Development, Appraisal and Characterization of a Novel Hydrophobic Kapok Fiber Reinforced Polymeric Composite

Author

SANIA ABDUL SATTAR

Fall 2019-MS Biomedical Engineering

Regn Number

00000318440

Supervisor

DR. MURTAZA NAJABAT ALI

DEPARTMENT OF BIOMEDICAL ENGINEERING AND SCIENCES

SCHOOL OF MECHANICAL & MANUFACTURING ENGINEERING

NATIONAL UNIVERSITY OF SCIENCES AND TECHNOLOGY

ISLAMABAD

DECEMBER 2021

Declaration

I certify that this research work titled **"*Development, Appraisal and Characterization of a Novel Hydrophobic Kapok Fiber Reinforced Polymeric Composite*"** is my own work. The work has not been presented elsewhere for assessment. The material that has been used from other sources it has been properly acknowledged / referred.

Signature of Student

Sania Abdul Sattar

2019-NUST-Ms-BME-00000318440

Copyright Statement

Acknowledgements

I am grateful to Allah Subhana-Watala, my Creator, for guiding me through this endeavor at every step and for every new concept He implanted in my head to help me improve my work. Without Your invaluable assistance and direction, I would have been unable to do anything. It was Your will that whoever supported me over the duration of my thesis, whether it was my parents or anybody else, therefore no one else is worthy of praise but You.

I would also like to thank my supervisor, Dr. Murtaza Najabat Ali, for his unwavering support and advice during my thesis, as well as for providing me with resources for my research. I would like to express my gratitude to him for assisting me in learning and for giving me his precious time and attention.

I am profusely thankful to my beloved parents Mr. Abdul Sattar and Mrs. Sajida Sattar for providing me this opportunity to study at one of the best institutes. Without their support this journey was impossible.

I would also like to thank my siblings, particularly Zoya Abdul Sattar, for their unwavering faith in me. She has always been a tremendous source of inspiration and provided me the financial assistance as and when required.

Dedication

Dedicated to my lovely parents, Abdul Sattar and Sajida Abdul Sattar, whose unwavering support, selfless love, cooperation, and a lot of prayers helped me to achieve this wonderful accomplishment.

Abstract

Fabricated novel kapok fiber Reinforced polymeric composite can be used for many bio medical applications. The fiber - reinforced polymer composite material made up of a polymer matrix with fiber embedded in it. The matrix material is EVA polymer, while the reinforced material is kapok fiber. Among different categories of composites being made, fiber reinforced composites can be designed by incorporating low-cost natural fibers as reinforcing materials in EVA matrix. Fabricated Kapok fiber reinforced composite by solvent casting method exhibited strong mechanical properties along with stress relaxation property inherited from EVA observed by mechanical testing and contact angle and XRD analysis shows that increased hydrophobicity inherited from Kapok fiber, semi-crystalline nature respectively. morphology, functional groups, degradation rate and non-hemolytic nature.

Based on these characteristics, the fabricated composite can be used for biomedical applications.

Key Words: *EVA, kapok fiber, solvent casting, reinforcement, degradation*

Table of Contents

List of Figures

List of Tables

CHAPTER 1: INTRODUCTION

Polymers, metals, and ceramics are the most common materials used in the manufacture of medical devices and implants. With some deficiencies, these materials address the needs and requirements of the respective device. Metals and ceramics are rigid, and therefore devices made from these materials result in poor mechanical strength and environment.[1]

Most composite materials are stable and non-biodegradable, so medical devices developed from such composite materials must be surgically removed from the human body when they are no longer needed. This prompted the researchers to study biodegradable materials.[1]

In recent years, the use of biodegradable materials has grown in importance over stable materials that do not degrade. In the field of biomedical sciences, the use of biodegradable materials is emphasized by the fact that the devices developed from biodegradable materials degrade over a certain period of time and no longer have to be physically removed when they are no longer needed avoiding the risks that come with the longer Presence of stable materials associated. [2]

Different composites are available for the medical purposes, natural fibers and Artificial fibers composites and are utilized as wound dressings and in orthopedic applications. Their use in artificial limbs draws on their specific properties, for example fatigue resistance and flexibility of composites.

Composites are commonly used worldwide. Extensive research is being conducted to meet the quest to develop cost effective, light weight, flexible and bio compatible material having high tensile strength. The quest to develop a cost-effective natural Fiber reinforced composite with desirous properties are the reasons for the selection of this topic.

Because of its lightweight, flexibility, high-stiffness qualities, and biocompatibility, fiber-reinforced polymer composites (FRPs) are finding more uses in the medical field. External components, such as artificial limbs, benefit from composites' high specific characteristics, fatigue resistance, and flexibility. Because of its lightweight, flexibility, high-stiffness qualities, and biocompatibility, fiber-reinforced polymer composites (FRPs) are finding more uses in the medical field. External components, such as artificial limbs, benefit from composites' high specific characteristics, fatigue resistance, and flexibility. Therefore, development cost-effective intelligent

solutions like FRP may benefit the country not only in terms of technological advancement but also in economic growth.

Kapok trees are found in Asia, Africa, and South America and are members of the Bombacaceae family. Kapok is a silky fiber that encases the seeds of kapok trees (Ceiba pentandra). It is yellowish or light brown in colour and has a silky gloss. Kapok fiber is an organic seed fiber that is highly lignified and mostly composed of cellulose, lignin, and xylan.[3]

The hollow tube structure of this fibers has a wall thickness of 0.8-1.0 μm. and a diameter of 8-10 μm.[4] KF is a highly light material with a low density. KF has an inherent water resistance and a high affinity for oils due to the presence of a waxy material on the surface of the fiber. The functional development of KF has gained the attention of researchers due to its unusual structure and capabilities, and KF-based functional materials now have application potential in a range of aspects.[4]

Kapok fiber is a natural Fiber with benefits such as softness, a high porous ratio, antibacterial properties, and mite elimination, among others.[5] However, kapok fibers also have obvious disadvantages such as short length, poor strength, poor elasticity and poor cohesion. Because of these disadvantages, kapok fiber cannot be spun alone, resulting in poor application in the clothing sector. At present, sound-absorbing materials made of kapok fibers can be developed by processing.

Wahi stated that utilizing Fiber sorbents as an adsorbent for removing oil from waste is the proper choice because it is highly biodegradable, easily available, feasible, and effective in addition to being able to remove oil. Due to their high rate of adsorption, stable structure, and high reusability, alkali treatment can transform the hydrophobic nature of kapok fibers into new hydrophilic fiber that have the potential to be used as natural sorbent oil.[6]

Kapok fibers are commonly utilized as filler fiber inserts and oil sorbents due to their hydrophobic-oleophilic qualities, a homogenous hollow tube shape, and low density. Hori [5] discovered that kapok fibers may be employed as an efficient oil sorbent in her research..[7]

Low cost, biodegradability, intrinsic hydrophobicity, and high sorption capacity are just a few of the benefits of kapok over traditional oil-absorbing materials.

Therefore, the goal of our study was to develop a novel fiber reinforced composite based on biocompatible polymer for the application of biomedical, characterization of fiber composite and evaluation of its mechanical testing, physical testing, chemical testing, and hemolytic activity.

Fiber-reinforced polymer composites can also contain additives, fillers, and core materials that help improve the performance, appearance, and manufacturing process of the product. Advantages of composite materials include:

1. In comparison to metals, it is inexpensive
2. Flexibility in design
3. Resistance to chemical attack to a wide range of substances
4. The material is light.
5. Durability
6. Insulation from electricity
7. Impact resistance is high.

CHAPTER 2: LITERATURE REVIEW

2.1 Executive Summary

The main idea of the research is to create resin-based composite material using polymer resin i.e., Ethylene vinyl acetate reinforced with kapok fiber. The composites will be prepared using solvent casting method and the purpose is to enhance the tensile, impact and flexural properties of the material. The composites will be created by varying the composition of resin and then the concentration of fiber.

2.2 Over view

Because of their exceptional mechanical properties and ease of processing, advanced composites are sought after for high performance applications. Composite materials are generally multiphase. This is a broader term and all naturally occurring materials fall into this category. Like steel, it is made up of two or more different phases. Composites are made up of two or more macroscopically dissimilar materials that have been bonded together. The individual physical and chemical properties of the phases are retained. The fibers are held by a matrix that transfers the load to the fibers (reinforcement phase), protects them from the atmosphere and maintains their orientation. Reinforcement is the mainstay. The reinforcement phase in the polymer matrix composite can be in a number of different forms; they can either be discontinuous, that is, in the form of whiskers, particles, flakes, short fibers, or in the form of continuous long fibers, woven fabric and sheets.[7]

2.2.1 Composite Materials

Composite materials as multifunctional materials that have properties that cannot be achieved by any ordinary material. Cohesively structured composites are formed by joining more than two compatible materials with different compositions and properties. Naturally occurring substances such as bones, wood, and shells as well as handmade products such as electrical insulators, powder metallurgical products, powdered plastics, resin-bonded magnetic materials, and paper laminates can also be classified. The disadvantage of this definition, however, was in the way that it allows it to be placed between the composites; any combination of materials without showing neither their peculiarity nor the rules that should give them that distinguish them from other ordinary and senseless mixtures.[8]

characterizes the difference between the alloys and composite materials and shows that individual elements of the composite materials retain their properties. In addition, these materials synergistically improve the properties during incorporation and suppress their disadvantages.[9]

Emphasizes that composites should not be viewed simply as a mixture of two materials. Furthermore, these mixtures have unique properties regarding strength resistance, heat resistance or other advantageous properties. The properties of these materials are unique as the individual components involved.[10]

Composites are diverse materials that are made up of at least two solid phases that are in close microscopical contact. These materials can also exhibit a homogeneous behavior microscopically in such a way that each part of them has a similar physical property. Ready-made structure in order to obtain newly processed materials with unique properties for the individual components, either alone or in combination.[11]

2.2.2 Composites Characteristics

One or more discontinuous phases are immersed in a continuous phase in composite materials. Usually, Non - continuous phases are tougher and more powerful than continuous phases, and are

referred to as reinforcement materials or reinforcement. On the other hand, the continuous phase is called a matrix.[11]

The properties of composite materials depend heavily on the material properties, their propagation, and the interaction between them. The properties of the composites can be the sum of the volume fraction of the properties of the components, or the components can combine in a synergistic manner, resulting in much better and improved properties.[12]

Apart from the idea of the components of the composites, the reinforcement geometry (size, size distribution and shape) generally influences the properties of the composites. The orientation and direction of the reinforcement materials as well as the concentration distribution also influence the properties of these materials.[13] The concentration, usually estimated as a percentage by weight or volume, controls the contribution of an individual component to the overall composite properties.[14]

Not only is this the main factor affecting the properties of the composite, but this manufacturing variable can easily be controlled and used to change its properties.[15]

2.2.3 Classification of the Composites

Composite materials can be classified in two ways, as shown in Fig 2.1; (i) Composite classification based on reinforcement (ii) Matrix-based composite classification (binder).Classifying composites based on reinforcement is helpful as it is responsible for the high performance and mechanical properties of the composites. It is further divided into three main types: (i) Particle Reinforced Composites (ii) Structural Composites (iii) Fiber Reinforced Composites.[16]

The reinforcement phase and matrix phase can be classified as in the following figure: 2.1

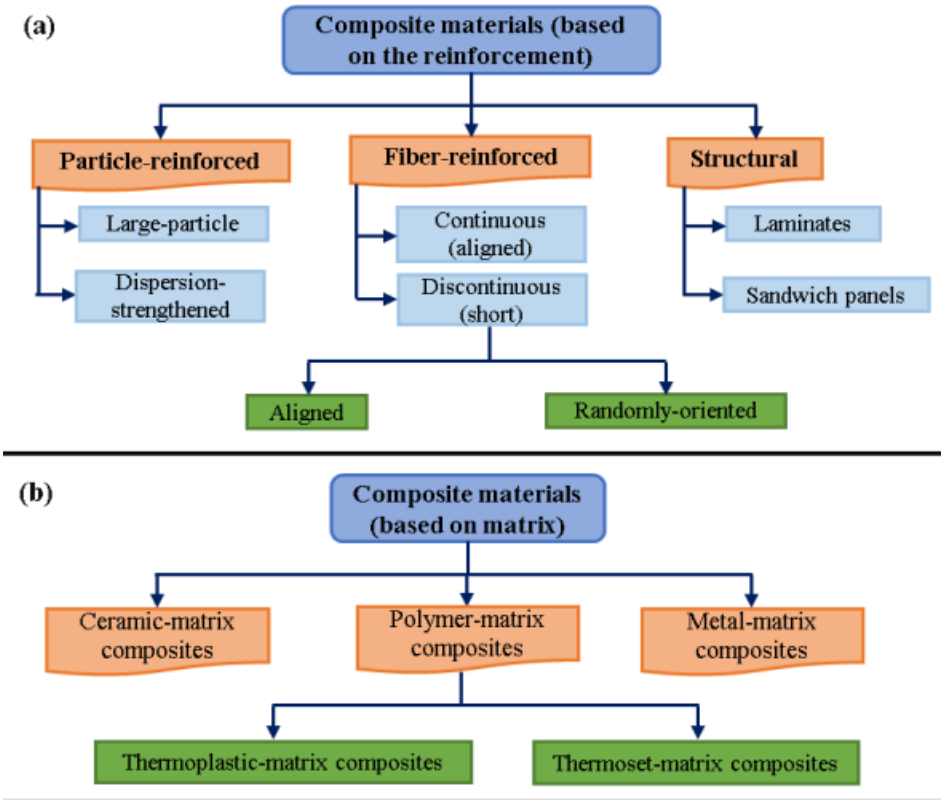


Fig. 2.1 Classification of Composites Manufacturing Techniques

2.2.4 Fiber-Reinforced Composites (FRCs)

Fibers are described by the fact that their length is significantly more pronounced compared to their cross-sectional measurements. The dimensions of the reinforcing fibers help assess their contribution to the composite properties. Fibers are exceptionally powerful in improving the fracture toughness of subject matrices. This is due to the large dimensions of the reinforcing fibers, which, particularly in the case of brittle matrices, prevent the crack initiation perpendicular to their surface, which would otherwise lead to failure. since large defects, which can show their presence in the bulk materials, are lowered due to the fibers' tiny cross-sectional area. The orientations molecular structure are responsible for the high rigidity and strength of the polymer materials.[17]

Fibers cannot be used directly in construction and design applications because of their small cross-sectional dimensions. They are then embedded in matrices to produce fiber composite materials.

These matrices serve as a load transfer and binding medium to hold the fibers together and protect them from strong environmental influences. The load transferability of the matrix is more critical in discontinuous composites compared to continuous composites.

On the other hand, fibers that are obtained from natural resources such as plants, animals or other living beings are called natural fibers. Natural fibers have gained incredible importance as a reinforcement material in polymer-matrix composites.[18] As shown in Fig. 1.3, animal, vegetable and mineral fibers are the types of natural fibers, depending on their origin.[19]
Natural fiber reinforced polymer composites have gained in importance due to the developing global energy crisis and environmental problems.[20]

Accessibility, sustainability, biodegradability, environmentally friendly, high specific properties, low density, improved energy recovery and good thermal properties, low energy consumption, low costs and non-abrasive behavior are the basic advantages of natural fibers.[21]

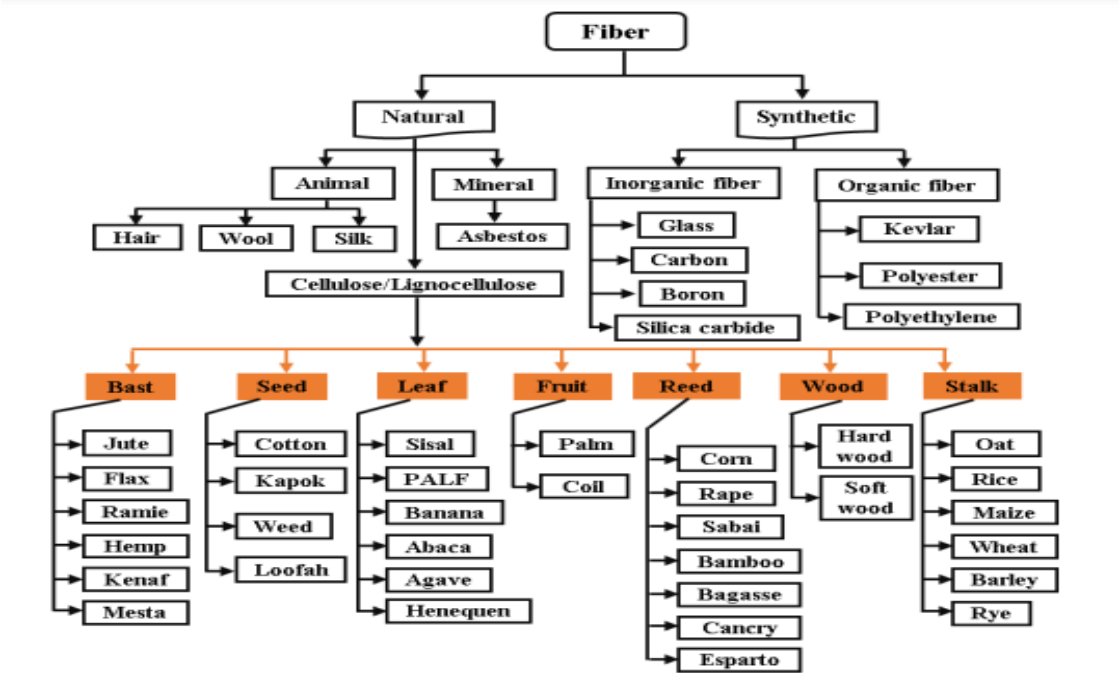


Fig. 2.2 Classification of Natural Fibers and Their Sources

2.3 Composites Based on Matrix

The composite materials consist of two or more than two elements that make up the substance with distinctive properties. Most composites are made up of some type of reinforcement (to add rigidity and strength to the lattice) and bulk material known as a matrix. As shown in Fig. 1b, Ceramic matrix composites, metal matrix composites, and polymer matrix composites are the three types of composites. Fibrous structures in many materials have excellent mechanical properties. However, these properties can only be achieved if the fibers are embedded in an appropriate matrix. The good matrix not only holds the fibers together, but also separates the worn fibers from the surrounding fibers, which prevents self-wear and the formation of new defects. A matrix is considered excellent if it exhibits the following properties: **(i)** Ability to deform under load. **(ii)** Transformation of the load on the fibers. **(iii)** Evenly distributed stresses within the composites.

2.3.1 Polymer-Matrix Composites (PMCs)

Both types of polymers (thermosetting and thermoplastic) are used as matrix media in these composites. For the production of these composite materials, polymer parameters are selected taking into account the desired mechanical properties such as fatigue resistance, adhesive strength, heat resistance, moisture and chemical resistance.[22] The resin matrix should have a mechanical strength equivalent to that of reinforcement, be easy to use and protect the composites against harsh environmental conditions. Besides this, the resins must have the wettability, penetrate the fiber bundles, reinforce by suppressing the dead air spaces, and have the properties necessary to improve the performance of the composites.

2.4 Introduction to Composite Processing

Composite parts can be made by several different conventional and unconventional techniques. The selection of this technique is based on the quality, quantity, and design of the part itself. In general, the manufacture of composites can be broadly divided into two types of techniques, open molds and closed molds.[23]

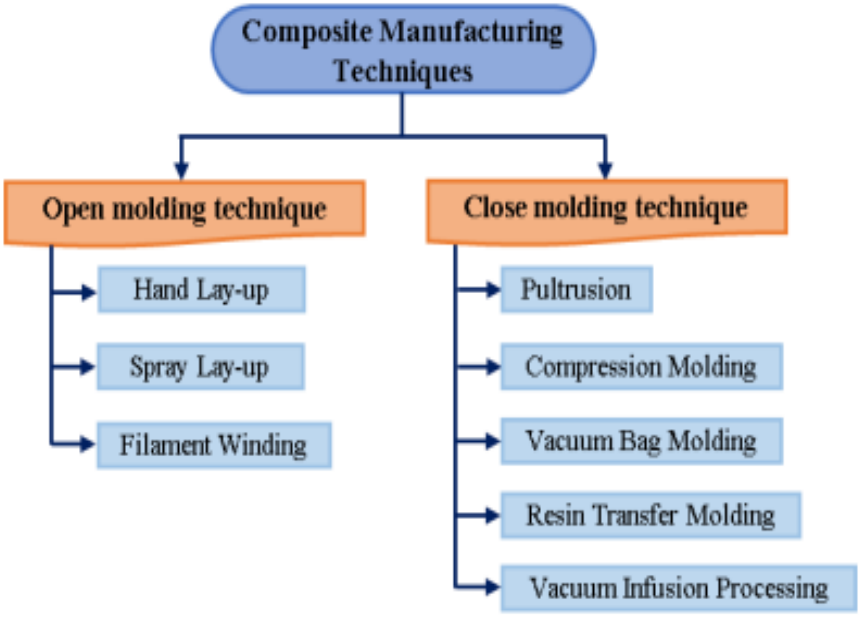


Fig. 2.3 Classification of Composites Manufacturing Techniques

2.5 Natural Fiber

Natural fibers have good physical and mechanical properties such as low cost, recyclability, non-toxicity, easy accessibility and mechanical properties such as good low density, Tensile strength and specific stiffness properties of fibers are also advantages of natural fibers and because of these properties they are preferred in various industries such as construction, Furniture and automobiles.[24]

2.5.1 Mechanical properties of NFPC

These include:

- Physical properties of fiber
- Orientation of fiber
- Strength of fiber
- Interfacial properties of fiber

2.5.2 Chemical Composition of Natural Fiber

Most vegetable fibers are made up of lignin, hemicellulose, cellulose, some water-soluble compounds, and waxes, with hemicelluloses, lignin and cellulose being the main components. The qualities of the components influence the fiber's overall properties. Thermal, micro-absorption, and biological breakdown of the fibers are all caused by hemicellulose.[25]

2.6 Kapok Characteristics

Kapok offers several advantages over crude oil material, including minimal cost, biodegradability, inherent hydrophobic characteristics, and high sorbent capacity, so it is preferred as an oil-absorbent material. Kapok Fiber demonstrated good water repellency, high oil absorption, and reusable qualities in these trials, indicating its potential as an alternative for oil spill control..[26]

Fig 2.4: Kapok tree

Kapok fiber have a thin cell wall with a large air-filled hollow lumen, resulting in low density, high bulk, good oil absorption, and water repellency. Water cannot easily infiltrate the lumen due to the negative capillary inlet pressure and the wide contact angle (> 90_) between water and the kapok fibers wall, as well as the large surface tension against air in the lumen. Furthermore,

although being mostly constituted of cellulose, the waxy cutin on the kapok fiber surface makes it water-repellent. Kapok fiber have a lower amount of cellulose and a larger portion of lignin than fibers.[27]

Fig: 2.5: kapok fiber

Malaysian kapok (Ceibapentadra, Gaertn.) grows in the northern regions of Peninsula Malaysia. The tree's delicate fruits are fluffy capsules with many dark brown seeds imbedded inside. For generations, floss has been used to fill pillows and cushions. Normally, the seeds are discarded. However, in rural regions, the seeds are roasted and eaten after the peel has been removed. They are sometimes sprouted before usage. They are only ingested in modest amounts because they are said to upset the stomach.

2.6.1 Kapok as natural sorbent

Ceibapentandra (L.) Gaertn, sometimes known as Kapok, is a type of crop. Its fibre is glossy and yellowish-brown in colour, and it is made up primarily of cellulose, lignin polysaccharide, and a tiny amount of waxy covering characteristics. It is derived from the fruits of the silk cotton tree. The hollow nature of the kapok fibre, combined with its huge lumen, has contributed to its high oil absorption and retention capability. Furthermore, the high waxy cutin content of the kapok surface contributed to its strong water repellency..[28]

Chemical composition	Percentage
Cellulose	64%
Lignin	13%
Pentosan	23%

Table 2.6.1: Chemical composition of kapok fiber[29]

The microstructure of Malaysian kapok is revealed by an investigation of its physicochemical and sorption properties, which reveal a hollow tubular structure with an average outer diameter of 2.15 to 6.5 m. Smooth surfaces were visible on the raw kapok fiber, which had a density of 1.3 g/cm3. Furthermore, physicochemical factors such as surface wax, molecular structure, physical configuration, and porosity influenced the ability of kapok fibers to absorb and hold oil.[28]

2.7 Ethylene vinyl acetate (EVA)

EVA copolymer is a heat processable, versatile and cheap material. Because of its good biocompatibility, EVA has been used over a long period of time as a material for artificial heart applications and for drug delivery to treat diseases of the blocked arteries. In addition, EVA has also been used as a drug carrier Stent material and other drug delivery equipment For oral infection treatment The percentage by weight of vinyl acetate in EVA ranges from 10 to 40%.[30]

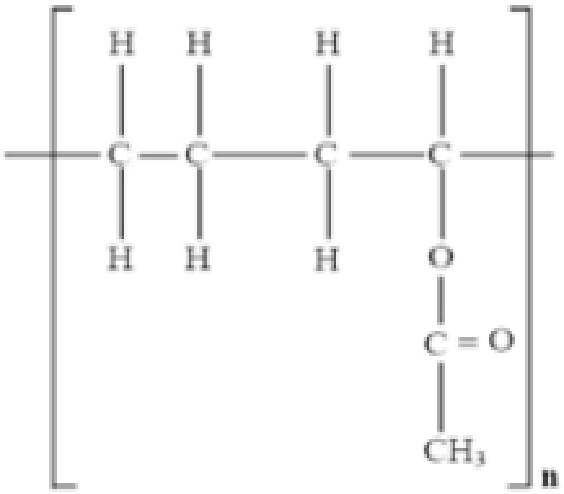


Figure 2.7: Structural formula of EVA

CHAPTER 3: MATERIALS AND METHODS

3.1 Materials

Ceiba pentandra (Kapok Fiber), Ethylene vinyl acetate (EVA), Tetrahydrofuran (THF), Glutaraldehyde, PBS, X-triton.

3.2 Selection criteria

For Composite fabrication, EVA was selected as polymeric matrix for kapok fiber as it has widely been used in biomedical application, drug delivery and wound dressing applications due to its biocompatibility, biodegradability. THF was selected as solvent due to excellent solubility of EVA in it. As purpose of our study was to develop hydrophobic reinforced polymeric composite so EVA were used as a matrix in this study based on their physicomechanical properties.

3.3 Optimization of solvent for Fiber and EVA concentration

Fiber and EVA concentration was optimized by using different concentrations in different solvents. For this purpose, Fiber concentrations were dissolved in different solvents separately such as Dichloromethane (DCM), Dimethyl sulfoxide, Dimethyl sulfoxide + Toluene, Dimethylformamide (DMF), Tetra hydro furan (THF). Fibers were not dissolved in these solvents. Fiber was dispersed in Tetra hydro furan (THF). PVA was used as a matrix material but it's not compatible with fiber because PVA was hydrophilic, and fiber was hydrophobic in nature. That's why we use EVA as a matrix material that was compatible with fiber then increased the EVA concentration 2%, 3% and observed the strength and hydrophobicity of composites. Based on these observation, Tetra hydro furan (THF) and EVA was used further for Composite fabrication in this study.

Material Selection and Optimization

Solvent selection for fiber

Solvent	Kapok fiber	Quantity(ml)	Outcome
DMSO	0.2gm	20ml	Not dissolved
Toulene + DMSO	0.014gm	30ml	Slightly dispersed
DCM	0.012gm	20ml	Slightly dispersed
DMF	0.011gm	20ml	Not dissolved
THF	0.022gm	50ml	Complete dispersion or segregation occurs

Polymer (Matrix) selection

Polymer	Property	Outcome
PVA	Hydrophilic	It makes a lump of fiber not compatible
EVA	Hydrophobic	Compatible with fiber

Table 3.3: Optimization and material selection

3.4 Composite fabrication

To fabricate fiber Reinforced composite using solvent casting method.[31] first2% EVA was dissolve in 40ml THF at 60°c for 45mins under constant stirring at hotplate to get a clear and uniform EVA/THF solution.[32]After dissolving EVA, 0.040gm kapok fiber is added in dissolved solution at 60°c for 1 hour then added 0.5% glutaraldehyde at 60°c the homogenous solution was poured into petri plate and kept at room temperature for overnight to get composite by evaporating solvent. After evaporation of solvent the composite was peeled off from petri dish.

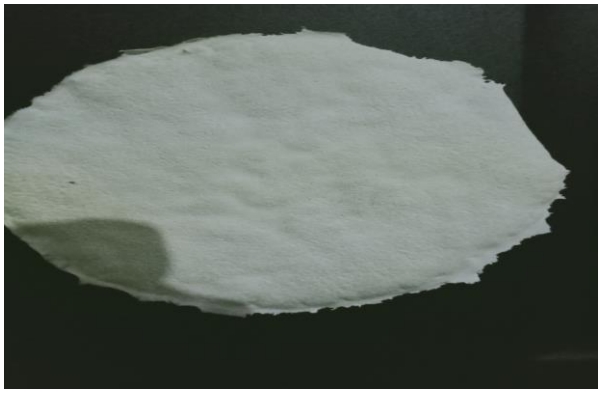


Figure 3.1: Fiber Reinforced polymeric Composite

3.5 Compositions

Samples	Polymer EVA (%)	Fiber (gms)	Crosslinker Glutaraldehyde (%)
2% EVA CL Film	2%	-	0.5%
2% EVA NCL Film			-
2% crosslinked composite		0.04gms	0.5%
2% non-crosslinked composite			-
3% EVA CL Film	3%	-	0.75%
3% EVA NCL Film			-
3% crosslinked composite		0.04gms	0.75%
3% Non crosslinked composite			-

Table 3.4: Illustrates the compositions of Composites

3.6 Characterization Techniques

3.6.1 Scanning electron microscope (SEM)

In this process, a fine electron beam is concentrated on the surface of a sample. These electrons communicate with the surface of the sample, which results in photons or electrons being knocked off the surface of the material. The detector then focuses on these knocked-off electrons[33]

The detector output obtained increases the brightness level of the cathode ray tube (CRT). Here the contact between the electron surfaces enables the release of secondary electrons (SE) (of the material), backscattered electrons (BSE) and X-rays. A common SEM detection method is carried out through these secondary electrons (SE). These secondary electrons are emitted from close to the sample surface. In this way a distinct and clear picture of the sample is obtained. It can even show sample details less than 1 nm in size. There is also elastic scattering of incident electrons and releases backscattered electrons. Compared to secondary electrons, they occur from deeper places. Therefore, their resolution is comparatively low. Fig. 3.2 shows the SEM apparatus (JSM 6490LA) that is present in SCME, School of Chemical and Materials Engineering, NUST, Islamabad.

Figure 3.2 SEM (JOEL JSM-6490LA) present at SCME, NUST

3.6.2 UTM (Universal Testing Machine)

The mechanical properties of the resulting mixture samples such as% elongation, tensile strength, ductility, brittleness was tested with UTM, a universal testing machine. The tensile and compressive strengths of the material are evaluated using a universal testing machine (UTM). The name "universal testing machine" comes from the fact that these machines can perform a wide range of tests on a wide range of materials, parts, and structures. The majority of UTM designs are modular and may be adjusted to meet specific customer needs.[34]

Universal testing machines can evaluate different types of materials, from different types of specimens including metals and concrete to soft specimens such as rubber.

Figure 3.3 Schematics of UTM

The UTM is a versatile and valuable test device that may evaluate material qualities such as

- Tensile resistance (percent)
- Elasticity of a material
- Material compression
- Yield strength in percent
- Deformation of elastic and plastic materials
- Material bending resistance
- Hardening of the strain

3.6.3 TGA (Thermogravimetric Analysis)

Thermal quantitative analysis, also known as thermal quantitative analysis (TGA), is a thermal analysis approach in which the mass of a sample is assessed over time intervals based on temperature variation. This procedure gives data about phenomena that occur in the physical world, such as

- Transitions between molecules
- Absorption of heat,
- Surface assimilation
- Desorption at the surface
- Chemisorption's,
- Thermal decomposition
- Solid-gas reactions.

  The thermal reactions required in this process can take place in a variety of atmospheres, including dense air, vacuum, or inert gas[35]

Figure 3.4: TGA analyzer

3.6.4 FTIR Spectroscopy

An FT-IR Bruker Alpha spectrometer was used to obtain an FTIR spectrum for the mixed films. The spectrum obtained from the Bruker Alpha spectrometer was analyzed and examined with the essential FTIR software to determine the nature of the peaks.

The name Fourier transform stems from the fact that it takes a Fourier transform method (a mathematical process) to turn raw data into real spectrum form.. The FTIR of the composite samples was carried out to determine the bond elongation in structures of to know fiber reinforced polymeric composite. Figure 3.5 depicts an FTIR spectrometer.

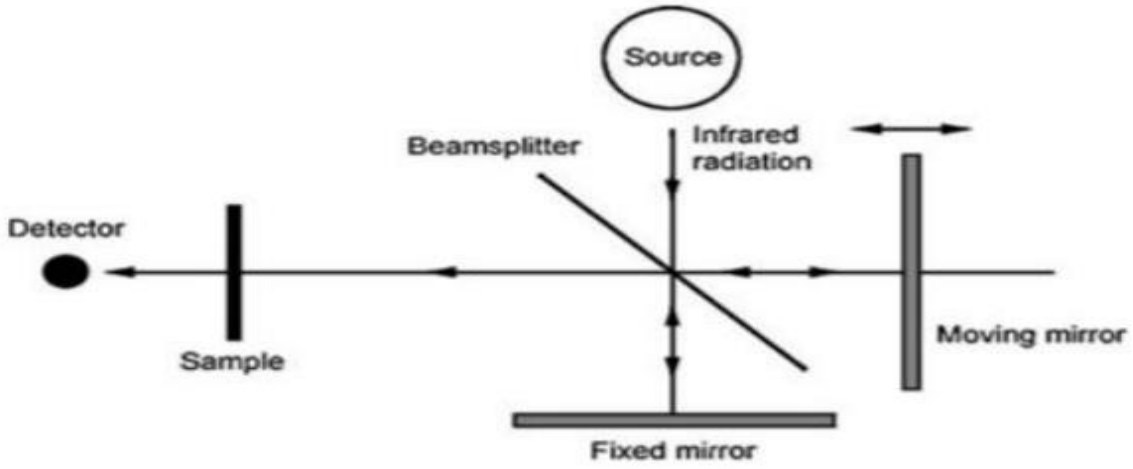


Fig 3.5 Schematics of an FTIR spectrometer

3.6.5 Contact Angle Analysis

The droplet is deposited on the substrate, which is subsequently tilted progressively during the tilting process. As soon as the droplet starts moving, the advance angle is measured at the front of the droplet. At the same time, the retreating angle is measured at the back of the droplet.

3.6.6 XRD Analysis

X-ray diffraction, or XRD, is a non-destructive test method for determining the structure of crystalline materials. The XRD analysis is used to investigate the crystal structure of a material in

order to determine the crystalline phases present and so acquire information about the chemical composition.

3.6.7 Hemolysis test

The hemocompatibility of fiber Reinforced polymeric composite, pure EVA, and kapok fiber composite (positive control) was checked by a hemolysis test. For the hemolysis test, fresh human blood was drawn into blood bags with anticoagulant drugs. To isolate red blood cells (RBCs), blood was centrifuged with PBS at 5000 rpm for 10 minutes. Erythrocytes were washed three times with PBS and then isolated erythrocytes were diluted with PBS solution for immersion of drug-loaded films. In this procedure, TritonX-100 and PBS solution were used as positive and negative controls. Drug-loaded films were immersed in diluted red blood cells and held at room temperature for 2 hours and then centrifuged at 5000 rpm for 5 minutes. Erythrocytes were discarded and the supernatant was collected for absorption measurement by UV spectrophotometry at 540 nm and then the percent hemolysis of each sample measured using the formula written below. As is the absorbance of sample films and an is the absorbance of the negative control and Apis is the absorbance of the positive control

Hemolysis (%) = [(As – An)/ (Ap)- (An)] x 100

Chapter 4 RESULTS AND DISCUSSION

4.1 Composite Samples

4.1.1 Prepared Fiber Reinforced polymeric composite

The composite was prepared according to the compositions listed in table 3.1. All the composites were prepared at constant temperatures as mentioned in chapter 3 according to the EVA compositions, with and without crosslinker. The composites prepared are shown in Fig: 4.1

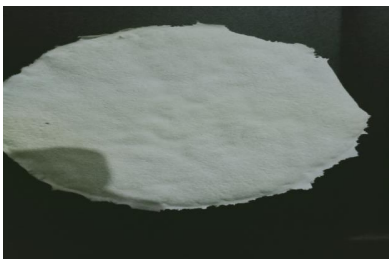


Fig 4.1: Composite

4.2 SEM Analysis of the Composite

Sem analysis was performed on the mix sheets to see the morphological changes in the composite. The following results were obtained because of the SEM analysis. The results show the change in surface structure because of mixing. With increasing EVA content, the composite surface shows a hardening which is visible in the SEM images.

The influence of EVA on fiber as a compatible material can be inferred from the SEM morphological analysis. The following are the various results obtained because of the SEM analysis. The differences in the morphological properties are clearly visible in these pictures. As the amount of EVA copolymer increases, so do the properties of fiber.

SEM analysis was done to check the pores, shape and size as well as the interaction between Polymer and fiber of the cross link and non-crosslink composites.[36]

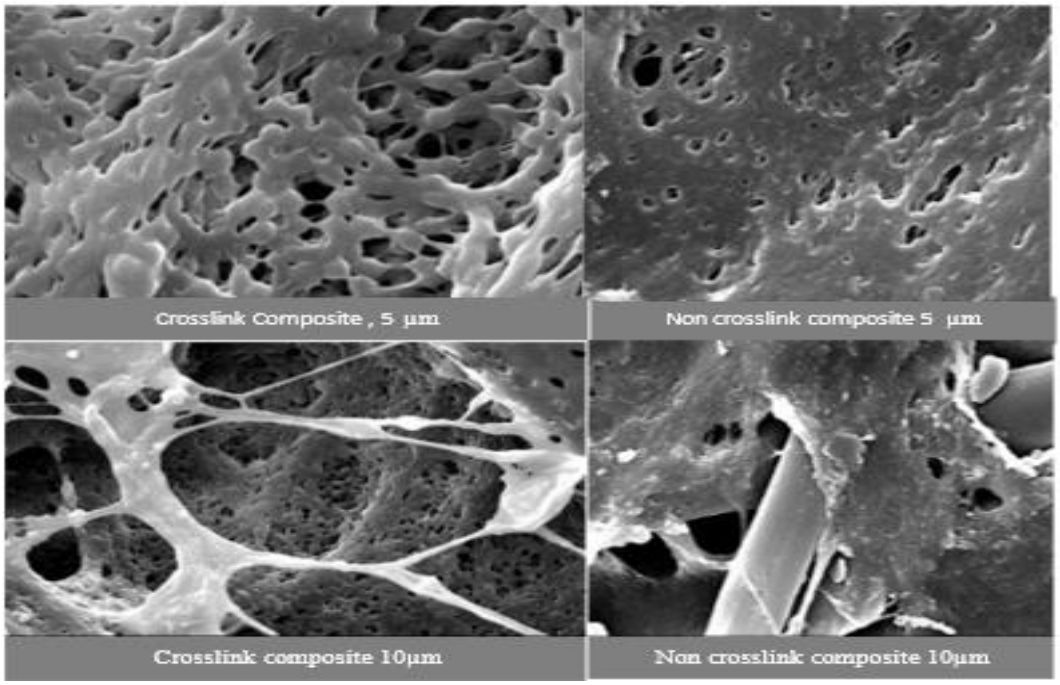


Figure 4.2: Scanning Electron microscopy

4.3 Mechanical Tests

4.3.1 UTM (Universal Testing Machine)

In order to examine the properties of this composite, we carry out various tests such as tensile, impact and bending tests.

In this test, the sample is placed between the clamps of the UTM (Universal Testing Machine) and loaded uniaxially in the longitudinal direction. The samples are prepared according to the following standards

ASTM D-638

(20x10x0.4mm)

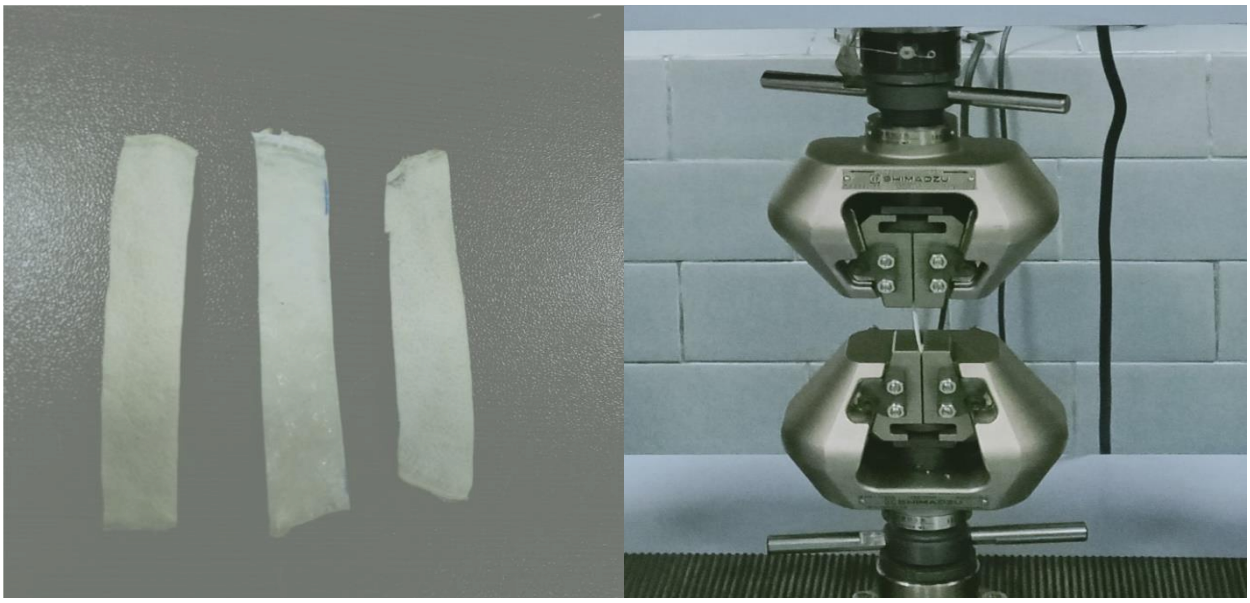


Figure 4.3: Composite samples

4.3.2 Tensile Test Results

As already mentioned, tensile tests according to ASTM D638 standard were carried out with a strain rate of 50mm / min on the Ultimate Testing Machine Shimadzu.

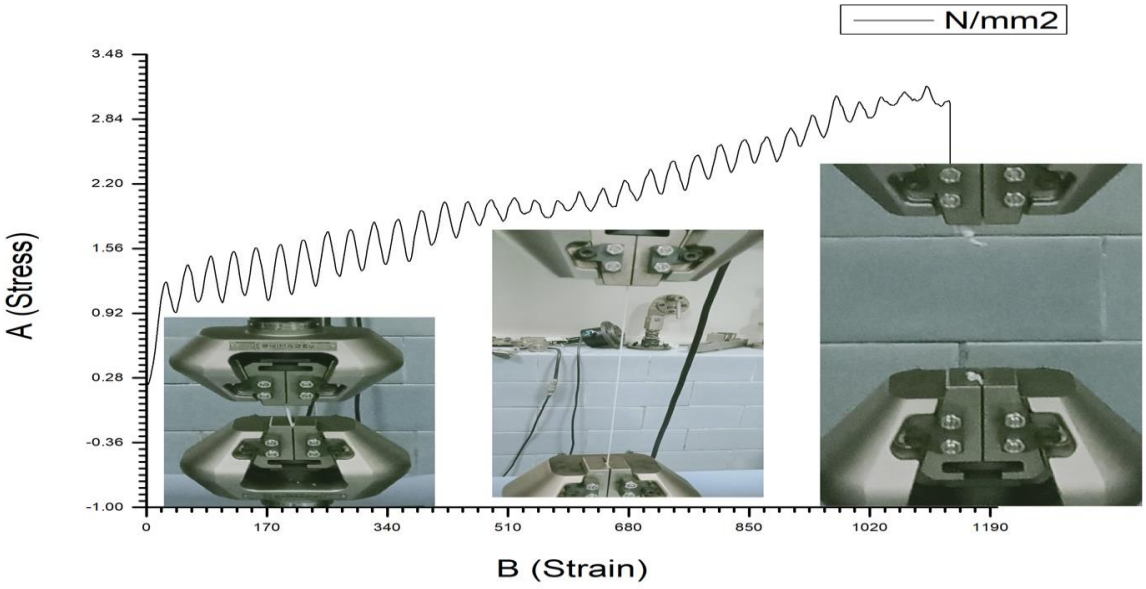
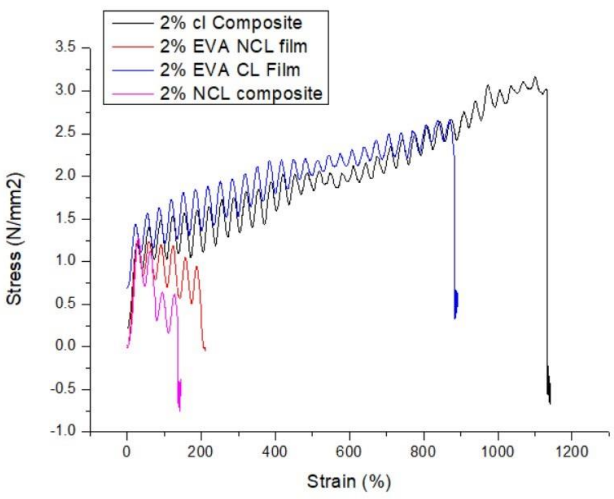
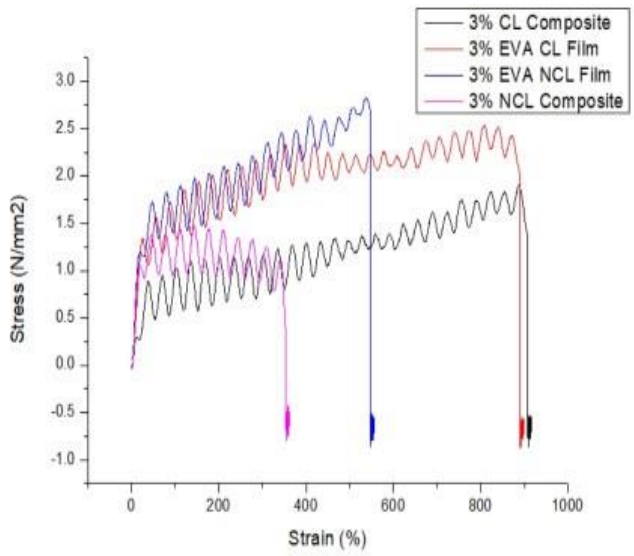


Figure 4.4: Tensile testing



Samples	Tensile Strength/Mpa	Elastic Modulus /Mpa	% Elongation	Toughness / Joules
2%EVA CL Film	2.66631	11.998	870.817	2.12547
2%EVA NCL Film	1.23580	22.122	58.381987	2.08728
2% CL Composite	3.16501	20.276	1100.70	2.75513
2% NCL Composite	1.27951	24.15	30.7710	0.09673

Figure 4.5: 2% EVA films and Composites



Samples	Tensile Strength / Mpa	Elastic Modulus / Mpa	% Elongation	Toughness / Joules
3%EVA CL Film	2.53320	17.943	809.789	2.11978
3%EVA NCL Film	1.90735	14.787	537.821	1.31558
3% CL Composite	2.82725	36.687	888.733	1.27513
3% NCL Composite	1.47621	15.807	144.659	0.46185

Figure 4.6: 3% EVA films and Composites

Figure 4.7: Behavior films and Composites

Because EVA is an elastomeric thermoplastic polymer with rubber-like softness and flexibility, these UTM results indicate stress fluctuation behavior.

The material is strong at low temperatures and resists stress cracking.

Relax oscillation is an intrinsic property of EVA.[37]

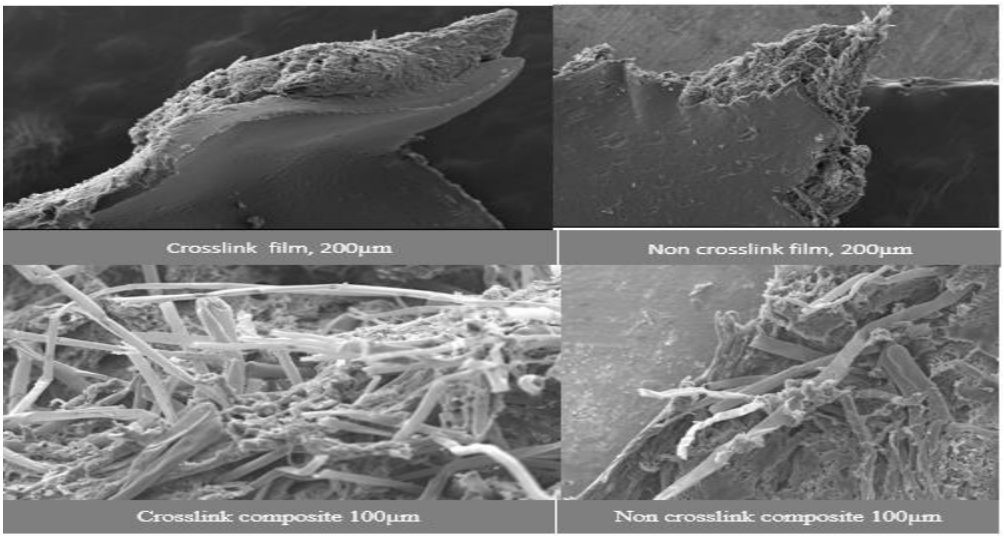


Figure 4.8: SEM results after fractured samples

- To check the behavior of fractured crosslink and non-crosslink composite.
- SEM results shows that the non-crosslink fibers break easily and completely fracturing occurred as compared to crosslink composite.

4.4 FTIR Spectroscopy Analysis

As seen in Fig. 1, kapok fibre solvents create intensity discrepancies in peaks. The -OH group stretching vibration is visible at peak 3342.32 cm-1.[38] The presence of cellulose is indicated by and.[39]. Asymmetric and symmetric aliphatic CH2 and CH3 stretching is indicated by peak 2916.26 cm-1. [40]. This indicates the presence of surface wax in general.

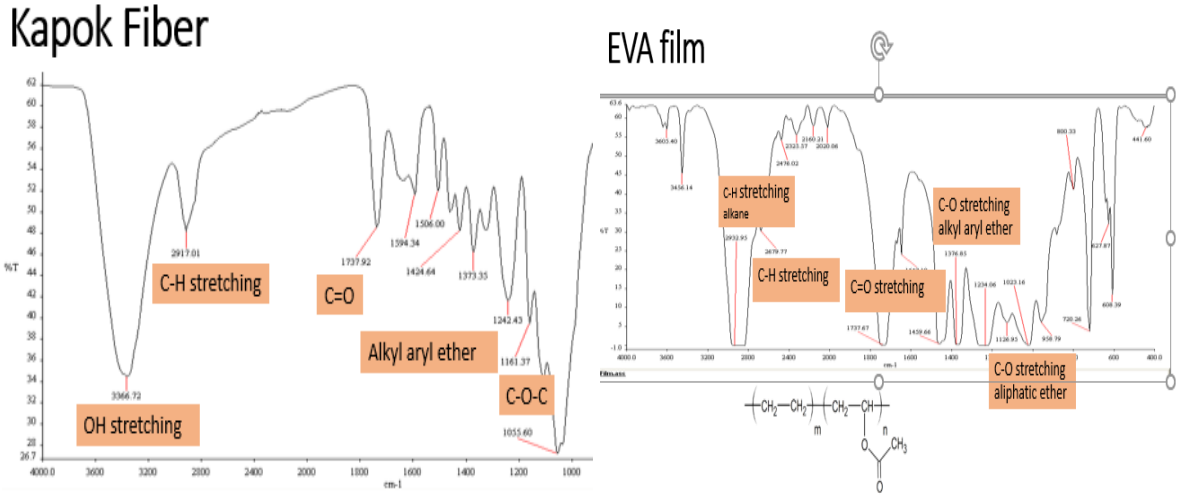


Figure 4.9: FTIR of EVA film and Kapok fiber[41]

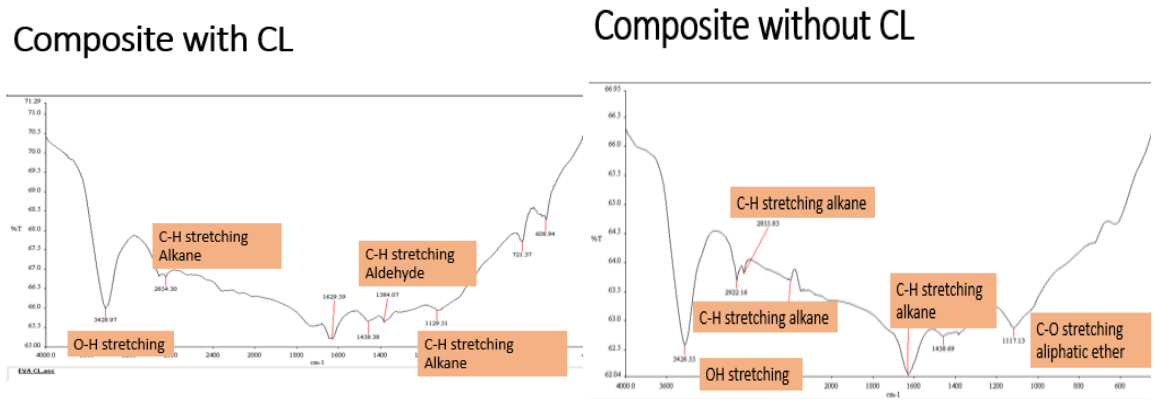
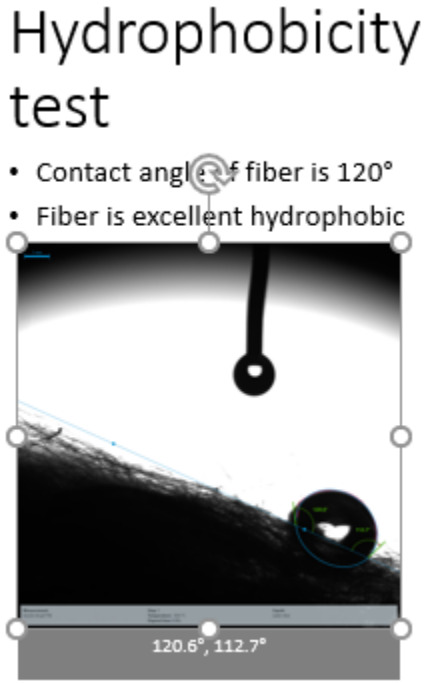


Figure 4.10: FTIR with and without crosslink of composite

4.5 Contact angle Analysis

When contact angle analysis was performed on the sample, it was analyzed that the crosslink composites has reduced the contact angle



Films and Composites	Contact Angle	Films and Composites	Contact Angle
2% EVA CL film	78.2°, 75.5°	3% EVA CL film	55.9°,53.5°
2% EVA NCL film	84.3°, 81.3°	3% EVA NCL film	61.2°, 58.3°
2% Cl Composite	95.6°, 93.9°	3% Cl Composite	84.4°, 80.8°
2% NCL Composite	96.2°, 95.9	3% NCL Composite	87.5°, 84.8°

Figure 4.11: Contact angle of films and composites

Fig 4.10 showed that the contact angle for the crosslink composites sample was 95°, 84 °, whereas the non-crosslink sample had a contact angle of 96°, 87.5°.

4.6 XRD Analysis

- To determine the degree of crystallinity in film and composite.
- Peaks show that the samples have semi crystalline behavior.
- content of the kapok fibers increased, indicating of the increase in crystallinity [42]

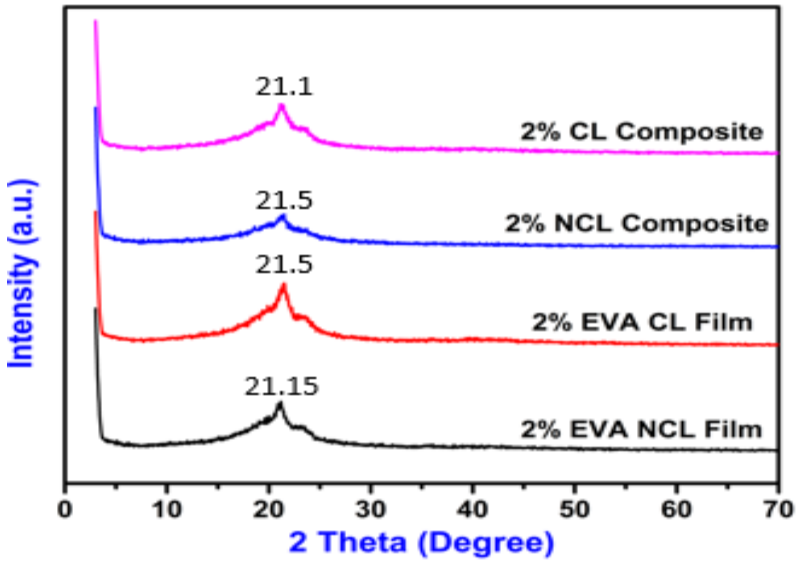


Figure 4.12: 2% Samples XRD

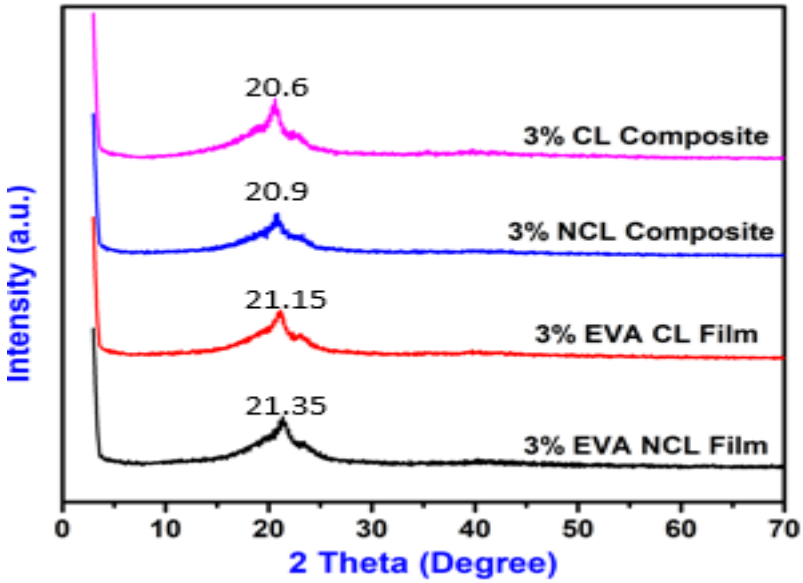


Figure 4.13: 3% samples XRD

Fig: 4.12 Peaks demonstrate a decrease in the content of kapok fibres, indicating a decrease in crystallinity.

4.7 Thermogravimetric (TGA) Analysis

To examine the decomposition temperature and thermal stability of materials.[42] these results showed that the 2% composite is more stable than 3% decomposition rate is slow as compared to 3% composite.

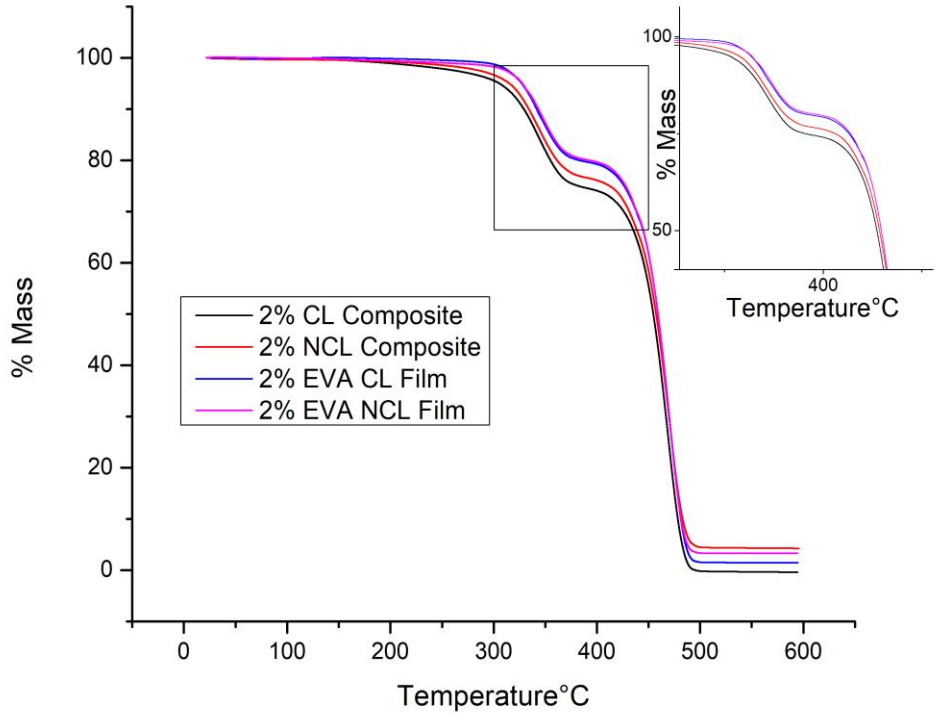


Figure 4.14 2% samples TGA

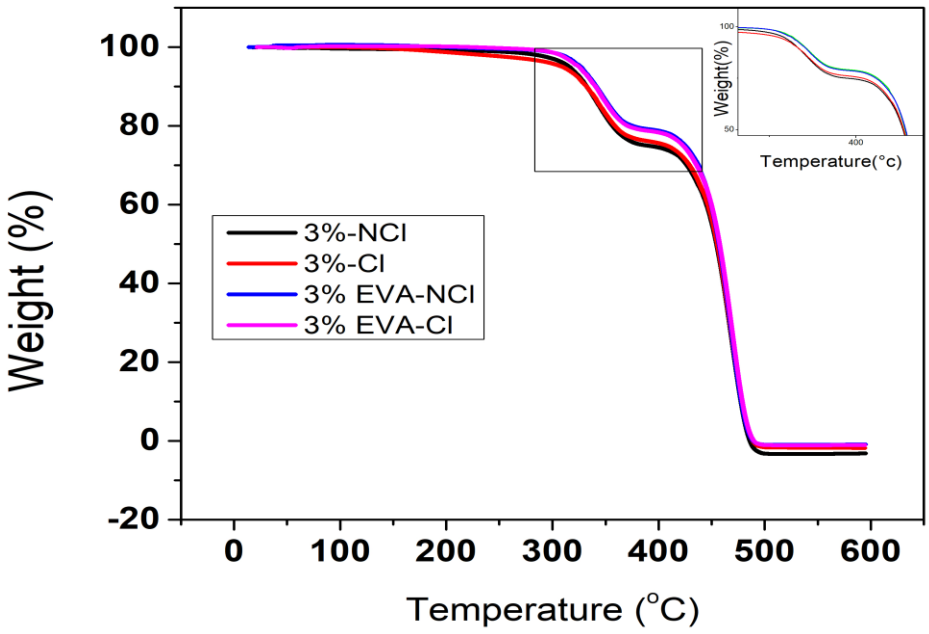


Figure 4.15 3% samples TGA

4.8 Hemolysis test

Composite can be used in any biomedical application it should have a hemocompatibility that was analyzed by two approaches: qualitative analysis and quantitative analysis using UV spectrophotometry.

4.8.1 Qualitative analysis

The color of the supernatant indicates the hemolytic and non-hemolytic nature of the films. From the qualitative analysis of the hemolysis test, The positive control (TritonX-100) resulted in 100% hemolysis, while the negative control resulted in no hemolysis.. The same was the case with our samples, they showed no color which means they are not hemolytic.

Figure 4.16: Qualitative analysis of hemolysis test

Figure 4.17: Positive control

4.8.2 Quantitative analysis

The results of the qualitative analysis were further confirmed by calculating their percentage hemolysis (%). UV spectrophotometry was used to calculate the percent hemolysis of

Composites and films all samples were found to be non-hemolytic since their percent hemolysis was in the range of the non-hemolytic materials defined by the ASTM F756 standard.

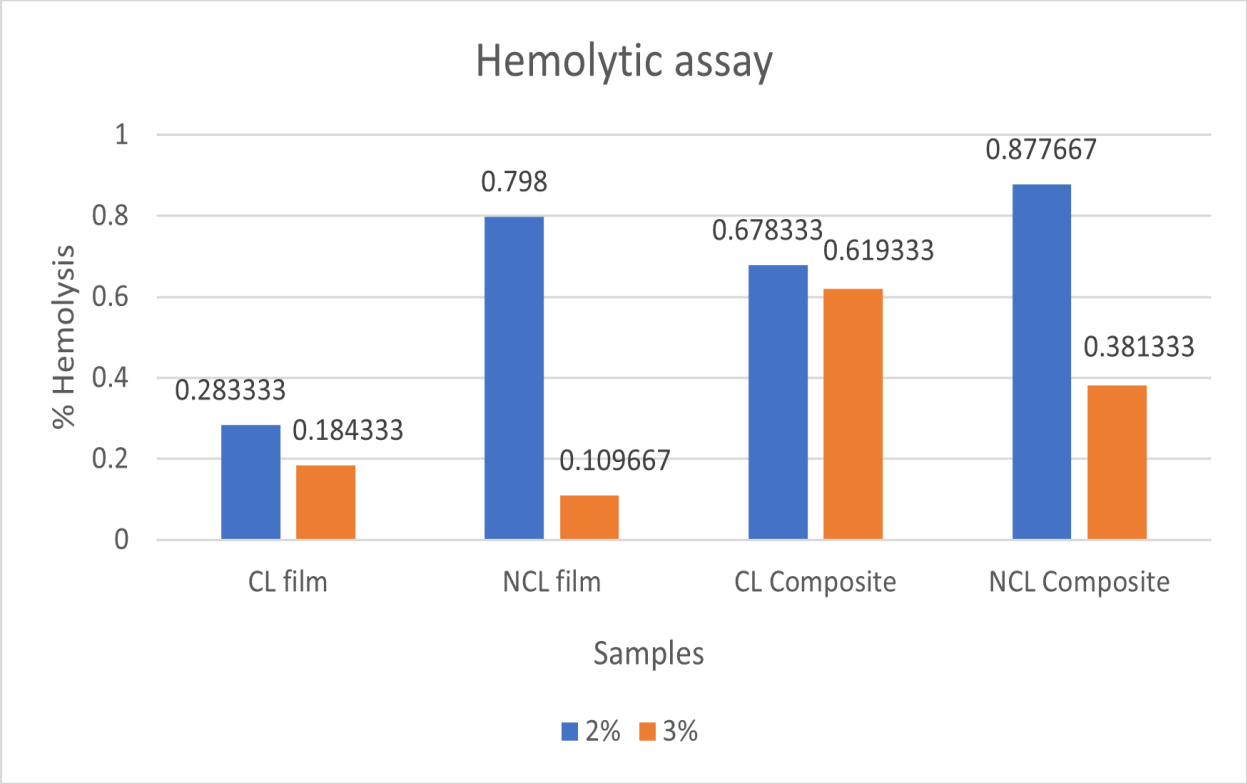


Figure 4.18: Hemolytic activity of 2%, 3% EVA films, and composite

Chapter 5 CONCLUSION

This research work aimed to increase the usability of kapok fiber by creating its composite with EVA. Based on the results, it was concluded that composite thus formed exhibits strong mechanical properties along with stress relaxation property inherited from EVA, increased hydrophobicity inherited from Kapok fiber --semi-crystalline nature, thermal stability till 200°C, non-hemolytic nature., Therefore, it can be used for biomedical applications.

Chapter 6 FUTURE PROSPECTS

- ✓ This research work can be carried out in future as well by considering the following suggestions.
- ✓ Evaluation of tensile and physical test of composite.
- ✓ Evaluation of mechanical strength of fibers reinforced composites
- ✓ Mechanical characterization of other natural fibers (e-g honeycomb) based structured reinforced laminates.
- ✓ Moisture's effect on natural fiber mechanical qualities
- ✓ Hydrophobic treatment of natural fibers before reinforcing into matrix
- ✓ Numerical simulation and analysis of results to compare with the experimental results.

Chapter 7 REFERENCES

1. ALiu, Y. and M. Wang, *Developing a composite material for bone tissue repair*. Current Applied Physics, 2007. **7**(5): p. 547-554.
2. Chen, L. and M. Wang, *Production and evaluation of biodegradable composites based on PHB–PHV copolymer.* Biomaterials, 2002. **23**(13): p. 2631-2639.
3. Fengel, D. and M. Przyklenk. *Studies on Kapok. 2. Chemical Investigation*. 1986.
4. Zheng, Y. and A. Wang, *Kapok fiber: applications*, in *Biomass and Bioenergy*. 2014, Springer. p. 251-266.
5. Chu, J., S. Liao, and H. Li, *Performance and application progress of kapok fiber. Tech.* Text, 2018. **36**: p. 6-10.
6. Wahi, R., et al., *Oil removal from aqueous state by natural fibrous sorbent: an overview.* Separation and Purification Technology, 2013. **113**: p. 51-63.
7. Hori, K., et al., *Excellent oil absorbent kapok [Ceiba pentandra (L.) Gaertn.] fiber: fiber structure, chemical characteristics, and application.* Journal of wood science, 2000. **46**(5): p. 401-404.
8. Kumar, A., S. Kumar, and R. Garg, *A review paper on stir casting of reinforced aluminum metal matrix composite*. International Journal of advance research in science and engineering, 2015. **4**: p. 62-74.
9. Kumar, D., *Mechanical characterization of treated bamboo natural fiber composite.* International Journal of Advanced Mechanical Engineering, 2014. **4**(5): p. 551-556.
10. Vinay, H.B., H. Govindaraju, and P. Banakar, *Experimental study on mechanical properties of polymer based hybrid composite.* Materials Today: Proceedings, 2017. **4**(10): p. 10904-10912.
11. Van Suchtelen, J., *Product properties: a new application of composite materials.* Phillips Research Reports, 1972. **27**: p. 28-37.
12. Prashanth, S., et al., *Fiber reinforced composites-a review.* J. Mater. Sci. Eng, 2017. **6**(03): p. 2-6.
13. Deve, H.E. and C. McCullough, *Continuous-fiber reinforced composites: A new generation.* Jom, 1995. **47**(7): p. 33-37.
14. Sood, M. and G. Dwivedi, *Effect of fiber treatment on flexural properties of natural fiber reinforced composites: A review.* Egyptian journal of petroleum, 2018. **27**(4): p. 775-783.
15. Fuqua, M.A., S. Huo, and C.A. Ulven, *Natural fiber reinforced composites.* Polymer Reviews, 2012. **52**(3): p. 259-320.
16. Mohd Nurazzi, N., et al., *A Review: Fibres, Polymer Matrices and Composites.* Pertanika Journal of Science & Technology, 2017. **25**(4).
17. Rubino, F., et al., *Marine application of fiber reinforced composites: a review.* Journal of Marine Science and Engineering, 2020. **8**(1): p. 26.
18. Al Rashid, A., et al., *Utilization of banana fiber-reinforced hybrid composites in the sports industry.* Materials, 2020. **13**(14): p. 3167.
19. Peças, P., et al., *Natural fibre composites and their applications: a review.* Journal of Composites Science, 2018. **2**(4): p. 66.

20. Khalid, M.Y., et al., *Developments in Chemical Treatments, Manufacturing Techniques and Potential Applications of Natural-Fibers-Based Biodegradable Composites.* Coatings, 2021. **11**(3): p. 293.
21. Elanchezhian, C., et al., *Review on mechanical properties of natural fiber composites.* Materials Today: Proceedings, 2018. **5**(1): p. 1785-1790.
22. Kessler, M.R., *Polymer matrix composites: A perspective for a special issue of polymer reviews.* Polymer Reviews, 2012. **52**(3): p. 229-233.
23. Lotfi, A., et al., *Natural fiber–reinforced composites: A review on material, manufacturing, and machinability.* Journal of Thermoplastic Composite Materials, 2021. **34**(2): p. 238-284.
24. Seydibeyoglu, M.O., A.K. Mohanty, and M. Misra, *Fiber technology for fiber-reinforced composites*. 2017: Woodhead Publishing.
25. Bledzki, A. and J. Gassan, *Composites reinforced with cellulose based fibres.* Progress in polymer science, 1999. **24**(2): p. 221-274.
26. Wang, J., Y. Zheng, and A. Wang, *Effect of kapok fiber treated with various solvents on oil absorbency.* Industrial crops and products, 2012. **40**: p. 178-184.
27. Liu, Y., et al., *Adsorption of methylene blue by kapok fiber treated by sodium chlorite optimized with response surface methodology.* Chemical Engineering Journal, 2012. **184**: p. 248-255.
28. Abdullah, M., A.U. Rahmah, and Z. Man, *Physicochemical and sorption characteristics of Malaysian Ceiba pentandra (L.) Gaertn. as a natural oil sorbent.* Journal of hazardous materials, 2010. **177**(1-3): p. 683-691.
29. Rengasamy, R., D. Das, and C.P. Karan, *Study of oil sorption behavior of filled and structured fiber assemblies made from polypropylene, kapok and milkweed fibers.* Journal of Hazardous Materials, 2011. **186**(1): p. 526-532.
30. Akl, M., H. Abd Elrazek, and E. Abdel Bary, *Poly (Ethylene-Co-Vinyl Acetate) Blends for Controlled Drug Release.* Am. J. Adv. Drug Deliv, 2018. **6**(1): p. 052-060.
31. Kong, I., K. Tshai, and M.E. Hoque, *Manufacturing of natural fibre-reinforced polymer composites by solvent casting method*, in *Manufacturing of natural Fibre Reinforced polymer composites*. 2015, Springer. p. 331-349.
32. Nguyen, C. and J. Kim, *Synthesis of a novel nitrogen-phosphorus flame retardant based on phosphoramidate and its application to PC, PBT, EVA, and ABS.* Macromolecular Research, 2008. **16**(7): p. 620-625.
33. NAWAN, N., *SEKUEN GEN 16S rRNA DAN PROFIL POTENSI ANTIBAKTERI PRODUK ISOLAT Streptomyces sp. GALUR ASIDOFILIK TANAH GAMBUT KALIMANTAN TENGAH TERHADAP Escherichia coli PENGHASIL ESBL*. 2019, Universitas Airlangga.
34. Mathew, A., S. Khandelwal, and N. Kaul, *Analysis of diurnal surface temperature variations for the assessment of surface urban heat island effect over Indian cities.* Energy and Buildings, 2018. **159**: p. 271-295.
35. Olatunji, O., et al. *Thermo-gravimetric characterization of biomass properties: A review*. in *IOP Conference Series: Materials Science and Engineering*. 2018. IOP Publishing.
36. Mwaikambo, L.Y. and M.P. Ansell, *Chemical modification of hemp, sisal, jute, and kapok fibers by alkalization.* Journal of applied polymer science, 2002. **84**(12): p. 2222-2234.
37. Liu, W., L. Zhang, and K. Mylvaganam, *Relaxation oscillation of borosilicate glasses in supercooled liquid region.* Scientific reports, 2017. **7**(1): p. 1-8.

38. Wang, J., et al., *Magnetically superhydrophobic kapok fiber for selective sorption and continuous separation of oil from water.* Chemical Engineering Research and Design, 2016. **115**: p. 122-130.
39. Liu, J. and F. Wang, *Influence of mercerization on micro-structure and properties of kapok blended yarns with different blending ratios.* Journal of Engineered Fibers and Fabrics, 2011. **6**(3): p. 155892501100600308.
40. Pakaya, F., H. Ardhyananta, and S.T. Wicaksono, *Mechanical Properties and Thermal Stability of Epoxy/RTV Silicone Rubber.* IPTEK The Journal for Technology and Science, 2017. **28**(1): p. 7-14.
41. Meszlényi, G. and G. Körtvélyessy, *Direct determination of vinyl acetate content of ethylene-vinyl acetate copolymers in thick films by infrared spectroscopy.* Polymer testing, 1999. **18**(7): p. 551-557.
42. Prachayawarakorn, J., et al., *Effect of jute and kapok fibers on properties of thermoplastic cassava starch composites.* Materials & Design, 2013. **47**: p. 309-315.