

**Mechanical Property Analysis of Titanium diboride and Cactus (Kulkual)
Fiber Reinforced Epoxy Composite**



Keyredin Selman Kiyar

A Thesis submitted to the Department of Mechanical Engineering,
School of Mechanical, Chemical and Material Engineering

Presented in Partial Fulfillment of the Requirement for the degree of
Master of Science in Manufacturing Engineering

Office of Graduate Studies

ADAMA SCIENCE AND TECHNOLOGY UNIVERSITY

November, 2022 G.C

Adama, Ethiopia

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Advisor: Dr. Venkatesh Chenrayan

Co-Advisor Dr. Kiran Shahapurkar

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Candidate's Declaration

I hereby declare that this master's thesis entitled “**Mechanical Property Analysis of Titanium diboride and Cactus (Kulkual) Fiber Reinforced Epoxy Composite**” is my original work. That is, it has not been submitted for the award of any academic degree, diploma or certificate in any other university. All sources of materials that are used for this thesis have been duly acknowledged through citation.

Keyredin Selman Kiyar

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Recommendation of Advisor

We, the advisors of this thesis, hereby certify that we have read the revised version of the thesis entitled “**Mechanical Property Analysis of Titanium diboride and Cactus (Kulkual) Fiber Reinforced Epoxy Composite**” prepared under our guidance by **Keyredin Selman Kiyar** submitted in partial fulfillment of the requirements for the degree of Masters of Science in Manufacturing Engineering. Therefore, we recommend the submission of revised version of the thesis to the department following the applicable procedures.

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Approval of Advisor

We, the advisor of the thesis entitled “**Mechanical Property Analysis of Titanium diboride and Cactus (Kulkual) Fiber Reinforced Epoxy Composite**” developed by **Keyredin Selman Kiyar**, hereby certify that the recommendation and suggestions made by the board of examiners are appropriately incorporated into the final version of the thesis.

Advisor: Dr. C. Venkatesh

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Approval by Board of Examiners

We, the undersigned, members of the Board of Examiners of the thesis by **Keyredin Selman Kiyar** have read and evaluated the thesis entitled “**Mechanical Property Analysis of Titanium diboride and Cactus (Kulkual) Fiber Reinforced Epoxy Composite**” and examined the candidate during open defense. This is, therefore, to certify that the thesis is accepted for partial fulfillment of the requirement of the degree of Master of Science in Manufacturing Engineering.

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LIST OF ACRONYMS AND ABBREVIATIONS

ASTM	America Society for Testing and Material
C	Central
CMCs	Ceramic Matrix Composites
EK	Epoxy-Kulkual composite
DMA	Dynamic Mechanical Analysis
FRCs	Fiber Reinforced Composites
HV	Vicker Hardness
IJERT	International Journal of Engineering Research & Technology
M	Middle
MAPP	Maleic anhydride grafted polypropylene
MMCs	Metal Matrix Composites
NaOH	Sodium hydro-oxide
NFRC	Natural Fiber Reinforced Composite
NFRPC	Natural Fiber-Reinforced Polymer Composite
P	Peripheral
PE	Polyester
PMCs	Polymer Matrix Composites
PP	Polypropylene
PRPCs	Particle Reinforced Polymer Composites
PTFE	Polytetrafluoroethylene
RTM	Resin Transfer Molding
SEM	Scanning Electron Microscope
UTM	Universal Testing Machine
VIP	Vacuum Infusion Processing
VHN	Vickers Hardness number

NOMENCLATURE

cc	Centimeter cube
M%	Average moisture content
M _c	Mass of composite (g)
M _f	Mass of fiber (g)
M _m	Mass of matrix (g)
M _p	Mass of particle (g)
V _F	Volume fraction of fiber (mm ³)
V _M	Volume fraction of matrix (mm ³)
V _c	Volume of composite (mm ³)
V _f	Volume of fiber (mm ³)
V _m	Volume of matrix (mm ³)
vol.%	Volume percentage (mm ³)
wt.%	Weight percentage (g)
$\mathcal{P}_c$	Density of composite (g/cm ³)
$\mathcal{P}_f$	Density of fiber (g/cm ³)
$\mathcal{P}_m$	Density of matrix (g/cm ³)

ABSTRACT

Pollution is now one of the most difficult tasks to address since synthetic products threatens the environment because they are difficult to degrade and build up in landfills. Polymer composites containing varying quantities of fibers (weight percentage, wt.%) were developed and analyzed for their mechanical properties. In this research, mechanical and physical characterization (such as density, tensile, compression and bending behavior) of epoxy reinforced by naturally available cactus fibre were carried out with various analytical and experimental studies. The main goal of this research is to investigate the mechanical properties of cactus fiber-reinforced epoxy composites with TiB_2 filler. The fibers were obtained by drying the leaf-like stem of *Opuntia* and collecting the skeleton structure found in cladodes. The influence of chemically treating the cactus fiber with NaOH on its physical properties was investigated. The composites were examined under the guidelines of ASTM. Mechanical property analysis was done in accordance with standards and earlier studies was reviewed. All the composites tensile moduli rise with increasing fiber content and were between 9.92 and 93.78%. Tensile strength of the composites increases ranged from 0.15 to 107.32%, with 10 vol.% cactus fiber reinforcement showing the greatest improvement of 82.64 MPa. Out of all the composite materials, the one with 10 vol.% reinforcement had the greatest flexural strength of 53 MPa. With a rise in fiber percentage to the resin, the cactus fiber-epoxy composites of neat epoxy, 0, 2.5, 5, 7.5, and 10 vol.% fiber have an average hardness of 28.8 HV, 36.2 HV, 38.1 HV, 40.1 HV, 44.6 HV, and 45.9 HV, respectively. The composites' maximum impact strength observed was 168.3 kJ/m². With the increase of fiber percentage in the resin, the compressive strength of cactus fiber-epoxy composites for 0, 2.5, 5, 7.5, and 10 vol.% is 77.03 MPa, 83.14 MPa, 89.92 MPa, 91.45 MPa, and 92.47 MPa, respectively. The highest water absorption obtained was 2.47%.

Keywords: *Polymer matrix composite, Mechanical properties, Opuntia fiber, Alkaline treatment*

CHAPTER ONE

1. INTRODUCTION

1.1 Background

Pollution is now one of the most difficult tasks to address since synthetic products are a threat to the environment because they are difficult to degrade and can build up in landfills. The technique of improving certain properties of traditional materials has resulted in the way of reinforcing polymers with particles, fibers, and whiskers. A composite is a macroscopic merging of different materials, with a distinguishable interface involving at least two components. It is a combination of reinforcement and matrix. Reinforcement is the material used to make fiber. A matrix is used to bond fibers together and propagate the applied force to the reinforcement. Fiber-reinforced polymer composites are used in transportation, sports, electronics, healthcare, construction, packaging, and energy industries. For modern academics, incorporating natural fibers of various types has become a subject of focus and a new research field. These composites were the first choice in the field of materials due to their simplicity in manufacturing, inexpensive, and improved performance (Alberto et al., 2017). These advantages make it possible to design the composites for a variety of applications. By affecting both the economy and the environment, environmental protection or the use of commodities for such dual-beneficial purposes will offer additional value.

According to the latest discoveries, natural fibers could be employed as reinforcement in polymer composites, replacing highly costly and non-renewable synthetic materials (Castellano et al., 2021). Extensive study has been spent in recent years on examining and investigating the capability of diverse plant fibers to serve as composite reinforcement materials. Sisal, banana, cactus, hemp, pineapple, bamboo, and other fibers are examples. NFRC materials can replace synthetics in light-weight applications. NFRCs are bio-composites that offer a promising product alternative for ending the greenhouse effect and producing healthier plastic products. Plant fibers have several benefits over synthetic ones, including recyclability, good biocompatibility, and light weight (Li et al., 2020). The best thing about NFRCs is that they can be made from a wide range of plant feedstock all over the globe. Even if NFRCs have

considerable promise, certain disadvantages are weaker mechanical characteristics and more water-holding capacity when compared with synthetic ones. The interfacial fiber-matrix interaction is one of the variables that influence both mechanical properties and water uptake in NFRCs (Robertson et al. 2013). Because of the incompatibility of fibers and matrix, a weak link often occurs between the matrix and fibers in many NFRCs. Rachini et al. (2012) suggest that mechanical characteristics could be improved by using fiber treatment and binding agents. Polymer matrix composites containing varying quantities of fibers (weight percentage, wt.%) are manufactured and tested for their mechanical performance. Reports of experimental findings suggest that combining a certain amount of fiber with polymers increases the strength and stiffness of the composite (Sen et al., 2011). This research involves the manufacturing of cactus fiber reinforcement epoxy and testing for its mechanical characteristics. In this study, cactus fiber reinforced composites will be fabricated and their tensile, compression, flexural, hardness, and impact strengths will be determined.



Figure 1.1: *Opuntia ficus-indica* (cactus) in parts of the dryland of Ethiopia.

Drylands add up to 75% of the Ethiopian main land (Peng et al., 2021). One such type of plant species that can adapt to such kinds of environments is the cactus pear, *Opuntia ficus-indica* (locally called kulkual or beles). Cactus pear is local to Africa, Mexico, South America, and the Mediterranean but is found in different parts of the world's drier areas. Given its larger spread as well as the broad range of habitats it covers, *Opuntia* is one of the most adaptable cacti in its family. *Opuntia* species have been utilized across a wide range of situations, including fruits, vegetables, animal food, medical goods, agro-industry (i.e. fruit juice), cosmetics, dye (pigment)

manufacture, and fencing (Vigueras et al. 2001). It has an excellent capacity to adapt to drier environments. Cactus pears have broadly spread over central and northern Ethiopia (Belay et al., 2011). Villagers cultivate cactus inside their yards, however, the majority of the fruit collected for food comes from wild cactus. Cactus pear became part of the customs of the Tigray people. Fresh cactus fruits are consumed as food from June up to August and their Cladodes for animal food. Inside the cactus, there is a fiber web. Different methods are used to pull out this fiber, including water soaking. When the cactus completes its life cycle, it dries and loses its greenish color, departing from the fiber. In Ethiopia, the economic viability of *Opuntia ficus-indica* has yet to be explored, and the role of cactus resources in national economies is negligible. As a result, in order to add value to the cactus plant, it is necessary to figure out the fiber extraction technique, characterize the recognizable properties of fibers, and incorporate the cactus fiber into a more valuable form of composite materials that can be used in a variety of industrial applications. The treatment of cactus with water and NaOH has been used in this study, which covers the study of the mechanical behavior of fiber-reinforced and unreinforced epoxy. This fiber is added to the polymer in the shape of short, randomly-distributed fibers to assure that the fibers have a significant length to diameter ratio in order to have a good reinforcing effect (Scognamiglio et al., 2019).

1.2 Statement of the problem

Synthetic fibers are man-made fibers which are much denser than natural fibers. Due to synthetic fibers (such as glass, carbon, kevlar, etc.), the environment is being polluted beyond repair. These fibers are unfavorable to the surrounding nature because they are magnified by using chemicals. Chemicals which are used for manufacturing synthetic fibers are harmful and can enter into the ocean and the seafood or our home supply systems, such as the water supply, and cause health problems. A portion of these pollutants, which are byproducts of the industrial process, get washed into household water, and the majority of such compounds are unbreakable. Synthetic substances decay slowly, resulting in long-term contamination. Synthetic fibers also suffer from heat damage, which can cause micro-plastic pollution and they have poor insulation capacity. Hence, people who constantly face dangerous chemicals will begin to get sick, and they may also get lung diseases. Materials which are made from a mixture of gas and petroleum take years to disintegrate, which creates a continuous need for waste disposal. Products made of synthetic materials that are thrown away into the ocean endanger marine life. Synthetic products

present softer and long-lasting materials, but their everlasting consequence on the environment overcomes the benefits they provide. Global awareness about natural fibers' ability to boost the quality of life inspires industries to discover more novel ways.

1.3 Objective

1.3.1 Main Objective

The general objective of this thesis is to investigate the mechanical properties and fabrication of cactus fiber-reinforced epoxy composites with TiB_2 filler.

1.3.2 Specific Objectives

- To study cactus fiber reinforced epoxy composite with TiB_2 filler by varying weight fractions of the fiber content,
- To analyze properties of cactus fiber reinforced epoxy composites such as impact strength, hardness, tensile, compression, and flexural behavior,
- To study the water absorption behavior of composite specimens,
- To study density and void content of the composites and their morphological structure,

1.4 Scope

In this study, cactus samples were taken from Awash Melkasa, which were at the early stage of growth with a height of not more than 1 meter. Samples were collected by cutting the cactus tree and removing all the spikes from its surface. The research looked at processing variables like fiber weight fraction and random fiber orientation. For this study, chopped random fiber orientation with 5 vol.% TiB_2 particles and five different volume fractions of cactus fiber (0, 2.5, 5, 7.5, and 10 vol.%) were used. Epoxy composite reinforced with TiB_2 particle and cactus fiber was fabricated by using the open molding technique. By varying the content of the fiber, physical and mechanical characterization in terms of tensile, compression, impact, hardness and flexural behavior of fiber unreinforced epoxy and cactus reinforced epoxy, were carried out.

1.5 Significance of the research

The substitution of natural fiber for synthetic fiber has massive consequences for the composite material industry. It implies that because natural fiber is lighter in automobile body parts, there will be a big market for farmers who can cultivate natural fiber and workshops that can fabricate composite parts. Ethiopia has a lot of potential for cactus plant cultivation. The

resource is easily accessible and can be used to fabricate automotive interior components in Ethiopia and enhance the efficiency of automotive internal accessories. This will assist in reducing the significant number of unemployed people in Ethiopia by providing job opportunities and a source of income for the rural community. This thesis will inspire local industries to think about using this composite because it might be a better option than synthetic ones. Some of the additional significance are:

- Low density and high specific strength and stiffness composite production.
- To expand our understanding of native natural fiber reinforcement for environmentally friendly products with the aim of replacing synthetic fibers.
- Production of composites at a lower cost than synthetic fiber by investigating previous work in other countries and introducing them to our country.
- To use this research as a reference for future studies in manufacturing industries, and automotive industries including automotive weight reduction, which has a direct impact on fuel consumption.

1.6 Research question

- What are the properties of cactus fiber?
- What is the result of adding cactus fiber on the strength characteristics of the epoxy matrix? (What are the mechanical characteristics of an epoxy composite reinforced with cactus fibers and TiB₂ filler?)
- Can the composite be formed using short fiber and TiB₂ filler?
- What quantity of cactus fiber provides the greatest strength?

1.7 Limitation of the study

The open molding technique, which is prone to porosity and flaws, is used due to the unavailability of other methods and processing budget for the manufacturing of cactus fiber-reinforced epoxy composite. Another drawback was the absence of a compressive molding machine in our vicinity for accurately preparing composite sample molds. In this instance, the open molding technique was used in place of compression molding. The study was constrained

to fibers from cladodes harvested by hand from a single location. There wasn't a highly competitive experimental laboratory available for the characterization of natural fiber composites, nor was there a workshop for composite material fabrication. Because the material chopping machine was absent, the fiber was chopped manually to the desired dimension. The fiber extraction technique was limited to manual fiber extraction from the cactus plant.

1.8 Research Motivation

In Ethiopia, there is a growing need to replace products with lightweight, cheaper, and locally available resources. These kinds of resources can enhance product performance while saving foreign currency for imported goods. Thus, cactus, which is widely available in Ethiopia, can be the best choice for such applications. Increasing environmental concerns have had a significant impact on government policy in several nations, inspiring "greener" materials research.

1.9 Structure of the thesis

This thesis work comprises five chapters, each covering a specific topic of the thesis work. Chapter one comprises the background of the problem, objectives, scope of the thesis, significance of the thesis, and structure of the thesis work. Chapter two deals with a brief literature review comprising studies on previous similar works relevant to the present study. Chapter three is about the study areas in which this research is being conducted, methodology, and sample preparation. Chapter four covers the evaluations, discussion, and explanation of test results. Chapter five concludes the current research work's results and forwards recommendations. Detailed information about the test results is included in the appendix.

CHAPTER TWO

2. LITERATURE REVIEW

2.1 Introduction

Fiber Reinforced Polymer Composites are made up of fibers as the reinforcing material and polymers as the binder. Because of their excellent room-temperature characteristics, simplicity of production, and affordability, these composites are employed in the widest range of applications and in the highest amounts. The matrix serves several roles that define the composite's performance. It links the fibers intact, keeping them connected. When a force is exerted on the composite material, it is carried into the fibers, which are the primary load-carrying constituents, via the matrix, allowing the material to sustain tensile, compression, bending, and shearing loads. Due to the need to create materials with novel features that are also environmentally friendly, composite materials made of natural fibers have drawn a lot of interest. The industrial sector has seen an increase in the demand for vegetal fiber over the past several years (Thyavihalli et al. 2019). Its use is on the rise because, when compared to synthetic fibers, it is less dense (Pickering et al. 2016), cheaper (Mohammed et al. 2015), uses less energy, and is biodegradable (Bourmaud et al. 2009). *Opuntia* is a kind of cactus that has historically been harvested for its fruit, according to Vigueras and Portillo (2001). Cladodes, which are articulated stems, make up its primary body. These cladodes have spines, which are distinctive *Opuntia* features with a defensive purpose, the presence of which varies according to the species. The most popular techniques for extracting fibers from these plants are dew retting, water retting, mechanical extraction, burial processes, and chemical treatments (Sanjay et al. 2019). Several layers of wood make up the woody skeleton found inside *Opuntia* cladodes. To extract this skeleton, various methods have been examined.

Although it takes so long to process, fibers can be prepared using the water retting process with a higher elastic modulus. Chemical techniques speed up the process but prevent access to the fiber network since they only function with ground cladodes rather than the cladode skeleton in the studies that have employed them. The mechanical characteristics of fibers have not been described because of this. These experiments demonstrate that an acid solution aids in the

splitting of the fibers, resulting in a homogeneous structure and removing some of the lignin that coats the fiber's exterior surfaces (Cheikh et al. 2018). Cleaning with hot water causes pressing the fibers later difficult, and cleaning with 1% KOH at 50°C results in fibers with little structural damage than boiling with 7% KOH, 2% KOH, or NaOH (Victoria et al. 2012).

2.1.1 Overviews of composite

Metals, ceramics, polymers, and composites are the most well-known material classifications. A composite is a macroscopic merger of different materials with a distinctive interface involving at least two components, the merging of which creates collective qualities that vary from their ingredients. The exploration of composite materials is not just for their different characteristics from their constituents, but also for the fact that composites usually possess significantly superior qualities. By mixing at least 2 distinct phases, a new product with superior properties can be produced that outperforms its constituents. The result is synergistic. The two phases in a composite material are primary and secondary. The matrix in which the secondary phase is introduced is formed by the primary phase. Because it normally serves to strengthen the composite, the implantation is called a reinforcing agent (Bourmaud et al. 2009).

2.1.2 Classification of composite

Composite materials are categorized in a variety of ways. Some of the different classification techniques are based on the materials' sources, matrix type, type of reinforcement utilized, and so on.

- a. Classification based on sources/origin of the materials

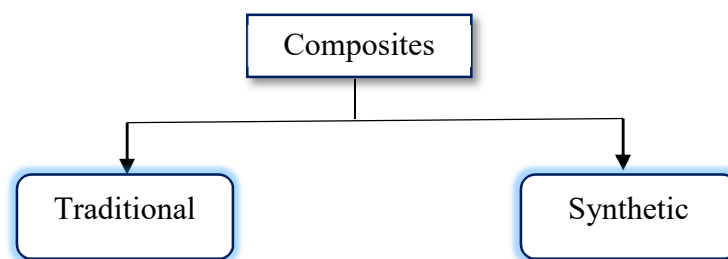


Figure 2.1: Classification of composite based on sources of the materials (David and Naidu, 2017)

Natural (traditional) composites are those which come from nature. Synthetic composites, on the other hand, are man-made and are enhanced through the use of chemical compounds.

b. Classification based on the primary phase (matrix)

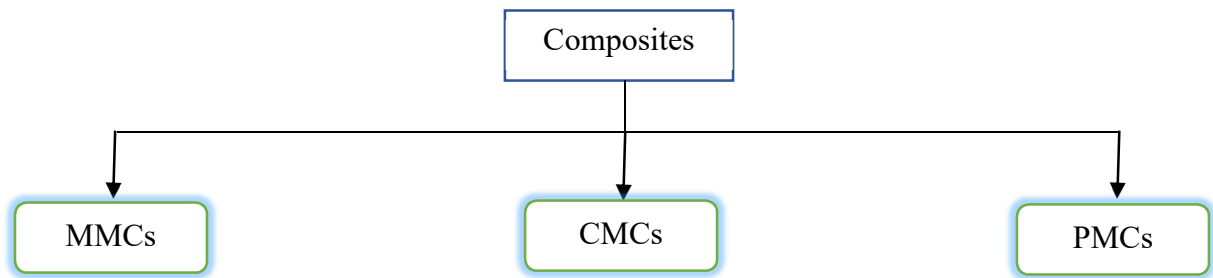


Figure 2.2: Classification of composite based on the primary phase (David and Naidu, 2017)

Metallic mixtures such as cermets, cemented carbides, Al, and Mg are used to make MMCs, which are reinforced by robust, high-strength fibers or ceramic particles. Other matrix substances, including titanium, copper, and iron, are being heavily utilized in MMCs. MMCs are categorized as being whisker reinforced, continuous fiber reinforced, or particle reinforced, depending on the kind of reinforcement employed. The biggest drawback of such composite materials is that they have higher densities and, as a result, inferior specific mechanical characteristics than PMCs. Another significant challenge is the enormous energy needed for the manufacturing of such composites (Mohammed et al. 2015).

CMCs are ceramic-based materials with a primary phase integrated into secondary phase. Alumina, boron carbide, boron nitride, silicon carbide, silicon nitride, and titanium carbide are some of the ceramic materials applied as matrices. Carbon, silicon carbide, and aluminum oxide are examples of fibers employed as secondary phases (Mohammed et al. 2015).

PMCs are made up of a polymer as the primary phase and a secondary phase embedded as fibers, particles, or flakes. In most industries, thermoset and thermoplastic PMCs are the most widely used polymer composites. Thermosetting PMCs cannot be restored to their previous condition once solidified. Since this thermoset cannot be reformed, only the reinforcing fiber can be retrieved from thermosetting PMCs (Groover, 2010). Thermoplastic PMCs, on the other hand, can be returned to their original state after solidification. Because they can be simply melted and remolded for new applications, thermoplastics are recyclable. This is how they can be utilized for an indefinite period of time (Mohammed et al. 2015).

c. Classification based on the secondary phase

The fiber size can be used to classify composites comprised of fibers reinforced into a matrix. Continuous fiber reinforced composites are those with long fiber lengths, whereas discontinuous fiber reinforced composites are those with short fiber lengths.

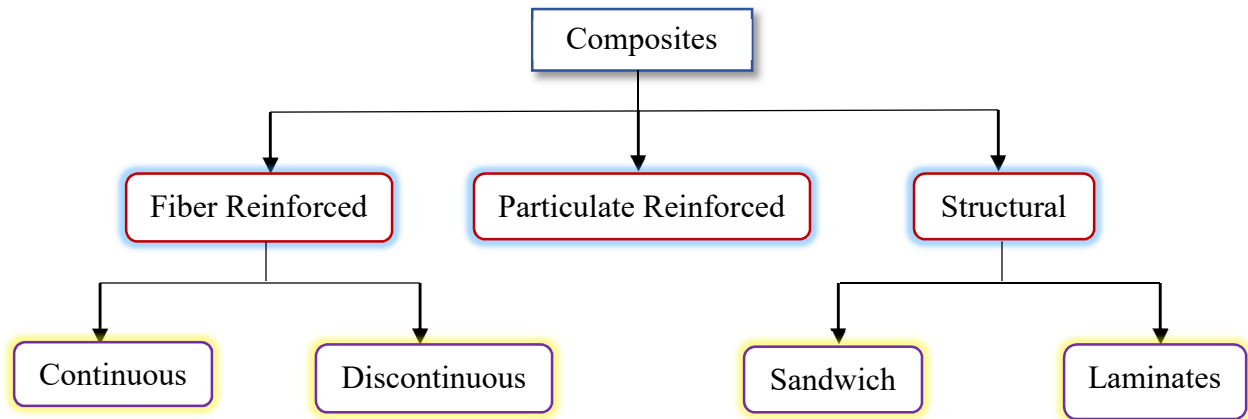


Figure 2.3: Classification of composite based on the primary phase (David and Naidu, 2017)

Particulate reinforced composites are made up of particles dispersed in a matrix. Because the particle distribution in the matrix is randomized, the strength and other properties of the composite material are often isotropic. The size of the particles has the greatest influence on the strengthening mechanism. Flakes are 2D particles that resemble tiny flat platelets. They are often less expensive than polymers and offer strength and stiffness to plastic molding compounds.

A structural composite is made up of at least 2 layers that have been bound with one another to create a single structure. The layers are usually, but not always, constructed from various materials. A laminar composite frequently uses various materials in its layers to benefit from integrating the unique qualities of each. A relatively thick core of low-density material is attached on both faces to thin layers of a separate material to create the sandwich structure. The low-density core could be foamed or honeycomb (Mohammed et al. 2015).

2.1.3 Natural fiber reinforced polymer composites

Because of the manufacturing industry's huge evolution, there is a demand for materials with improved qualities in regards to strength, rigidity, specific gravity, reduced price, and eco-friendliness with cleaner production. Fiber-reinforced polymer composites are lightweight,

high-strength materials that are used in a variety of applications including automotive, aviation, public transportation, sports, electronics, medical, building, packaging, and energy. To meet the needs of many of these applications, the characteristics of polymers are enhanced using fillers and fibers. In the past few decades, much emphasis has been placed on decreasing damage to the environment through the use of waste and abandoned pollutants in beneficial items.

Cellulose is a natural fiber that is usually used to reinforce polymer composites (Achaby et al., 2018). Cellulose is an abundantly available biopolymer made up of biomass on the planet, accounting for over 1/3 of a plant's chemical constituents, including hemp fiber, Alfa fibers, and tomato plant residues, among others. The technique relies on alkali treatment and is frequently suggested for separating cellulose and lignocellulosic networks (Mannai et al., 2018). Vegetal fibers are made up of both non-structural (waxes, fats, etc.) and structural elements (hemicellulose, lignin, and cellulose). This composition is affected by a number of parameters, including harvest time, climatic background, soil properties, vegetative or plant maturity level, fiber extraction method, and fiber characterization technique (Aruwa et al., 2018). Furthermore, the particulate material can be added to several polymeric matrices to save money and enhance properties. Several more composites now employ hard particles made of metal powders or ceramics to greatly enhance their properties (Das et al., 2016).

Natural fiber composites are replacing conventional materials without compromising their strength, and they have no negative environmental impact. Natural fibers have drawn the interest of researchers, scientists, and engineers in the past few years because of their unique advantages over synthetic fibers (Greco et al., 2013). The mixture of natural fibers and biopolymers resulted in the production of bio-composites, which degrade completely in soil (Lee and Wang, 2006). Natural fibers are also being used in place of synthetic fibers because synthetic fibers have a negative impact on the environment. Furthermore, research has demonstrated that introducing natural fibers into a poly (lactic acid) matrix enhances the composite's stiffness while lowering costs and improving environmental advantages, particularly concerning biodegradability. Natural fibers offer the advantages of being light weight, low density, cheap in cost, and eco-friendly (biodegradable and recyclable), but they also have drawbacks such as poor interaction with incompatible matrices and swelling qualities that cause cracks to form in inelastic matrices. Natural fibers' ability as reinforcement in polymer composites is significantly diminished by

their poor moisture resistance, inclination to agglomerate during processing, and inadequate interaction in hydrophobic polymer matrices. Because of the weak bond between the matrix and natural fiber, debonding and fiber pullout occur, which leads to the enhanced properties of polymer composites (Nayak et al., 2021). Such natural fibers have significantly reduced densities than E-glass fiber. As a direct consequence, some natural fibers have a very large specific strength. (Okubo et al., 2004).

2.1.4 Manufacturing Methods of Composite Materials

Depending on the geometry of the component to be produced, composite materials are fabricated in a variety of techniques. There are 2 types of composite fabrication techniques: open molding and closed molding. Reinforcement and matrix are placed in an open mold and hardened while subjected to air in an open molding method. The open molding method's tool costs are generally low, allowing it to be used for prototypes and short production runs. Hand lay-up, spray-up, and filament winding are all examples of open molding techniques. Reinforcement and matrix harden inside a two-sided mold or a vacuum bag in closed molding. Closed-molding methods are generally automatic and involve specialized equipment; thus, they are mostly used in major operations that create broad volumes of material. Closed molding comprises compression molding, pultrusion, vacuum bag molding, vacuum infusion processing, and resin transfer molding.

a. Hand lay-up

Reinforcements are manually put into the mold before resin is applied using a brush or roller. Hand lay-up can be used to manufacture small and huge products. However, this method is best suited for low-volume production. Of the open-molding processes, this is the cheapest and most frequently used method (Srinivas et al., 2017). This is due to the fact that, of all the operations in open molding, this one demands a minimal number of tools. The hand lay-up technique benefited from inexpensive tool costs and a huge variety of products. The drawbacks of this technique included that it was time-consuming, prone to generating trapped air, fiber disorientation, and production inconsistency, which were explored (David and Naidu, 2017).

b. Spray up

A chopper gun, a portion of specialized equipment, is employed in this technique. This specialized equipment aids in the chopping of reinforcement materials into small lengths and the subsequent addition of resin. Spraying pressurized resin and reinforcement in the form of chopped fibers is done with a spray gun. The polymer solution and reinforcement are sprayed one at a time (Srinivas et al., 2017). The chopped ingredients will be spread out on a molding platform. The curing procedure will take place at room temperature after spraying to the desired thickness. Following this, the product will be removed from the mold. This method is typically used to produce large quantities, and it is more automated than hand lay-up. The advantages of this technique include a continuous process, the flexibility to use any substance as a mold, and the capacity to fix errors simply by re-spraying, while the drawbacks include a low volume fraction and the ability to manufacture only the sheet as researched (David and Naidu, 2017).

c. Filament Winding

Filament winding is an automated method that involves wrapping uninterrupted threads of resin-saturated fiber reinforcement across a revolving cylindrical mold. It is used to make hollow products such as rocket motor shells, tubes, stacks, and chemical storage tanks (Srinivas et al., 2017).

d. Compression Molding

Compression molding is a fabrication method that involves composite materials being smashed between two solid molds and subjecting them to extreme temperature and pressure (up to 205 °C) until the component cures. Compression molding is recognized as a quick molding process and has great consistency. The operation is simple to automate. Compression molding is a high pressure and mass production process for rapidly forming complex polymer composite products. Compression molding reduces additional cutting and machining processes while keeping manpower costs to a minimum (Srinivas et al., 2017).

e. Pultrusion

Pultrusion is a technique for shaping composite materials into longer and more uniform sizes—for example, into rods. Uninterrupted yarns of fiber are immersed in a liquid polymer before being pulled into hot steel molds to shape the composite material into continuous lengths.

Pultrusion is a nonstop method of manufacturing items having a uniform geometry (Srinivas et al., 2017).

f. Vacuum Bag Molding

A vacuum is generated to drive air pockets and surplus resin from the laminate, compacting it (at least 2 layers of fiber reinforcement are bonded with a resin). Vacuum bag molding aids in the removal of extra resin that accumulates when structures are built using (open-molding) hand lay-up methods. Ambient air pressure exerts a force on the bag by lowering the pressure inside the vacuum bag. The pressure applied to the laminate eliminates trapped air and surplus resin while also compacting the laminate, causing a high percentage of fiber reinforcement (Srinivas et al., 2017).

g. Vacuum Infusion Processing (VIP)

VIP is a method of driving resin into a laminate using vacuum pressure. Vacuum infusion is commonly used to create very huge structures. Vacuum infusion generates durable, lighter laminates while reducing emissions greatly (in comparison to open-molding and wet lay-up vacuum bagging). The laminate is already crushed when the resin is forced into the mold, resulting in no space for more resin (Srinivas et al., 2017).

h. Resin Transfer Molding (RTM)

RTM, also known as liquid molding, is a closed-molding process where reinforcing material is placed into a closed mold, the mold is clamped, and resin is poured in under pressure (through injection holes). This technique gives complicated pieces with smooth surfaces on all exposed sides (Srinivas et al., 2017).

2.2 Review of previous researches

Bouakba et al. (2013) studied fabrication, quasi-static mechanical, and fatigue characterization of cactus fiber/polyester biocomposites with zig-zag-style fiber used to create the fiber sheaths of random orientation and three different fiber networks. For a half month, cactus cladodes stems were buried in soil that was 0.3 meters deep. This allowed CaC_2O_4 to ferment, which made it simpler to remove the fiber. *Opuntia ficus-Indica* was used to obtain the 3 fiber network structures (architectures), and no chemicals were used in the fiber extraction process. Following

water washing, the fibers are dried by air outdoors within 2 days. Fibers from the outside core make up architecture one, those from the intermediate part make up architecture two, and those from the inner core make up architecture three. Presumably because of a larger fiber density (0.75g/cc) and a lesser amount of mesh holes (lower porosity), exterior fibers had the highest mechanical properties (better elastic modulus, tensile strength, and deformation at break).

Mannai et al. (2018) investigated the removal, characterization, and mechanical, thermal, and morphological behaviors of fiber networks from the Cactaceae (*Opuntia*), and they found that the major fibers of the exterior area have lower pore sizes. Southern Tunisia was used to collect the *Opuntia ficus-indica*, and its fibrous systems were separated into several portions by using the water immersion method. The fibrous systems were manually removed from various roots after being submerged in hot water for two hours and kept in a sealed tank for more than half a month. The freed fiber portions were cleaned with clean water and allowed to dry for seven days at a temperature of 24 °C and a mean humidity of around 25 percent.

Mannai et al. (2020), investigated the use of a straightforward hand lay-up technique to reinforce polyvinyl alcohol and styrene-butadiene rubber matrices with *Opuntia* fibers of 3, 6, and 9 weight percentages. Due to correct interfacial bonding between the polymer and fiber, they were able to get acceptable thermomechanical and thermal properties as well as positive outputs regarding the composite's biodegradability. Manual labor was used to separate the fibrous networks, which were then allowed to air dry for a week at ambient temperature. The 56 layers that were discovered are a continuous phase that was found in the P, M, and C sections of the stem. In order to create fiber-reinforced polymer composites, such as cactus fiber/polyester and cactus fiber/polylactic acid, cactus fibers were employed as a natural filler.

Greco et al. (2015) studied looking at *Opuntia*'s application by polylactide matrix reinforcement. Due to its excellent mechanical qualities, which have been sustained for a long time even in moist places, polylactic acid is the most popular biopolymer. *Opuntia cladodes* and *Opuntia* fibers showed the same crystalline peak at $2\theta = 22.2^\circ$, due to crystalline cellulose. They discussed how to make slabs out of wood flour-*Opuntia*-PLA composites using cladodes. They discovered that the young's modulus of *Opuntia* fiber is higher in bending of 354 MPa than in tensile of 157 MPa.

Greco et al. (2013) created plates of composite by compression of polylactic acid with a thickness of 3 mm, wood flour (30 wt.%) and opuntia fibers (20 wt%) were combined. The mix was heated to 170°C in a forced convection oven and pushed with a force of 20 tons until it reached 180°C. Mechanical qualities were inferior to those of a composite made from wheat flour, flax, and PLA synthesized under the same circumstances. Opuntia composite demonstrated a modulus comparable to the matrix and a higher toughness due to higher resistance values (70 MPa vs. 48 MPa) and deformation (0.027 percent against 0.012 percent). The mechanical qualities are reported to change based on the type of matrix utilized, the amount of fiber, and also the production method.

Scaffaro et al. (2018) investigated the interaction between polylactic acid matrix and cactus fiber bio-composites' structure and their properties. The Italian cladodes utilized in this research were procured, and they underwent a series of washing, grinding, meshing, and vacuum-drying procedures in a furnace set to 90 °C for a minimum of twelve hours. The reinforcement and matrix were dried under vacuum for 12 hours at 90°C before being melted in a mixer for four minutes at 190°C and 64 revolutions per minute. Following this step, opuntia particles were sieved through sieve sizes ranging from 75-150 microns (A) and 150-300 microns (B).

Malainine et al. (2003) found that Opuntia spines have a possibly higher content of crystalline cellulose. Alkaline processing can be a useful option; pure cellulose from Opuntia was already obtained using NaOH, and the amount of cellulose was increased from 21 to 63 percent. Thus, it's indeed reasonable to assume that alkaline treatment will enable the separation of hemicellulose and lignin as well as other contaminants from Opuntia fibers and enhance matrix compatibility with the fiber.

Malainine et al. (2004) studied the water uptake and mechanical properties of composite materials that included powdered cladodes of opuntia as a chip filler and a poly (propylene) matrix. They collected opuntia cladodes in Marrakech, Morocco. They were cut into 1 cm cube sides and heated at 105 °C for 4 hours. Due to the fact that this drying period was long enough to balance the fiber's weight, it was considered that very little moisture was still present. The pieces were afterward crushed to fit through a sieve with a 100 mm gap. They examined the impact of using Opuntia cladodes for compression-molded Polypropylene (PP) matrices' reinforcement. They used a compatibilizer chemical to increase the adhesion of reinforcement-

matrix while they mechanically, thermally, and morphologically characterized the composites. A commercial compatibilizing chemical, Maleic anhydride-grafted polypropylene, with 1 wt.% of MAPP and physical properties similar to polypropylene (melts at 175°C), was used. It was mixed with ground cladodes of less than 100 microns.

Zampetakis et al. (2018), studied polypropylene discs with a diameter of 40 mm and thickness 10 mm, reinforced with Maleic anhydride-grafted polypropylene and 0.4 weight percent Opuntia fiber powder. Sigma Chemical Company and Aldrich Chemical Company provided the polypropylene that was used. Steel balls were used in a steel vessel during the spinning ball milling of the fiber to create the fiber flour. These discs, which were created through hand mixing and pressed at 190°C.

Scognamiglio et al. (2019) studied Opuntia plants' fibers, which were found in Anguillara, Italy. The Opuntia leaves were first scrubbed with a brush and pressurized air before being cleaned, scraped, and then chopped into segments around 20 mm in size. Afterwards, they were exposed to ultrasonic vibration in clarified water for half an hour, dried in an oven for a day, and then ground for two hours in a ball mill. The fiber length produced in their ultimate forms did not exceed 1 mm. Consequently, it was recommended that the fibers be crushed to a much slightly reduced size, such as 1 mm. They assessed the effectiveness of using Opuntia to strengthen a thermoplastic film made of starch and glycerol. To make the film, 10 grams of potato starch, 4 milliliters of glycerol, and 400 milliliters of distilled water were combined and stirred for 10 minutes. To retain the mixture, a Teflon-coated steel mold was employed for 12 hours at 45°C and 1 hour at 70°C. The films were made by swirling for different amounts of time while adding 8 and 16 weight percentages of opuntia. The production of three different forms of thermoplastic starch films which introduced powdered cactus fibers included composites with 16 weight percent of fiber stirred for one hour (A), composites with 16 weight percent of fiber stirred for five hours (B), and composites with 8 weight percent of cactus fiber (C). In comparison to pure thermoplastic film, the mechanical characteristics of films containing opuntia are reduced; this could be attributed to ineffective reinforcing and the use of too big and variable-sized fibers (which is smaller than 1 mm).

Lahouaria et al. (2018) investigated the DMA, flexural modulus, and tensile strength of a Cactus fiber reinforced epoxy composite. The extraction procedure revealed three distinct fiber

structures, each of which corresponds to a separate section of the plant's trunk. Architecture #One is fibers taken from the inner part, Architecture #two is fibers from the intermediate region, whereas Architecture #three is fibers taken from the outer. In this research, steam contact and chemical retrieval were applied to remove the fiber. The retrieval process involves placing the trunk in the ground not more than 0.3 m. The fermentation was accomplished throughout the one and a half month, which made it easier to remove the fiber.

According to Arévalo et al. (2010), tensile test findings reveal a rise in the composites' young's modulus, demonstrating effective reinforcing. Since the fibers are stiffer and hence less distorted, elongation generally decreases. Opuntia fibers were injected into a high-density polyethylene matrix to enhance it, increasing the matrix's young's modulus of elasticity by 36 percent.

Yuan et al. (2017) investigated the thermal and mechanical properties of TiB₂ filled hybrid composites used in high-temperature tribological applications. To investigate temperature, tribological, and mechanical characteristics, TiB₂ particles of various sizes were incorporated into hybrid composites of Nomex and PTFE fibers supplied by DuPont Plant, China. Xing-Guang Chemical Reagent Co., Ltd. in China supplied the phenolic resin. Meryer Chemical Technology and Alfa Aesar Chemicals Co., Ltd., both in China, supplied TiB₂ powders having average diameters of 4 μm and 47 μm. The fabric was washed chemically and dried at 50 degrees Celsius. The two types of TiB₂ particles were then introduced in various weight fractions into the resin, and the particles in the matrix were equally stirred with the help of ultrasonic. A Rockwell hardness test was done on every specimen with a spherical indenter, and 10 indentations were obtained to get an average result. A UTM was used to determine the tensile characteristics of the hybrid composites. Tensile testing was performed at a rate of 50 mm/min. The average result of at least 3 experiments was used to determine the tensile property for every sample.

Chary and Mohan (2017) studied the influence of TiB₂ addition on the mechanical properties of an epoxy-resin composite. The research looked at how nano-crystalline TiB₂ particles affect the mechanical behavior of TiO₂ powder-reinforced epoxy composite materials. Ciba Geigy India Ltd. supplied both the LY556 epoxy resin and the HY951 hardener. The resin and bonding agent are combined in a 10:1 weight ratio. TiB₂ and TiO₂ powders of 200-micron dimensions were

purchased from India. The hand lay-up method was used to manufacture epoxy composites. To achieve uniform mixing, the TiO_2 particles were combined into the epoxy-hardener mixture with a 2, 4, 6, 8, and 10 volume fraction and stirred continuously. Prior to being reinforced in resin, TiO_2 and TiB_2 particles were carefully milled together by using a mortar mixer for 15 minutes. A Vickers hardness testing machine has been utilized, with a dwell period of 30 sec for indentation. Every sample had ten indentations, with the average hardness value recorded. A UTM was used to determine the tensile and flexural characteristics.

Chary et al. (2017) studied the influence of TiB_2 addition on the mechanical and thermal properties of a ZrO_2 -epoxy composite. Ciba Geigy India Ltd. supplied all of the LY556 epoxy resin, ZrO_2 powder, TiB_2 , and HY951 hardeners. The resin and hardener are combined in a 10:1 weight ratio. The hand lay-up method was used to manufacture composite material. To guarantee consistent mixing, the ZrO_2 particles are mixed in the epoxy-hardener mixture at a ratio of 2, 4, 6, 8, and 10% by volume.

2.3 Summary

Table 2. 1: Summary of literatures

Author and Year	Reinforcement	Matrix	Fiber extraction	Manufacturing Process	Results
Mannai et al., (2020)	Opuntia fiber Wt. %: 3, 6 & 9	Polyvinyl alcohol & styrene butadiene rubber	Manually after drying	Hand lay-up	Young's modulus: 1.83 GPa
Scaffaro et al., (2019)	Opuntia powder ρ : 1.54 g/cm ³	Poly-lactic acid	Water retting	Compression molding	Tensile modulus: 2.5 GPa & Tensile strength: 55 MPa
Scognamiglio et al., (2019)	Opuntia fiber Wt. %: 8 & 16	Thermoplastic starch	Water washing and brushing	Open molding	Tensile strength: 3.75 MPa & % elongation: 23.4
Lahouaria et al., (2018)	Cactus fiber	Epoxy	Burying then washing	Molding	Tensile modulus: 0.37 GPa & Tensile strength: 15.5 MPa

Mannai et al., (2018)	Opuntia fiber ρ : 1.54 g/cm ³	-	Manually after water immersion	-	Tensile modulus: 2.93 GPa, flexural modulus : 2.36 GPa
Zampetakis et al., (2018)	Cactus powder wt. %: 0.4	Polypropylene	Ball-milling	Hand lay-up	Compressive module: 2.8 MPa, specific flexural modulus: 2831 MPa
Greco et al., (2015)	Opuntia ρ : 0.94 g/cm ³	Poly-lactic acid	Manually extracted after boiling	Rotational molding	Flexural modulus: 354 MPa & tensile modulus: 157 MPa
Bouakba et al., (2013)	Cactus fiber ρ : 0.75 g/cm ³ wt. %: 25%	Polyester	Burying underground & washing	Low pressure Compression molding	Tensile modulus: 1.48 GPa , Tensile strength : 15.8 MPa, flexural modulus: 7.95 GPa, flexural strength: 66.5 MPa
Greco et al., (2013)	Opuntia fibers ρ : 0.94 g/cm ³ wt. %: 20% flax fiber	Poly-lactic acid & wood flour	manually extracted	Compression molding	Flexural modulus: 4.6 GPa & flexural strength : 70.3 MPa
Arevalo et al., (2010)	Cactus powder Wt. %: 15%	High Density Polyethylene	Drying then grounding	Injection molding	Tensile modulus: 1.3 GPa, tensile strength: 20 MPa
Malainine et al., (2004)	Opuntia powder Wt. %: 0-70% ρ : 1.5 g/cm ³	Poly(propylene)	Drying	Compression molding	Tensile modulus: 260 MPa, tensile strength: 10 MPa & Max Water Uptake: 26.27%

2.4 Research gap

The most commonly produced materials in industries are polymers, which include thermoplastics (polyethylene, polypropylene, poly (ethylene terephthalate), polyamide, poly (lactic acid), etc.) and thermosets (epoxy, phenolic, polyurethane, etc.). This is mostly due to their low cost, lightweighness, and simplicity in manufacturing. And yet, due to their toxicity and lack of biodegradability, they are among the main contributors to the world's pollution. By significantly increasing worldwide waste generation and contamination at an astonishing speed, synthetic polymers pose a hazard to public health. Improvements are imperatively needed if the use of these materials is to be sustained. Natural fibers can be added to the composites as a means of enhancing the material. The plant *Opuntia ficus-indica* is regarded as invasive. Despite its untapped potential, *Opuntia* is currently not utilized in Ethiopia for any composite purposes. *Opuntia ficus-indica* has a high potential to be a viable filler in the composite field due to its inexpensive price, good characteristics, biodegradable nature, and availability.

Research on several kinds of *Opuntia ficus-indica* powder as reinforcement for polymer matrices has been performed. However, the primary goal of the current work, which has not yet been reported, is the use of short-form cactus fiber as reinforcement in epoxy matrix composites with TiB_2 filler particles. Research and experimentation were conducted in order to determine the characteristics of an epoxy composite reinforced by an indigenous species of cactus fiber (*Opuntia ficus-indica* fiber) with TiB_2 filler.

CHAPTER THREE

3. MATERIALS AND METHODS

The materials and procedures employed for carrying out the experiment, as well as the testing techniques and characterizations undergone, are all thoroughly explained in this chapter.

3.1 Materials

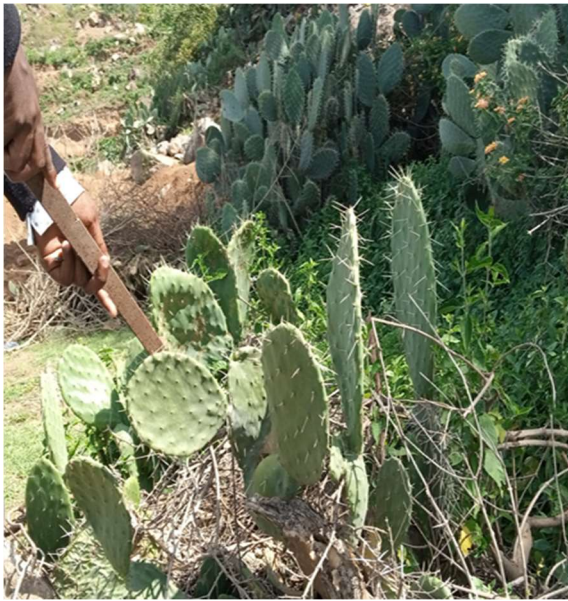
3.1.1 Cactus fiber

Opuntia ficus-indica (locally known as beles/kulkual) has been investigated in this study with the goal of utilizing Ethiopia's largely untapped resources. A few studies, based on the literature, report the manufacturing and application of cellulose from cactus species. *Opuntia* is a cactus (non-forest and home-grown plant) native to subtropical, tropical, semiarid, and arid areas that grows as a shrub and has an original-looking/unique morphology, reaching up to 5 m in height and producing a durable trunk when it gets older (Mannai et al., 2020). The fiber was collected from Awash Melkasa, which is located in Adama Wereda, Oromia's East Shewa district. It lies at an altitude between 1300 and 1800 m and is found at 8° 24' 0" North latitude and 39° 19' 59" East longitude. The area is classified as semi-dry, with unpredictable rain and recurring drought, as well as a hard cropping condition. The town's typical lowest and highest monthly temperatures are 20 and 35 degrees Celsius, respectively (Yihun et al., 2013).

Cactus fiber preparation

A suitable quantity of cactus cladodes was collected in June 2022 from the local area of Awash Melkasa after cutting at their base from the cactus tree. The fibers were manually removed from the leaf-like stems through hand removal by using sharp material to remove their spines. The removal operation was done by hand, and after that, it was dried for three days at room temperature. The fibrous structure was separated from various parts of the cactus cladodes. The fiber connections were carefully extracted from the various cladodes after they had been submerged in water and kept in a tank for 6 hours. The freed fiber layers were then thoroughly cleaned and dried for two days under ambient conditions, with an average temperature of about 25°C. The fibers were chopped into shorter ones (approximately 1 cm in length and a diameter of 300-400 microns), and then they were left to dry for a whole day. This amount of drying time

was adequate to balance the filler's weight. The residual water content was indeed thought to be quite low.



(a)



(b)

Figure 3.1: collecting of cactus (a) cactus plant, (b) cactus cladodes



(a)



(b)

Figure 3.2: extracting the cactus fiber (a) splitting cactus cladode, (b) dried cactus cladodes

Figure 3.1 shows the cactus cladodes are collected at their early stage from the cactus plant and then their spines are removed by using a sharp tool.

Figure 3.1 shows the cactus cladodes are collected at their early stage from the cactus plant and then their spines are removed by using a sharp tool. Figure 3.2 shows a cactus cladode which is split into two after being pressed inside a sheet metal bending machine. This cladode is then allowed to dry and the fibrous network is pulled out from the rest of the other parts.



Figure 3.3: cactus fiber preparation (a) cactus fiber pulled out from the cladode, (b) cactus fiber immersed in 0.1N of NaOH

Figure 3.3 shows a fibrous network that is pulled from the cactus cladode and then immersed in a bath of 0.1N NaOH. Every 100 ml of sodium hydroxide was mixed with 1 liter of water. NaOH is used to remove the fiber network from the lignin. The NaOH treatment enabled parts other than the fiber (the hemicelluloses and surface impurities) to be separated from the fiber, and a very clean and good-looking fibrous structure was acquired. To obtain optimum reinforcement, it is generally recognized that a good fiber-matrix interface bond is essential. Alkaline treatment is among the most effective approaches to improve fiber-matrix interaction. Chemical treatment using NaOH is a widely used approach in the literature; it eliminates the lignin, hemicellulose, and amorphous components, resulting in a roughened fiber surface (Sanjay et al., 2019). Natural fiber materials were simply treated with a NaOH 1M solution at ambient temperature. The extracted fibers were cleaned using distilled water and dried at room temperature. When compared to the untreated fiber, the treated fiber showed approximately a 3 to 20% increase in diameter and about a 15% increase in mass, measured with a digital caliper with 0.01 mm accuracy and a digital balance with 0.001 g accuracy.



Figure 3.4: cactus fiber after being chopped



Figure 3.5: SEM image of cactus fiber

Table 3. 1: Properties of cactus fiber

Physical properties	
Density	0.84 g/cm ³
Shape	irregular
Color	yellowish
Length	10 mm

3.1.2 Titanium diboride particles

These are microparticles that are used to enhance the mechanical plus surface properties of the composite materials. Many studies claim that this particle has the ability to increase the interfacial adhesion between epoxy and fiber. The particles utilized here are 75-120 microns in size.

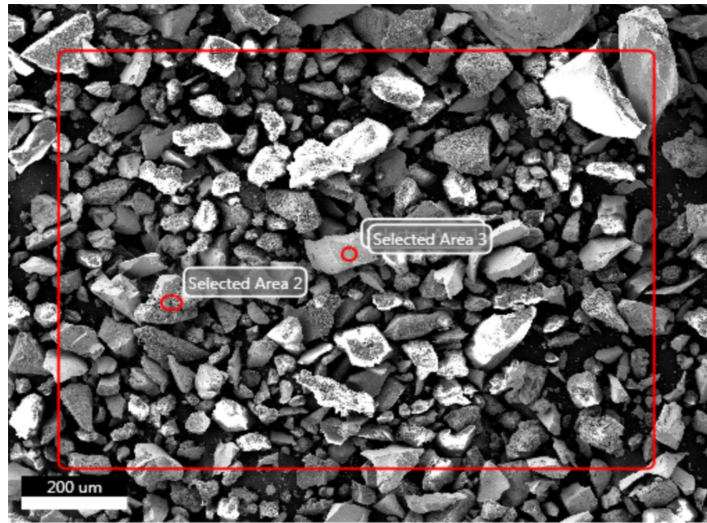


Figure 3.6: SEM image of TiB₂

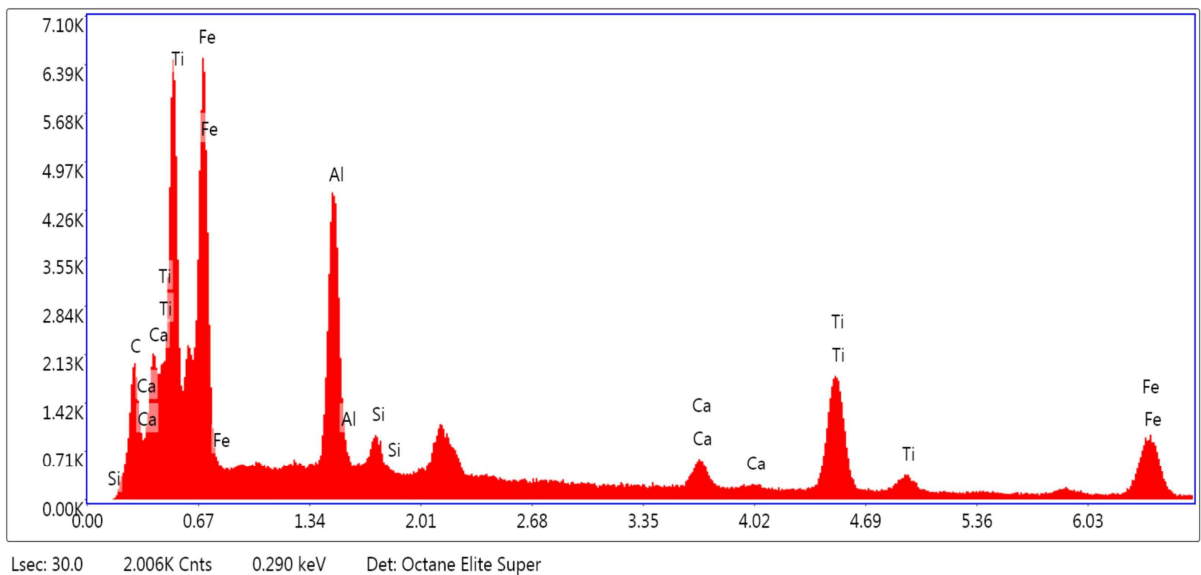


Figure 3.7: XRD of TiB₂

Figure 3.7 illustrates the XRD analysis of TiB₂. The spectrum contains a peak of Ti.

Table 3.2: Elemental composition of TiB₂.

Element	Weight %	Atomic %
C K	4.26	13.68
FeL	26.85	18.53
AlK	15.80	22.57
SiK	2.60	3.56
CaK	6.61	6.36
TiK	43.87	35.30

3.1.3 Matrix

Table 3.3: Properties of lapox L12 epoxy resin and K6 hardener.

Lapox L-12 epoxy resin		
Description	Measurement unit	value
Viscosity at 25 °C	MPa	11850
Volatility at 105 °C/h	Percentage	40
Colour	Blue	-
Epoxy value	Equivalent/kg	5.4
K6 hardener		
Description	Measurement unit	value
Viscosity at 25 °C	MPa	10
Colour	Colorless	-
Size	gram	500
Moisture content	%	1

The matrix material used is Lapox L-12 epoxy resin with K6 bonding agent, provided by World Glass Fiber Plc, Addis Abeba, Ethiopia. When combined with different hardeners, Lapox L-12, a liquid, unaltered moderate viscosity epoxy, can be used to create composites. Low viscosity liquid hardener with room-temperature curing is known as K-6.



Figure 3.8: Epoxy resin and hardener for sample preparation

3.1.4 Composition

The volume percentage compositions of cactus fiber and titanium diboride in the composite were established using reviews of the literature and trial testing. The designations for the samples manufactured with various wt.% of fiber and filler are included in Table 3.3 and Table 3.4.

Table 3. 4: Nomenclature of different sample composites

Designation	Vol. % of fiber	Vol.% of TiB_2	Total Wt. (g)	Mass of epoxy (g)	Mass of fiber (g)	Mass of Hardner (g)
EK	0	5.00	145.92	145.92	0.00	14.59
EK2.5	2.5	5.00	145.92	142.83	3.09	14.28
EK5	5	5.00	145.92	139.96	5.96	14.00
EK7.5	7.5	5.00	145.92	137.28	8.63	13.73
EK10	10	5.00	145.92	134.78	11.14	13.48

While EK stands for Epoxy-Kulkual composite, E stands for neat epoxy, and the number denotes the volume percentage of the fiber introduced to the composite. On two different molds, mold number one (101x101x12 mm) and mold number two (302x302x3 mm), the samples were created using equivalent volume percentages.

3.1.5 Mold preparation

Steel, steel plate, and bolts were readily available resources that were used to create 2 different molds. Steel was used to create two mold cavities of differing sizes, each measuring 101 x 101 x 30 mm and 302 x 302 x 30 mm, respectively. The composite was cast into the molds at the correct size to prepare the samples. Utilizing acetone of the highest caliber and paper towels, the molds were completely cleaned. Acetone is a multi-purpose solvent and all-purpose cleanser. To make it simple to remove the sample, petroleum jelly was coated on the surfaces of the mold cavity. A cheap and readily available release agent is petroleum jelly. Additionally, it aids in bridging the space between the steel and the steel plate.

3.1.6 Composite preparation

In an epoxy resin matrix, short cactus fiber and TiB_2 filler are used as reinforcement. The TiB_2 filler has a density of 4.52 gm/cc and an average particle size of 100 to 120 micrometers. By adjusting the fiber content (0, 2.5, 5, 7.5 and 10 vol. %) and fiber's length (10mm) with constant filler content (5 vol. %), composites of different proportions were created. Properly measured in digital balances, the epoxy and kulkual fiber were then hand combined with a glass rod in a glass beaker. The mixture is mechanically agitated for uniform dispersion and to prevent powder agglomeration, and then a magnetic stirrer set to 200 rpm is employed for 40 minutes. A thin layer of releasing agent was applied to the interior surfaces of the molds before the mixture was poured inside. The composite was made with the open mold casting method, where it's a process of pouring a mixture into a mold and letting it cure.



(a)



(b)

Figure 3.9: Constituents used for the composite (a) measuring fiber in electronic balance (b) powder TiB_2 filler.



(a)



(b)



(c)

Figure 3.10: mixing and pouring the ingredients (a) epoxy resin in a beaker (b) mixture of powder TiB_2 and epoxy (c) Poured mixture in the prepared mold.

3.1.7 Curing and removal of the sample

After a day of curing, the samples were ready to be extracted from the molds with ease. As TiB_2 powder concentration and particle size increased, agglomeration and sedimentation became more and more important. Higher EK compositions showed minor flaws and cracks. The dispersion and agglomeration of the particles were significantly influenced by the size and irregularity of the particles as well. Magnetic stirrers were used to mix materials, which

increased homogenization and decreased sedimentation. Every composite material's mold is cured, and the samples with the proper dimensions are sliced with a hacksaw for different tests.

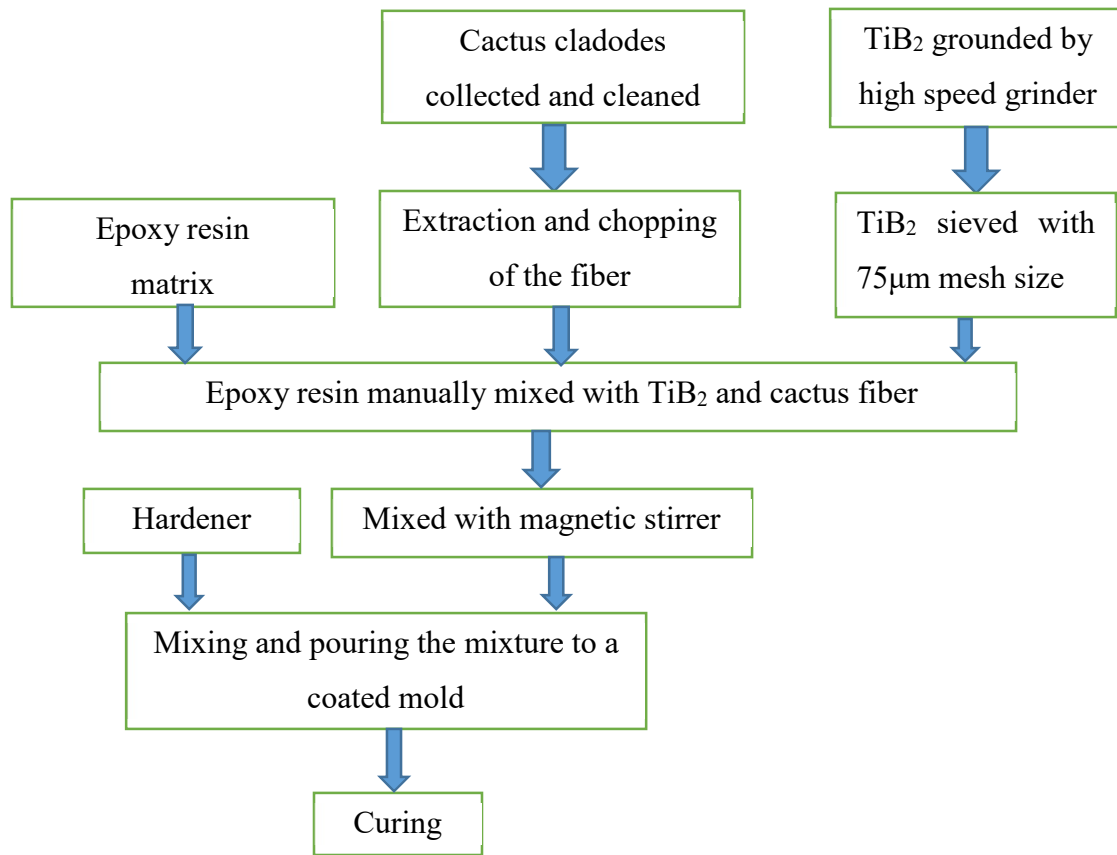


Figure 3.11: Process flow chart of epoxy reinforced with cactus fiber and TiB₂ powder

3.2 Methodology

The cactus fiber and TiB₂ filler were examined for this research, and the samples were made by guidelines of ASTM. Additionally, testing is done in accordance with standards and carefully considered presumptions derived from earlier studies.

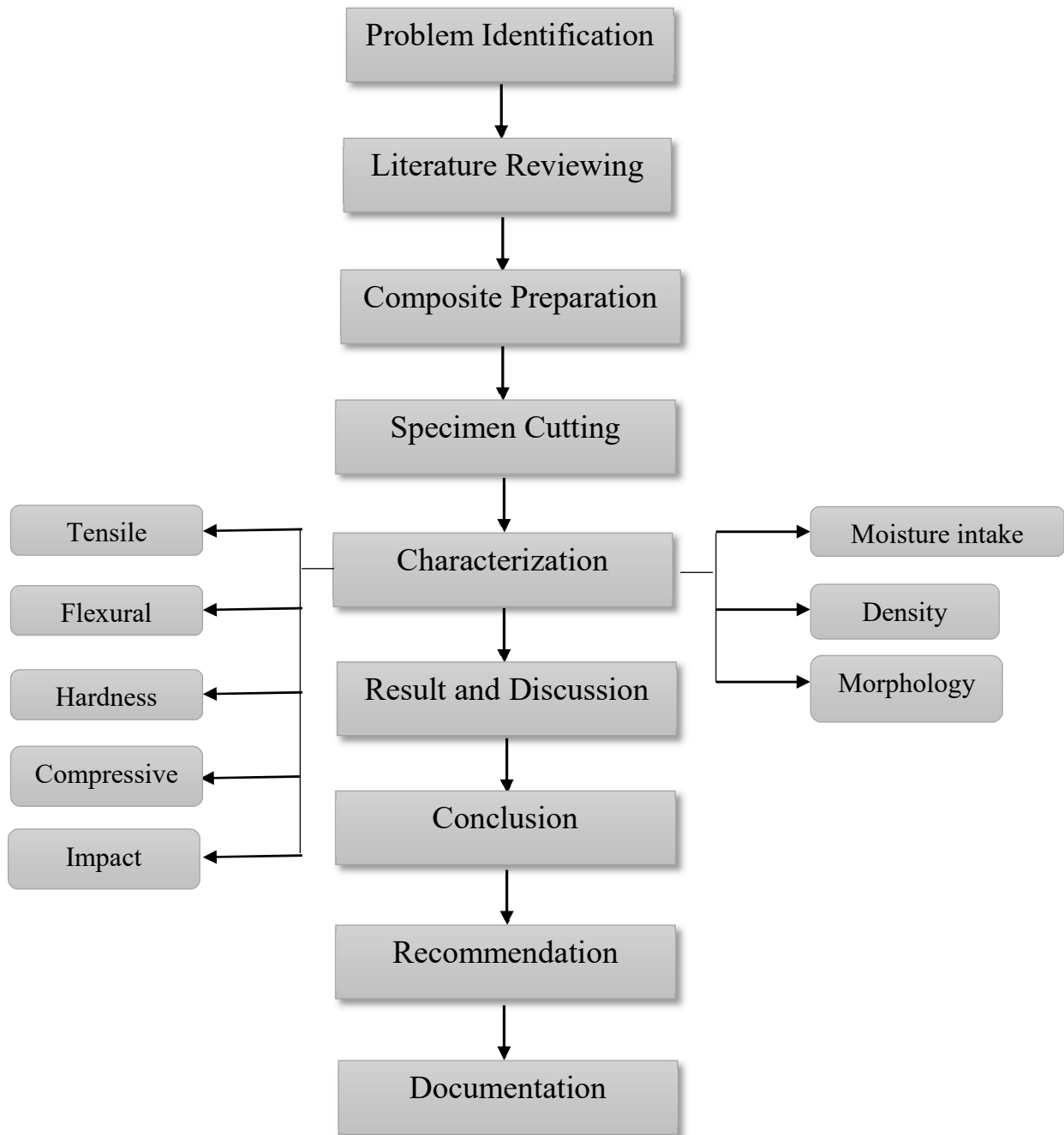


Figure 3.12: Research methodology

3.2.1 Density measurement

Every produced specimen is measured for density by the guidelines of the ASTM D792-13 standard. The average values of the densities of five specimens are provided. The substance's degree of homogeneity can be determined by that substance's density. In comparison to other reinforcement phases, natural polymer reinforcement has the advantage of having a lower

density and weight in composite materials. The samples were taken out of the mold once the solution had dried and specimens were made.

Calculating the theoretical and experimental densities of the specimen, then determining any distinction among the two, yields the sample's void content. The Archimedes principle is used to quantify experimental density, while the rule of mixtures is used to compute theoretical density.

$$\rho_C = \rho_M \times V_M + \rho_K \times V_K \quad \text{Equation 3.1}$$

ρ_C = Density of composite

ρ_M = Density of matrix

V_M = Volume of matrix

ρ_K = Density of kulkual fiber

V_K = Volume of kulkual fiber

The Archimedes principle uses weights in the air and submerged in liquid to measure a sample's density experimentally. Applying equation 3.2 below, the density of a sample can be computed.

$$\rho_C = \rho_{liq} \times \frac{m_{air}}{m_{air} - m_{water}} \quad \text{Equation 3.2}$$

ρ_{liq} = Density of water

m_{air} = Mass of sample in air

m_{water} = Mass of sample in water

The amount of void content in a sample is calculated as:

$$\text{Void content } (\phi_V) = \frac{(\text{Theoretical density} - \text{Experimental density})}{\text{Theoretical density}} \quad \text{Equation 3.3}$$

3.2.2 Tensile testing

Tensile testing is carried out according to the ASTM D638 standard using a computer-controlled universal test system (Z020 Zwick Roell, USA). Samples with measurements stated in figure

3.7 are taken into account. The gage length for each sample is 50 mm, and a 115 mm gap is retained between grippers.

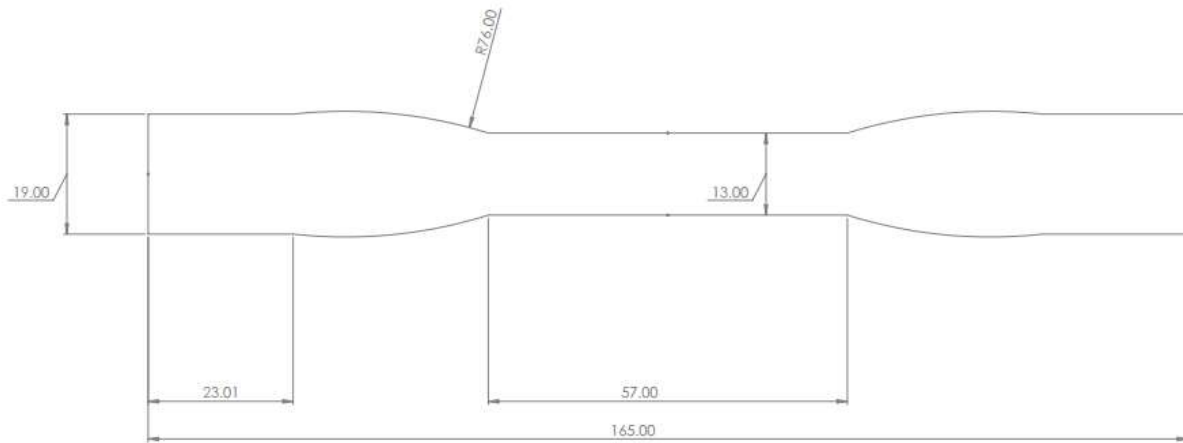


Figure 3.13: Dimension of specimen for tensile testing

Throughout the experiments, a steady cross-head speed of 5 mm/min is maintained using a 50 kN load cell. The test is carried out till the specimen breaks. The stress and strain are determined using the obtained load and displacement data, respectively. The tensile modulus is calculated by the 0.2% offset method on the stress-strain graph, and the tensile strength is calculated as the stress at maximum load. The strain measurement at breakage is being used to calculate the percentage elongation. The composite material's tensile strength was the highest tensile stress attained prior to actual failure. It was the highest tensile stress, which was the limit of the composite's ability to sustain a tensile load prior to failing.

For each sample, the average modulus and strength values of five specimens are presented.

$$\sigma_t = \frac{P}{A} \quad \text{Equation 3.7}$$

P = Load applied

A = the cross-section area

Tensile strength is calculated by dividing the maximum load sustained by the specimen in newtons by average initial specimen's cross-sectional area in m². The specimens were put in an upright position in the testing machine's grips then uniformly and properly tightened to avoid

slipping. The material elongates as the test begins; the resistance of the material increases and is recorded.



Figure 3.14: Test setup for tensile test

3.2.3 Flexural testing

The material's capacity to resist bending loads exerted perpendicular to a bar-shaped test piece is referred to as flexural strength. A flexural experiment is carried out using a Zwick (Z020, ZHU) machine with a capacity of 20 kN. Flexure tests are performed by the guidelines of the ASTM D790-17 standard. A cross-head speed of 1.4 mm/min is used with a 0.1 MPa pre-load. To preserve a 16:1 span length/thickness ratio, all specimens have span lengths of 52 mm. Each volume fraction combination is tested five times, with the average data being supplied for analysis. Up until the specimen fails and the stress-strain data is collected, tests are conducted.

The calculation of flexural modulus is as follows:

$$E_{fm} = \frac{L^3 m}{4bd^3} \quad \text{Equation 3.5}$$

L is the length of the support (mm)

b is the width of beam (mm)

d is the thickness of beam (mm)

m is gradient of the tangent line to the load-deflection curve's earliest straight-line segment.

The flexural stress is estimated by:

$$\sigma_{fs} = \frac{3PL}{2bd^2} \quad \text{Equation 3.6}$$

P denotes the force at a specific position on load-deflection curve. The reported flexural strength values are taken post fracture for comparison.



Figure 3.15: Test setup for flexural test

3.2.4 Compression testing

A UTM with a 50 kN load capacity is used to perform the compression tests. For each sample, the compressive strength and modulus are determined. The maximum stress at the tip of the elastic zone determines the compressive strength. Figure 3.- displays the compression testing setup. The load cell, which had been positioned on the top of the frame and also supported the top grip, can be utilized for tensile or compression testing. The UTM was linked to a display device, allowing us to record the test results (stress-strain curve).

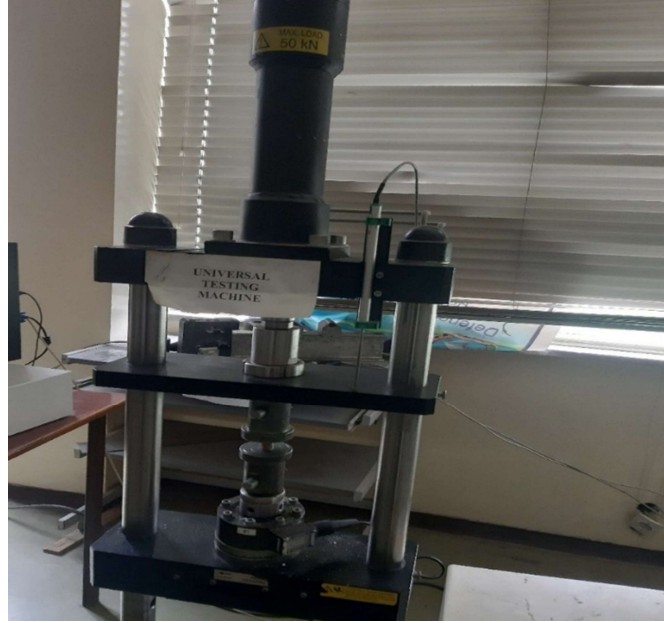


Figure 3.16: universal testing machine for Compression test setup

The test specimen used for the compression strength test is twice as long as it is wide or thick. The specimen sizes are made by the guidelines of ASTM D-695 and have dimensions of 12.7 x 12.7 x 25.4 mm. The testing standard speed is 1.2 mm/min. The compression strength is determined by simply dividing the highest compressive load generated by the specimen during the experiment by the specimen's initial minimum cross-sectional area.

$$\sigma_c = \frac{P}{A} \quad \text{Equation 0.1}$$

Where: σ_c = compressive strength

P = maximum compressive load carried by the specimen

A = specimen's original minimum cross-sectional area

The load held by the test sample at the yield point is divided by the specimen's initial minimum cross-sectional area to determine the compressive yield strength. By creating a tangent line to the load deformation curve's earliest stages linear portion, choosing points along this linear region, and dividing the stress value indicated by this point by the corresponding strain, one can determine the modulus of elasticity.

3.2.5 Impact Test

A composite material's impact strength indicates the energy it takes to shatter a sample of a particular size. The impact strength value represents the material's capacity to withstand a sudden impact. The experiment was carried out utilizing a dial display impact testing machine (Charpy/Izod Hensgrand) with an impact speed of 3.5 m/s. The ASTM D256 impact test is carried out using specimens sized 63.5 x 12.7 x 12.7 mm. The test specimens for the V-notch had notches that were 2 mm by 2 mm and were cut at a 45° angle. A notched specimen is held as a horizontal simple beam and is smashed by swinging hammers placed on the testing machines with the striking line in the middle. The charpy impact testing shows how much energy is required to shatter a certain type of specimen when certain circumstances are met. The energy used to start the specimen's fracture and the energy used to fly the fractured specimen's free end are combined to make up the energy dropped by the swinging during the specimen's breaking.



Figure 3.17: Impact testing machine

3.2.6 Hardness test

Hardness testing has been discovered to be quite helpful for studying composite development and research activities. A predetermined load is exerted directly by an indenter with a set geometry on the specimen's surface. After the force has been entirely released, the indentation is calculated in order to assess the sample's hardness.



Figure 3.18: Vicker Hardness Tester

The Vicker hardness testing machine HV50 was employed. It was used to measure the composite's surface hardness. A standard specimen made in accordance with ASTM-E384 is carefully laid on the vicker hardness tester's surface. Utilizing the Vicker Hardness Tester, a micro hardness testing tool, the specimen's hardness characteristics are examined. The test is carried out on a flat specimen with an appropriately prepared surface. Most flaws that can affect the indentation or the following measuring of the diagonals are absent from the face. The indentation periphery & edges are virtually always distinct in the microscope field of view during evaluation. Since etched surfaces might hide the edge of the indentation, making an accurate measurement of the depression's size difficult, the specimen surface is not etched prior to generating an indentation.

A square-based pyramid diamond indenter with an angle of 136° between the opposing sides at the vertex is used and is driven into the test specimen's surface with a specified force, F .

$$HV = \text{Constant} \times \frac{\text{Test force}}{\text{Surface area of the indentation}}$$

$$= 0.102 \times 2F \left(\frac{\sin\left(\frac{136^\circ}{2}\right)}{d^2} \right) \quad \text{Equation 3.8}$$

Where: HV = Vicker Hardness

F = Test force

d = Diagonal Length

The force of 10 kg.f is applied to the samples to generate a star-shaped Vickers impression. The diagonals are used to calculate the vicker hardness. The measurement is performed at a single point on one specimen and then again at another specimen of the same wt.%. For one composite's composition, at least 3 tests were conducted.

3.2.7 Water Absorption

The water absorption test is performed as per the ASTM D-5229 standard. According to the ASTM D-5229 guideline, the water uptake test's size was set at 20 x 20 x 6 mm. During ordinary ambient conditions, the standard water uptake test is performed using pure distilled water, salt-water, and underground tap water. Specimens from each weight fraction were immersed in distilled water, salt-water, and tap water containers in a controlled environment (50 percent humidity, 25 °C or room temperature).

Average moisture content, stated as a %, is the average quantity of water accumulated in a substance and is calculated as moisture's mass divided by mass of the oven-dry material as shown below:

$$M \% = \frac{W_i - W_o}{W_o} \times 100 \quad \text{Equation 3.8}$$

where: W_i = current specimen in grams (following contact with water)

W_o = oven-dry specimen in grams

M = Average moisture content

The specimens were brought out of the water, cleaned to exclude any extra water, and then measured. The desired accuracy of 0.1 mg is achieved by using a digital balance. The digital balance was used for measuring the mass of cactus fiber, TiB_2 , and epoxy resin used for sample preparation and also the test specimens. By changing the wt.% of the reinforcing materials in the epoxy matrix with TiB_2 filler particles and chemically treated cactus fiber reinforcement, the composite material yielded the findings that are presented below. To investigate the moisture

absorption ability of treated cactus fiber reinforced composite specimens, water uptake experiments were conducted.



Figure 3.19: Digital balance used to measure specimens for moisture absorption

CHAPTER FOUR

4. RESULT AND DISCUSSION

In this section, the results of all the experimental work are discussed in detail. The tests performed in this paper are flexural, tensile, hardness, impact, compression, water absorption, density, void content, and morphological analysis of the fabricated composite material, which is epoxy resin reinforced with cactus fiber and TiB_2 powder.

4.1 Density Test

The quality of production, the fiber and filler dispersion, and the amount of voids caused by air trapped all have an impact on the mechanical characteristics of fabricated samples. Hence, it is essential to determine these characteristics in order to effectively understand the link with the researched qualities. The ratio of reinforcing fiber to matrix determines density significantly. Composites become denser as their fiber content rises. As the amount of fiber in a composite material grows, so does the void content.

Table 4. 1: Density and Void content estimation of specimens

Composition	Theoretical Density (kg/m^3)	Experimental Density (kg/m^3)	Void Content (%)
E	1192.00	1192.00	0
EK-0	1292.90	1284.4	0.661
EK-2.5	1379.37	1365.7	1.001
EK-5	1400.35	1385.2	1.093
EK-7.5	1421.32	1406.5	1.054
EK-10	1442.3	1426.4	1.114

Theoretical densities are determined by employing Equation 3.1 and are estimated to be greater than experimental densities (Table 4.1). The difference is due to air entrapment in the polymer during ingredient mixing. Equation 4.3 is used to compute void content (%). The void content grows as the cactus fiber content increases. When compared to composites without

reinforcement, the findings demonstrate that the density and void content of composites increase as the fiber content increases. When the fiber content is low, the TiB_2 filler reduces the void content (fills the gap between matrix and fiber) of the composites by reducing the likelihood of air entrapment, which minimises the occurrence of pores and voids. When the fiber content increases to a higher percentage, the TiB_2 filler will not be able to fill all the gaps between matrix and fiber. So the likelihood of air entrapment will increase. Higher void contents are typically associated with greater vulnerability to moisture absorption.

4.2 Tensile Test

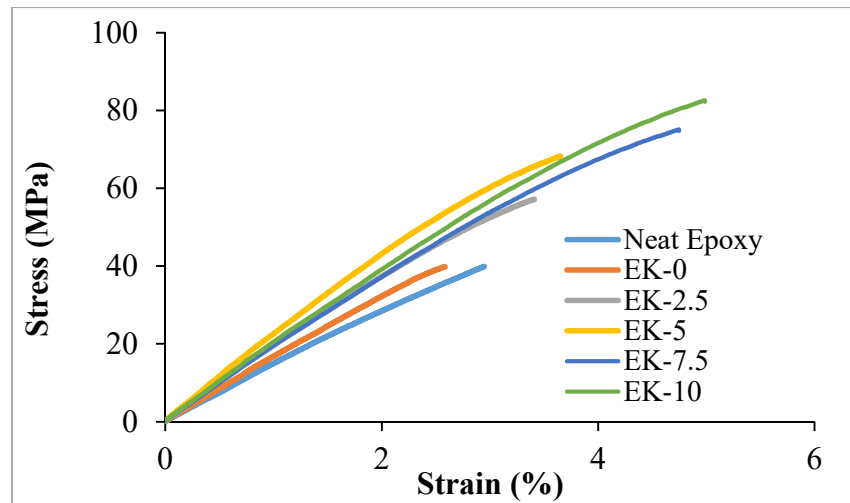


Figure 4.1: Representative Tensile stress-strain curves of composites.

Figure 4.1 represents the tensile stress-strain curves for neat epoxy and epoxy composites with cactus fiber and TiB_2 particle reinforcement. All of the specimens' stress-strain curves show a similar pattern till the elastic area's maximum stress, which is followed by a sudden brittle failure. Specimen E has a 2.56% strain at 39.92 MPa tensile stress. Specimen EK-0 has a 2.83% strain at a 39.86 MPa tensile stress. Specimen EK-2.5 has a 2.54% strain at 57.23 MPa tensile stress. Specimen EK-5 has a 3.50% strain at 68.31 MPa tensile stress and so on. The stress-strain curves show tensile stress has increased up to fiber content of 10 vol.%. From the specimens, E, EK-0, and EK-2.5 showed more brittle behavior, and EK-5, EK-7.5, and EK-10 specimens showed less brittle behavior.

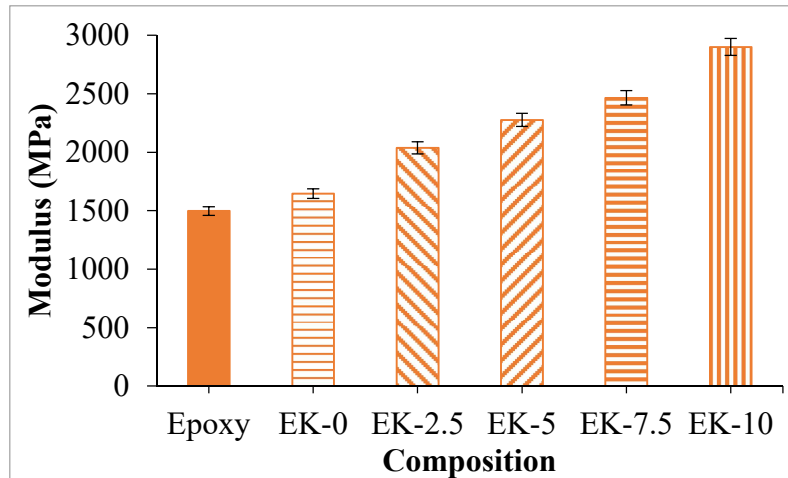


Figure 4.2: Young's modulus measured experimentally

Figure 4.2 shows the experimentally determined young's modulus. When compared to E (pure epoxy), the young's modulus of EK-0, EK-2.5, EK-5, EK-7.5 and EK-10 increased. The reinforcement of cactus fiber and TiB_2 particles into the matrix is mostly responsible for the rise in modulus. Specimen E had a tensile modulus of 1.496 GPa which was fabricated from 0 wt.% of TiB_2 and 0 wt.% of cactus fiber. Specimen EK-0 had a tensile modulus of 1.645 GPa which was fabricated from 5 vol.% of TiB_2 and 0 vol.% of cactus fiber. Specimen EK-2.5 had a tensile modulus of 2.037 GPa which was fabricated from 5 vol.% of TiB_2 and 2.5 vol.% of cactus fiber. Specimen EK-5 had a tensile modulus of 2.276 GPa which was fabricated from 5 vol.% of TiB_2 and 5 vol.% of cactus fiber and so on. All composites' tensile moduli rise with increasing fiber content and are between 9.92 and 93.78% higher than in E.

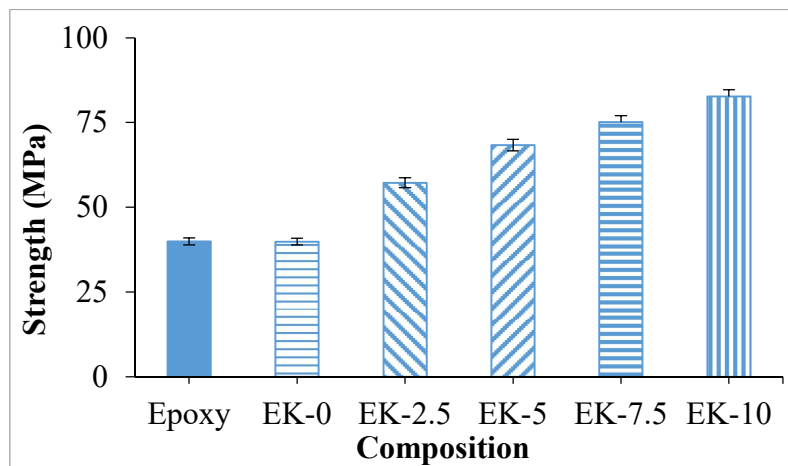


Figure 4.3: Tensile strength measured experimentally

Figure 4.3 shows the experimentally determined tensile strength of the specimens in response to fiber volume fraction.. Strength increases ranged from 0.15 to 107.32%, with EK-10 showing the greatest improvement. The combination of TiB₂ particles with cactus fiber in a load-carrying matrix increases the fiber-filler-matrix bonding and thus strength. The composite with 10 vol.% fiber had the highest values, approximately 82.64 MPa. Furthermore, the increase in stress transferred from matrix to fibers as a consequence of strong interfacial adhesion can justify this significant improvement in tensile strength (Alamri et al. 2012).

Several parameters, including distribution of reinforcement, porosity, interfacial bonding, and fiber wt.%, affect the tensile characteristics of cactus fiber-reinforced epoxy (Essabir et al. 2013). Rising the fiber vol.% to 10% with 5 vol.% TiB₂ filler results in a high interface region between the fibers and the matrix, and the fibers in the sample facilitate load transmission from the matrix to the fibers across the interface.

4.3 Flexural Test

Figure 4.4 shows the flexural stress-strain characteristics of the samples that were put through a flexural test. Every one of the specimens exhibits nearly linear stress-strain profiles up to failure, suggesting that both neat epoxy and cactus fiber reinforced epoxy composites fail brittlely.

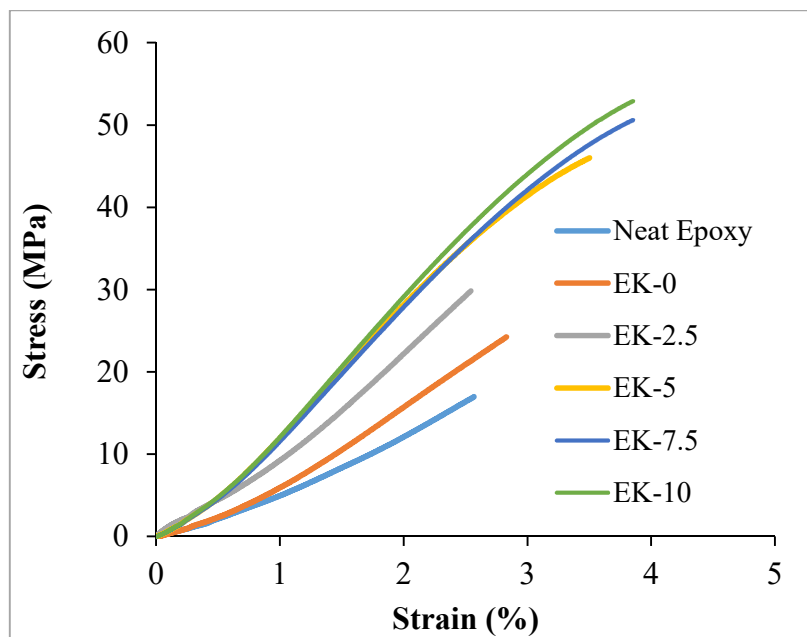


Figure 4.4 Representative Flexural stress-strain curve of the composite specimens.

Specimen E has 2.94% strain at 17 MPa flexural stresses. Specimen EK-0 has a 2.58% strain at 24.24 MPa flexural stresses. Specimen EK-2.5 has 3.41% strain at 29.85 MPa flexural stresses. Specimen EK-5 has a 3.65% strain at 46.03 MPa flexural stresses and so on. The flexural stress-strain curves show flexural stress has increased up to fiber content of 10 vol.%. From the specimens, E, EK-0 and EK-2.5 showed more brittle behavior, and EK-5, EK-7.5, and EK-10 specimens showed less brittle behavior.

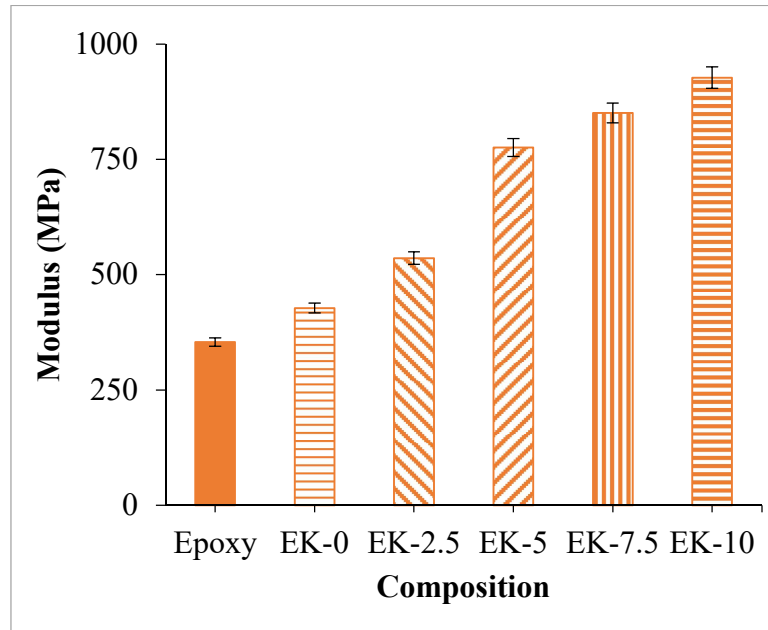


Figure 4.5: Flexural modulus measured experimentally

The flexural modulus of all composites shows an increasing trend as the cactus fiber volume fraction increases. Out of all the composite materials, EK-10 had the greatest modulus, showing a 161.96% improvement in modulus above neat epoxy. The robust interaction between the cactus fiber and the epoxy matrix is confirmed by the elevated flexural modulus.

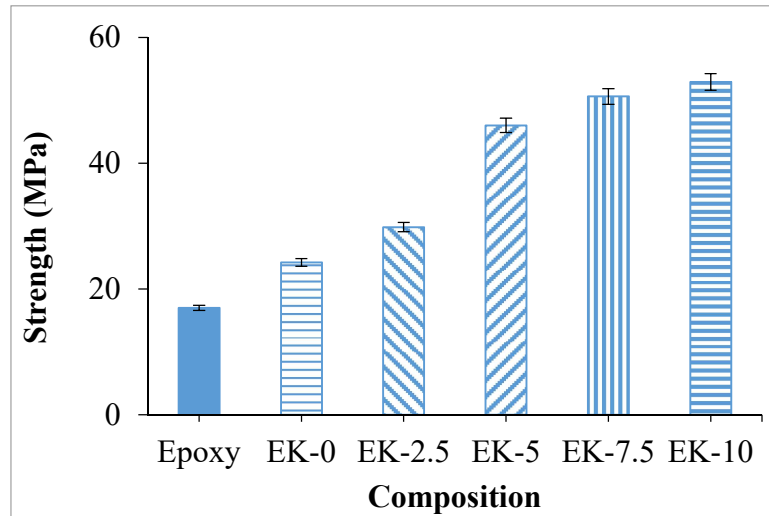


Figure 4.6: Flexural strength measured experimentally

The flexural strength of all composites shows an increasing trend as the cactus fiber volume fraction increases. The strength of EK-0, EK-2.5, EK-5, EK-7.5 and EK-10 rises almost uniformly and is determined to be greater than that of neat epoxy. Out of all the composite materials, EK-10 had the greatest flexural strength, showing a 211.29% improvement in strength over neat epoxy. As a result, flexural strength rises with increased cactus fiber reinforcement. Flexural strength improves as the fiber vol.% rises to 10 vol.%; this is due to the result of fiber concentration and TiB_2 filler providing the composite with increased resistance to crack starting and spreading. The reason for the improvement in flexural strength is the rise of fiber volume percentage up to 10%, which causes the bonding between the fibers to be stronger

4.3.1 Scanning Electron Microscopy (SEM)

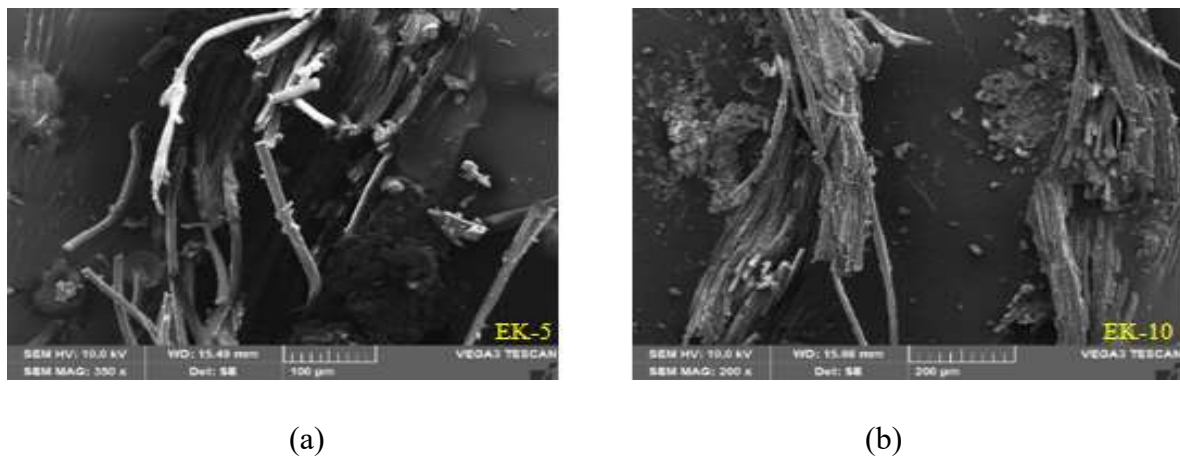


Figure 4.7: Flexural tested SEMs of (a). EK-5, (b). EK-10

Figure 4.7 shows SEM images of the surfaces for a better understanding of the composites' properties. In comparison to the EK-5, the composite EK-10 clearly demonstrates bundled fibers. In addition to TiB₂ particles, the behavior of clustered fibers contributed to the increase in flexural strength.

4.4 Compression Test

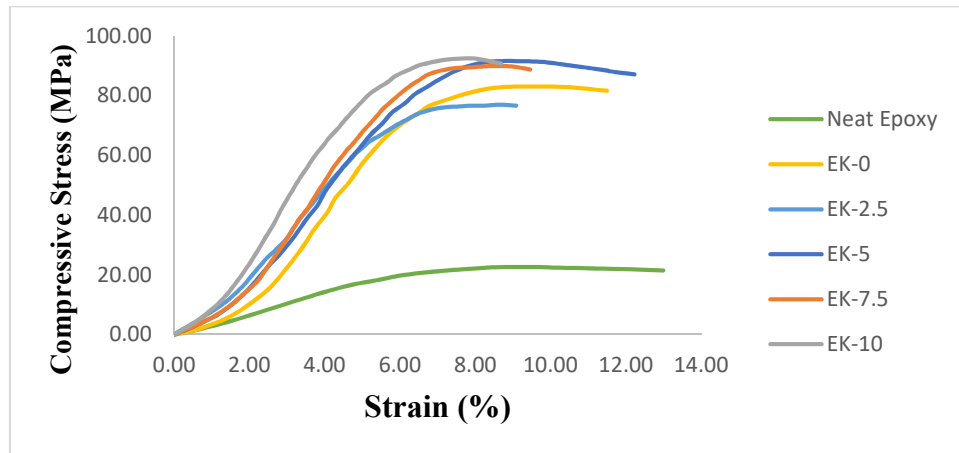


Figure 4.8: Representative compressive stress-strain curves of composite samples

The percentage volume of reinforcements has a major impact on the composite's failure mechanism. When compared to epoxy samples without fiber reinforcement, stress-strain curves for composites reinforced with cactus reveal similar patterns. Epoxy without fiber reinforcement has a linear elastic zone that is followed by a region that decreases strain, which is seen by a decline in stress values. The stress value rises as this epoxy sample is compressed farther, eventually causing rupture.

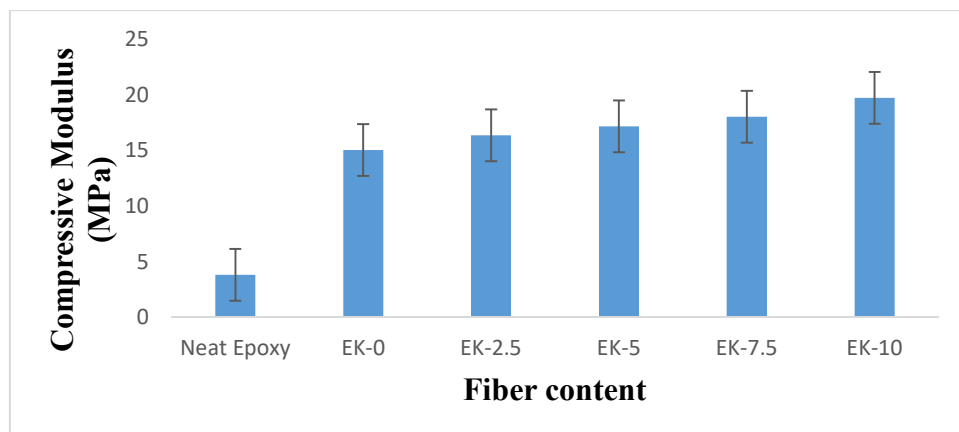


Figure 4.9: Experimentally measured compressive modulus

By measuring the gradient of the linear range on the compression stress-strain curve, the compressive modulus of the cactus fiber-reinforced epoxy composites was determined. The compressive modulus of TiB_2 particles and cactus fiber reinforced epoxy composites is improved in comparison to neat epoxy samples with 0% fiber content. When compared to neat epoxy samples (E), the improvement in compressive modulus is 292.65, 327, 347.63, 370, and 414.85% for EK-0, EK-2.5, EK-5, EK-7.5, and EK-10 respectively.

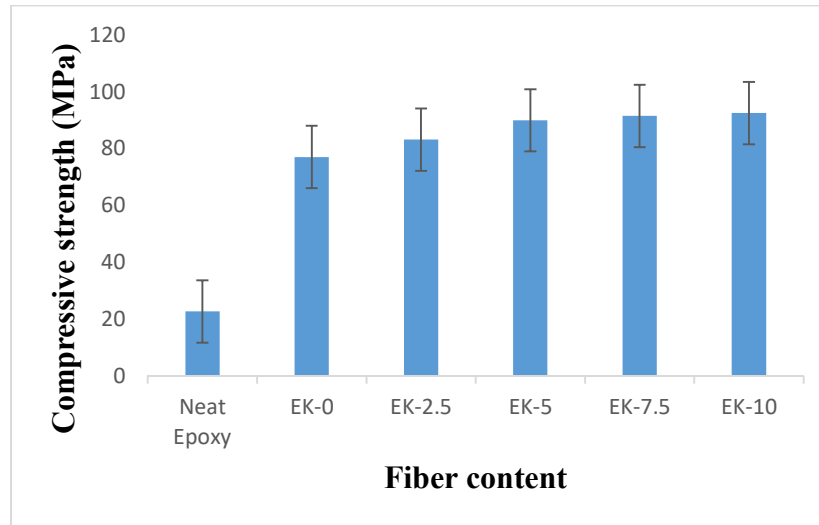


Figure 4.10: Experimentally measured compressive strength

When compared to no fiber-reinforced epoxy samples, all cactus fiber-epoxy composites have superior strength. With the increase of fiber percentage in the resin, the compressive strength of cactus fiber-epoxy composites for 0%, 2.5%, 5%, 7.5%, and 10% vol.% is 77.03, 83.14, 89.92, 91.45, and 92.47 MPa, respectively. When cactus fiber is added to an epoxy resin, it is observed that the mechanical properties improves due to adequate interfacial contacts between the matrix, fiber, and filler until a certain amount of reinforcement (i.e., EK-10). In the current study, it was discovered that adding 0, 2.5, 5, 7.5 and 10 vol.% of cactus fiber reinforcements enhanced the compressive strength of the composite material. The compressive strength of the EK-10 composite is found to be greater (92.47 MPa).

4.5 Impact Test

Three specimens from the epoxy composite EK-0 with 0 wt.% fiber reinforcement were studied, and those tests' average results were recorded. The rest of the epoxy compositions were

evaluated similarly. It has been found that till 10 vol.% fiber reinforcement, the impact strength was higher.

Table 4. 2: Results of impact test on various composite of varying composition.

Sample type	Trials	Energy absorption (J)	Average impact energy (J)	Angle (degrees)
Neat Epoxy	1	5.5	5.33	107
	2	5		105
	3	5.5		109
EK-0	1	6	6.17	129
	2	6.5		132
	3	6		126
EK-2.5	1	8	7.5	132
	2	7		129
	3	7.5		135
EK-5	1	7.5	7.67	138
	2	8		132
	3	7.5		139
EK-7.5	1	9	6.17 8 6.	135
	2	7		133
	3	8		126
EK-10	1	16	5.33 16.83	137
	2	17.5		135
	3	17		137

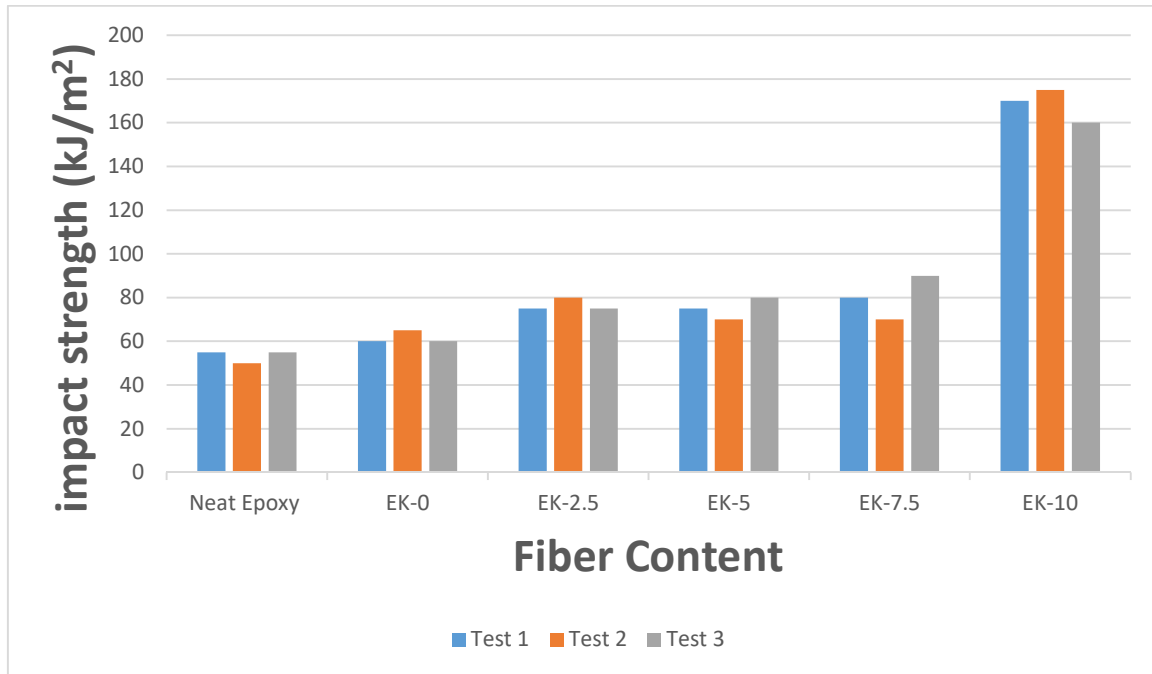


Figure 4.11: Impact strength of individual test specimen

From figure 4.11, it can be observed that the impact strength is high EK-10

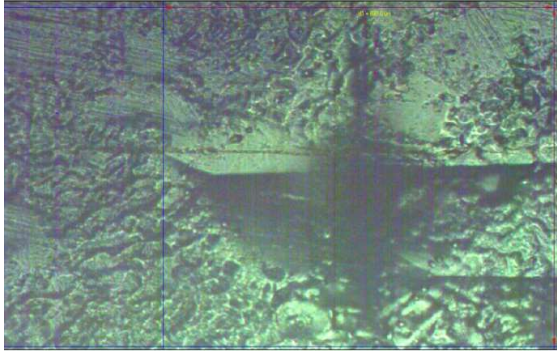
Table 4. 3: Average impact strength

Sample type	Average impact energy (J)	Average impact strength (kJ/m ²)
Neat Epoxy	5.33	53.3
EK-0	6.17	61.7
EK-2.5	7.5	75
EK-5	7.67	76.7
EK-7.5	8	80
EK-10	16.83	168.3

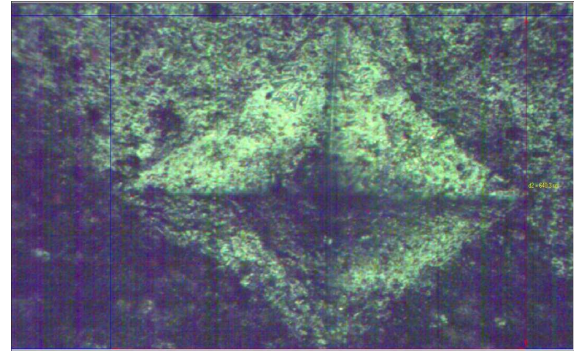
Table 4.3 displays the impact strength and energy of cactus-reinforced epoxy composites with the addition of TiB₂ particles. As the quantity of reinforcement increases, impact capabilities are observed to increase. The strongest samples, which were EK-10, have an impact strength of 168.3 kJ/m². Impact strength increases due to the composite material's increased capacity to withstand deformation before failure as a result of increased reinforcement % and also because the interface between the fiber & matrix is stronger

4.6 Hardness Test

The hardness experiment was conducted to investigate the hardness of the composites by calculating the degree of penetration of the indenter tip.



a)



b)



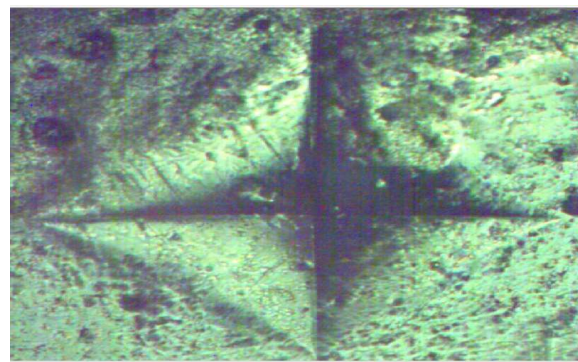
c)



d)



e)



f)

Figure 4.12: Optical microscopic view in Vicker hardness tester (a) Neat epoxy (b) EK-0 (c) EK-2.5 (d) EK-5 (e) EK-7.5 (f) EK-10

The minimum scale value the optical microscope measures is 0.5 micrometers with a magnification of 125X. Three different places on the surfaces of three specimens of the epoxy composite EK-0 with 0 wt.% fiber reinforcement were studied, and those tests' average results were recorded. The rest of the epoxy compositions were evaluated similarly.

The impact of fiber loading on the hardness of the composite materials is depicted in figure 4.13 below. The data shows that the composite's hardness improves when the volume percentage of fiber reinforcement increases from neat epoxy to 10 vol.% in the composite. The hardest composition, a 10 vol.% cactus fiber-epoxy composite, measured 45.9 HV. With the increase of fiber percentage in the resin, the hardness of cactus fiber-epoxy composites for neat epoxy, 0%, 2.5%, 5%, 7.5%, and 10% vol.% of fiber has an average hardness of 28.8, 36.2, 38.1, 40.1, 44.6, and 45.9 HV, respectively.

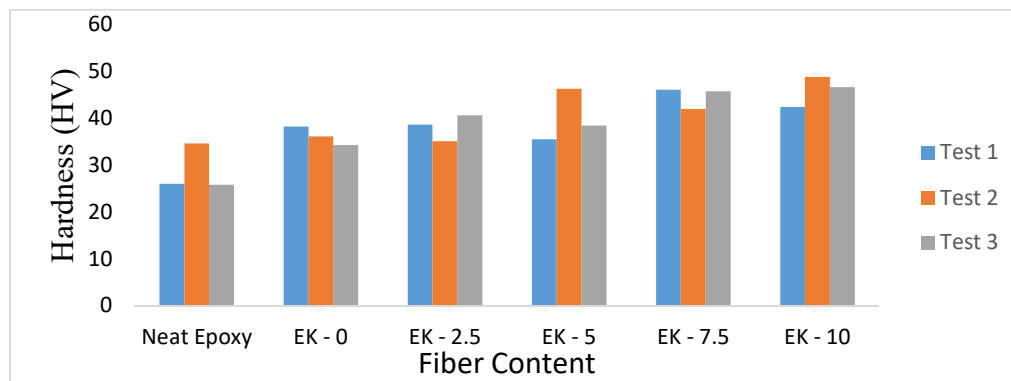


Figure 4.13: Relationship of fiber wt.% and hardness of the composites

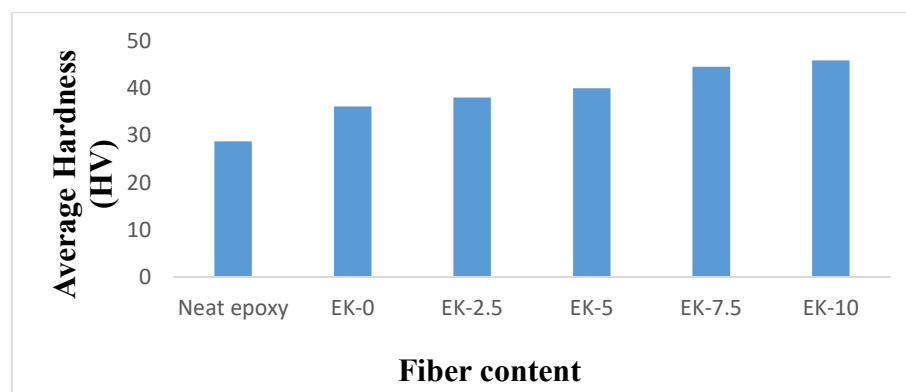


Figure 4.14: Average hardness of the composites

From figure 4.14, it can be observed that the addition of fiber from neat epoxy to 10 vol.% increased the Vicker's hardness by 59.47%. Surface characteristics are highly correlated to

hardness. Cactus fibers, which are understood to have different strengths than epoxy, and are located near the surface, significantly affected the hardness of the composite material.

The rise in hardness value of the samples when the fiber concentration rose from 0 to 10 vol.% could be because of TiB₂ particles scattered inside the composite being effective at preventing dislocation by indentation, hence improving the composite's distortion resistance. The hardness of a composite material increases because the interaction between the matrix, filler particles, and fiber phase increases as they are pushed together and contact one another more firmly.

4.7 Water absorption

The specimens were taken out of each water container, cleaned to eliminate any extra water on them, and then measured. The desired accuracy of 0.1 milligrams is achieved by using a digital balance. Water absorption experiments were carried out to investigate the water uptake characteristics of treated cactus fiber-reinforced composite specimens. The tables below show the percentage of water that the composite specimens absorbed after 24, 48, 72, 96, and 120 hours.

After 24 hrs. of immersion in the water containers, the cactus fiber reinforced composite samples showed the following water content: According to the experimental findings, after 24 hrs. of immersion in a tap water container, the water absorption % for each composite sample was 0.089, 0.37, 0.51, 0.99, and 1.00% for the EK-0, EK-2.5, EK-5, EK-7.5, and EK-10 samples, respectively.

Table 4. 4: Specimens' water absorption test results immersed in a salt-water

Sample type	Mass of specimen before (mg)	M % after 24hr (%)	M % after 48hr (%)	M % after 72hr (%)	M % after 96hr (%)	M % after 120hr (%)
EK-0	5.141	0.065	0.065	0.098	0.13	0.16
EK-2.5	5.743	0.40	0.49	0.59	0.69	0.78
EK-5	5.391	0.42	0.59	0.74	0.87	0.98
EK-7.5	5.977	0.50	0.68	1.08	1.26	1.45
EK-10	5.559	0.51	0.69	1.11	1.28	1.46

Table 4. 5: Specimens' water absorption test results immersed in a tap water

Sample type	Mass of specimen before (mg)	M % after 24hr (%)	M % after 48hr (%)	M % after 72hr (%)	M % after 96hr (%)	M % after 120hr (%)
EK-0	5.621	0.071	0.089	0.12	0.16	0.21
EK-2.5	5.364	0.32	0.37	0.47	0.55	0.63
EK-5	5.443	0.40	0.51	0.68	0.83	0.94
EK-7.5	5.946	0.75	0.99	1.31	1.50	1.66
EK-10	5.233	0.81	1.00	1.33	1.53	1.67

Table 4. 6: Specimens' water absorption test results immersed in a distilled water

Sample type	Mass of specimen before (mg)	M % after 24hr (%)	M % after 48hr (%)	M % after 72hr (%)	M % after 96hr (%)	M % after 120hr (%)
EK-0	5.508	0.072	0.16	0.19	0.23	0.25
EK-2.5	5.714	0.41	0.75	0.80	0.86	0.89
EK-5	5.733	0.46	0.78	0.85	0.96	1.01
EK-7.5	5.047	0.76	1.28	1.41	1.53	1.59
EK-10	5.470	0.85	1.39	1.54	1.67	1.78

The experimental findings indicated that the water intake percentages for all composite specimens were 0.065, 0.40, 0.42, 0.50, and 0.51% when submerged in salt water; 0.071, 0.32, 0.40, 0.75, and 0.81% when submerged in tap water; and 0.072, 0.41, 0.46, 0.76, and 0.85% in distilled water for EK-0, EK-2.5, EK-5, EK-7.5, and EK-10 specimens, respectively, after 24 hours.

Following a 48-hour period, the water absorption percentage of all composite specimens was found to be 0.065, 0.49, 0.59, 0.68, and 0.69% when submerged in salt water; 0.089, 0.37, 0.51, 0.99, and 1.00% when submerged in tap water; and 0.16, 0.75, 0.78, 1.28, and 1.39% in distillate water for EK-0, EK-2.5, EK-5, EK-7.5, and EK-10 specimens, respectively.

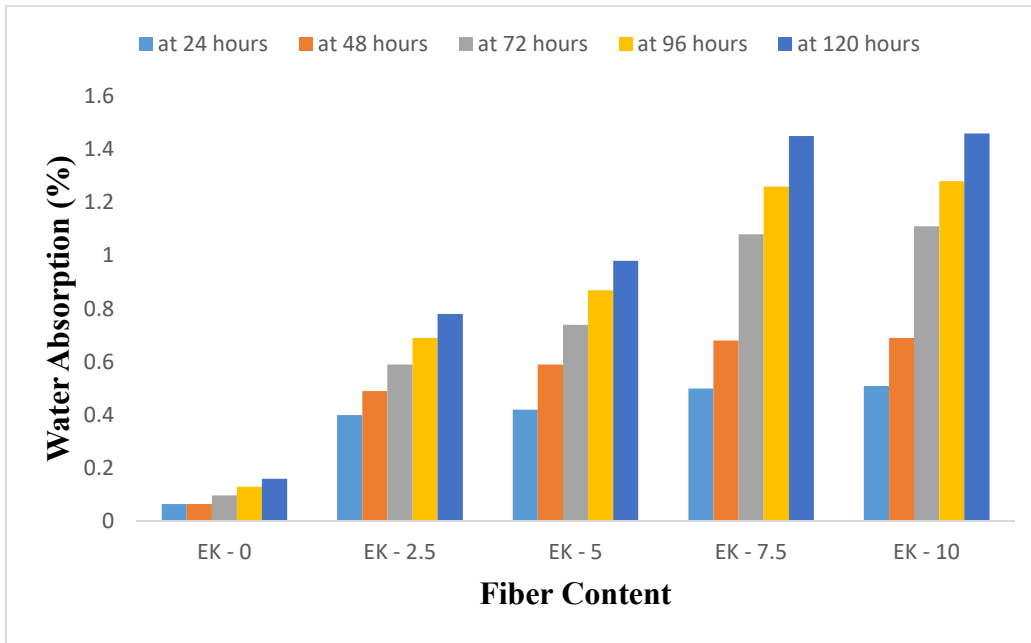


Figure 4.15: Water absorption percentage of epoxy composites with cactus fiber and TiB_2 particles reinforced specimens after immersed in salt-water

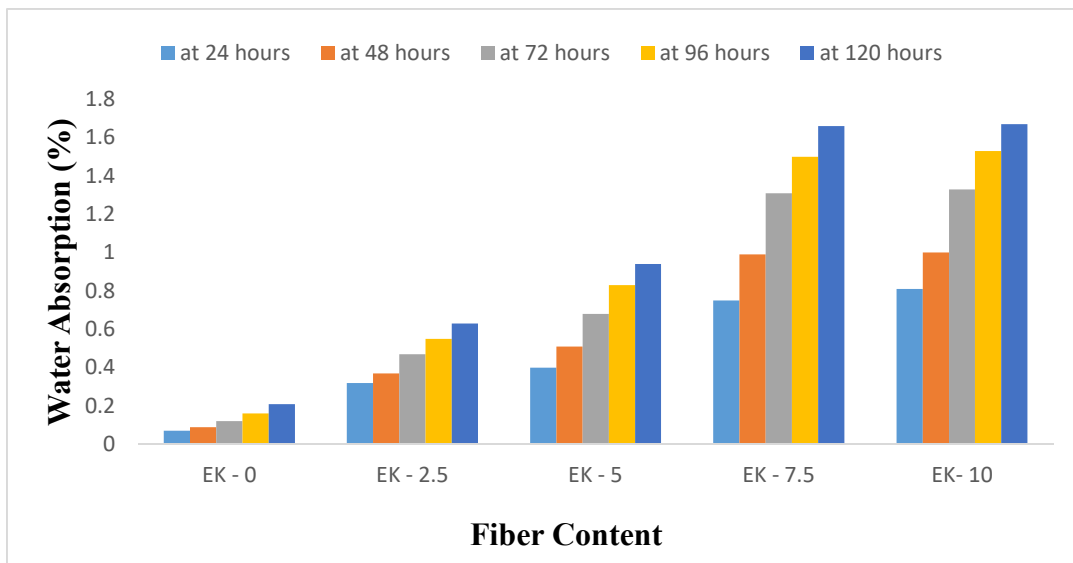


Figure 4.16: Water absorption percentage of epoxy composites with cactus fiber and TiB_2 particles reinforced specimens after immersed in tap water

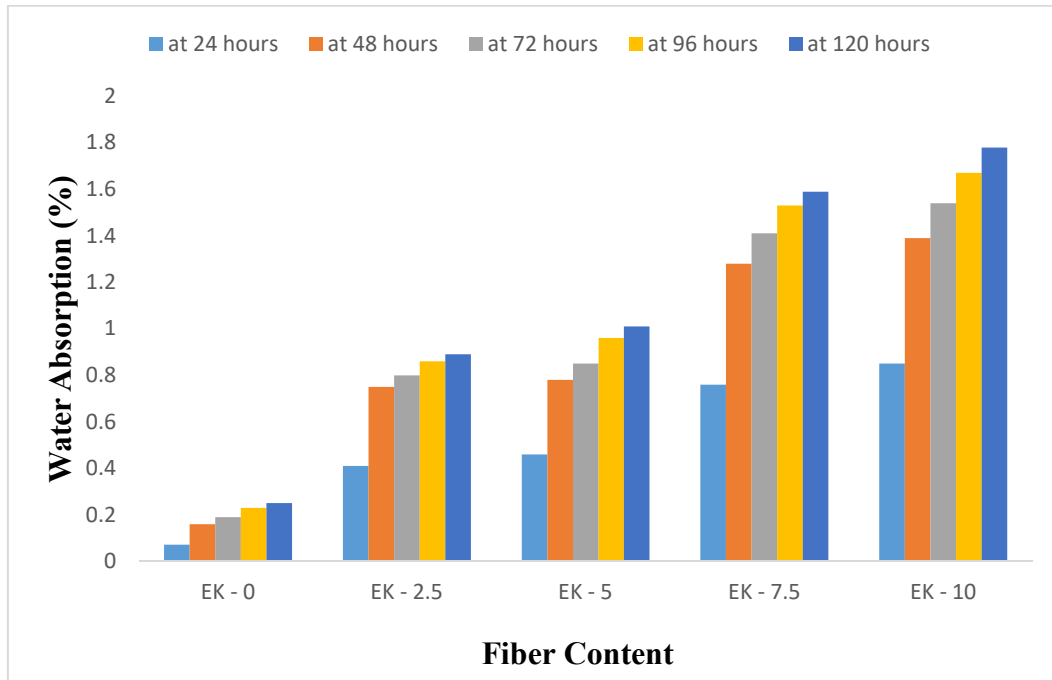


Figure 4.17: Water absorption percentage of epoxy composites with cactus fiber and TiB_2 particles reinforced specimens after immersed in distilled water

It was found that the composite specimen (EK-0), which contains 0 vol.% cactus fiber and 5 vol.% TiB_2 particles, takes far less water than other composite specimens over the course of the water absorption test. The spaces in the cactus fiber were filled with the addition of 5% of the filler powder, which is TiB_2 particles, enhancing the quantity of interfacial interaction between the fiber and the epoxy matrix. The process of absorbing water is found to begin fast before leveling out as it approaches equilibrium. The composites' densities & the empty spaces have a big influence on how quickly water is absorbed (Das et al., 2.16). Furthermore, it is evident that the amount at which water is absorbed increases with fiber content. Composites containing 10 vol.% cactus fiber show a greater amount of water absorption than those that contain 0 vol.% fiber.

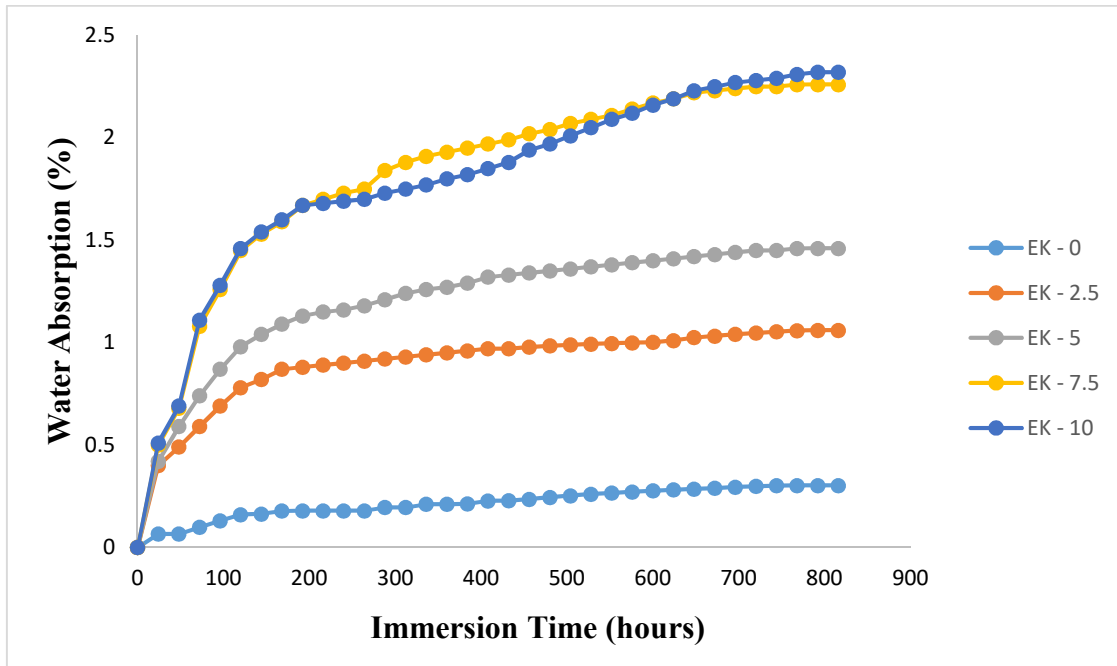


Figure 4.18: The influence of fiber percentage on cactus fiber-epoxy composite water absorption properties immersed in salt-water

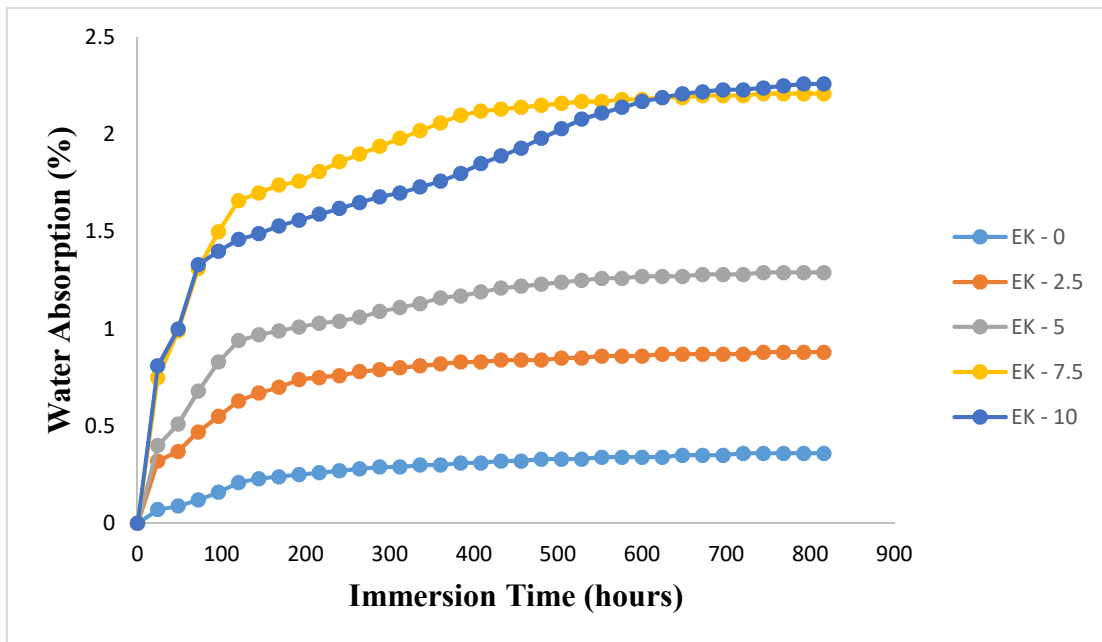


Figure 4.19: The influence of fiber percentage on cactus fiber-epoxy composite water absorption properties immersed in tap water

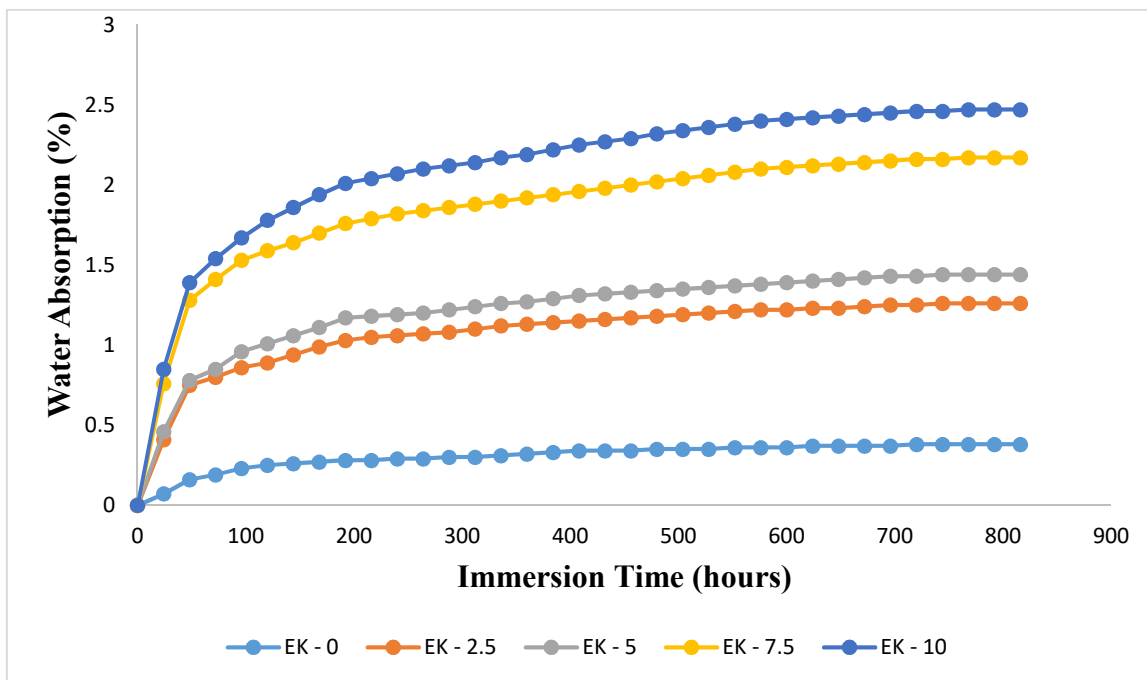


Figure 4.20: The influence of fiber percentage on cactus fiber-epoxy composite water absorption properties immersed in distilled water

Figures 4.18, 4.19, and 4.20 demonstrate that the water absorption process begins sharply and settles off for a while as it nears equilibrium. It is also obvious from the graphic that the water absorption rate rises with the amount of fiber. In comparison to composites with 2.5 vol.% fiber, 10 vol.% cactus fiber shows a higher rate of moisture uptake. The cause may be because cactus fibers possess more polar hydroxide groups, which cause fiber-based epoxy composites to absorb moisture at a rapid rate.

Testing on EK-0 composite samples revealed a very small ($<0.40\%$) increase in water content during the test period. As a fact, the cactus fibers themselves were responsible for all the water uptake in the composite samples. Generally speaking, larger fiber percentages led to greater water uptake. The likelihood of fiber-to-fiber interaction increases when the fiber percentage is raised (Das et al., 2.16). This increased fiber contact and interconnection, which results in more transport of moisture content, is most likely the cause of the spike in water uptake seen between 5, 7.5, and 10 vol.% fiber fractions.

After 32 to 34 days of immersion in water, equilibrium was reached. The results for the three types of water are plotted as a result of opuntia fiber content on water absorption in Figures 4.18, 4.19, and 4.20. While the specimens attained equilibrium level after 34 days immersed in

water, the maximum water absorption measured was 0.30, 1.06, 1.46, 2.26, and 2.32% when submerged in salt water; 0.36, 0.88, 1.29, 2.21, and 2.26% when submerged in tap water; and 0.38, 1.26, 1.44, 2.17, and 2.47 in distillate water for EK-0, EK-2.5, EK-5, EK-7.5, and EK-10 specimens, respectively.

4.8 Comparison with the Previous Works

No.	Authors	Matrix	Fiber type and filler	Results
1	Bouakba et al., (2013)	Polyester	Cactus fiber	Tensile strength=15.8 MPa Flexural strength: 66.5 MPa
2	Malainine et al., (2004)	Poly(propylene)	Opuntia powder	Tensile strength=10 MPa Max Water Uptake=26.27%
3	Greco et al., (2013)	Poly-lactic acid & wood flour	Opuntia fibers	Flexural strength=70.3 MPa
4	Scaffaro et al., (2019)	Poly-lactic acid	Opuntia powder	Tensile strength=55 MPa
5	Scognamiglio et al., (2019)	Thermoplastic starch	Opuntia fiber	Tensile strength=3.75 MPa % elongation=23.4
6	Arevalo et al., (2010)	High Density Polyethylene	Cactus powder	Tensile strength=20 MPa
7	Current work	Epoxy	Cactus fiber and TiB ₂ filler	Tensile strength=82.64 MPa Flexural strength=53 MPa Compressive strength= 92.47 MPa Impact strength= 168.3 kJ/m ² Hardness= 45.9 HV Water absorption= 2.47%

CHAPTER FIVE

5. CONCLUSION AND RECOMMENDATIONS

5.1 Conclusion

Recently, many researchers are beginning their study to replace metallic products of with composite materials in order to reduce weight. For reasons of reduced price and eco-friendliness with cleaner production, NFRPCs are preferred for such kinds of applications. In this research, cactus fibers placed in random orientations and TiB_2 particles are utilized to produce composite materials using an open molding process, and their mechanical behaviors like tensile, compression, flexural, and impact properties are examined. Various mechanical tests were done in order to assess the mechanical qualities of an epoxy composite reinforced with opuntia fiber and TiB_2 filler. The fibers were placed in the direction of random orientation.

The flexural and tensile properties of epoxy composites with cactus fiber and TiB_2 particle reinforcement are highly impacted by the fiber vol.% of fiber in the epoxy matrix. As the fiber content increases to a 10% fiber volume fraction, these strength properties also increase. With a rise in fiber percentage to the resin, the cactus fiber-epoxy composites of neat epoxy, 0, 2.5, 5, 7.5, and 10 vol.% fiber have an average hardness of 28.8, 36.2, 38.1, 40.1, 44.6, and 45.9 HV, respectively. Composites' hardness is greatly influenced by fiber. The addition of fiber from neat epoxy to 5 vol.% increased the Vicker's hardness by 59.47%. The composites' impact strength decreases from neat epoxy, 0, 2.5, 5, 7.5, and 10 vol.% with an average impact strength of 168.3, 80, 76.7, 75, 61.7, and 53.3 kJ/m², respectively, as fiber percentage to resin increases.

Using the experimental findings, the following conclusions were drawn from water absorption tests on different weight percentages of cactus fiber reinforced and 5 vol.% TiB_2 filled epoxy composites: When specimens that had an equal amount of fiber content were immersed in distilled water rather than salt water or tap water, the percentage of water absorption increased. This means that the percentage of water absorbed in distilled water is greater than in tap water, and the percentage of water absorbed in tap water is greater than in salt water. It is also discovered that the proportion of water absorption increases as the weight percentage of fiber reinforcement in the composite increases. The majority of composites need five weeks to

achieve equilibrium moisture content (saturation). Once the specimens achieved equilibrium after 34 days in the water, the maximal water absorption was 0.30, 1.06, 1.46, 2.26, and 2.32% when submerged in salt water; 0.36, 0.88, 1.29, 2.21, and 2.26% when submerged in tap water; and 0.38, 1.26, 1.44, 2.17, and 2.47 in distillate water for EK-0, EK-2.5, EK-5, EK-7.5, and EK-10 specimens, respectively.

The compressive modulus of TiB₂ particles and cactus fiber reinforced epoxy composites is improved in comparison to neat epoxy samples with 0% fiber content. When compared to neat epoxy samples (E), the improvement in compressive modulus is 292.65, 327, 347.63, 370, and 414.85% for EK-0, EK-2.5, EK-5, EK-7.5, and EK-10 respectively. When compared to no fiber-reinforced epoxy samples, all cactus fiber-epoxy composites have superior strength. With the increase of fiber percentage in the resin, the compressive strength of cactus fiber-epoxy composites for 0, 2.5, 5, 7.5, and 10 vol.% is 77.03, 83.14, 89.92, 91.45, and 92.47 MPa, respectively.

In this research work it is achieved that the fabricated composite is suitable for some industrial applications. Based on the studies, no extensive studies on cactus fiber exist in Ethiopia. This work demonstrates that hand lay-up can successfully fabricate composites containing cactus fiber with random orientation. To improve bonding capabilities with a matrix, the fibers were treated with NaOH to enhance surface roughness. The mechanical properties of the composite were influenced by the wt% fiber content. It was also established that combining natural fiber with TiB₂ filler improved the mechanical characteristics of the composites, yielding results that were greater than those achieved for neat epoxy composites.

5.2 Recommendations

The current work illustrates the feasibility and manufacturing of epoxy-based composite materials reinforced with cactus fiber and TiB₂ particles. These materials are environmentally safe, lighter, and have reasonably good characteristics. Furthermore, the use of opuntia ficus-indica fibers minimizes waste and environmental problems. The recommendations below were made after considering the fabrication and characterization of epoxy composites with cactus fiber and TiB₂ particle reinforcement.

- ❖ This composite material is recommended for automotive, manufacturing, and construction sectors to use this abundantly available opuntia fiber resource.

- ❖ Any agricultural industry seeking to replace heavy metals with lightweight, recyclable, sustainable, ecologically safe, and green composites should give attention to such kinds of materials.
- ❖ They can be used to make automobile parts such as car body parts because they enhance strength and durability while also minimizing the total machine weight. This results in low fuel consumption and longer machine life.

Since the composites are lighter in weight and have high strength, they are highly recommended for automobile body parts production. Their primary advantages is decrease car weight and fuel consumption, increase impact strength and renewable usability.

5.3. Future scope of the research

Cactus fiber in the range of 0 to 10 vol.% was investigated in this research. This can be expanded to include a greater cactus fiber volume fraction to investigate the influence of weight percentage on the physical and mechanical properties of the composite. It is also possible to explore the mechanical and physical characteristics of opuntia fiber in combination with other kinds of natural fibers. The open molding method can also be changed to other precise manufacturing techniques and use the powder filler materials at a nanoparticle size. One of the future plans is to use more effective treatment chemicals other than NaOH to treat opuntia fiber. Across the country, opuntia fibers of varying ages, weather, and soil conditions should be employed by doing more research into the material's physical, mechanical, morphological, thermal, and chemical properties. A comparison between alkaline-treated opuntia fiber reinforcement and untreated ones can also be carried out. This research can also be expanded by including the wear characteristics and machinability of the developed material.

Published conference paper at Bule Hora University:

M. Yasin, K. Selman, Gossaye. K, P.J Ramulu, M. Gelaw, K. Shahapurkar. (2021). Comprehensive Review on the flexural behavior of polymer matrix composites reinforced with waste materials.

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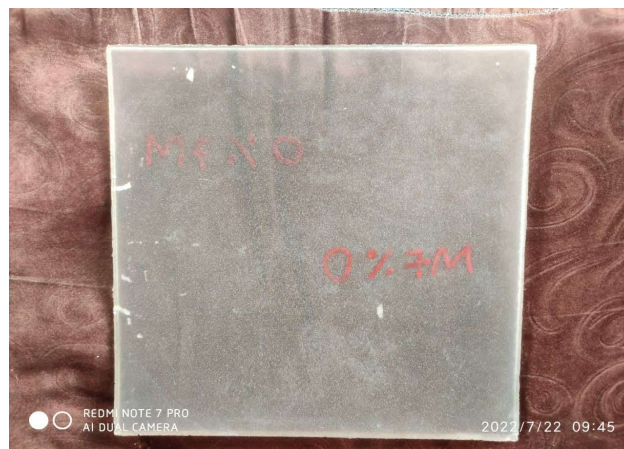
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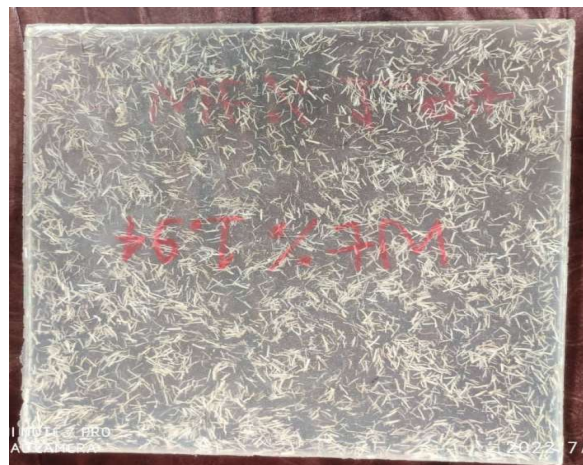
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APPENDIX

Appendix A: epoxy with 5 vol.% filler TiB_2 and 0 wt% cactus fiber



Appendix B: 2.16 and 1.94 wt.% cactus fiber reinforced epoxy composite



Appendix C: 4.26 and 3.92 wt.% cactus fiber reinforced epoxy composite



Appendix D: 6.29wt.% (7.5vol%) cactus fiber reinforced epoxy composite



Appendix E: 6.29wt.% (7.5vol%) cactus fiber reinforced epoxy composite

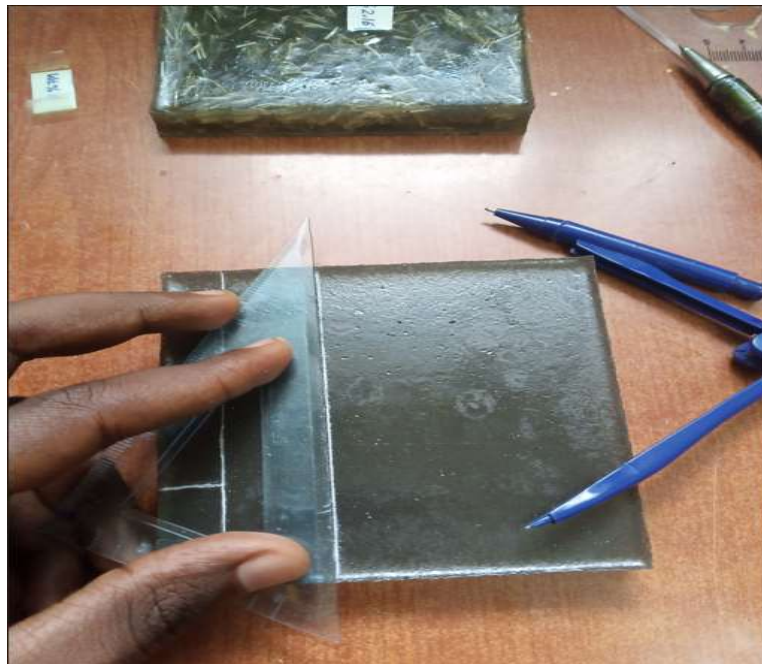


Appendix F: 4.26wt.% (5vol%) cactus fiber reinforced epoxy composite

This sample was made with 4.26 wt.% cactus fiber and 5 vol.% filler TiB_2 which were sieved with coarse sieve size. The size of the filler were the key factors for the high sedimentation and the formation of flakes and cracks.



Appendix G: Specimen cutting







Appendix H: Compression testing setup



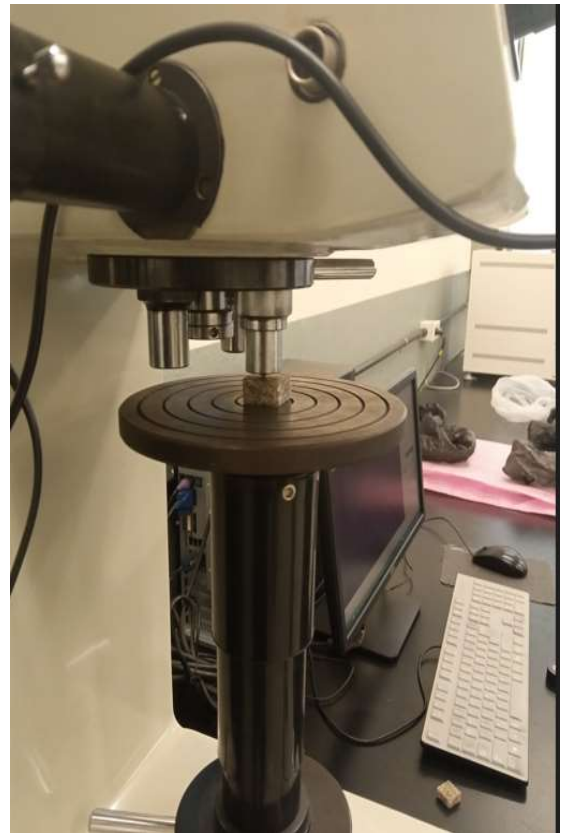
Appendix I: Charpy impact test setup



Appendix J: Specimens during Water Absorption Test



Appendix K: Hardness test setup



Appendix L: Tensile test setup



Appendix M: Flexural test setup



Appendix N: Specimens after compression test



Appendix O: Specimens after Impact test

