

Experimental Investigation of Pumice Particulate Reinforced Epoxy Composites



Yordanos Zelalem Mengistu

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
Master's in Manufacturing Engineering

Office of Graduate Studies
Adama Science and Technology University

June, 2023G.C

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DECLARATION

I hereby declare that this Master Thesis entitled “**Experimental Investigation of Pumice Particulate Reinforced 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 are used for this thesis have been duly acknowledged through citation.

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LIST OF ACRONYMS AND ABBREVIATIONS

ASTM	America Society for Testing and Material
CM	Composite Material
CMC	Ceramic Matrix Composites
E	Neat Epoxy
EDS	Energy dispersive X-ray spectrometer
ER	Epoxy Resin
GHG	Green House Gas
HV	Vickers Hardness Number
MMCs	Metal Matrix Composite
PMCs	Polymer Matrix Composite
PPE	Pumice Powder Epoxy
PRPCs	Particle Reinforced Polymer Composite
RSH	Rectangular Hollow Section
SEM	Scanning Electron Microscope
UTM	Universal Testing Machine
XRD	X-Ray Diffraction

NOMENCLATURE

E_{fm}	Flexural modulus
m_1	Mass of sample in air
m_2	Mass of sample in water
V_M	Volume of matrix
V_{PP}	Volume of pumice powder
V_v	Void Content
ρ_a	Actual density of composites
ρ_M	Density of matrix
ρ_{PP}	Density of pumice powder
ρ_T	Theoretical density
ρ_w	Density of water
σ_f	Flexural stress
E_t	Tensile modulus
σ_c	Compression stress
σ_i	Impact strength,
σ_t	Tensile strength
σ_t	Tensile stress
A	Cross-sectional area
b	Width of specimen
d	Surface of indentation
E	Energy absorbed by the material during test
F	Applied load
h	Height of the specimen
HV	Vickers Hardness
M	The slope of the load/deflection curve
S	Support span
t	Thickness
$W\%$	Percentage of water absorption
W_a	Weight of the sample after soaking in water

W_b

Weight of the sample before soaking in water

ε

Tensile strain

ABSTRACT

Pumice stone is found in nature during the rapid cooling and solidification of molten lava and it is available in Ethiopia abundantly. The characteristics, compositions, abundance, biodegradability, eco-friendliness, lightweights and high thermal resistance of it, makes pumice a competitive candidate among other commercial particulate reinforcements for polymer matrix. In the present work, pumice particulates are utilized to prepare epoxy based composites. Four types of composites are prepared by open mold casting method by varying pumice content (0, 10, 20, and 30 vol.%). Physical characterization in terms of particle size analysis, X-Ray Diffraction (XRD), Energy dispersive X-ray spectrometer (EDS), density and void content estimation was carried out. Mechanical characterizations in terms of tensile, flexural, compression, impact, and hardness test finally water absorption studies were performed as per American Society for Testing and Materials (ASTM) standards. Density of composites decrease with increase in pumice content due to the low density pumice particles. Compressive properties of composites enhance significantly by reinforcing pumice content. Modulus of all the composites is increased by 54-58% when compared to plain epoxy while specific modulus also reveal increasing trends with increase in pumice content and noted to be in 65-93% higher than neat epoxy. The strength of all pumice/epoxy composites is found to be 41-49% higher than that of neat epoxy. Flexural, tensile and impact properties are in line with the compressive results observed and are mainly credited with the hard pumice particles to enhance the properties. Water absorption behavior of composites reveals that composites with 10 vol. of pumice reveal the lowest moisture absorption as compared with all other compositions. The property map underlines the importance of using pumice/epoxy composites over numerous syntactic foam composites, suggesting the effective use of naturally occurring lightweight materials for envisaged applications. Morphological study of compression tested specimens is evaluated by using scanning electron micrographs.

Keywords: *Epoxy, Lightweight composites, Physical properties, Property Mapping, Pumice, Water absorption,*

CHAPTER ONE

INTRODUCTION

1.1. Back ground of the study

Materials are used in structural and non-structural, electrical, electrochemical, biological, biomedical, thermal, and other applications to improve human productivity and living conditions. Many automotive industries are using metals to manufacture vehicle frame components due to its availability and simplicity to manufacture. The weight of the material inside the car production sector influences the fuel consumption that is directly associated with the corresponding amount of emission to the ecosystem (Lamrot, 2021). Modern businesses everywhere need materials with complex material characteristics that are not available from more common materials like ceramics, metals, and polymeric materials. Engineers and material scientists are particularly interested in composite materials because of these requirements (Jose et al., 2021).

The term “composite material” further clarifies that it is a combination of materials. When two or more constituent materials with noticeably different physical or chemical properties are mixed, the result is a material with distinct properties that are different from those of the constituent materials. When compared to the properties of individual materials, the enhancement renders composite materials superior. Some composites are well suited for use in applications like tennis rackets, fishing rods, car bodies, boat hulls, and aircraft parts because they combine high strength with low weight (Gholizadeh, 2019; Rajak et al., 2019). A composite material is made up of embedded reinforcement (fibers, particles, flakes, and/or fillers) in a matrix (polymers, metals, or ceramics). The reinforcement improves the matrix's overall mechanical properties while assisting the matrix in assuming the proper shape. When properly constructed, the new material combines the advantages of the individual components for increased strength (Nagavally, 2017). Composite materials, by definition, comprise at least two materials. In a combination, each takes up a distinct phase i.e. the matrix phase or the dispersed phase.

1.2. Types of Composite Materials

Composite materials are commonly classified in to two distinct phases:-

- a) **Matrix phase:** The matrix is the continuous primary phase. It shares weight with the scattered phase and keeps it together. The matrix is frequently more malleable and ductile (Zaghloul et al., 2022).
- b) **Reinforcing phase:** The secondary phase with a discontinuous form contained in the matrix is known as the dispersed phase. Because it typically outperforms the matrix in strength, it is referred to as the reinforcing phase (Zaghloul et al., 2022).

1.2.1. Classification Based on the Matrix Phase

The three subcategories that can be made within the composites field are ceramic-matrix composites, metal-matrix composites, and polymer-matrix composites. These materials are widely employed in a range of technical applications as shown among other places in Figure 1.1.

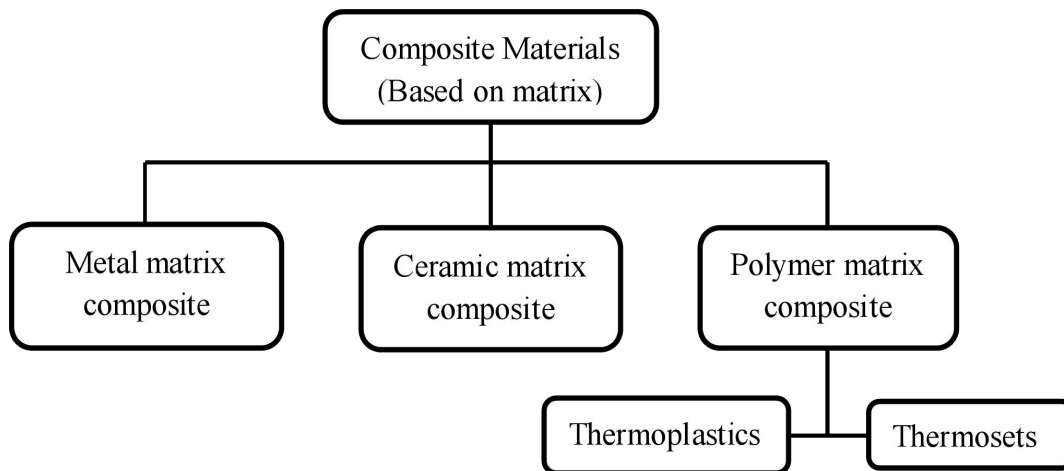


Figure 1.1 Classification of composite based on matrix (Nazeer and Safiulla, 2018).

i. Metal Matrix Composites (MMCs)

Metal matrix materials like Mg, Al, and Fe are used to make metal matrix composites. Due to their greater elastic modulus, tolerance to high temperatures, and unique characteristics like high strength, stiffness, dimension stability, improved moisture resistance, conductivity, and

toughness of fracture, MMCs are frequently employed in industrial applications (Singh et al., 2020).

ii. Ceramic Matrix Composites (CMC)

In this kind of composite, the ceramic matrix phase contains ceramic fibers. This design solves the issue of conventional ceramic constructions' low crack resistance. The matrix and dispersion phases are both excellent choices for materials like carbon, silicon carbide, alumina, etc (Rajak et al., 2019).

iii. Polymer Matrix Composites (PMCs)

In this type of composite, several short or continuous fibers are connected by polymer matrices. Polymer matrices are widely used in composite materials due to their cost-effectiveness, ease of production, and low research costs. Compared to ceramic and metal matrices, they also offer superior properties at room temperature. The matrix could be thermoplastic, elastomeric, or thermosetting (Hsissou et al., 2021). Due of their greater strength and heat resistance, thermoset are more widely used than thermoplastics. Thermoplastic polymers melt when heated and repeatedly get softer due to heat. Most frequently, the matrices are thermoset resin systems such as epoxies, phenolic, and polyamides (Xavier and Ramesh, 2023). The matrix connects the reinforcing fibers, distributes the constraints, provides chemical resistance for the structure, and provides the final product with the proper shape. Strength, stiffness, and light weight are some of PMCs' benefits (Rajak et al., 2019). Polymers are categorized in to three as shown in Figure 1.2.

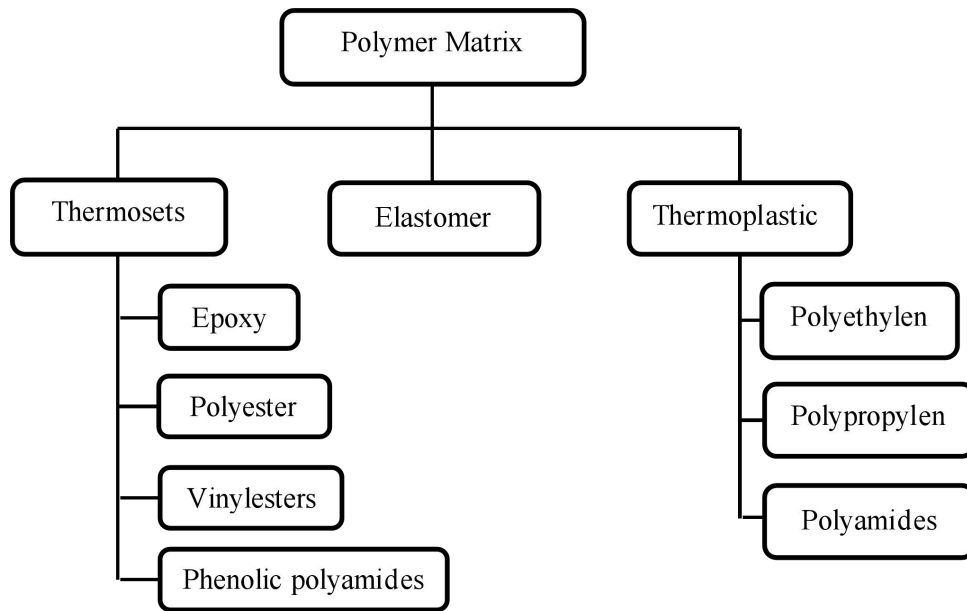


Figure 1.2 Classification of polymer matrix (Chohan et al., 2022).

1.2.2. Classification based on reinforcing materials

Based on how the reinforcing material is classified, polymer composites can be separated into three groups: structural composites, fiber reinforced polymer matrix composites, and particle-reinforced polymer matrix composites (Figure 1.3).

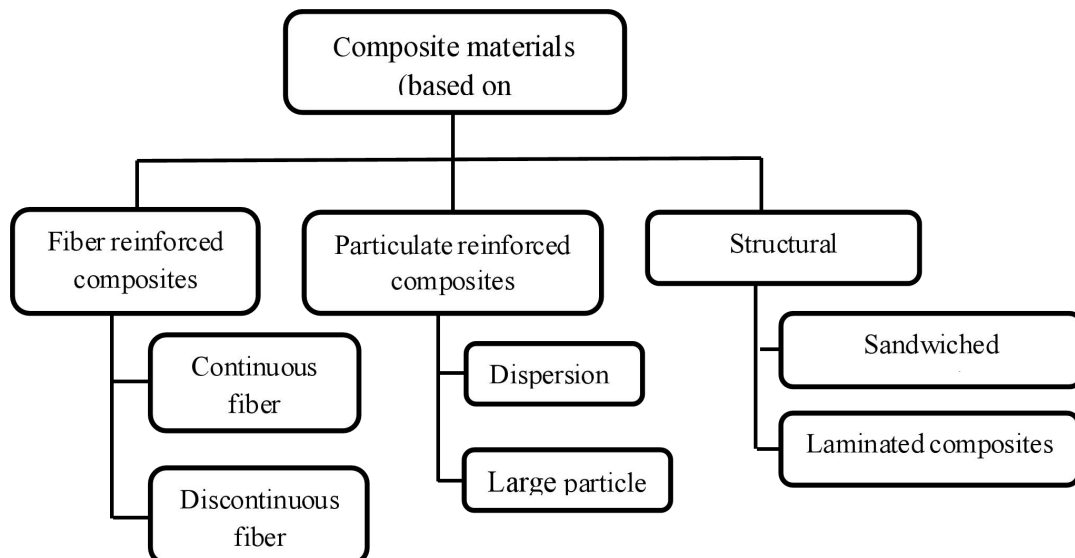


Figure 1.3 Classification of composite based on reinforcement (Rajak et al., 2019).

i. Fiber-Reinforced Composites

Materials are strengthened by the use of fibers. Improved mechanical qualities including strength and strength-to-weight ratio are offered by fiber-reinforced composites. It combines brittle, stiff, strong fibers with a softer, ductile matrix. The fibers that do the majority of the heavy lifting are transferred the load through the matrix, which serves as a conduit (Dixit et al., 2017). They are further classified as either continuous or discontinuous fibers (Li et al., 2022).

➤ Continuous fiber-reinforced composites

The length of the fibers in continuous fiber composites can range from a few feet to several thousand feet. Composites made of long, continuous fibers are often more expensive than those made of short, discontinuous fibers. But it makes up for it by greatly enhancing the mechanical qualities and performance of composites (Li et al., 2022).

➤ Discontinuous fiber-reinforced composites

A few of the several factors that impact discontinuous fiber-reinforced composite qualities are the strands' length, thickness, and orientation in- and out-of-plane. These materials typically display a wider variety of mechanical properties due to their random layout and typically shorter is further divided into two sub-sub-divisions. They are made up of discontinuous fibers that are aligned and dispersed fibers that are randomly orientated (Shah et al., 2020).

ii. Particle-Reinforced Composites

Particulate composites are some of the composite fabrication classes that are utilized the most commonly since they are readily available and inexpensive. Particulate composites are formed of embedded or distributed particles in a matrix body. Particle-reinforced composites have the benefits of being inexpensive, simple to produce, and simple to mold. Depending on the manner of strengthening, they might be further separated. This kind of composite can be categorized as large particle and dispersion-strengthened, depending on the typical size of these reinforcements (Egbo, 2021; Nagavally, 2017).

- **Dispersion Strengthened:** In this instance, the particles are noticeably smaller. The strengthening takes place at the atomic and molecular levels, just like precipitation hardening does in metals. When mechanical stress is applied, unlike large-particle composites, the matrix carries the majority of the load. In this situation, the scattered particles prevent the propagation of dislocation lines along the matrix, which is the strengthening mechanism that takes place at the atomic level (Egbo, 2021).
- **Large Particulate:** This kind of composite contains a large number of coarse particles that enters; tend to reduce matrix deformation at shared surfaces. These constraints are the basic mechanism for strengthening large-particle composites. Instead of necessarily improving them, they are frequently created to yield odd combinations of properties. They are applicable as the primary load carriers and, because they have millimeter-diameter or larger diameter to the MMC, CMC, and PMC matrix types (Egbo, 2021).

iii. Structural composites

Structural composites are combination of laminate or sheet which is constructed layer by layer. Their properties depend not just on those of the constituents but also the geometrical design of various structural elements. Two classes of widely used structural composites are laminar composites and sandwich composites types (Egbo, 2021).

1.2.3. Pumice stone

From the above classification of composites this thesis focuses on particulate reinforced polymer composites. Particulate composites offer several advantages. They provide reinforcement to the matrix material thereby strengthening the material, cheaply available and most importantly reduces health hazard when working with it. Pumice is a rock with an amorphous aluminium silicate composition and a high void ratio that is produced as a result of volcanic activity. The term Pumice is an Italian word which meaning foam. For medium- and fine-grained Ponce, the English names pumice and pumicite, respectively, are used (Soyaslan, 2020). The pumice stone is a naturally occurring lightweight aggregate that resembles a sponge that was formed during the quick cooling and solidification of molten lava. Lava is largely rhyolitic in composition if it is extrusive in nature. When gases in the molten volcanic materials are trapped during cooling, the porous structure of pumice is created by trapping

millions of small air gaps or bubbles (Rashad, 2021; Redick, 2023). Pumice has low density, strong fire resistance, good thermal and acoustic insulation, adequate compressive strength and satisfactory elasticity, a porous structure with low permeability, and non-combustibility in the event that pumice stones float on water due to their low density in comparison to water (Rashad, 2019).

It can be found particularly in Turkey, Italy, Greece, Ecuador, Chile, Ethiopia, Uganda, Guatemala, Saudi Arabia, the United States, Cameroon, Algeria, Tanzania, France, Syria, Spain, Guadeloupe, and other nations (Ash, 2019). Pumice is a glassy volcanic rock fragment with a high vesicularity of 50–80%, and its characteristics vary based on the circumstances of its creation, including melt viscosity, temperature, and the length of the pressure drop. Due to these qualities, pumice is used as a good reinforcing element for composite materials (Bhatia, 2022; Inoue and Fujita, 2023). Pumice has been extensively used, domestically and abroad, for infrastructure works, lightweight concrete, lightweight masonry blocks, and horticultural applications. It has a reduced CO₂ footprint because thermal energy is not needed for its expansion compared with the artificial lightweight aggregates (Nomikou et al., 2022).

From polymer matrix composite thermoset resins have large molecular structures, which give them excellent thermal, mechanical, and electrical properties. Among the different thermoset matrix, epoxy, polyesters and vinyl esters are commonly used in automotive, infrastructure, aircraft, marine, chemical, and electrical applications (Chen et al., 2021). Epoxy resins were discovered in 1909 by Prileschajew (Y. Jin et al., 2018). Epoxy is used very effectively as an adhesive and as a laminating resin for most of the engineering applications. It offers excellent moisture barrier qualities when used in polymer composites (Jeyapragash et al., 2020). ER has been increasingly used in the applications such as structural adhesives, surface coatings, engineering composites, in the aerospace, aircraft industries and electrical insulation, due to their manufacturing characteristics, and product performance. In fact, replacing tools made from metal, wood, and other materials with epoxy resin has been shown to improve efficiency, reduce production costs, and speed up processes in various industries (Sukanto et al., 2021). For advanced composite materials Epoxy resins are widely used as heavy-duty anti-corrosion coatings because of their exceptional properties, such as easy processing, high safety, excellent solvent and chemical resistance, toughness, low shrinkage on cure,

mechanical and corrosion resistance, and excellent adhesion to many substrates (F.-L. Jin et al., 2015). Epoxy resins are commonly used in various applications Because of their excellent mechanical properties and high adhesiveness to many substrates currently epoxy resins are intensively used across a wide range of fields (F.-L. Jin et al., 2019; Kumar et al., 2019).

In the present work, particulate based epoxy composite from pumice powder is developed and its mechanical behaviour under various loading conditions is investigated for varying vol. % of pumice powder.

1.3. Statement of Problem

Pumice stone is a remarkable natural lightweight aggregate that possesses both strength and lightness, making it an excellent material for a marine structures. Found abundantly around the river beds in Ethiopia, it offers a sustainable and readily available resource. However, the pollution around river beaches has resulted in drifts of pumice particles that harm aquatic life, particularly fish, by entering their respiratory organs due to their lightweight nature.

To address this issue, the present study develops a solution by reinforcing pumice particles in a commonly used epoxy matrix, develop composites that showcase a range of unique physical and mechanical properties. This innovative approach not only provides a sustainable solution for lightweight materials but also helps decrease pollution around the rivers, protecting aquatic life and preserving the environment.

1.4.Objectives

1.4.1. General Objective

Preparation and characterization of pumice powder reinforced epoxy composite to investigate the different mechanical and physical properties.

1.4.2. Specific Objectives

- Fabricating pumice powder reinforced epoxy composite by open mold casting method.
- Determining the effect of volume percentage of pumice on the characteristics of composites.

- Study the physical properties in-terms of particle size analysis, density, hardness, XRD, EDS and SEM.
- Investigating mechanical properties in-terms of tensile, compression, flexural, impact and water absorption.
- Proposing specific application for the composite based on the properties exhibited and analysis obtained.

1.5. Scope of the study

- Synthesised of composite material with varying weight percentage of pumice particulate and epoxy resin.
- Focused on testing of mechanical property and characterization of the composite.

1.6. Significant and expected outcome of the study

1.6.1. Significance of the Study

- Processing of novel pumice particulate for potential applications in marine structures wherein the materials known as buoyancy materials have specific gravity considerably lesser than water.
- Utilization of natural resources to fabricate composites that can meet envisaged applications.
- Low apparent weight of pumice can be used to lower the overall density of composites for potential lightweight applications.
- Publications in peer reviewed journals to signify the use of reinforcement.

1.6.2. Expected Outcome of the Study

The expected outcome from the experimentation and analysis were:

- Improving the properties and characteristics of composite when compared to the polymer without filler
- Investigation on the characterization of pumice and comparison with the commercial ones as well as the composites made with them.
- Proving that pumice particles are indeed well suitable for making composites for industrial and domestic applications.

1.7. Organization of the thesis

The thesis is organized as follows: Chapter one provides an introduction that outlines the background, statement of problem, objectives, scope of the study, significance of the study, and limitations. Chapter two includes a literature survey, a summary of the literature, and identifies research gaps. Chapter three covers the materials and methods, including the fabrication and characterization of physical and mechanical properties. Chapter four presents the results and discussion of different test performed under physical and mechanical characterization. Chapter five includes the conclusion and recommendations, where the interpretations of the obtained results are analyzed, and future work is recommended.

CHAPTER TWO

LITERATURE REVIEW

2.1. Introduction

This chapter presents a comprehensive literature analysis of particle-reinforced epoxy/polymer matrix composites. A review of the literature is provided, which includes information about particulate polymer-based composites. The chapter offers a critical review of previous research conducted on this topic, providing valuable guidance for the current study. Additionally, this chapter covers the examination of previous research papers.

- Natural particulate and mechanical properties of natural particulate polymer composites
- Pumice particulate and mechanical properties of pumice particulate reinforced polymer composites
- Manufacturing techniques of polymer composites and finally summary and research gap were presented.

2.2. Natural particulates and mechanical properties of natural particulate polymer composites

The use of particulates in polymers is increasing for a variety of reasons, including enhanced thermal conductivity, improved processing, cost savings, improved physical qualities including hardness and density, and improved mechanical and tribological capabilities. For example, in cable applications, alumina trihydrate particles are used as fire retardants, while metakao-li particles are used to improve mechanical and tribological capabilities. Numerous factors, including the form, size, and surface chemistry of the particles, influence the distinctive features of various fillers (Bhatia, 2022). By combining different reinforcing agents (fibers and particles), polymer matrix composites are created. Natural or synthetic materials may be employed as reinforcing agents in polymer matrix composites. However, reinforcing elements like fibers and particles made from plants, vegetables, fruits, and seeds can replace and minimize the use of plastic components (Mallick, 2018). In recent years, the usage of polymer composites reinforced with natural fibers or particles has increased in a

variety of engineering applications, including automotive, structural, cosmetic, and household appliance uses. In polymer-matrix composites, the use of particles as reinforcing agents can be employed to reduce the ductility and enhance the modulus of the polymer matrix. Particulate composites strengthen the matrix material by adding reinforcement, which also increases its availability at a reduced cost and, most importantly, reduces the risk to human health when employed (Jose et al., 2021). Dispersion Particle interactions with the matrix in reinforced composites are molecular in nature and have substantially lower particle sizes. The total strength of the composite is increased by these molecular-scale particle-matrix interactions. In a manner similar to how precipitate-hardened metals pin, the tiny particles also prevent dislocation motion throughout the composite (Kundu et al., 2022). Waste natural materials such as coconut shells, groundnut shells, oil palm shells, coir pith, cashew nut shells, bamboo fibre, eggshell powder, pistachio shell powder, rice husk, and wood dust have a remarkable potential as reinforcing fillers in polymer resin matrixes (thermosetting and thermoplastic). All of these natural waste products have strong mechanical properties and can be used more effectively to develop composite materials for various applications. These materials have various benefits, including low density, low cost, lesser pollution, excellent toughness and hardness, good thermal characteristics, reduced tool wear, and biodegradability (Hadi and Fadhil, 2021).

The following list of natural particle reinforced polymer composites includes some of the aforementioned particulates depending on various qualities.

Jose et al. (2021) investigated on the impacts of calcium carbonate on groundnut shell powder/vinyl ester composites mechanical characteristics. The scientists discovered that adding more calcium carbonate enhances the composites mechanical properties, and they discovered that the composite specimen with particles as small as 0.5 mm exhibited the lowest rate of water absorption and the highest impact strength. Composite specimens with 1 mm-sized particle spacing showed the maximum ductility and tensile strength.

Hadi and Fadhil (2021) researched done on the qualities of the epoxy resin reinforced with pistachio shell powder in the impact, tensile, and flexural tests. By using the hand lay-up method, a pistachio shell powder reinforced epoxy resin with three distinct weight fractions

(5%, 7%, and 9%) has been developed. When compared to other samples, the sample (epoxy +7% wt. pistachio shells) performs best in the tensile test, flexural test, and impact strength. The main findings of this study are that samples containing 9% weight fraction of pistachio shell in epoxy resin have decreased elasticity, tensile strength, flexural strength, impact strength, and flexural modulus, while samples containing 5% and 7% weight fraction of pistachio shell in epoxy resin have increased elasticity, flexural strength, and impact strength.

Sekaran et al. (2020) investigated the tensile and flexural strength of composites. To create hybrid composites, epoxy resin is combined with the powdered coconut shell, walnut shell, and wood apple (Bael Fruit) shell as reinforcements. In the investigation, two combinations were created: walnut shell flour-coconut shell flour/PP composites and wood apple shell flour-coconut shell flour/PP composites. Resin and powder are divided 60:40. The ratio of the powder in each combination (coconut shell powder with walnut shell powder and coconut shell powder with wood apple shell powder, respectively) is 30:10. It has been demonstrated through evidence that adding powdered coconut, walnut, and wood apple shells to composite materials can enhance their mechanical qualities. In both tensile and flexural tests, hybrid composites outperformed both individual particle reinforced composites in terms of characteristics. In compared to wood apple shell flour and coconut shell flour/PP composites, walnut shell flour and coconut shell flour/PP composites produce superior outcomes with better characteristics.

Gossaye et al. (2023) studied the Ethiopian eggshells known as Habeshan eggshells to make epoxy-based composites. By altering the Habesha eggshell content (0, 2.5, 5, 7.5, and 10 vol.%), five different types of composites are studied. All eggshell/epoxy composites show increased modulus compared to neat epoxy specimens, regardless of the strain rates. Increases in composites' modulus occur for strain rates of 1.43, 0.1, and 0.001 and range from 17% to 57%, 3% to 107%, and 30% to 114%, respectively. Additionally, for all of the strain rates examined, the compressive yield strength of all Habesha eggshell/epoxy composites is lower than epoxy specimens.

2.3. Pumice particulates and mechanical properties of pumice particulates reinforced polymer composites.

Around the world, natural stones are frequently employed in construction. Natural stones can be divided into groups based on their mineralogy, formation circumstances, and physical and chemical characteristics. Environmental and atmospheric factors also have an impact on the composition of stones (Aygun and Aygun, 2016). Pumice stone is a type of natural stone that was created during volcanic activity, when the lava from a volcanic eruption is suddenly cooled. Pumice is a naturally occurring glassy, light-colour volcanic rock. It has a porous structure, and the dissolved gases that form those pores throughout the cooling process as the lava erupts through the atmosphere are caused by the cooling process. Due to its low density, pumice stone floats on the surface of the water unlike other types of rock (Sever et al., 2019).

2.3.1. Availability of pumice in Ethiopia

In 2000, 12 million metric tonnes (Mt) of pumice (and related minerals) were produced globally. Pumice and pozzolan are still primarily produced in Italy, where annual production is estimated to reach 4.6 Mt. The United States, Chile, Ecuador, Ethiopia, France, Germany, Greece, Spain, and Turkey were among the other top nations. when come to Ethiopia, Pumice is abundant in the Ethiopia Rift, which makes up around 30% of the country's surface (Fetene and Addis, 2020). They are primarily found in regions close to Bishoftu, Adama, Ziway, Butajira, and Ambo.

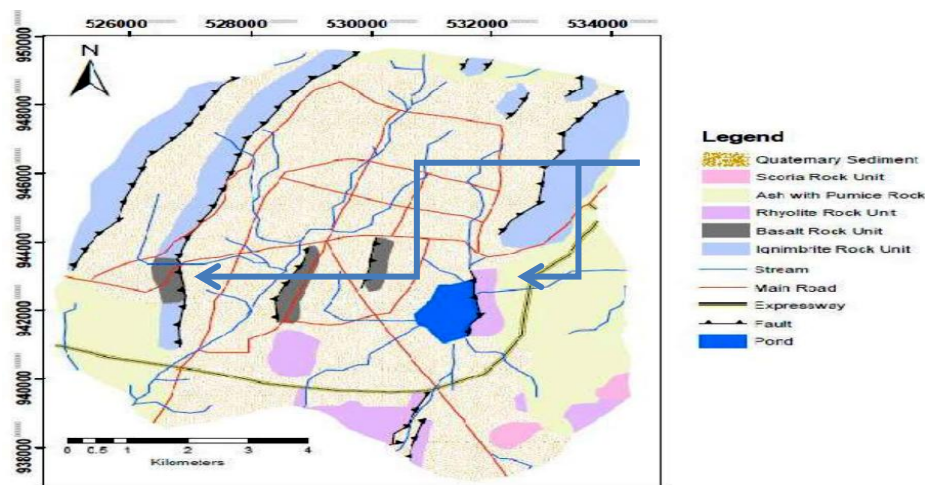


Figure 2.1 Geological map of Adama city (Shiferaw, 2020).

For instance the above Figure 2.1, depicts the map of Adama city which shows the location of pumice stone and other minerals in and around Adama.

Pumice ore's chemical composition is primarily made up of SiO_2 (60–75%), Al_2O_3 (13–17%), $\text{Na}_2\text{O} - \text{K}_2\text{O}$ (7-8%), Fe_2O_3 (1-3%), and CaO (1-2%), with only trace amounts of TiO_2 and SO_3 . Powder, fine aggregate, and coarse aggregate, respectively, all had varying specific gravities ranging from 2.04 to 2.85, 1.18 to 2.52, and 0.82 to 2.17. According to ISO 10390, its pH value is around 7, and its hardness (measured on the Mohs scale) is in the region of 5.5 to 6. Only 15% of the overall volume is made up of solid material, and 85% is air, depending on the particle porosity, which might exceed 85% (Rashad, 2021; Son et al., 2021). Due to its exceptional qualities, such as its low density, high heat and noise insulation, air conditioning characteristics, ease of plaster holding, and elasticity in case of seismic stress and behaviour, pumice offers a number of benefits. Most conventional particle fillers are more expensive than pumice powder (Abdulkarem et al., 2020). Only a small amount of research has looked into the usage of pumice particles as a reinforcing ingredient in composites. Pumice powder is increasingly being used as filler in composite applications today.

(Fleischer and Zupan, 2010) studied the process of making composite panels using pumice stones and epoxy binder. The density of the stones was found to be 490.24 kg/m^3 for both 8 and 4 mm nominal diameter stones. Three epoxy coatings such as 0.67 ('as fabricated'), 0.72 (second coating), and 0.75 (third coating) were applied to achieve different relative densities. Compressive behaviour was evaluated for each density and peak strengths were 2.4, 3.7, and 5.0 MPa, respectively. The results showed that smaller stone diameters which was 4 mm resulted in stronger composites due to increased interaction between the binder and pumice. as relative density was increased the peak strength of the pumice/epoxy structure increased. The structure was cost and weight efficient and has potential for energy absorption applications. The mechanical performance was compared to other core materials and scaling laws were developed.

Gencel (2015) studied about analysis of the raw materials using various methods such as SEA-EDS, XRD, XRF and TG-DTA. Pumice was combined with brick- making materials in different ratios up to 40% by weight. The resulting semi-dry mixtures were then pressed into

molds using a hydraulic press at 20MPa pressure. This study also examined the bulk density, apparent porosity, thermal conductivity, mechanical strength and water absorption of the burned samples using Archimedes principle, as well as their ignition values. The study found that incorporating pumice into the brick as reduced their burned density, while significantly decreasing the thermal conductivity of the 40 % pumice – added brick by over 30% compared to the reference brick without any additive. The bricks also surpassed the standards requirements in terms of compressive strength.

Sahin et al. (2016) studied the mechanical and thermal characteristics of composites packed with pumice particles and made of polyphenylene sulphide and also discovered that adding pumice powder reinforcement increased the mechanical and thermal properties of composites. Furthermore, it was asserted that pumice powders may take on the role of conventional particle fillers.

Dagwa and Adama (2018) aimed to investigate the impact of incorporating pumice powder as a reinforcement material at varying weight percentages (2-8 wt% in 2 wt% increments) into recycled beverage cans using the manual stir casting technique. The resulting mechanical properties (Rockwell hardness, impact strength, and tensile strength) and physical property (density) were evaluated through standard methods. Results showed that when 8 wt% pumice was added, the unreinforced recycled beverage can exhibited an 11.08% increase in hardness and a 28.39% increase in tensile strength. However, the impact strength decreased by 24.24%, and the density was reduced by 39.51% compared to unreinforced recycled aluminium. This composite material could potentially be used in the development of lightweight materials for various applications.

Soyaslan (2020) investigated a traditional hand layup technique in a laboratory setting; this study assessed the mechanical and flammability characteristics of wood-plastic composite panels manufactured from formulas containing pumice powder. To enhance the mechanical qualities of the wood flour-epoxy composites, pumice was blended with the wood flour. Based on composition by weight, the wood flour was combined with five amounts of pumice powder: 5, 10, 20, 30, and 50%. In comparison to unstirred ones, it was discovered that the tensile strength of the composites made with pumice-stirred wood flour increased

significantly. With increasing pumice powder content, up to 50% weight, the composites' tensile and stiffness strengths increased. Investigated was how pumice affected the characteristics of wood plastic composites (WPCs) that make them flammable. Pumice inclusion reduced the composite materials' flammability qualities.

Sari, Suteja, et al. (2022) examined the performance of a cornhusk fiber (CHF)-reinforced polyester composite filled with pumice powder (PP). The impact of various PP volume fractions on the composites tensile, bending, impact, and fracture morphology was investigated. Polyester-CHF composites with different PP volume fractions (5-30%) were created using the hot press process. Results indicated that increasing the PP volume fraction from 5% to 15% improved the tensile strength of the polyester-CHF composite. The modulus of elasticity and bending modulus tended to increase as the PP filler decreased from 5% to 30%, but elongation value decreased. The best bending strength and impact toughness were achieved at a PP filler volume fraction of 20%. Scanning electron microscopy (SEM) images revealed the presence of CHF pull-out in all composite variations, as well as the number of voids dependent on the PP filler volume.

Çoban and Yilmaz (2022) examined the pumice powders with polypropylene packed for its mechanical, viscoelastic, thermal, morphological, crystallographic, and thermo-mechanical behaviours. The composites were created by using a high-speed thermo kinetic mixer, compression molding, and various weight fractions of pumice powder (0–40 wt.%) added to polypropylene . When 10 wt.% of pumice powder was poured into polypropylene, the maximum tensile and flexural strength values of 26.5 and 46.4 MPa, respectively, were attained. The addition of pumice powder also enhanced the storage and loss modulus of polypropylene. With pumice powder loaded into polypropylene, the beginning decomposition temperature of PP rises by 39°C. The crystallinity of polypropylene significantly increased once pumice powder was added. It's noteworthy to note that polypropylene's thermal conductivity increased as pumice powder's weight percentage did. It may be concluded that pumice particles are a useful filler material for enhancing mechanical and thermal qualities.

Saba et al. (2019) investigated the effects of pine resin on the thermo-mechanical characteristics of cement and pumice composites. Pumices are ground and then divided into

three categories: $d_{\max}$ 5 mm, $d_{\max}$ 10 mm, and $d_{\max}$ 20 mm, depending on their largest grain diameter. Each group receives cement as the binding agent. The ratios of pumice in the samples are thought to be 20, 40, 60, and 80% of the overall volume. Additionally, to create artificial pores in each specimen group, concrete grout contains pine resin (about 1% of the cement-pumice combination). The produced samples are dried for 28 days at room temperature before thermo-mechanical measurements are made. Porosity and water absorption were found to rise as pumice ratio and particle diameter increased, but density, thermal conductivity, and compressive strength decreased. Concrete blocks are made with some artificial pores (in addition to pumice pores), which improve the material's insulating capabilities. For resin-mixed samples with $d_{\max}$ 5 mm, $d_{\max}$ 10 mm, and $d_{\max}$ 20 mm, the thermal conductivity drops by 8–15%, 7–12%, and 5–13%, respectively. For each group of diameters, the values for compressive strength decrease by 10–13%, 6–10%, and 8–12%, respectively.

Koyuncu (2018) examined the mechanical, thermal, and morphological properties of composite materials made from discarded maize husk fibres treated with pumice powder were investigated. At different volume fractions of 5:30, 10:25, 15:20, 20:15, 25:10, and 30:5, maize husk (CHF) and pumice powder (SBA) were combined in a polyester matrix. The outcomes show that adding SBA to the CHF composite improved the adhesion between the CHF and polyester matrix. The SBA/CHF composites' mechanical and thermal properties consequently saw a significant improvement. Among all the composites and the polymer matrix, the composite with a 5:30 volume fraction (JPA) had the highest values of tensile strength (18.61 MPa), tensile modulus (3.23 GPa), flexural strength (36.12 MPa), and flexural modulus (3.39 GPa), which was attributed to the composites' robust SBA/CHF interface bonds. While the lower values of composite specimens' were recorded as a result of many CHF pull outs. In terms of thermal stability, JPA composites performed better than the other composites. According to the morphological investigation, CHF pull out rose with more CHF present and fell as SBA concentration rose and became evenly distributed throughout the composite. The results showed that the CHF/SBA composites had superior mechanical properties compared to the fiber-glass composites.

Çoban and Yilmaz (2022) analyzed various volcanic materials using Fourier transform infrared (FTIR), X-ray diffraction (XRD), and scanning electron microscopy (SEM) at the beginning of the review. Then, it discusses the thermal and mechanical characteristics of polymer composites that contain various volcanic particle constituents, including volcanic ash, pumice, perlite, and tuff. Volcanic particle materials have been shown in peer-reviewed studies to be useful as fillers for polymer composites. However, there are a number of variables that need to be taken into account and optimized in order to produce a balanced polymer composite with the necessary qualities, including volcanic particle type, size, surface treatment, dispersion, filler concentration, and interfacial interaction with the polymer matrix. In particular, it was advised to concentrate on the study of volcanic particle materials in various polymers, particularly bio-based and recycled ones, according to uses that are both economical and environmentally friendly.

Kösedağ (2023) studied on mechanical properties of epoxy mixed with cheap pumice to create a stronger composite material. The objective was to evaluate the worth of the pumice and improve the mechanical properties of the epoxy. Composite samples with no filler, 10%, 20%, and 30% filler were produced. Samples were made by using an ultrasonic mixer and vacuuming. The composites were tested for mechanical properties and the distribution of the powder was observed using an optical microscope. XRF analysis was used to determine the composition of the pumice powder. The results showed that adding filler greatly improved the strength and strain values of the composites, with the best mechanical values found in the 20% pumice composite. The maximum stress value of this composite was 30.6 MPa and the corresponding unit strain was 0.049%.

2.4. Manufacturing techniques of polymer composites

Manufacturing processes are divided into thermoplastic and thermosetting plastics due to differences in their physical and chemical properties. Several fabrication methods can be utilized to create polymer matrix composites, as shown in Figure 2.2 (Chohan et al., 2022). The focus of this research is on the fabrication of thermosetting matrix composites.

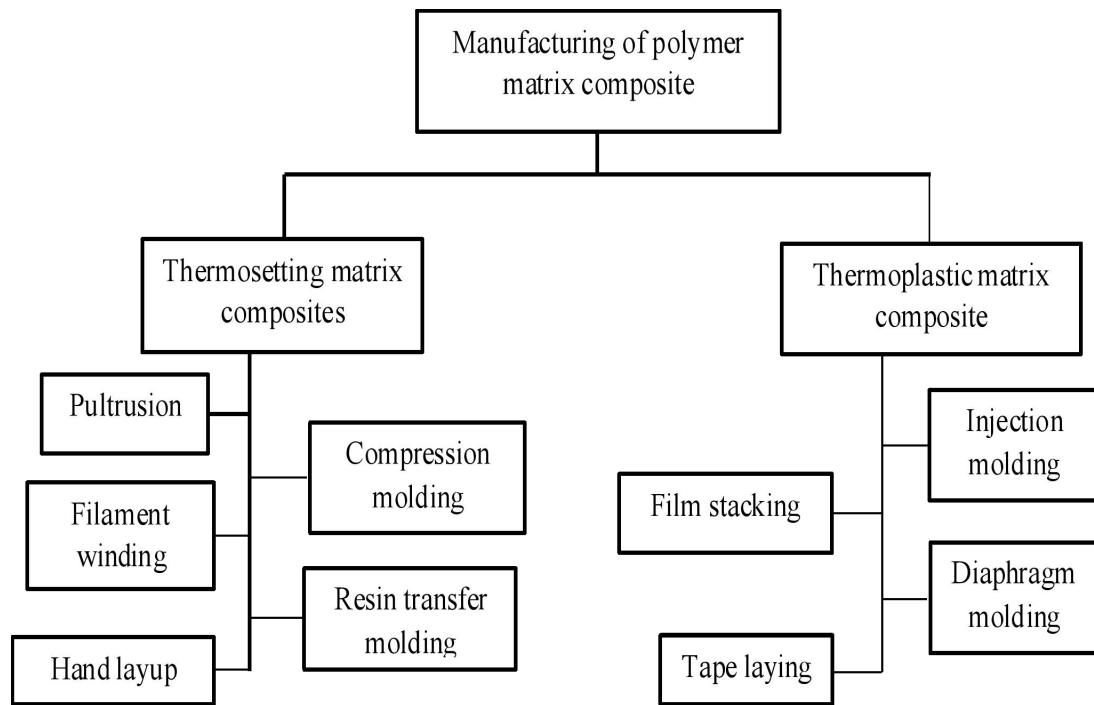


Figure 2.2 Classification of manufacturing techniques used for polymer matrix composites.

2.4.1 Pultrusion

Pultrusion is a manufacturing process used to create polymer matrix composites. The process involves pulling continuous fibers through a resin bath before passing through a die, where the resin cures and hardens, forming a laminate with the same cross-sectional profile as the die. The resulting profiles are then cut to the required lengths using a travelling cut-off saw after being constantly pulled through the die by a puller (Cavaleri et al., 2019). This process is considerably simpler than using alternative techniques like filament winding or resin transfer molding and allows for the creation of large composite structures using affordable facilities, tools, and materials which is shown in Figure 2.3.

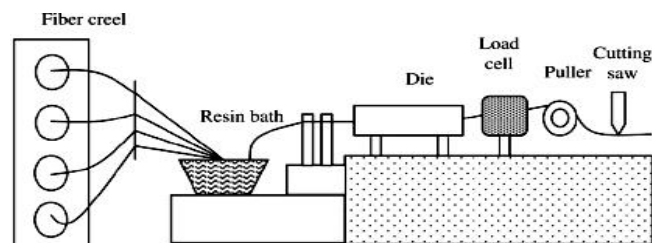


Figure 2.3 Pultrusion process (Balasubramanian et al., 2018)

2.4.2. Compression molding

Compression molding is another polymer processing method used to produce polymer composite products. This technique is commonly employed for processing the most popular rubber compounds and thermoset polymers in powder form (Verma et al., 2019). Compression molding is especially beneficial for automotive applications, such as the fabrication of sheet molding which is shown in Figure 2.4.

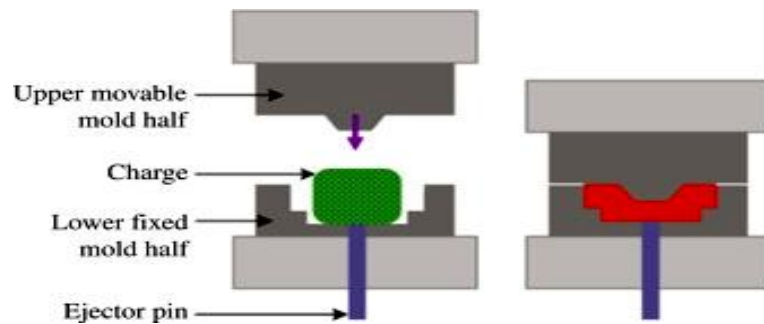


Figure 2.4 Compression molding process (Edebali, 2021).

2.4.3. Filament winding

Filament winding is a composite manufacturing technique that is typically used to produce circular components. As illustrated in Figure 2.5, continuous fiber rovings are drawn through a resin bath by a puller. Each pre-impregnated composite is then coiled around the revolving mandrel. To achieve a uniform distribution of the composites along the length of the mandrel, the composites are also traversed in the x direction (Verma et al., 2019). There are three different types of winding patterns used in filament winding: hoop, helical, and polar windings. These patterns are selected based on the specific requirements of the component being produced.

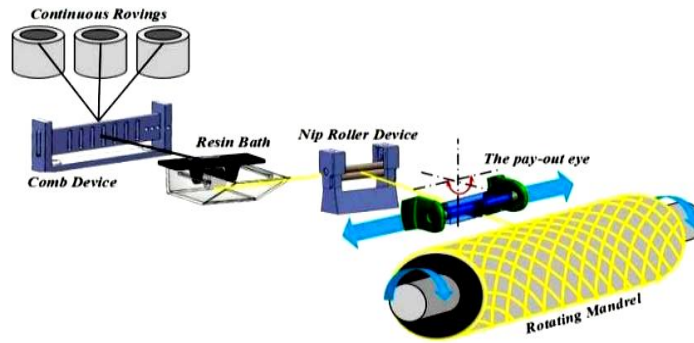


Figure 2.5 Filament winding process (Q. Ma et al., 2019).

2.4.4. Resin transfer molding

Resin transfer molding involves the use of a rigid, closed mold. In this process, the preform is arranged using a half-mold and then compressed by sealing the mold. The resin is transferred into the mold using various tools, such as pressure or vacuum, while maintaining a positive gradient pressure, which ultimately releases trapped air from the preform. Polyester, epoxy, vinyl ester, and phenolic resins are commonly used in this technique. To remove trapped air from the mold, a vacuum is used at specific vents. The resin impregnation of the preform and subsequent curing of the composite constitute another phase (Sapuan and Yusoff, 2015). As the final stage, the item must be taken out of the mold. Figure 2.6 demonstrates how fiber compression improves the mold closing order, enabling it to have the proper thickness for the composite. RTM is frequently employed to create parts for cars and airplanes.

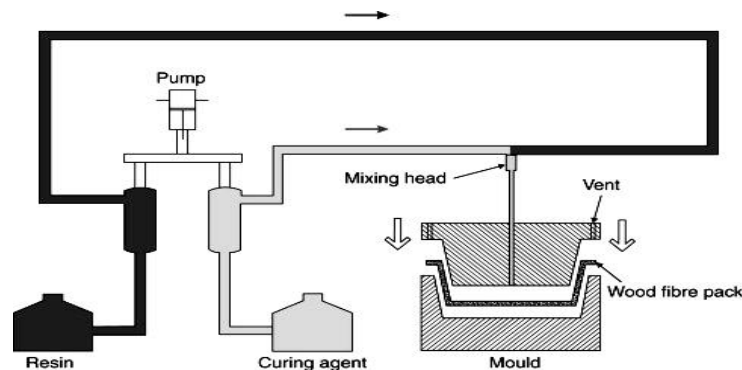


Figure 2.6 Resin transfer molding process (Dai and Fan, 2014).

2.4.5. Hand layup

Hand lay-up is an open molding technique commonly employed to produce polymer composite products. It is a straightforward and widely used method for processing thermoset-based fiber and particle composites. This technology offers several advantages, including lower mold costs, easy mold maintenance, and accessibility to manufacturing equipment. To ensure a good surface finish for the product and to prevent the polymer from sticking to the mold surface, thin plastic sheets were placed at the bottom of the mold, and a releasing agent was applied (Getahun et al.; Lamrot, 2021). Molds can be made from a variety of materials, including wood, metal, and plastic. The process involves manually placing fiber reinforcements and resin on the mold surface which as shown in Figure 2.7.

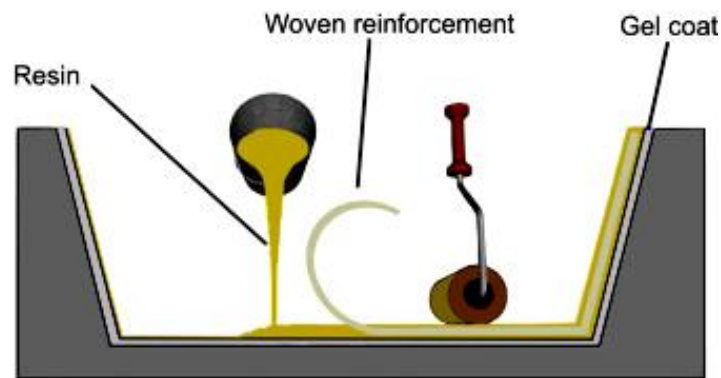


Figure 2.7 Hand layup process (Raji et al., 2019).

2.5. Literature Summary

Table 2.1 Summary of literatures.

Authors	Title	Their works	Result
Gencil (2015)	Characteristics of fired clay bricks with pumice additive.	Mixing the brick raw material with pumice (10, 20, 30, and 40) wt.%. SEM-EDS, XRD, XRF, TG-DTA, Thermal conductivity and mechanical strengths are measured	Thermal conductivity of bricks with pumice reduces >30% compared to without additive of pumice. compressive strength becomes higher than the required one with additive of pumice
M. Koyunchu (2018)	Mechanical and flammability properties of wood plastic composite with pumice powder	Mixing wood flour with pumice up to 50 wt.% mechanical property and flammability are measured	Tensile strength increases and flammability properties decreased with addition of pumice compared to without addition of pumice
Sari et al. (2021)	Evaluation of mechanical, thermal and morphological properties of corn husk modified pumice powder reinforced polyester composites.	Corn husk (CHF) and pumice powder(SBA) were mixed in polyester matrix at varied volume fractions of 5:30, 10:25,15:20, 20:15, 25:10, and 30:5.	The average compressive and tensile strength of concrete containing 10% Ground Pumice as a cement replacement is highest.

Kösedağ (2023)	Investigation of the effect of filling ratio on mechanical properties of pumice filled epoxy-based composites.	Mixing epoxy with no filler, 10%, 20%, and 30% filler of pumice and fabricate by using ultrasonic mixer and vacuuming. Then after compressive strength tested.	Best compressive strength values obtained in the 20% pumice composite With maximum stress value was 30.6 MPa and strain was 0.049%.
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2.6. Research Gap

Based on the available literature, it is inferred that most of the works are focused on the use of pumice as a filler or partial replacement of cement to make lightweight concrete, lowering production costs and GHGs emissions while also improving concrete performance. Furthermore, pine resin, polypropylene and polypropylene sulphate and few thermoplastic matrices are used for preparation of composite. However, there are no works that are reported on the use of pumice as reinforcement in preparation of composites that is abundantly available in Ethiopia. Therefore, pumice particulate reinforced epoxy composites with varying content of pumice were investigated in this work.

CHAPTER THREE

MATERIALS AND METHODS

3.1. Introduction

In this chapter, the materials chosen for the study and the fabrication method used to produce the composites are presented. Additionally, the tests utilized for characterizing the microstructure, mechanical, physical, and water absorption properties of the composites are described.

3.2. Materials

3.2.1. Matrix Material - Epoxy Resin

Epoxy polymers are a popular choice of matrix in composite materials due to their exceptional resilience to humidity and fatigue, high hardness, strong adherence to reinforcements, and minimal shrinkage after curing. Epoxy also possesses robust mechanical, thermal, and corrosion-resistant properties, making it suitable for a wide range of industrial applications (Koyuncu (2018)). Due to numerous benefits over other polymers, the thermosetting matrix used in this study was a medium viscosity epoxy resin (LAPOX L-12) and a room temperature curing polyamine hardener (K-6) procured from World Glass Fiber Plc, Addis Ababa, Ethiopia.

Hardener (K - 6)

Hardener K-6 is a liquid hardener with low viscosity that can cure at room temperature and is typically used in applications that involve hand layout. It has a fast curing time at room temperature, but due to its strong reactivity, it has a limited pot life. Table 3.1, provides detailed information on the constituent properties of hardener K-6 as supplied by the manufacturer. (Rampur et al., 2020).

Table 3.1 Properties of epoxy and hardener.

Constituent	Trade name	Chemical name	Epoxide equivalent	Density (g/cm ³)
Resin	LAPOX L-12	Diglycidyl Ether of bisphenol A (DGEBA)	182-192	1.192
Hardener	K-6	Tri ethylene Tetra amine (TETA)	---	0.954



Figure 3.1 Epoxy (L-12) and hardener (K-6).

3.2.2. Reinforcement - Pumice Powder

To conduct this study, pumice stones were collected from Adama in Oromia region, Ethiopia specifically around Awash river as tried to show in Figure 3.2.

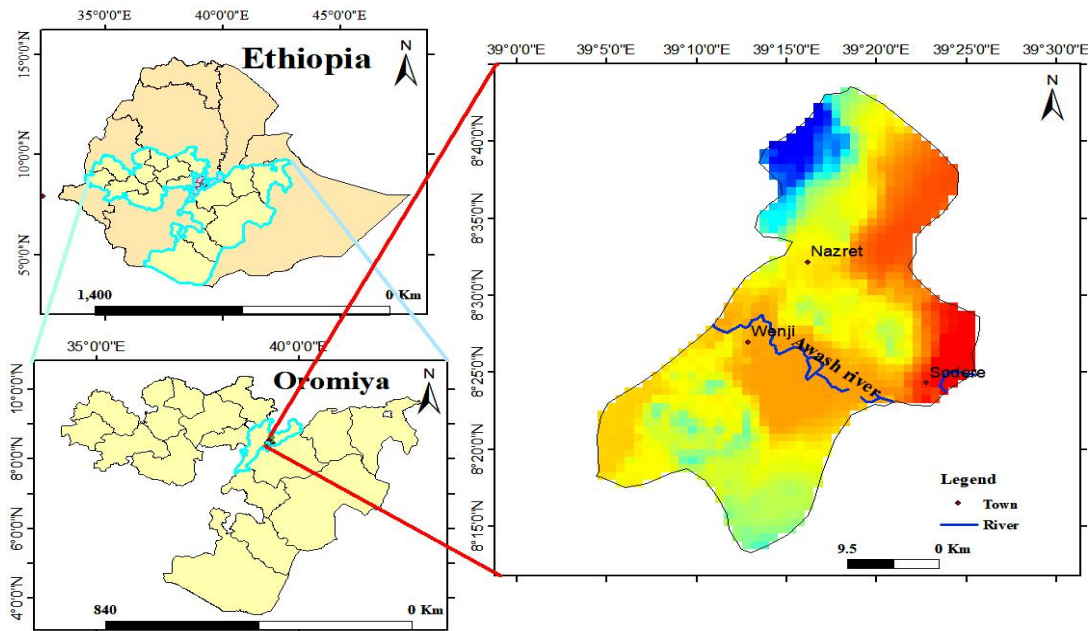


Figure 3.2 Location of pumice stone used for this study.

The pumice stones were cleaned with water to remove the dust particles then dried under sunlight for 24 hours to remove the moisture content. The stones were crushed manually with the aid of hammer to reduce the size of stones into fragments and ground into a fine powder size using a dry ball milling machine. The powder was then sieved using a 200-mesh sieve with a mesh size of 75 μm . Density of pumice powder is determined to be 514 kg/m^3 using an automatic density analyzer (QUANTACHROME ULTRAPYC 1200e).

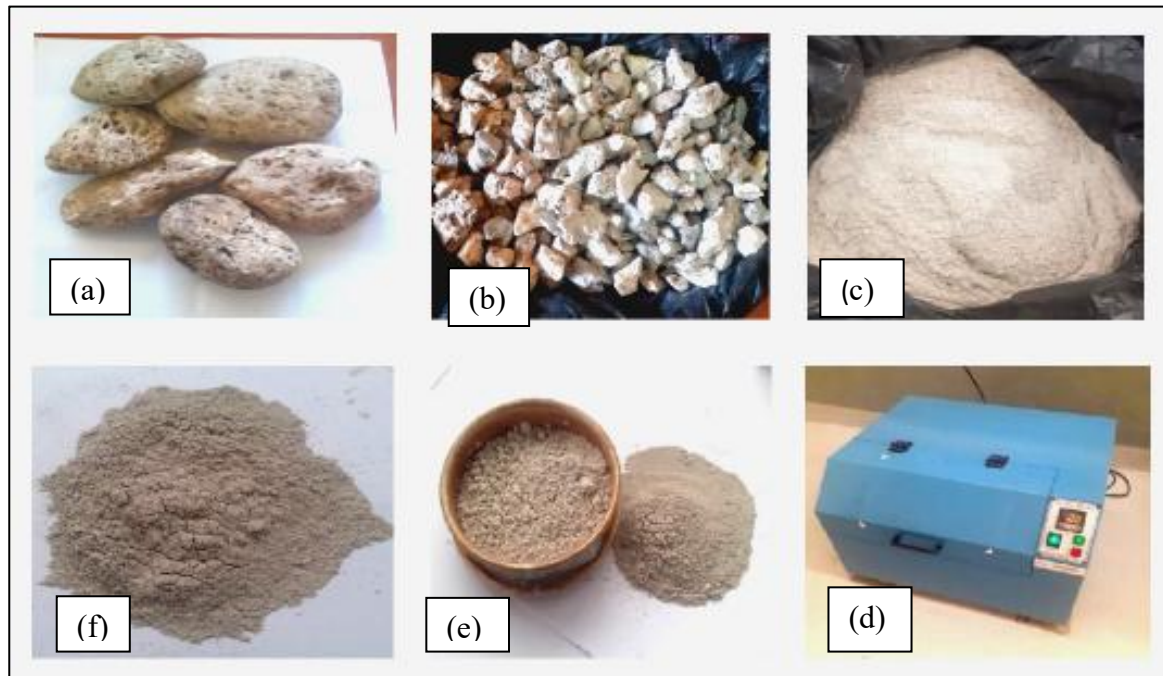


Figure 3.3 Pumice Powder Preparation (a) raw pumice stone, (b) manually crushed pumice stone, c) Manually grinded pumice powder, (d) grinding process of pumice powder, (e) sieving process, and (f). sieved powder

3.2.3. Distilled water

Distilled Water, which has a neutral pH of 7, was utilized in this study for washing pumice stones and during the water absorption test for immersing samples.

3.2.4. Pure Petroleum Releasing Agent

This agent helps to effortlessly remove the composite material from the mold substance, ensuring that the manufacturing process is smooth and efficient. This will ensure that the material has a smooth and flawless surface finish.



Figure 3.4 Pure petroleum jel.

3.3. Process Parameter

Based on literature reviews the smaller particle sizes were preferred for enhancing composite strength. Therefore, pumice powder with a size of 75 μm was used in this study, this was due to keep its light weightiness. When pumice powder is finely ground, its density increases, as noted in Fleischer and Zupan 2010, who found that stones with nominal diameters of 8 and 4 mm had a density of 490.24 kg/m³. In this study, the density of 74.3 μm pumice powder was found to be 514 kg/m³. For marine structures to remain buoyant, their density must be less than that of water. For this case 75 μm were selected. Additionally, in this particle size the strength of the pumice-epoxy composite also impressive when compared to that of syntactic foam composites Pumice powders were added to the composite material in weight percentages of 0, 10, 20, and 30 vol.%.

Table 3.2 Compositions of samples in vol.%.

Codes	Composition (vol.%)		
	Pumice powder	Epoxy	Hardener
E	0	100	5
PE-10	10	90	5
PE-20	20	80	5
PE-30	30	70	5

E refers to pure epoxy whereas PE belongs to pumice particulate-epoxy and the number indicates the volume percentage of filler in the composite. The samples were made from two different sized molds: mold number 1(larger) and mold number 2 (smaller) with equivalent volume percentages.

3.4. Sample Preparation Method

The materials were selected depending on availabilities, economies, and environmental factors. And some these materials were purchased from the markets and some of them were collected from the environments.

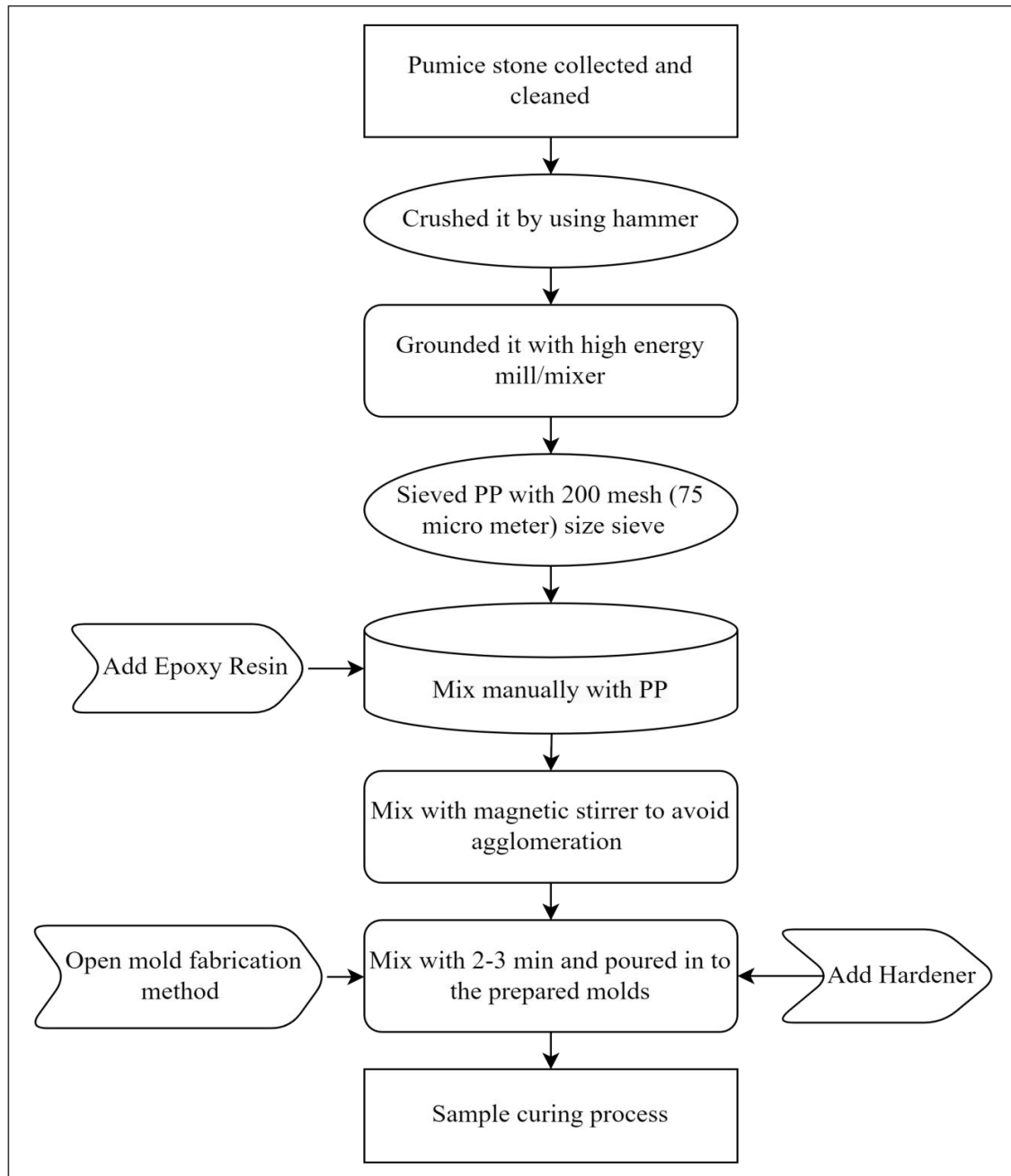


Figure 3.5 Flow chart for sample preparations.

Open mold casting method

The composite material will be produced using the open mold casting technique. This process is the simplest way to manufacture thermoset-based composites and offers several advantages, including lower mold costs, readily available production tools, and easy mold maintenance. To begin the process, a mold is prepared from metal, wood, or plastic, and its surface is coated with a releasing anti-adhesive agent to prevent the mixture from sticking. Then the mixed powder and resin are mixed together to get a consistent slurry and further hardener by 5% is added to the mixture before finally pouring in the mold. Finally, the solution is left to cure and harden in the mold. Four types of epoxy composites were fabricated using pumice powder in 0, 10, 20, and 30 vol.%, respectively.

This process involves the following steps:

- Step 1. The masses of pumice powder and epoxy resin were measured according to the calculated weight fractions as shown in Figure 3.6.

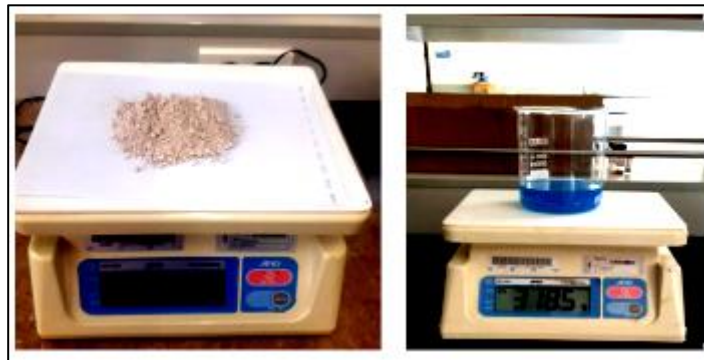


Figure 3.6 Measured pumice powder and epoxy resins.

- Step 2. Pumice powder and epoxy resin were slowly and constantly blended together. A glass rod was used to stir the ingredients in the mixing bowl. Use a magnetic stirrer plate spinning at 500–600 rpm and add a stirrer bar to the container for around 50 minutes to prevent agglomeration and ensure thorough mixing which is shown in Figure 3.7. The mixes were then given a final 5% hardener addition and stirred for 2-3 minutes.

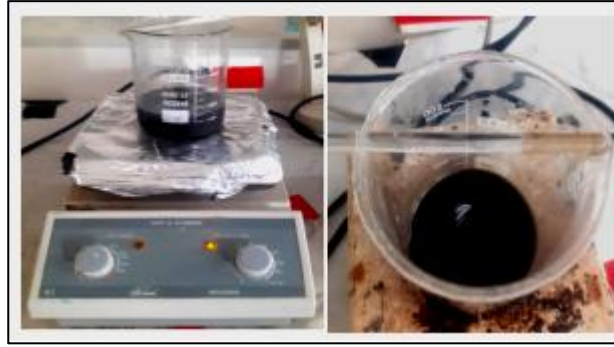


Figure 3.7 Mixing epoxy resins and pumice powder on magnetic stirrer plate.

- Step 3. Mold preparation: RHS, steel plate, and bolts, which are items that can be found nearby, which are used to create two different sized milled steel molds. The first one is 100x100x100 mm in size, while the second one is 300x300x100 mm in size. Before coating the mild steel mold with releasing agent, it should be cleaned with sanitizer. Then, mold release was used to keep the epoxy from adhering to the mold. Finally, the slurry is placed into a mild steel mold that has been impregnated with a pure petroleum-releasing agent. It was selected due to its superb release qualities, simple application, and great mirror finish shine. The samples are cured for 4 hours before being taken out of the mold. The process is illustrated in Figure 3.8 and 3.9.

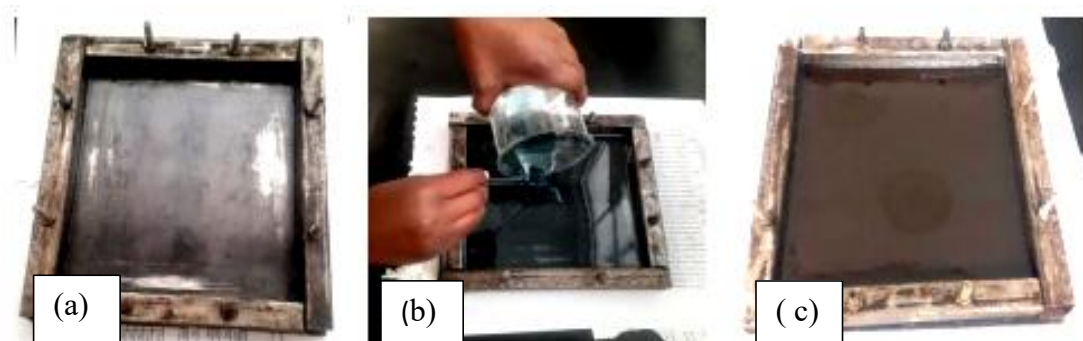


Figure 3.8 Mold number 1, (a) Empty mold (b) Pouring mixed liquids, and (c) Cured samples.

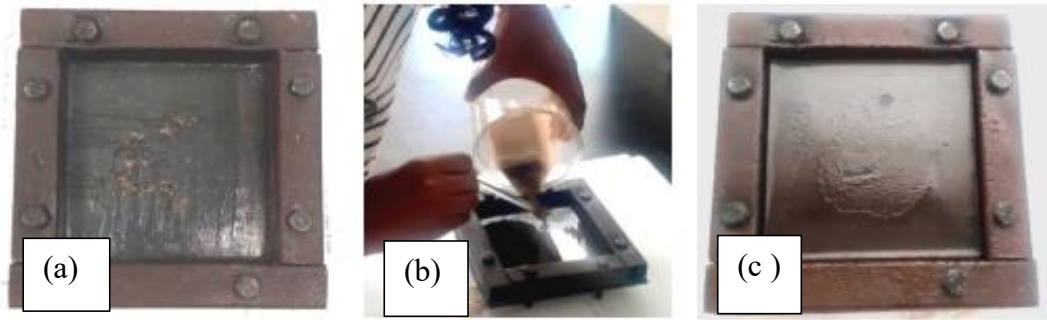


Figure 3.9 Mold number 2, (a) Empty mold, (b) Pouring mixed liquids, and (c) Cured samples.

- Step 4. The slurry is put into a mild steel mold, where it cures for 4 hours before the samples are removed. Fabricated samples were shown in Figure 3.10 and 3.11.

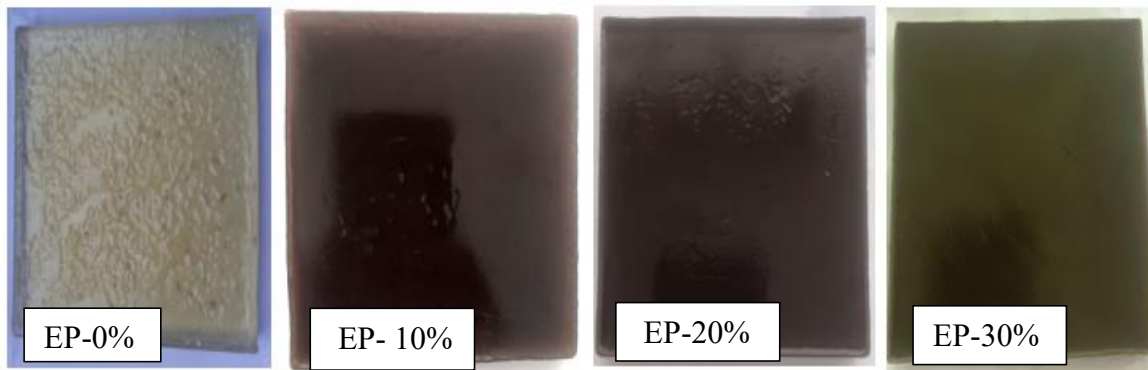


Figure 3.10 Prepared samples on mold number 1.



Figure 3.11 Prepared samples on mold number 2.

- Step 5. The specimens were cut using a jigsaw and a water jet machine in accordance with the ISO and ASTM standards for each test (tensile, compression, flexural, impact, SEM, hardness, density, and water absorption tests) as shown in Figure 3.12.



Figure 3.12 Cutting samples using jig saw.

3.5. Equipment's and Machines used

The instruments and tools used for characterization of composite material were given as follows:

3.5.1. High energy mixer/mill

The ball milling device grinds tough or fragile samples into a fine powder for examination. It uses a grinding vessel with one or more balls that shake ferociously to produce large G-forces. As shown in Figure 3.13, the device has a timer that can be adjusted up to 100 minutes. In case of mechanical failure, a vibration detection switch automatically switches off the grinder. This machine is available in material Engineering department, ASTU.



Figure 3.13 High energy mixer/mill.

3.5.2. 200 Mesh size testing sieve

This sieve is made of stainless steel wire mesh fabric and a brass frame. The No. 200 mesh is a fine-mesh U.S. standard with a nominal sieve aperture of 0.0029" (75 μ m). Die-formed frames offer a perfect fit, superior quality, and performance, ensuring that the American Society for Testing Materials (ASTM) requirements are always met. It is accessible in the laboratory of the ASTU Department of Material Engineering, as depicted in the figure 3.14.



Figure 3.14 200 Mesh size Testing Sieve.

3.5.3. Magnetic stirrer & Hotplate MS300HS

Magnetic stirring machines are used for mixing liquids with precision and control which is available in material engineering department at ASTU. The machine shown in Figure 3.145, are designed to handle even the most demanding mixing tasks with a maximum speed of 1500 rpm and temperature of 380 °C.



Figure 3.15 Magnetic stirrer & hotplate MS300HS.

3.5.4. Analytical (digital) balance

The digital mass balance is a precision tool that can measure mass with great sensitivity, within range of 0.01 to 0.1 mg. This equipment is essential for accurately characterizing physical properties and measuring the mixing of composites. The material engineering department at ASTU provides this machine, and Figure 3.16 shows the mass balances used in this experiment.



Figure 3.16 Digital mass balances.

3.5.5. Hardness testing machine

To determine the Vickers hardness of a material, a square-based pyramid diamond indenter with an angle of 136 degrees is used. The HVS-50 machine, featured in this thesis and shown in Figure 3.17, has a measuring range of 5 to 2900 HV and a test force of 9.807, 49.03, 98.07, 196.1, 294.2, and 490.3N. It can accommodate test pieces up to a maximum height of 180 mm, with a throat depth of 125 mm. The machine offers measuring system magnifications of 125X and 250X, and the minimum scale value of the optical micrometer is 0.5m. Equation 3.1 used for this measurement was obtained from the HVS-50 machine manual. Overall, this study was conducted at the Department of Material Science at ASTU in Ethiopia, and the Vickers hardness testing apparatus was used to accurately measure the hardness of the specimens.

$$\begin{aligned} \text{HV} &= \frac{\text{Constant} \times \text{Test force}}{\text{Surface of indentation}} \\ &= \frac{0.102 \times 2F \left(\sin \frac{136^\circ}{2} \right)}{d^2} \end{aligned} \quad 3.1$$

Where, HV - Vickers hardness value,

F - Load in kgf

d - Arithmetic mean of the two diagonals ($\frac{d_1+d_2}{2}$), d_1 and d_2 in mm.

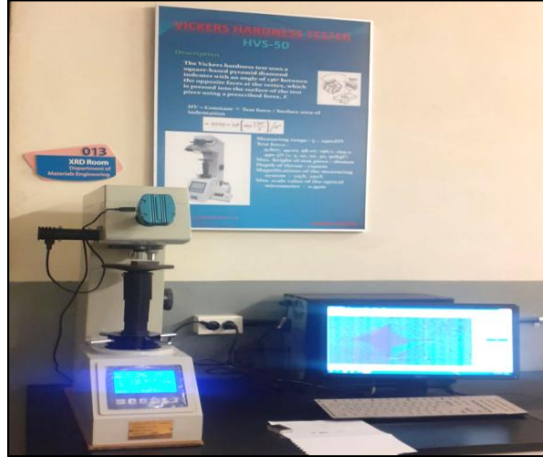


Figure 3.17 Vickers hardness testing machine.

3.5.6. Pendulum impact testing machine

Impact testing measures a material's ability to resist a swinging pendulum strike, and its impact strength is its capacity to absorb and release energy during impact or shock loading. The Charpy V-notch test is a standardized high-strain-stress test that measures the amount of energy absorbed by a material during fracture. The apparatus used was displayed in Figure 3.18.

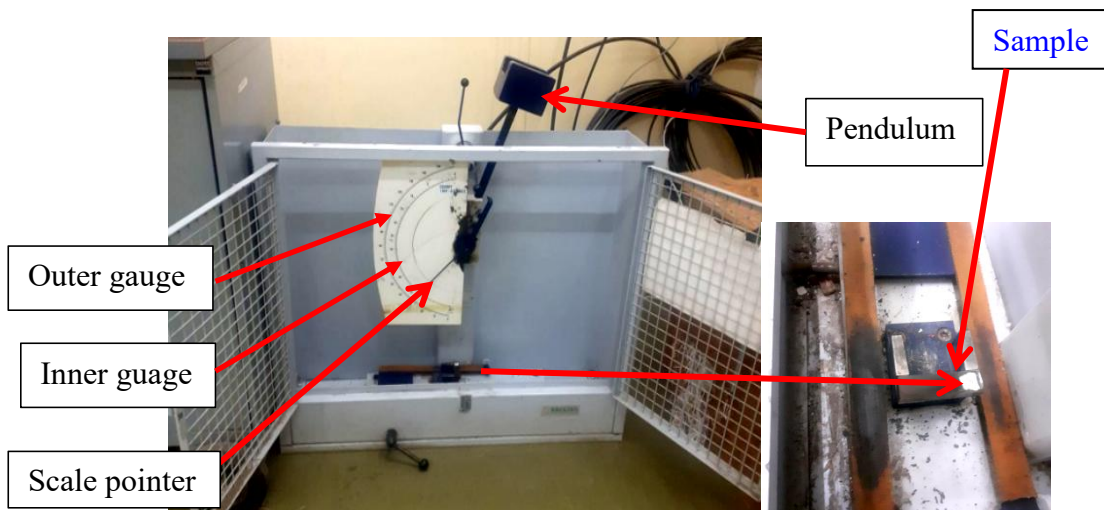


Figure 3.18 Pendulum impact testing machine and set of specimen.

3.5.7. Universal testing machine (UTM)

The universal testing machine (UTM) measures mechanical properties of various materials under different stress conditions. It performs tests like tensile, compression, bending, and shear by switching jaws and modes. Key parts include the actuator, attachment kit, safety devices, engine, gear, screws, and crosshead grip extensometer, as well as computer and control system for stress, strain, and displacement control modes. Test data can be stored, reanalyzed, and printed for all mechanical properties (Selvam and Dinaharan, 2017). The UTM machine was as shown in Figure 3.19.

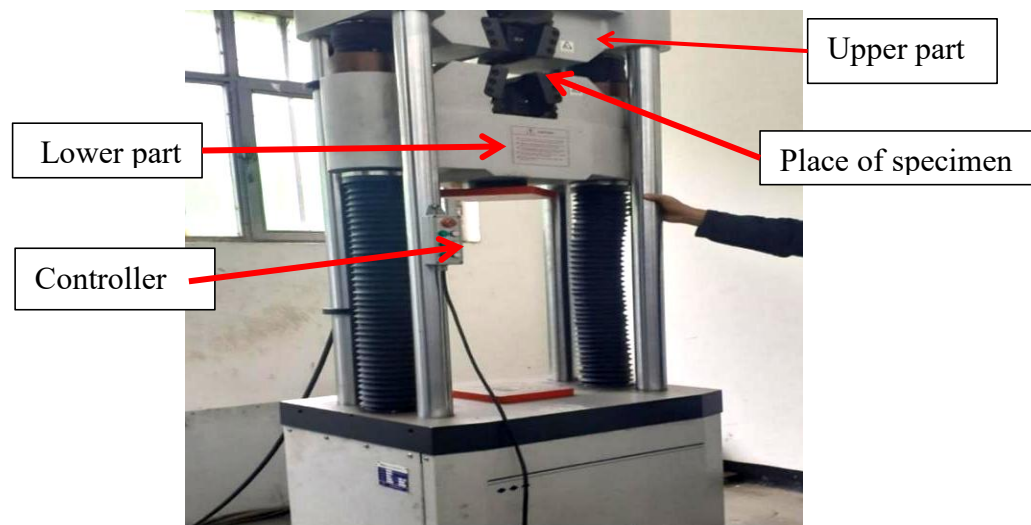


Figure 3.19 Universal testing machine.

3.6. Physical Characterization

3.6.1. Particle size analyser

Particle size examination is performed using CILAS 1064 liquid with a range of 0.04-500 μm . Pumice powder is scattered with the aid of an injection disperser while defined dosage and feed of powder into the jet of compressed air is accomplished with a vibratory feeder. The powder particles are illuminated with a laser as they move in front of a camera that captures the particles at a speed of 175 frames /sec. For every particle captured, the corresponding size is estimated by equating the projection imaged by the camera to the size of the particle having a projected area. Three runs are carried out and the average values are presented. A scanning

electron microscope (JEOL JSM 6380 LA) is used to examine the morphology of specimens subjected to compression testing.

3.6.2. XRD analysis

To describe the crystalline structure of pumice powder, X-ray diffraction (XRD) XRD-7000 X-RAY DIFFRACTOMETER, SHIMADZU Corporation (Japan) was employed (Sever et al., 2019). The powders are tested in material engineering department, ASTU.

3.6.3. SEM -EDS

When it comes to analysing the structure and composition of ultra-small objects, researchers often turn to a cutting-edge technique known as SEM-EDS. By combining scanning electron microscopy with energy dispersive X-ray spectroscopy, this powerful approach gain a detailed understanding of the morphology of tiny structures while simultaneously identifying their elemental compositions (J. Ma et al., 2020). By scanning the surface of a sample with a low-energy electron beam, SEM provides a highly detailed visualization of the sample's structure. But SEM doesn't work alone; it's often paired with another powerful technique called Energy Dispersive X-ray Spectroscopy (EDS). By identifying the distinctive X-rays released by the sample's constituent elements using an electron beam, EDS can determine the elements present in a sample with remarkable accuracy (J. Ma et al., 2020). It's possible to detect microplastics in sample matrices using cutting-edge techniques. By directing a beam into the material, a series of complex interactions occur that result in the emission of photons and electrons near the sample surface. With various modes available for SEM characterization, researchers can detect and analyse the signals created by these interactions to produce highly-detailed images of the sample (Son et al., 2021).

3.6.4. Density and Void fraction

Density is one of the most significant factors in defining the properties of polymer composite materials. The sample was prepared according to ASTM D-792 and is defined as the mass of the material per unit volume. The densities of the specimen was measured by calculating the theoretical and experimental density then the difference between the two gives void content in the sample. In order to determine the actual density of composites the Archimedes principles are used (Chaves et al., 2023). During measurement the distilled water are used as a medium.

Then sinker attached to the wire to ensure that the sample was fully submerged in the water and to prevent it from floating. By measuring the weight of the sample in the air and then again while it is suspended on the wire and submerged in water, this is used to obtain accurate measurements of the sample's density.

The actual density of the composites is given by the following equation 3.2.

$$\rho_c = \rho_w \left(\frac{m_1}{m_1 - m_2} \right) \quad 3.2$$

Where, ρ_a - Actual density of composites

ρ_w - Density of water

m_1 - Mass of sample in air

m_2 - Mass of sample in water

The theoretical density of composite materials in terms of rule of mixture can be obtained as per the following equation 3.3.

$$\rho_T = \rho_M * V_M + \rho_{PP} * V_{PP} \quad 3.3$$

Where, ρ_T - Theoretical density

ρ_M - Density of matrix

V_M - Volume of matrix

ρ_{PP} - Density of pumice powder

V_{PP} - Volume of pumice powder

The amount of void content in a sample is calculated as per equation 3.4.

$$V_v = \frac{\rho_T - \rho_a}{\rho_T} \quad 3.4$$

Where, V_v - Void Content

ρ_T and ρ_a - Theoretical and actual density

3.7. Mechanical Characterization

The mechanical test (tensile, flexural, compression, impact, and water absorption) were performed as per ASTM standards. To ensure the reliability and repeatability of the test results, each test was conducted five times for each composition.

3.7.1. Tensile test

Tensile tests were carried out on computer-equipped universal testing machines with a 20 KN load cell capacity at room temperature. The tests were carried out on the standards of ASTM D-638-14 with a sample size of 165mm overall length, 57mm narrow section length, 19mm overall width, and 13mm narrow width. The distance between the grippers was 115mm. The test specimen was loaded under tension at a fixed speed of 5 mm/min, and important properties such as tensile strength and elongation were measured until the samples became fractured or failed completely as shown in Figure 3.20. The data on load and displacement were taken to compute the stress and strain. Based on this data, the average values of strength and modulus of four samples were discussed.

As shown in Equation 3.5., tensile strength of the samples was calculated.

$$\sigma_t = \frac{F}{A} \quad 3.5$$

Where, σ_t - Tensile Strength

F - Applied Force

A - Cross-sectional Area

As shown in Equation 3.6., tensile modulus of the samples was calculated.

$$E_t = \frac{\sigma_t}{\varepsilon} \quad 3.6$$

Where, E_t - Tensile Modulus

σ_t - Tensile Stress

ε - Tensile strain

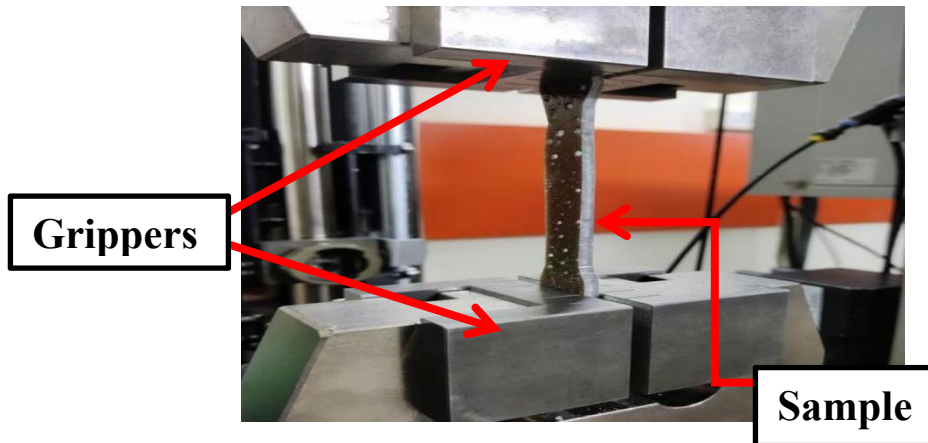


Figure 3.20 The process of tensile test.

3.7.2. Flexural test

The required forces to bend a beam were measured under three points of loading conditions utilizing a computer-controlled Zwick (Zwick Roell Z020, ZHU) machine with a load cell capacity of 20 KN. To ensure accurate and standardized testing, the ASTM D-790-17 standard was used. The measured sample size was 127 mm in length, 12.7 mm in width, and 3.2 mm in thickness. The test was conducted at an across-head speed of 1.4 mm/min with a preload of 0.1 MPa. All specimens had a span length of 52 mm, and each specimen was tested three times before the average data were analyzed which is shown in Figure 3.21. The stress-strain data was collected after the tests were carried out until it became fractured. Flexural stress was determined by equation 3.7

$$\sigma_f = \frac{3FS}{2bh^2} \quad 3.7$$

Where, σ_f - Flexural stress

S - Support span between the two outer points

b - Width of the specimen

h - Height of the specimen

F - Applied Force

Flexural modulus is given by Equation 3.8

$$E_{fm} = \frac{S^3m}{4bt^3} \quad 3.8$$

Where, E_{fm} - Flexural modulus

S - Support span

m - The slope of the load/deflection curve

b - Width of the specimen

t - thickness of the sample

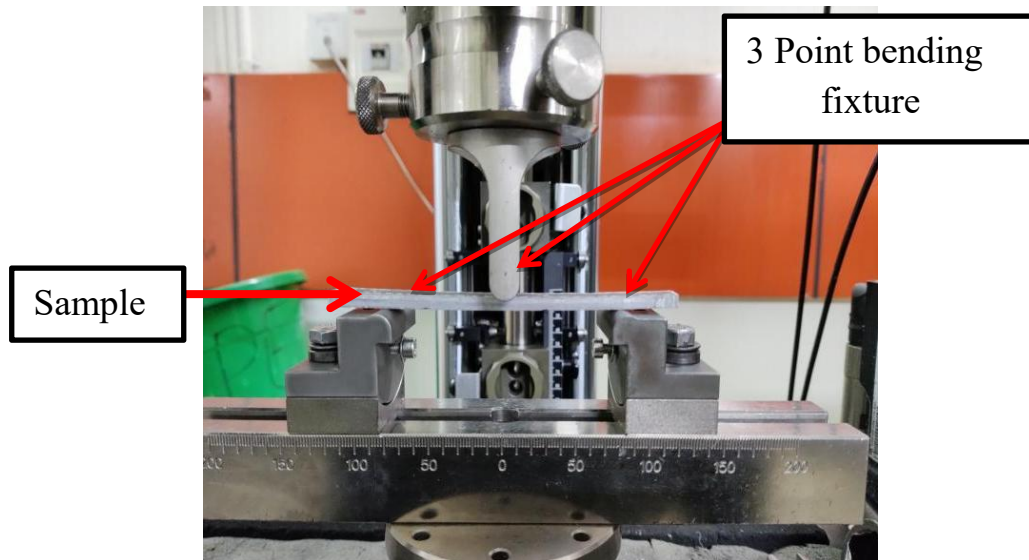


Figure 3.21 The process of 3 point bending test.

3.7.3. Compression test

Composite compression testing is a method of applying compressive loads to materials without risking buckling. Compression tests conforming with ASTM D695-15 standards (12.7×12.7×25.4 mm) are done on the specimen's 12.7×12.7 mm face using a Zwick Roell Universal test equipment. The tests are carried out with a constant strain rate of 1.3 mm/min. The average values of four specimens of each arrangement are provided. By measuring the compression and deformation at various loads, the compressive stress and strain of the material can be calculated. This type of testing is crucial for understanding how materials behave under crushing stresses.

$$\sigma_c = \frac{F}{A} \quad 3.9$$

Where, σ_c - Compression Stress

F - Load applied force

A - Cross-sectional area

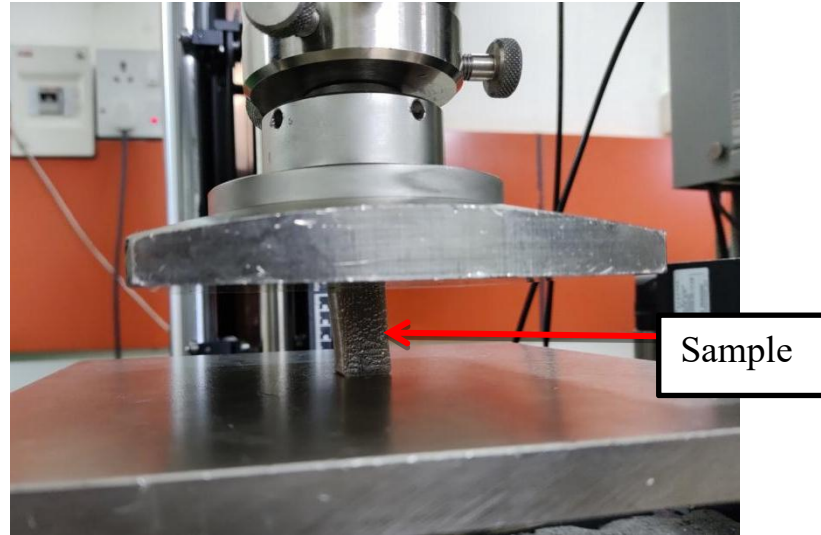


Figure 3.22 The process of compression test.

3.7.4. Impact strength test

To determine how well a material can withstand sudden increases in stress, researchers use an impact test to measure the energy absorbed during fracture. This type of test is particularly useful for assessing the toughness, brittleness, notch sensitivity, and impact strength of engineering materials under high-rate loading conditions. In this case, researchers will be conducting a Charpy impact test on composite materials using a Model IT-30 impact testing equipment and following the ASTM D-256 standard for test specimen preparation. During the test, a pendulum with a 30 kg weight will be used to deliver impact loads to the specimen until it cracks. The impact speed will be 3.8 m/s, and the specimen will have a 2.5mm deep notch cut in it with the striker's opposite direction pointing up. The test will be conducted on samples with dimensions of 55x10x (3-6) mm. Overall; impact testing is an important tool for evaluating the strength and durability of materials. The test was conducted in Bishoftu, Ethiopia, at the Defense University Engineering Collage.

The energy absorbed by the specimen can be calculated by using equation 3.10

$$\sigma_i = \frac{E}{t \times b} \quad 3.10$$

Where, σ_i - impact strength,

E - energy absorbed by the material during test

t - thickness

b - width of specimen.

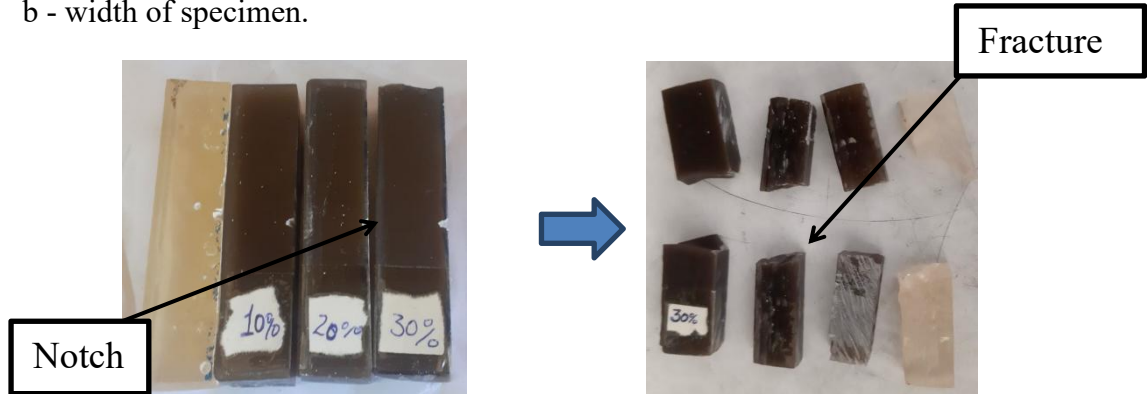


Figure 3.23 Samples before and after testing respectively.

3.7.5. Water absorption studies

To evaluate the water absorption characteristics of a pumice particulate-reinforced epoxy composite, the samples were subjected to a water absorption test. This involved immersing the samples in distilled water at room temperature for a period of eight days, as shown in Figure 3.24(c). Prior to immersion, the samples were weighed and recorded, as seen in Figure 3.24(b). After the eight-day immersion period, the samples were removed from the water, cleaned with dry materials, and once the samples were dry, they were weighed again using a precision digital scale to determine the amount of water absorbed. Using an equation 3.10, the water absorption rate of the samples was calculated (Somaiah Chowdary et al., 2022).

$$W\% = \frac{W_a - W_b}{W_b} * 100 \quad 3.11$$

Where, $W\%$ is percentage of water absorption, W_b = Weight of the sample before soaking in water and W_a = Weight of the sample after soaking in water, and absorption coefficients are calculated by using the formula.



(a)

(b)

(c)

Figure 3.24 (a) Water absorption specimens (b) measuring weight before and after immersion
(c) Specimens immersed in water.

CHAPTER FOUR

RESULTS AND DISCUSSION

4.1. Introduction

In this chapter, the results of the experimental works were discussed in detail. In different sub section particle size analyses, XRD, SEM-EDS, Density and void content, hardness, impact, tensile, flexural, and compression analysis water absorption test results were discussed.

4.2. Particle Size Analysis

Particle size analysis is performed on powdered pumice particles, and the findings are shown in Figure 4.1 (a), Pumice particles have a volume-weighted mean particle size of $74.3 \mu\text{m}$. The particles have a huge extension at the tail end of the curve, indicating that there has been a significant amount of cluster formation. The shear forces generated by manual stirring, on the other hand, are likely to disperse the majority of the clusters. Figure 4.1 (b) depicts a scanning electron micrograph of pumice particles. The form and size of pumice particles are found to be uneven.

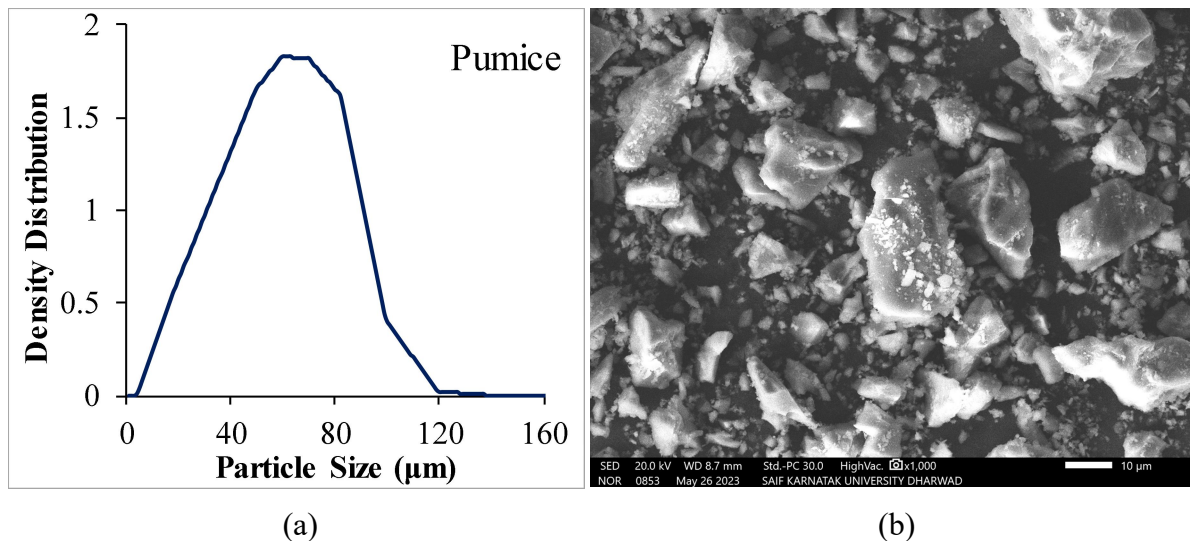


Figure 4.1 (a). Particle size analysis and (b). Scanning electron micrograph of pumice powder.

4.3. XRD

X-ray diffraction (XRD) analysis used to identify the crystalline phases present in the pumice particles. By analyzing the position and pattern of the diffraction peaks, they were able to determine the exact composition of the pumice powder. Figure 4.2, shows the X-ray curve of the pumice particles, revealing three prominent diffraction peaks at $2\theta = 23.7$, 27.7 , and 29.5 , as well as a few other peaks. These peaks indicated the presence of silicate and aluminates groups in the pumice particles, which in turn confirm that the pumice powder picks were indeed crystalline.

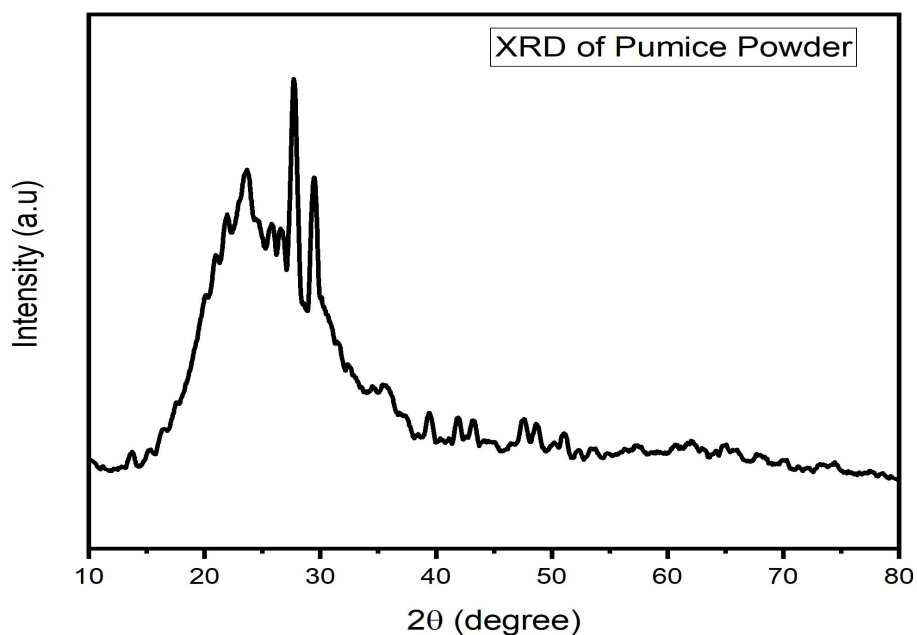


Figure 4.2 XRD graph.

4.4. EDS Analysis

The energy dispersive X-ray spectrometer studies representative X-ray spectra to determine X-ray energy. To identify components present in the specimen, quantitative analysis of X-ray spectra is done. The peaks depicted in the graph are used to calculate the chemical composition. Figure 4.3, demonstrates the EDS spectrum of pumice particles. Major peaks corresponding to oxygen and silicon can be seen while several smaller peaks corresponding to iron, aluminum and carbon can be clearly observed. Table 4.1, displays the energy peaks

corresponding to the main components found in pumice. The peak high ratio of pumice reveals that oxygen, carbon and silicon constitute 77% of the total composition while iron and aluminium combined together amount for 13%. The remaining composition is made-up by calcium, sodium, titanium, magnesium and potassium. Results of EDS spectrum clearly demonstrate the use of pumice as an effective filler due to the presence of elements like silicon, iron and aluminium known for their excellent properties (Sari, Setyawan, et al., 2022)

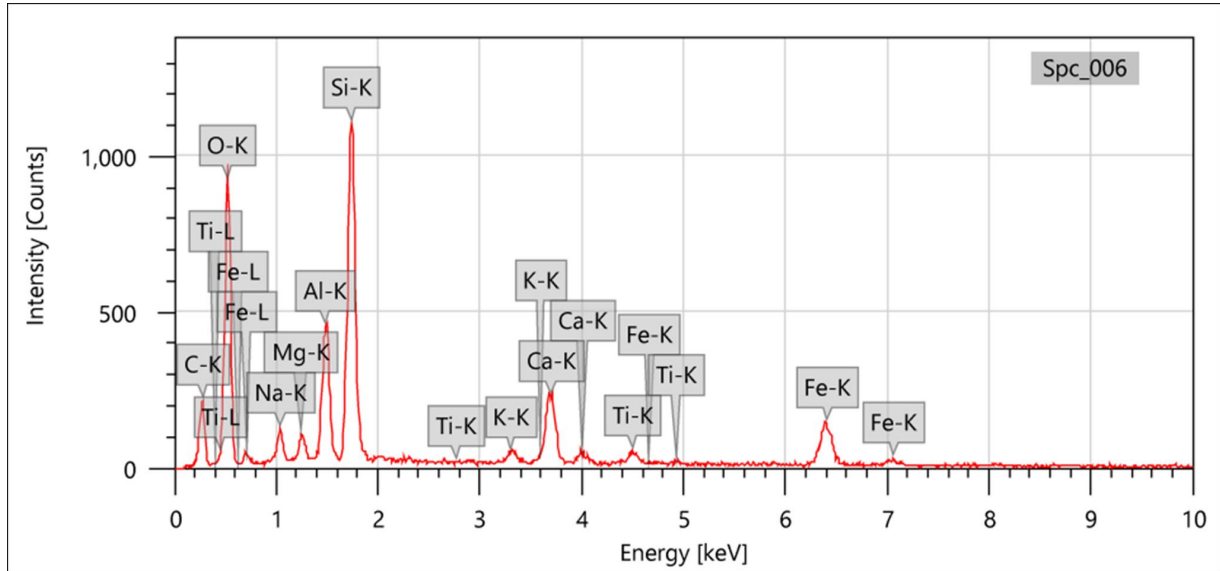


Figure 4.3 EDS spectrum of pumice particles.

Table 4.1 Elemental composition of pumice particles.

Element	Mass (%)	Atom (%)
C	18.91±0.23	28.70±0.35
O	44.92±0.47	51.17±0.53
Na	2.09±0.09	1.66±0.07
Mg	1.17±0.06	0.88±0.05
Al	5.27±0.11	3.56±0.07
Si	13.19±0.17	8.56±0.11
K	0.70±0.05	0.33±0.02
Ca	4.78±0.11	2.17±0.05
Ti	1.08±0.06	0.41±0.02
Fe	7.88±0.20	2.57±0.0

4.5. Density and void content

The open mold casting method is used to create the samples. Even distribution of particles in matrix is a difficult issue since it can lead to agglomeration and poor sample quality. Therefore, estimation of density and void content in the composites is generally considered as a measure of quality of samples. Density and void estimation of composites are presented in Table 4.2, The density of composites reduces as the pumice content increases. Density of PE-30 is noted to be lower than 1 g/cc signifying the light weightiness of composites. The experimental density of all composites is found to be lower than the theoretical density owing to minor traces of entrapped air known as void. Inclusion of voids is a very common phenomenon during fabrication of samples. However, higher voids in the composites lead to very inferior samples. Wherein it can be observed that the void content in the composites were very small and thereby can be ignored. Insignificant deviances observed in the void fractions confirm the consistency in the preparation of samples.

Table 4.2 Density and void fraction estimation.

Composition	Theoretical Density (kg/m ³)	Experimental Density (kg/m ³)	Void fraction (%)
E	1192	1192	0.00
PE-10	1124.2	1109	1.37
PE-20	1056.4	1042	1.38
PE-30	988.6	975	1.39

4.6. Tensile Test

4.6.1. Tensile Stress Strain Curves

Figure 4.4, Shows the results of tensile stress-strain curves for neat epoxy and pumice-loaded epoxy composites. From the graph, it was observed that all the specimen stress-strain profiles have similar trends up to 20 MPa stress. Sample PE-30 has a maximum tensile strength of 48 MPa. Sample E is in a linear elastic area followed by sudden brittle failure and has lower elongations than other pumice particulate-filled composites. Sample PE-10 is in the plastic zone, but its elongation is similar to that of sample E; this implies that the material was less

brittle and stiffer. Samples PE-20 and PE-30 are in the plastic zone and have higher elongation values; this means the materials were ductile and stiff. The higher stress were obtained from PE-20 closely followed by PE-30. The closeness of PE-20 and PE-30 indicates that after PE-30 when pumice powder increases the stress value also decreases. This is due to agglomeration of particles at higher filler content. Nevertheless, the effect of reinforcing naturally available pumice in matrix can be effectively realized through the stress strain curves. According to the results, the maximum stress value increased when compared to the unfilled composites.

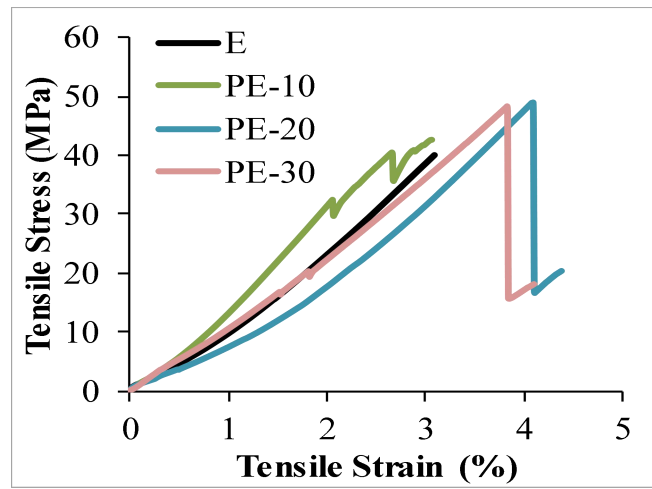


Figure 4.4 Representative tensile stress strain curves of pumice epoxy composites.

4.6.2. Tensile modulus

As shown in Figure 4.5 (a), an experimentally measured tensile modulus is presented. The tensile moduli of samples PE-10, PE-20, and PE-30 increased when compared with E sample. Sample PE-30 has a maximum tensile modulus of 1213 MPa, which is closely followed by samples PE-20 and PE-10, respectively. The tensile modulus of sample E is lower than that of all samples, which is 726 MPa. The results indicate that as the filler concentration increases, the tensile modulus of composites also increases. Rigid or harder filler reinforcement improves the stiffness of epoxy-based composites (Iyyadurai et al., 2023). In Figure 4.5 (b), the specific modulus of prepared samples was shown. All pumice-reinforced composites have a greater specific modulus compared with neat resin E. The strengths were raised linearly up

to sample PE-20 then after the rates were increased simultaneously until reaching sample PE-30.

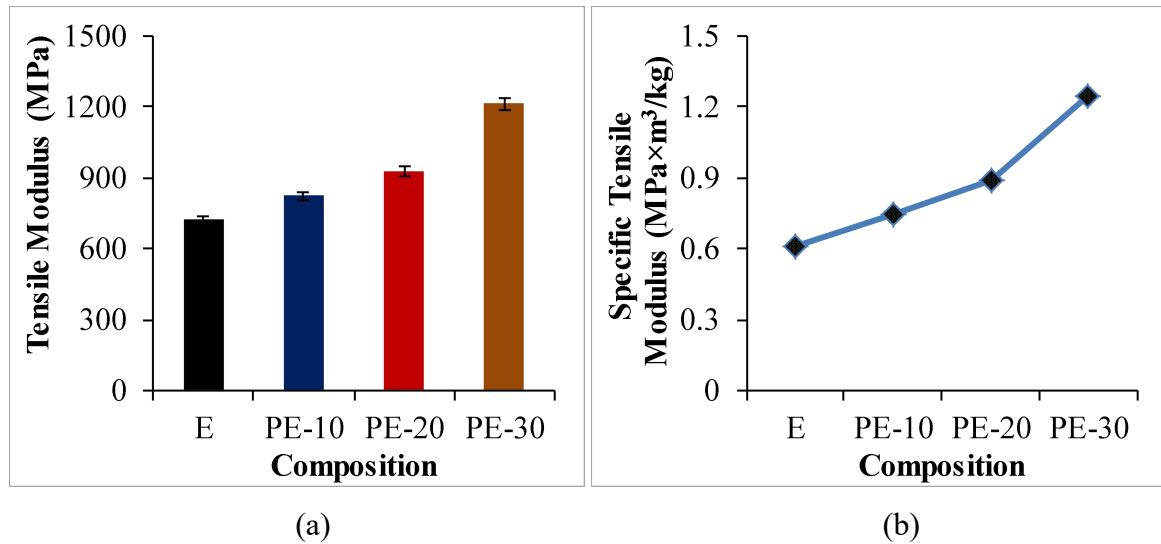


Figure 4.5 Experimentally measured tensile (a) modulus and (b) specific modulus.

4.6.3. Tensile strength

Figure 4.6 (a) shows the experimentally tested tensile strengths of the specimens. From the graph, all pumice powder-reinforced epoxy composites indicate higher tensile strengths than neat epoxy specimens. Sample PE-30 has the largest tensile strength, which was 48 MPa, and samples PE-20 and PE-10 increased closely with respect to sample PE-30. the pumice particles act as reinforcing elements, helping to distribute stresses more evenly throughout the material. As more pumice particles are added, the composite becomes better able to resist deformation and fracture under tensile loads.. Narrow epoxy has a lower tensile strength, which is 40 MPa. Figure 4.6 (b), shows the specific tensile strength of the composted material. The specific strengths were increased linearly from sample E to PE-30.

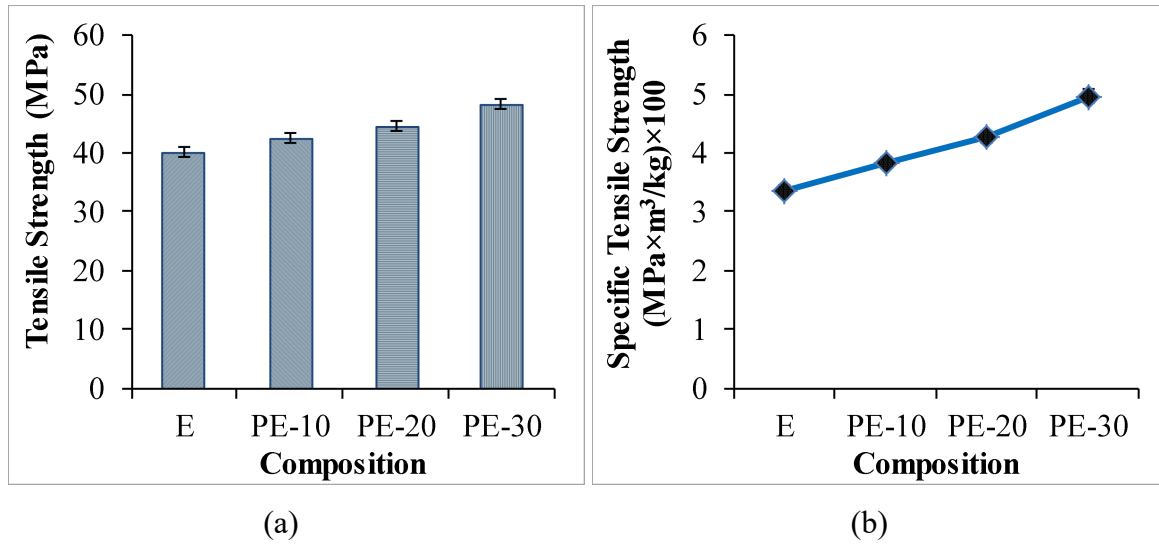


Figure 4.6 Experimentally measured tensile (a) strength and (b) specific strength.

4.7. Flexural Analysis

4.7.1. Flexural Stress Strain Curves

Figure 4.7, displays the stress-strain profiles for sample specimens put through flexural testing. When stress increases at a steady rate up until the point of fluctuations, all samples initially deform elastically. The non-linear flexural stress-strain curves are present in all composites. Sample EP-30 led to higher flexural strength, closely followed by samples PE-20 and PE-10, respectively. In Sample PE-30 and PE-20 results were much closed from each other this was due to agglomeration. In PE-30, when pumice powder content increases the epoxy content decreases so it becomes difficult to stir the mixture at this time agglomeration was created. This was the one cause to decrease the result beyond 30%. Sample E, had the lowest flexural stress-strain curves in the case that a sudden failure resulted in a drop in stress value. Due to the varying tensile and compressive strengths of composites, the failure of internal plies caused the stress variation in those samples. This is because specimens that were subjected to flexural loading were under combined load, with the top face being compressed and the bottom face being tensed (Yousif et al., 2012)

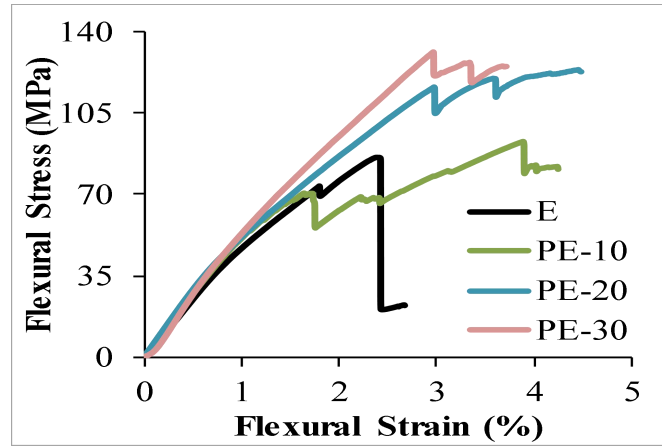


Figure 4.7 Representative flexural stress strain profiles.

4.7.2. Flexural modulus

Figure 4.8 (a), shows the influence of pumice powder composition on the flexural modulus of pumice powder/epoxy composites. The modulus of all composites shows an increasing trend as the volume of pumice powder content increases. The neat epoxy composites reveals lowest flexural modulus of 4887 MPa while the maximum flexural modulus of 5896 MPa was depicted by PE-30 composites. As compared to neat epoxy all the composites reveal an increase in the flexural modulus in the range of 10-21%. Figure 4.8 (b) shows specific modulus of flexural strength of composites. The specific modulus slope shows that adding pumice powder leads to a linear increase up to PE-30 composites and the increase is in the range of 18-47% as compared with neat epoxy (Iyyadurai et al., 2023).

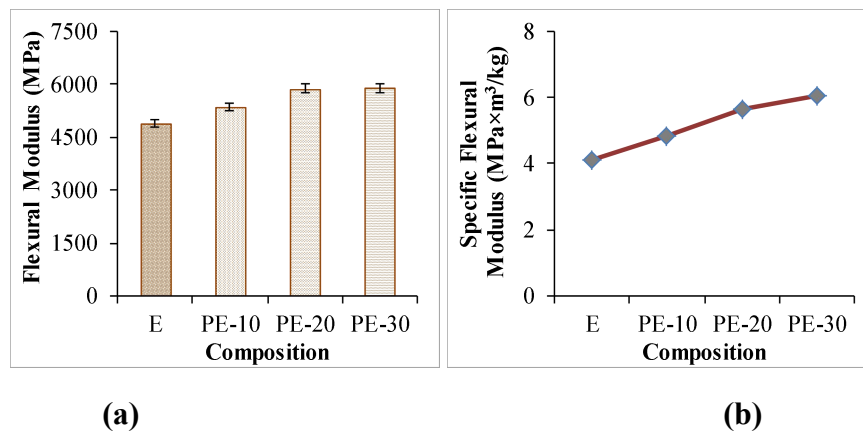


Figure 4.8 Experimentally measured flexural (a) modulus and (b) specific modulus.

4.7.3. Flexural strength

The flexural strength of pumice powder/epoxy composites was shown in Figure 4.9(a). In comparison to neat epoxy, the strength of PE-10, PE-20, and PE-30 is found to be higher. However, the pumice powder composition appears to have a considerable impact on flexural strength. The sample PE-10 and PE-20 the strengths are increased in high rate than others (Iyyadurai et al., 2023). Figure 4.9 (b) shows the specific flexural strength of composites. This result comes when the specific strength divided by their densities. PE-30 has a higher specific strength since PE-30 has lower density than others. So a numbers divided by small numbers rather than larger one they gives large number Then which was consistent with composite strength values.

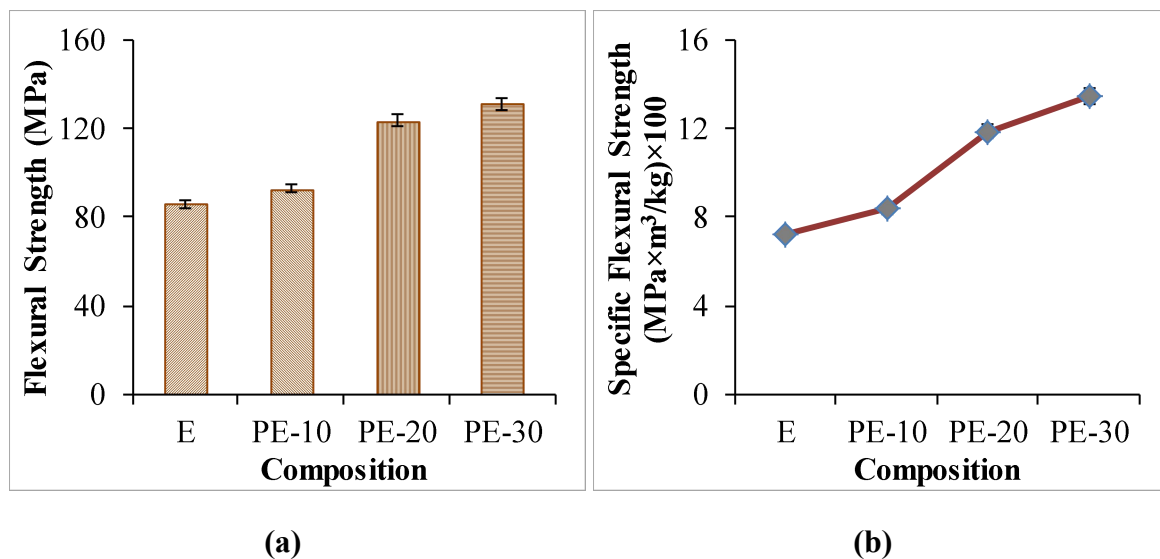


Figure 4.9 Experimentally measured flexural (a) strength and (b) specific strength.

4.8. Compressive Test

4.8.1. Compressive stress strain curves

Figure.10 depicts the compressive stress strain curves of all composites, including neat epoxy. Until the elastic area, all of the composites exhibit identical stress strain curves. However, neat epoxy specimen reveals sudden decrease in stress after attaining plasticity and finally fails at stress lower than all the composites. Decrease in stress and resulting failure of neat specimen is

mainly attributed to the inherent brittle behavior. Pumice reinforced composites show better response to loading post the elastic limit. It can be clearly observed that reinforcing pumice in epoxy matrix has strengthened the composites by effectively transferring the stress from epoxy matrix to pumice. Thereby, the composites depict increase in stress as the load increases. PE-10 and PE-20 composites reveal similar stress strain curves while PE-30 reveals higher stress compared with all other composites. Although PE-30 reveals higher stress, the response to loading decreases after a certain point. This may be attributed to agglomeration of particles at higher filler content. Nevertheless, the effect of reinforcing naturally available pumice in matrix can be effectively realized through the stress strain curves.

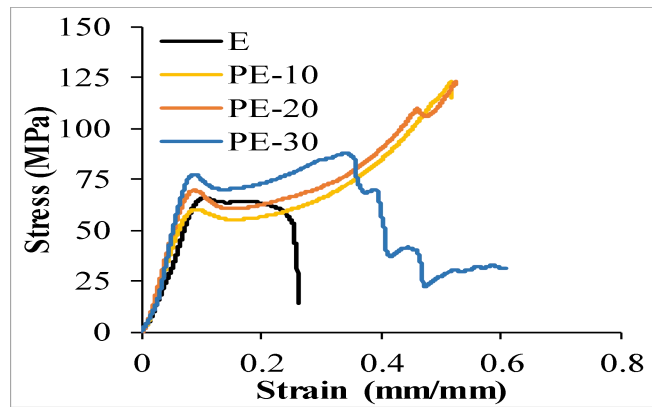


Figure 4.10 Representative compressive stress strain curves of pumice epoxy composites.

4.8.2. Compressive modulus

Figure 4.11(a) depicts the experimentally measured compressive modulus of pumice reinforced epoxy composites. Composite moduli are empirically determined using stress-strain profiles defined by the slope of the linear region. The modulus of all composites increases with increasing pumice filler amount and is found to be higher than neat epoxy. PE-30 composites registers the maximum modulus followed closely by PE-20 and PE-10, respectively, indicating that addition of pumice has contributed significantly to enhance the modulus of composites. However, addition of pumice beyond 10 vol.% does not yield more improvement and the increase is more or less even. Nevertheless, the increase in modulus of composites for all compositions as compared to neat epoxy is noted to be in the range of 54-58%. Addition of pumice in widely used matrix material has shown significant improvement mainly attributed to the presence of compounds such as SiO_2 , Al_2O_3 , and other iron oxides

(Sari, Setyawan, et al., 2022). These compounds are known to be one of the hardest and stiffer particles and thereby the presence of these particles in naturally available pumice readily enhances the modulus of composites. Specific modulus of the developed composites is shown in 4.11(b). When compared to neat resin, all pumice reinforced composite compositions have a greater specific modulus. Increase in specific modulus is noted to be in the range of 65-93% as compared with neat resin. As a result, utilizing pumice/epoxy composites can result in significant structural weight savings (Mills, 2007). Such weight savings are especially important in marine applications, where buoyancy is a key design criterion (Tagliavia et al., 2010).

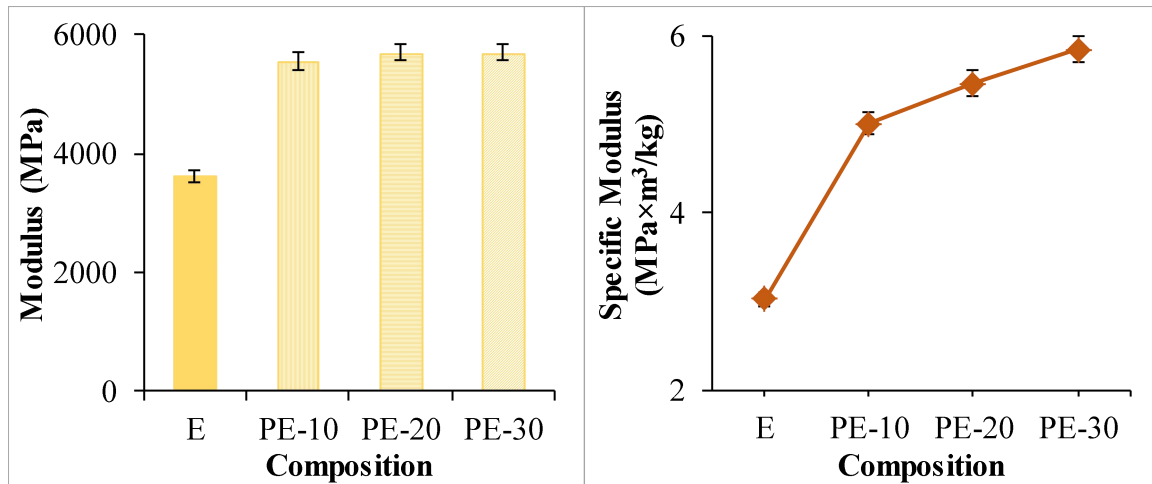


Figure 4.11 Experimentally measured compressive (a) modulus and (b) specific modulus.

4.8.3. Compressive strength

Experimentally measured compressive strength and specific strength of pumice reinforced epoxy composites are presented in Figure 12. As illustrated in Figure 12 a) strength of all the pumice/epoxy composites are noted to be higher than neat epoxy. Increase in strength of composites is noted to be in the range of 41-49% as compared to neat epoxy. Addition of pumice particles has significantly enhanced the strength of composites. Although it is well known that substituting the load bearing matrix with different fillers reduces composite strength, nonetheless, strong bonding between constituents and even dispersion of pumice particles aided in effectively transmitting the load from the matrix to the pumice particles. As a result, strength of composites increases with addition of pumice. However, the strength of

PE-30 is lower than that of PE-20 and PE-10, which might be attributed to attaining optimum filler dispersion. As a result, some particle agglomeration may have occurred, resulting in a drop in strength at PE-30. Nonetheless, the strength of PE-30 is much better than that of plain epoxy, and because of its low density, it is well suited for many buoyancy applications. Specific strength of pumice/epoxy composites are presented in 12 b). In-line with the observed compressive strength trends, the specific strengths of all compositions are higher when compared to neat epoxy. Higher specific strengths are preferred for structures demanding higher load bearing capacity.

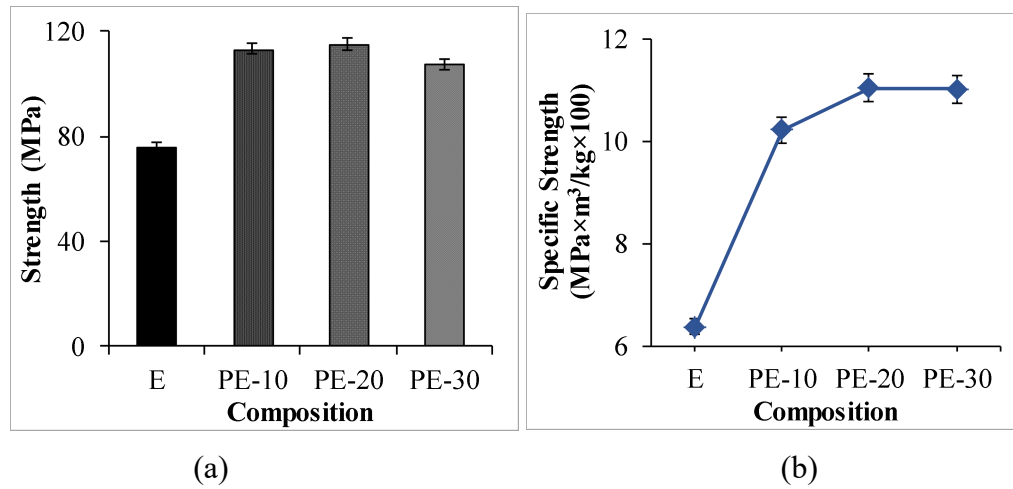


Figure 4.12 Experimentally measured compressive (a) strength and (b) specific strength.

4.9. Impact Test

An impact test is one type of distractive test that describes the amount of energy absorbed by a material during fracture. It describes the flexibility properties, or the ability to deform until fractured, by absorbing a specific amount of energy. Impact test can be used to determine the toughness and impact strength of the composite material (Vigneshwaran et al. 2021). For each composition, three specimens are tested and their average values are reported. Table 4.3 displays the average energy absorbed by specimens (S). Neat epoxy, PE-10, PE-20 and PE-30 composites reveal 6, 7, 8.75 and 10 J of impact energy absorbed. Results clearly demonstrate that all pumice reinforced composites reveal significantly increase in impact strength as compared to neat epoxy.

Table 4.3 Results of impact strength.

S. No.	Sample Name	Energy Absorbed in (J)
1	E	6
2	PE-10	7
3	PE-20	8.75
4	PE-30	10

4.10. Hardness Test

Hardness is considered one of the most important factors that govern the wear resistance of any material. The aim of the hardness test was to compute the resistance of material to plastic deformation. A higher value of hardness indicates that the material is harder, offering more resistance to penetration by other materials. The maximum hardness was 100.14 HV which was obtained by sample PE-30 as reported in Table 4.4. It has been firmly established that the oxides of silica, alumina, and iron are classified as hard materials, and their presence in pumice is indicative of the material's strength and hardness (Dagwa and Adama, 2018). This is why the hardness values were notably high in sample PE-30. And the bare minimum hardness was 72.28 HV, which is shown by neat epoxy. According to the findings, composites get harder as their pumice powder content rises.

Table 4.4 Tabular description hardness test result.

Code	Trial 1	Trial 2	Trial 3	Trial 4	Trial 5	Average
E	63.4	66.7	72.4	75.8	83.1	72.28
PE-10	78.1	74.8	77.3	73.5	78.7	76.48
PE-20	84.6	82.5	82	92.8	87.6	85.9
PE-30	101.6	99.9	93.1	100.3	105.8	100.14

Figure 4.13, shows the results of sample PE-30 and the other sample results are illustrated on appendix E.

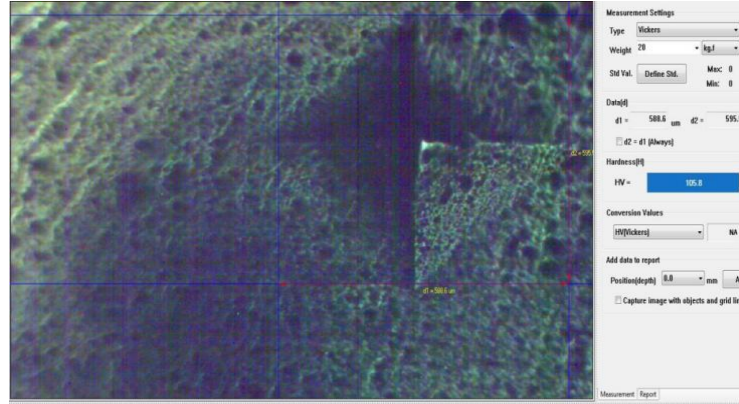


Figure 4.13 Hardness test results for sample PE-30.

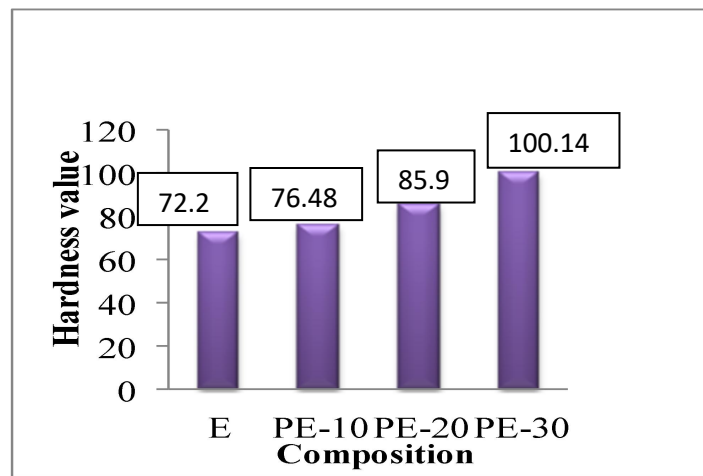


Figure 4.14 Hardness test results.

4.11. SEM

Scanning electron micrographs of specimens post compression test are depicted in Figure 4.15. Micrographs are captured on the fracture surfaces to understand the correlations between the property and structure. Figure 4.15(a) illustrates the micrograph of neat epoxy where it can be observed that striations marks similar to cabbage like structures can be observed indicating brittle mode of failure. Fracture features of PE-10, PE-20 and PE-30 can be seen in Figure 4.15(b), (c), and (d) respectively. Pumice particles can be observed uniformly distributed in the matrix; additionally it can be observed that striation marks visible in neat epoxy specimen cannot be observed in all the pumice reinforced composites indicating that pumice particles have effectively absorbed the compressive loading. Moreover, the surfaces are better

compacted revealing that maximum crushing of constituents also composites are integrated indicating good response.

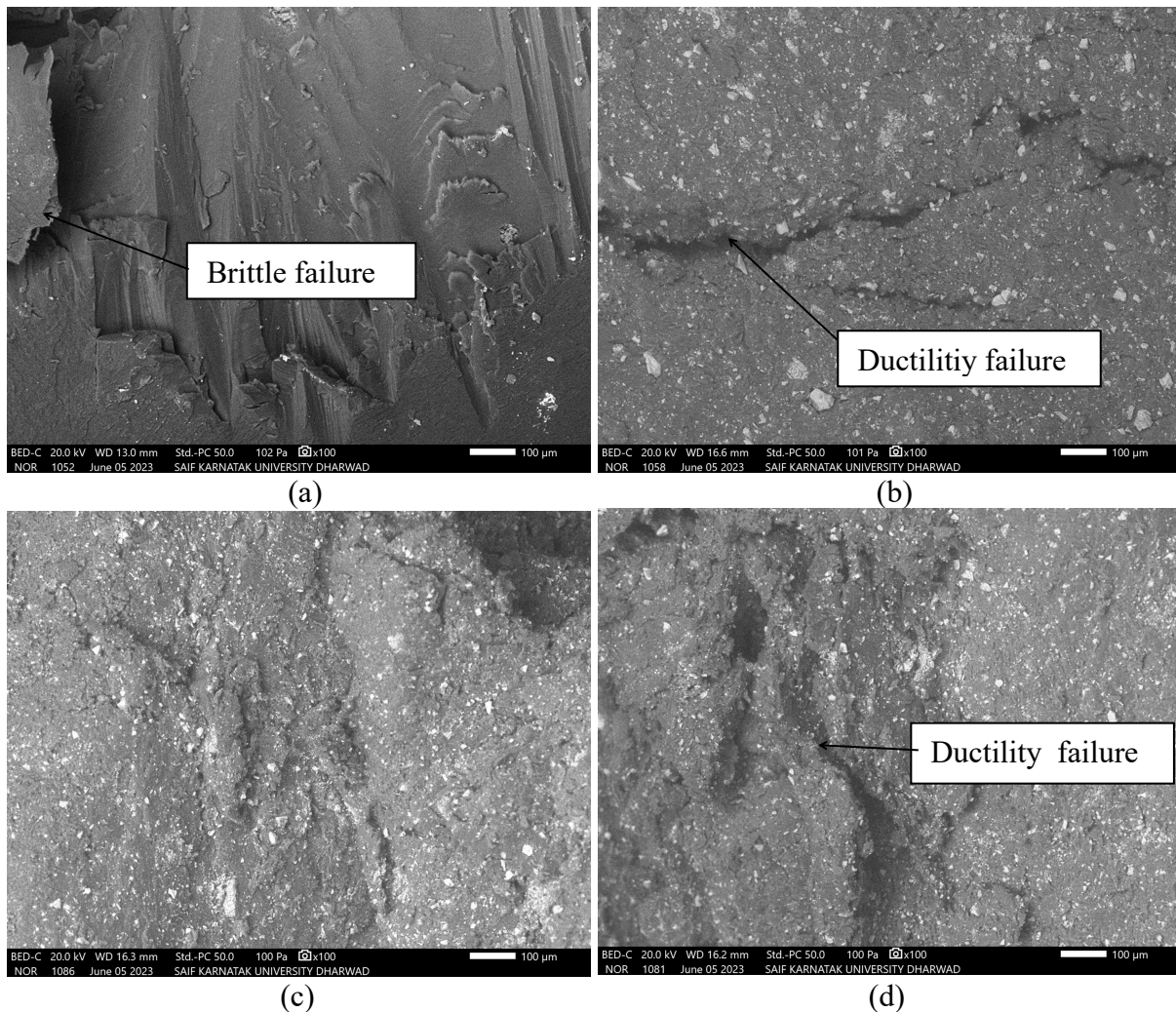


Figure 4.15 Scanning electron micrographs of (a) E0, (b) PE-10, (c) PE-20 and (d) PE-30.

4.12. Water absorption Test

It was essential to consider that moisture absorption might affect the physical and mechanical properties of composites in order to promote their continuous usage in high-performance applications. Prior to using this test for upcoming outside jobs, it was necessary to research it. Increased water absorption may affect the mechanical properties and dimensional stability of a composite. A water absorption test was performed to determine the volume of water absorbed under particular conditions. The sample weights used for the water absorption test in Table 4.5 as determined prior to submersion.

Table 4.5 Measured samples before immersion.

Sample	E	PE-10	PE-20	PE-30
Average Weight of sample before immersion (gm)	12.436	11.008	14.168	12.61

Table 4.6 Measured samples after immersion from day 1 to 8.

S. No.	Immersion Period in hr.								
	24	48	72	96	120	144	168	192	Avg.
E	0.23	0.349	0.422	0.473	0.528	0.564	0.63	0.6	0.475 %
PE-10	0.179	0.237	0.216	0.225	0.243	0.253	0.315	0.201	0.233%
PE-20	0.474	0.545	0.553	0.586	0.603	0.619	0.685	0.654	0.589%
PE-30	0.572	0.663	0.705	0.755	0.796	0.826	0.889	0.870	0.750%

Figure 4.16, shows the water absorption behaviour of pumice reinforced epoxy polymer composites for 8 days in distilled water conditions. Water absorption increases as the vol. % of pumice increases. It's also been noticed that moisture absorption increased at first then reduces daily, finally at the last day it becomes decline compared to the previous days. When compared 0, 10, 20, and 30 vol.%, water absorption in 10 vol.% pumice powder reinforced composite was lower. 0 and 20 vol.% were varied by very small amount and increased parallel but 30 vol.% pumice powder reinforced composites absorb water more compared to all samples.

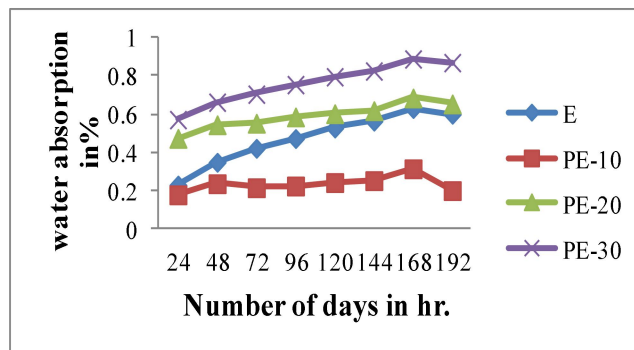


Figure 4.16 Water absorption graph.

4.13. Property Map

Figure 4.17 shows a property mapping of compressive modulus plotted against density of different lightweight composites and compared to the current work. Property maps have been used as a guideline for industry workers and researchers to choose certain compositions depending on anticipated applications. For comparative analysis, the current study results and data retrieved from previous studies are plotted with respect to sample density. For comparison, the compressive modulus results from the current study and data from earlier studies on lightweight composites, notably syntactic foam composites, are displayed with relation to sample density. The map clearly shows that hollow glass micro balloon and fly ash cenosphere reinforced composites have very low density as compared to the current study. However, compressive modulus of all the compositions (PE-10, PE-20 and PE-30) is substantial higher than compared with all existing studies signifying the use of pumice particles in developing promising composites. Furthermore, PE-30 has a density of 975 kg/m^3 less than water and can thus be termed lightweight. Figure 4.13, clearly shows the benefit of integrating pumice particles into a regularly used epoxy matrix. Despite the fact that the density of pumice is higher than that of hollow particles and fly ash cenosphere, the compressive properties of these composites cannot be overlooked. PE-30 composites have the highest modulus of all composites, indicating the suitability of using these particles in larger filler ratios for applications requiring higher modulus with low density. Finally, it can be concluded that the plentiful availability of pumice particles in certain nations can pave the way for efficient exploitation in the production of composites that meet the specific requirements of a wide range of applications.

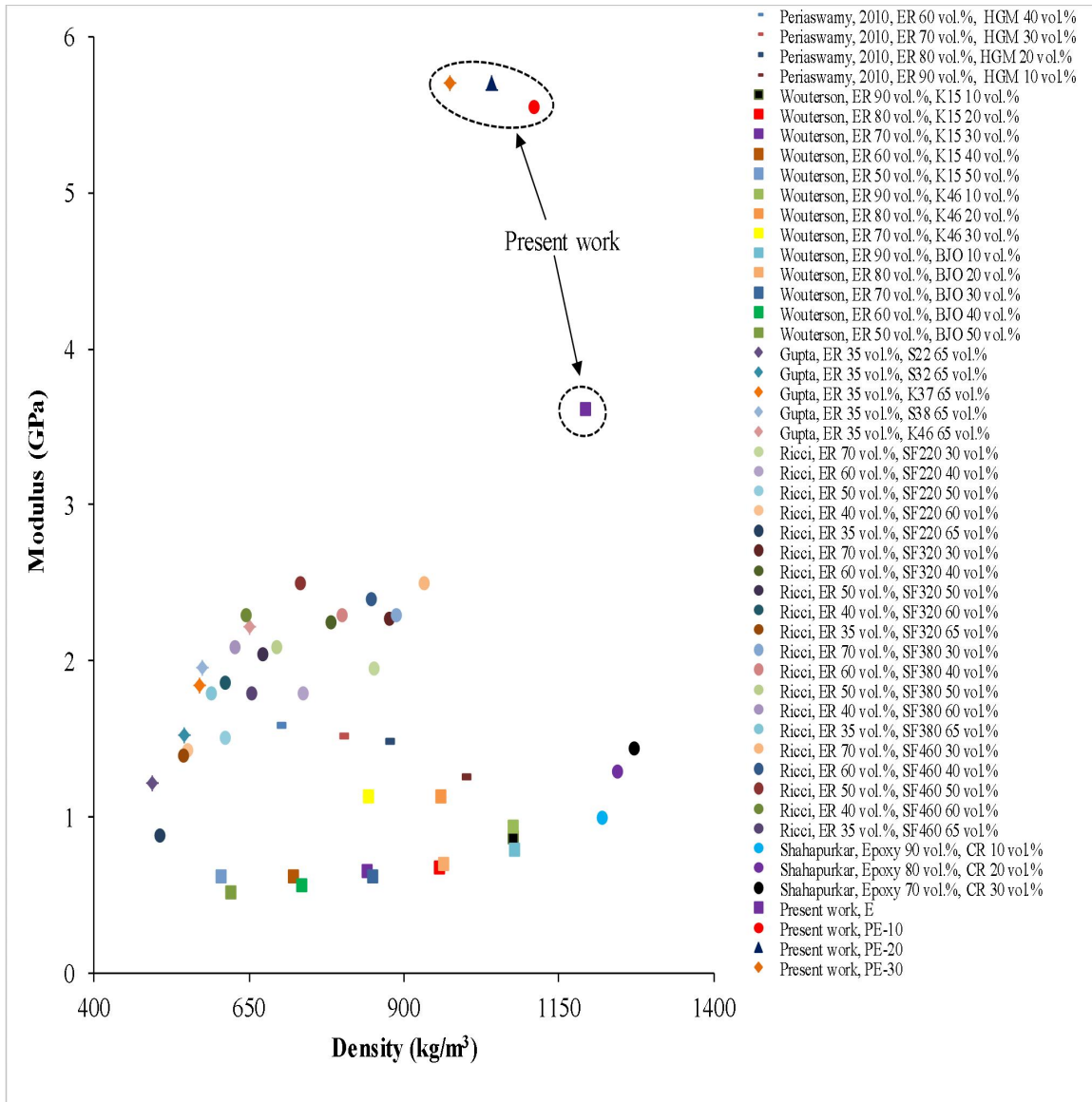


Figure 4.17 Mapping of compressive modulus and density of different lightweight epoxy composites (Alam and Al Riyami, 2018); (Shahapurkar, 2021) (Swetha and Kumar, 2011); (Dimchev et al., 2010; Gupta and Ricci, 2006); Periasamy et al.; (Mazzanti et al., 2019).(Tsuji et al., 2009)

[Note: ER stands for Epoxy resin, HGM represents Hollow glass micro-balloons, HSGM represents – Hollow soda glass micro-balloons, K15 and K46 stands for hollow microspheres, BJO – Hollow phenolic microspheres, S22, S32, S37 and S46 represents cenospheres with density of 220, 320, 370 and 380 kg/m³, K46 – Cenospheres with density of 460 kg/m³, UCIPC – Uncoated interpenetrating phase composite, SCIPC – Silane coated interpenetrating phase composite].

Syntactic foams are a type of composite material that consist of hollow spherical particles, such as micro-balloons or microspheres, embedded in a binder matrix. The spheres are arranged in an ordered manner, giving rise to the name 'syntactic', while the closed cellular structure of the material is referred to as 'foam'. These lightweight composites are made by adding hollow glass micro-balloons to the binder matrix (Sankaran et al., 2017). One of the most widespread applications of syntactic foams is in the marine industry because of their low moisture absorption and buoyancy. These materials can additionally be employed for deep water pipe insulation, spacecraft, boat hulls, underwater vehicles, and even soccer balls. As shown in Table 4.7, SF-46 indicates syntactic foam of hollow micro-balloons of glass which is mixed with 60% of epoxy and 40 % of hollow micro-balloons (Ullas et al., 2019).

Table 4.7 Comparison of pumice/epoxy composite and hallow micro-balloon/epoxy.

	Compressive Strength MPa	Tensile Strength MPa	Flexural strength MPa	Density Kg/m ³
PE-30	105	48	130	975
SF-46	80	22	28	700

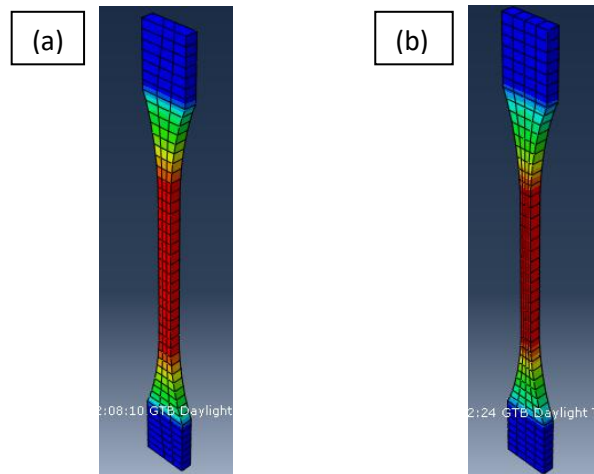


Figure 4.18 Comparison of tensile test in abaqus a) PE-30 and b) SF46

Deformation scale factor f for PE-30 was become 2.502 and for SF46 was 1.733.

CHAPTER FIVE

CONCLUSION AND RECOMMENDATION

Conclusion

In this research work, pumice particulate reinforced epoxy composites were investigated by varying the pumice content in 0, 10, 20 and 30 vol.%. Composites are prepared by open mold casting method. From the selection of process parameters to physical and mechanical characterization were discussed briefly. The physical and mechanical properties such as density, XRD, SEM-EDS, tensile, flexural, compression, impact, hardness and water absorption were evaluated through the experimentation. Based on the results following conclusion were made.

Epoxy with pumice particulates has high mechanical and water absorption properties compared with neat epoxy. Sample PE-30 has a maximum tensile modulus of 1213 MPa, which is closely followed by samples PE-20 and PE-10, respectively. The tensile modulus of sample E is lower than that of all samples, which is 726 MPa whereas the maximum tensile strength is shown by PE-30 at 48 MPa. Neat epoxy has a lower tensile strength, which is 40 MPa. Tensile result shows that as the pumice content increases the tensile modulus and strength also increases.

Flexural behaviour of pumice/epoxy composites reveals stress strain profiles of PE-20 and PE-30 specimens display higher stress. As compared to neat epoxy all the composites reveal an increase in the flexural modulus in the range of 10-21%. The specific modulus slope shows that adding pumice powder leads to a linear increase up to PE-30 composites and the increase is in the range of 18-47% as compared with neat epoxy. The flexural strength of PE-30 is found to be maximum while neat epoxy found minimum. Specific strength also shows similar trends to flexural strength. All flexural results show that when the pumice powder increases flexural values also increase.

In comparison to the neat epoxy sample, all of the composites experience greater strain in compression testing. The compression modulus (43%-61%) as well as the strength values (53%-55%) are higher than the neat epoxy sample. Results clearly demonstrate that all

pumice reinforced composites reveal significantly increase in impact strength as compared to neat epoxy. E, PE-10, PE-20 and PE-30 composites reveal 6, 7, 8 and 10 J of impact energy absorbed. The maximum hardness was 100.14 HV, as reported in Table 4.4, and it was produced by PE-30 composite sample. The bare minimum hardness was 72.28 HV, which is shown by neat epoxy. According to the findings, composites get harder as their pumice powder content rises. Minimum water absorption rate was obtained from sample PE -10 whereas the other sample also reveal good results but relatively the sample PE-10 results were better. SEM images of tested samples show that pumice particles are good in stopping deformation and increasing energy absorption capacity. The property map demonstrates the importance of pumice/epoxy composites in comparison to hollow particle reinforced composites and some functionally graded foams.

In general, most of results have revealed enhanced performance when the pumice powder content increases. Thus, the selection of the appropriate pumice powder content should be based on the specific application requirements of the composite material thereby concluding that EP-20 and EP-30 exhibit the best outcomes. Overall, the study highlights the potential of pumice powder particulate as a low-cost and effective reinforcement material in epoxy composites, and provides valuable insights for further research and development in this area. As a result of their toughness and lightness, these materials could be suggested for applications requiring buoyancy and wear resistance materials.

Recommendations

Considering the experimental investigation and results obtained the following recommendation is made:

- As the pumice powder content increase it becomes difficult to stir it by hand and the magnetic bar can't stir it due to its viscosity this leads to agglomeration. So it's better to use other machine
- Use the weight percentage of the pumice powder greater than 30% to know the decline points.

Future work

- Use additional fibers or powders to get a composite which will have unique properties for light weight application.
- Thermal and wear property of the composite can be studied for potential applications in aerospace and automotive industries.
- For water absorption test use more than eight days to know its result briefly.

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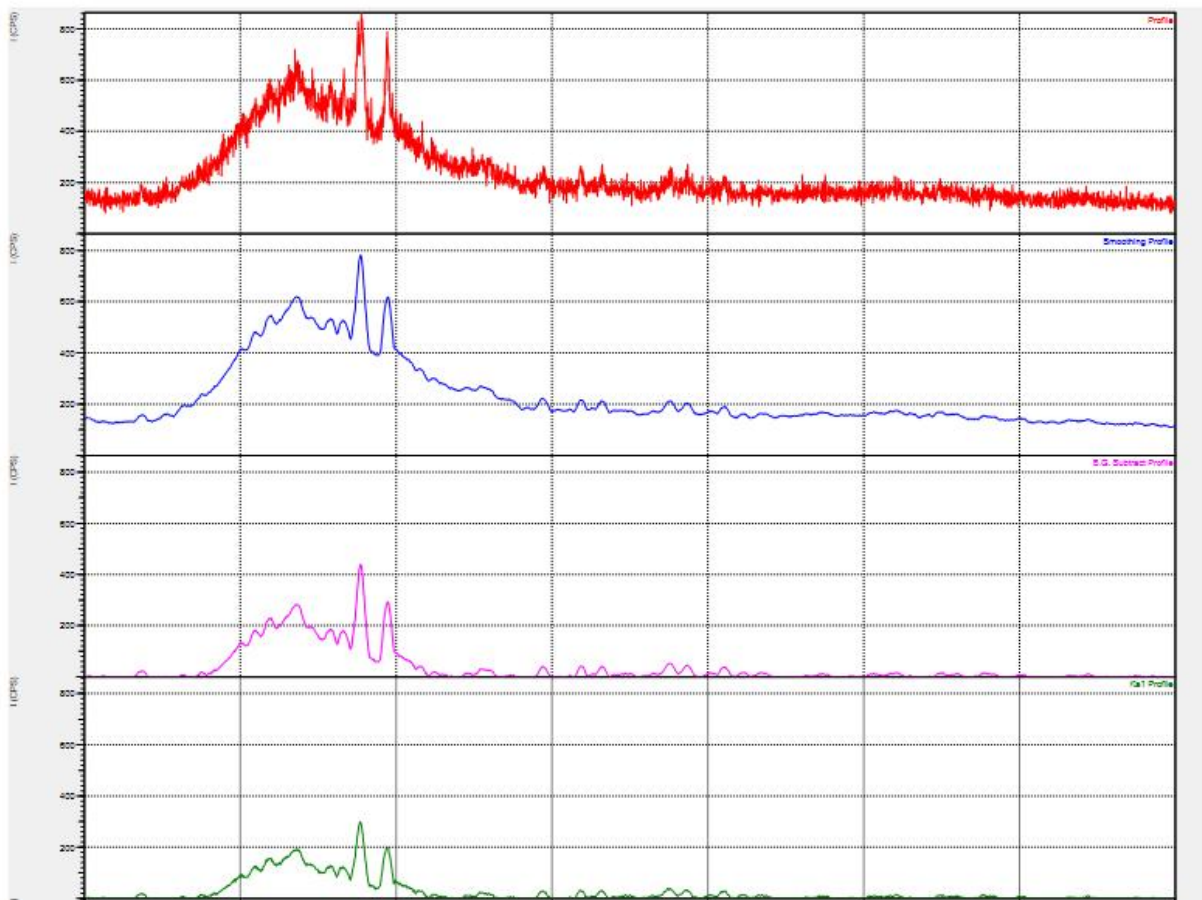
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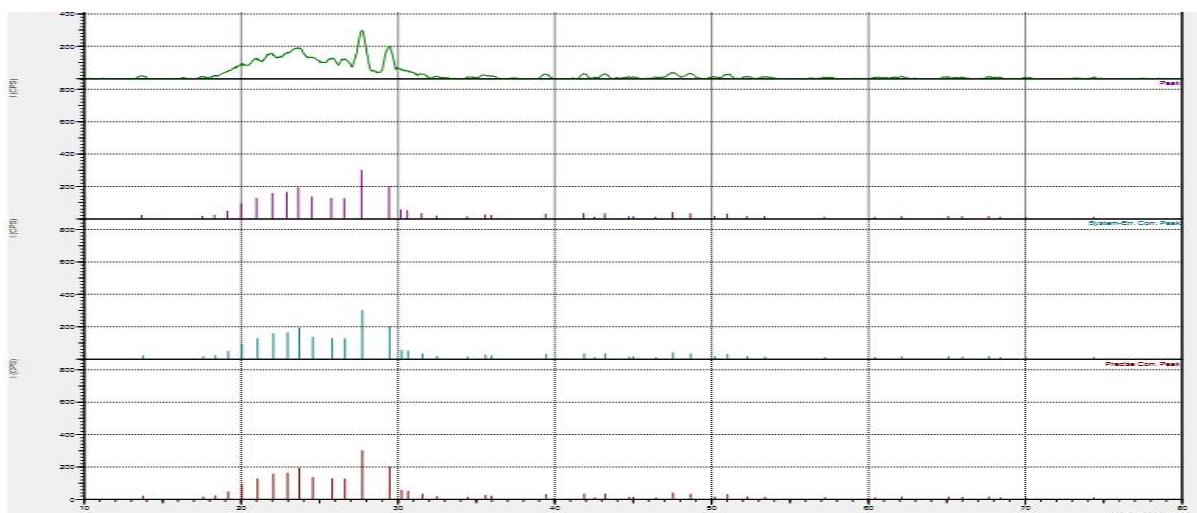
APPENDIXES

Appendix A

XRD Result

< Group: Standard Data: Pumica >





*** Basic Data Process ***

Group : Standard
Data : Pumica

Strongest 3 peaks

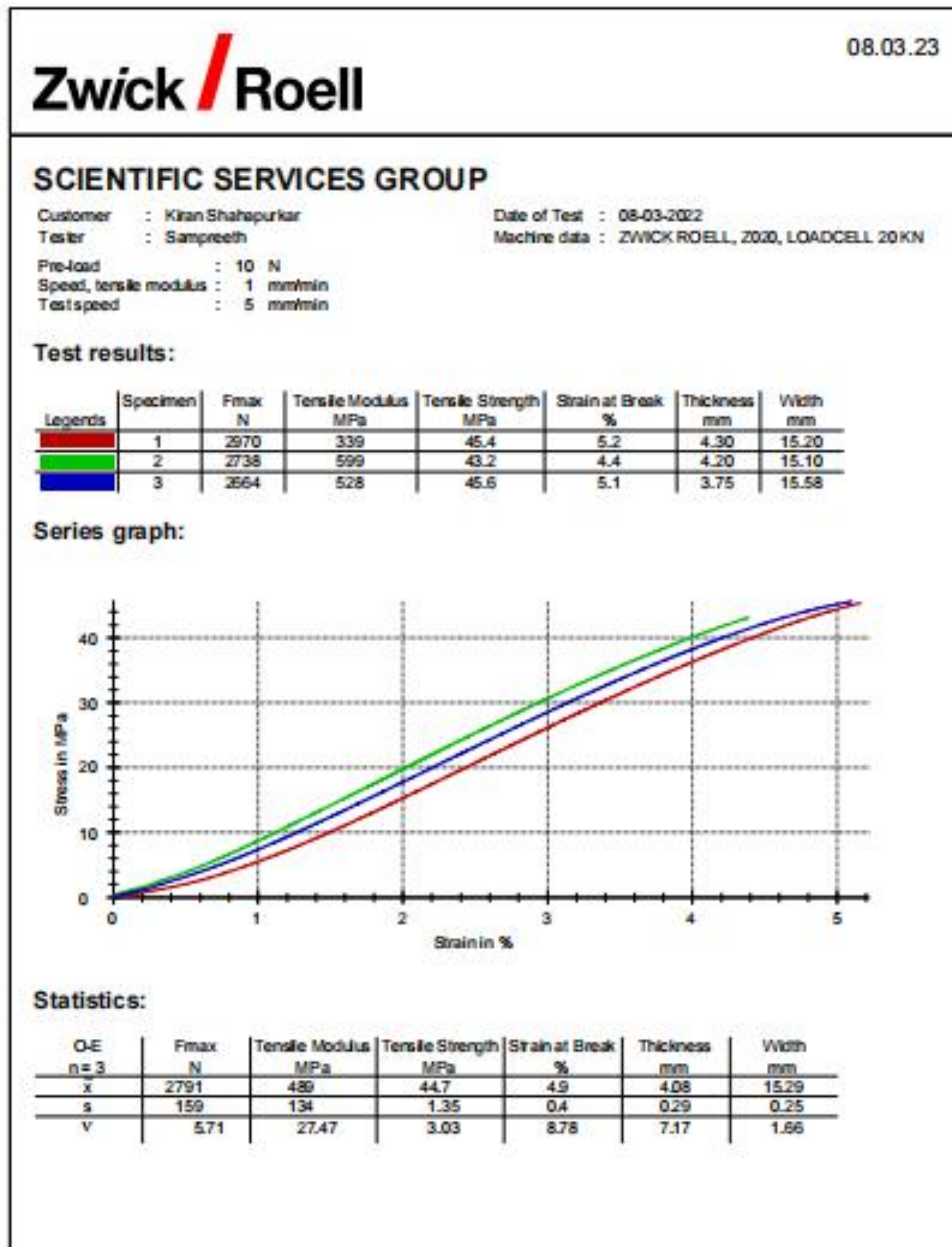
no.	peak no.	2Theta (deg)	d (Å)	I/I1	FWHM (deg)	Intensity (Counts)	Integrated Int (Counts)
1	13	27.7345	3.21396	100	0.69600	120	5662
2	14	29.4798	3.02753	66	0.66170	79	3252
3	9	23.7136	3.74903	64	0.00000	77	0

Peak Data List

peak no.	2Theta (deg)	d (Å)	I/I1	FWHM (deg)	Intensity (Counts)	Integrated Int (Counts)
1	13.7540	6.43320	7	0.51000	8	195
2	17.6022	5.03448	5	0.25330	6	162
3	18.3676	4.82638	8	0.08000	9	76
4	19.1853	4.62248	16	0.88000	19	774
5	20.0626	4.42228	31	1.52000	37	2778
6	21.0598	4.21508	43	0.00000	51	0
7	22.0573	4.02666	53	0.00000	63	0
8	22.9752	3.86783	54	0.00000	65	0
9	23.7136	3.74903	64	0.00000	77	0
10	24.5719	3.61999	45	0.00000	54	0
11	25.8096	3.44913	43	0.00000	51	0
12	26.6282	3.34492	42	0.00000	50	0
13	27.7345	3.21396	100	0.69600	120	5662
14	29.4798	3.02753	66	0.66170	79	3252
15	30.2430	2.95285	18	0.00000	22	0
16	30.6425	2.91525	17	1.22000	20	914
17	31.5614	2.83244	11	0.52000	13	348
18	32.5004	2.75272	6	0.20000	7	160
19	34.4584	2.60065	4	0.08000	5	44
20	35.5973	2.52001	8	0.64000	10	323

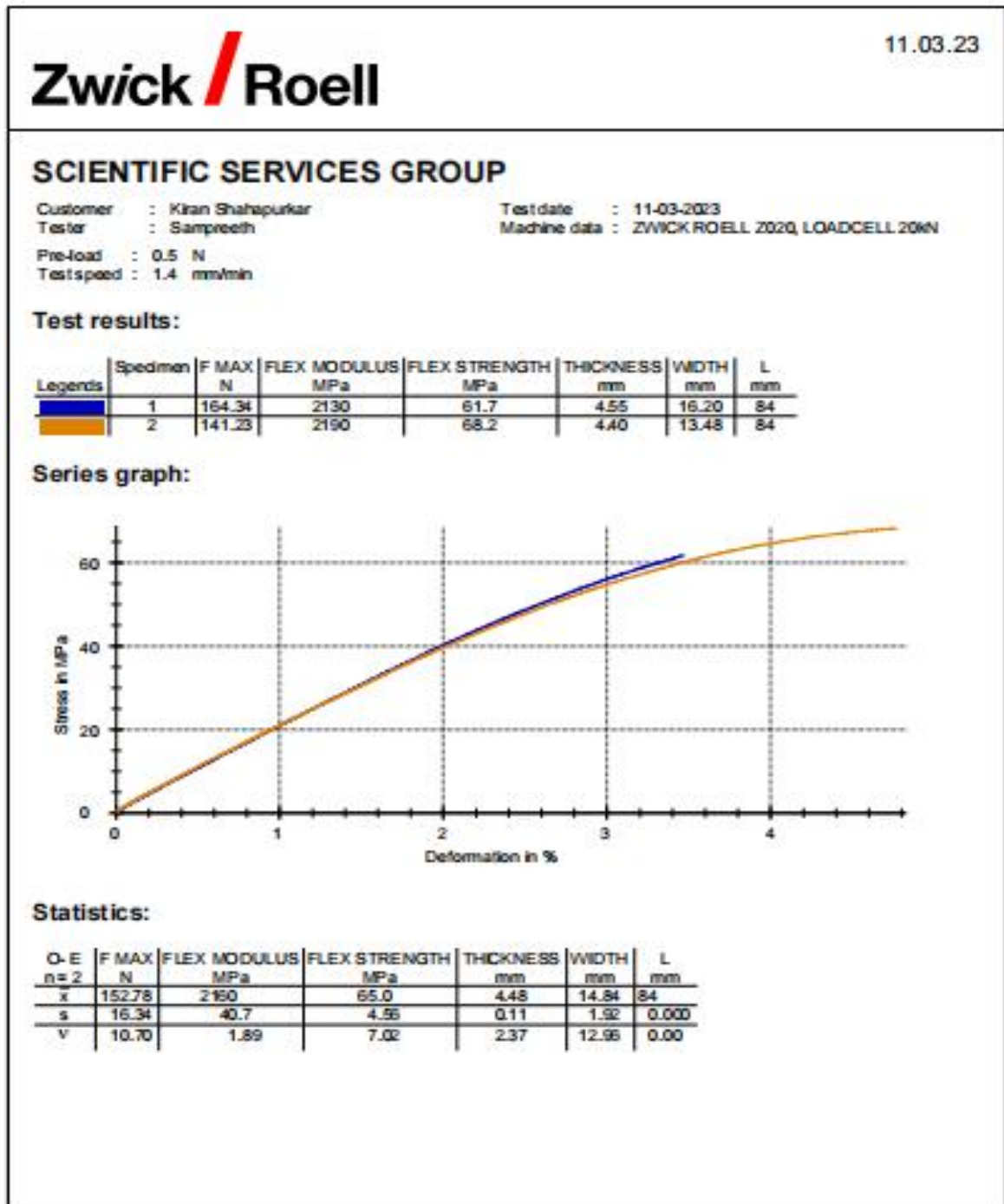
Appendix B

Tensile test



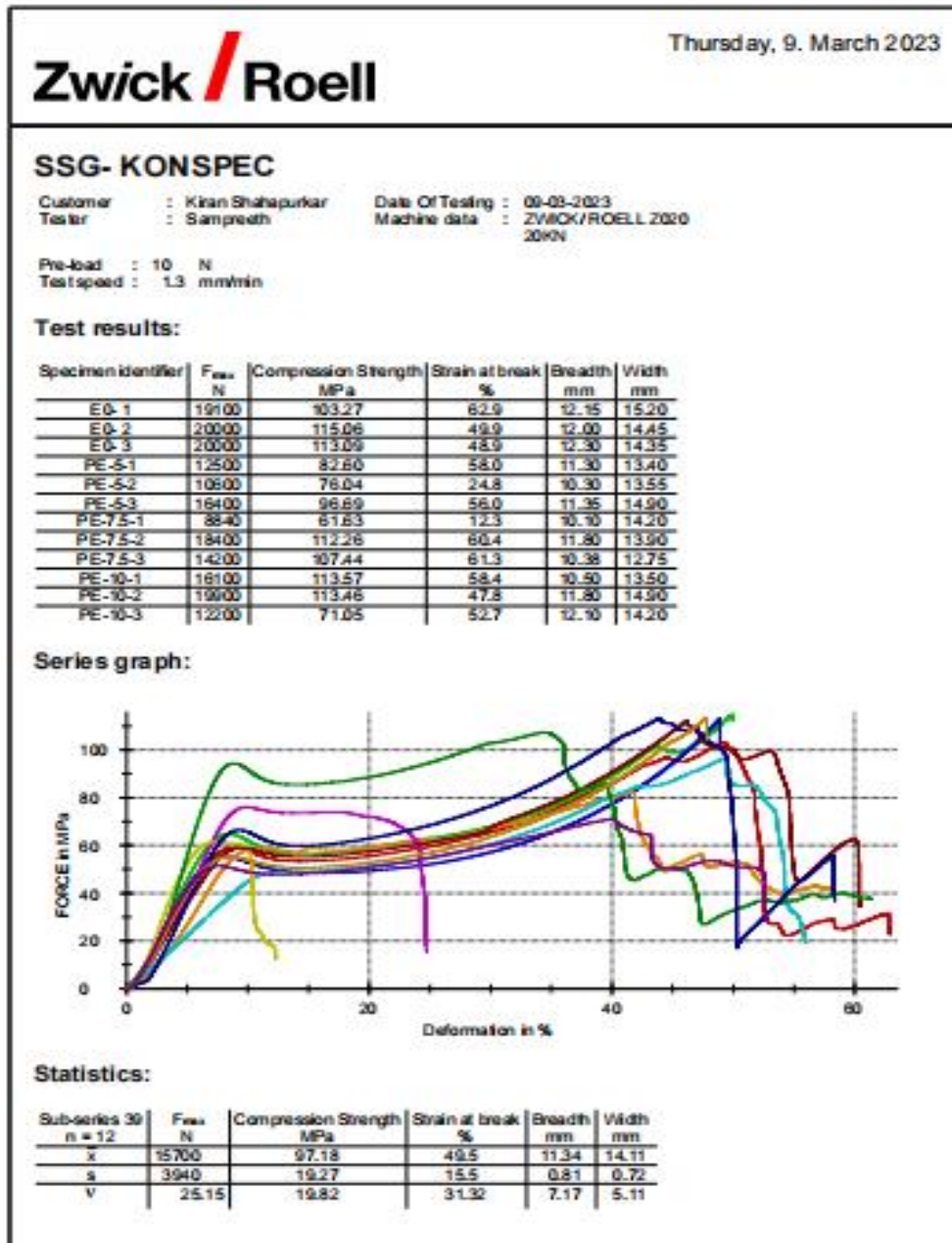
Appendix C

Flexural Strength



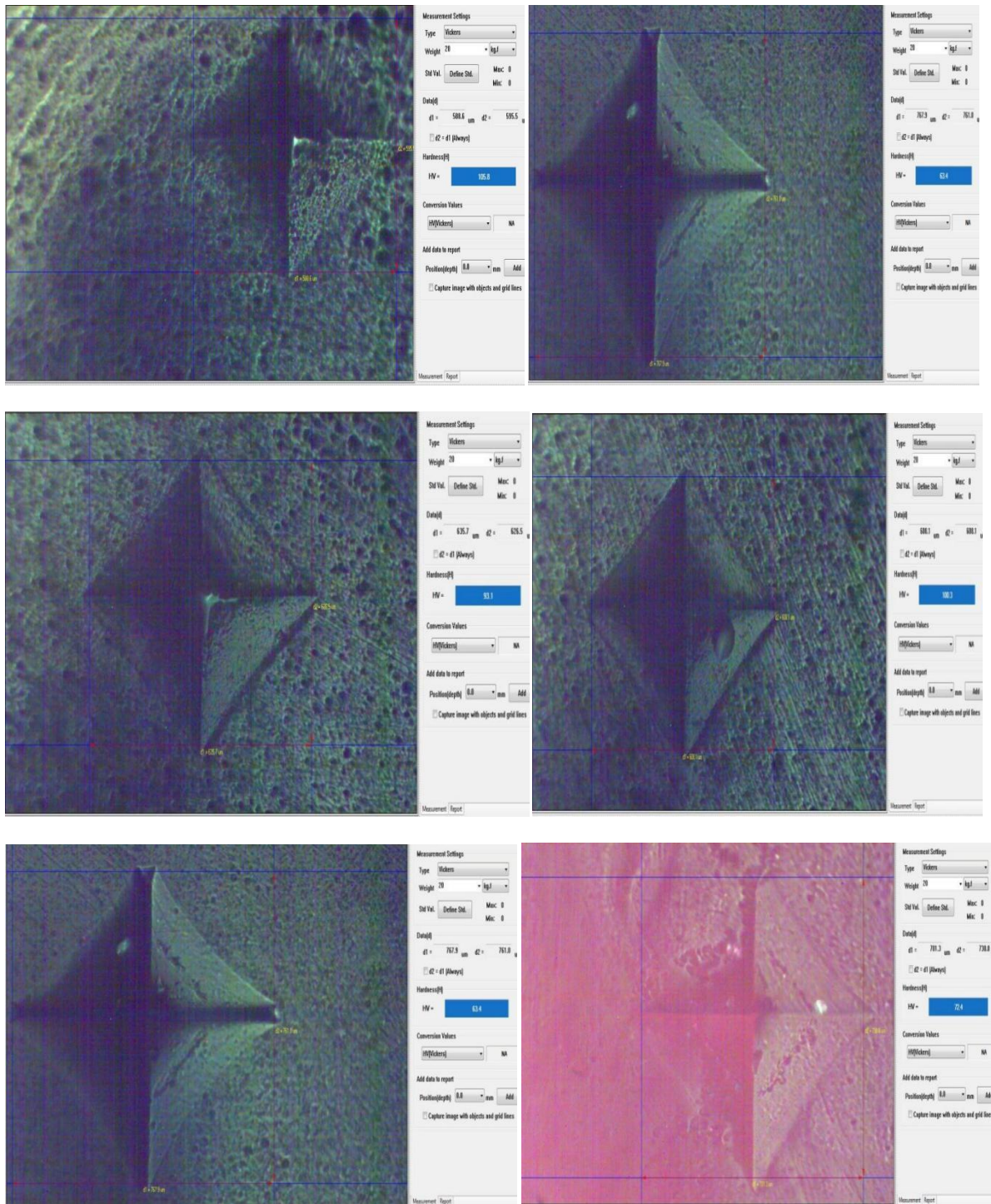
Appendix D

Compression test



Appendix E

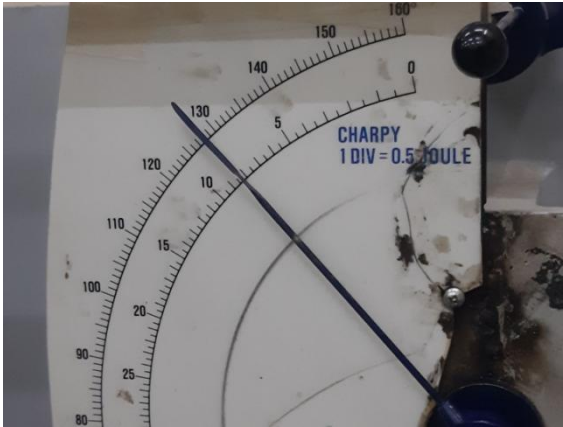
Hardness test



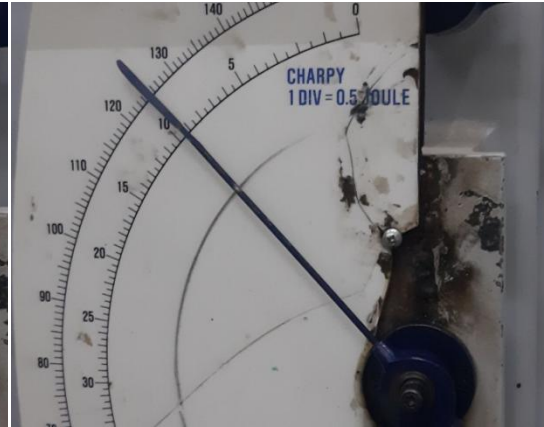
Appendix F

Impact test

For sample PE-20



8 jule, 128 degree



9.5 jule, 125degree

The average value becomes 8.75 jule