluated by varying the relative volume fraction of cabuya fiber and coir fiber. The effect of fiber loading on the tensile strength of composites, the increase in tensile strength is due to the reinforcing action that fibers fulfill. In addition, the tensile modulus and elongation at break at different fiber loading are increased, indicating that transferring of stress from the polymer matrix to the stiffer fiber occurred.

It was found that a cabuya fiber-reinforced composite with 25% fiber content, demonstrated better tensile strength than all the samples. The use of coir fiber showed a lowest strength compared with cabuya fiber. This means that composites reinforced with cabuya fibers present better mechanical properties than composites reinforced with coir fibers. This could be attributed to the less adequate adhesion between coir fibers and polyester resin, resulting in the poor interfacial interaction and debonding of the matrix from the fiber during the tensile deformation.

The results indicate that in cabuya fiber-reinforced composites it is observed that a much longer plateau is located between a strain where the peak stress is reached and the strain at break. It can be concluded that the proper content addition of cabuya fiber shows a positive effect on elongation to break for polyester resin, which was expected because of cabuya fibers acting as bridges to prolong the fracture process of the composite and that the failure of the composite was controlled by the bridging effect of cabuya fiber inside the composite.

5.5 Future Outlook

Key considerations in creating these composites are fiber dispersion in the polyester resin matrix, interaction between the two phases, and sintering parameters. In order to decide the most appropriate strategy to be processed in a quick and economical way, also to avoid the loss of mechanical properties of the fibers. In addition, composites have good

tensile properties could be well developed by the judicious selection of coir and cabuya fiber.

The morphology, chemistry, and mechanical properties of fibers give them excellent potential to achieve this if used as a reinforcing phase in a composite. Although natural fibers are generally considered to be viable for use in composites, the problem of compatibility of the fiber with the matrix is an area that must be considered. Plant fibers are generally hydrophobic in nature because of an abundance of hydroxyl groups, so they are not compatible with hydrophobic matrices such as polyester. This incompatibility leads to a low fiber–matrix interfacial bond strength, poor wetting of the fibers by the matrix resin, and a reduction in mechanical performance when the composite is exposed to moisture. To overcome this, a surface treatment of fibers by physical and chemical treatments or coupling agents need to be used prior to composite fabrication of coir fiber or cabuya fiber-reinforced composites, so this consideration need to be studied in future.

Several examples of composites are abundant in literature, in order to obtain composites to be used in major load-bearing situations to replace bone, its strength and toughness must be improved. The topic is relatively new so much more work must be carried out to improve the properties of these composites, if natural fibers are to be incorporated into composite materials for medical applications, evidence of their bioactivity and toxicity is essential. Depending on the dissolution rate of the matrix material in vivo, natural fibers could be released into the body, possibly inducing a harmful response. On the other hand, the presence of natural fibers in the composite may have no detrimental effects, and could even enhance its bioactive properties.

In addition, some studies indicate that one of the most important factors in the biocompatibility of fibrous materials, with regard to the removal of foreign particles including debris, is fiber length. If biocompatibility were most dependent on length, this would suggest that shorter fibers might be nontoxic. However, there are still discrepancies in toxicity reports, so more studies are necessary.

Chapter 6

Conclusions & Outlook

Biodiversity found in Ecuador is a large source to extract natural fibers, which could be considered for the use in diverse areas. Since natural fibers are low-cost, recyclable and eco-friendly material, natural fiber reinforced-composites have potential to replace conventional synthetic composites on both performance and cost basis.

In this work, coconut and cabuya fibers were studied, physical and mechanical characterization of the fibers was made with the purpose of demonstrating the potential of these fibers as a material for reinforcing polymers in biomedical applications and as an environmentally friendly material. Results show that natural fibers, when used as reinforcement, compete with such technical fibers as glass fiber. Mechanical properties of plant fibers are much lower when compared to those of the most widely used competing reinforcing glass fibers. However, the specific properties, strength, and stiffness of plant fibers are comparable to the values of glass fibers.

The materials of natural origin provide characteristics similar to particular tissue, especially in hard tissues because of the mechanical, physical and chemical properties that these materials show. However, mechanical and physical properties of natural fibers have distinct properties between them. The abundance and affordability of cabuya fibers and their better physical and mechanical properties compared to coir fibers make them promising materials for biomedical applications.

For composite manufacture, composites were prepared at 1, 5, 10 and 25 wt.% fiber content. In all composites mechanical properties increases a bit with fiber loading. Composites having 25 wt .% showed better performance. In addition, the mechanical properties of composites are influenced mainly by the adhesion between matrix and fibers. In this study we can observed that adhesion between coir fiber and polyester resin matrix is poor. However, the adhesion can be improved by surface modification of the fiber. In conclusion, this research is a contribution to the first steps to know the properties and behavior of coconut and cabuya fibers and composite materials reinforced with these fibers. It is required further research efforts to elucidate the long-term durability of composite materials in human body conditions, since it has been seen that natural fibers have a good potential.

Appendix A

Manufacturing Factors

Advantages	Disadvantages
High repeatability	Mold and tool design are critical
Good surface finish in both surfaces with a net shape geometry	Critical layup of preforms
Lower labor intensity and skill levels	Medium to high production rates
Short cycle times	Requires matched, leak-proof molds
Low-pressure infusion operation availability	Restricted resin choice (due to viscosity)
Relatively low costs for prototyping	Air entrapment
Controlled Volatile emissions (for example, styrene)	Full wet-out of fibers can be difficult (resin flow path)

Table A.1: Advantages and Disadvantages of resin transfer molding (RTM) (Strong, 2008)

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Abbreviations

FRCs fiber reinforced composites 2
FTIR Fourier-transform infrared spectroscopy 22, 28

HCl hydrochloric acid 22

IM injection molding 14

MEKP methyl ethyl ketone peroxide 21, 24

NaOH sodium hydroxide 22

RTM resin transfer molding xi, 2, 21, 23, 41

SEM Scanning electron microscopy 23, 28, 29

XRD X-ray diffraction 23