

Experimental Investigation on the Mechanical and Tribological Properties of Caesalpinia Decapitala Shell Epoxy Composites



Hailemariam Biratu Gutema

A Thesis Submitted to the Department of Mechanical Engineering

School of Mechanical Chemical and Materials Engineering

Presented in Partial Fulfillment of the Requirement for the Degree of

Masters of sciences in Manufacturing Engineering

Office of Graduate Studies

Adama Science and Technology University

November, 2022

Adama, Ethiopia

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DECLARATION

I hereby declare that this Master Thesis “**Experimental Investigation on the Mechanical and Tribological Properties of Caesalpinia Decapitala Shell Epoxy Composites**” 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 were used for this thesis have been duly acknowledged through citation.

Hailemariam Biratu Gutema

Name of student

Signature

Date

RECOMMENDATION

We, the advisors of this thesis, hereby certify that we have read the revised version of the thesis entitled “**Experimental Investigation on the Mechanical and Tribological Properties of Caesalpinia Decapitala Shell Epoxy Composites**” prepared under our guidance by Hailemariam Biratu Gutema submitted in partial fulfillment of the requirements for the degree of Masters of Science in Manufacturing Engineering. Therefore, we recommend the submission of a revised version of the thesis to the department following the applicable procedures.

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Date

APPROVAL SHEET

We, the advisor(s) of this thesis entitled “**Experimental Investigation on the Mechanical and Tribological Properties of Caesalpinia Decapitala Shell Epoxy Composites**” and developed by Hailemariam Biratu Gutema, hereby certify that the recommendation and suggestions made by the board of examiners are appropriately incorporated into the final version of the thesis.

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We, the undersigned, members of the Board of Examiners of the thesis by Hailemariam Biratu Gutema have read and evaluated the thesis “**Experimental Investigation on the Mechanical and Tribological Properties of Caesalpinia Decapitala Shell Epoxy Composites**” and examined the candidate during the 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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ACKNOWLEDGEMENT

In the beginning, I want to declare all the credit to my unforgettable helper Almighty GOD for giving me the strength and ability to complete this research.

I would like to thank my main advisor, Dr. Kiran Shahapurkar who has given his best in making me understand how this research has to be done and has guided me throughout the research. In addition, I would like to express my sincere gratitude to my co-advisor Mr. Mengistu Gelaw for his invaluable guidance, continuous encouragement, and constant support in making this research possible. I am always impressed with their effort in putting up with my attitude and still treating me well as his student after giving him such a difficult time. My sincere thanks go to all staff of the Mechanical Chemical and Material Science School who helped me in many ways whenever I needed them. Thanks for always putting up your best effort in helping me to finish this research.

The best thanks go to my family and friends. I am very thankful to have them as a family because they never gave up on me and constantly encourage me morally and financially which are things that I needed the most to complete this research.

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LIST OF ACRONYMS AND ABBREVIATIONS

CMC	Ceramic Matrix Composites
FRC	Fiber Reinforced Composite
GRA	Grey Relational Analysis
GRC	Grey Relational Coefficient
GRG	Grey Relational Grade
ECD	Epoxy Caesalpinia Decapetala
CD	Caesalpinia Decapetala
PE	Polyester
MMs	Metal Matrix Composites
OA	Orthogonal Array
PMCs	Polymer Matrix Composites
PRP	Particle Reinforced Composites
S/N	Signal to noise ratio
COF	Coefficient of Friction
SEM	Scanning Electron Microscope
UTM	Universal Testing Machine
VHN	Vickers Hardness number
ANN	Artificial Neural Network
ANOVA	Analysis of Variance

NOMENCLATURES

μ	Coefficient of Friction
w_s	Specific wear rate ($\text{mm}^3/\text{N}\cdot\text{km}$)
w_r	Wear rate (mm^3/km)
W_v	Volumetric wear rate (m^3/s)
F_n	Normal force (N)
V_s	Sliding velocity (m/s)
Δw	Weight loss (g)
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)
$\mathcal{P}_c$	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

In the present study a comprehensive analysis of the mechanical properties and tribological behavior of an epoxy-based composite reinforced with Caesalpinia Decapetala (CD) Shell were investigated. Caesalpinia Decapetala is available in abundance all over the world, especially in Ethiopia, particularly in East and West Oromia near West Harar. The seeds from Caesalpinia plant are extracted, and made it in powder form by using a coffee gridding machine and sieving it to 100-105 μ m. Four types of composites were prepared by varying the Caesalpinia Decapetala shell content by 0, 10, 20 and 30 vol.%. Open mold casting technique with the assistance of magnetic stirrer is used to fabricate the samples. Further, composites were mixed for 10, 30, and 60 minutes by using a magnetic stirrer for each percentage composition of the composite sample and trimmed according to ASTM standards. Physical characterization, including density and void content, was, performed. Additionally, mechanical behavior, including compression, tensile, impact, and hardness tests, are, carried out according to ASTM standards. Furthermore, dry sliding wear tests were performed by varying the applied load (30, 40 and 50 N), sliding velocity (5 m/s) and sliding distance (3, 4 and 5 km). The mechanical test results show that a 30 percent Caesalpinia Decapitala composition mixed for 60 minutes with a magnetic stirrer has a high Vickers hardness of 150.8 HN, a compression strength of 64 MPa, the tensile strength is 62MPa at 20% of ECD composition and, a high-energy absorption of 7.5J during impact testing at 30% of ECD composite. In addition, the water absorption capability of the produced composite material was examined, and, the results show that when compared to all compositions for 24,48,72,96,120 164, 188, 212, and 236 hours, less water is absorbed in 10%. Experiments on dry sliding wear tests reveal minimum wear rate and coefficient of friction for composites with 30 vol.% of Caesalpinia Decapetala depicting an increase in wear resistance as compared to other compositions. Statistical tools such as Taguchi Grey Relation Analysis and Artificial Neural Network were utilized to validate the wear results. Finally, SEM and EDS are used to characterize the morphological and physiochemical structure of the synthesized composite.

Keywords: *Caesalpinia Decapetala Shell; Epoxy Matrix; Mechanical test; Dry Sliding Wear; Polymer Matrix Composite.*

CHAPTER ONE

INTRODUCTION

1.1 Background of the Study

Due to the rapid development of advanced manufacturing industries, materials with good qualities in terms of strength, stiffness, density, lower cost, and better sustainability are in high demand. Composite materials are one of the materials that can provide these properties. This material can be described as a substance that combines two or more different materials with different properties on a macro scale to create a new material with qualities that are completely different from the individual constituents. Due to these properties, polymer matrix composites reinforced with natural and synthetic fiber are highly recognized in a variety of fields, inclusive of maritime, aviation, and automotive (Pattanaik et al., 2016).

In composite material, the primary face of the composite is a matrix that acts as a binder and holds the fibers and fillers within the desired position, thus transmitting the external load to reinforcement. Additionally, the matrixes are less hard and more ductile by its property. The secondary face of composite materials is reinforcement. These reinforcements are either natural or manmade, and they are used to enhance the strength of the composite. Natural fibers include jute, bamboo, sisal, grass, sugar cane bagasse, flax, and others, while, etc.

Artificial fibers include Carbon, graphite, glass and aramid fabrics are the most commonly used fabrics for fiber reinforced polymer composites especially for making tribo-components and aircraft structures that encounter harsh operating conditions such as high stresses, speeds, temperatures, (Shukla, 2011). Natural fibers like banana, coir, jute, kenaf, oil palm, and sisal have been used in composites for advanced work like aircraft and aviation, as well as everyday usages like furniture, low-cost housing, and constructions (Erdogan et al., 2019).

Currently, natural fillers are gaining more attention than the synthetic ones due to their low cost, low density, abundance, biodegradability and eco-friendly nature. Polymer composites made with organic fillers usually show exceptional specific properties. Many of such reinforcements were

studied and experimented on; such as Rice husk, Coconut shell (Prabhu et al., 2021), Eggshell (Owuamanam et al., 2021), Doum palm shell (Audu et al., 2018).

Natural polymer matrix composites, on the other hand, have several advantages, including renewable resources, minimal toxicity, and biodegradability. From home appliances to wear-resistant components, these composites have a diverse variety of applications (Biswal & Satapathy, 2016).

Researchers are interested on such fillers because they are both natural and considered as wastes with good potential to be utilized for industrial applications. *Caesalpinia Decapetala* shell filler is one of most commonly available material in our country. This natural reinforcement have advantages due to its light in weight, non-corrosive, low density, lower cost, ease of manufacturing, etc. Due to these properties, it is highly recommended for wear application areas with good mechanical properties. In most of applications, natural polymer composites have been subjected to a range of wear. Dry sliding wear is a common wear mechanism that involves the loss of materials from the surface of the material. Based on the literature review the tribological and mechanical properties of *Caesalpinia Decapetala* reinforced composite were investigated. In this study, the *Caesalpinia Decapetala* shell reinforcement epoxy-based composite was explored by varying the applied load, filler composition, sliding distance and velocity. The composite was fabricated by slowly mixing the ingredients with a glass rod and stirring it with a magnetic stirrer to make a uniform mixture. After that, the composite plate was made using a solid casting technique, and the specimens were trimmed according to ASTM standards. The result of wear rate, specific wear rate, and coefficient of friction were reported. In addition to this, mechanical properties like hardness, compression strength, tensile strength, impact strength, and water absorption of the CD reinforcement epoxy-based composite were examined. Physical characterization was carried out, which included density and void content. Finally, scanning electron microscopy is used to examine the microstructure-properties of the composite material by the utilization of EDS to characterize the morphological and physiochemical structure of the synthesized composite. Statistical tools such as Taguchi Grey Relation Analysis and Artificial Neural Network were utilized to validate the wear results. Based on the outcome results it can be recommended for different applications like construction, transportation, and house-holding applications areas.

1.2 Statement of the problem

Fabrication of composite material has many advantages for meeting human demand in special equipment, system components, and household products. Importing different machines, tools and equipment need lots of investment costs and a long delivery time. While manufacturing in the country will solve such problems. Then again, making sure sustainable development of our economic system should focus on indigenous resources.

Furthermore In our country Ethiopia, there are many fiber content plants are available but the applicability of these plant are not much seen. From those materials *Caesalpinia Decapetala* is the one which is available mostly in our country and it have very strong seed in appearance. Due to this properties significantly increase the mechanical and tribological behavior of CD reinforced based composites. However, this *Caesalpinia Decapetala*'s seeds are not consumed and thereby are considered as waste. It is abundant in our country Ethiopia, especially, in East and West Oromia around Harar. Therefore, in the present work, an investigation of the tribological and mechanical behavior of *Caesalpinia Decapetala* shell epoxy composite was carried out and the application area of these was determined after it composed with the reinforce material.

1.3 Objectives of the Study

1.3.1 General Objective

The general objective of this work is to evaluate the experimental investigation on the mechanical and tribological behavior of *Caesalpinia decapetala* shell particulate reinforced epoxy matrix composites.

1.3.2 Specific Objectives

The specific objectives are as follows:

- Fabrication and physical characterization in terms of density and void content of *Caesalpinia decapetala* polymer matrix composite by varying the *Caesalpinia decapetala* content in 0, 10, 20 and 30 vol.%.
- Examine the mechanical response of *Caesalpinia decapetala* composites in terms of hardness, impact, and compression strength.

- Investigate the dry sliding wear behavior of composite under varying applied load, *Caesalpinia decapetala* composition, velocity and sliding distance. And optimizing Taguchi Grey Relation Analysis and Artificial Neural Network
- Study the structure-property correlation using scanning electron microscopy and elemental composition using EDAX.

1.4 Significance of the Study

Natural fillers are available as low-cost, lightweight, and environmentally friendly alternatives to synthetic fibers in composites. The major importance of this study is:

- It is used for several application areas like construction, transportation and household equipments.
- Fabricating locally reinforced composites from *Caesalpinia Decapetala* shell will increase the use and replacement of the manufacturing process.
- It provides many advantages in household equipment, and construction materials.
- *Caesalpinia Decapetala* based composites help to develop rural industries. They are non-abrasive, reduce corrosion to some extent and hence tool wear costs can be reduced.
- The directed beneficiaries are farmers, engineers, technologists, the transportation industry, researchers in this field, society, and the country as a whole.

1.5 Scope of the Study

This thesis work was delimited to the preparation of composite samples with varying proportions of *Caesalpinia Decapetala* shell through different wt.%. Also, study the tribological and mechanical properties of *Caesalpinia Decapetala* shell particulate reinforced epoxy matrix composite. Evaluation of composites by varying the *Caesalpinia Decapetala* content was carried out to assess the impact of reinforcement. They are very strong in appearance and can significantly increase the tribological behavior of composites. Therefore, physical and tribological

characterization of Caesalpinia Decapetala/Epoxy composite is targeted for the present work and is the prime focus of the work.

1.6 Research Motivation

It is widely regarded that our country is an agricultural-primarily based country. It has a sufficient wet, sunny season and fertile soil. Having this natural appearance gives it different plant species. The Caesalpinia decapetala plant is one of them. It has number of seeds that are visibly hard in appearance. However, these substances are waste in our country, and they grow over a year. Moreover, dried eight or nine months after its seedlings. Nobody can produce it, and it truly degrades. To remedy this problem it is critical to apply its seed shell as an effective reinforcement that can be used to prepare composite materials for envisaged applications.

1.7 Structure of the Thesis

This section provides a top-level view of the thesis work. It is categorized into five chapters for this work. Chapter one is introduction and background of the study. Chapter two deals with literature review of related works in specific sections and also highlights the key inferences. In chapter three, materials and methods are discussed. Chapter four shows the result and discussion of the fabricated composites theoretically as well as graphically represented. The final chapter provides a summary, the conclusion of the work, and recommendations for future research.

CHAPTER TWO

LITERATURE REVIEW

2.1 Introduction

A literature review is a summary of previously published research on a particular subject. A complete academic paper or a component of an academic publication, such as a book or article, might be referred to as a thesis. Diverse literature from various sources relating to the current topic was studied in this section, and a research gap was identified.

2.2 Composite Materials

Over the previous 21th century composite materials plastics and ceramics have been the main developing materials (Mikellp. Groover, 2010). The amount and number of fabrication of composite materials have grown progressively, piercing and leading new markets persistently. Current composite materials constitute an important amount of the engineered materials market ranging from everyday goods to sophisticated place applications (Venkatesh & Venkatesan, 2015). While composite has already established their value as mass-saving materials, the present challenge is to make their price effective. The efforts to produce economically good-looking composite components have resulted in several innovative manufacturing techniques currently being used in the composite industry.

Composite material is multi-phase engineered material in which two or more dissimilar materials are combined together but remain distinctively identifiable in the mixture. It not only maintains the characteristics of the constituent components but also exhibits new characters which are not possessed by any of the individual components Mostly they have strong fillers surrounded by a weaker matrix material to obtain enhanced properties which is different from the individual components (Jiao et al., 2019).

The Reinforcing fillers (particulate or fiber) offer the physical and mechanical properties, for stiffness and strength of the composite while the matrix serves to hold, keep and dispense the reinforcement, and also to transmit and distribute the load to the fillers (Ganesan et al., 2018).

Composite are applied in a wide-ranging areas: from straw mud walls to steel bar reinforced concrete, and tire cord, etc.

From analysis of the composition and the internal structure of composite materials we found that it includes three basic physical phases. The term phase indicates a homogeneous material, such as a metal or ceramic in which all of the grains have the same crystal structure, or a polymer with no filler. The primary phase forms the matrix, which is continuous, while the secondary phase (reinforcement) is scattered and imbedded in the matrix (Doddamani, 2019).

The other phase is formed at the interface between the reinforcement phase and matrix phase. In microstructural level, it was found that near the interface of reinforcement phase and the matrix phase becomes a complex structure, which creates a strong adhesion bond between them. The bond forms with direct bonding, formation of solution between the phases or by adding a third component to bond the primary and secondary phases. The structure and morphology have an impact on macroscopic performance of composites, so the microscopic area in the interphase changes in structure and properties. Thus, it becomes the third phase of composites, which is called interphase. Therefore, a composite material is composed of primary (matrix) phase, secondary (reinforcement) phase and interphase. The structure and the nature of these three phases, their configuration and interaction, as well as the relative content determine the performance and characteristics of composite materials.

2.3 Classification, of composite

Several criteria are taken into account for classifying composites. Based on its existence it is classified into two: natural (traditional) and synthetic composites. The naturally existing ones such as wood are classified under traditional while humans combine modern composites in a controlled way to get the desired properties and structure. Based on the type of reinforcement used, and the matrix material.

Composites are classified based on the type of strengthening: Figure 2.1 was classification of Composites based on the type of reinforcement.

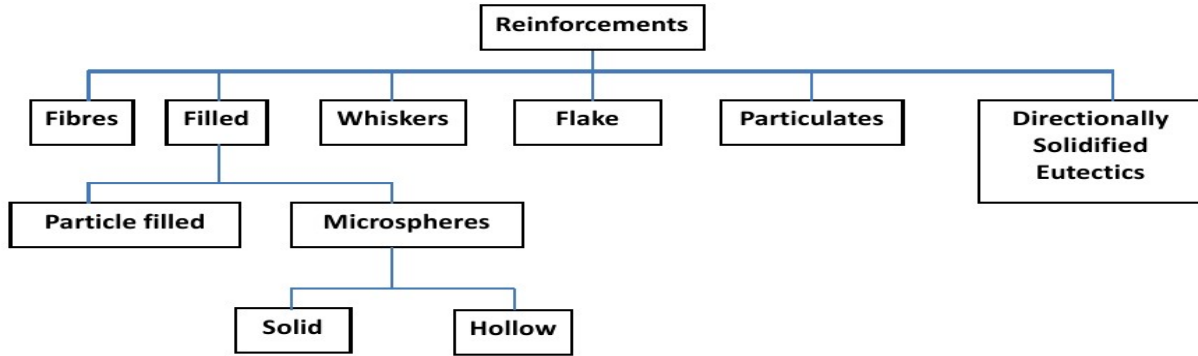


Figure 2. 1 Classification of composite based on types of reinforcements (Sumithra and Reddy, 2017)

a) Particulate Reinforced Composite

Particulate, ranging in size from microscopic to macroscopic, a composite that strengthens is a particle of all the dimensions more or, less equal is called particulate reinforced composites. As shown in figure 2.2 the arrangement of particulate reinforcement with a matrix. Particulate fillers are working to improve high-temperature performance, reduce friction, increase wear resistance, and reduce shrinkage (Ashik and Sharma, 2015). The particle will also donate the load with the matrix, but to a smaller extent than a fiber. The presence of these powders results in dispersion hardening of the matrix, in which dislocation movement in the matrix material is restricted by the microscopic particles. A particulate reinforcement will therefore get better stiffness but will not normally strengthen (Mikell, 2010).

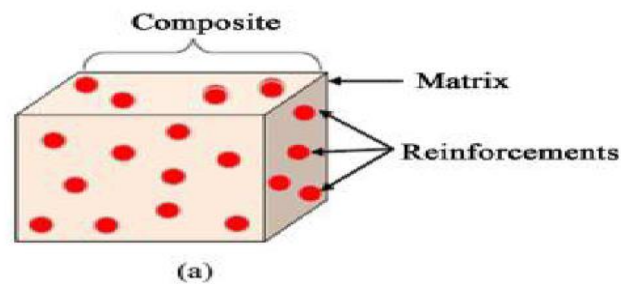


Figure 2. 2 Particulate reinforced composite (Ashik and Sharma, 2015)

b) Fiber-reinforced Composite

Fiber-reinforced composites include reinforcements with lengths greater than the cross-sectional dimension. Fiber reinforcement represents a physical rather than a chemical means of modifying

material to make it suitable for various engineering applications. Composites are made up of fibers in the matrix structure and can be classified by fiber length. Composites with long fiber reinforcements are, referred to as continuous fiber reinforcement composites, while composites with short fiber reinforcements are referred to as discontinuous fiber reinforced composites. These can usually be classified as shown in figure 2.3 below

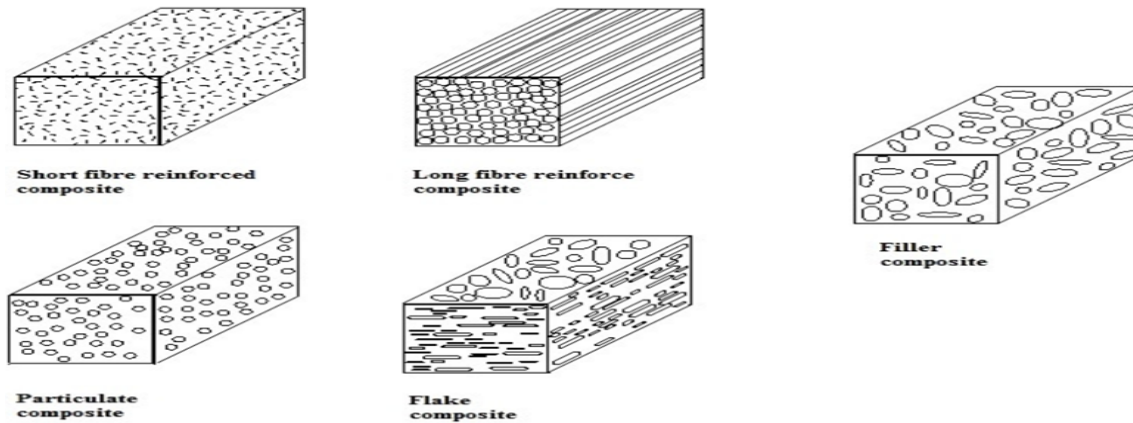


Figure 2. 3 Classification of composites based on the structure (Dipen Kumar, 2019)

c) Hybrid fiber-reinforced composites

Hybrid fiber-reinforced composites are those in which two or more types of fibers are reinforced in a single matrix structure. Fibers can be incorporated unidirectional or bidirectional into the matrix structure of continuous fiber composites and transfer loads from the matrix to the fiber in a very simple and effective manner. Discontinuous fibers must be of sufficient length to ensure effective load transfer and limit crack growth to avoid material failure in the case of brittle matrices. The arrangement and orientation of fibers determine the properties and structural behavior of composite materials. An improvement in properties such as impact strength and fatigue strength can be observed when using chemically treated natural fibers.

d) Structural composite:

Structural composites are the combination of laminate or sheets, which are, constructed layer over layer. The laminates are usually fibers oriented in changing manner from layer to layer as shown in figure 2.4 (a). It also includes sandwiched panels, where they are joined by a form of adhesives and a rigid and light weighted composite is formed as shown in figure 2.5 (b). Sandwich panels have the benefits of lightweight, large bending stiffness, and energy absorption. Laminates are

sheet constructions by stacking layers (also called plies or lamina) in a specified sequence as shown in figure 2.4. The layers are often in the form of prepare“ (fibers prefilled with partly cured resin) which are consolidated in an autoclave.

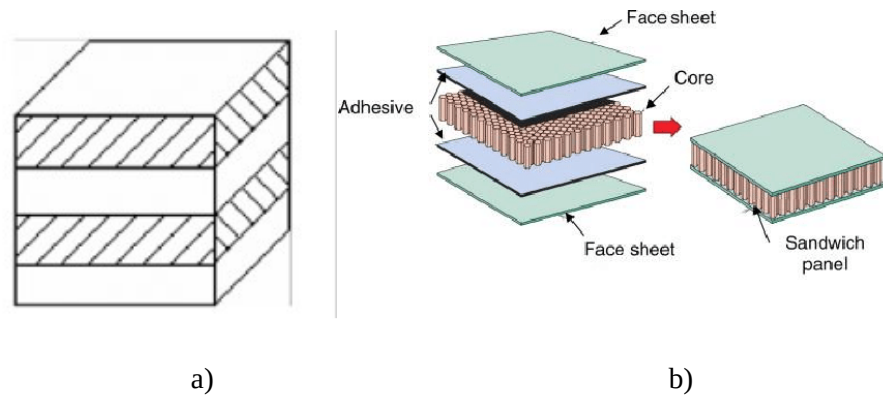


Figure 2. 4 Structural composites a) Composite laminate b) Sandwich panels (Groover, 2010).

e) Synthetic fiber

Synthetic fibers are synthetic fibers created by humans, as opposed to natural fibers generated directly from living organisms such as plants (such as cotton) or animal fur. They are the outcome of scientists' significant research into replicating naturally existing animal and plant fibers. Synthetic fibers are, made by extruding fiber-forming ingredients via spinnerets and then weaving them into a fiber. Synthetic fibers are shown in figure 2.5 below.

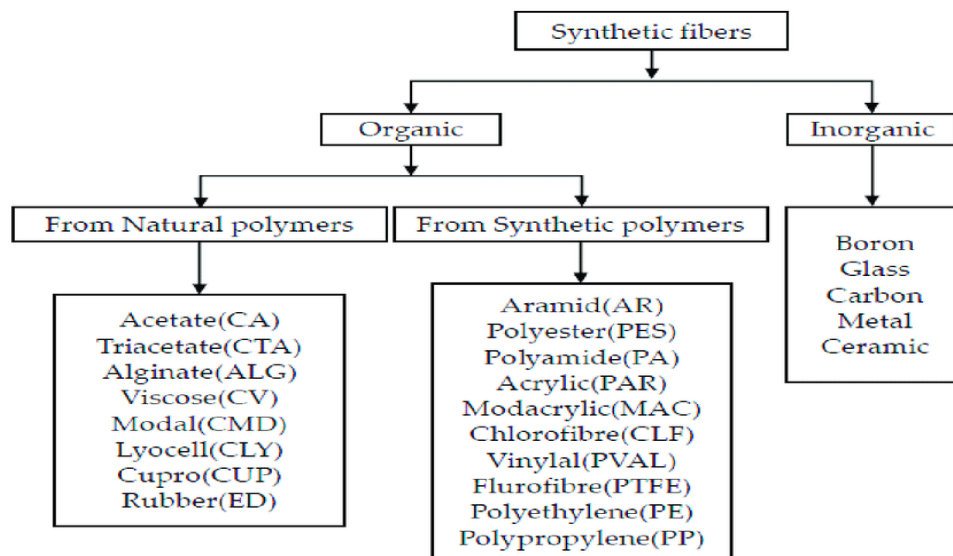


Figure 2. 5 Classification of synthetic fiber (Muda and Mustapha, 2018).

f) Natural fibber

The major advantage of fiber-reinforced composites is to offer high strength and modulus that are either comparable to or better than many traditional polymer materials. Natural fibers are light in weight and low cost when compare to synthetic fibers these natural fibers are classified as illustrated in figure 2.6 below.

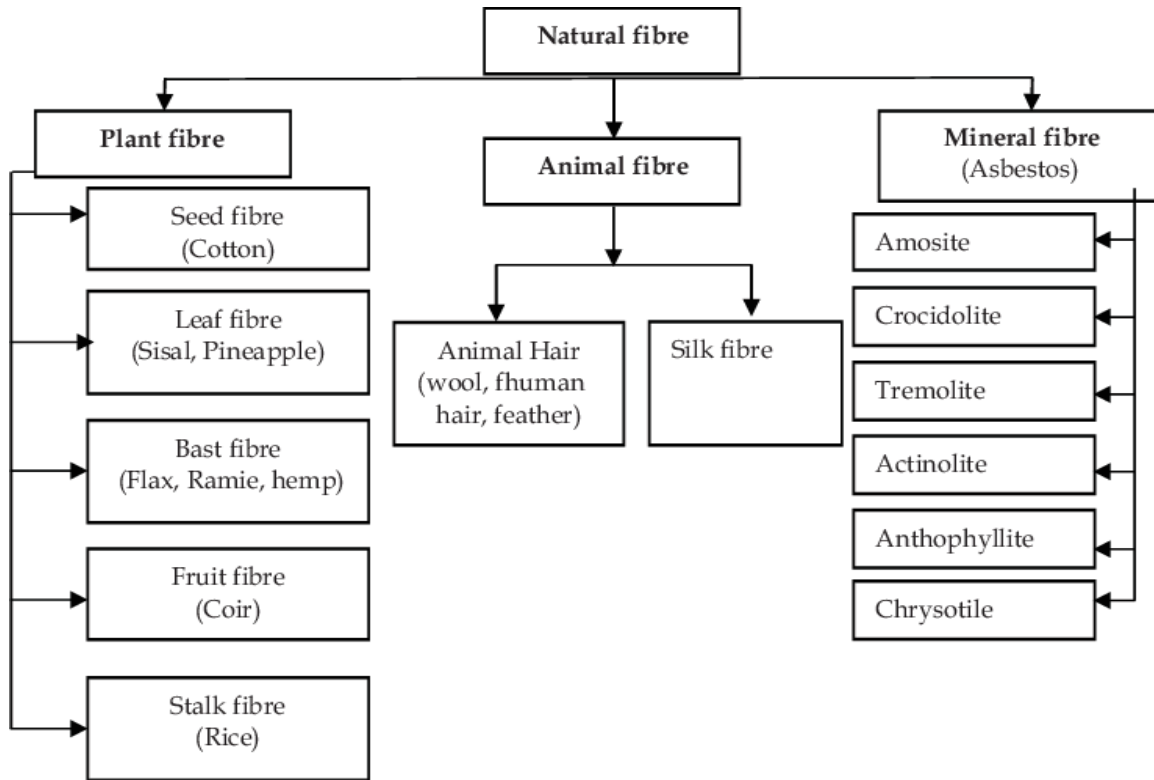


Figure 2. 6 Classification of natural fiber (Muda and Mustapha, 2018).

2.3.1 Matrix material

Matrix is a fundamental material phase of a composite that functions as a binder, holding the fibers in place and transferring external load to the reinforcement and protection to the reinforcements from environmental damage. Furthermore, the matrixes are thought to be softer and more ductile. Because of their lightweight, great strength, multiple degrees of design freedom, and simplicity of

processing and shape, polymer matrix composites, were selected for several application area(Kumar & Madurai, 2010)

Composite materials based on the matrix can be classified as polymer matrix composite metal matrix composite and ceramic matrix composite as shown below in figure 2.7

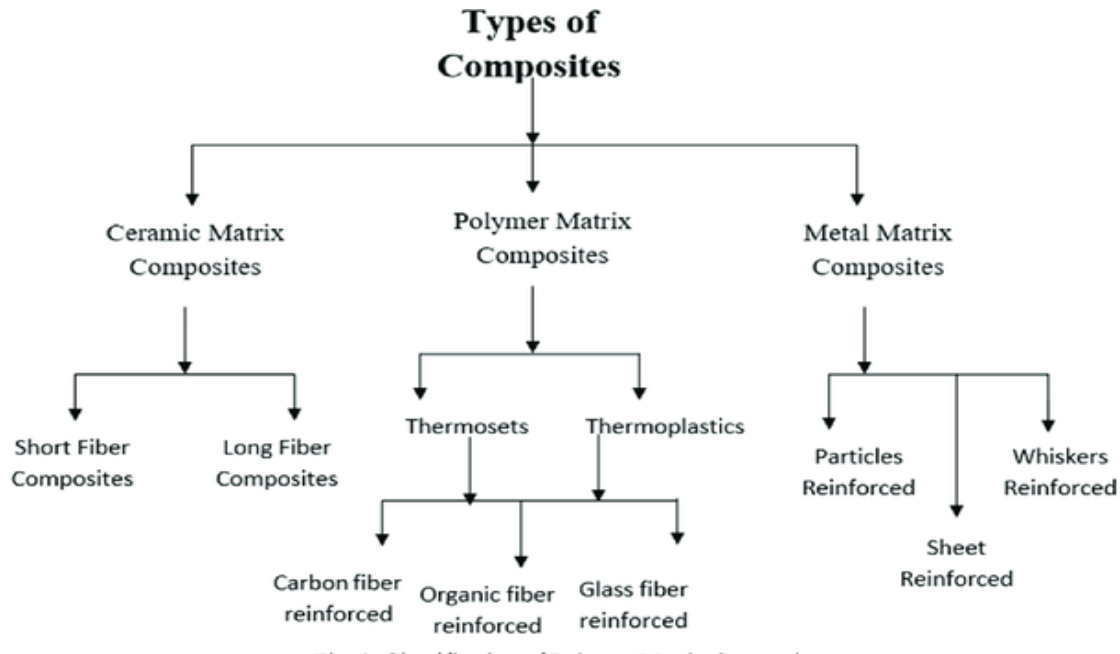


Figure 2. 7 Classification of composite based on the matrix material (Kashyap, 2014)

a) Polymer Matrix Composites

The most commonly used matrix materials are polymeric. In general, the mechanical properties of polymers are insufficient for structural purposes. In particular, their strength and rigidity are low compared to metals and ceramics. These difficulties are overcome by reinforcing other materials with polymers. Second, the processing of PMC does not have to involve high pressure and does not require high temperature. In addition, the equipment required to produce PMC is simpler. For this reason, PMC quickly evolved and soon became common for structural applications, of which glass fiber reinforced plastic (GRP) is the best known example, using ceramic fibers in a plastic matrix. For the above reason, this work is only interested in polymer matrix composites. They are made up of long-chain molecules made up of many simple repeating units. The most commonly used polymer composites in more industries are thermoset and thermoplastic polymer matrix

composites. These fiber-reinforced polymer composites are found in products that people interact with in everyday activities (Mikell .P. Groover, 2010).

Thermosetting polymers are polymers that have been set to a solid form and cannot be restored to their unusual uncured form. Composites made with thermoset matrices are strong and have very good fatigue strength. They are extremely brittle and have low impact strength. They are often used for high-temperature applications due to their thermoplastics. Thermoset composites are generally cheaper and easier to make because the liquid resin is very easy to work with. Thermoset composites are difficult to recycle because the thermoset cannot be reshaped or reshaped; only the reinforcing fiber used can be recovered (Mikell. P. Groover, 2010)

Thermoplastic polymers are polymers that can be shaped, melted, and reshaped without changing their physical properties. Thermoplastic matrix composites are tougher and less brittle than thermosets, with very good impact strength and damage tolerance. Thermoplastic composites are less dense than thermosets, making them a viable alternative for weight-sensitive applications. The manufacturing process of thermoplastic composites is more energy intensive due to the high temperatures and pressures required to melt the plastic and impregnate the fibers with the matrix. The energy required makes thermoplastic composites more expensive than thermosets (Patil, 2012).

B) Metal-matrix composites

MMC typically uses silicon carbide fibers embedded in a matrix composed of an alloy of aluminum and magnesium, but other matrix materials such as titanium, copper, and iron are increasingly being used. Typical applications of MMCs include bicycles, golf clubs, and missile guidance systems; An MMC made of silicon carbide fibers in a titanium matrix is currently being developed for use as the skin (hull material) of the US National Aerospace Plane. (Groover, 2010)

b) Ceramic matrix composites

CMC is the third main type and examples include silicon carbide fibers fixed in a matrix of borosilicate glass. The ceramic matrix makes them particularly suitable for use in light, high-temperature components such as parts for aircraft jet engines. The main advantages of ceramic materials over other materials are their resistance to oxidation and deterioration at elevated temperatures, their high melting points, and their high compressive strength. Unfortunately,

ceramics suffer from susceptibility to brittle fracture and therefore have relatively low fracture toughness values. One method of increasing the fracture toughness of ceramic is to reinforce it with fibers, whiskers, or particles. Crack propagation is severely hampered by the presence of the reinforcing phase. (Groover, 2010)

2.4 *Caesalpinia decapetala*

Caesalpinia decapetala is a recurring plant that grows in tropical and subtropical areas. It is a sprawling, thorny, and noxious poisonous shrub that invades forest margins, smothering edges, and choking native vegetation. Widely grown as a live fence, often becoming naturalized in wooded grasslands and upland evergreen bushlands, this robust thorny hardy, thorny, evergreen shrub grows from two to four meters high or climbs to 10 meters or more (Iem et al., 2005). It often forms dense thickets. The stems are covered with minute golden hair, tiny golden hairs, and thorns which are spines, straight or hooked, numerous, and not in regular rows or confined to nodes. The leaves are dark green, paler lighter underneath, not glossy, and up to 30 cm long with leaflets up to 8 mm wide. The flowers are pale yellow in elongated erect clusters of upright racemes 10 cm to 40 cm long. Fruits are brown, woody pods, flattened, unsegmented, smooth, sharp, sharply beaked at the apex, and the tip, is about 8 cm long as shown in Figure 2.8. It is used perversely before used before this study for several applications, like Medicine, fodder (leaves, pods), bee forage, feed, mulch, ornamental, live fence, necklaces (seeds) so on (Chaudhary et al., 2021).

Local Medicinal Uses *Caesalpinia decapetala*: Seed powder is used against malaria and is also used to remove freckles from the face (Shah, 2015). The plant root is used as a laxative (Hassan et al., 2020). Used in Ayurveda and Sidha. Roots, stems, and pods for pain relief. Decoction of root juice is used to treat sprains and muscle swelling (Shah, 2015). Seeds astringent, anthelmintic, analgesic, antipyretic, antimalarial; Insecticide for seeds and roots. Flower infusion for bronchitis, asthma, and malaria fever. *Caesalpinia decapetala* fruits can be eaten, but the seeds are toxic (Ahmad Jan et al., 2017). Furthermore local handicraft and used as a landscaping plant as a hedge or an ornamental in China and elsewhere (Chaudhary et al., 2021). The fruits and bark are rich in tannin. With an oil content of 35%, the seeds serve as a source of lubricant and soap (Gargiullo, 2005). Plants are grown as field boundaries in Nepal (Chaudhary et al., 2021) finally

environmental uses include Agroforestry; Boundary, barrier or support; soil improvement. It is used as an ornamental plant.

2.4.1 *Caesalpinia decapetala* originates

Caesalpinia decapetala originates from is native to tropical and eastern Asia but is now a serious weed in many locations such as South Africa, Tanzania, Zimbabwe, Hawaii, Portugal, New Zealand, and Norfolk Island. It has become an important invasive plant in South Africa and Hawaii, where it can take over large tracts of agricultural land, limiting land and restricting animal movement. *C. decapetala* has been introduced around the world as an ornamental, hedge, and erosion control species, species and has been reported to tend towards weediness and be weed prone in some situations such as in South Africa, Australia, New Zealand, and the Pacific islands. It is known to have been introduced to New Zealand in 1965 and has subsequently become invasive. In Hawaii, *C. decapetala* was first recorded in 1888 (Starr et al., 2003). In the West Indies, *C. decapetala* was first recorded in Puerto Rico in 1900 (US National Herbarium). In South Africa, *C. decapetala* was introduced around 1904, 1904, and by 1961 it is reported as invasive in the Kruger National Park at the same time in Ethiopia.

2.4.2 *Caesalpinia decapetala* in Ethiopia

Ethiopian originated stinging *Caesalpinia decapetala* particulate filled /local name Qontr particulate filled is a plant from Acacia or Fabaceae or Leguminosae family which is used as the source of particulate filled for domestic use as west in rural parts of our country having a comparable mechanical property with other particulate filler fibers. So far, this fiber was not commonly well known as reinforcement in the preparation of composite. In the case of domestic application, *Caesalpinia decapetala* /Qatar particulate filler has a comparable property with other particulate filler reinforcement composite. It is classified under the category of fibers extracted from the best the plant like a palm trees, coconut, fly ash, etc. The genus *Caesalpinia* contains more than 100 species. It prefers fertile soil to grow 6 up to 10 m in height. *Caesalpinia decapetala* is used as a landscaping plant as a hedge or an ornamental, and pale in Ethiopia. As shown in figure 2.8 below.

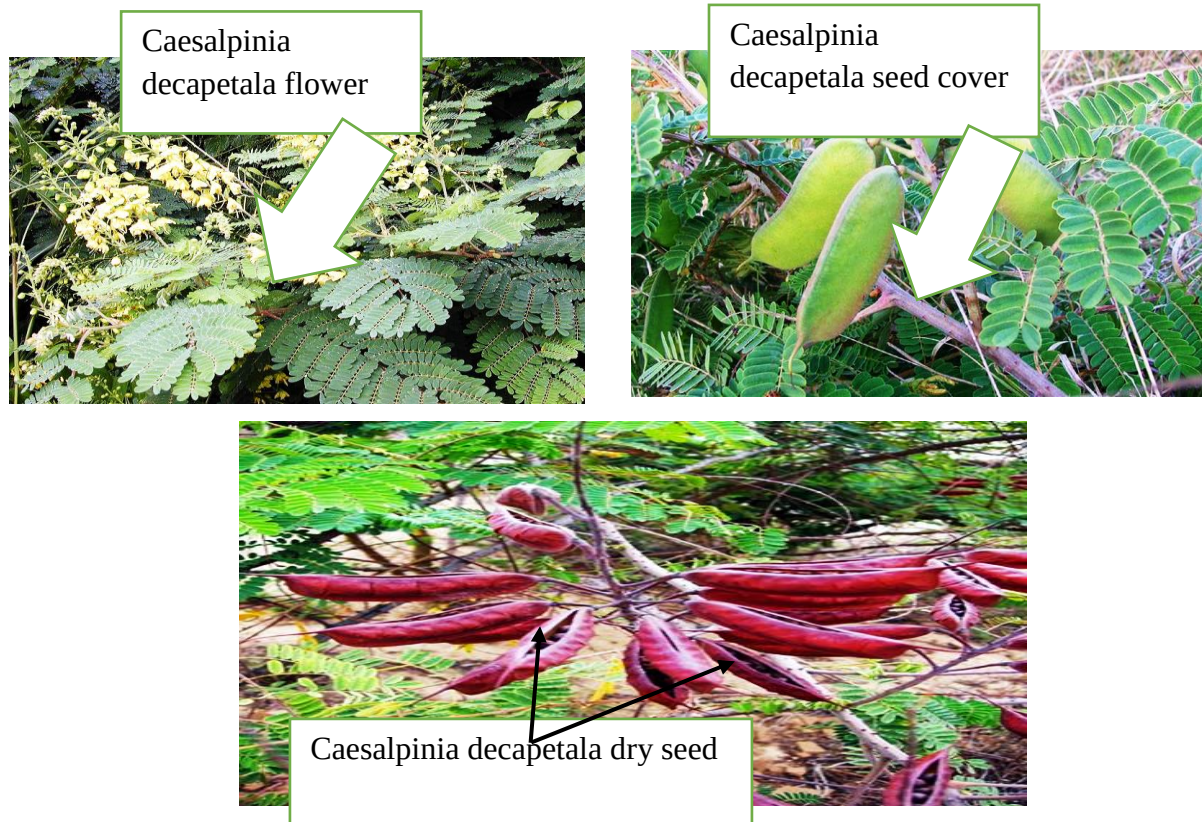


Figure 2. 8 *Caesalpinia decapetala* seed and seed coat

Table 2. 1 Properties of natural fiber and synthetic fiber

Aspects Property	Natural fiber	Synthetic fiber
Mechanical property	Moderate	High
Moisture sensitivity	High	Low
Technical Thermal sensitivity	High	Low
Resource	unlimited	Limited
Environmental Production	Low	High

Recyclability	Good	Moderate
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2.4.3 Applications of the natural composite

Natural fiber composites can be extremely cost-effective material for the accompanying applications:

- Storage devices: post-boxes, grain storage silos, etc.
- Furniture: chair, table, shower, bath units, etc.
- Electric devices: electrical appliances, pipes, etc.
- Everyday applications: lampshades, suitcases, helmets, etc.
- Transportation: automobile and railway coach interior, boat, etc.

2.5 Mechanical behavior

The mechanical behavior means how the composite material behaves when it is under mechanical loading or action. As the composite materials form according to our required mechanical loading and thermal conditions, they have good strength and durability. The mechanical properties of the various samples prepared were, determined by tensile compression, impact, flexural, and hardness tests. The International Standard ASTM (D638, D3410, D256, D790 E-384,) standard test method, respectively, carried out these tests. Each prepared specimen was, secured on the machine platform. The defamations of the sample for all tests were, based on the above ASTM standard.

Kiran et al., 2018, Conducted the experiment with a large strength-to-weight ratio; composite materials can fulfill numerous qualities required for diverse technical applications. Because of their high specific strength and stiffness, polymer-based composites are suitable materials for engineering applications. The addition of fillers to polymer matrix composites increases their mechanical properties. Mechanical parameters of polymer matrix composites with various fillers, such as tear strength, impact strength, and hardness, are considered based on this. To increase mechanical qualities, polymer matrix composites of various polymers/fibers are blended with various quantities of fillers such as aluminum oxide (Al_2O_3), silicon carbide (SiC), and calcium. From the analysis, fillers were found to exhibit improved mechanical properties of polymer matrix

composites. Also the main factors like dispersion of fillers and distribution, adhesion, and interface between filler and polymer matrix influence the mechanical properties of polymer composites.

Awad et al., 2019, used marble dust particles as reinforcement materials in a polymer composite matrix for the experiment. In this study, these particles will be used as reinforcement in a polypropylene (PP) matrix to create a novel composite material. The thermal and mechanical properties of varied particle sizes (35 - 1350 μm) and the weight percentage of marble dust particles (10 - 70 percent by weight) were examined. The addition of dust particles to the PP matrix increased the composite materials' thermal and mechanical properties (such as flexural strength, compressive strength, Young's modulus, and hardness). The mechanical behavior of these composites depends on numerous parameters, such as the contact area between the dust particles and the PP matrix, the amount of dispersion, the dust particle size, and the dust to PP ratio. The test results were examined, and the best circumstances were identified. A scanning electron microscope was used to investigate the fracture surface of the tested samples as they were flexed (SEM). The sample decomposed in three phases, according to the TGA curves. A mass loss of 1.879 percent happened in the temperature range of 25 to 596.77 $^{\circ}\text{C}$. Between 597 $^{\circ}\text{C}$ and 770 $^{\circ}\text{C}$, there was a considerable mass loss of 10.3 percent, whereas, between 770 $^{\circ}\text{C}$ and 940 $^{\circ}\text{C}$, there was a mass loss of 1.866 percent (0.4685 mg). In the temperature range of 25–1100 $^{\circ}\text{C}$, the total mass loss of marble dust particles is 14.09 percent. In addition to metals, ceramics, and polymers, a fourth material classification can be identified: composites. Composites are multifunctional material systems that offer properties not available with any single material. They are cohesive structures made by physically combining two or more compatible materials that differ in composition and properties, and sometimes in shape.

Suresh and Sudhakara, 2019, used marble dust particles as reinforcement materials in a polymer composite matrix for the experiment. An innovative method was, used in this research. Powdered red mud and aluminum industry waste were, employed as particle reinforcement in this study to create a reinforced polymer composite. The effects of particle loading on the composites' mechanical and tribological properties were, examined. For material characterization, the red mud particles were, submitted to an X-ray diffraction investigation. Light microscopy and scanning electron microscopy were, used to examine the morphology of composite materials. Due to its outstanding impact strength and superior wear resistance, an ecologically friendly composite with

great mechanical and tribological properties was, created and researched, which can be employed in non-metallic gears.

Raghul et al., (2020) investigated the effect of the addition of silica nano particles content in the sisal-palm-glass fiber-reinforced composite. The composite was fabricated by hand lay-up method followed by hot press molding and their mechanical behaviors like tensile strength, flexural strength and impact strength were evaluated. The results showed that maximum tensile and flexural strength were achieved when 2% Nano-silica added to the sisal-palm-glass fiber and the result showed that the incorporation of 1% of Nano-silica has significant effects on the Charpy impact behavior.

2.6 Dry sliding wear

This topic contains a summary of the research conducted by several authors on the dry sliding wear behavior of polymer matrix composites. The authors have conducted research on the analysis of composites based on their properties. This summary of the literature contains fabrication techniques, methodology, and methods for experimenting on various composites. The dry sliding wear behavior of composites is investigated by varying applied loads, and sliding distance.

Wear tests were generally performed on pin on disc apparatus. On the other hand, a wear test measures the changes in conditions caused by friction, and the result is, obtained from deformation, scratches, and indentations on the interacting surfaces. Wear arises when there is relative motion between two solid bodies under the impact of the load, which can slide or roll. The kind of wear depends on the respective material qualities in addition to movement or the impact of the tribological load, which is why the following forms of wear were, separated into abrasive wear, erosive wear, impact wear and surface fatigue wear, which are briefly detailed below.

a). Abrasive Wear

The loss of material from the surface in abrasive wear happens as a result of sliding abrasive particles in the form of abrasion under load, as well as dust particles from the surroundings that get stuck in the sliding and cause the loss (H. Sharma et al., 2020).

b). Erosive Wear

Erosive wear can be defined as the process of metal removal due to impingement of solid particles on a surface. Erosion is, caused by a gas or a liquid, which may or may not carry entrained solid particles, impinging on a surface. When the angle of impingement is small, the wear produced is closely analogous to abrasion. When the angle of impingement is normal to the surface, material is, displaced by plastic flow or is dislodged by brittle failure (Naik et al., 2021). Same other types are illustrated in table 2.2.

Table 2. 2 Symptoms and appearance of different types of wear

Types of wear	Symptoms	Appearance of the worn-out surface
Abrasive	Presence of clean furrows cut out by abrasive particles.	Grooves
Erosion	Presence of abrasives in the fast moving fluid and short abrasion furrows.	Waves and troughs.
Impacts	Surface fatigue, Small sub-micron particles or formation of spalls.	Fragmentation, peeling and pitting.
Delamination	Presence of subsurface cracks parallel to the surface with semi-dislodged or loose flakes.	Loose, long and thin sheet like particles

Patnaik et al., 2016, manufactured epoxy resin matrix composite reinforced with fly ash particles using ultrasonic stirring technology. The composite pin-on-disc laydown test was completed and compared according to the Taguchi layout test. An orthogonal array showed and tested the impact parameters such as % fly ash debris, traditional load, sliding speed, and track spacing on the composite. Optimizes a minimum wear rate, friction pressure, and friction coefficient. Due to this investigation the minimum wear rate was obtained at 40 wt.% of fly ash, 400 RPM of speed, 10N of load, and 40cm of truck distance.

Shahapurkar et al., 2021, developed the crumb rubber reinforced epoxy matrix composite and studied the influence on the dry sliding wear behavior of epoxy composites. Application tests are conducted for three grades of crumb rubber (10,20 and 30 wt.%), applied load,30,40, and 50N,

and three ranges of sliding distance 1, 3, and 5 km and optimize by using Taguchi-coupled gray relational assessment (GRA) essential component analysis (PCA) the influence of crumb rubber on the tribological response of composites. Additionally, the coefficient of friction shows a decreasing trend with an increase in crumb rubber content, indicating the effectiveness of reinforcing crumb rubber in an extensively used epoxy matrix that means at a load of 50N, 30 wt.% of crumb rubber and 3km sliding distance they get minimum wear rate and coefficient of friction. The post-test temperature of the disks will increase with an increase in the applied load, even if it decreases with an increase in filler loading.

H. Sharma et al., 2020, conducted the effect of food scrap fillers (citrus lime peel) on the wear behavior of epoxy composites experimentally. The Citrus Limetta peel fillers, 15% by weight of the resin, were used to make epoxy composites with three different particle sizes in micrometers i) 100–250 μm ; ii) 350–500 μm and iii) 650–800 μm). The dry slip properties of the developed epoxy composites were tested using the pin on disc art on test equipment. The investigations were carried out with a track radius of 60 mm and a constant sliding distance of 3000, 4000, and 5000 m. The results show that the incorporation of Citrus Limetta peel fillers into the epoxy matrix significantly improves the wear resistance of the developed composites. It was observed that the maximum wear resistance and coefficient of friction were found with the smallest packing's at 100–250 μm of filler particle size, 3000 sliding distance, and 30N load.

P. Kumar et al., 2018, Studied the effect of hybrid reinforcement on dry sliding wear behaviour of vinyl ester (VE) composites. With the coconut shell powder (CSP) content the specific wear rate fell more rapidly. Percentage CSP is more protective against wear from sliding Reinforcement form, geometry and dispersion lead to improved wear resistance. These outcomes are very important for the manufacture a new composite integrated with surface-treated reinforcing materials such as Carbon fiber and CSP that will have foremost application in automotive sliding or bearing components.

Waste marble dust is solid waste generated from the cutting and polishing of pieces of marble on construction sites and in the marble processing industry (Nayak and Satapathy, 2020). This paper reports the use of this waste as a filler material in particle-filled polymer composites. Polyester-based composites are produced with different weight fractions of marble waste and the dry sliding

wear behavior of these composites is investigated. Wear tests are performed using a pin-on-disc tester based on Taguchi's Orthogonal Array under ASTM G 99-05 standard. The effects of different parameters on the specific wear rate of the composites are studied and an optimal combination of parameters for the lowest wear rate is obtained. The morphologies of the worn surfaces are studied with a scanning electron microscope to determine the mechanism of wear of the composites under different conditions.

The tribological conduct of epoxy polymer composites strengthened with periwinkle shell particles was studied using a pin-on-disc wear rig below dry sliding conditions (Asuka and Aigbodion, 2016). By utilizing non-carbonized and carbonized periwinkle shell particles composites were fabricated at 0, 10, 20, 30, and 40 wt.%. The effects of wear parameters like applied load, sliding speed, and percent of periwinkle shell particles addition on wear rate were investigated. Confirmation of the experiment's behavior using ANOVA testing parameters suggests that periwinkle shell particles, sliding velocity, and implemented load had a considerable impact on the wear rate. The outcomes show that the addition of periwinkle shell particles as reinforcing substances in epoxy composites increases the wear resistance of the composite significantly.

The wear output of coconut shell powder (CSP) and coir fiber reinforced polyester resin composites was experimentally investigated by K Vignesh et al., 2019, The effect of the concentration of coconut shell powder and coir fiber and the sliding distance in the composite weight loss was analyzed. The result shows that the friction coefficient increases with load increases in composites containing polymer matrix in the CSP and coir fiber. The wear rate is observed to increase with the increases in the load applied. With the addition of CSP and coir fibre, the wear rate decreases. When the load applied increases, friction also increases at the material's contact surface and spinning disk. For the polymer matrix composites, the addition of the CSP and coir fiber serves as reinforcement. The wear resistance improves thanks to this addition.

Z.Robin et al., 2017, Studies Tribological work has an impact on the global production of natural composites. The composites filled with Musaceae fiber bundles and cured with two hardeners; rises of about 15% in global styrene double bond conversion observed using both hardeners for tidy matrix and composites, indicating that any changes in resin structure occur during the tribological study. Determining the usable alternatives for controlling the therapeutic environment to monitor tribological behavior.

A S Singha and Thakur, 2008, Studied on new series of green composites involving Hibiscus sabdariffa fiber as a reinforcing material in urea formaldehyde (UF) resin based polymer matrix. Initially urea formaldehyde resin prepared was subjected to evaluation of its optimal properties. Then reinforcement of the resin with Hibiscus sabdariffa fiber was attained using engineered resin in the particle sizes of three different types, short fiber and long fiber. Reveals traction resistance. Madhu and Balasubramanian, 2020, focused on the utilization of natural fibers as reinforcement in polymer composites and they have been persistently utilized in the field of auto, airplane, and biomedical ventures. In this work, carbon fiber polymer composites (CFPC) with seaweed fillers were manufactured utilizing the hand-layup technique. Sliding wear tests were conducted under dry condition on the created specimen. The impact of working boundaries like sliding speed, load, sliding distance, and seaweed filler stacking on explicit wear rate was explored. In this examination, an optimum wear rate was acquired for the sliding speed of 1.5 m/sec, applied load of 2 N, and sliding distance of 1000 m in 10 wt. % of seaweed-filled composites. The rising pattern of effect strength has been all around perceived from the dunking pattern in specific wear rate on seaweed growth in the CF10SW epoxy composite.

Vigneshwaran et al., 2019, investigated the use of waste marble dust as a filler material in particle-filled polymer composites. Polyester-based composites are available with varying weight amounts of marble dust debris and the dry sliding wear performance of these composites is considered. Wear trials are performed using a pin-on-disc mechanical test rig based on the Taguchi L25 orthogonal rig per ASTM G 99-05. The effects of different limits on the respective wear rate of the composite materials are taken into account and an optimal combination of parameters for the lowest wear rate is determined. Given the experimental information, a predictive model using the neural network is used to determine the determined wear rate of the composites over a broader range of working limits inside and outside the test area.

N. Doifode and P. Nalawade, 2018, Studied Epoxy Composite Friction and Wear Behavior Reinforced under Dry Condition with Ipomoea Carnea Fiber. This paper describes the effect of normal wear load (micron) and friction coefficient of reinforced / reinforced Ipomoea Carnea epoxy composites with different weight percentage of the fiber fraction (10 percent, 20 percent, 30 percent, 40 percent wt). To measure the micron wear and friction coefficient of Ipomoea Carnea

reinforced epoxy composites, the 12 mm diameter fiber composite pin was manufactured and tested for each fiber weight concentration under three separate loading conditions (10N,20N,30N). Measurement of friction and wear was done. The Pin-on - Disk Tribometer acquired wear data in real time, and time (Sec) friction coefficient. Each test was performed under dry condition for 10 min (600 Sec), and data for wear (micron) and friction coefficient with time variation (100 Sec interval) were recorded from the monitor.

N Vijaya Kumar et al., 2014, Investigated Properties of Wear on Jute Reinforced Polypropylene Composites at different loads and at different sliding velocities. The composites with varying percentage of fiber weight (5%, 7.5%, 10%, 12.5% and 15%) using injection moulding process. The friction and wear loss coefficient is studied for different loads and sliding speeds. Thimmana Gouda [26], Studied wear characteristics on Natural Fiber Hybrid Polymer Composite for Orthopedic Implants. Identified foremost changes in tribological properties of 36% Hybrid Natural Fiber Polymer Composite compared to 12% and 24%. Finally, 36% Material can be used for orthopaedic implants or replacement of human bone.

G Rajasekhar et al., 2019, Studied on Coconut Fiber Reinforced Polyamide Matrix Composite to its Wear Behavior. The coconut fiber reinforced composites have substituted the most commonly produced plastic fiber (Glass, Kevlar) strengthened polymer composites in many applications. Concluding with an increase in normal force results in an increase in wear rate due to bonding between the coconut fiber and matrix is inadequate to withstand normal force resulting in damage to the matrix thereby increasing wear rate. The coefficient of friction decreased with regular force, but it declines less with extreme force.

P. Rajasekhar et al., 2017, Examined the impact of date palm seeds on tribological polyester composite behavior under Various Research Conditions. It revealed that the friction coefficient for polyester composites packed with date seed powder falls by 15% with a rise in filler under heavy contact strain. In addition to rising the size, the friction coefficient rises by up to 10%. Unfortunately, the date seed filler continuously decreases from 15% to 25% lower the wear resistance of the planned composites under heavy contact pressure and fast slipping pace. The load applied (contact pressure) and the sliding velocity have a direct impact on the polyester composite wear rate, which is filled with date seed.

H Aireddy, and SC Mishra 2011, Examined tribological actions of polymer matrix composites reinforced by Bio-waste. The composite is observed to show the maximum corrosion intensity at an impingement angle of 90 °. Worn rate of erosion is minimised by increasing coir dust volume. Abrasive wear resistance declines with decreased usual load, and improves with increased amount of fibre.

2.7 Taguchi Gray Relational Method and An artificial neural network

Taguchi's technique has been used to find optimum process parameters at optimal cost (Hussain et al., 2020). This method has restraint to optimization of single response problems. To increase the usefulness of this technique for multi-response optimization problems, Grey relational analysis (GRA) proposed by Deng Julong can be incorporated with Taguchi method (Liu and Lin, 2010). In GRA, the multi-response problem is converted into a single response problem and then optimal solutions are evaluated. The GRG is used to optimize process parameters and the optimal process parameters setting can be evaluated by the Taguchi method using GRG as a measure of performance index. Selection of Orthogonal array provides that all controlled variables are equally considered in experiments. The minimum number of trial run must be more than or equal to the total degrees of freedom. Each row of orthogonal array represents different combination of level parameters (Hussain et al., 2020). An artificial neural network (or simply neural network) consists of an input layer of neurons (or nodes, units), one or two (or even three) hidden layers of neurons, and a final layer of output neurons (Rout & Satapathy, 2012).

2.8 Research Gap

Caesalpinia decapitata are undervalued materials considered as wastes. In spite of the hidden potential within these wastes, they are not currently used for any purpose in Ethiopia. With its composition, low cost, good properties, biodegradable nature and abundance, it has a great capability to be a competitive bio filler. There are researches made, of Caesalpinia decapitata being utilized as a medicine and seed powder is used against malaria and is also used to remove freckles from the face. There are also studies conducted on different types of Caesalpinia decapitata shell powder as reinforcement for polymer matrices. However, there are no works reported on Caesalpinia decapitata as reinforcement in polymer matrix composites and is the main focus of the

present work. Further researches are required for using this organic filler in adding value and enhancing the composite's properties. In this paper, experimentation and testing were done to find out the properties of a *Caesalpinia decapitala* reinforced composite and suggest applications for it.

CHAPTER THREE

MATERIALS AND METHODS

3.1 Introduction

In this section, the materials and methodology of the composite materials were listed for this study. The methodology for planning the test procedure is based on the related publication journals. Based on this we prepared the composites. This composite is the mixture of two phases namely matrix and reinforcement.

3.2 Materials

Composite materials are the combination of matrix and reinforcement materials; the matrix materials are metal matrix, polymer matrix and ceramic matrix. The reinforcement materials are natural and synthetic materials can be used to enhancing the property of composite materials.

3.2.1 Matrix

Lapox L-12 epoxy resin with K-6 hardener, supplied by World Glass Fiber Plc, Addis Ababa, Ethiopia is the matrix resin used and is shown in Figure 3.1. Lapox L-12 is a liquid, unmodified epoxy resin of medium viscosity that can be used with various hardeners for making composites. K-6 hardener is a low viscosity room temperature curing liquid hardener. Table 3.1 presents the properties of epoxy resin and hardener.

Epoxy resins also referred to as poly-oxides, are epoxy-based reactive pre-polymers and polymers. Epoxy refers to the epoxy functional group as a whole (Shetty, 2019). Curing is the term for the crosslinking reaction, and the co-reactants are known as hardeners or curing agents. Epoxy resins are low-molecular-weight monomers that shrink very little when cured. In this state, they can be partially cured and stored. The chemical and corrosion resistance of the hardened epoxy resins is excellent, as are the mechanical and thermal qualities (Mousavi et al., 2021).



a)

b)

Figure 3. 1 a) Epoxy resin and b) hardener used for sample preparation

Table 3. 1 Property of epoxy resin and hardener.

Description	Unit	Value
Epoxy resin (LAPOX L-12)		
Color	Blue	0.8
Epoxy value	Eq./kg	5.35
Viscosity at 25 °C	MPa	11850
Volatile content at 105 °C/h	%	0.4
Hydrolysable chlorine	wt. %	0.08
Marten's value	°C	150
Hardener (K-6)		
Color	GS	0.8
Viscosity at 25 °C	MPa	10
Pot life at 80 °C	S	4140
Gel time at 80 °C	S	7080

3.2.2 Reinforcement

The reinforcement materials are the secondary face in composite materials. It is used for enhancing the strength of the materials. Reinforcement materials made up of synthetic and natural. In this study, we use *Caesalpinia decapetala* particulate as reinforcement.

A suitable quantity of *Caesalpinia decapetala* seed was collected from different areas of our country, Ethiopia. The filler was extracted by hand as shown in figure 3.2 B. The size of the particulate filler is between 100 and 105 μ m as illustrated in figure 3.2 E. while the all preparation parameters are shown in figure 3.2 A, E, C, D, and E. *Caesalpinia Decapetala*'s abundant availability in Ethiopia makes it a viable option to use in utilitarian composites. They are very strong in appearance and can significantly increase the tribological behavior of composites in this material. *Caesalpinia Decapetala* is not consumed and thereby is considered a waste. Therefore, in the present work, an investigation of the tribological and mechanical behavior of the aforementioned composites was carried out so that the developed composites can be utilized for some envisaged applications. Furthermore, several equipment's are used for the preparation of this polymer matrix composite. Those are listed in table 3.2 below.

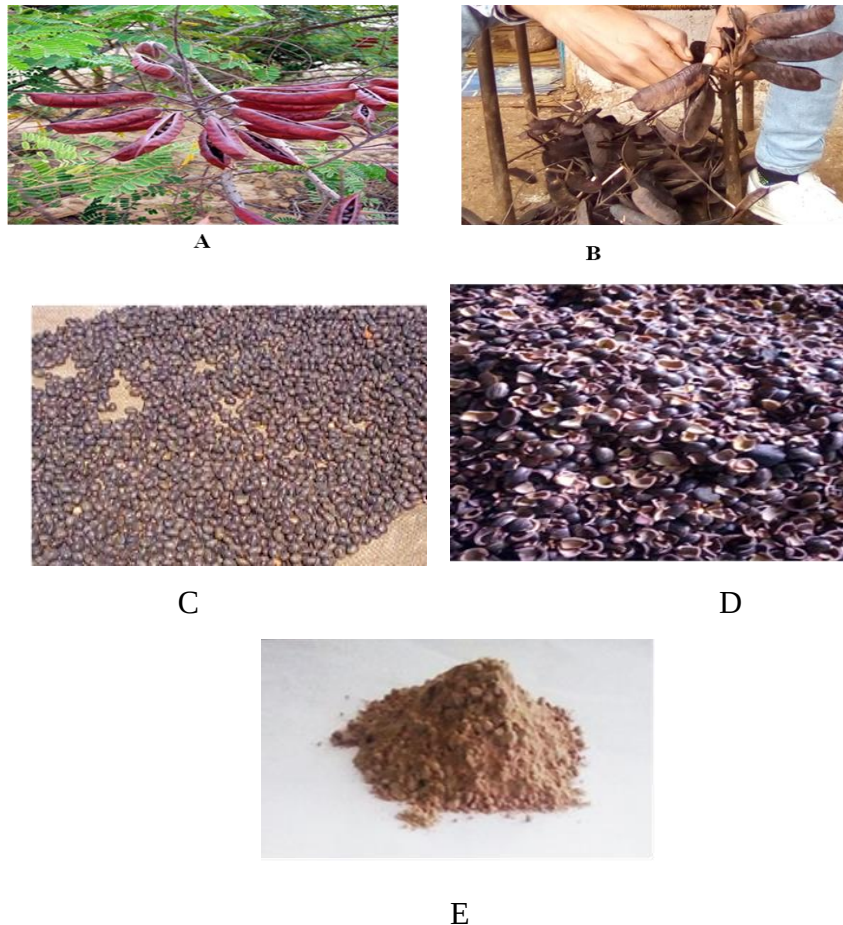


Figure 3. 2 Process parameter for preparations of CD particulate reinforcement.

Table 3. 2 List and amount of material required to prepare the composite

Item	Unit	Quantity
Caesalpinia decapetala	Kg	1
Hardener	Liter	1
General purpose resin	Liter	2
Sandpaper	pics	15
Soft	pics	5
Molds	pics	2
Acetone	Liter	1
Glass stirrer	pics	2
Mixing container baker	pics	60, 500 and 1000 ml
Steel rule	pics	1 m
Magnetic stirrer	pics	1
Weighing machine	pics	1

3.2.3 Petroleum jelly

The petroleum jelly is shown below in Figure 3.3. It is a releasing agent during the fabrication of composite. A release is a chemical that prevents other materials from adhering to surfaces. It can be used to solve problems with mold release or die cast in composite fabrication. Applying a thin coat of jelly and wiping off or polishing the haze/obscure/part using a cloth towel is recommended for better performance of the material.



Figure 3. 3 Pure petroleum jelly

3.3 Methodology

3.3.1 Composition of *Caesalpinia decapitala*

Based on researches reviewed and trial experimentation, the volume percentage compositions of *Caesalpinia decapitala* shell particles in the composite were determined. Table 3.3 illustrates designations given for the samples prepared with varying weight percentages of filler. Table 3.3 shows the nomenclature of different sample composites. Designation Filler content (vol.%)

Table 3. 3 Nomenclature of different sample composites

Designation	Vol.%
E	0
ECD-10	10
ECD-20	20
ECD-30	30

E refers to pure epoxy whereas CD designates *Caesalpinia decapitala* shell and the number indicates the weight percentage of filler in the composite. The samples were made with equivalent volume percentages on two different molds; mold number 1 (smaller) and mold number 2 (larger).

3.3.2 Mold preparation

Two different-sized molds were made from locally available materials: RHS steel, steel plate and bolts these molds were made from a steel material with dimensions: (100x100x20 mm) and (300x300x20 mm) mold cavity. The molds were used for casting the composite to the desired size

for sample preparation. The size of the sample was (300x300x3.4 mm) and (100 x 100x12 mm). The molds were thoroughly cleaned with laboratory-grade acetone and tissue paper as in Figure 3.4. Acetone is a universal cleaner and solvent used for several purposes. Petroleum jelly was applied on the surfaces of the mold cavity for easy removal of the sample. Petroleum jelly is an economical and easily available releasing agent. It also supports in filling the space between the RHS steel and the plate steel.



Figure 3. 4 Preparation of mold a) Cleaning mold 2 (larger mold 300x300x30mm) with laboratory grade acetone, b) Petroleum jelly applied on mold 1 (Smaller mold 100x100x30mm).

3.3.3 Composites preparation

Fiber-built up composite created by a few techniques relying on the state of the part to be made. That large number of strategies fall under a standard called polymerization. Polymerization is the most common way of joining an enormous number of synthetic molecules together to shape an inflexible construction. Composite manufacturing procedures are divided into two categories: open molding and closed molding. The composite is processed in a two-piece mold of open molding. The processing method of composite preparation is an open molding type.

Based on this method, the CD shell particle reinforcement epoxy-based matrix composites were prepared and the tribological and mechanical behavior of the composites were investigated for construction, automotive parts, and housing applications. The process parameter was shown in figure 3.5 bellow

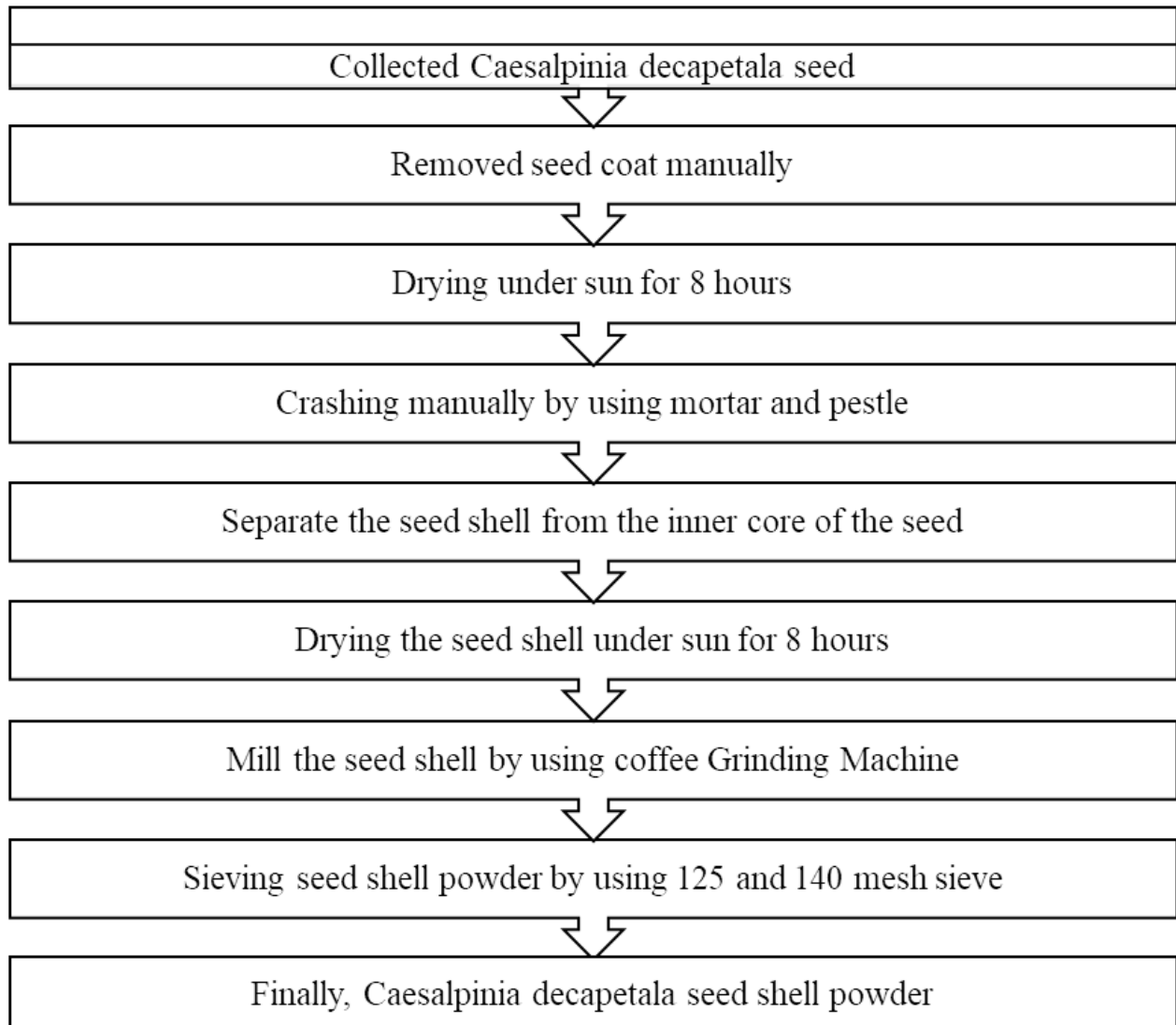


Figure 3. 5 Flow diagram of Caesalpinia decapetala seed shell particulate reinforcement preparation

Table 3. 4 General work specification

Factors	Specifications
Matrix type	Epoxy resin (Thermoset polymer)
Fiber/matrix volume content	Determined after fabrication

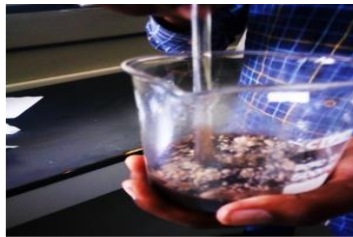
Size of Caesalpinia decapetala seed shell particulate	100 to 105 micrometer
The wt.% of the matrix	90,80,70
wt% of filler material (CD)	10, 20, 30
Steel mold dimension (mm)	100x100x12 and 300x300x12
Fabrication method	Solid casting techniques

3.3.4 Solid casting process

It is one method of fabrication of composite materials in ceramic and polymer matrix composite. The mold has been cleaned jauntily by using acetone and polyvinyl alcohol to avoid the dust and rust from the surface of the rectangular steel mold. Then the mold was coated with jelly as a releasing agent to easily remove from the mold. A jelly coat is applied to the prepared mold with the help of our hands slowly with a uniform thickness to get the better surface finish expected. The epoxy resin matrix was prepared with a pre-determined composition of 10% with a catalyst, which facilitates the drying process of the composite. It was stirred with simple mechanical stirring to mix properly, in addition to this stirring by using, a magnetic stirrer to make homogeneity of solution before pouring the mixing solution till the mixture became warm (exothermic reaction) as shown in figure 3.6b&c. Furthermore, pore the homogeneous solution into the mold to get the final product of Caesalpinia decapetala particulate reinforcement epoxy based composite as illustrated in figure 3.6d. Finally, keep the sample in the mold for the prepared cured sample at room temperature for 24 to 72hrs, as indicated in figure 3.6e.



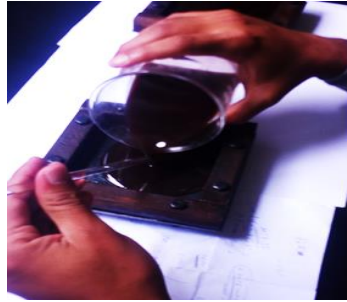
a)



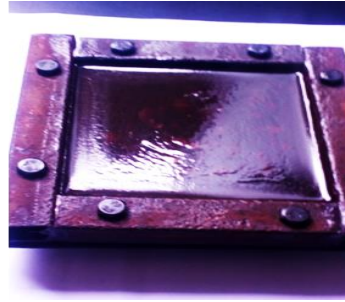
b)



c)



d)



e)

Figure 3. 6 composite preparations procedure.

3.3.5 Curing and removal of sample

The composites were removed from the mold and post cured in the air for 72 hours. The samples were left to cure for hours and they were easily removed from the molds as shown in figure 3.7. The size and unevenness of the particles also had a great impact in the dispersion and agglomeration of the particles. The use of magnetic stirrers for mixing improves homogenization and reduced sedimentation. After curing, the samples were taken out of the mold and changed to the required shape and size for conducting the test after 72 hours. Flow diagram of the proposed work is depicted in figure 3.8.

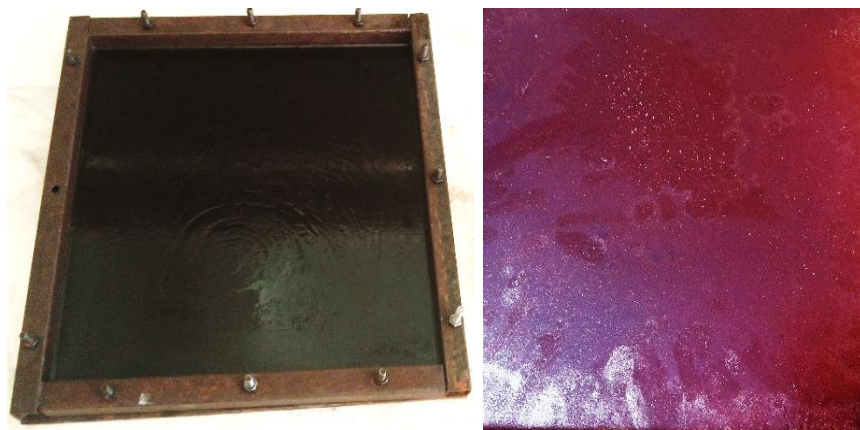


Figure 3. 7 Samples cured and removed from mold

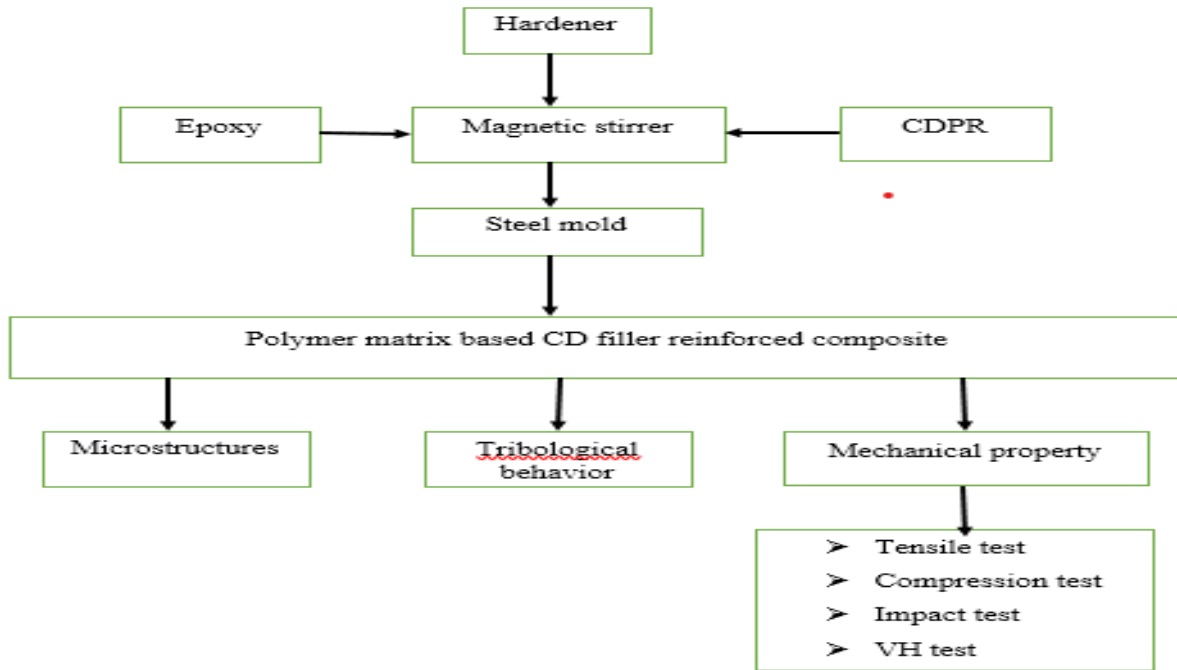


Figure 3. 8 The flow diagram of mixing CD reinforcement with matrix and conducted test.

3.4 Experimental procedure and setups

The experimental design is a set of procedures that are designed to test a hypothesis. The process has five steps: define variables, formulate a hypothesis, design an experiment, assign subjects, and measure the dependent variable

3.5 Measurement of Density and Voids content:

The density of composite samples has been determined as per ASTM-C 639 standard through the rule of mixture. Certain properties of composite material can be computed using a rule of mixtures, which involves calculating a weighted average of the constituent material properties. Density is an example of this averaging rule. The ways of determination of density are illustrated in equations (3.5 to 3.10).

$$m_c = m_r + m_m \dots\dots\dots 3.1$$

$$V_c = V_m + V_r + V_v \dots\dots\dots 3.2$$

$$\rho_r = \frac{m_r}{V_r} \dots\dots\dots 3.3$$

$$\rho_m = \frac{m_m}{V_m} \dots\dots\dots 3.4$$

Form Equation (3.1 and 3.2) we obtain equation (3.4)

$$\rho_c = \frac{m_c}{V_c} \dots\dots\dots 3.5$$

$$\rho_c = f_m \rho_m + f_r \rho_r \dots\dots\dots 3.6$$

Where:

m_c :- mass of composite (Kg)	f_m :- volume fraction of matrix
m_r :- mass of reinforcement (Kg)	f_r :- volume fraction of reinforcement
m_m :- mass of matrix (Kg)	ρ_c :- density of composite in (Kg/cm3)
V_c :- volume of composite (cm3)	ρ_r :- density of reinforcement in (Kg/cm3)
V_r :- volume of reinforcement (cm3)	ρ_m :- density of matrix in (Kg/cm3)
V_m :- volume of matrix in (cm3)	

The void content of composite samples has been determined as per ASTM D-2734-70 standard procedure through Archimedes principle. The volume fraction of voids (V_v) in the composites was calculated by using the equation 3.7.

$$V_v = \frac{\rho_t - \rho_a}{\rho_t} \dots\dots\dots 3.7$$

Where: V_v is Voids content, ρ_a actual (experimental) density in (Kg/cm³) , ρ_t is theoretical density in (Kg/cm³)

3.6 Mechanical testing

Mechanical testing is a method of determining a material's mechanical properties. It can be used to assess a material regardless of its geometry, as well as under specific geometric constraints. When analyzing a material, a range of mechanical test methods can be utilized, some of which can

indicate the acceptability of a corrosion-prone material. In addition to the tribological test, a mechanical test was also conducted to identify the mechanical properties of the material. From these mechanical tests, one can conduct the tensile tests, compression tests, impact tests, Vickers hardness tests, characterization, and finally conduct water absorption test to know the property of the materials.

3.6.1 Compression test

Compression tests are conducted using computer-controlled universal test system with a 50 kN load cell. This test is one of the characterize mechanical properties of Caesalpinia decapetala particulate reinforced with epoxy resin composite and it is executed based on the ASTM D3410 standard. The overall dimension of the tested sample according to ASTM standards and based on the universal testing machine of Bishoftu Ethiopia Deface University College to test the compression strength, 120mm of length, 30mm of width, and 3mm of thickness were prepared for testing. The specimens were subjected to axial compression load until fracture using UTM. Samples were placed between upper and lower compression anvils shown in figure 3.9 B. The compressive strength is defined by the peak stress at the end of the elastic region. At least five samples of each volume fraction are tested to check for repeatability.

$$\sigma_c = \frac{P}{A} \dots\dots\dots 3.8$$

Where: P- Load applied and A is the cross-section area

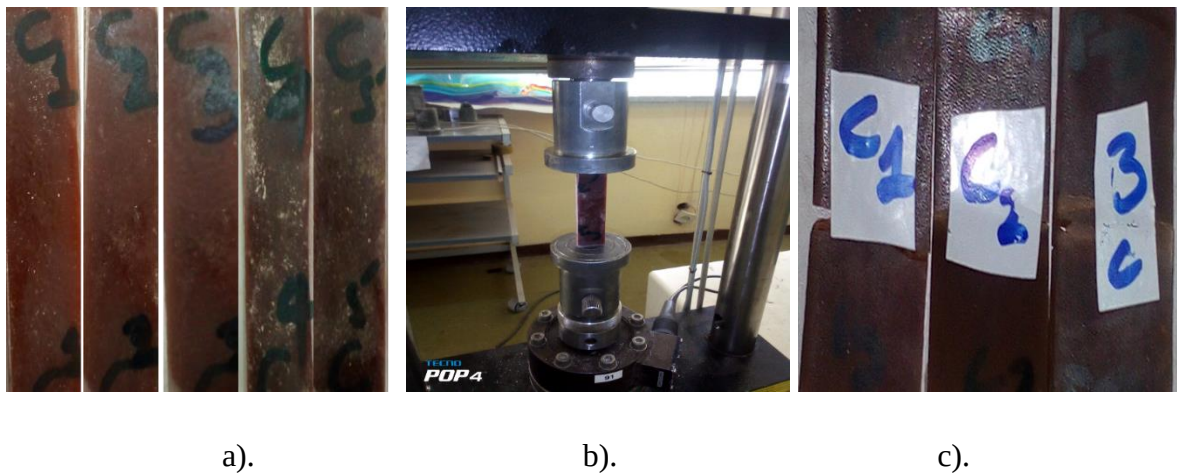


Figure 3. 9 Compression test setup a) before test b) during c) after test.

3.6.2 Tensile testing

A computer-controlled universal test system with a 50 kN load cell is used for tensile. Samples with the dimension of narrow width 13 mm, length of the narrow section 57 mm, overall width 19 mm, and overall length 165 mm are considered. All the samples have a gauge length of 50 mm and the distance between grippers is maintained at a distance of 115 mm as shown in figure 3.10. A constant crosshead displacement rate is maintained at 5 mm/min during the tests. The test is conducted until the fracture of the sample. The acquired load and displacement data are used to calculate the stress, respectively. The average modulus and strength values of five specimens for each sample are reported.

$$\sigma_t = \frac{p}{A} \dots\dots\dots 3.9$$

Where, P- Load applied and A is the cross-section area

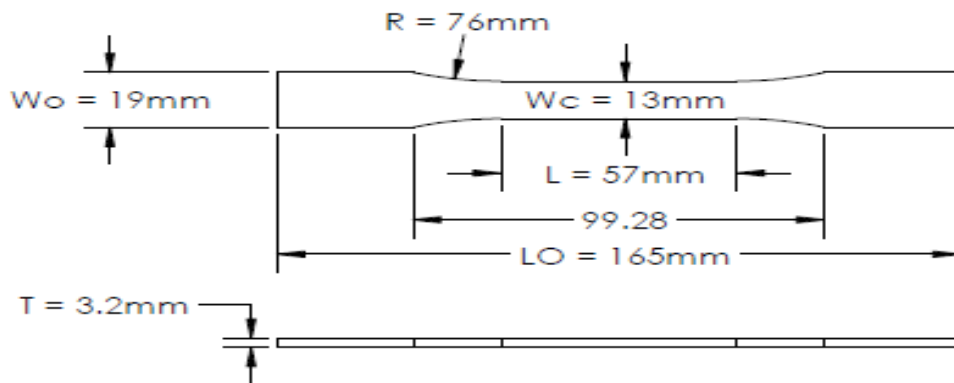


Figure 3. 10 Overall dimensions of a tensile test specimen



Figure 3. 11 Tensile test setup and tested sample.

3.6.3 Charpy Impact Test

The Charpy impact test is also known as the Charpy V- notch test. It is a standardized high strain-stress test that determines the amount of energy absorbed by a material during fracture. This absorbed energy is a measure of a given material's notch toughness and acts as a tool to study \ temperature-dependent ductile-brittle transition. It is widely used in the industry since it is easy to prepare and conduct, and results can be obtained quickly and cheaply. Figure 3.12 shows the dimension of the specimen for the impact test.



Figure 3. 126 Chirpy test specimens (Naik et al., 2021)

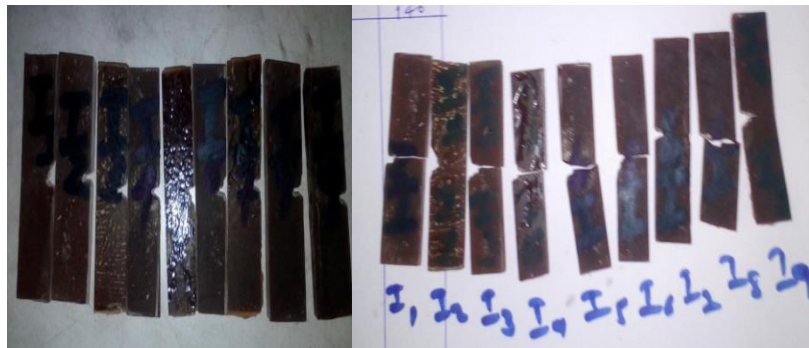


Figure 3. 13 Impact test specimen

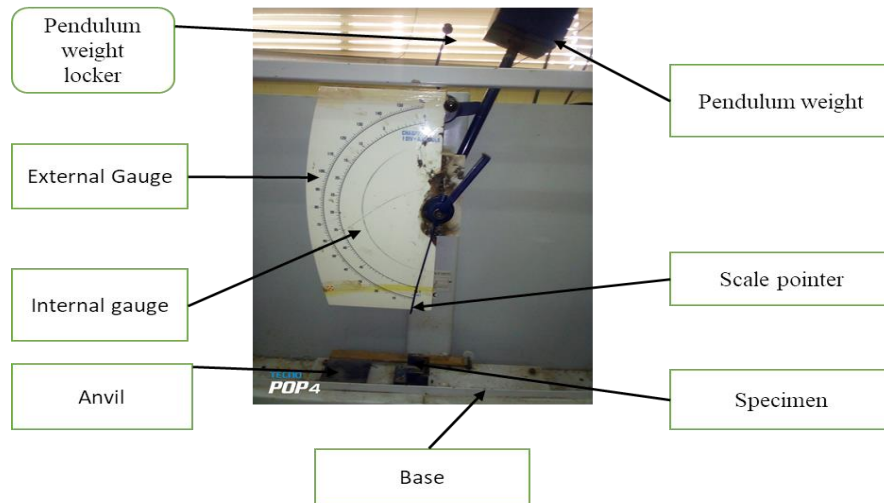


Figure 3. 14 Specimen set up and parts of Charpy impact tester machine

The impact test was carried out on samples measuring 55 mm in length, 10 mm in width, and 5.5 mm in thickness. With an 18-kilogram weight-mounted pendulum, the hammer has an impact at high speed as shown in figure 3.14 above. The specimen's notch should be cut in the opposite direction as the striker. Impact testing quantifies the energy required to shatter a material and can be used to determine the material's toughness.

3.6.4 Hardness Test

The hardness of a substance is a loosely defined concept that has a variety of connotations based on the individual involved experience. Hardness is frequently associated with deformation resistance. Depending on how the test is carried out, there are three broad types of hardness measures. Scratch hardness, indentation hardness, and rebound, or dynamic, hardness are the three types. Only indentation hardness is of significant engineering significance.

3.6.5 Vickers hardness testing

Vickers hardness testing, also known as micro hardness testing, is mostly utilized for small components, thin sections, and case depth work. An optical measurement system is used in the Vickers method. The micro hardness test protocol, ASTM E384, describes a range of low stresses that are measured and converted to a hardness value using a diamond indenter as shown below in Figure 3.15 and all the rest of the images are in the appendix. It is great for evaluating a variety of materials, but test samples must be properly cleaned so that the size of the impressions can be

measured. The Vickers scale uses a square base pyramid-shaped diamond for testing. Typical loads are 5 and 10 kilograms, however, "Macro" Vickers loads can weigh up to 30 kg. The indenter for the Vickers hardness test is a square-base diamond pyramid. The included angle between the pyramid's opposite faces is 136°. This is known as the diamond pyramid hardness test because of the shape of the indenter as shown below. The diamond-pyramid hardness number (DPH), or Vickers hardness number (VHN, or VPH), is defined as the load divided by the surface area of the indentation. This area is computed in practice using microscopic measurements of the impression's diagonal lengths.

$$VH = 0.102 \frac{2F \left(\frac{\sin 136^\circ}{2} \right)}{d^2} \dots\dots\dots 3.10$$

where:

Hv is Vickers hardness value

F is applied force for indentation

d2 is indentation surface

d2 = (d1+d2)/2 d1, d2 value of diagonal 1 and 2



Figure 3. 15 Hardness test setup

3.6.6 Water absorption test

The water absorption capabilities of a *Caesalpinia decapiala* particulate reinforced epoxy matrix composite were investigated as illustrated in Figure 3.16 using ASTM D570. The content of water

absorbed was determined using a precision 4-digit scale after samples were removed at regular intervals and weighed immediately after wiping away water from the surface. The samples were immersed in distilled water at room temperature as shown in figure 3.17, and the water absorption test was performed using distilled water a variety of times. The samples were withdrawn from the container and their surface moisture was removed after predetermined time intervals. Accurate digital weighing equipment was used to determine the sample's water uptake content. The absorption of distilled water was tested for 24, 48, 72, 96, 120, 144, 168, 192, 216, and 240 hours. Natural lowland *C. decapitala* was synthesized with epoxy to make the samples. The following formula was used to compute the water absorption percentage: shown in equation 3.11 below. The Chemistry department at Adama Science and Technology University conducted a water absorption test.

$$W_{\%} = \frac{W_2 - W_1}{W_1} * 100\% \dots\dots\dots 3.11$$

Where,

$W_{\%}$ is percentage of water absorption

W_1 is initial weight of sample before immersed

W_2 is final weight of sample after immersed

The test was carried out by measuring the initial weight of the sample using a precise weight balancer having accuracy of measuring weight 0.0001g

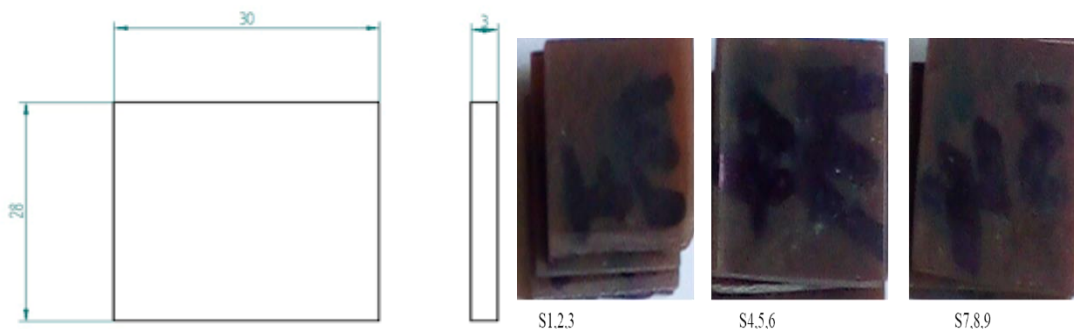


Figure 3. 16 Specimen dimension for water absorption (Raju et al., 2012)

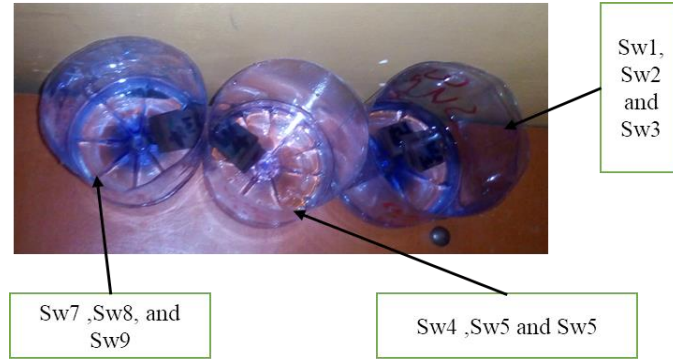


Figure 3. 17 Water absorption test

3.7 Preparation of specimen for tribological test

Caesalpinia decapitala reinforcement was used to make composites with mixing polymer matrix. After that, 10 percent by weight of hardener was added to the solution of the composite, then mix manually for 5 minutes before being poured into steel molds with the dimension of 100X100X20 mm (Shahapurkar et al., 2021). Cast slabs were cured for 72 hours at room temperature and trimmed to a uniform size according to the ASTM G-99 standard (12 x 12 x 25.4 mm thick) for dry sliding wear tests. Then they were mounted on pin-on disc tester machine to conduct the experiment. This prepared sample was trimmed by a water jet to minimize the residual stress from the trimmed sample.

Calculation for Wear

Wear rate was estimated by measuring the weight loss of the specimen after each test. The weight loss was calculated by taking the weight difference of the sample before and after each test. The weight loss:

$$\Delta w = (w_a - w_b) \dots\dots\dots 3.12$$

Where Δw is the weight loss in (g) and w_a and w_b are the weight of the sample after and before the abrasion test in (g). The abrasive wear rate (W_r), is calculated by using the following formula:

$$w_r = \frac{\Delta w}{L} \dots\dots\dots 3.13$$

Where ‘ W_r ’ is the wear rate in m^3/km , Δw is the weight loss in gm. and ‘ L ’ is the sliding distance in m.

The volumetric wear rate W_v (m^3/s .) of the composite is related to density (ρ) and the abrading time (t) was calculated using the expression,

$$w_v = \frac{\Delta w}{\rho_t} \dots\dots\dots 3.14$$

For characterization of the abrasive wear behaviour of composite, the specific wear rate is employed. This is defined as the volume loss of the composite per unit sliding distance and per unit applied load. Often the inverse of the specific wear rate can be expressed in terms of volumetric wear rate. The specific wear rate (W_s) can also be calculated by using the equation:

$$W_s = \frac{\Delta w}{V_s F_n} \dots\dots\dots 3.15$$

where ‘ w_s ’ is the specific wear rate in m^3/Nm , ‘ Δw ’ is the weight loss in grams, ‘ V_s ’ is the sliding velocity in meters per second, and ‘ F_n ’ is the applied load in N. Experimental results of the wear test of different test pieces (10, 20, and 30% by weight of *Caesalpinia decapetala*) at different test conditions.

3.7.1 Dry sliding wear test

The tribological characteristics of polymer matrix and *Caesalpinia Decapetala* shell powder particulate reinforcement composite were experimentally assessed by dry sliding wear tests conducted as per ASTM G 99 standard. The dry sliding application of *Caesalpinia Decapetala* Shell Powder polymer matrix composite was performed utilizing a pin on disc tester system and also characterized according to ASTM standards (Kumar and Anand, 2019) illustrated in figure 3.18. The system disc is composed of steel with insignificant surface roughness and hardness of 76 HRC and the disc was spun while the assembled sample was maintained steady. The wear data were plotted against different load situations to study the impact of the applied normal load all plots are mentioned in Appendix A. With the use of a sample holder and screws, the sample is held constant with screw fixtures perpendicular to the turntable, and with a lever, fixture loads were practiced. Various parameters, comprising rotation time, with a constant speed and sliding

distance, were set manually before each test. The weight of the sample was determined with an electronic balance before and after the dry sliding control with a precision of ± 0.01 mg at the collapse of the behavior test. Wear tests were conducted three times on each created Caesalpinia Decapetala shell powder epoxy composite, reinforced with Caesalpinia decapetala shell powder particles in various weight percent, and the average CoF value was reported in this study.

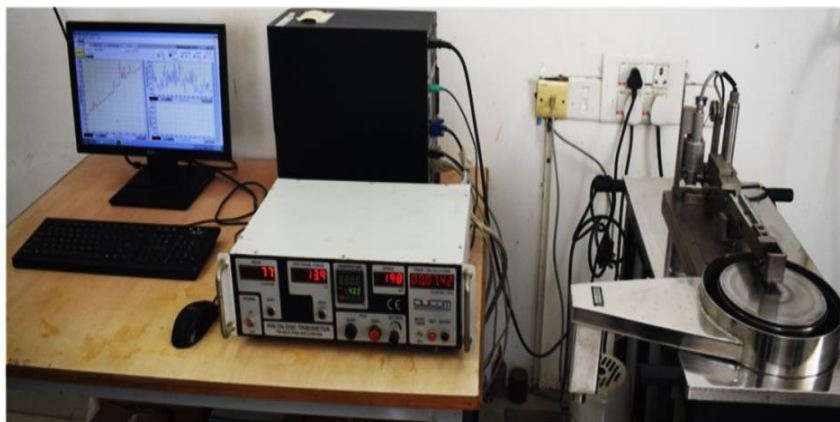


Figure 3. 18 Sliding wear experimental setup

3.8 Characterizations

Scanning electron microscopes (SEMs) are scientific instruments that use a strong electron beam to investigate materials at a microscopic level. These were developed in reaction to the limits of optical microscopy, which are constrained by the laws of physics. Scanning electron microscope uses a wide beam of electrons to view an object in a sequential scan pattern. SEM is one of the best and most widely used techniques for the chemical and physical characterization of Caesalpinia decapital particulate polymer matrix composite. An electron in the beam interacts with the sample to generate signals that carry information on the sample's geometry, shape, interfacial properties, crystallography, as well as other properties of a material. Specimens can be viewed in wet conditions or at a wide variety of temperatures, from cryogenic to any high temperature, as well as in high and low vacuum (in environmental SEM). The most frequent detection method uses secondary electrons generated by electron beam atoms. ESM has a broad depth of field, which allows a large portion of the sample to be in focus at once, resulting in a high-resolution image of the three-dimensional sample. Due to its combination of high magnification (10X–500KX), great depth of field (30mm), grating resolution of 1-2 nm, and crystallographic information, the ESM is one of the most extensively used instruments in universities, national labs, research, and industry.

Scanning the sample and detecting secondary electrons produces a picture. Examining the specimen and detecting backscattered electrons produces a picture. By taking samples and identifying secondary electrons, the surface tilt can be seen. The three most common modes of operation in SEM analysis are backscattered electron imaging (BSE), secondary electron imaging (SEI), and energy dispersive spectroscopy (EDS). In this study, EDS were used to characterize the *Caesalpinia decapital* particulate samples and to identify the element from the composite.

3.9 Optimization of Parameters

In order to optimize the process parameters of multi response experiment Taguchi gray relation analysis and Artificial Neural Network method were followed. Experimental design is verily required to know the total degree of freedom for proper selection of suitable orthogonal array. The degree of freedom is the number of comparisons between process parameters that need to be made to determine which level is better and specifically how much better it is (Wennberg, 2013).

F exhibitive was used. For tribological, water absorption and void study “Smaller is better ” quality exhibitive was used.

The degree of complexity of the proposed problem increases with the restricted number of data. However, GRA advocated by Prof. Deng is considered to be the right choice to address this kind of issue. GRA employs a unique approach to minimize the variance in the data called normalizing which transforms all the available data from 0 to 1. The existing multi objective problem can be transformed in to a single objective without affecting the weightage of an individual objective. The uncontrolled variables called noise factors in the experiments are the key reason to induce the inconsistency in the wear rate and coefficient of friction. The effect of noise factors is being minimized with the help of signal to noise ratio approach. The nature of response, whether higher or lesser is a prime factor to arrive a reasonable S/N ratio. The present study advocates to minimize the wear rate and coefficient of friction of the developed polymer composite, hence the lesser the better concept is followed to arrive S/N ratio as per the following equation.

$$\frac{S}{N} \text{ ratio} = -10 \log \frac{1}{n} \sum_{i=1}^n Y_i^2 \dots\dots\dots 3.15$$

The variance in S/N ratio is being normalized with the help of following equation,

$$Z_{ij} = \frac{\max(Y_{ij}, i=1, 2, \dots, n) - Y_{ij}}{\max(Y_{ij}, i=1, 2, \dots, n) - \min(Y_{ij}, i=1, 2, \dots, n)} \dots\dots\dots 3.16$$

The relevant grey relational coefficient is arrived by using the following relation with the involvement of corresponding quality loss ($\Delta_{\min}, \Delta_{\max}$). Table 4.4 shows and comprises the calculated grey relational coefficient value, S/N ratio, and normalized S/N ratio.

$$GC_{ij} = \frac{\Delta_{\min} + \lambda \Delta_{\max}}{\Delta_{ij} + \lambda \Delta_{\max}} \dots\dots\dots 3.17$$

CHAPTER FOUR

RESULTS AND DISCUSSIONS

4.1. Introduction

The prepared composite samples were examined according to ASTM standards, as described in the previous chapter three. The test findings reflect the qualities of the *Caesalpinia decapetala* particulate reinforced epoxy matrix composites that have been prepared. The reinforcements are mill (grind) to 100 – 105 micrometers in powder form in all composites. Three wt.% compositions and three different mixing parameters of composite samples were used. Based on these analyses, the result discusses the output result of dry sliding wear test, compression test, hardness test, impact test, water absorption, and physical characterization of the composite. The results of each composite were examined and discussed below. For all mechanical as well as tribological tests, the graph displayed on testing machines during the experiments is shown in the appendix.

4.2 Density measurement and void content

4.2.1. Density

Table 4.1 Density and void content estimations

Composition	Theoretical Density (Kg/m ³)	Experimental Density (Kg/m ³)	Void Content (%)
E	1192.00	1192.00	0.00
ECD-10	1254.60	1235.45	1.53
ECD-20	1317.20	1295.60	1.64
ECD-30	1379.80	1350.40	2.13

Density of the composites are evaluated by measuring the experimental density using Archimedes principle while theoretical density are calculated by using rule of mixtures. The density and void estimations are presented in 4.2. Density of composites increase with increase in content of *Caesalpinia Decapetala*. Density of *Caesalpinia Decapetala* and epoxy are noted to be 1818 and

1192 kg/m³, respectively. Void content estimations are also presented in Table 4.2 wherein the void content increases with increase in filler content owing to small air entrapment in the composites. Void content estimations noted are very small and therefore can be neglected. Lower void content estimations also reveal the higher quality of composites achieved.

4.2.2 Void content

The void content can be measured according to the Archimedes principle of the weight of material in the air being equal to the weight of material in the liquid. Various fiber volume fractions were used to reinforce composite samples, and voids were discovered in the samples. Composites with high porosity (or void content) have a higher risk of failure due to stress concentration and lower strength. To calculate the porosity of samples, the density of the specimens from the measured value was compared to the density calculated theoretically. The compressive strength of the samples is diminished because of the high porosity in the samples. Equations 3.8 are used to calculate porosity. The porosity of the CD/epoxy composite samples was also measured using volume and density measurements, as shown in Figure. 4.1. The porosities of the 2% by volume fiber samples were below 20% in all cases. When the fiber volume fraction was increased to 30%, the porosity rates increased significantly to around 1.6% to 2.13%. Numerous pores and cracks were also observed on the surface of composite samples, indicating a porous sample. These defects are expected to be the cause of the decrease in bond strength at a higher fiber volume fraction

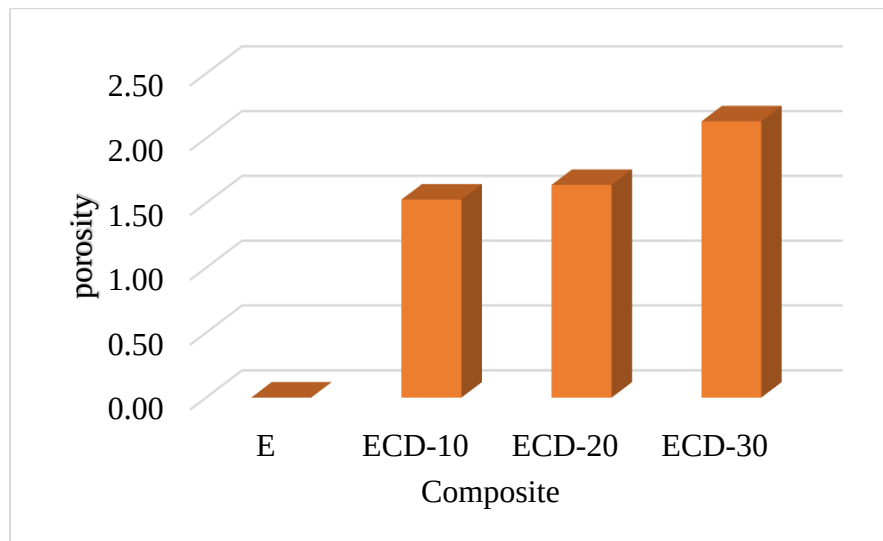


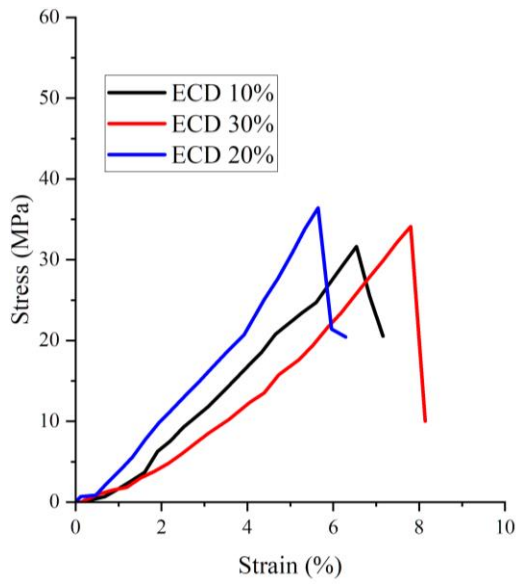
Figure 4. 1 Results of void content

4.3 Mechanical testing

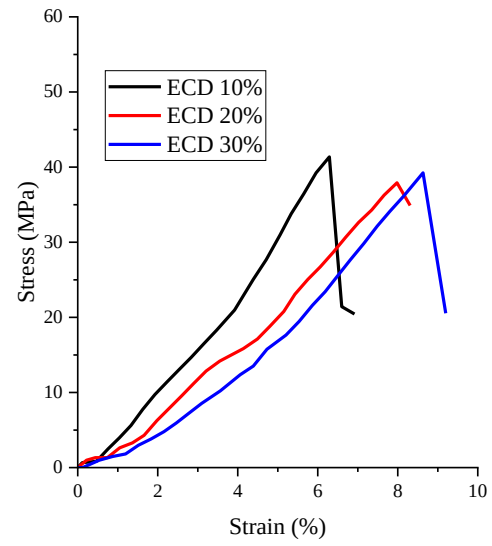
Mechanical testing is a method of determining a material's mechanical properties. It can be used to assess a material regardless of its geometry, as well as under specific geometric constraints. When analyzing a material, a range of mechanical test methods can be utilized, some of which can indicate the acceptability of a composite material. In addition to the tribological test, conduct the mechanical test for this thesis to know the same mechanical properties of this material. So these mechanical tests include compression test, impact test, Vickers hardness test, and characterization of the result after it conduct the test.

4.3.1. Compression test

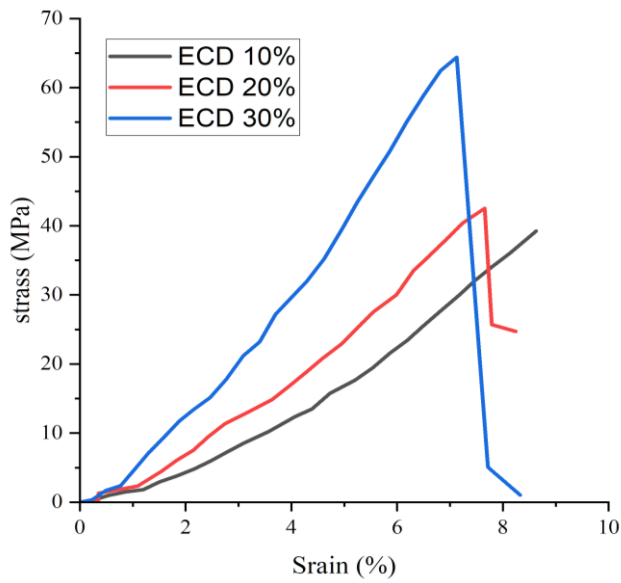
Compressive strength one of the most essential and widely evaluated qualities of materials used in many applications is their capacity to resist fracture under compressive pressure. This test was correlated with tensile strength. The compressive strength of a material is defined as the value of the uniaxial compressive stress attained when the material fails (Singha & Thakur, 2008). Compressive strength is, normally assessed by an experimental compression test. Typical compressive stress–strain curves of the ECD-10, ECD-20, and ECD-30 mixed specimens are presented in Figure 4.2. Compressive mechanical properties, including 0.2% compressive yield strength (0.2% CYS), ultimate compressive strength (UCS), and ultimate elongation of the CES epoxy based composites, were determined. The mechanically milled composite, ECD-30, showed a great enhancement, not only in strength but also in elongation, as compared to the mechanically milled composite, ECD-10 and ECD-20. This suggests that, although the plasticity is not reduced, the compressive strength can be retained for long of periods. The simultaneous increase in strength of the ECD-30 is also outstanding when compared to the mixed nanocomposite (ECD-10 and ECD-20).



a)



b)



c)

Figure 4. 2 Representative compressive stress strain profiles of composites at (a) with 10 minute mixed (b) With 30 minute mixed and (c) with 60 minutes mixed

Though the strain rate sensitivity is mainly credited with the matrix, percentage volume of reinforcements also significantly affect the failure mechanism. Stress strain plots for *Caesalpinia decapitala* particles show similar trends compared to neat epoxy sample. Neat epoxy profile consists of a linear elastic region tailed by a strain-relaxing region depicted by descent in stress values. Further compression of neat epoxy sample leads to rise in stress value thereafter till final rupture. On the other hand, *Caesalpinia decapitala* shell reinforced composites profile show a linear elastic region followed by a broad plateau region. Further compression loading leads to slight strain softening and decrease in stress; thereafter stress increases with increase in strain owing to the densification attained by the *Caesalpinia decapitala* shell composites. Higher plateau region observed with *Caesalpinia decapitala* shell composites with 30 vol.% of *Caesalpinia decapitala* shell at 60 minute mix with magnetic stirrer is attributed to higher resistance offered by *Caesalpinia decapitala* shell particles as illustrated in Figure 4.2C. Additionally, it is observed from Figure 4.6 that stress increase is considerably faster in case of neat epoxy sample whereas for *s Caesalpinia decapitala* hell composites, it depends on the volume content of *Caesalpinia decapitala* shell particles and mixing technique. Since stress–strain response is a strain rate dependent occurrence, therefore the compression rate is maintained constant in this study as per ASTM standard recommendations. Irrespective of the strain rates, all the *Caesalpinia decapitala* shell composites depict higher stress values compared with other compositions and neat epoxy specimens. Due to homogeneity, the matrix with reinforcement and percentage composition of filler material investigates maximum mechanical properties. The composite specimen, which have the maximum compression strength and compressive modules at ECD-30 than ECD-20 and ECD-10 also with neat epoxy as illustrated in figure 4.3.

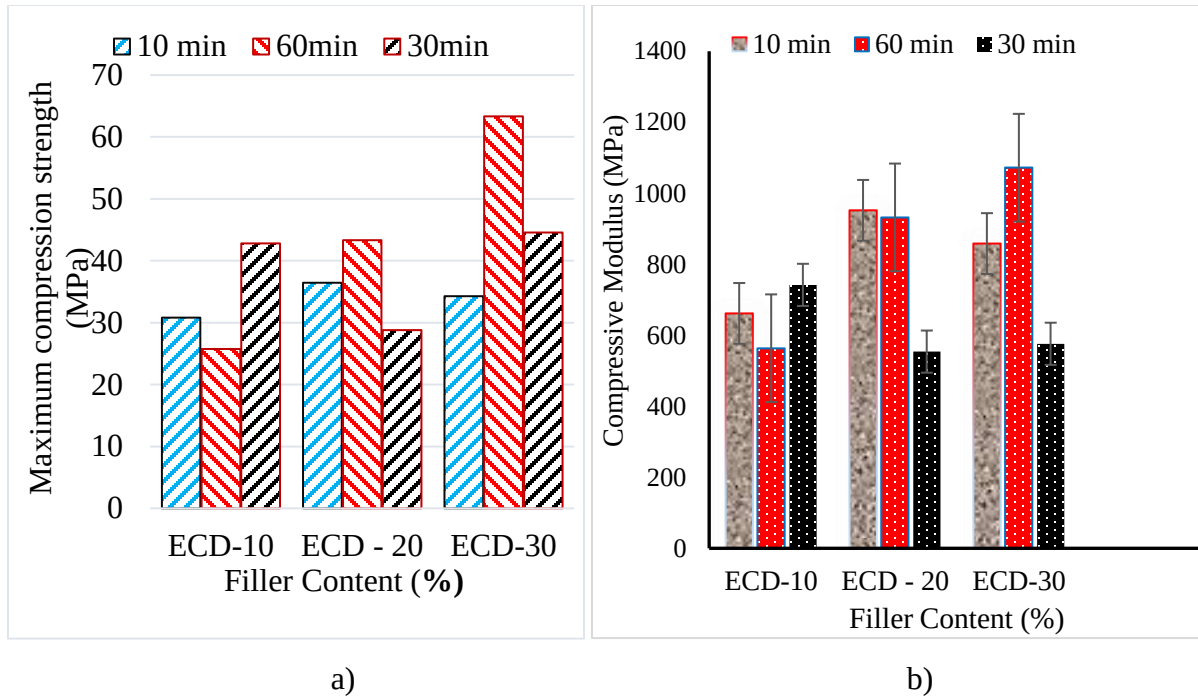


Figure 4.3 Experimental results of compression test a) Maximum compression strength and b) Maximum compression modulus

Modulus of the *Caesalpinia decapitata* shell particles reinforced epoxy composites samples was measured from the stress-strain curve by determining slope of the linear region (Figure 4.3 (B)). Compressive modulus of *Caesalpinia decapitata* shell particles reinforced epoxy composites show increasing trends in comparison with neat epoxy samples except at EESP-10 Figure 4.3b. Reinforcement of *Caesalpinia decapitata* shell particles in epoxy resin is known as toughening method. The toughening effect is instigated by the blocks generated from the reaction between the reactive end groups of the *Caesalpinia decapitata* shell particles and the active group of the epoxy resin. However, incorporation of *Caesalpinia decapitata* shell particles into the polymer matrix often results in deterioration of mechanical performance that is associated with the insufficient interfacial interactions between matrix and filler after a certain percentage of reinforcement. In the present work, 30 vol.% of *Caesalpinia decapitata* shell particles was found to be the highest enhancing the modulus of composites than 10 vol.% and 20 vol.% *Caesalpinia decapitata* shell particles. Furthermore, enhancement of these interactions can be realized by the addition of proper compatibilizer, modification or functionalization of *Caesalpinia decapitata* shell particles.

4.3.2. Tensile test

Stress Strain Curves

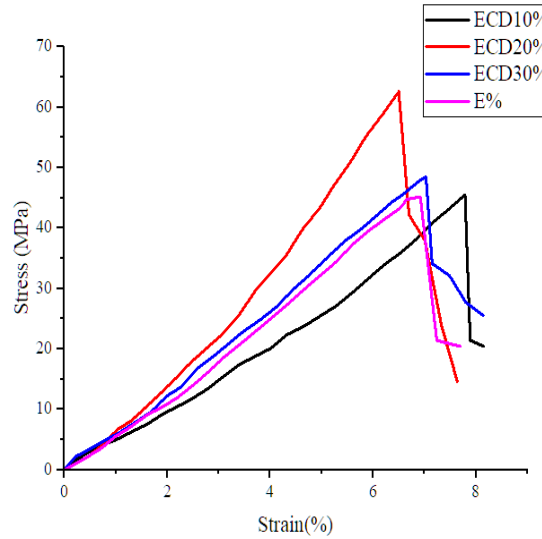


Figure 4. 4 Representative tensile stress strain curves of composites

Representative tensile stress–strain curves for neat epoxy and *Caesalpinia decapitala* particles filled epoxy composites are presented in Figure 4.4. Stress–strain profiles of all the specimens demonstrate similar curves up until maximum stress consisting linear elastic area followed by abrupt brittle failure. Similar trends are observed in earlier studies on epoxy based composites (Mohan & Kanny, 2018). Reinforcing *Caesalpinia decapitala* shell particles in epoxy matrix enhances the stress notably as evident from Figure 4.3.

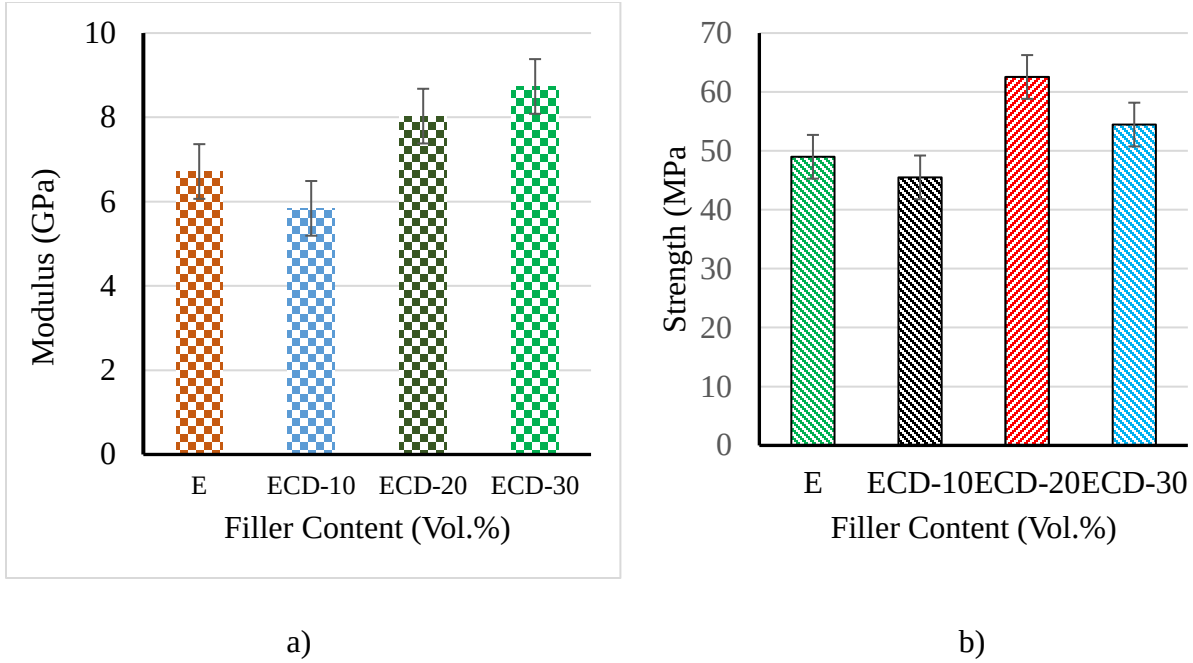
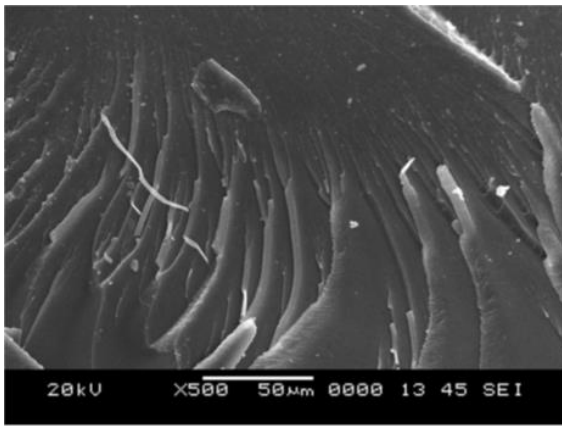


Figure 4. 5 Experimentally measured a) modulus of the composites and b) strength of the composites.

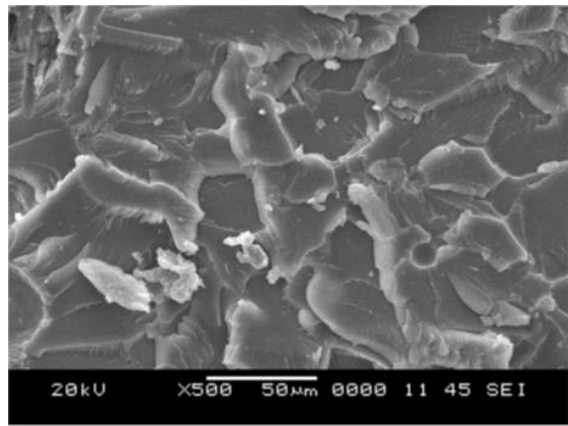
Experimentally measured tensile modulus is presented in Figure 4.5a. Tensile modulus of ECD-10, ECD-20, and ECD-30 increased as compared with E. The increase in modulus is mainly attributed to reinforcing hard *Caesalpinia decapitala* shell particles into matrix. Tensile modulus of all the composites increases with increase in filler content and are in the range of 0-3% higher as compared with E. Relatively hard particles dispersed in epoxy matrix assist in the load transfer and contribute in enhancing the modulus of the composites. Reinforcement of rigid fillers increases the stiffness of epoxy-based composites (Mohan & Kanny, 2018). Experimentally measured strength of the specimens is presented in Figure 4.5b. All the *Caesalpinia decapitala* shell/epoxy composites reveal higher strength as compared to neat epoxy specimens. Increase in strength is in the range of 25-38% and maximum increase is observed with ECD -20. Reinforcing *Caesalpinia decapitala* shell in load bearing matrix increases the filler-matrix bonding and thereby increases the strength.

Fracture surfaces of post tensile tested specimens as observed in scanning electron microscopy are presented in Figure 4.6. Fracture features in neat epoxy sample can be seen in a wherein striation marks and brittle failure marks can be clearly observed (Figure 4.6 (a)). Micrographs of ECD-10, ECD-20, and ECD-30 are presented in Figure 4.6 (b), Figure 4.6 (c), and Figure 4.6 (D)

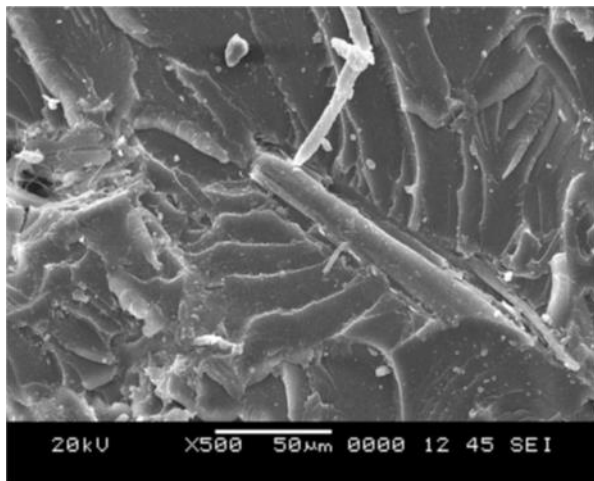
respectively. It can be clearly observed that striation and deformation marks are less in number for *Caesalpinia decapitala* shell/epoxy composites compared with neat epoxy. Good bonding between the *Caesalpinia decapitala* shell and epoxy matrix can also be seen. In line with the trends observed for modulus and strength higher filler loadings reveal low deformation marks and contribute significantly in enhancing the properties of composites.



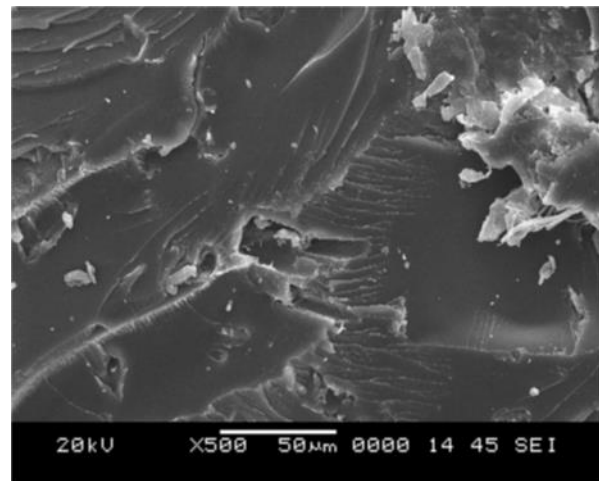
A)



B)



C)



D)

Figure 4. 6 Tensile tested scanning electron micrographs of (a). E, (b). ECD-10 (c). ECD-20 (d) and ECD-30

4.3.3. Charpy Impact Test

The Charpy impact test is also known as the Charpy V- notch test. A standardized high strain-stress test determines the amount of energy absorbed by a material during fracture. Figure 4.7 shows the experimental results of the impact strength of ECD composite specimens. The line demonstrates a consistent increase in impact strength as the filler content increases, indicating 7.5J with 30 wt. % filler. However, as the filler content is increased, the impact strength decreases when the sample is mixed manually. This could be due to the weak interfacial interaction between the filler and the matrix material when the filler content is homogeneous (Raju et al., 2012) discovered similar behavior in composite specimens, namely that impact strength diminishes as filler loading increases. The CDSPC board absorbs the potential energy in the drop weight during an impact test. The energy absorbed in the drop weight impact test increases with the increase in the weight percentage of filler loading, which is *Caesalpinia decapetala* shell particles, as shown in Figure 4.7. It almost exhibits a linear incremental response up to 30% filler loading, indicating that the sample S8 absorbed the maximum energy of 7.5 J because of the homogeneity of the solution (with 30 wt. percent filler loading and 1 hour mixing time by magnetic stirrer). This could be because reinforcing particles absorb more potential energy as their quantity in the matrix material increases. Drop weight impact strength diminishes as filler loading increases, and also, if the composition was not properly mixed, the final output result of the impact test was decreased or low energy absorbed (Sathish et al., 2021).

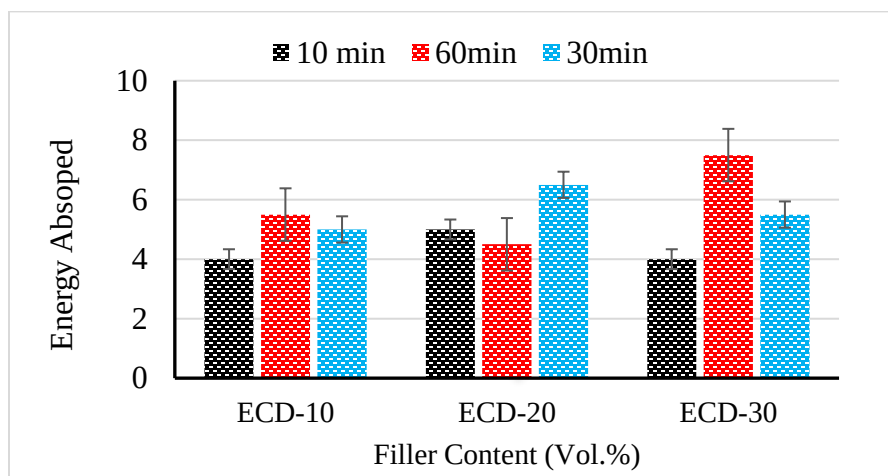


Figure 4. 7 Impact test result

4.3.4. Hardness test results

Figure 4.8 shows that the C. decapitala particulate reinforced epoxy composite has the highest Vickers hardness number in 10 kgf, with a tester machine hardness of 150 HV. The peak hardness value was obtained in unidirectional 150 HV using 10kgf. From the synthesized composite material, the 10% particulate fiber reinforced epoxy composite shows the least value and obtained high hardness at 30% during Vickers hardness test because of the similar parameters of compression and impact test result. Because of reinforcement and mixing properties, raise the value of hardness from (124.3 – 150) VKR for ECD-10 to ECD-30 composite.

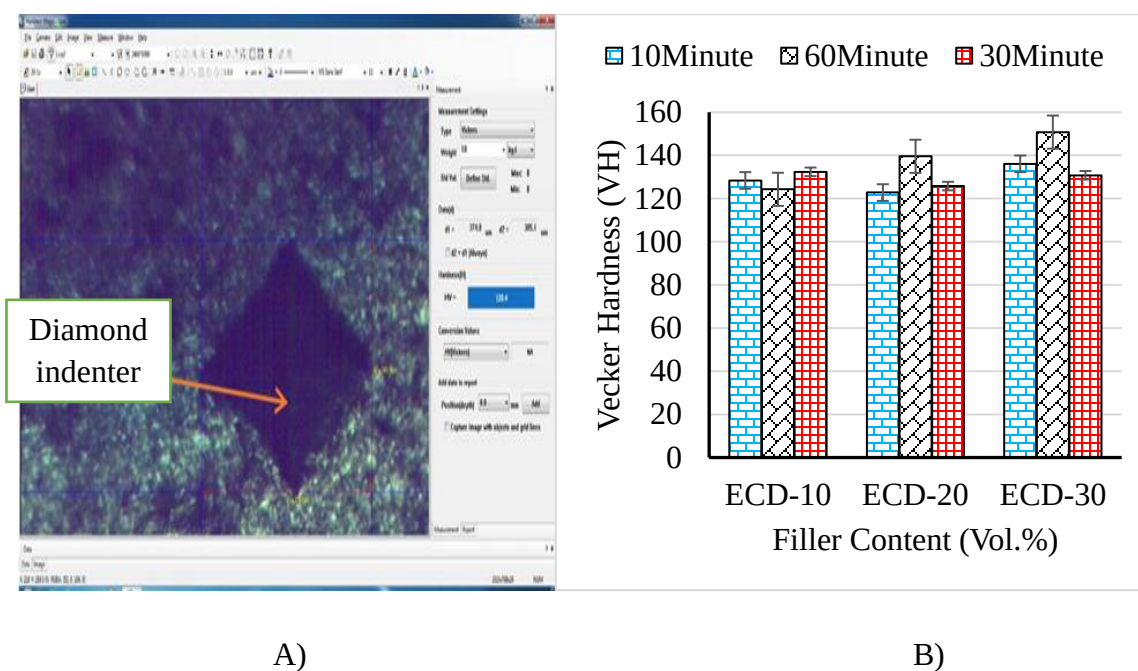


Figure 4. 8 Vickers hardness A) indentation area and B) result of hardness value

4.3.5. Discussion for mechanical test

Epoxy matrix composites were fabricated with 10, 20, and 30 weight percentage *Caesalpinia decapetala*, and the mechanical characteristics were studied as per ASTM standard. The influence of mixing time, mixing method, and filler content are analyzed on compression test by universal testing machine, Impact test by Charpy machine, and hardness test by Vickers hardness machine. The effects of each variable on the mechanical properties was analyzed and the results show that:

- With increasing *Caesalpinia decapetala* contents at 30% and mixing uniformly, the compression strength, high energy absorbed during impact testing, and maximum hardness are reported because of mixing homogeneity, irregular shape of reinforcement material, and maximum *Caesalpinia decapetala* contents.

The results of this study can be used to select material composition as per the mechanical requirement of the component. The selection of appropriate material can increase the Mechanical property well as reduce the density of the material

4.3.6. Water absorption test

The results obtained from the water absorption test are shown in Figure 4.9. The weight of each sample soaked in the water was weighted by the interval of 24 hrs. (24, 48, 72, 96, 120 144, 168,192, 216, and 240 hrs.). The result recorded was the average of those times. In the case of CD reinforced epoxy matrix composite, the water absorption, which is a significant attribute to consider when choosing a building material, is only 1.51–8.82 percent for 15 days and 0.656-3.28 percent for 24 hours (Sandeep Kumar et al., 2021). This could be attributed to a decreased space in the composite because of stronger particle to resin bonding. It has greater outdoor applications because of its characteristics. As illustrated in Figure 4.9, water absorption increases as the percentage number of *Caesalpinia decapetala* particles increases. For in this test, the specimen was immersed in distilled water for 240 hours. ECD-30 at 10 minute mixed is the highest water absorption sample because of the maximum percentage value of filler material and is not mixed uniformly because of minimum mixing time. For this reason, in the composite materials, there is segregation and they are not homogeneously mixed. However, after 10 days the composite was at equilibrium there is no value change during measuring the sample.

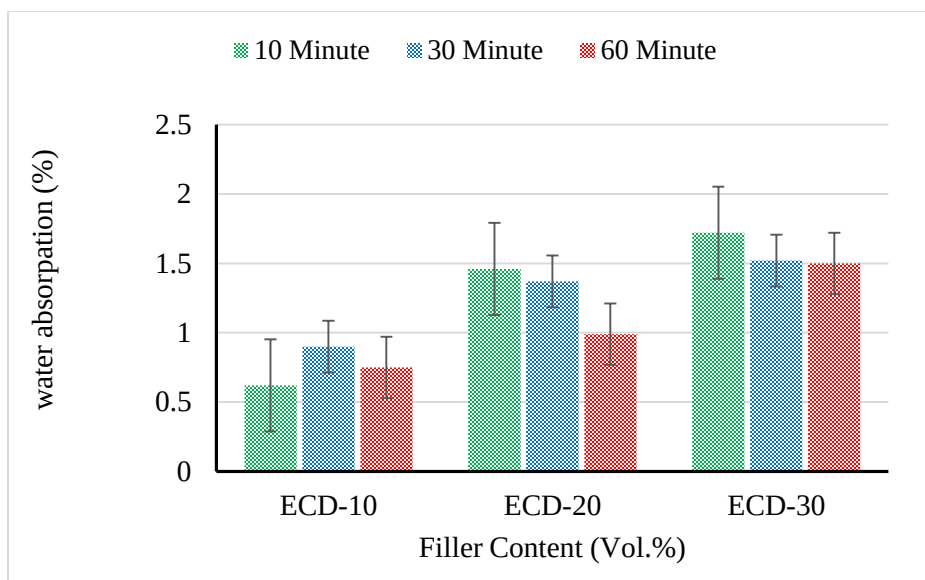


Figure 4. 9 Water absorption result

4.4 Scanning Electron Micrographs

Caesalpinia decapitala shells are very hard, brittle in apparatus and brown in color elliptical in shape during dry time. Due to these properties, it has a better mechanical property and high resistance of tribological characteristics in the fabrication of composite. This material has a low density-to-weight ratio, is low cost, and is easily available anywhere. Additionally, it has high wear resistance, high compression, and impact strength, and it has high hardening properties when it mixes with epoxy resin in composite material. Furthermore, Caesalpinia decapitala seed shells are easily soluble with a matrix to prepare a composite sample.

EDS spectra of Caesalpinia Decapetala particles are presented in figure 4.10. Energy peaks equivalent to the existing different elements of Caesalpinia Decapetala are depicted in the tabular form (Table 4.2). The spectra reveals major constituents as oxygen, bromine, aluminium, calcium and manganese. Aluminium is one of the widely used non ferrous metal in wear resistant applications. Presence of aluminium in the CD shell augurs for the enhanced wear resistance.

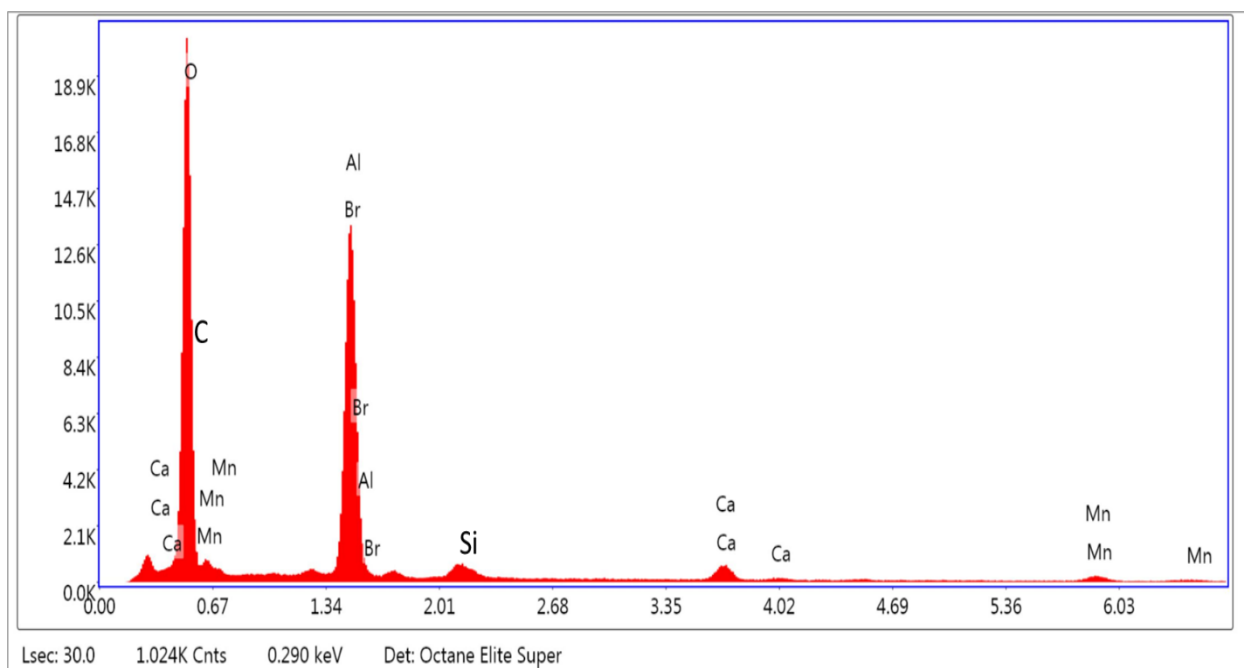


Figure 4. 100 Energy dispersive analysis X-rays of Caesalpinia Decapetala particles.

Table 4.2 Chemical composition of Caesalpinia Decapetala.

Element	Weight %	Atomic %
O K	28.23	40.23
Br K	16.23	7.21
Al K	12.69	20.14
C K	14.28	14.61
Si K	10.27	7.12
Ca K	7.57	5.43
Mn K	10.73	5.24

4.4.2 Characterisation of the crystal scale

Based on the article published for this study, EDS were used to characterize the Caesalpinia decapital particulate samples. A representative portion of Caesalpinia decapital particulate was sprinkled onto double-sided carbon tape mounted on a SEM stub. This grain mount enable to determine the particle morphology, external surface structure and external elemental distribution of individual Caesalpinia decapital particulate. The majority of the particles ranged in size from approximately 1 μ m to 10 μ m and consisted of solid spheres Figure.4.16. Agglomerated particles and irregularly shaped amorphous particles shown in figure 6.16 may have been due to inter-particle contact or rapid curing.

The SEM microstructure of Figure 4.11A shows that the CD particles are dispersed uniformly in the epoxy resin when compared to Figure 4.11B of filler materials. Figure 4. 11 shows the morphology of fractured structure of epoxy based /CD particulate composites with minimum defects at low volume fractions of CD particles, while the morphologies of composite surface show that non uniformly distribution and dispersed on the minimum mixing time sample. Due to this the strength of the composite was less.

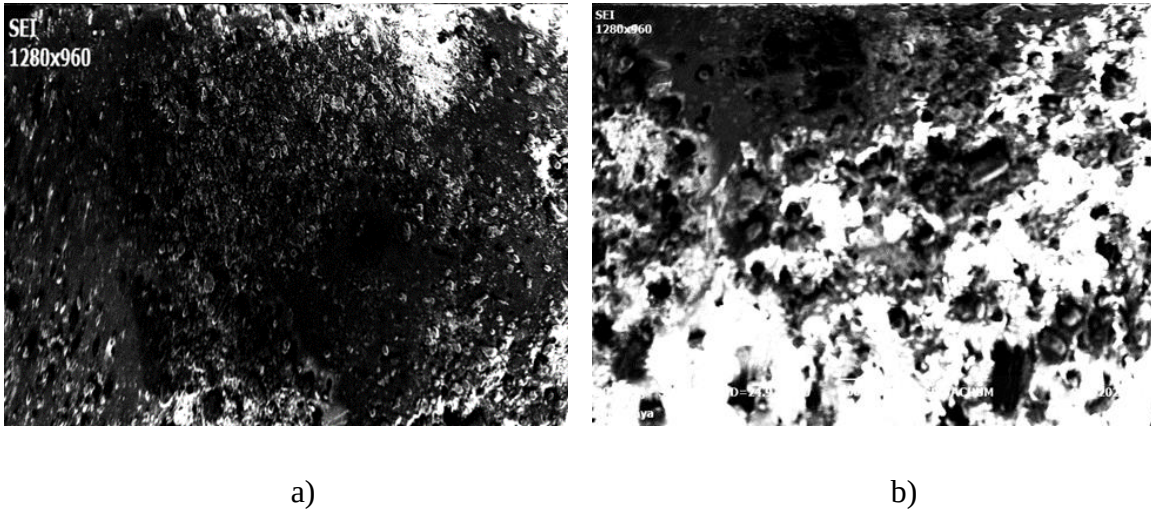


Figure 4. 11 SEM Image of CD particles epoxy based composite a) 60 minutes mixed sample b) 10 minutes mixed sample

4.5 Dry Sliding Wear Test

Tribological tests were performed on pin on disc apparatus. Wear test measures the changes in conditions caused by friction, and the result is obtained from deformation, scratches, and indentations on the interacting surfaces. Wear arises when there is relative motion between two solid bodies under the impact of the load, which can slide or roll. The kind of wear depends on the respective material qualities in addition to movement or the impact of the tribological load.

4.5.2 Experimental Wear rate

Wear of *Caesalpinia Decapetala* reinforced epoxy composites are depicted in Figure 4.12. Wear rates of specimens tested at constant sliding velocity (5 m/s) is seen to decrease with increasing filler content and applied load. Epoxy is known to be brittle in nature and reveal very high wear

ate. Reinforcing Caesalpinia Decapetala particles consisting of aluminium as one the major constituents which reduces the wear rate significantly. As the Caesalpinia Decapetala content in the composite is increased from 0-30 vol.%, the ability of composites to resist wear further enhances and thereby the decrease in wear rate can be observed. Additionally, decrease in wear rates with increase in applied normal load from 30 to 50 N is observed. Although, the volume loss in epoxy based composites depict higher wear rates at higher applied loads, caesalpinia decapetala effectively resists wear of composites by absorbing the applied load and thereby lowers the wear rates significantly. Furthermore, reduction in brittle epoxy content as compared with caesalpinia decapetala content further decreases the wear rate. Consequently, ECD-30 at 50 N load depicts lower wear as compared with other compositions and test conditions which in turn upholds the results of statistical and ANN prediction . Wear rate decreased in the range of 10-35% as the applied load increases from 30 to 50 N whereas the wear rate reduces in the range of 13-30% as the caesalpinia decapetala content is increased from 10-30 vol.%.

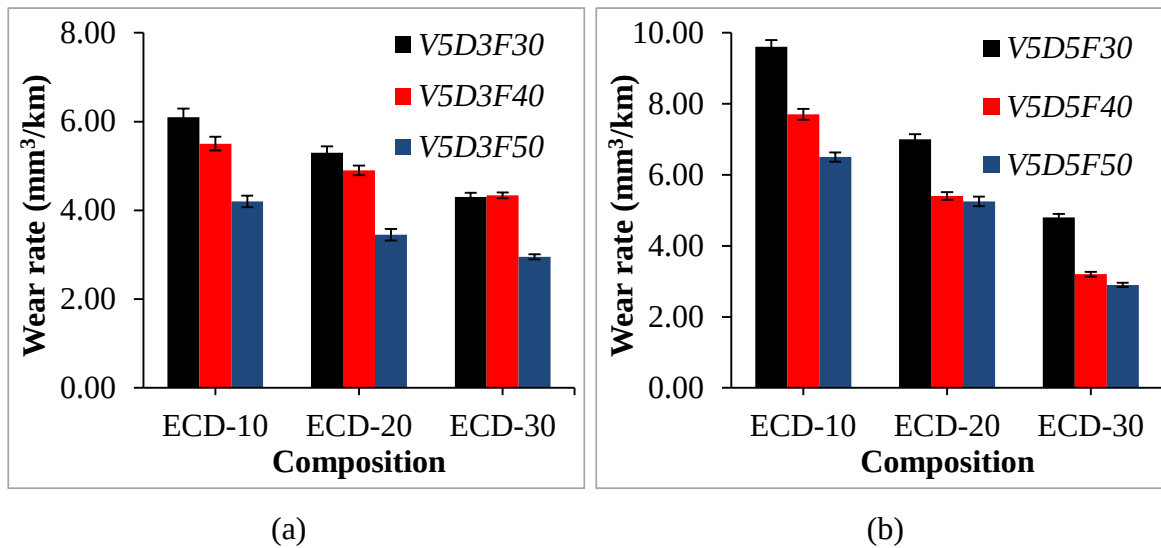


Figure 4. 11 Wear rates of composites at sliding distance of (a) 3 and (b) 5 km.

4.5.3 Experimental Specific wear rate

Specific wear rate revealing the load bearing capability of the specimens is presented in Figure 4.13. In line with the wear rate trends, specific wear rate of all the composites decrease with increasing applied load and caesalpinia decapetala content. ECD-30 at 50 N load depicts the lowest specific wear rate of 0.059 mm³/N-km inferring higher wear resistance of composites.

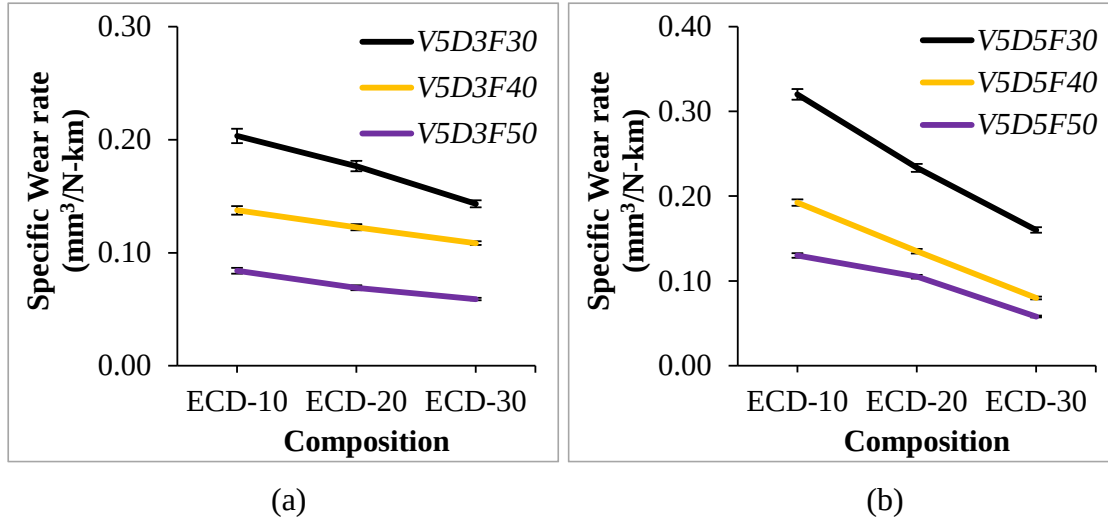


Figure 4. 12 Specific wear rates of composites at sliding distance of (a) 3 and (b) 5 km.

4.5.4 Experimental Coefficient of friction

Coefficient of friction of specimens subjected to different test conditions are presented in Figure 4.14. Irrespective of the sliding distance, coefficient of friction decrease with increase in filler content and applied normal load. Increase in *Caesalpinia Decapetala* content results in lower fluctuations at the interface of pin (specimen) and rotary disc owing to the higher resistance offered to wear by alumina available in *caesalpinia decapetala* particles. As a result, surface roughness of specimen is very minimal and thereby low coefficient of friction is reported at higher filler loadings. Furthermore, higher applied loading also reveal low coefficient of friction due to better compaction of asperities on the disc surface.

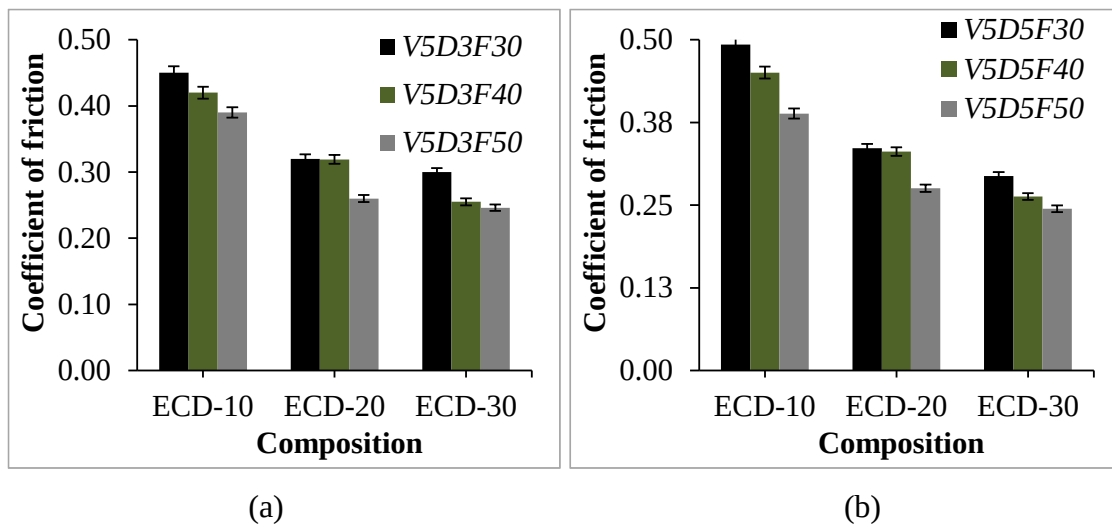


Figure 4. 13 Coefficient of friction of composites at sliding distance of (a) 3 and (b) 5 km.

4.5.5 Post wear scanning electron micrographs

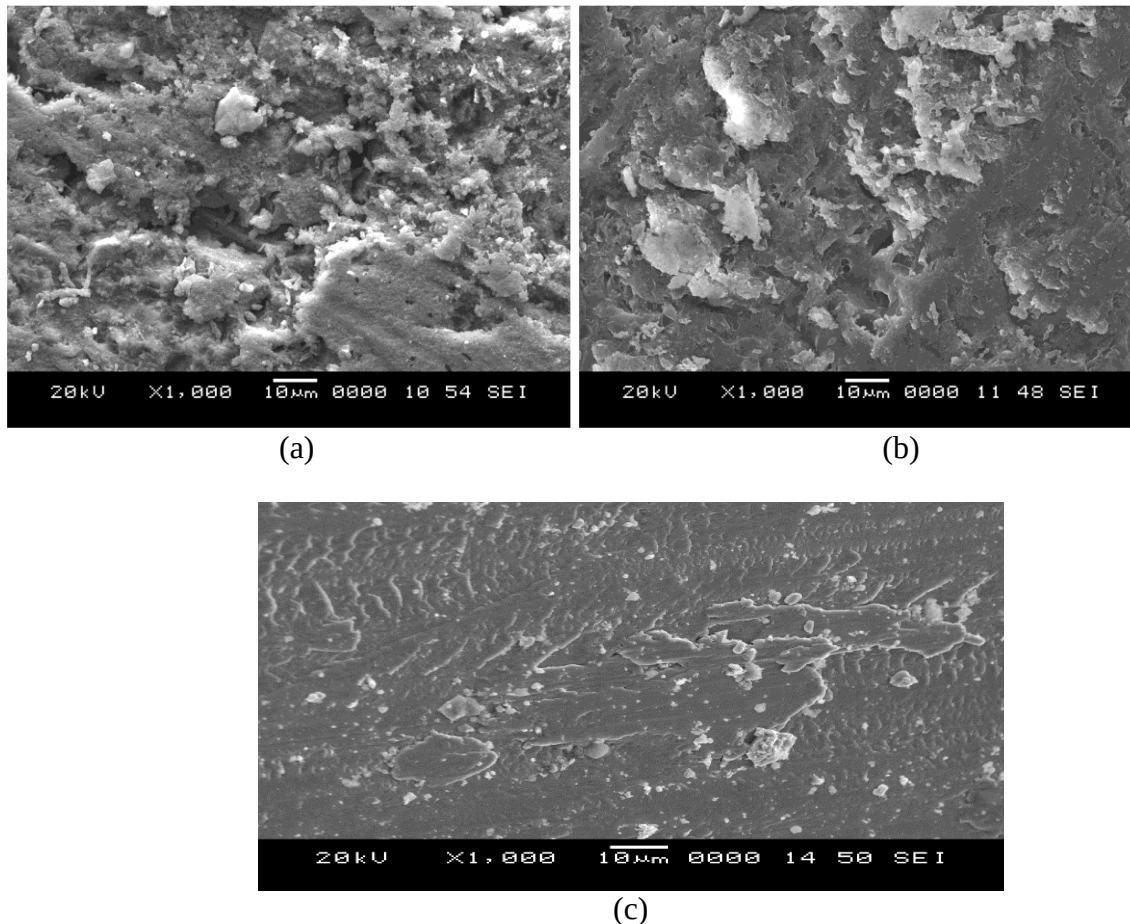


Figure 4. 14 Scanning electron micrographs of (a). ECD-10, (b). ECD-20 and (c). ECD-30 at 50 N applied load.

Post wear surfaces of specimens are shown in Figure 4.15. Micrographs of ECD-10, ECD-20 and ECD-30 are depicted in Figure 4.14 a, b and c, respectively. From the micrographs, it is evident that the wear tracks depict decreasing severity with increase in CD shell content. Lower content of caesalpinia decapetala and dominance of matrix at ECD-10 induces more wear while increasing the caesalpinia decapetala content from 10-30 vol.% enhances the wear resistance significantly owing to the ceramic elements seen in caesalpinia decapetala. Furthermore, micrographs for ECD-30 reveal relatively smoother surface compared to ECD-10 and ECD-20 indicating an effective film formation that assists in decreasing the wear rate by compacting the undulations and wear debris between disc and pin to minimize wear further. At 50 N load and 30 vol.% of caesalpinia decapetala content combined together effectively reduce the wear asperities and result in reduced wear even at elevated test conditions.

4.6 Statistical Analysis

4.6.2 Taguchi's design of experiment

Taguchi's robust design of experiments is a versatile tool to deal with minimum number of experiments whose variable are in more number. Since the considered parameters are in three number and objective are in two number, L9 orthogonal array has been implemented in this study so as to bring down the number of experiment as nine. Table 4.3 shows the observed values of both the objectives for the nine experimental run.

Table 4. 1 Experimental observation

S.No.	Normal load (N)	Sliding Distance (km)	Percentage of Composition	Wear rate (mm)	Coefficient of Friction (mm)
1	30	3	10	0.13	0.388
2	30	4	20	0.11	0.276
3	30	5	30	0.06	0.245
4	40	3	20	0.14	0.33
5	40	4	30	0.08	0.26
6	40	5	10	0.19	0.45
7	50	3	30	0.16	0.29
8	50	4	10	0.32	0.49

4.6.3 Grey Relational Analysis (GRA)

Table 4. 2 S/N ratio, normalized S/N ratio, and grey coefficients.

S/N Ratio		Normalized S/N ratio		Deviation sequence		Grey Relational Coefficient (GRC)	
Wear rate	CoF	Wear rate	CoF	(Δ_{WR})	(Δ_{CoF})	Wear rate	CoF
17.72	8.22	0.46	0.66	0.54	0.34	0.4817	0.5980
19.17	11.18	0.36	0.17	0.64	0.83	0.4395	0.3766
24.44	12.22	0.00	0.00	1.00	1.00	0.3334	0.3335
17.08	9.63	0.51	0.43	0.49	0.57	0.5032	0.4674
21.94	11.70	0.17	0.09	0.83	0.91	0.3765	0.3537
14.42	6.94	0.69	0.88	0.31	0.12	0.6164	0.8036
15.92	10.75	0.59	0.24	0.41	0.76	0.5471	0.3980
9.90	6.20	1.00	1.00	0.00	0.00	1.0004	1.0013
12.77	9.37	0.80	0.47	0.20	0.53	0.7173	0.4870

Evaluation of Grey Relational Grades (GRG)

The GRG is manipulated with the help of the following equation, and the Table 4.5 shows the GRG and their rankings.

$$G_i = \frac{1}{m} (GC_{ij}) \dots\dots\dots 4.1$$

Where, G_i is the GRG of an i^{th} experiment; m is the number of responses;

Table 4. 3 Grey relational grade and their ranking

S. No.	Grey Relational Grades		CGRG	Rank
	Wear Rate	CoF		
1	0.27	0.25	0.3716	9
2	0.32	0.25	0.4008	8
3	0.25	0.24	0.3492	4
4	0.71	0.27	0.6928	6
5	0.24	0.28	0.3647	1
6	0.25	0.25	0.3541	5
7	0.35	0.24	0.4171	3
8	0.30	0.26	0.3946	2
9	0.29	0.28	0.3968	7

4.6.4 Optimal combination of wear parameters

The analysis of GRG is carried out to minimize the wear rate and coefficient of friction for the developed novel polymer composite. The ascendancy of the quality characteristic is measured through the value of GRG. The experimental combination with larger value of GRG is being assigned as rank 1 and recognised as an optimal wear testing parameters, which yield the minimum wear rate and COF. In table 9, fifth experimental combination has got highest mean value whose parameter combinations are 40 N of normal load, 4 km of sliding distance and 30 % of composition of reinforcement and subsequently are considered as optimal wear parameters.

4.6.5 Analysis of variance (ANOVA)

ANOVA analysis is conducted based on the weightage of means scored by each parameter. The purpose of this statistical analysis is to expose the significant parameter to minimize the wear rate and COF. The ANOVA table shown in Table 4.6 & 4.7 affirms the degree of influence of the parameters over the response and is given by ranking. It is observed from ANOVA analysis of both the responses that R^2 value is more than 90% confirms the suitability of the model. Since P value of percentage of composition of CD is less than 0.005 among the three wear test parameter, then the percentage of composition of CD plays a significant role in minimizing the wear objectives. However, the normal load secured the secondary importance over the objectives. The effect of sliding distance declared to be insignificant when comparing to the rest of two parameters. The following regression equation arrived from the analysis clearly exhibits the relation between the parameters considered at three levels and each of the response.

Table 4. 4 Analysis of Variance for wear rate.

Source	DF	Adj SS	Adj MS	F-Value	P-Value
Normal load (N)	2	0.11809	0.059045	8.58	0.104
Sliding Distance (km)	2	0.01349	0.006746	0.98	0.505
Percentage of composition	2	0.18553	0.092763	13.48	0.003
Error	2	0.01377	0.006883		
Total	8	0.33087			

$R^2 = 94.84\%$; $R^2 (adj) = 83.36\%$

Table 4. 5 Analysis of Variance for coefficient of friction.

Source	DF	Adj SS	Adj MS	F-Value	P-Value
Normal load (N)	2	0.05589	0.027944	2.49	0.286
Sliding Distance (km)	2	0.01215	0.006073	0.54	0.649
Percentage of Composition	2	0.32730	0.163651	14.60	0.004
Error	2	0.02242	0.011209		
Total	8	0.41775			

$R^2 = 94.63\%$; $R^2 (adj) = 88.53\%$

$$\text{Wear rate} = 0.5573 - 0.1391x_1 - 0.0586x_2 + 0.1977x_3 - 0.0466y_1 + 0.0482y_2 - 0.0016y_3 + 0.1422z_1 - 0.0039z_2 - 0.1383z_3$$

$$\text{Coefficient of friction} = 0.5355 - 0.0994x_1 + 0.0061x_2 + 0.0933x_3 - 0.0477y_1 + 0.0417y_2 + 0.0059y_3 + 0.2655z_1 - 0.0918z_2 - 0.1737z_3$$

Where x_1 , x_2 , and x_3 are the normal load at three levels 30, 40 and 50 N respectively,

y_1 , y_2 , and y_3 are the sliding distance at three levels 3, 4 and 5 km respectively, and

z_1 , z_2 , and z_3 are the percentage of composition of CD shell powder at three levels 10, 20, and 30% respectively

4.6.6 Artificial Neural Network (ANN)

ANN is an algorithm supported technique to solve engineering problems with limited data, which is inspired by the system and functioning of human biological neural network. The technique involves input, hidden layer and an output. The input data are processed by the hidden layer with the help of propagation function which adds the values based on assigned weightage. The resulting value will be compared by the certain threshold value. The neuron sends an output on its outgoing weights of all connected neurons subjected to the activation, the activation is being initiated if the resulting value exceeds the threshold value. The assignment of weightage, activation function is governed by the algorithm. The algorithm employs different type of propagation function according to the nature of the problem. In this study, the wear parameters including percentage composition of the reinforcement are considered to be an input whereas wear rate and coefficient of friction are output in MATLAB R2018. Levenberg-Marquardt (LM) algorithm with feed – forward propagation function is employed for the testing and the gradient descent with momentum weight and bias learning function is employed for the training. ANN architecture shown in Figure 4.15 confirms the presence of single hidden layer with 10 number of neurons and is used to process the data. One third of the data are used for the testing and rest of the data are used for the training.

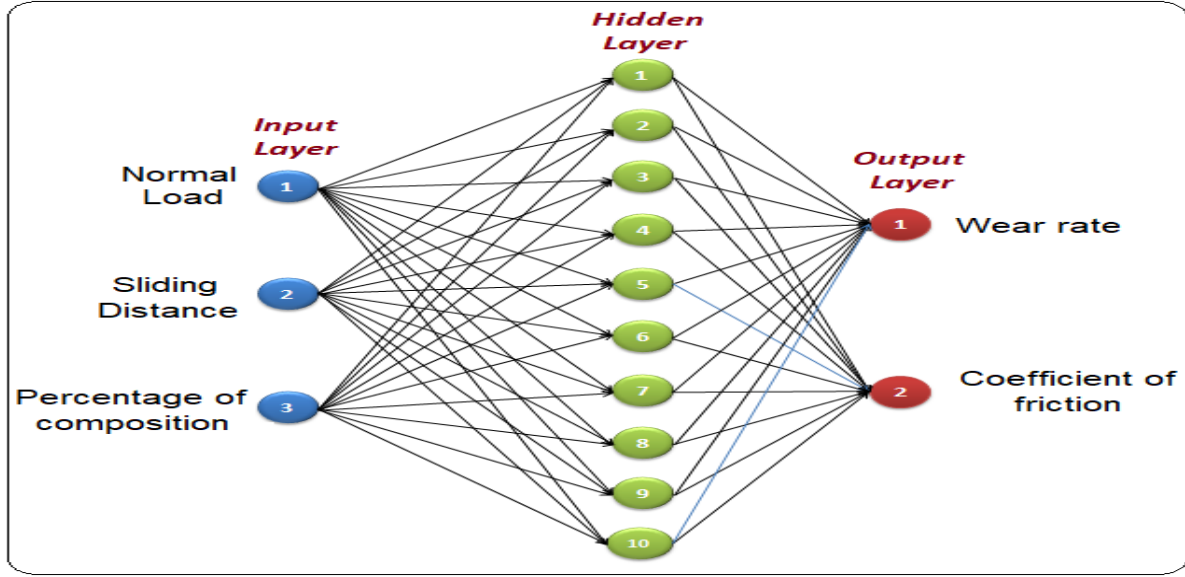


Figure 4. 15 ANN Architecture.

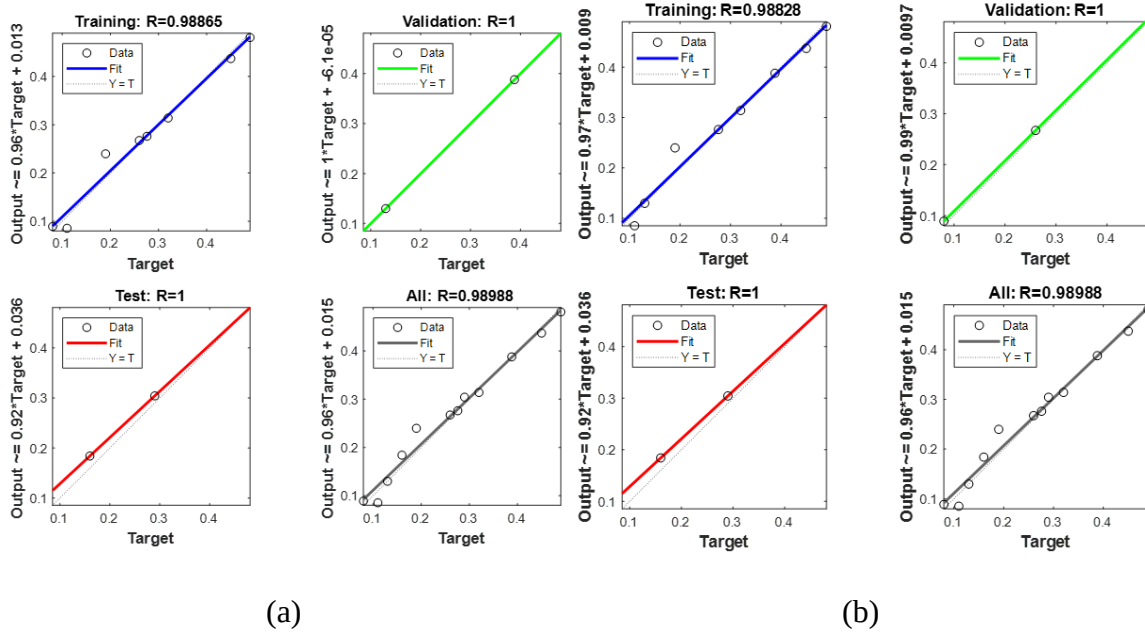


Figure 4. 16 Prediction performance of ANN model (a) Prediction indicator for wear rate (b) Prediction performance for CoF.

Figure 4.17 (a) depicts the prediction performance of wear rate response and (b) depicts prediction performance of CoF, the results expose degree of genuineness of the prediction. The results affirm that all the linear line are aligned exactly and with negligible outlier points in turn expresses the reliability of the proposed ANN model. The range of R^2 value also from 0.988 to 1 highlights the

negligible error of the model. The following relation shows the percentage of error prediction between experimental and ANN model and the error in prediction has been tabulated in Table 4.8.

$$\text{Percentage of error in prediction} = \frac{\text{Experimental results} - \text{ANN results}}{\text{Experimental results}} \times 100 \dots\dots\dots 4.2$$

Table 4. 6 Error prediction between experimental and ANN

Run No	Experimental		ANN model		percentage of error	
	wear rate	CoF	wear rate	CoF	wear rate	CoF
Training set						
1	0.13	0.388	0.12998	0.38980	0.0154	-0.4639
6	0.19	0.45	0.19000	0.45080	-0.0018	-0.1778
8	0.32	0.49	0.32005	0.48976	-0.0150	0.0490
4	0.14	0.33	0.13997	0.33040	0.0214	-0.1212
Testing set						
5	0.08	0.26	0.07989	0.25972	0.1375	0.1077
2	0.11	0.276	0.11005	0.27603	-0.0455	-0.0109
7	0.16	0.29	0.15998	0.29000	0.0125	0.0000
Validation set						
3	0.06	0.245	0.06000	0.24589	-0.0017	-0.3633
9	0.23	0.34	0.23000	0.33965	0.0009	0.1029

5 CONCLUSIONS AND RECOMMENDATIONS

5.1. Summary

This chapter presents the summary and conclusion derived from the conduct of the study, which is the mechanical response and tribological study of the *Caesalpinia decapetala* seed shell particulate fiber reinforced epoxy composites. The purpose of this research is to see if *Caesalpinia decapetala* seed shell particles can be used to make composite samples. The test findings suggest that using *Caesalpinia decapetala* seed shell particles and epoxy as an matrix, a composite sample can be made. The addition of particles improved the tribological and mechanical characteristics by a few percentage points before deteriorating further as the particle content in the samples increased.

5.2. Conclusions

Caesalpinia decapetala is a plant that has more than 100 species in the world and has not yet been studied in detail. The stinging *Caesalpinia decapetala* shell of Ethiopian origin has a particulate fiber that can yield composite materials with comparable tribological and mechanical properties. According to the research, there are no detailed studies on *Caesalpinia decapetala* in Ethiopia. This means that no studies are available on composite material using Ethiopian origins of stinging *Caesalpinia decapetala* shell particle fibers instead of being used for medicines and animal feed raw materials from its root and leaves. A researcher is only trying to analyze its properties related to medicine and animal production. In other ways, *Caesalpinia decapetala* plants grew abundantly in various countries including our country Ethiopia. Researchers are investigating the different properties of *Caesalpinia decapetala* plans concerning food and drug applications. However, according to an investigation, there is still no study on the composition of *Caesalpinia Decapetala* plans. Because of availability of *Caesalpinia decapetala* in our country. This study focused on the particulate fiber-reinforced polymer composite of the *Caesalpinia decapetala* shell to waste material converts into useable.

The following results can be reached after analyzing the material properties of *Caesalpinia decapetala* seed shall particulate reinforcement fiber reinforced composites with three different percentage compositions and with three different mixing parameters:-

1. *Caesalpinia decapetala* seed shell, a by-product of the *Caesalpinia decapetala* plant, can be successfully compounded with resin for a value-added product and used to manufacture composite materials with different fiber compositions and mixing methods possible by the solid casting process.
2. Different mechanical properties of the product were determined from different weight percentage compositions and different mixing methods. Sample of 30 wt.% of CD contents has a maximum compression strength and maximum energy absorbed during impact test also the the hardness values are maximum, it is 150.8HV. Additionally value of tensile strength was maximum at 20wt.% of CD of composite. This sample was a compassion *Caesalpinia decapetala* particle reinforcement and mixing 1 hour by a magnetic stirrer. The mechanical strength of the composite was also affected by the percentage composition of reinforcement and mixing method. Composite 30% shows better mechanical strength than 10% and 20% of wt.% reinforced composite.
3. The maximum water-absorbed sample was 30 wt. % CD content composite. It absorbed 1.7% before 10 days due to the maximum weight present of reinforcement and inappropriate homogeneity because of being minimum mixing time. The composite after 10 days at the equilibrium no value change.
4. Inclusion of CD particulate improves the wear resistance capacity of the proposed composite in a versatile manner. 30 % volume percentage of CD particulate declared to be a more wear resistive under at higher load.
5. The SEM and EDS results corroborates the homogenous dispersion of reinforcement and existence of aluminium respectively.
6. The natural hardness inhibited with CD particulate found to be the root cause for the improvement of wear resistance.
7. The implementation of hybrid GRA-ANN mathematical model predicts the optimal wear parameters as 40 N of normal load, 4 km of sliding distance and 30 Vol. % of composition of reinforcement.
8. ANOVA results earned from GRA announces the prime significance of CD composition among rest of other wear parameters. The accuracy of the proposed model is validated with R^2 value near to 95 %.

9. The error comparison between the experimental and ANN prediction assures the negligible error of less than 0.49 % which in turn upholds the adequacy and accuracy of the proposed hybrid model.

5.3. Recommendations

In this investigation, fabrication of epoxy-based *Caesalpinia decapetala* seed particulate reinforced composite had an important and a potential application. Many researchers have conducted plenty of work on natural fiber and synthetic fiber composites in recent decades due to these reasons *Caesalpinia decapetala* seed shell particulate was utilized in the present study. It was an abundant natural resource with the characteristics of its low density and weight properties. This natural filler results in a better tribological and mechanical property of the composite. Based on the results, the following application areas of the composite can be recommended that include industrial, automotive (seat backs, doors panel and dashboards), household like ceiling flooring, tables, partition walls and for cabinets.

5.4. Future works

Future researchers can investigate many other features of particle loaded with *Caesalpinia decapetala* composites as a result of this study. Future research recommendations include the following:

1. Possible use with the addition of other ceramic/metallic fillers, polymeric resins other than epoxy and natural fibers in the development of hybrid composites and also metal/ceramic matrix.
2. It is known that filler surface modification increases the bonding strength between filler and the matrix. Therefore, filler modification can be done to improve the strength.
3. These composites are showing promising results for tribological and mechanical applications. Therefore, this work can be further extended to other mechanical, chemical and tribological tests.

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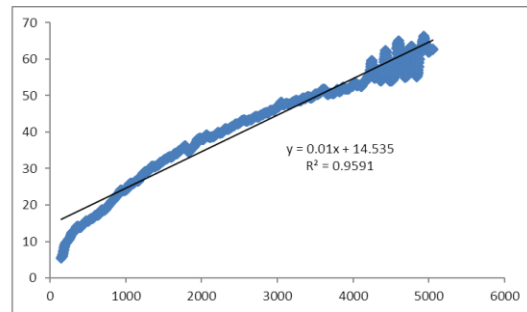
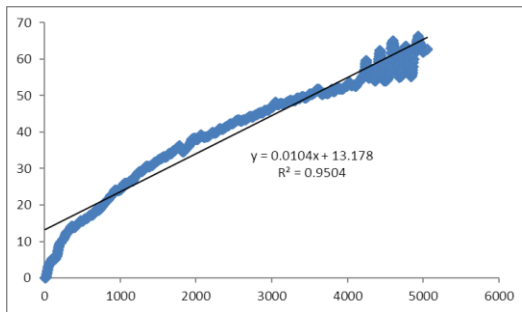
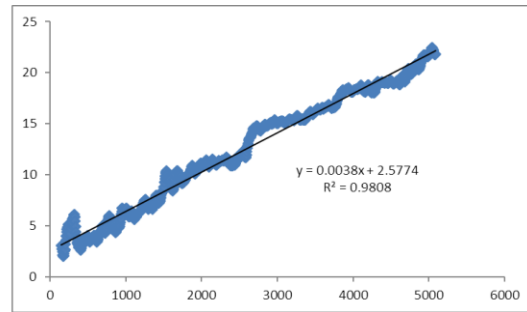
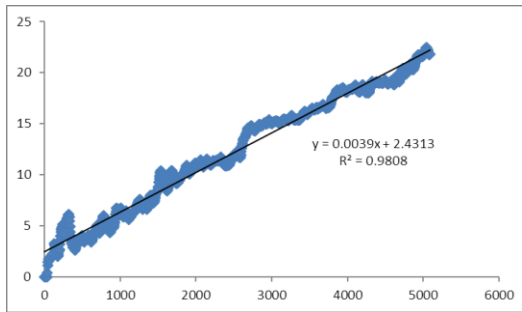
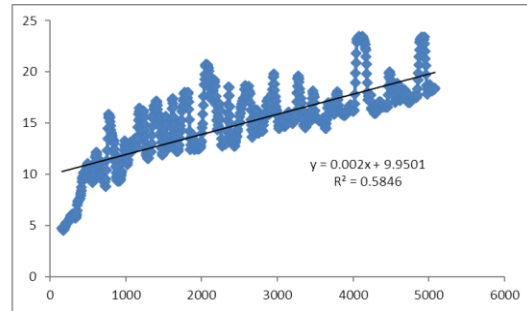
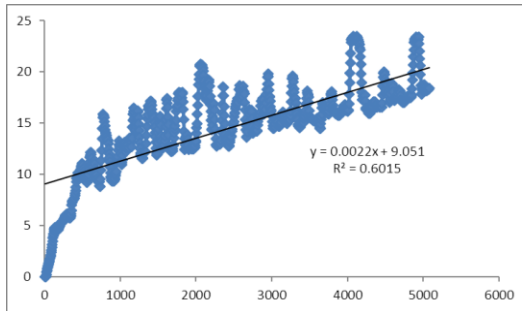
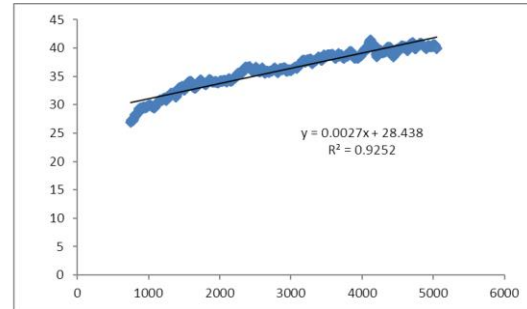
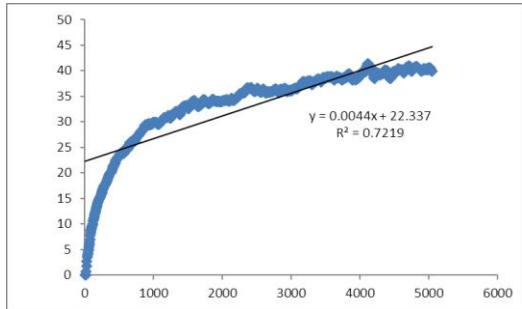
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Appendix A

The graph display during conduct the wear test on pin on disc uprates



Appendix B

Compression test

To: - Adama Science and Technology University
Adama

Subject :- Regarding composite Testing

Upon your request dated on May.13/2022 to perform, compression, and Impact Test of Composite for your post graduate student (**Hailmariam Biratu**), the respective test is conducted in our Mechanical testing laboratory.

Accordingly the analysis is completed as per request and hence, you find the report attached here.

Attached : - 4

With Regards



Dr. Col. Bisrat Yosef (Dr.)
Defence University College of Engineering
Academic Dean (Acting)

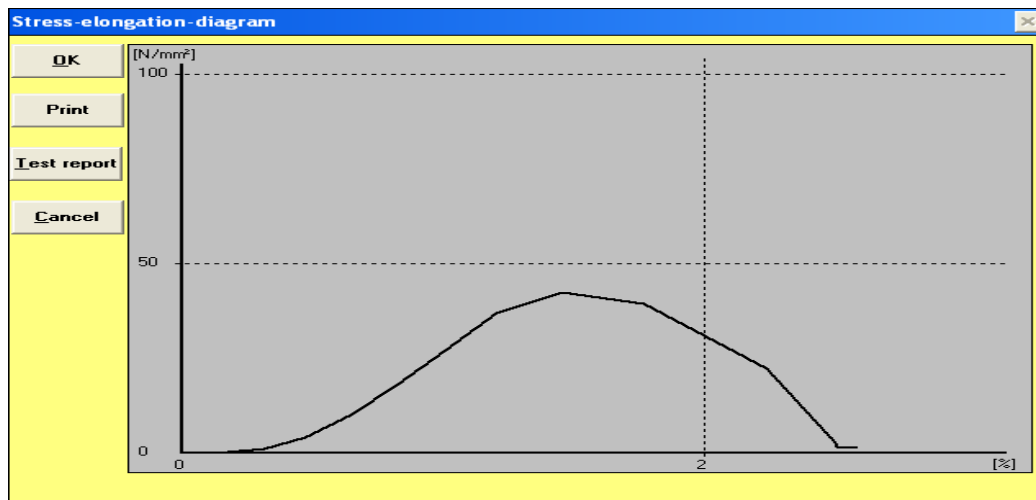
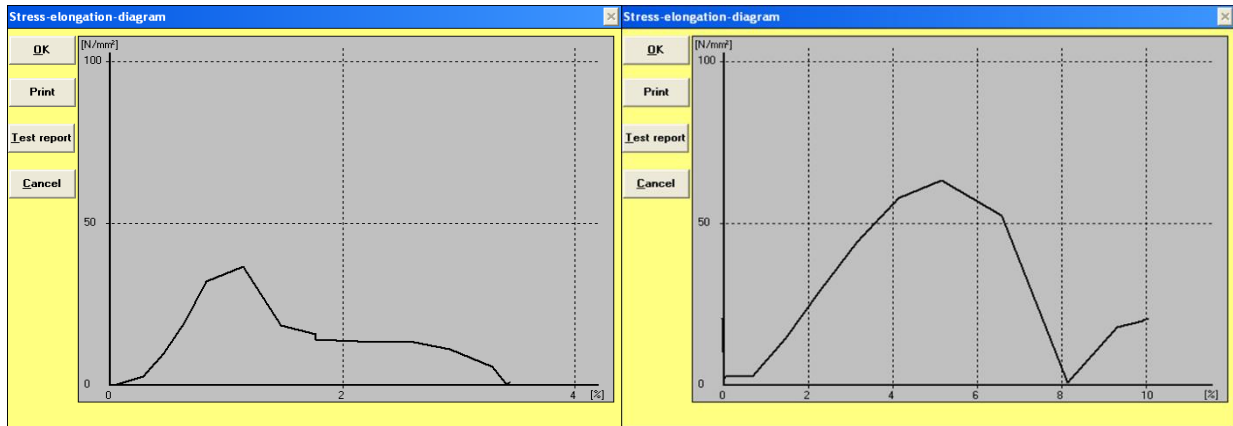
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Bishoftu -Eth



Appendix C

Hardness

