Experimental Investigation of Mechanical Performance of Hybrid Fiber Reinforced polymer Composites for Structural Application



A Thesis Submitted in Fulfillment of the Requirements for the award of

MSc. in Manufacturing System Engineering

By Samueal Tadesse

Under the guidance of Dr. Anil Kumar (Ass. prof.) and Mrs. Hanna B.

> Faculty of Mechanical Engineering Jimma Institute of Technology Jimma University Jimma, Ethiopia

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ABSTRACT

Natural fibers which are sourced from plant and animals will have a significant role in developing 'green' economy. Because it has attractive features of abundant availability, renewable resources, environmentally friendly, low cost, and biodegradability. Most of these natural fibers are discarded into landfill areas as wastes and removed by open burning that may lead to air pollution.

The present study focus on using natural fibers like sheep wool and Enset (Enset Ventricosum) fiber for manufacturing the hybrid fiber composite material (HFCM) for structural application. This HFCM was synthesized by hand lay-up technique with design consideration of principal parameters such as Fiber volume ratio (30%,50%, and 75%), ply arrangement (EWE, WEW) and fiber orientation (MMM, MUM, MRM, URU, RUR). Then mechanical tests, thermal and machining tests were conducted to characterize experimentally beside theoretical studies.

In the experimental section, the task was completed in four main stages. In the first stage, the physical properties of the fibers such as the crystallinity index and moisture absorption of HFCM were investigated. The result shows that the 5% Na OH treated fiber enhances the fibers crystallinity and cellulose fibers are more crystal than keratin-based fibers.

In the second stage, the thermal stability nature of the fiber was investigated. Almost 5-6% of weight loss was seen in the range of 105-280°c. It shows that, the stability of fiber and comparable results with others.

In the third stage, the mechanical performance investigation of the HFCM was conducted. Tensile, impact and flexural tests were performed. The results showed that increasing fiber content increases the strength of the composite except up to the saturation limit of 56.1% fiber volume ratio. The woven orientation of the fiber also shows better strength than the other.

HFCM with MMM fiber orientation, EWE ply arrangement and 56.1 % fiber volume ratio are the optimum model with 22 MPa, 7.12 MPa, and 28 KJ/m² tensile strength, flexural strength and impact strength respectively. Optimal custom response surface design experimentation with the quadratic model has been intended to model the tensile strength and flexural strength response.

Finally, the machinability of the HFCM was investigated. The result shows, cutting quality of the HFCM depends on fiber to matrix adhesive strength and the direction of cutting relative to fiber orientation. After all, the goal of this HFCM is to use as an alternative for structural material. This HFCM suggests being used for refrigerator body, car dashboard, and gas cylinder manufacturing industries.

Keywords: - HFCM, mechanical test, machining test, Enset Ventricosum, TGA, XRD



DECLARATION

This MSc. Thesis entitled as "Experimental Investigation of Mechanical Performance of Hybrid Fiber Reinforced Polymer Composites for Structural Application" is the result of my own effort and has not been previously included in a thesis, exposition or report submitted to this university or to any other institution for degree, diploma or other requirement, excluding where due acknowledgment and reference is made. It is submitted to the Faculty of Mechanical Engineering, Jimma Institute of Technology, in partial fulfillment of the requirement for the award of masters of sciences in manufacturing system engineering.

Submitted by

Samueal Tadesse			
candidate	Signature	Date	
Advisor			
Dr. Anil Kumar	D. Aml Kun	4/12/2019	
	Signature	Date	
Co- Advisor			
Mrs. Hanna B.			
	Signature	Date	
	<u>Approved by Board of Examiners</u>		
External Examiner			
	Signature	Date	
Internal Examiner			
	Signature	Date	
Chairman			
Mr. Srinivasa R. K.			
	Signature	Date	



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LIST OF ABBREVIATIONS AND SYMBOLS

A

ASTM : American society of testing materials

С

CM : Composite material CMC : Ceramic matrix composite

Ε

E : Enset fiber EWE : Enset -Wool- Enset

Η

HFCM : Hybrid fiber composite material

М

M : Mat or woven MMC : Metal matrix composite MMM : Mat-mat-mat MR : Modulus of rupture MRM : Mat-Random-Mat MUM : Mat-unidrectional-Mat

Р

PE : Polyethylene PEEK : Poly-ether-ether-ketone PLA : Polylactic acid PMC : Polymer matrix material PP : Polypropylene



PPS : Polyphenyle sulphide

R

RH : Relative humidity RPM : Revolution per minute RR : Random random RTM : Resin transfer molding RUR : Random -unidirectional-random

Т

TGA : Thermo- gravimetric analysis

U

UR : Unidirectional random URU : Unidirectional-random- unidirectional UTM : Universal tenisle testing machine

W

W : Wool fiber WEW : Wool- Enset-Wool

X

XRD : X-ray diffraction, X-ray diffraction, X-ray diffraction



CHAPTER 1 INTRODUCTION

The present study will focus on developing hybrid fiber composite material and conducting an experimental investigation to characterize its mechanical performances for structural application. In this chapter, stated a general introduction of the research with a brief background of the study. It also includes the objective of this paper, and the scope of the work.

1.1 Composite material, CM

The word composite has a raw meaning of 'put together'. It means that it is made up of a combination of two or more dissimilar materials by putting together. These materials will be reinforcing and matrix material. The reinforcing material may be fiber, particle, or layer. Its main functions are supporting the load and increasing the mechanical strength of CM, such as rigidity and flexural strength[1]. These matrix materials classified into polymer, ceramic or metal. Its functions are binding the reinforcement together and transfer stresses within reinforcements and fixing the material with its shape[2].



Figure 1.1 Composite material structure

1.1.1 Composite material classification

Composite materials are classified into a variety of groups based on the nature of constitutes (reinforcement and matrices). Based on matrices it will classify into polymer, metal or ceramic composite materials. Based on reinforcement it will classify into a particle, fibrous or structural composite materials [3]. CMs are also sub-classified into different classes based on reinforcements' origin. For instance, fiber-reinforced CMs will divide into natural fiber and synthetic fiber CM. Particle reinforced CMs classified into large particles, and dispersion strengthened CM. Structural composite divide into laminates and sandwich CM. Natural fiber also



classified into three classes according to their origin plant fibers, animal fibers, and mineral fibers [4]. Plants are also two types. These are primary and secondary depending on their consumption.



Figure 1.2 Classification of composite material [7,20].

Primary plants are those cultivated for the sake of its fibers while the secondary plants are plants in which its fibers are produced as a by-product. It is also classified into seed fiber, leaf fiber, bast fiber, core fiber, stalk fiber and so on based on parts of the plant that sourced for fiber. Animal fibers also categorized into animal hair, silk fiber, and avian fiber. Mineral fibers are fibers that derive from the particular treatment of minerals[5]. It also classified into asbestos, ceramic fibers, and metals fibers [6]. Detail of its classification presented above in the Figure.

1.1.2 Hybrid fiber composite material

Hybrid fiber composite material, HFCM is another kind of composite material. Most recent published journals and studies are focused on mono-fiber composite materials. In these mono-fiber composite materials, it uses a single class of fiber as reinforcement. Class means a collection of fibers, which have the same family to be originated. For instance, in a class of plant fiber, it uses



a variety of plant parts as a fiber source. Such as sisal, cotton, rice, and Enset will be listed. In the class of Animal fiber, it uses animals' part as a fiber source. Such as fiber-like human hair, silk, feathers, and sheep wool are grouped into the class of animal fiber [9].

Fiber hybridization is the main task of synthesize HFCM. Fiber hybridization is a process of fixing two or more class of fibers together with a matrix to form single composite materials. Its product is also called hybrid fiber composite material, HFCM. HFCM is a new opportunity for developing innovative material with improved features. Such as strength, stiffness, and others, which cannot be achieved by mono-fiber reinforced composites[10]. Now, very few studies are done in the natural fiber hybridization of HFCM.

In this study, to develop hybrid fiber composite material Enset and sheep wool was used as reinforcement and epoxy as binding material. Enset is an abundantly available, fibrous, multipurpose, and drought-tolerant indigenous plant in Ethiopia. Its scientific name is Enset Ventricosum. It covers almost 65% of the total crop production in the southern region of Ethiopia [11]. Its productivity is very high as compared to other crops and it is not affected by excess rain or drought. Enset fiber is obtained on Enset tree pseudostem and it is a by-product of traditional food called *kochho*[12]. This Enset fiber will collect from southeast of Ethiopia and extract manually by knife or other sharp edge tool and dry with the sun.





Figure 1.3 a) Enset tree b) Enset fiber

Another fiber type selected in this study will be Sheep wools from the class of animal fiber. Since it is abundantly available in Ethiopia especially in high land regions [13]. According to yensesw

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A. et al.[14], estimation Ethiopia has currently 23.6 million sheep. One sheep can produce averagely 2.3 up to 3.6 kg of raw wool annually[15]. Sheep wool is a by-product of sheep farming. Increasing the application area of sheep wool in added-value engineering material will contribute to the environmental, economical, and social sustainability of the societies [16]. Sheep wool is a fiber with attractive features of its softness, durability, can withstand wear and resist flames. For this study, the sheep wool is collected from DebreBerhan area, a high land region of Ethiopia. Its preparation will follow with sorting, cleaning, carding, spinning, weaving, and finishing processes.



Figure 1.4 Sheep wool

Generally, in this study, Enset fiber and sheep wool will be used as reinforcement and Epoxy as a matrix to develop HFCM. During synthesizing this HFCM, various controllable parameters will be considered. These parameters will be fiber orientation, fiber-volume ratio, and composite ply. After successfully developing HFCM, an experimental investigation will be conducted to characterize the mechanical performance of it. In order to find significant parameters that affect the design of the developed HFCM, optimization of the parameter will also have done. Finite element analysis will also perform to compare experimental results with respective numerical models.

1.2 Background of the study

The use of composite material was started with Egyptians and Mesopotamian carpenters. They have used a mixture of mud with grasses to create a strong and durable building, around 1500 B.C [17]. The modern era of composite starts with the invention of plastic in the early 19th century that replaced metallic and timbered materials. Later in 1935, high-performance fibers like glass fibers



were developed and binding with a plastic polymer to get strong and rigid materials. In the 1970s, the composite industry began to advance since improved resins and reinforcements were developed. It can be an alternative material for many applications like civil, transport, aerospace, medical, and sports industry[18].

For the last three decades, researchers and engineers are interested in doing extensive researches and developing innovative materials due to the steadily need for efficient and effective engineering materials. Composite materials, ceramics, and plastics can be listed as emerging materials. Its application and type also have grown steadily and conquered the market relentlessly. For instance, composite materials can use for both conventional and advanced application areas. For conventional applications, like consumer goods, furniture, housing structure, etc. Moreover, for advanced applications such as the medical industry, the automotive industry, aircraft, and aerospace structure will be listed [19].

For advanced applications, like aerospace and automotive industries, the uses of composite materials are not only decreasing the dead weight but also good for absorbing shock and vibration. It also creates a stable indoor environment for passengers. For common applications, like consumers' goods, furniture, and housing structure, the selected composite materials will be designed with consideration of its area of application and structural aspects. The need for lighter and shock-resistant structures leading a high emphasis on the use of composite material on civil works applications [20].

Composite materials are defined as bi-component in terms of its constituents such as the reinforcing phase and matrix phase or binding phase. However, it can achieve improved properties over their individual constitutes. Composite materials are classified based on the nature of constitutes. Based on the matrix or binding material, it will be polymer, metal or ceramic. Based on reinforcing material type composite may classify in to structural, particulate, and fibrous [21].

Polymer matrix materials (PMC) are a type of composite material it uses polymer matrix reinforced with natural or synthetic fibers. These polymers based matrix are classified as thermosetting or thermoplastics polymers. Thermosets polymers matrix has the qualities of creating a well-bonded three-dimensional molecular structure after the curing process. It can be decomposed instead of melting. Due to that thermoset matrix have a wide range of application in



the fiber composite. Low density, good corrosion resistance, low thermal conductivity, and lower electrical conductivity, and so on are listed as common merits in PMC[22]. Epoxy, polymer, and phenolic polyamide resins are a sample of thermoset matrixes. Epoxy resin has a wide range of industrial applications because of its high strength and mechanical adhesiveness characteristics [23].

Fibrous composites constituents fiber as reinforcement and polymers or others as matrices. Fibers in the composite are the load-carrying element and provide strength and rigidity. Polymer matrices in the composite maintain the alignment of the fibers (position and orientation). Fibers are very effective in discouraging the instant growth of cracks if it is normal to fiber orientation. Fibers are classified into natural fiber (plant fiber and animal fiber), and synthetic fibers such as glass fiber, carbon Nanofiber, Aramid so on based on its origin [24].

The concern of environmental sustainability and the demand for cost-effective material forcing engineers to develop and brought natural fiber-reinforced composite material to the market. Natural fibers also further divided into different classes such as plant, animal, and mineral-based on its source. Besides that natural fiber composite have many advantages like abundantly available, non-toxic, thermal and sound insulation, hydrophobic, corrosion-resistant, non-abrasive, renewable, strong and light-weight and cheap to process and manufacture it [25]. various sectors like automotive industries, furniture industries, construction field, sporting industries, packaging, and consumable goods are commonly listed current application of natural fiber reinforced composite [21].

1.3 Statement of the problem

Nowadays, the need for efficient and effective engineering material for a structural application is a vital issue for researchers.

Agricultural wastes are becoming an alarming global problem. These waste materials usually discarded into landfill areas and removed by open burning that may lead to environmental pollution. On the other hand, the rapid growth of the world's population demand will continue to rise in all kinds of sectors, but the world's resources remain limited.





Figure 1.5 Discarded sheep wool

The structure is the basic component for every static and dynamic physical system. It may be the load-bearing or non-load bearing. The need for economical, safe, lightweight, and durable material is a vital issue for the researchers to get an alternative solution for today's structural application. Nowadays, metals, woods, stones, ceramics, and plastics are mostly used materials for structural application. These materials have their tensile drawbacks. For instance, metals may expose to corrosion and expensive to manufacture. Woods are limited in resources and lead to deforestation and difficult to process. Plastics are petroleum-based innovative material but due to environmental polluting and depletion of the source; it is incompatible and not viable as an alternative material.

Synthetic fiber-reinforced composite materials are vastly used in today's composite material industries. But these synthetic fibers have serious limitations such as non- recyclable, non-renewable, non-biodegradable, healthiness risk and high energy consumption during manufacturing. Therefore, searching for a positive solution for the above-mentioned problems and an alternative material is the main goal of this study.

Natural fiber composite material can be used as an alternative and immediate solution for structural material in some areas of application. Natural fiber composite material offers a good opportunity to use an alternative to the above-listed materials due to its privileged inherent characteristics. Lightweight, neutral to emission CO_2 , abundantly available, biodegradable, reduce energy consumption, non-abrasive during machining, and non-irritation to the risk, are some of the inherent characteristics of natural fiber composite material.



Most of the recent good studies are giving emphasis on mono-fiber composite material. That means uses a single class of fibers as reinforcement to develop the composite material. Very few studies are done in hybrid fiber composite material. That has been developed with fiber hybridization. HFCM is a new opportunity to develop composite material with enhanced mechanical performance over mono-fiber composite material. Developing HFCMs with the attention of locally available, cheap and abundantly available natural fibers are the main objective of this study. Enset fiber and sheep wool as fiber reinforcement and epoxy as matrices will be used.

1.4 Objective

1.4.1 General objective

The main aim of this study will be focus on developing, characterizing and optimizing the hybrid natural fiber-reinforced polymer composite materials for structural application.

1.4.2 Specific objective

- Preparing the mold used to cast the composite
- To develop hybrid fiber composite material by using sheep wool and Enset fiber as reinforcement and polymer-based thermosetting matrices like Epoxy as binding material.
- Conducting an experimental investigation to characterize the mechanical performance of the developed HFCM.
- Determining the thermal stability of the fibers using thermo-gravimetric analysis (TGA) and crystallinity index using x-ray diffractograms (XRD).
- Optimizing parameters, which particularly affect the mechanical strength of HFCM; such as fiber orientation fiber volumetric ratio and ply arrangement.
- To investigate the effect of fiber hybridization in mechanical properties of composite material with respect to the mono-fiber composite
- Comparing the results obtained from the experimental result with a respective mathematical model used for modeling and predicting the response and validate the model.

1.5 Scope of the research

The scope of this research will include

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- Developing hybrid fiber composite material using sheep wool and Enset fibers as reinforcement and Epoxy as resin
- Conducting mechanical test like tensile test, flexural test, impact test, thermal test using, TGA, crystallinity test using, XRD
- Conducting Machining tests like drilling, and cutting operation
- Performing comparison on the mechanical performance of Hybrid fiber composite with its respective mono-fiber composite
- Statistical analysis will be done to model and predict the results that obtained from the experiment with a respective mathematical model

1.6 Thesis outline

In order to accomplish fully the objective of this thesis, the hole studies divide into the following five chapters.

In the *first chapter*, a general introduction to the composite material, background studies, problem of the statement, objectives, and outline of the thesis are presented in this section.

In the *second chapter* review of the previous work related to natural fiber-reinforced composite material, an overview of composite material, its constitute, classification, method of manufacturing and merits and demerits are presented.

In the *third chapter* methodology of the thesis with detail of experimental material and methods and laboratory testing used throughout the studies are presented in detail and literature review concerning to the test also presented.

In the *fourth chapter*, the result of the experimental studies and consequent analysis are presented. The physical investigation, mechanical investigation and machinability studies of the HFCM are recorded in this chapter. The response surface methodology (RSM) with optimum custom parametric optimization also presented in this chapter.

In the last *chapter five*, the summary of the result conclusions drawn from the above analysis and future recommendations are presented.



CHAPTER 2 LITERATURE REVIEW

2.1 Introduction

Composite material is one of the material types used in the everyday life of human beings. It is prepared with a physical combination of two or more materials together on which one of the materials is acts as reinforcement and the other is as a matrix or binding element [26].

Composite material is made with the proportional composition of reinforcements (fibers, particles, laminas) embedded in a matrix (polymer, metal or ceramics). The matrixes are responsible to form the desired shape and to transfer the load while the reinforcement is the stiff part, which is responsible for composites' mechanical strength [27].

Properties of composite material are strongly dependent on the properties of their constituent materials, their distributions and the interaction among them. Besides the nature of the constitutes, the geometry of the reinforcement (shape, size, and weight) and the volume concentration and geometrical orientation of reinforcement influence on strength of the composite largely [28]. Composite materials are classified into different groups. Based on its constitutes properties and nature. Shown in chapter one Figure 1.2.

2.2 Merits of composites

Some benefits of composite material over a conventional one

- Enhanced torsional stiffness and impact strength
- Better tensile strength
- ✤ Higher fatigue endurance limit (up to 60% of ultimate tensile strength)
- ✤ 30%- 40% lighter than aluminum alloy structure
- Lower embedded energy compared to other structural metallic materials like steel, aluminum, etc.
- ✤ Good damping effect, provide lower vibration transmission than metals
- More versatile and flexible during manufacturing to meet the design requirement
- Less life cycle cost than metals due to its long life offer and reduce maintenance
- Composite exhibits excellent fire retardancy with improved appearance.



Due to the shape flexibility of Composite parts can eliminate joints/ fasteners, which providing part simplification and integrated design compared to conventional metallic parts [10, 28, 29].

2.3 Constitutes of Composite material

Composite material is materials that differ from others are it is made from a macro-scale combination of the reinforcement and matrix material.

2.3.1 The reinforcement

Reinforcement is the discontinuous and stiff phase of composite constitutes which has the role of withstanding the mechanical loadings and rigged the composite material.

Based on its shape size or aspect ratio and structure it divides into three types these are fiber, particle or lamina.

- Particle reinforced composite
- Lamina or structure reinforced composite
- Fiber-reinforced composite

According to the reinforcement type, the fiber composite may be classified into the following types[30].

2.3.1.1 Particle reinforced composite

In the particle reinforced composite material, the discontinuous phase is found in the form of particle or dispersion form. From 0.01µm to 200µm particle size is used in particle reinforced composite material. Based on particle size it also categorized into two groups [31].

The particle can be found in different shapes like spherical, disk-shaped, rod-shaped and plateshaped. it has an aspect ratio close to unity. Particle reinforced composites have enhanced hardness and wear resistance but decreased tensile strength. Some of the commonly used particles include Aluminum oxide AL_2O_3 , Titanium carbide (TiC), and silicon carbide(SiC) [32].



a) Dispersion hardened composite material

In this, type of material, the size of the particle ranging from 0.01 μ m up to 0.14 μ m. These materials possess improved high-temperature strength and creep resistance [32].



Figure 2.1 Particle reinforced composite [32].

b) Particulate or flake composite

In this type of composite material, 1µm to 200-µm size particles are used as reinforcement.



Figure 2.2 Particulate or flake composite [32].

2.3.1.2 Laminar composite

It is composed of two or more layers or lamina and bind together by the matrix binder. It can be a sandwich or serious of a lamina. Laminate composite is composed of layers of material held together by matrix. Generally, these layers are arranged alternately for better bonding. These layer lamina may vary with fiber orientation. These laminates can have unidirectional, angle ply, crossply, and symmetric laminates [33]. A hybrid laminate composite material can also be fabricated by using a different class of component material.



Figure 2.3 Laminar composites [32].



The greatest emphasis of this study is on hybrid structural laminates composite material.

2.3.1.3 Fiber-reinforced composite

In fiber-reinforced composite material, the discontinuous or the reinforcement phase are found in the form of a thin thread or in a hairy structure. Fibers are in circular in cross-section with high specific strength and stiffness. Its length is many times greater than its diameter, having an aspect ratio greater than 100. It can be used as continuous or chopped fibers [34].



Figure 2.4 Fiber with a) Continuous longitudinal oriented fiber and b) Random oriented chopped fibers [32].

Due to its excellent structural performance, fiber-reinforced composite is gaining potential in tribological applications. The fiber in the composite material has a role of carries the major shared load. There are two types of fiber reinforcement namely; synthetic or man-made and natural fiber

a) Synthetic fibers

Synthetic fibers are man-made fibers such as glass fibers, carbon fibers, and aramid fibers. It can be used as reinforcement in polymer and metal-based high-performance composite material. Composite reinforced with synthetic fiber have good mechanical performance but it also possesses severe drawbacks like high cost, poor recyclability, an environmental pollutant, and non-biodegradability [35,39].

b) Natural fiber

Natural fibers are fibers that originated from a plant, animal or mineral sources naturally it can be used as reinforcement in composite material. Natural fibers based on its origin categorized into plant fibers, animal fibers, and mineral fibers.

In the last three decades, the use of natural fibers has been growing area of interest due to its important features like low specific weight, biodegradability, renewability and low production



cost. The main focus of this research is composite material with natural fiber reinforcement and bind with polymer matrix [36].

2.3.2 Matrix

Matrix is a substance that has the role of fixing the reinforcement together which has adhesive properties. Matrixes are the continuous phase of the composite material, which has the role of fixing reinforcements as its orientation and keeping the composite in its shape as designed [37, 38].

The role of the matrix in the composite material:

- Hold the fibers in place and isolated one another to prevent the fibers from the formation of surface flaws
- Transfer and distribute the load into the fibers and evenly distributive stress concentration
- Provides resistance to crack propagation and damage tolerance to the plastic flow at cracked tips
- ✤ Keep the reinforcing fibers in the proper orientation and position
- Protect the surface of the reinforcement from environmental effect and
- Protect the reinforcements from handling damage
- Withstand all of the interlaminar shear strength of the composite
- Determine the service temperature limitation of the composite [39–41].

2.4.2.1 Metal matrix

Light metals like Magnesium, aluminum, and titanium alloys are commonly used metals matrixes reinforced by ceramic fibers and particles. It used to improve the stiffness and strength of the material. high-temperature cobalt and nickel-based alloys are also reinforced with ceramic particles to create a class of MMCs called cermet. Ceramic fiber reinforced aluminum alloys have been developed for making propeller shafts used in an automobile. In aluminum alloy $383-SiO_2$ particle composites plasma electrolyte oxidation (PEO) method was used for the production of engine block cylinder liners. It minimizes the weight, increases wear resistance and production cost [42, 43].

However, metal matrix composite, its higher densities, and lower specific mechanical properties are the main design disadvantage when compared to polymer matrix composite [43].



2.4.2.2 Ceramic matrix

Ceramic has important features of high stiffness, hardness, compressive strength, and relatively low density. However, it is also brittle and low fracture toughness. Ceramic matrixes composites are designed to retain the ceramics important properties and compensating for their weakness like brittleness and poor fracture toughness in the material. the main reason to develop ceramic matrix composite is to increase the material's toughness [7].

Ceramic matrix composites (CMC) are mostly used in the high-temperature application and where the environmental attack is an issue. For instance, TiC fiber reinforced alumina (AL_2O_3) and silicon carbide, zinc and calcium phosphate are listed [44, 1].

2.4.2.3 Polymer matrix

Polymers are low density and good chemical resistant matrix that is suitable for aerospace applications especially in the fabrication of the fuselage and wings.

Advantages of polymer matrix

- Low density
- Low thermal conductivities
- Translucence
- Low electrical conductivities
- Good corrosion resistance
- Aesthetic color effects

Limitation

- Low transverse strength
- Poor stiffness
- Low operational temperature limit
- The poor mechanical properties of polymers are overcome by reinforcements with suitable particles, whiskers or fibers [3, 40, 45].

2.4 Natural fiber

Natural fiber can add many environmental benefits when compared with glass and other synthetic fibers. Natural fibers are green to the environment from their extraction up to disposal after usage.



Especially those natural fibers, which are agricultural by-product totally have no impact on the environment even during producing, extracting, purifying, processing and disposing of after use [46].

Plant and animal-based natural fiber have attracted researchers to use it as an alternative to synthetic fiber such as glass and carbon fibers. These natural fibers reinforced composite has received much more in the structural application. For instance, the interior part of the car such as door trim panels from natural fiber polypropylene, engine transmission cover, and dashboard in the automotive industry are the listed one. Based on its origin natural fibers are classified into three categories; these are plant fibers, animal fibers and mineral fibers [50].

2.4.1 Mineral fiber

Asbestos is one of mineral-based natural fiber which is used now mostly in the composite industry due to its important feature of thermal and sound insulation, friction properties, adsorption capacity, inflammability, and chemical inertness. In 2006, 2.3 million tons of asbestos were mined worldwide. Besides this, useful features it has many drawbacks especially with its silicate content related to human health and environment pollution. These mineral-based fibers are drawn by the electrospinning process and other modifications [49].

2.4.2 Animal fiber and their sources

Animal fibers like wool, silk, human hair and feathers contain proteins.

a) Silk fibers

Silk is collected from dried saliva of bugs or insects during the preparation of cocoons of the larvae. Example silk from silkworms [52,53].

b) Human hair

Human hair is a filamentous biomaterial that grows from follicles found in the dermis. it is made up of 95% protein, is called keratin [53].

c) Avian fiber or Feathers

Feathers are found in birds which formed in tiny follicles in the epidermis or outer skin layer, which contain keratin proteins [54].

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2.4.3 Plant fibers and their sources

Plant fibers are fibers extracted from vegetables and plants. It is a rigid and crystalline cellulose structure composed of cellulose, hemicellulose, lignin, waxes and some soluble compounds. Generally, the fiber contains 60-80% cellulose, 5-20% lignin and up to 20% moisture [55, 44, 56].

Bast fiber

It is extracted from the ribbon of the stem of the plant. Like jute fiber, Enset fiber.

Leaf fiber

Leaf fiber is scraped from the soft tissue of the plant by manually or mechanically. Such as sisal, banana. According to the ASTM D123-52 standard, banana fiber is classified as a leaf fiber of natural vegetable fiber.

Seed fiber

In this type of fiber, seed fibers are extracted from the seed of the plant. For instance, Hemp, Cotton, Flax, and Ramie are listed.

Fruit fibers

It is found from the husk of the fruit. Coir which originates from coconut fruit is the listed one

Stalk fiber

Stalk fibers are collected from the stalks of the plant and seeds. For example, rice, straws of wheat, grass, and bamboo.

2.5 Natural fiber-reinforced polymer composite

Natural fibers in composite material are used as reinforcement and can be bind with both thermosetting and thermoplastic polymer matrices. The matrix plays has a role in the strength of the composite material. It is liable to transfer the load between the stiff fibers through shear stresses at the interface region [55].

The polymer matrix or resins are of two basic types, these are thermoplastic and thermosets.



a) Thermoplastic

Thermoplastic matrices are found in solid-state and liquefy when heated above their melting temperature, and then re-solidify when it gets cooled below the re-crystallization temperature. Common thermoplastic materials polylactic acid (PLA), polypropylene (PP), polyethylene (PE), poly-ether-ether-ketone (PEEK), and polyphenylene sulfide (PPS) are listed [58,59].

b) Thermoset

Thermoset matrixes are found in very low viscosity liquid state due to its chemical bonding of molecules. In the thermoset matrix, the chemical bonding of low weight monomers and prepolymer forms it. Common thermoset matrixes include vinyl ester, epoxies, polyester and so on [58].

Thermoset matrices due to its branched molecular chain cannot re-melt after initial curing. Heating or UV light radiation, catalyst or pressure or combinations of both are used as the initial curing processes during composite molding and non-reversible reactions are held [45].

Thermoset resins have an important feature of high dimensional stability, high thermal stability, high creep properties, and chemical resistance. During composite fabrication, the liquid state resin is converted to a hard rigid solid by chemical cross-linking during a curing process. These curing processes include heating, UV light radiation, catalyst or pressure or combination of these [60, 61].

2.5.1 Comparison of thermoset and thermoplastic matrix

Thermoplastic composites are high recyclability, better toughness, high fracture resistance, low manufacturing time, infinite shelf life, high-temperature damage tolerance, ease of material handling, and large strain to failure [61].

Both thermoset and thermoplastic matrices are different in their chemical composition and molecular structure. Due to that, the fabrication of polymer composite material with this thermoset or thermoplastics matrixes are followed a different procedure. For instance, for thermoplastic matrix composites use a combination of heating, pressing and cooling procedures [62].





The manufacturing procedure of composites with polymer is presented below.



In this study, the thermoset polymer matrix is used as a binding material. That is called Epoxy resin. Epoxy resins are relatively low molecular weight pre-polymers that can be processed under a variety of conditions. The aim of the epoxy matrix is to fix the fiber as it is and transfer the loads to the fibers [63].

The advantages of epoxy resins are it can be partially cured and stored in that state, low polymerization shrinkages during cure, good mechanical strength, excellent resistance to chemicals and solvents and excellent adhesion to fibers.

System 2000 epoxy with 2020 hardener was used in this study which chemically belongs to the epoxide family. The two ring groups at the epoxy molecule center help it to absorb both mechanical and thermal stresses better than linear groups. It helps the epoxy resin to have a feature of very good stiffness, toughness, and heat resistance [52]. Some of the disadvantages of epoxy resin are it needs long curing time and poor mold release characteristics due to its high adhesive characteristics.

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Properties	Polyester resin	Epoxy resin	Vinyl ester resin
Density (g/cm3)	1.2-1.5	1.1-1.4	1.2-1.4
Young's modulus (GPa)	2-4.5	3-6	3.1-3.8
Tensile strength (MPa)	40-90	35-100	69-83
Compressive strength(MPa)	90-250	100-200	
Tensile elongation to break (%)	2	1-6	4-7
Water absorption 24hr at 20 C	0.1-0.3	0.1-0.4	
Cure shrinkage (%)	4-8	1-2	

Table 2.1 Typical thermoset polymer matrix and their Properties[52,62].

Thermosetting polymer resins in which the resin molecule contains one or more epoxide groups are known as Epoxies. Two main types of epoxies, Glycidyl epoxy and Non-Glycidyl are available. Glycidyl epoxy contains Glycidyl amine, Glycidyl ester or Glycidyl ether. Non-Glycidyl epoxy resins are either Cycloaliphatic or Aliphatic resins[59].



Figure 2.6 Typical resin stress/ strain curves redrawn from [62]



	Polvester	Vinvl ester	Enory ester resin
	1 olyester	r ingi ester	
4.1.		X 7 1'1 1 '1	YY' 1 1 1 1 1 1 1 .'
Advantage	Easy to use the	Very high chemical	High mechanical and thermal properties
	lowest cost of	resistance, higher	High water resistance
	resins available	mechanical properties	Long working times available
		than polyester	Temperature resistance can be up to
			140°c (wet)/220°c (dry)
			Low cure shrinkage
Disadvant	Only moderate	Posture generally	More expensive than vinyl esters
age	mechanical	required for high	Critical mixing
	strength, high	properties	Corrosive handling
	cure shrinkage,	High styrene content	
	limited range of	Higher cost than	
	working times	polyester	
		High shrinkage	

Table 2.2 Advantge and disadvantage of different resins[66].

The selection criteria of the matrices are depending mainly on the composite end-use requirement. For instance, thermoplastic matrices are chosen when a composite material with high damage tolerance and recyclables is needed. whereas thermoset matrices are preferred If the composite needs to be good chemical resistance and work with elevated temperature.

The setting reaction of the epoxy resin occurs in four stages

- Pot life stage: in this stage, the resin remains in a workable liquid form
- Gel stage: the time to resin starts to be a soft gel. a liquid epoxy starts to exhibit pseudoelastic properties
- Hardening stage: the resin starts to become hard, during this stage the part can be removed from its mold
- Maturing stage: it is the further stage, the resin gains harness, and complete stability. the composite has maximum strength, hardness, and stability in this stage[9,66].





Figure 2.7 Processing stages of epoxy resin[9].

Important features of Epoxy [67]

- Lightweight
- Resists most alkalis and acids
- Low moisture absorption
- Non- staining
- Resists stress cracking
- Retains stiffness and flexibility
- Easily fabricated

2.5.2 Mechanical performance of natural fiber composite

During developing natural fiber, reinforced composite material the main consideration is its mechanical performance. It depends on many factors. Such as

- Fiber type and its strength
- Fiber length
- ✤ Fiber orientation in the composite
- The amount of fiber in the composite (fiber weight fraction)
- The thickness of the laminate
- ✤ Fiber matrix interfacial bond strength (the interface) and so on [57,66]

In addition to these, processing conditions, fiber extraction methods, fiber's origin in the plant part have also an undeniable impact on the strength of the fiber-reinforced composite. Several factors that significant in selecting suitable natural fiber for use in composites. Physical factors such as fiber dimension, structure, crystallinity, variability, and so on. Mechanical properties like fiber


strength, bonding with matrix, environmental factors such as availability, cost and production are considered [68]. Enset and sheep wools are chosen for this research because it is abundantly available in Ethiopia and a new fiber which does not get researchers focus.

The matrix that used to adhere to fibers plays a vital role in the mechanical strength of the composite. Its main role is transferring the load to the stiff fibers through shear stresses at the interface.

2.5.3 Production of natural fibers

Natural fibers such as Jute, pineapple, banana, and coir are mostly used in today's composite material industry. Some studies stated that natural fibers like flax, jute, hemp, and sisal have good strength and comparable with glass fiber in specific strength and modulus. S. Marathe. [69] and Srinivas et al. [34] Have studied the physical and mechanical properties of banana fiber and report that banana fiber has better flexural strength and tensile modulus.

Flax	Borneo
Hemp	Yugoslavia, china
Sun hemp	Nigeria, Sierra Leone, India
Jute	India, Egypt, Ghana, Malawi, Sudan
Kenaf	Iraq, Tanzania, Jamaica, South Africa, Cuba, Togo
Roselle	Borneo, Malaysia, Sriylanka, Togo
Sisal	East Africa, Bahamas ,Tanzania, India
	Malaysia, Uganda, Philippines, Bolivia
Coir	India, Lanka, Philippines, Malaysia

Table 2	2.3 Some	natural	fibers	and	their	abundant	origin	69
								_

2.6 Wool

Wool is made up of a basic unit of a protein called keratin. It has three essential constituents such as cuticle, cortex, and medulla. The cuticle is the outer layer. The cortex is the inner structure made up of millions of cigar-shaped cortical cells. Wool has usually from 2-3.5 centimeters length depending upon the breed of the sheep[70].

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Wool has non-sticky characteristics due to that it shows good elongation and elastic recovery properties. it is more hygroscopic than any other fiber. Wool such as sheep wool, goat hair, alpaca hair, horsehair, etc. has several qualities such as elastic, grows in staples, and crimped.

2.6.1 Important features of wool [44,70, 71]

- ✤ Hygroscopic
- Good elasticity
- Good resilience
- Retain its shape fairly well during normal use
- Fairly resistant to bacteria
- Good thermal conductivity
- Moisture absorption is less as compared to silk fibers
- Slight sputtering when a flame is presented

Disadvantage

Poor dimensional stability

Wool fibers have a low tenacity with excellent elongation and elastic recovery. During burning wool fiber burns slowly with slight sputtering.

Fiber	Density	Service	Melting	Tensile	Elongation,	Young's
	Kg/m3	temperature	temperature	strength,	%	modulus
		(°C)	(°C)	(MPa)		(MPa)
Wool	1.3	100-400	570	125-200	20-40	

Table 2.4 Physio -mechanical properties of wool[44]

2.7 Enset fiber

It contains no woody material and can grow from 2.0 m to 7m. The stalk, which ranges in diameter from 300 mm to 800mm. it consists of layers of the overlapping leafstalk. At the end of each stalk is a dark greed oblong leaf, measuring about 600mm to 2300 mm. Enset fibers are obtained from the pseudostem of the Enset tree. It obtains by manual or mechanical scraping [11]. According to the American society of testing materials (ASTM) standard D123-52, banana fibers are classified

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as leaf fiber of plant fiber even though they are extracted from pseudostem. Enset plant and banana fibers are found in the same species their difference is due to its size and the Enset plant has no fruit sometimes it is also called false banana. Whereas banana plant has fruit is called banana [72].

Table 2.5 Comparisons o	f natural fiber and	glass fiber towards	environment impact[41]
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Environmental impact	Glass fiber	Natural fiber (reed fiber)
Non-renewable energy requirements (MJ/Kg)	44.7	3.64
Energy use (MJ/Kg)	48.33	3.64
CO_2 emissions (g/Kg)	2.04	0.66
CO emission (g/Kg)	0.80	0.44
SOx emission (g/Kg)	8.79	1.33
NOx emission (g/Kg)	2.93	1.07
BOD to water(mg/Kg)	1.75	0.36
COD to water (mg/kg)	18.81	2.27
Phosphate to water (mg/kg)	43.06	233.6
CML-human toxicity (Kg 1.4 dichl)	21.2	9.04
CML- terrestrial eco- toxicity (Kg 1.4 dichl)	5250	4480
CML- greenhouse effect (Kg CO_2)	75.3	4.04

2.8. Interface region of fiber and matrix

The interface region of fiber and matrix is the boding surface or region where a discontinuity occurs, whether physical, mechanical or chemical. The applied load effectively transferred from the matrix to the fiber is via this interface region. in this region, the loads are transmitted between the reinforcement and the matrix [26, 38].

The interface region is the main factor to effects the strength of composite material. Since fiber to matrix bonding and load transferring capability between fibers in the composite are directly correlated to fiber to matrix interfacial bonding. Due to that for the better mechanical strength of the composite, a strong fiber-matrix bonding is important. This matrix/ fiber interface plays a role of transferring the stress and increasing the fracture energy. This interfacial bonding strength of the composite is mainly dependent on surface adhesion of fiber to the matrix. This surface adhesion



of fiber is affected by surface impurities for the formation of a weak interface between the fiber and the matrix.

Natural fibers are hydrophilic in nature due to its hydrogen bonding. as a result, the fiber-matrix interfacial strength is reduced and its mechanical performance is also reduced. In order to increase the interface adhesion between the fiber and matrix, fiber surface cleaning and chemical treatment are done [37].

2.8.1 Chemical fiber surface treatment

Despite the important features of natural fiber, it has also drawbacks such as higher moisture absorption, lower fire resistance, lower mechanical properties, durability, quality variability of the fiber and difficulties during the manufacturing process [73].

Using natural fiber in reinforcing polymer is not a challenge on its own. However, the major challenges are poor interfacial adhesion and poor compatibility between the hydrophilic fiber and the hydrophobic matrix causing fiber swelling within the matrix. In order to improve these weaknesses of natural fiber, it passes through several chemical treatments.

These fiber surface treatments may include both physical and chemical treatment techniques in order to achieve one or more of the following objectives [46,73].

- Remove surface impurities and undesirable fiber constituents (such as oils, waxes, pectin)
- Roughen of the fiber surface topography
- Change the surface chemistry of the fiber
- Separation of individual fibers from their fiber bundles
- Chemical modification of the cellulose fiber surface
- Reducing the hydrophilicity of the fibers

For instance, Chemical like sodium hydroxide treatment is done in order to decrease the hydroxyl group in the fibers. It is responsible for the rate of moisture absorption in natural fiber composite. Fibers' chemical treatments are grouped into fiber pretreatment, coupling agent or dispersing agent.

The most known chemical treatments include the use of alkalis and acid hydrolysis in order to improve the adhesion between the fiber surface and the matrix by modifying the fiber surface and the fiber strength [74].



2.9.1.1 Alkali treatments

Alkaline treatment or mercerization is the process of plunging the fiber into aqueous alkaline solution with water. The importance of this treatment is used to disrupt the hydrogen bonding in the network structure, which increases the surface roughness of the fibers, which results with better mechanical interlocking of fiber and matrix [75].

These alkaline treatments especially the use of NaOH solution to modify the surface of the fiber in the composite has been extensively studied. H. Kim et al, Donath et al, Gassan et al.[41-44] Studies, this NaOH plays a critical role in removing hemicellulose and artificial impurities by means of alkaline cleavage and pectin also removed in this solution. It results in exposed cellulose of the fiber, to the possible reaction in the sites. This aqueous sodium hydroxide (NaOH) treatment of natural fibers endorses the ionization of the hydroxyl group to the alkoxide from fiber surface [38].

Fiber – OH + NaOH fiber
$$O^-Na^+ + H_2O$$

Many scholars report that Fibers, which immersed in 5% NaOH and cleaned with distilled water, are used to remove amorphous compounds and increased the crystallinity index of cellulose fiber. They recorded that due to crystallinity increasing the tensile strength of the fiber also better than the untreated one.

NaOH alkali treatment has been a simple and economically viable tool to modify fibers' surface chemistry[76]. NaOH treatments are selected for this study in order to enhance the surface morphology of the Enset fiber and sheep wool that lead to better bonding [46].

M Mazumder, [102] Conduct a study on the effect of NaOH treatment of fibers on the mechanical strength of the composite. The result showed that composites that are reinforced with treated fiber had better mechanical performance than the untreated one. This may due to that; this chemical treatment enhances the adhesive characteristics of the fiber surface.

2.9 Manufacturing technique of composite material

Composite material manufacturing process is a goal-oriented process. The goal includes End-use, production volume, size of the product, economic targets, labor intensity, material involved, required skills, surface complexity and appearance, production rate, tools, and required equipment are the factors that are considered while choosing a manufacturing process to produce a composite



with desired specification [26]. For each type of application area, it will have done with a different procedure. It depends on the availability of technology, facilities, and personnel skills. It commonly produced in hand lay-up technique, press molding, injection molding, and extrusion.

There are many fabrication techniques in manufacturing natural fiber reinforced thermoset composites. These are:

- Hand lay-up technique
- Resin transfer molding (RTM)
- Compression molding
- Injection molding

2.9.1 Hand lay-up technique

The hand lay-up technique is the most widely used and simplest fabrication process for composite material. It is a process of placing the fibers in the mold manually and poring the resin followed by hand brush or rollers smearing [45].

Four essential procedures in hand lay-up techniques

- 1. Mold preparation
- 2. Gel coating
- 3. Lay –up and
- 4. Curing

In otherwise, hand lay-up techniques are called wet lay-up techniques. However, in this technique high soluble resin is sprayed, poured or brushed into the mold then the reinforcement is wet with resin. After that, the wet reinforcement is placed in the mold, rolled or squeezed to distribute the resin and compact it, are followed respectively. However, additional fiber loading is not possible.

These hand lay-up techniques are suited for one-offs or short production runs and used for large components such as mainly in the application of marine and aerospace structures. Easy to operate, low cost, versatile, efficient manufacturing method with no size limitations, and produces a high gloss finish on one surface are the listed advantages of hand lay-up techniques. On the other hand, the quality of the product is very much subjected to operator skill and only one finished surface is the principal limitation of hand lay-up techniques. It is labor-intensive technique [26, 46].



2.10 Merits of Natural fiber in composite material

- Acceptable mechanical performance such as higher specific strength with a less specific weight
- High tilling level
- ✤ Favorable processing properties, for instance, low wear on tools
- Low energy consumption
- Options for new production technologies and material
- Highly acceptable specific strength properties
- ✤ Favorable accident performance, high stability, less splintering, weight saving
- ✤ Favorable Eco balance for part production with less investment cost
- Biodegradability
- Occupational health benefits compared to glass fibers during production
- Reduced fogging behavior
- ✤ Abundance availability with a renewable source
- ✤ Good thermal and acoustic insulating properties [37,77,78].

2.11 Application of natural fiber composites

Building and construction industry; panels for partition and false ceiling, partition board, floor, window and door frames [37,79,80].

- Low-cost housing
- Emergency shelter material
- ✤ Furniture; chair, table, bath unit
- Aerospace industries (wing and rotor assembly of helicopters)
- ✤ Storage device; boxes, biogas containers, etc.
- Sports equipment (handles of rackets, helmets in bicycle)
- Marine structures (ships bodies, swimming pool, boats)
- ✤ Automotive industry automobile and railway coach interior, boat, etc.
- ✤ Toys



2.12 Current trend of natural fiber composite

Nowadays, natural fiber-reinforced composites are an interesting area for both academic and industrial sectors due to its attractive features. its demand is increasing at 15-20% annually in an automotive application, and 50% or more in the construction area [69].



Figure 2.8 The use of natural fiber-reinforced composite used in industries in 2002[69].

2.13 Mechanical and thermal characterization

In this work, in Mechanical characterization tensile strength, flexural strength and impact strength of the HFCM were examined. In thermal characterization, the thermal stability of the fibers was investigated.

2.13.1 Tensile test

Tensile test is conducted in order to investigate the tensile strength of the composite material. Material's tensile strength is defined by measuring the force that pulls a specimen to the point where it will break. The maximum tensile stress of the material is defined before the specimen gets failure or breaking that states the tensile strength of the material.





Figure 2.9 Tensile test diagram [62].

2.13.2 Flexural test (Bending test)

The load is applied in the mid of specimens that are simple supported at both ends to produce curvature of the sandwich facing planes. Flexural stiffness, core shear strength, and shear modulus or the facings compressive and tensile strengths of sandwich construction can be evaluated [79]. Flexural stress values were determined by using an equation

$$\sigma_f = \frac{3FL}{2bh^2} \tag{2.1}$$

Where σ_f flexural stress, F is the load, L is the span length, h is the thickness of the specimen and b is the width of the specimen.

The flexural modulus was calculated by the equation of

$$E_f = \frac{L^3}{4bh^3} \left(\frac{\Delta F}{\Delta S}\right) \tag{2.2}$$

Where

E_f is the flexural modulus of elasticity

 ΔS is the difference in deflection

 ΔF is the difference in load



2.13.3 Impact test (drop weight test)

This test is used to determine the damage resistance of composites facing a drop-weight impact event. The impact test is used to measure the materials' capability to resist the fracture under highspeed stress or impact load. It is related to the toughness of the material under high strain rate deformation.

2.13.4 X-ray Diffraction (XRD) studies

XRD experiments were conducted to investigate the crystalline nature of the fiber. The higher the crystalline, the better mechanical strength. The X-ray diffractograms show that the intensity of the crystallographic plane in the fiber[19].

The fiber crystallinity index (I_c) of the fiber calculated by using an equation

$$I_c = \frac{I_K - I_{Am}}{I_K} \tag{2.3}$$

Where I_c is the fibers crystallinity index, I_k is the maximum intensity or lattice reflection peak of diffraction and I_{Am} is the minimum peak position or the intensity of diffraction of the amorphous fraction, which is taken at a 2 theta angle.

2.13.5 TGA (Thermogravimetric Analysis) studies

Thermogravimetric analysis (TGA) is used for determining the thermal stability and fraction of volatile components in a material. It is by measuring a material's weight loss as a function of temperature when a material is heated.

2.14 Hybrid natural fiber reinforced polymer composite

When two or more types of reinforcements are combined in a single matrix, it is called a hybrid composite. Fiber hybridization is a new opportunity for tailoring the composite material with new properties. One of the objectives of this study is to develop and investigate the hybrid composites containing sheep wool and Enset fibers [1].

Several studies reported that hybrid fiber composite has enhanced thermal and mechanical performance. For instance, Thiruchitrabalm et al. [53] Conduct an investigation on banana/ Kenaf hybrid composites and report that chemical treatment had provided better mechanical properties, for both the random mix and woven hybrid composites. Ekhlas abudo, [76] conduct studies on



Kenaf and recycled jute fibers hybrid composite and report that tensile and flexural strength of randomly oriented Kenaf composites were dependent on the fiber treatment, fiber length, and fiber weight fraction. The maximum tensile strength was around 24.8 MPa for fiber length of 30 mm.

kuan et al. [112] investigate hybrid composite, the result showed that increasing the volume fraction of the matrix could enhance the impact strength of the hybrid composites. According to the arrangements of fibers and layers, hybrid fiber composites are classified into the following groups.

Intermingled

Different fiber materials are mixed together and passed through a matrix simultaneously.



Figure 2.10 Models for intermingled HFCM [80]

Interlaminated

Each separate laminate containing just one type of fiber. The laminae are bonded together in a matrix.



Figure 2.11 Interlaminated HFCM [80]

Interwoven

Composed of fabric reinforcement where each fabric contains more than one type of fiber.



a)

Figure 2.12 a) Plan weave or (Mat) orientation b) Interwoven HFCM [38, 80]



Natural fibers composites are used in Load-bearing and less dimensional stability structural applications. For example, flax reinforced polyolefin is extensively used in the automotive industry. Plant fibers like Enset, Banana, flax, and sisal have a large potential as reinforcement in structural material due to its high aspect ratio and high specific strength and stiffness.

Now a day, natural fibers composites are found in every day-to-day life. For instance, jute fibers with polyester resin are used in buildings, elevators, pipes, and panels in India[80].

Natural fiber composites are very cost-effective material in different application areas. It can be used as a partition wall, ceiling, window and door frames in construction and building industries. It can be used in biogas containers and storage devices in the storage and packaging industries. It can be used to manufacture tables and chairs in furniture industries. It can also use in electronic industries to use as an outer casting and case. In the automobile and railway industries, it also used to make coach interior parts, and dashboards [81].

2.15 Research and related works

M. Ramesh T. et al. [93] studies on Processing and mechanical property evaluation of banana fiber reinforced polymer composites. Composite material with banana fibers was developed by using the hand lay-up process with 40 %, 50 % and 60 % fiber volume ratio and reaming epoxy. Tensile, flexural and impact tests were conducted to investigate mechanical properties. SEM analysis is carried out to evaluate the fiber-matrix interface and analyze the structure of the fractured surface. The maximum tensile strength, flexural strength, and impact strength are 26 MPa, 6 .2 MPa, and 8 joules respectively with 50 % banana fiber and 50 % epoxy resin composite. They suggest that banana fiber-reinforced composite with a 50 % fiber volume ratio can be used as an alternative for conventional fiber-reinforced polymer composites.

R. Gopinath et al. [6] studies on Characterization and structural performance of hybrid fiberreinforced composite deck panels. Glass and jute hybrid composite material with a vinyl ester matrix were developed. Experimental and Finite element evaluation of the flexural performance of the composite deck was carried out. A hybrid deck made using glass and jute fiber with woven fiber orientation was found that it can be used for practical engineering applications.

Tyagi S. et al. [39] Experimental and numerical analysis of tensile strength of unidirectional glass/ epoxy composite laminates. Unidirectional glass/ epoxy composite laminates were used to develop



the composite with volume fraction percentage of 40 %, 50%, 60%, 70 % and 80 % were prepared using hand lay-up manufacturing technique. The tensile modulus of the specimen with a fiber ratio of 80 % fraction has less strength with 651.6 MPa. Tensile strength increases with the increased volume fraction of glass fiber in glass/ epoxy laminates. The contribution of epoxy resin in tensile strength is negligible in comparison to the glass fiber.

K. Ram et al. [7] Polymer composite for industrial safety helmets. Replacing existing industrial safety helmet made from acrylonitrile butadiene styrene (ABS), with Banana, sisal and jute hybrid composite was the main aim of their work. The drop test was carried out to measure the impact strength of the developed composite. The observed impact strength was 9.95 J/mm2. It is comparable strength with the existing helmet made from plastic. The authors concluded that the composite material with hybrid polymers have good potential to replace ABS in safety helmets.

Jauhar Fajrin. [14] works on Mechanical properties of natural fiber composite made from Indonesian grown sisal. Sisal fiber with epoxy was used to fabricate using a vacuum bagging process. Unidirectional and randomly oriented sisal fiber was used as a design parameter. Tensile, flexural and compressive properties of the composite were studied. The result shows that the tensile, flexural and compressive stress of the composite 22.52 MPa, 8 MPa, and 12 Mpa respectively. It shows the mechanical properties of such composite was comparable with some of the previous reported studies. The authors also show unidirectionally oriented sisal fiber has better mechanical strength than randomly oriented sisal fiber.

Akkimaradi K. et al.[109] Design and analysis of composite cylinder. E-glass epoxy composite pressure vessels are studied. Structural analysis of steel and fiber-reinforced composite pressure vessels were employed and compare the result. The analytical results for steel pressure vessels were compared with Ansys results. The weight and structural efficiency of the composite pressure vessel are compared with steel pressure vessels. Ansys results of steel and composite pressure vessel are compared for different internal pressure. And they conclude that composite pressure vessel has more strength than steel depending upon design criteria. Comparison of the weights shows that composite pressure vessel has 75 % less weight than steel pressure vessel. The structural efficiency of the composite pressure vessel is 76 % more than the steel pressure vessel.

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Debnath K. et al. [87] Works on Characterization and optimization of the mechanical performance of plant fiber composites for structural application. The objective of the study was to demonstrate whether plant fiber composite was a potentially structural replacement to E glass composites. Flax/ polyester and E- glass/ polyester were used to develop the composite. The effect of fiber volume ratio on the physical and tensile behavior of the Plant fiber-reinforced composite has been investigated. The fatigue performance of plant fiber-reinforced composite and glass fiber reinforced composite were compared. Investigate the manufacture and mechanical testing of a full scale 3.5-meter composite rotor blade suitable for 11 KW turbines built from flax/ polyester and E-glass/ polyester. It found that the flax/ polyester blade is 10 % lighter than the E glass/ polyester blade almost 45 % mass was saved. It also a lightweight, low cost and sustainable alternative to conventional aligned E glass reinforcements. From static testing of the blades, Flax/ polyester blade satisfies the design and structural integrity requirement for an 11 KW turbine under normal operation and worst case.

Abiy A. [108] works on the Design and analysis of bamboo and E glass fiber reinforced epoxy hybrid composite for wind turbine blade shell. Use bamboo and E glass fiber reinforced epoxy hybrid composite with unidirectional orientation with 5% NaOH treatment. Tensile, in-plane shear, compressive and flexural tests were carried out. A bamboo to E-glass fiber ratio of 0:100 was found a higher tensile strength; Whereas bamboo to E-glass fiber ratio of 50:50 had higher compressive strength. Similarly, a bamboo to E-glass fiber ratio of 50:50 had a higher elastic modulus for all the tests carried out. Finally, the author suggests that the bamboo and E glass fiber ratio of 50:50 output can have the potential to be used for wind turbine blade shell construction.

2.16 Literature summary

Different researches develop composite material with natural fibers that are locally available and had specific strength as reinforcement and thermosetting matrix as a binding element. They suggest to use it for many different engineering applications starting from safety helmets up to the airplane body. Due to its inherent characteristics of high specific strength to weight ratio. They also use different methodologies and investigation techniques for characterization and optimization of the experimental result. Different design parameters also considered fiber length, fiber chemical treatment, fiber type, fiber volume ratio, fiber ratio, matrix type, and so on.



Some researchers also studied hybrid composite material especially natural fiber with synthetic fiber. These composite also found that its mechanical performance is closely comparable with steel structure and others. Different design parameters are considered to fabricate composite material with maximum specific strength.

2.17 Research gap

Based on the above and most of the recent good studies are giving emphasis on mono-fiber composite material. i.e. using a single class of fibers as reinforcement to develop the composite material. Very few studies are done in hybrid fiber composite material. That has been developed with fiber hybridization. HFCM is a new opportunity to develop composite material with enhanced mechanical performance over mono-fiber composite material. Enset and Wool fiber were used to develop HFCM in this study. Up to know there is no published studies towards these fibers hybridized composite. Therefore, Developing HFCM with the attention of locally available, cheap and abundantly available natural fibers are the main objective of this study. Enset fiber and sheep wool as fiber reinforcement and epoxy as matrices will be used.



CHAPTER 3 METHODOLOGY

This chapter presents the materials and methods used for the fabrication of composites under study. The whole experimental work was designed into three stages as presented in the figure below. The first stage was raw material preparation, the second stage attempted to composite development and in the last third stage conduct laboratory test. These three stages also subdivided into phases as preferred.



Figure 3.1 Experimental work procedures

3.1 Materials

In this study, it uses a variety of materials, like matrix, fibers, mold, and testing types of equipment. These materials used during composite preparation and during the experimental investigation are presented below.

3.2. Raw material preparation

The matrix material and the reinforcement materials are the main listed raw materials used to composite fabrication. Raw materials used in this experiment:



- ✤ Matrix material
 - Epoxy resin and its hardener
- ✤ Reinforcement
 - ✤ Sheep wool
 - ✤ Enset fiber

3.2.1 Matrix material preparation

Matrix material or adhesives are the constitutes of composite material which is continuous and adhesive characteristics. Its main function is fixing the reinforcement together and transferring the load to the fibers. Thermoset polymer matrix is used in this study due to its inherent characteristics of good chemical and corrosion resistance, creep resistance, good affinity to heterogeneous materials, low specific gravity, chemical stability, easy processability, and manufacturability as well as low cost.

The matrix used in this work is Epoxy resin with the trademark name of SYSTEM # 2000 Epoxy RESIN, and # 2020 Hardener which are manufactured by FGDCC, or Fiber Glast Development Corporation Company. The number 20 indicates in #2020 that the mixture of epoxy and hardener will change from liquid to solid state after 20 minutes' pot life after it impregnated. This hardener has a feature of less toxicity than amine type hardeners, lower exothermic characteristics, have a long pot life, and due to its liquid state easy to mix with epoxy resins. It also cures completely at room temperature.

System 2000 epoxy resin is a medium viscosity, lightweight designed to offer the highest ultimate strength and used at room temperature. The mix ratio of epoxy resin to hardener was 4:1 on their volume after that it mixed and stirred for a few minutes using deep glass stick thoroughly.



Mechanical properties	Epoxy 2000 with hardener 2020
Mix ratio, volume	4:1
Pot life	77°F
Flexural strength	65308 psi
Tensile modulus	45326 psi
Glass transition temp. Tg	180 °F

Table 3.1 Typical properties of system 2000 Epoxy with hardener 2020

Source; Fiber Glast Development Corporation, 385 Carr Drive-Brookville, Ohio 45309, USA

3.2.1 Reinforcement material preparation

Reinforcement materials are the dis-continues phase of composite material which is the harder and load-bearing element. Its main function is supporting the load and increasing the mechanical strength of the composite material. Enset and Sheep wool fibers were used as reinforcement fiber during composite preparation.

For Enset fiber, the fiber was collected from the Jimma area, Ethiopia with a mean length of up to 1.3 m. The fibers were extracted from steam manually by using the sharp edge of the bamboo. To remove the flesh and debris from the fiber surface by using dry cleaning then washed with distilled water. After that, Enset fiber was soaked for an hour in 5% NaOH solution and later rinse with distilled water. This Chemical treatment by Sodium hydroxide solution was used to increase the fiber surface adhesive characteristics by removing impurities.





b)

Figure 3.2 Reinforcement material a) sheep wool b) Enset fiber

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After rinsing the fiber goes to the process of drying. The fibers were dried for 48 hours under laboratory conditions at 22°C near the window and followed by 8 hours outside under the sun.





Figure 3.3 a) Manual extraction of Enset fiber and b) Enset fiber For sheep wool, the fiber was collected from the Debrebrhan area, Ethiopia. It was collected in the form of wool and yarn from the local market. Then it passes through the following process. Sorting, cleaning carding spinning weaving finishing.

After cleaning, both Enset fiber and wool was treated with 5% of NaOH for one hour. Then the fibers washed thoroughly with distilled water and dried in sunlight for 72 hours to remove the moisture content in it. For the yarn wool, the treatment was followed after the spinning process.

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No	Process	Description	Image
1	Sorting	Organizing the wools and separating fibers from its kink and hard particles.	
2	Cleaning	Raw wool contains mess and wax. to clean these dirty particles the wool is washed with detergent and shampoos with water and followed by soaking with alkaline that is sodium hydroxide, NaOH.	
3	Carding	The wool after washing makes a kinked structure, the process of carding is necessary to separate the wool from its kink and also useful to remove residual dirt and other matter left in the wool.	
4	Spinning	Spinning is the process of forming a thread of wool or to form one strand yarn. The spinning process is done by a locally available manual rod-like device is called" <i>Enzirt</i> ".	

Table 3.2 Sheep wool and fiber preparations

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Sodium hydroxide (NaOH) with water is an alkaline solution used to enhance the surface morphology of the fiber in order to increase the surface adhesive characteristics.





Figure 3.4 a) Sheep wool and b) yarn of sheep wool



3.3 Composite development

The composite materials in this study were developed by hand lay-up technique with the following of the lightweight compression process. In this stage, the pattern preparation and composite preparation procedures were done respectively.

3.3.1 Mold preparation

The pattern that was used to develop the composite materials were prepared from 700mm *660mm MDF type wood. It was cut into six patterns with a dimension of length 250mm*width 180mm and thickness of 15mm by using a power-driven manual cutting machine. The detail dimension of the pattern is presented below in the figure.



Figure 3.5 Mold preparation a) Detail dimension and b) Pattern preparation from MDF wood

The inner side of the frame and the upper and bottom of the plate were covered with non-reactive plastic film and coated with petroleum jelly to lubricate the mold section that used to avoid the sticking of the composite to the surface of the mold. Tin plastic sheet also used between the top and bottom plate of the mold. Its functions were to avoid the sticking of the composite to the plate surface and to get a good surface finish of the composite.

3.3.2 HFCM preparation

The Hybrid fiber composite materials were prepared by using hand lay-up techniques. The hand layup method is the simplest processing step. It needs minimal infrastructure with easy procedure. It includes the design of fiber orientation and the casting of composite material. Generally, hand lay-up processes are more flexibility in composite design.

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Steps followed during specimen preparation: -

- *i.* Measure the weight of fibers and matrix as per the design
- *ii.* Stir the epoxy as per company instruction carefully to avoid the formation of air bubbles.
- *iii.* Putting a releasing film on the mold surface and coated with plastic film
- *iv.* Properly placing the fiber as required orientation in the mold
- v. Pour the resin and smear it gently on the fiber
- *vi.* Again placing the designed fiber ply and pour epoxy and smear it for HFC preparation as design requirement unless finishing it here
- *vii.* The preform of fiber (fiber powder) was used to minimize the air gap and composite thickness variation during casting HFCM.
- *viii.* On the top of the last play cover with plastic film and do rolling
- *ix.* Finally, close the mold by a designed plate with releasing agent then followed by a lightweight compression step

The thin plastic film was required to prevent the mold from air entry and to get a good surface finish at the end product. The lightweight compression step also performed with 15 kg pressing weight for 24 hours.





Figure 3.6 Composite material preparation a) cutting the woven fiber with mold dimension and b) pouring of epoxy into the fiber



The fiber was tailored with different orientations. These are categorized into Random and geometrical orientation. Random orientation includes random continuous and random discontinuous fibers. In Random continuous orientation, it contains long fibers. In random discontinuous orientation, it uses short randomly oriented fibers. Whereas, in geometrical orientation include unidirectional orientation and multidirectional orientation of fibers. In unidirectional orientation longitudinal and transverse direction, orientations were used. In multi-direction orientation, woven orientations were used.

a) Unidirectional orientation

In this type of orientation, the fibers are all arranged in the same direction either longitudinal or transverse.

Unidirectional longitudinal orientation

In this fiber orientation, the fibers were arranged longitudinally to "y"-direction or wide side of the mold. The fibers are aligned longitudinally which is parallel to the direction of the applied load. After uniformly arranged the fibers in the mold, pour the resin and smear it carefully throughout the mold. It followed with compression with a load of 15kg for a curing time of 24 hr.



Figure 3.7 Unidirectional longitudinal orientation

Unidirectional transverse orientation

In this fiber orientation, the fibers were arranged horizontally towards x-direction or narrow side of the mold. It was prepared to transverse loading that means the direction of the applied load is perpendicular to fibers arrangement. The fiber was placed uniformly in the mold then pour the resin and smear it carefully after that press it with 15kg load with curing time of 24 hr.





Figure 3.8 Unidirectional transverse orientation

b) Multidirectional orientation

The fibers are arranged in many directions as desired either geometrically systematic or randomly oriented.

Geometrically systematic (bi-directional orientation)

In this orientation, the fibers were placed by consideration of the geometrical axis. The fibers were oriented in two directions (warp 90 and weft 0) to form a woven type structure. It uses the same ratio of fibers in both directions. As shown in chapter two Figure 2.12 (a).

c) Random orientation

In random orientation, the fibers were arranged casually but with uniform distribution of fiber throughout the mold. Either random discontinuous fiber or random continuous fibers were used.

Random discontinuous fiber

Chopped fibers were used and placed thoroughly in the mold with uniform distribution. Then pour the resin carefully to avoid redundant assemblage of fibers due to the flow pressure of poured resin. After that, followed by a lightweight compression process.



Figure 3.9 Random discontinuous fiber orientation



Random continuous fiber

Long fibers were used and placed in the mold arbitrarily but with uniform distribution. After that, the resin was poured thoroughly to keep the fibers from the redundant assemblage and irregular distribution of it in the mold. Then followed with a lightweight compression process.



Figure 3.10 Random continuous fiber orientation

d) Layer or sandwich orientation

In this orientation use layer by layer of the above-listed orientation as desired.



Figure 3.11 Layer or sandwich orientation

3.3.3 Hybrid fiber composite development

The required hybrid fiber composite material was manufactured with a different type of fiber orientation, ply arrangement and fiber volume ratio with hand lay-up technique. The manufacturing composition as presented below in the table.



Orientation	Arrange	Compositions			No of sp.
	ment	EF (%)	W (%)	Ep (g)	
MMM	ESE	75	25	750	6
	SES	25	75	750	6
URU	ESE	75	25	750	6
	SES	25	75	750	6
MRM	ESE	75	25	750	6
	SES	25	75	750	6
RUR	ESE	75	25	750	6
	SES	25	75	750	6
MUM	ESE	75	25	750	6
	SES	25	75	750	6
М	Е	100	0	250	6
	S	0	100	250	6

Table 3.3 Designed HFCM

3.4 Laboratory Test

After preparing the composite, the next step was spacemen preparation and followed by lab testing and recording.

3.4.1 Spacemen preparation

During composite preparation, the prepared composites were with a dimension of length 300 mm width 180 mm and a thickness of 15 mm equal with mold dimension. It was cut by using an electrical saw machine to standard specimen dimensions.





Figure 3.12 a) cast composite b) cutting of composite with an electrical saw and c) specimens

3.4.1.1 Specimen preparation for tensile test

The dimensions of the samples are according to the ASTM standard D-3039. For each designed composite, it was prepared five specimens with a rectangular shape with a dimension of length 200 mm, width 20 mm and variable thickness. Sheet metal taps were attached at both ends of the specimen during griping in order to minimize the premature failure of the specimen.

The most commonly used specimen geometries such as the dog- bone specimen and straight-sided specimen with end tabs were prepared from flat samples. Square cut tabs were mounted on the specimens according to the specifications given by ISO 527. It used to minimize the complicated stress field in the gripping area that causes premature failure. That leads to the underestimation of the ultimate tensile strength of the composites.

The failure should have occurred inside the gauge section. Mostly the stress-strain curves obtained not be used to determine the true ultimate tensile strength of the composite since failure occurred outside the gauge section in the tab region.





Figure 3.13 prepared specimen for tensile test

3.4.1.2 Specimen preparation for flexural test

The three-point bending flexural test provides the modulus of elasticity in bending and bending stress-strain response of the material. The specimens were prepared according to ASTM D 790. It has a rectangular shape having a dimension of length 200mm, and width 20 mm. The span (center to center) distance for each specimen is 100mm.





Figure 3.14 Prepared specimen for flexural test

3.4.1.3 Specimen preparation for Impact test

Charpy digital testing machines were used to determine the resistance of impact or the sudden load of composite material. The specimens were prepared according to ISO 179: 1997 standards. Three specimens with 80 *10 *8 mm, with a V-notch 2mm deep at 45° on one side at the center were prepared at each sample.

Si.	Mechanical test	ASTM standard	Sample dimension, mm	Testing facility
No				
1	Tensile	ASTM D638 ISO 527-4	200*20*10	UTM testo meter
2	Flexural	ASTM D790 ISO 178	200*20*10	M350-5 CT testometric
3	Impact	ISO 179 ASTM D6110	80*10*8	Charpy digital impact test
4	Water absorption	ASTM D 570	40*40*10	

Table 3.4 Material testing standard

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Source: Materials Testing Standards for Additive Manufacturing of Polymer Materials, Aaron M. Forster, National Institute of Standards and Technology, 2015

3.4.1.4 Specimen preparation for TGA and XRD test

Thermogravimetric analysis (TGA) is used for determining the thermal stability and fraction of volatile components in a material. It is by measuring the material's weight loss as a function of temperature when the material is heated. The specimen used in the TGA test was prepared in the form of powder from each Enset and Wool. The fibers were crushed with mortar.

Similarly, in X-ray diffractograms (XRD) test the specimen was prepared in the form of powder with a mortar and compressed in the prepared mold. As shown in Figure 3.16 (b).

3.4.2 Testing and recording

The designed laboratory testing was conducted as per the ASTM standard. The temperature and humidity chamber was used to conditioning the specimen with 65% RH humidity and 20°C temperature for 24 hrs. Since too much dry or wet specimens had an effect on the result of testing.

3.4.2.1 Physical properties test

It covers length, diameter, density, water absorption, etc.

Length measurement

After washing and drying the fibers, 10 fibers were randomly chosen and their length measured with an optical microscope and meter. Then the average fiber length was reported.



Water absorption test

The water absorption test objective was to determine the capability of the fiber to absorb the water and measured with the weight gaining of the sample which is dipped in the water. Water absorption and thickness swelling tests were carried out according to ASTM D570-81. For water absorption test the procedures were first to prepare three specimens with a dimension of 40 mm length * 40 mm width for each factor. From both Enset and wool fiber the specimen was prepared. Then dip it in distilled water at room temperature. The dipping is done consequently with the interval of every 24 hr. At every 24 hours, the specimens were removed and the wiped the surface moisture with a hanky and measure the weight and dimension. Then again, dip it until the samples reached their saturation limit.



Figure 3.15 Water absorption test specimen and weight measurement apparatus

3.4.2.2 Thermal properties test

In order to study the thermal properties of fibers and the respected composite TGA thermal gravimetric analysis was conducted.

3.4.2.3 X-ray Diffraction (XRD)test

The X-ray diffractograms show that the intensity of the crystallographic plane within the fibers. The X-ray diffraction test was carried out using a DRAWELL artist of science X-ray. diffractometer with a rotating stage and goniometer in 2 Θ configuration. The wavelength of co radiation is 0.179mm with continuous scanning mode. The intensity was measured from 10^o up to 70^o at 2 with a step size of 0.0167^o and scan speed of 0.03 deg./sec.

The specimen for XRD testing was prepared by compacting the fiber powder in the prepared glass mold. The fiber was crushed with mortar.





Figure 3.16 a) DW-XRD Y3000 testing machine and b) Compacted fiber powder specimen

The degree of crystallinity (crystallinity index, Ic) was estimated by using this equation.

$$I_c = \frac{I_k - I_{am}}{I_k} \times 100 \tag{3.1}$$

Where I_c is the fibers crystallinity index, I_k is the maximum intensity or lattice reflection peak of diffraction at a 2 theta angle of b/n 15 and 25 and I_{am} is the minimum peak position or the intensity of diffraction of the amorphous fraction, which is taken at a 2 theta angle between 11 and 20 where the intensity is at a minimum.

a) Thermogravimetric analysis (TGA)

TGA measures the rate of weight loss of a material as a function of temperature when it was burning with the gas-conditioned environment. It used to investigate the thermal stability and fraction of volatile components in a material by measuring the weight changes as the material is heated. The thermal decomposition temperature T_d is increased when the heating rate increased. T_d is obtained from the point where weight loss suddenly increased.





Figure 3.17 TGA analysis equipment (PerkinElmer TGA 4000)

For this study, both Enset and wool fibers are investigated separately to analysis its thermal stability. And also this TGA analysis was conducted to the composites of these fibers with epoxy

During conducting the test, each specimen used in the TGA test was heated from 30 °C up to 700 °C with the incremental increase in temperature 30°c/min and weight loss was investigated. TGA curves for fibers and its composite samples are demonstrated with percent of weight loss as a function of temperature change.

3.4.2.4 Temperature and humidity conditioning process

Environmental conditions especially temperature and humidity have an undeniable impact on the test results' accuracy and consistency. Monitoring and control of laboratory conditions are to be acquired throughout the testing process. Achieving balanced relative humidity (RH) and maintaining it at a regulated level is essential before conducting the experiment. Not only the laboratory environment condition is necessary but also specimen condition is also necessary. Very dry or excessively moist specimen leads to an incorrect test result. Therefore, laboratory conditioning and specimen conditioning is the first action before investigation. In this study, the ETS 5506 series mid-size temperature and humidity chamber were used to conditioning the specimen with 65% RH humidity and 20 °C temperature for 24 hrs. Since too much dry or wet specimens had an effect on the result of testing.





Figure 3.18 Temperature and humidity curing machine (ETS 5506 series)

3.4.3.4 Mechanical test

During the development of new material, the main thing is recognizing its mechanical performance to withstand and tolerate stresses for the intended application.

Different types of Mechanical tests have been conducted in order to investigate the mechanical performance of composite material. Such as tensile strength test, flexural strength test, impact strength test and hardness of a material test. Most of these testing results are expressed in terms of stress-strain correlation.

Stress =
$$\frac{force}{area}$$
, $\sigma = \frac{F}{A}$ (3.2)

Strain =
$$\frac{deformation}{original area}$$
, $\epsilon = \frac{\Delta L}{lo}$, $\Delta L = lf - lo$ (3.3)

a) Tensile test

Tensile test is conducted in order to investigate the tensile strength of the composite material. Material's tensile strength is defined by measuring the force that pulls a specimen to the point where it will break. The maximum tensile stress where before specimen failure or breaking is defined as the tensile strength of the material. Tensile strength of the material is expressed in terms of tensile modulus and ultimate tensile strength obtained from the tensile test results. Tensile

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elastic modulus is an indication of the relative stiffness of a material and found from the stressstrain diagram of the linear region.

Test procedures

The tensile test was performed based on the ASTM D790 testing standard by using UTM with a load cell of 5KN and a fixed crosshead of 5 mm/min. Before conducting the test, the end edge of the specimen was a tap with sheet metal, in order to prevent it from premature failure during gripping. Tensile test set up as shown below and its set up drawing is in chapter two shown in Figure 2.12.



Figure 3.19 a) Tensile test set up

Calculation

During the tensile test, ultimate tensile strength and young's modulus are the main properties to be evaluated. The tensile strength of the composite material highly dependent on fiber concentration and orientation.

Ultimate tensile strength

$$\sigma_{ut} = \frac{p_{max}}{bh} \tag{3.4}$$

Where p_{max} is the maximum load before failure, b is the specimen width and h is the specimen thickness



Tensile modulus

$$E_t = \frac{\Delta\sigma}{\Delta\varepsilon} \tag{3.5}$$

where E_t is the tensile modulus of elasticity, $\Delta \sigma$ is the difference in applied tensile stress between the two strain points and $\Delta \varepsilon$ is the difference between the two strain points within the elastic region.

b) Flexural test

The flexural strength of HFCM is investigated from flexural test and expressed in terms of modulus of rupture (MR) in Psi (MPa) that defined by the measure of stress in a material just before it yields in the bending test.

V *Test procedure*

Flexural test of material is performed using center point loading in the three-point bend test. Based on the ASTM D790 testing standard, flexural or three-point bend test ware carried out by using a Testometric universal testing machine.



Figure 3.20 Experimental setup of the flexural test

Calculation

✤ Flexural stress

$$\sigma = \frac{_{3PS}}{_{2bh^2}} \tag{3.6}$$

Where σ is the normal stress on the outer surface of the specimen, p is the applied load and b is the specimen width h is the thickness of the specimen


Flexural modulus

$$E_{\text{flex}} = \frac{S^3 m}{4bh^3} \tag{3.7}$$

Where m is the slope of the tangent of the initial load-deflection curve

c) Impact test

The impact test is used to measure the materials' capability to resist the fracture under high-speed stress or impact load. It is related to the toughness of the material under high strain rate deformation. The test was carried as per ASTM D 256 using a Charpy digital impact tester. The specimen with MMM fiber orientation, and with EWE and WEW ply arrangement hybrid reinforced composite materials were used to investigate the impact strength of the material. The initial energy of the impactor was 7.5J at the initial height and released at 2.9 m/s speed.



Figure 3.21 Charpy digital impact tester

Impact strength
$$= \frac{E}{A}$$
 , $E = J_0 - J_1$ (3.8)

Where

 J_0 = Initial energy at the initial height

 $J_1 = Resultant energy$

A= Area of the specimen



3.5 Design of experiments (DOE)

Experiments are carried out to explore and analyze, the effect of the controllable variables on the final response. Design of experiments is a systematic technique used in the investigation of a system or process. It is a technique of maximizing the amount of information gained with the minimum amount of run experiment with fewer resources required. It allows on efficient judgment with the significance of input variables either individual or combination effect on output response.

Factorial design of experiment, Taguchi design of experiment, full factorial design of experiment and response surface methodology (RSM) were widely used techniques. RSM concept was used in this study.

it includes: -

- *i.* Designing a set of experiments
- *ii.* Determining a mathematical model
- *iii.* Testing of the adequacy of the model developed (statistical significance) and
- *iv.* Determining the optimal value of the response.

The overall flow chart is presented in Figure 3.22.





Figure 3.22 RSM optimal design experiment flow chart



RSM is one of design experiments technique which is useful for modeling and predicting how the response of interest is influenced by several input variables with the goal of optimizing this response. It combines both statistical and mathematical methods of experiment design, regression analysis, and optimization in order to deliver useful information.

If all independent variables are measurable and can be repeated with negligible errors, the response surface will be

$$y = f(x_1, x_2, \dots, x_k)$$
 (3.9)

Where: K is the number of the independent variables

In order to optimize the response "Y", it is necessary to find an appropriate estimate for the true functional relationship between the independent variables and the response surface. Typically, a second-order polynomial Eq. is used in RSM.

$$y = bo + \sum_{i=1}^{K} b_i x_i + \sum_{i=1}^{k} b_i X_i^2 + \sum_{i=1}^{k} \sum_{j=1}^{k} b_{ij} x_i x_j + \varepsilon \qquad \text{for } i < j \qquad (3.10)$$

in response to surface design, the central composite design (CCD), the Box-Behnken design (BBD) or optimal designs are used to develop mathematical models.

3.5.2 Optimal RSM designs

Optimal design is a good design choice when central composite and Box-Behnken do not fit the design needs. Since these particular methods are recommended choice when you have categorical and numerical factors, constraints, need to fit a cubic or higher-order model or are trying to fit a custom model. Unlike the CCD and BB design, where there is a specific pattern to the design points, points in these designs are chosen by an algorithm.

3.5.3 Steps in RSM

Deigning experiment, process characterization, optimization, and verification are often carried out phases in RSM optimal DOE.





planning experiments

This planning session carried out with first-hand knowledge of the project develop from the literature review. Determining the process parameters and critical factors through literature review and preliminary study.

Fiber orientation, ply arrangement and fiber volume ratio are the input parameters that affect the output response. Tensile strength and flexural strength are the output response which can suggest the strength of the HFCM. Parameter Fiber orientation (MMM, MUM, MRM, RUR, URU) levels, play arrangement (EWE, WEW) and fiber volume ratio (30, 50 & 75 %) with respective level. Design expert 12. software was used to design the optimum run of the experiment which helps to suggest overall responses with respect to input variables.

Designing and running experiments

After determining the factors and levels in the planning phase, the experiment now can be designed and running of the experiment carried out. RSM optimal design of the experiment suggests 27 runs of experiment with a combination of levels. After this, conduct the experiment and record the data.

Development of the mathematical model and analysis of variance

Design expert software develops and displays the possible modulus which can fit on the input data from the experiment and suggest the mathematical model that best fits the distribution of the experimental data.

Analysis of variance is a standard statistical technique used to interpret experimental data. Look at the p-values to determine if the model explains a significant portion of the variance. It also employed to review the relative influence of multiple variables and their significance. Generally, ANOVA is a test method look for low p-values to identify important terms in the model.

Testing the adequacy of the model developed

The adequacy of the models was tested by using the analysis of variance (ANOVA). ANOVA which a technique for studying sampled data relationships. It is used to test the significant difference between class means, and this done by analyzing the variance. F-test, lack of fit test and other adequacy measures like R^2 , adj- R^2 , pred. R^2 and adeq are used to examine the model is statistically significance or not.



F- test: - ANOVA calculates the fishers F- ratio, which is the ratio between the regression mean square and the mean square error. If the calculated value of the F-value is higher than the tabulated F-value, then the model is said to be adequate at the desired significance level α . In the current work the α – level is set at 0.05, i.e. the confidence level set at 95%.

The p-value (or prob > *F*): - P-value of the model and each factor in the model can be calculated by means of ANOVA. If the p-value less or equal to the selected α – level, then the effect of the presence of the variable term is significant. If the p-value is greater than the selected α – level, then it is considered that the presence of the variable term in the model is not significant. Sometimes the individual variables may not be significant. If the effect of interaction terms is significant, then the effect of each factor is different at different levels of the other factors.

Lack of fit test: - The lack of fit would be considered insignificant if the p-value of the lack of fit exceeds the level of significance.

The adequate precision: - compares the range of the predicted value at the design points to the average predicted error. Adq $R^2>4$ indicates the model is adequate.

The R^2 is the proportion of the variation in the dependent variable explained by the regression model. On the other hand, R^2 adj is the coefficient of determination adjusted for the number of independent variables in the regression model. For a good model, values of R^2 and R^2 adj should be close to each other and also they should be close to 1. If the fitted surface is an adequate approximation of the true response function, then the analysis of the fitted surface will be approximately equivalent to the analysis of the actual system.

Model reduction

Model reduction is a process of eliminating terms that are not desired or which are statistically insignificant (terms that have p-value greater than the level of significance α). There are three types of automatic model regression these are forward selection technique, backward elimination procedure, and stepwise regression method.

The stepwise regression method is used in this study. Which is a combination of the forward and backward regressions, where the calculations used for the inclusion and deletion of variables are the same as they are for the forward and backward procedures. Its advantage is assuming different



or similar levels of significance for inclusion or the deletion of variables from the regression equation.

Development of the final reduced model

After the final model has been tested and found to be adequate, the program automatically suggested a polynomial model which is best fits the criteria discussed in the fit summary section. This model includes only the significant terms and the terms that are essential in order to maintain the hierarchy.

After all, by using diagnostic graphs can make sure about the model that provides a good estimate of the true response surface.

3.5.4 Optimization

Optimization is a process of searching the combination of levels of factors that used to satisfy the goal placed on each of the factors and responses.

In this numerical optimization, the desired goal for each factor and responses will be on target, maximized, minimized, within range, none (for responses only) and set to an exact value (factors only). Weight and the importance of each goal can be changed in relation to the other goals. The numerical optimization process includes combining the goals into an overall desirability function (D).

3.6 HFCM machining test

Composite materials are widely used in structural applications due to their inherent valuable physical and mechanical performance. Still, its areas of application are limited due to its machining characteristics with currently available machines. Machining natural fiber-reinforced composite is a tedious task since there are many parameters that should be considered to prevent the material from machining damage and to use it for a long time effectively.

Machinability is the main factor to select one material to use in a structural application. Machining can have required to cut a material in the desired dimension to, assembly, repair, decoration and so on. In this section, the machinability of the designed HFCM and its characteristics during machining with currently available machines can be investigated. The surface of the machined edge will be presented and analyzed as below. The two main machining operation such as drilling



and cutting was performed on fabricated HFCM surface of the machined edge quality and machining damage are analyzed in the result section.

Manual toothless hack saw and Makita XVJ03Z 18V LXT Lithium-Ion Jigsaw, with speed motor of 2600strokes per minute for cutting operation and PED 5008 Electric Drill 10 mm Ingco power tools for drilling operation were used.



Figure 3. 23 HFCM composite machining a) Electric motor cutting b)Hand hacksaw and c) Drilling motor driven



CHAPTER 4 RESULT AND DISCUSSION

In this section, the result of the experiment and its analytical analysis of data are presented. The result of the model also discussed with the related literature review results and inspected with real-world areas of applications.

The first thing during developing new material is investigating its physical and mechanical properties with the experiment. Both physical and mechanical investigation has been conducted. Physical properties like length, water absorption, crystallinity index and so on. Whereas investigating mechanical properties include mechanical strength (flexural, tensile, and impact), thermal properties and so on.

4.1 Physical characterization

a) Length of the fibers

The average length of Enset fiber was 90 cm the shortest fiber 80 cm and the longest 110 cm. it shows a wide length distribution. The average length of wool fiber was 3 cm the shortest fiber 2cm and the longest 4.5cm.

b) Water absorption of the composite

The optimized HFCM with the highest mechanical strength was demonstrated in the water absorption test. In this test EWE and WEW with 50 % fiber volume ratio HFCM were inspected. In addition to that individual fibers composite i.e. E and W, fiber composites are investigated for four consecutive days. The result is also presented below in the graph. Percent of water absorption was used to analyses the capacity of HFCM that absorbs water.

The result shows that WEW, HFCM absorbed more water on the fourth day than the other with 25.28 %. The reason was in this composite material the wool fibers are more concentrated. Due to the amorphous nature of Wool, it is highly moisture absorbent than plant-based fibers.

In all samples, on the first day of immersion, the percent of water absorption is high and linear. For the next days, the linearity shows decreased and approach to constant. This implies that after some days the model reaches to saturation level. Continuing dipping after these days is not much necessary.





Figure 4.1 Percent of water absorption for samples with respect to time Percent of water absorption for E fiber was lesser than wool fiber. Besides that, the model that the hybrid of E and W shows more water absorption. The reason is the more fiber means the more water-absorbent, and also the amorphous nature of the wool and porosity in the composite are responsible for that.

Moisture absorption is maximum for composite with WEW, HFCM, having moisture absorption of 25% after 72 hr. dip in the water. The more amorphous behavior of the wool, porosity content, fiber matrix-adhesion are responsible for the moisture absorption behavior of the HFCM. This moisture absorption behavior had an impact on the strength and stiffness of the composite material. Since, this hydrophilicity behavior of the fiber consequence the fibers to swell, then developing shear stress at the interface, which responsible for fiber debonding and ply delamination. However, the fiber surface modification with chemical treatment can minimize this moisture absorption behavior of the fiber. Therefore, benzoyl chloride or other chemical treatment analysis will be studied in the future. This suggests the water absorption behavior of these composites is a great drawback. That limits its area of application. The fibers need additional chemical treatments other than NaOH. Besides that, waterproof paints and water-insoluble resins should be used to avoid water absorption behavior in composite material.



4.2 XRD test result

The X-ray diffractograms show that the intensity of the crystallographic plane within the fibers.



Figure 4.2 X-ray diffraction patterns of Wool and Enset fiber treated and untreated with 5% NaOH

The figure shows the main diffraction intensity was about $2\theta = 22.5$ for Enset fiber which treated with 5% NaOH. Enset fiber demonstrated relatively high crystallinity as compared to wool fiber. From this, the treated Enset fiber has better crystallinity than the untreated one. Similarly, the main diffraction intensity for wool fiber about $2\theta = 20$, which is treated with 5% NaOH. Whereas, wool fiber shows relatively low crystallinity as compared to others. The possible reason is wool fiber contains keratin, while Enset fiber contains cellulose. Cellulose is better crystalline than keratin. For the treated Enset and wool fibers, the diffraction intensity is better. Since the chemical treatment removes the non- cellulosic material like lignin, wax, hemicellulose. And also the crystal structure of these fibers was modified to some extent.

The diffraction intensity pattern shows for both Enset and wool fiber it was not sharp. It indicates that the crystallinity order in the fibers was lower. The molecules in the fiber do not form appropriate crystals.



	Untreated E fiber	Treated E fiber	Untreated W fiber	Treated W fiber
Ik	1716	2250	943	1129
I _{am}	930	1210	621	742
I _c (%)	45.80	46.22	34.15	34.27

Table 4.1 Crystallinity indices, of 5% NaOH treated and untreated Enset (E) and wool (W) fiber.

The estimated crystallinity index I_c of the treated Enset fiber is 46.22 % which is higher than that of untreated wool fiber (34.27%). This low crystallinity index shows that the wool fibers have relatively high amorphous regions than Enset fiber. The crystallinity index for treated fiber is higher than the untreated one. This indicates that during NaOH treatment the removal of non-crystalline material from the fibers and the crystal are better oriented in the fiber. The higher amorphous region in the fiber also shows the fiber is more water absorbents. Therefore, besides increasing crystallinity of the fiber during 5%NaOH treatment the water absorption characteristics of the fibers are also minimized. From the result, the plant cellulose has better crystallinity than wool keratin.

4.3 Thermal characterization

In order to study the thermal properties of fibers and the respected composite TGA thermal gravimetric analysis was conducted.

a) Thermogravimetric analysis (TGA)

During conducting the test, each specimen used in the TGA test was heated from 30 °C up to 700 °C with the incremental increase in temperature 30°C/ min and weight loss was investigated. TGA curves for fibers and its composite samples are demonstrated with percent of weight loss as a function of temperature change.



TGA test result of fibers



Figure 4.3 TGA analysis of fibers shows the mass loss of Enset (E), wool (W)fiber and composite (C) as a function of temperature

TGA test result of Enset fiber

7.537mg Enset fibers were used for the test with nitrogen media.

During the investigation, it was observed that there is a distinct phase in the analyzed graph

- i. Initial weight loss of about 4-5 % for the temperature of 0- 115 °C was considered to be the evaporation of water in the fiber.
- ii. The second phase shows almost negligible change in 4% of weight loss from 115-280°C it implies the stability of fiber in this range.
- iii. In the third phase, a sharp decrease in weight approximately up to 55% from 280- 450°
 C indicates a major thermal decomposition of Enset fiber. This sharp drop in the mass of the samples indicates the thermal degradation of cellulose from the fiber.
- iv. Finally, complete loss of organic matter at a very slow rate between 450°C 525°C suggesting complete combustion of residual carbon-based material or ashes.

Cellulose is the main component of natural plant fibers that control and determine the degradation behavior of the fibers. In the third phase, the sudden drop in the mass was due to the degradation of this cellulose. Almost 55-60% of weight loss is occurring due to this cellulose degradation.



The main decomposition temperature was obtained at 350 °C. The value was close value to compare with other plant fibers' findings from literature such as sisal, flex, and banana.

TGA result for Sheep wool fiber

6.105652 mg sheep wool fibers were used for the test with nitrogen media.

During the investigation, it was observed that there is a distinct phase in the analyzed graph.

- i. Initial weight loss of about 1-3 % for a temperature of 0- 105 °C was considered to be the evaporation of water from the fiber.
- The second phase shows, almost negligible change in 5-6 % of weight loss from 105-280° C. it implies the stability of fiber in this range
- iii. In the third phase, a sharp decrease in weight approximately up to 60% from 280-525° C indicates a major thermal decomposition of wool fiber. This sharp drop in the mass of the samples indicates the thermal degradation of the keratin from the wool fiber. In mass 3.5mg
- iv. Finally, complete loss of organic matter at a very slow rate between $525^{\circ}C 650^{\circ}C$ suggesting complete combustion of residual carbon-based material or ashes.

keratin is the main component of wool fibers that control and determine the degradation behavior of the wool fibers. In the third phase, the sudden drop in the mass was due to the degradation of this keratin. Almost 55-60% weight loss is occurring due to this keratin degradation.

The main decomposition temperature was obtained at 380 °C. The value was close value to compare with other fibers' findings from the literature.

TGA result for HFCM

14.510435 mg of HFCM powder was used for the test with nitrogen media.

During the investigation, it was observed that there is a distinct phase in the analyzed graph

- i. Initial weight loss of about 2-3 % for the temperature of 0- 105 °C was considered to be the evaporation of water from the fiber.
- The second phase shows, almost negligible change in 10-11 % of weight loss from 105-280° C it implies the stability of fibers in this range



- iii. In the third phase, a sharp decrease in weight approximately up to 35-40% from 280-375°C indicates major thermal decomposition of fibers. This quick drop in the mass of the samples indicates the thermal degradation of cellulose from E fiber and keratin from W fiber.
- iv. Finally, complete loss of organic matter at a very slow rate between 400°C 670°C suggesting complete combustion of residual carbon-based material or ashes.

The main decomposition temperature was obtained at 310 °c. The value was close value to compare with other composite material findings from the literature.

4.4 Mechanical characterization of the developed composites

Tensile strength, flexural strength, and impact strengths are significant to recommend the suitability of any composite material for structural application.

4.4.1 Tensile test result of HFCM

Tensile test result of the prepared HFCM is presented below with tabulated form and with graph interpretation.

Ply arrangement, orientation, fiber volume ratio are factors considered during the HFCM specimen. Ply arrangement is laying off the respective lamina in the sandwich model of HFCM. These arrangements are grouped in two levels these are Enset –Wool- Enset (EWE) and Wool-Enset- Wool (WEW). In fiber orientation, the orientation of fibers in the lamina, which are unidirectional, Mat or random fiber orientation are considered. These orientations were grouped into five levels. These are MMM, MRM, MUM, RUR, and URU. M stands for Mat or Woven fiber orientation, R stands for random fiber orientation, U stands for unidirectional longitudinal fiber orientation.

From these, for each designed HFCM three specimen were prepared. The mean value of the result is presented and used for further analysis.

a) Effect of fiber orientation on tensile strength of HFCM

The result of the measured tensile strength of HFCM is presented below with respect to its orientation and ply arrangement.



Table 4.2 Tensile test experimental result of HFCM with respect to parameters

Run	Volume	Ply arrangement	Orientation	Tensile	Force @peak	Young's modulus
	%			strength	Ν	MPa
				MPa		
1	75	EWE	MMM	20.86	3906.3	519.2
2	30	EWE	MMM	18.87	3923.23	513.28
3	75	EWE	URU	30.06	4808.6	990.54
4	30	WEW	URU	1.74	293.8	73.72
5	30	EWE	RUR	4.24	1103.3	340.64
6	50	EWE	URU	31.82	4937.9	1090.5
7	50	WEW	MMM	5.64	1087.3	160.372
8	50	WEW	RUR	11.78	1887.56	439.06
9	50	WEW	MRM	4.02	805.8	69.6
10	50	EWE	RUR	7.82	828.9	104.5
11	50	WEW	MRM	4.02	805.8	69.6
12	30	EWE	URU	28.63	4833.6	1076.6
13	30	EWE	MRM	17.04	3523.6	409.06
14	50	WEW	MMM	5.64	1087.3	160.372
15	30	EWE	MUM	20.08	4396.9	1049.2
16	75	WEW	RUR	10.93	1723.26	405.04
17	75	EWE	RUR	5.89	498.9	101.96
18	75	EWE	MUM	23.1	4619.6	951.72
19	50	WEW	MUM	9.28	1856.16	337.64
20	50	WEW	RUR	11.78	1887.56	439.06
21	75	EWE	MRM	19.18	3456.62	828.52
22	75	EWE	MUM	23.1	4619.6	951.72
23	50	WEW	URU	2.76	394.21	84.64
24	30	WEW	МММ	4.18	839.95	156.5
25	50	WEW	MUM	9.28	1856.16	337.64
26	50	EWE	МММ	22.06	3996.2	915.28
27	75	WEW	URU	2.06	394.21	82.52



The result of the measured tensile strength of HFCM is presented below in the graph with respect to its orientation and ply arrangement.



Figure 4.4 Effect of considered parameters on maximum tensile load and respective tensile strength a) force @peak versus designed parameters and b) ultimate tensile strength versus designed parameters



The value in the x-axis and z-axis in the graph is the code that stands for orientation and plies arrangement that listed in the table. For instance, code 1 is HFCM with MMM orientation and EWE ply arrangement and code 2 implies HFCM with MMM orientation and WEW ply arrangement and continued like that. Shown in the Figure 4.4 below.

The above data implies that the ultimate tensile strength (UTS) for the developed HFCM is affected by the ply arrangement in the sandwich model of the composite. HFCM with Enset- Wool-Enset (EWE) ply arrangement shows preferable tensile strength. Since in this ply arrangement more Enset fiber lamina were used than wool fibers lamina. Concentrating Enset fiber in the composite gives better strength than concentrating sheep wool in HFCM.



Figure 4.5 The effect of fiber-matrix volume ratio on the tensile strength of the HFCM

The fiber matrix volumetric ratio in the composite also had its own impact on the tensile strength of the composite. When the fiber ratio increased from 30% to 50%, it shows the tensile strength also increases. Whereas, in 75 % fiber volume ratio the tensile strength was not higher as expected instead the fiber was pullout instead of bearing the load was shown during failure. Since in this concentration of fiber, the matrix cannot impregnate completely the fibers or there was a formation of Fiber Bridge in the composite. It indicates there is poor interface contact between fibers. This follows a weak load transfer and underestimated strength of the composite. Therefore, in order to get optimum tensile strength of the composite the fiber to matrix ratio should be proportional. The



HFCM with 50 % fiber volume concentration has superior tensile strength than the other. The matrix can impregnate the fiber appropriately then the Fiber matrix interaction bond is strong to withstand the applied load.

The concentration of Epoxy in this study is expressed in mass unit grams and excluded its effect on developed HFCM. The reason to express the concentration of epoxy in gram was the initial weights of the epoxy were not found at the final product. Because after curing and drying composite the resin shows a huge difference in weight change. There is a wide variation of weight of the designed composite before and after the drying process. However, for all designed composite material the volume ratio of the matrix and the reinforcements should be proportional in order to minimize fiber bridging and voids in the composite.

The URU fiber orientation with EWE plies arrangement and 50% fiber volume ratio had superior tensile strength than the other with a tensile strength of 31.26 MPa and 1090.5 MPa modulus of elasticity. This result provides there is excellent adhesion between the reinforcement and the matrix and within the plies.

b) Comparison of E and W with epoxy

Investigating tensile strength of each fiber type with keeping its fiber volume fraction constant and with similar woven fiber orientation. Enset fiber composite shows better tensile strength. Enset fiber composite observed to have tensile strength and tensile modulus of about 23.01 N/mm2 and 626.26 N/mm2 respectively.



Figure 4.6 Tensile strength on reinforcement type



The investigation shows that Enset fiber has the highest ultimate strength than the sheep wool and can resist the highest applied tension load. The measured tensile strength of HFCM in this study is closely comparable with the results of human-made fibers reported in the literature.

c) Tensile test failure behavior of HFCM

The failure characteristics of composite material are quite complex to analyze. Since the failure took place in the gage area or tab (gripe) zone by fiber pullout, breakage or delamination of the lamina. Its failure behavior is also highly dependent on each fibers straightness and placement. In the hand lay-up technique, the main difficulty is placing fibers in parallel. Some fibers are tilted or wrinkled or weaker than the other. It has a great impact on the failure behavior of the specimen. Due to that, to minimize the effect of fiber straightness in the composite the testing machine applied 10 N pre-tension loads.



Figure 4.7 Force vs. elongation graphs of HFCM

In lower strain, the specimen behaves elongate nearly linearly in the liner region. However, its linearity is also not smooth because of micro-crack and gradually straighten of the fiber in the composite. At higher strain, micro-cracks propagate and gradual debonding and pulled out of the fibers are take place. After the non-linear region, the fibers are completely pulled out from the matrix and the specimen gets failure.







Figure 4.8 a) Fracture behavior of MMM with EWE HFCM and b) Schematic view of failure

The failure modes of the developed HFCM are different for each type of fiber orientation and ply arrangement. For the above HFCM, the failure is with a large area of longitudinal splitting.

For 50% fiber volume ratio with MMM fiber orientation and EWE ply arrangement the failure mode is less-brittle manner. The failure surface was with less fiber pullout and large longitudinal splitting. For MMM fiber orientation with WEW ply arrangement, the failure was more serrated with extensive fiber pullout and the delamination of the layer also seen.

HFCM with 75% fiber volume ratio produces a more ragged and uneven fracture surface was seen. Delamination of a layer also occurred. Since the insufficient matrix prevents transmitting the load among fibers. Whereas, HFCM with 25% fiber volume ratio the fracture occurred on the tap region. Such typical failures in the gripping of the composite specimen are leading to underestimation of the ultimate strength of the composite. The strain for the failure of the MMM oriented HFCM was lower than URU oriented HFCM.



The fracture surface for unidirectional fiber with EWE ply arrangement and 50% fiber volume ratio shows the fracture was in a brittle manner with short pullout fiber. Whereas, for maximum fiber volume ratio, the fracture surfaces are rougher and higher pullout fibers due to the inadequate availability of the matrix in the composite. This is creating porosity and Fiber Bridge in the HFCM.

The premature failure in the gripping area of the tensile test specimen was due to the presence of a stress singularity at the tab wedge tip. These types of failure will underestimate the strength of the material. That is why rectangular specimen with fabric tab was selected according to ISO 527.



Figure 4.9 Failure of the tensile test specimen in grip and gauge zone respectively.

4.4.2. Flexural or three-point bending test result of HFCM

For this test, three specimens were taken for each designed HFCM sample test but the mean values of the test result were presented as below. The samples were grouped according to play arrangement, fiber orientation, and fiber volume ratio. Flexural test or three-point bend test from Table 4.3 shows that for each considered parameter the required load to bend the specimen was varying for every change of parameters.



Sample	Fiber	Ply	Orientation	Force	Flexural	Max. strain
code	Volume	arrangement		@peak, N	strength, MPa	
	ratio, %					
1	75	EWE	MMM	95.86	6.57	0.016
2	30	EWE	МММ	88.23	5.82	0.058
3	75	EWE	URU	65.18	4.35	0.018
4	30	WEW	URU	27.13	1.89	0.012
5	30	EWE	RUR	35.63	2.36	0.057
6	50	EWE	URU	74.13	5.03	0.037
7	50	WEW	MMM	80.27	5.04	0.046
8	50	WEW	RUR	31.24	2.09	0.043
9	50	WEW	MRM	70.22	4.2	0.014
10	50	EWE	RUR	35.63	3.26	0.049
11	50	WEW	MRM	70.22	5.5	0.067
12	30	EWE	URU	62.45	4.02	0.034
13	30	EWE	MRM	72.4	4.88	0.067
14	50	WEW	МММ	80.27	5.04	0.052
15	30	EWE	MUM	76.46	5.11	0.047
16	75	WEW	RUR	29.24	1.86	0.043
17	75	EWE	RUR	38.45	2.03	0.034
18	75	EWE	MUM	81.92	5.53	0.023
19	50	WEW	MUM	71.82	4.73	0.034
20	50	WEW	RUR	31.24	2.09	0.059
21	75	EWE	MRM	74.2	5.12	0.016
22	75	EWE	MUM	81.92	5.53	0.023
23	50	WEW	URU	33.13	2.51	0.012
24	30	WEW	MMM	77.23	4.9	0.052
25	50	WEW	MUM	71.82	4.73	0.034
26	50	EWE	MMM	110.01	7.53	0.057
27	75	WEW	URU	30.83	2.03	0.026

Table 4.3 Three-point bending test result design expert output



This implies the parameters such as fiber orientation, ply arrangement and fiber volumetric ratio are the main factor to be considered in the flexural strength of designed HFCM in the Figure 4.10.







This implies, from these design parameters, HFCM with EWE ply arrangement, 50% fiber volumetric ratio and MMM fiber orientation required maximum force during deforming in the three-point bend test. since in this EWE ply arrangement more Enset fiber with a mat or fabric orientation were used and also in 50 % fiber volumetric ratio the matrix to fiber interactions were strong which means the fiber and the ply were fully impregnated in the matrix.

In woven or Mat fiber orientation with EWE play arrangement and 50% fiber volume ratio the bending force was maximum. Since the interwoven orientation of fiber protects the composite from quick crack propagation or failure during flexural loading than unidirectional and random fiber orientation. However, in woven with 75% fiber volume ratio HFCM, during flexural loading showed delamination of ply in the upper region without fracture. It indicates poor interaction and a formation of layered structure between respective lamina. This may be due to the creating of a layered structure of the lamina in the composite. Besides that, in the 50 % fiber volume ratio, MMM fiber orientation, and EWE play arrangement, flexural strength was maximum compared to other HFCM. The possible reasons will be, (1), There is strong ply-to-ply interaction due to adequately impregnation of the lamina in the matrix and the fiber also strongly bonded with each other in these orientations. (2), The waviness of these fibers in the lamina significantly increases the load transfer capability in the composite.

The flexural strength was better in the EWE ply arrangement than WEW ply arrangement. It happens because the more Enset fiber was found in the EWE ply arrangement than WEW. This implies the stiffness of Enset fiber is higher than that of woven fiber. Therefore, a higher Enset fiber percentage in the composite can improve the flexural strength of the composite.

Flexural strength test is all about testing the stiffness of the lamina and its dimensional stability during bending load. It is the simultaneous testing of tensile and compressive strength of the sample. During bending load, the upper side of the specimen loaded by compressive stress with the loading noise and simultaneously the bottom surface loaded to tensile stress.

The result also shows, when the fiber volume ratio in the composite with respect to the matrix was increased from 30-50, the flexural strength of the composite also increases with 20 %. However, in increasing volumetric ratio '50-75%,', the flexural strength was decreasing with 12 % even if more fiber was found in the composite. The possible reason was the fibers were not fully



impregnated in the matrix or it creates the fiber bridge or void in the composite. It also shows the delamination of lamina during bending failure because of poor interaction of lamina in the sandwich model.



Figure 4.11 Fiber volume ratio with respect to flexural strength

Therefore, during HFCM design increasing the reinforcement volumetric ratio is not always increase the strength of the material. However, the proportional fiber and matrix ratio should be considered for good fiber to matrix interaction in the composite.

The flexural strain of HFCM is reduced with increasing fiber volume fraction, from 0.057mm/mm to 0.016 mm/mm. The flexural strength is decreasing with increasing fiber volume fraction above 50%. The highest HFCM selected from this result is MMM with a EWE ply arrangement with a 50% fiber volume ratio. Its flexural strength 7.53 N/mm2 with deflection loads of 110.43N and 0.057mm/mm flexural strain.

a) Flexural test Failure behavior of HFCM

When the bending strain increases due to bending load, both tension and compression stress is developed in the specimen. The crack formation was taking place on the lower side of the specimen where the matrix cracked, which is the opposite side of the loading. As the bending load continued, the crack was propagated to the upper side of the specimen. Then fiber pullout in the



tension zone and delamination of plies in the compression side took place. Finally, the failure of the specimen is existing.

This implies the parameters such as fiber orientation, ply arrangement, and fiber volumetric ratios are the main factor to be considered in the flexural strength of designed HFCM.



Figure 4.12 Failed samples of flexural testing of HFCM

In the lower fiber volume ratio, the specimen shows that the fibers breakage instead of the pullout of fibers from the matrix. These indicate that there is better interfacial strength between the laminas, especially in A (30 %) fiber volume ratio samples. However, in C (75%) fiber volume ratio the broken out fiber was almost null but the failure was due to fiber pull out and delamination of plies in HFCM. This indicates that the fiber-matrix bond was weak due to insufficient wetting of fibers with a matrix. Similarly, in Unidirectional orientation HFCM the broken out fiber in tension region is almost null and delamination of the plies shown in the compressive region of the specimen. Whereas for woven or mat orientation HFCM broken out fibers and delamination of plies are existing.



4.4.3 Impact test result of HFCM

The impact test is used to measure the materials' capability to resist the fracture under high-speed stress or impact load. It is related to the material's toughness under high strain rate deformation. The initial energy of the impactor was 7.5J at the initial height and released at 2.9 m/s speed to strike the specimen. The result has been presented in the table and graph.

Ply arrangement	Orientation @ 50% fiber ratio	Impact strength, J	Impact strength (J/m^2)
WEW	MMM	3.28	21.13
	URU	3.77	18.65
EWE	MMM	1.740	28.8
	URU	2.34	25.8

Table 4. 4 Impact test result

Both ply arrangement and Fiber orientation of the HFCM can have a great effect on the impact strength of the specimen. EWE ply orientation has preferred impact strength than WEW. Whereas, MMM fiber orientation had better impact strength than URU. From this, EWE ply arrangement with MMM fiber orientation had maximum impact strength as shown in the figure.







The maximum impact strength of 28.8 KJ/m² is obtained in the case of MMM fiber orientation with EWE ply arrangement.



Figure 4.14 Interaction graph of the factors on the impact strength of the HFCM Generally, impact strength suggests material toughness under high strain rate deformation. It is all about a measure of the materials' energy absorption during failure. Form experimental investigation, the impact strength of the developed HFCM improved with respect to ply arrangement and fiber orientation. In WEW ply arrangement HFCM the crack propagates more easily and with less energy than EWE. Similarly, in URU fiber orientation the high impact stress distribution is less. In MMM the stress distribution is not only in transverse direction but also in the longitudinal direction. Due to that the crack growth and propagations in this specimen are less.

The failure mechanism is also due to fiber pull out, shear of fiber and delamination of ply. The interaction region also has a negative impact on the impact strength of the material. In URU fiber orientation the interaction between the fiber and the matrix is good. but it leads to fiber breakage instead of the pullout and debonding. This shows brittle type failure to the composite. Whereas in MMM there is less interaction between the fibers that leads to progressive fiber pullout and fiber breakage. Fiber pullout dissipates more energy compared to fiber breakage.



4.5 Design of experiment (DOE)

After properly determining the factors and levels, the experiment can be designed and run as below shown in table 4.7. RSM optimal design of the experiment suggests 27 runs of experiment with a combination of levels.

4.5.1 RSM

Optimal custom design is used in this study. It is a design choice when central composite and Box-Behnken do not fit your needs. Since this particular method is the recommended choice when the design has both categorical and numerical factors, constraints, need to fit a cubic or higher-order model or are trying to fit a custom model.

4.5.1.1 Effect of design parameters on the strength of HFCM

Table 4.5 shows the HFCM input parameters and experimental design levels that have been used for developing the HFCM. The average of three measurements is recorded for each response. Two mathematical models were successfully developed to predict the response of tensile strength and flexural strength of the HFCM.

Fact or	Name	Un its	Туре	Mini mum	Maximu m	Code d Low	Code d High	Mean	Std. Dev.
A	volume	%	Numeric	30.00	75.00	$-1 \leftrightarrow$ 30.00	+1 ↔ 75.00	52.22	17.17
В	ply arrangement		Categorical	EWE	WEW			Levels:	2
С	orientation		Categorical	MMM	URU			Levels:	5

Table 4.5 Design parameters and corresponding level for developing HFCM

Table 4.6 Ext	perimental re	esponse fo	or tensile	test and	flexural	test
		sponse ic		test and	IICAului	test

R1	tensile strength	MPa	27	Polynomial	1.84	30.92	13.08	9.33	16.80	Quadratic
R2	flexural strength	MPa	27	Polynomial	1.86	7.53	4.21	1.60	4.05	Quadratic



Run	Volume ratio,%	Ply arrangement	Orientation	Tensile strength, MPa	Flexural strength, MPa
1	75	EWE	MMM	20.86	6.57
2	30	EWE	MMM	18.87	5.82
3	75	EWE	URU	30.06	4.35
4	30	WEW	URU	1.74	1.89
5	30	EWE	RUR	4.24	2.36
6	50	EWE	URU	31.82	5.03
7	50	WEW	MMM	5.64	5.04
8	50	WEW	RUR	11.78	2.09
9	50	WEW	MRM	4.02	4.2
10	50	EWE	RUR	7.82	3.26
11	50	WEW	MRM	4.02	5.5
12	30	EWE	URU	28.63	4.02
13	30	EWE	MRM	17.04	4.88
14	50	WEW	MMM	5.64	5.04
15	30	EWE	MUM	20.08	5.11
16	75	WEW	RUR	10.93	1.86
17	75	EWE	RUR	5.89	2.03
18	75	EWE	MUM	23.1	5.53
19	50	WEW	MUM	9.28	4.73
20	50	WEW	RUR	11.78	2.09
21	75	EWE	MRM	19.18	5.12
22	75	EWE	MUM	23.1	5.53
23	50	WEW	URU	2.76	2.51
24	30	WEW	MMM	4.18	4.9
25	50	WEW	MUM	9.28	4.73
26	50	EWE	MMM	22.06	7.53
27	75	WEW	URU	2.06	2.03

Table 4.7 Experimental data and result for HFCM



✤ Correlation graph of design parameters and responses (tensile strength and flexural



A:volume (%)





Figure 4. 15 Correlation graph for fiber volume ratio and response a) tensile strength and b) flexural strength

4.5.2 Development of Mathematical models and Analysis of variance

As per data distributions of the response, the fit summary output indicated that the quadratic model was statistically significant for both tensile strength and flexural strength. Therefore, the quadratic or curve fit model was used for further analysis. By using the stepwise regression method, the insignificant terms in the model are eliminated.

The resulting ANOVA analysis for the quadratic model of both flexural and tensile strength is presented below in table 4.8 and 4. 10 respectively. The other adequacy measures for both models are carried out. And the result shows that the model is adequate. This means the model can be used to navigate the design space.

4.5.2.1 For flexural strength model

The analysis of variance indicates that the main effects are; in this case, B, C, BC, A² are significant model terms.



			1		1	
Source	Sum of Sauares	df	Mean Sauare	F-value	n-value	
	Suit of Squares	•••		1 / 0.00		• • • •
Model	64.21	11	5.84	45.03	< 0.0001	significant
A-volume	0.1243	1	0.1243	0.9589	0.3430	
B-ply arrangement	12.57	1	12.57	96.99	< 0.0001	
C-orientation	47.08	4	11.77	90.81	< 0.0001	
BC	2.17	4	0.5428	4.19	0.0179	
A^2	1.99	1	1.99	15.37	0.0014	
Residual	1.94	15	0.1296			
Lack of Fit	1.10	10	0.1099	0.6505	0.7371	
Pure Error	0.8450	5	0.1690			
Cor. Total	66.16	26				

Table 4. 8 ANOVA for the reduced quadratic model of flexural strength

The Model F-value of 45.03 implies the model is significant. There is only a 0.01% chance that an F-value this large could occur due to noise.

P-values less than 0.0500 indicate model terms are significant. In this case, B, C, BC, A² are significant model terms. Values greater than 0.1000 indicate the model terms are not significant. The Lack of Fit F-value of 0.65 implies the Lack of Fit is not significant relative to the pure error. There is a 73.71% chance that a Lack of Fit F-value this large could occur due to noise. Non-significant lack of fit is good -- we want the model to fit.

Fit Statistics

Std. Dev.	0.3600	R^2	0.9706
Mean	4.21	Adjusted R ²	0.9491
<i>C.V.</i> %	8.55	Predicted R ²	0.8958
		Adeq. Precision	22.8084

The *Predicted* R^2 of 0.8958 is in reasonable agreement with the *Adjusted* R^2 of 0.9491; i.e. the difference is less than 0.2. *Adeq Precision* measures the signal to noise ratio. A ratio greater than 4 is desirable. Your ratio of 22.808 indicates an adequate signal. This model can be used to navigate the design space.



Then the next step will be developing the mathematical model which can represent the experimental result.

Final mathematical model in terms of the actual factors

The response Fs, flexural strength, of HFCM in the function of fiber volume ratio. Since the other two parameters are categorical data.

Orientation	Ply arrangement	Equation, Fs (A)
MMM	EWE	2.95498 + 0.153666 * A -0.00141421 * A ²
	WEW	$1.11573 + 0.153666 * A - 0.00141421 * A^2$
MUM	EWE	$1.89756 + 0.153666 * A - 0.00141421 * A^2$
	WEW	$0.582202 + 0.153666 * A - 0.00141421 * A^2$
MRM	EWE	$1.54637 + 0.153666 * A - 0.00141421 * A^2$
	WEW	$0.702202 + 0.153666 * A - 0.00141421 * A^2$
RUR	EWE	$-1.13502 + 0.153666 * A - 0.00141421 * A^2$
	WEW	$-1.94188 + 0.153666 * A -0.00141421 * A^2$
URU	EWE	$0.781647 + 0.153666 * A - 0.00141421 * A^2$
		$-1.54169 + 0.153666 * A - 0.00141421 * A^2$

Table 4. 9 The final mathematical model in terms of the actual factors for flexural strength

Where:

- M: Mat or woven fiber orientation
- R: Random fiber orientation
- U: Unidirectional fiber orientation
- EWE: ply arrangement with Enset wool Enset
- WEW: ply arrangement with wool Enset wool
- A: fiber volume ratio, Fs: flexural strength



The equation in terms of actual factors can be used to make predictions about the response for given levels of each factor. Here, the levels should be specified in the original units for each factor. Then the next step will be validating the model.

4.5.2.2 Validation of the models

In figure 4.16 below shows the relationship between the actual and predicted values of the tensile and flexural strength. The residual tends to close to the diagonal line it shows that the developed models are valid and adequate. Since the residuals in the predictions of each response are small and all the actual values of the percentage error for response are in reasonable agreement.



Figure 4.16 Predicted vs. actual graph

A graph Actual vs. predicted helps to detect observations that are not well predicted by the model. The value of R- squared, $R^2 = 0.9706$ is a measure of the amount of variation around the mean explained by the model. The model is 97.06 % fitted with the variability in flexural strength.




4.5.2.3 For tensile strength

The analysis of variance indicates that the main effects are; in this case, A, B, C, AB, BC, A², are significant model terms.

Source	Sum of Squares	df	Mean Square	F-value	p-value	
Model	2260.11	12	188.34	792.00	< 0.0001	significant
A-volume	8.89	1	8.89	37.40	< 0.0001	
<i>B-ply arrangement</i>	1026.51	1	1026.51	4316.59	< 0.0001	
C-orientation	254.56	4	63.64	267.61	< 0.0001	
AB	1.51	1	1.51	6.36	0.0244	
BC	800.52	4	200.13	841.57	< 0.0001	
A^2	13.45	1	13.45	56.57	< 0.0001	
Residual	3.33	14	0.2378			
Lack of Fit	3.33	9	0.3699			
Pure Error	0.0000	5	0.0000			
Cor. Total	2263.44	26				

Table 4.10 ANOVA for Quadratic model of tensile strength

The Model F-value of 792.00 implies the model is significant. There is only a 0.01% chance that an F-value this large could occur due to noise.

P-values less than 0.0500 indicate model terms are significant. In this case, A, B, C, AB, BC, A² are significant model terms. Values greater than 0.1000 indicate the model terms are not significant.



✤ Fit Statistics

Std. Dev.	0.4877	R^2	0.9985
Mean	13.18	Adjusted R ²	0.9973
<i>C.V.</i> %	3.70	Predicted R ²	0.9942
		Adeq Precision	88.6675

The Predicted R^2 of 0.9942 is in reasonable agreement with the Adjusted R^2 of 0.9973; i.e. the difference is less than 0.2. Adeq Precision measures the signal to noise ratio. A ratio greater than 4 is desirable. Your ratio of 88.668 indicates an adequate signal. This model can be used to navigate the design space.

Table 4.11 The final mathematical model in terms of the actual factors for tensile strength

Orientation	Ply arrangement	Equation, Ts
MMM	EWE	9.32811 + 0.432184 * <i>A</i> - 0.00367677 * <i>A</i> ²
	WEW	$-4.86138 + 0.397977 * A - 0.00367677 * A^2$
MUM	EWE	$11.0532 + 0.432184 * A - 0.00367677 * A^2$
	WEW	$-1.42695 + 0.397977 * A - 0.00367677 * A^2$
MRM	EWE	$7.41581 + 0.432184 * A - 0.00367677 * A^2$
	WEW	$-6.68695 + 0.397977 * A - 0.00367677 * A^2$
RUR	EWE	$-5.28522 + 0.432184 * A - 0.00367677 * A^2$
	WEW	1.30321 + 0.397977 * A - 0.00367677 * A ²
URU	EWE	18.9014 + 0.432184 * <i>A</i> - 0.00367677 * <i>A</i> ²
	WEW	$-7.31456 + 0.397977 * A - 0.00367677 * A^2$

Where:

M: mat or woven fiber orientation

R: random fiber orientation

U: unidirectional fiber orientation

EWE: ply arrangement with Enset wool Enset

WEW: ply arrangement with wool Enset wool



A: fiber volume ratio

Ts: Tensile strength

The equation in terms of actual factors can be used to make predictions about the response for given levels of each factor. Here, the levels should be specified in the original units for each factor.

4.5.2.4 Validation of the models

In figure 4.17 below shows the relationship between the actual and predicted values of the tensile strength. The residual tends to close to the diagonal line it shows that the developed models are valid and adequate. Since the residuals in the predictions of each response are small.



y = 0.9985x + 0.0199 $R^2 = 0.9985$

Figure 4.17 Predicted vs. actual graph of tensile strength model

A graph Actual vs. predicted helps to detect observations that are not well predicted by the model. The value of R- squared, $R^2 = 0.9985$ is a measure of the amount of variation around the mean explained by the model. The model is 99.85 % fitted with the variability in flexural strength.



4.5.2.5 Diagnostic report and plots

This section contains a description of each instance statistically. Taken directly from state ease DE.12.1 Software recommendation.

- *Run order*: the randomized order for the experiments
- ✤ Actual value: the measured response data for this particular run
- Predicted value: the value predicted from the model, generated using the prediction equation, includes block and center-point corrections, when they are part of the design.
- ✤ Residual: Difference between Actual and predicted values for each point
- Leverage: Leverage of a point varies from 0 to 1 and indicates how much an individual design point influences the mode's predicted values. A leverage of 1 means the predicted value at that particular case will exactly equal the observed value of the experiment, i.e. the residual will be 0. A leverage of 1 means that any error (experimental, measurement, etc.) associated with observation is carried into the model and included in the prediction.
- *Residual vs run*: it tests the assumption of constant variance. The plot shows a random scatter constant range of residual across the graph. Shown in figure 4.19.
- Predicted vs. run: it checks for lurking variables that may have influenced the response during the experiment. The plot should show a random scatter. Trends indicate a time- related variable lurking in the background. Blocking and randomization provide insurance against trends ruining the analysis. Shown in figure 4.20. the plot shows a random scatter as recommended.
- Box-Cox plot for power transforms this plot provides a guideline for selecting the correct power law transformation. A recommended transformation is listed, based on the best lambda value, which is found at the minimum point of the curve generated by the natural log o the sum of squares of the residuals. If the confidence interval around this lambda includes 1, then the software does not recommend a specific transformation. The current lambda and best lambda were 1 and 0.37 respectively, and it was in the 95 % confidence interval (-0.37, 1.12). Shown in figure 4.21. the recommended transformation also none.



Run Order	Actual Value	Predicted Value	Residual	Leverage	Standard Order
1	6.57	6.53	0.0450	0.433	3
2	5.82	6.29	-0.4722	0.433	1
3	4.35	4.35	-0.0017	0.433	24
4	1.89	1.80	0.0945	0.433	25
5	2.36	2.20	0.1578	0.433	16
6	5.03	4.93	0.1006	0.444	23
7	5.04	5.26	-0.2235	0.369	5
8	2.09	2.21	-0.1159	0.369	19
9	4.20	4.85	-0.6500	0.500	15
10	3.26	3.01	0.2472	0.444	17
11	5.50	4.85	0.6500	0.500	14
12	4.02	4.12	-0.0988	0.433	22
13	4.88	4.88	-0.0036	0.571	12
14	5.04	5.26	-0.2235	0.369	6
15	5.11	5.23	-0.1248	0.460	7
16	1.86	1.63	0.2318	0.476	21
17	2.03	2.44	-0.4050	0.433	18
18	5.53	5.47	0.0624	0.365	9
19	4.73	4.73	0.0000	0.500	11
20	2.09	2.21	-0.1159	0.369	20
21	5.12	5.12	0.0036	0.571	13
22	5.53	5.47	0.0624	0.365	8
23	2.51	2.61	-0.0961	0.444	26
24	4.90	4.45	0.4471	0.476	4
25	4.73	4.73	0.0000	0.500	10
26	7.53	7.10	0.4272	0.444	2
27	2.03	2.03	0.0016	0.433	27

Table 4.12 Diagnostic reports of flexural strength, Fs





Figure 4.18 Diagnostic plot of residual vs. predicted



Figure 4.19 Diagnostic plot of residual vs. Run







• Effect of process parameters on the strength of HFCM on model graphs



Figure 4.21 The effect of process parameter on flexural strength a) volume b) ply arrangement c) orientation of fiber on flexural strength



Interaction effect plot







✤ For Tensile strength





Figure 4.23 The effect of each design parameter on flexural strength



Interaction effect plot of tensile strength







4.5.3 Optimization

The optimization is a process of searching for a combination of factor levels that simultaneously satisfy the criteria placed on each of the responses and factors.

For this study, the optimization criteria are maximizing the strength of the HFCM. Therefore, the maximum strength of the material can be suggested with maximum flexural and tensile strength. The table below summarizes the criteria utilized to maximize the response.

Name	Lower	Upper	Lower	Upper	Importance	Goal
	Limit	Limit	Weight	Weight		
A:volume	30	75	1	1	3	is in range
B:ply	EWE	WEW	1	1	3	is in range
arrangement						
C:orentaion	MMM	URU	1	1	3	is in range
tensile strength	1.74	31.82	1	1	3	maximize
_						
flexural strength	1.86	7.53	1	1	3	maximize

Table 4.13 Constraints and optimization criteria used for design parameters of HFCM

Number	volume	ply	orientation	tensile	flexural	Desirability	
		arrangement		strength	strength		
1	56.112	EWE	MMM	22.002	7.125	0.791	Selected
2	55.842	EWE	MMM	21.997	7.126	0.791	
3	54.995	EWE	MMM	21.976	7.129	0.791	
4	55.811	EWE	MUM	23.721	6.069	0.736	
5	55.524	EWE	MUM	23.715	6.070	0.736	
6	55.245	EWE	MUM	23.708	6.071	0.736	
7	55.268	EWE	URU	31.556	4.955	0.736	
8	54.992	EWE	URU	31.549	4.955	0.736	
9	55.896	EWE	MRM	20.086	5.717	0.644	
10	56.179	EWE	MRM	20.091	5.716	0.644	
11	55.616	EWE	MRM	20.079	5.718	0.644	
12	54.223	WEW	MUM	9.342	4.756	0.359	
13	54.185	WEW	MMM	5.908	5.290	0.290	
14	54.468	WEW	MMM	5.908	5.290	0.290	
15	54.749	WEW	MMM	5.907	5.290	0.289	
16	54.170	WEW	MRM	4.082	4.876	0.204	
17	55.890	EWE	RUR	7.384	3.036	0.197	
18	54.310	WEW	RUR	12.072	2.232	0.150	
19	54.215	WEW	URU	3.455	2.633	0.088	
20	53.935	WEW	URU	3.455	2.632	0.088	

Table 4.14 Optimal solutions as obtained with design expert software

MSc. Thesis



The report solution gives to see which ones best meet the specified criteria. The desirability of 1 means the goals were easy to reach and better results may be available. In the highest desirability scores indicate there is an island of acceptable outcomes.



Figure 4.25 Graphical views of each optimal solution

The flexural strength and tensile strength of the optimum HFCM are 7.12 and 22 MPa respectively with MMM fiber orientation, EWE plies arrangement and 56.11 fiber volume ratio of design parameters.

Fiber orientation plays an important factor in the mechanical performance of HFCM. If the HFCM Fiber volume ratio had a significant effect on the strength of HFCM. The increased fiber volume ratio had a significant positive effect on the tensile and flexural strength of the HFCM up to getting saturated means the matrix can impregnate the fiber sufficiently. After saturation increasing, the fiber volume ratio had a negative effect on the strength of the HFCM.



Point prediction shows the prediction at the levels of the selected factors along with error estimates used to compute statistical intervals.

Solution	Predicted	Predicted	Std	SE	95%	95%	95%	95%
1 of 20	Mean	Median	Dev	Mean	CI	CI	TI low	ΤI
Response					low	high	for	high
					for	for	99%	for
					Mean	Mean	Pop	99%
								Рор
tensile strength	22.0023	22.0023	0.487653	0.32548	21.3042	22.7004	19.5955	24.4091
flexural strength	7.12479	7.12479	0.360039	0.240222	6.61277	7.63681	5.37072	8.87887

Table 4.15 Point prediction

SE Mean: the standard error of the mean, was the measure of the dispersion of means around the total mean.

Tolerance interval: is a statistical interval within which, with some confidence level, a specified proportion of sampled falls.

Confirmation of optimum factors level and response

Confirmation compares the prediction interval of the model to a follow-up sample average. If the average of the sample is inside the prediction interval, then the model is confirmed. It shows the factor setting used for the confirmation sample level and the limits for the area of interest from the design (low and high level). Confirmation with Two-sided confidence= 95% shown in the table below and compares the prediction interval of the model. That the prediction interval is between 95% PI low and 95% PI high to confirm the model.

For tensile strength, (20.7448 < 22.0023 < 23.2598) the model is confirmed.

For flexural strength, (6.20225 < 7.12479 < 8.04733) the model is confirmed.

volume



	1 2	0					
56.1125		VE	MMM				
Solution 1 of	Predicted	Predicted	Std	n	SE	95% PI	95% PI
20 Response	Mean	Median	Dev		Pred	low	high
tensile	22.0023	22.0023	0.487653	1	0.586296	20.7448	23.2598
strength							
flexural	7.12479	7.12479	0.360039	1	0.432821	6.20225	8.04733
strength							

Table 4.16 Confirmation location or optimum factors level and response

orientation

ply arrangement

It shows that fiber volume ratio '56.1 %', ply arrangement 'EWE', and orientation 'MMM' could be used to fabricate HFCM with maximum flexural and tensile strength. Therefore, this HFCM will be used for further investigation.

4.6 Composite machining and Fastening

In this section, the machinability of the designed HFCM and its characteristics during machining with currently available machines can be investigated. The surface of the machined edge will be presented and analyzed below.

4.6.1 Drilling of hybrid reinforced composite material

Drilling is a process of creating a hole in materials in order to join the parts through bolting or riveting.

In 50% fiber volume ratio, MMM, EWE of HFCM, as shown in the figure below, the drills bit first jerk the fibers and then shears them during the drilling operation. During that when the fiber pulls out it creates some void and twisted fiber in its surroundings. The fibers were not only strained from the drilled region but also drawn out from its nearby.







Figure 4.26 Drilled hole front and backside

In the back edge of the hole in HFCM built-in chips of fibers and the eroded surfaces were shown. The chips also found in the different long and short sizes of fibers. Better hole quality was observed in HFCM with a 50% fiber volume ratio.

For HFCM with WEW ply arrangement and MMM fiber orientation trim short wool fiber was observed in the inner side of the hole. This implies that the wool fibers are not enough wetted with the matrix. Wool epoxy bonds are weaker than Enset with the epoxy adhesive bonds.



Figure 4.27 Drilled WEW, MMM with 50% fiber volume ratio HFCM

For HFCM with a 75 % fiber volume ratio, drilling operation indicates some of the fibers were pulled out from nearby this might cause by not enough strength of fiber-matrix sticking. Delamination during drilling operation was existing Because of the generation of thrust force during drilling. If the generated thrust force exceeds the inter-ply bonding strength, then there is the formation of delamination around the drilled hole. There are two types of delamination peel up and push down delamination. Peel up delamination occurs during the entry of the drill bit. Peeling



force through the slope of the drill bit flutes separates the composite lamina and causes the formation of delamination around the entry side of the drilled hole. Push- down delamination occurs during the exit of the drill bit. As the drill bit approaches to the hole exit side, push down delamination is much dangerous as compared to the peel up delamination.

A sharp drill with a medium constant feed can produce a good quality hole. Always clean scissors are required to trim the pulled out fibers after cutting operation with post-curing. Drill-bit diameters, speed of the spindle and feed rate have a major influence in reducing the damage of a hole and quality of the hole in the HFCM. By appropriate selection of drill-bit diameter and angle, speed of the spindle, feed rate the machinability of the composite could be improved.

4.6.2 Mechanical cutting of composite

The cutting operation was conducted by using both hand hacksaw and motor-driven hacksaw. The pulled out fiber and small wear out of composite was shown when HFCM cut with motor-driven hacksaw especially in MMM, HFCM with a 75% fiber volume ratio. Hybrid fibers composite materials with WEW ply arrangement, the fibers were easily pullout and quickly wear out by high-speed motor-driven hacksaw.

In HFCM with MMM, EWE,50% fiber volume ratio better quality of edge was observed. Since there was better saturating of fiber with matrix in this composite material. Shown in the Figure below in Figure 4.28 a).



a)



Pulled out Trimmed fiber

b)

Figure 4.28 HFCM, MMM, EWE, with a) 50 % fiber volume ratio and b) 75% fiber volume ratio



For URU and RUR, hybrid reinforced composite material cut with motor-driven hacksaw with longitudinal cutting direction there was very little fiber pullout and wear of surface. Whereas, when the cutting direction was transversal to fiber orientation more fiber pullout and the non-smooth edge of the composite are shown. Toothless carbide saw blade with medium speed is recommended to machine the composite material by motor-driven hand hacksaw

When the HFCM cut with toothless hand hacksaw the composite damage was minimized almost less fiber pull out by tooth of hacksaw was seen. Sharp toothless blades should be used to minimize the composite damage during the cutting operations. Always clean scissors are required to trim the pulled out fibers after cutting operation and post-curing is recommending.

Generally, cutting the quality of the HFCM depend on the direction of cutting and fiber orientation. To get a good quality of edge and less damage of fiber the cuter should be parallel to the fiber orientation. The fiber direction of the composite should be taken in to account to ensure good surface quality.

4.7 Suggested area of application for HFCM

Structural materials should be sustaining external loads in addition to self-support. These structural materials are classified into the non-load bearing structures and load-bearing structures. Nowadays, natural fiber-reinforced composite materials are used for the structural and non-structural application.

In load-bearing structural material, the structure plays a role of supporting the structure of the final components like column and beam of the building, body of an aircraft, whereas, in non-bearing structural material enduring minimal load the primary functions are for aesthetic purposes like interior panels of a car, partition wall and so on.

To use natural fiber for these applications the primary consideration is its lightweight and can be tailored as user requirement as per design. In this study, the use of natural fiber-reinforced composites for gas cylinder application and refrigerator application will be investigated.

The suitability of the designed HFCM for the desired application can be investigated based on lab testing and computational modeling.



i. Car dashboard

New fuel economy and emissions standards are the driving challenge to automotive industries to use lighter material in the structure of the car body. The most important criteria that a material to be used in the car body are lightweight, economic effectiveness, safety in terms of impact load, aesthetic value and recyclability.

Steel, aluminum, magnesium, and its alloys are the Current most common material used in the automotive structure. Carbon- fiber composite material also used in some vehicle industries. To preserve optimum fuel efficiency, automotive industries need to use lighter material to minimize the Wight of the car. It also used to construct the non-load bearing element of the car like seat structures, bumpers, and hood.

Car dashboards are the basic structural element and play important roles in different aspects. Such as safety, appearance and as a box for the instrumental panel, audio and video devices, and switches. The basic criteria to the dashboard are mass of the dashboard, absorption capability of vibration and appearance.

Based on the design advantage of these composite materials this HFCM is recommended to use in the manufacturing of the car dashboard. This HFCM offers the advantage of high strength to weight ratio, its weight 50 % lighter than conventional steel and 30% lighter than aluminum, good to absorb vibration and resist it, good in stiffness and impact strength. It has also the advantage of improving fuel efficiency, improving safety and crashworthiness, design flexibility and easy to manufacture at the required shape. Durability and easy to maintenance is also an inherent advantage of composite material. This HFCM dashboard can absorb the vibration and its effect of produce noise during high speed and uneven road.

Besides that, this HFCM especially the wool has the advantage of creating a conditioned environment by maintaining humidity and temperature. Due to that, it is also favorable to use as interior ceiling and hoods.

ii. Refrigerators body

Due to its low conductivity characteristics, composites are good insulators and also Due to its high strength and lightweight it can also use as a refrigerator's external body, especially on portable vaccine refrigerators. Lightness is an significant feature to transport it from place to place.



Besides that, this HFCM has the advantage of dimensional stability. It can retain its shape and size in cold or hot or wet and dry conditioned. Unless the correct matrix was selected and painted with waterproof materials. It is also durable and easy to maintenance.

This HFCM can be used as refrigerator external body in substitution of aluminum and steel due to its above listed inherent characteristics such as lightweight, high specific strength to weight ratio, impact strength, dimensional stability, shock absorption, and good insulation.

Other suggested application

- For house appliances, like furniture and consumer goods cooking cabinet, tables, and other furniture.
- For construction, partition wall, roofing, blower housing, wall sleeves, roof panels, and HVAC insulation system
- Small scale wind energy as rotor blade material
- ✤ For energy, for solar panels and wind turbines blades and so on.



CHAPTER 5 CONCLUSION AND RECOMMENDATIONS

In this chapter, the major summary of the study, conclusions, and recommendations are made according to the objective. The content of each chapter and tasks result are also summarized.

5.1 Conclusion

- HFCM is successfully fabricated by using hand lay-up manufacturing techniques with Enset and wool fiber as reinforcement and epoxy as binding material. This hybrid composite gives good mechanical properties as compared with mono fiber composite.
- Fiber volume ratio (30%,50% and 75%), ply arrangement (EWE, WEW) and fiber orientation (MMM, MUM, MRM, URU, RUR) are the design parameters with respective level to develop the HFCM.
- From the water absorption test, composite with WEW, HFCM, having moisture absorption of 25% after 72 hr. dip in the water which is maximum. The more amorphous behavior of the wool, fiber volume ratio, porosity content, and fiber matrix-adhesions are responsible for the moisture absorption behavior of the HFCM.
- The thermal stability of HFCM is directly dependent on the thermal stability of both Enset and wool fibers. The result shows the major thermal decomposition of the fiber was in the range of 290 -330 °C.
- For 5% NaOH treated fibers, the diffraction intensity is better. During this chemical treatment removal of the non- cellulosic material. And It also modified the crystal arrangement in the fibers and minimize the hydrophilicity nature of the fibers. Not only that, but these NaOH chemical treatments also increase the crystallinity index of cellulose fiber and enhanced fiber to matrix interfacial bonds to improve the strength of the composite.
- From the tensile test, and flexural test result, fiber volume ratio, ply arrangement and fiber orientation was the principal parameters that affect the strength of the HFCM. Increasing fiber volume ratio from 30-50% results increasing the strength of the composite. whereas, increasing its volume above 50% shows the strength of the composite decline. Since excess fibers in composite materials create less wettability of fiber that means there is poor interfacial strength of fiber and matrix. This affects the stress transferring between fibers during tensile



and flexural loading. Due to that lower value of tensile strength and flexural strength was seen in 75% fiber volume ratio.

- The EWE ply arrangement has better strength. Since the mechanical strength of Enset fiber is better than wool fiber therefore, concentrating Enset fiber will result in better strength than concentrating wool in HFCM.
- Fiber orientation has a great impact on the tensile and flexural strength of the HFCM. From these MMM or HFCM with woven fiber orientation has maximum flexural strength than the other. EWE ply arrangement has also better strength than the WEW ply arrangement.
- From optimization result, fiber volume ratio '56.1%', ply arrangement 'EWE', and orientation 'MMM' could be used to fabricate HFCM with maximum flexural and tensile strength. The maximum values of tensile strength and flexural strength were 22. MPa and 7.2. MPa respectively. The result of this optimized model is in good agreement with some natural fiber composite models found in the literature.
- From the machining test, the above-considered parameters also had an undeniable impact on the surface quality of the machined edge and machining damage. Especially, fiber volume ratio, increasing fiber volume ratio above optimum value pulled out fiber and delamination of plies are observed. Since the fibers were not enough wetted with matrix to adhere strongly. Due to that, strained fiber from the drilled region and peel up and push down delamination were observed. However, in HFCM with MMM fiber orientation, EWE ply arrangement and 50 % fiber volume ratio, good quality of drilled hole and edge surface were shown with acceptable machining damage. Tooth less hacksaw is highly recommended during the composite cutting operation.
- Identifying these significant design parameters, and investigate its impact on the strength of the HFCM. Then, optimizing these parameter levels to design composite material with maximum strength. That can be used as an alternative to light-duty structural materials that fabricate from locally most available, lightweight, and fully effective and efficient materials were the main contribution of this research.



5.2 Recommendation for future work

From the conclusions of this research, the following recommendation is made for further studies in the future.

- In this work, the hand lay-up manufacturing technique was used to develop HFCM. By using another manufacturing process, it will be analyzed.
- In addition to the above-listed test, erosion, flame retardancy, chemical resistance and other tribological test are required to use this HFCM heart fully in gas cylinder manufacturing industries and another application area.
- Study on the effects of water absorption on the mechanical performance of natural fiber composites with respect to different fiber volume ratio and immersion time.
- Creep, wear, Dynamic and thermal properties study on natural fiber-reinforced composite material
- Study on the effect of the fiber length and fiber wrinkling on composite material for gas cylinder manufacturing.
- Investigate the effect of adhesive and mechanical bonding of the HFCM in mechanical performances.
- Additional machining operation and mechanical fastening investigation will be carried out to optimize the machining parameters and to discuss the effect of different assembling techniques.



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APPENDICES

Testing machine	Location	organization	Machines
Tensile testing machine with v- griping mechanism	Addis Ababa	Federal poly technique college, Ethiopia	Used for tough material, metal and so on.
Tensile test machine For soft material like polymer, plastic and textile limited to 5KN	Addis Ababa	Ethiopia , textile department	
Flexural test	Addis Ababa	ECAE	
TGA test	Jimma	JIT	



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XRD	Jimma	JIT	XRO-7000
Impact (digital charpy impact test)	Jimma	ЛТ	
Specimen conditioning equipment	Addis Ababa	ECAE	