Analysis of Mechanical Properties of Coffee Husk Reinforced Unsaturated Polyester Composite with Nano Silicon Dioxide Particles

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Thesis Approval sheet

We, the undersigned members of te Board of Examiners of the final open defense by **Engdasew Gezahgn** have read and evaluated his thesis entitled **"Analysis of Mechanical Properties of Coffee Husk Reinforced Unsaturated Polyester Composite with Nano Silicon Dioxide Particles"** and examined the candidate. Therefore, this is to certify that the thesis has been accepted in partial fulfillment of the requirement of the Degree of Master of Science in Manufacturing Engineering.

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DECLARATION

I hereby declare that the work which is being presented in this thesis entitled "*Analysis of Mechanical Properties of Coffee Husk Reinforced Unsaturated Polyester Composite with Nano Silicon Dioxide Particles*" is my work. In partial fulfillment of the requirement for the award of the degree of Masters of Science in Manufacturing Engineering is my work carried out February 2020 to January 2021 under the supervision of Dr. BK Singh, a program of Mechanical Design and Manufacturing Engineering, Adama Science and Technology University, Adama, Ethiopia.

The material embodied in this thesis has not been submitted for any other degree or diploma award or assessment. All relevant sources of information used in this thesis have been properly acknowledged.

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Name of the Student	ID Number	Signature	Date

This is to certify that the above declaration made by the candidate is correct to the best of my knowledge and belief. This thesis has been submitted for examination with my approval.

Dr. BK Singh	_____	_____
Advisor	Signature	Date

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ABSTRACT

Nowadays, there is an increasing concern toward substituting pollutant and non-renewable synthetic fiber, and the scarce wood fibers with alternative lignocellulosic fibers that originate from agricultural residues to reinforce bio-composites. Therefore, many industries are seeking more eco-friendly materials that will decrease the level of environmental contamination and economic cost. The use of bio-based materials for the production of biocomposites is of interest due to their low cost, abundance, and the fact that they are renewable and biodegradable. Coffee is one of the most valuable primary products in the world trade and a central and popular part of Ethiopian culture. However, coffees productions generate a lot of coffee wastes and by-products, which, could be used for more applications. This project has aimed at evaluating the performance of coffee husk powder with nano-silica reinforced unsaturated polyester composites. To investigate the performance of this composite the coffee husk was crushed, treated with 5% NaOH and dried naturally. Both treated and untreated coffee husk powder was used to produce the composite and tested. The composites were prepared as (15/85, 20/80, 25/75) weight composition for both treated and untreated coffee husk powder, the nano-silica was used 20/80 %wt of treated coffee husk. The results showed that the treated coffee husk has higher performance than the untreated coffee husk composite, while the addition of nano-silica improved the impact strength on contrary it decreased the tensile strength. The result indicates that treated coffee husk fiber can be used in composites as filler to decrease material cost. On this basis using coffee husk fiber is advantageous in decreasing cost and decrease wastes that come from coffee production. Generally treated coffee husk fiber is advantageous reinforcement to increase mechanical properties and decrease cost of the product.

Keywords: *Polyester resin, Agro-waste, coffee husk, bio-composite, thermoset, composite*

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LIST OF ABBREVIATION AND ACRONYMS

ASTM-American Society for Testing and Material

UTM-Universal testing machine

APC-Agro-waste plastic composite

AR- Agro-residues

MEKP- Methyl ethyl ketone peroxide

PSI-Pound per square inch

CSS- Coffee silver skin

CHF- Coffee husk fiber

LLDPE-Linear low-density polyethylene

WF- Wood fiber

PLA- Polylactic acid

HDPE- High-density polyethylene

MAPE- Maleated polyethylene

CH- Coffee husk

PBAT-Polybutylene adipate terephthalate

CA- Contact angle

PP- Polypropylene

HV – Vickers hardness

SEM – Scanning electron microscopy

S - Sample

SCG- Spent coffee ground

CHAPTER ONE

1. INTRODUCTION

1.1 Background of the study

A composite material in its essence is a combination of two or more different materials that are mechanically bonded together. Each of the various components retains its identity in the composite and maintains its characteristic structure and properties. The composite materials, however, generally possess a combination of properties such as stiffness, strength, weight, high-temperature performance, corrosion resistance, hardness, and conductivity which are not possible with the individual components. Indeed, composites are produced when two or more materials are used together to give a combination of properties that cannot be achieved otherwise. Composite materials especially the fiber-reinforced polymer depends on properties of an individual component, several different phases, and size, shape, orientation, and distribution of fiber. Composites are multifunctional material systems that afford characteristics not obtainable from any distinct material (Wright & Askeland, 2014).

Most commonly polymers are used as a matrix. Overall, the mechanical properties of polymers are insufficient for many structural purposes. Particularly their strength and stiffness are near to the ground compared to metals and ceramics. These difficulties are surmounting by reinforcing other materials with polymers. Secondly, the processing of polymer matrix composites doesn't require high pressure and high temperature. The, equipment required for manufacturing polymer matrix composites are simpler. For this reason, polymer matrix composites developed quickly and almost immediately became trendy for structural applications (Arpitha et al., 2014).

Most of the commercially available fiber-reinforced polymer composites that present a wide variety of applications (e.g. automotive, construction, marine, aeronautics, and home appliances) are based on petroleum-based polymers reinforced with synthetic fibers (Tarazona et al., 2017). Natural fibers are the best substitute that can help solve ecological and environmental problems as they substitute glass fiber, carbon fiber, or nylon fiber as reinforcing materials in polymeric matrices to improve mechanical properties (Ochoa et al., 2017). Natural fibers are getting increasing importance as reinforcing materials in composites due to some advantages they

provide such as cheap, lightweight, not poisonous, better mechanical possessions, and a clear lower environmental impact (García-García et al., 2015a). Presently steel and plastic are widely used in the manufacture of various products such as doors, false ceilings, toys, boxes for agricultural use, rims, and mobile panels. However, it is evident that both these materials are neither economical nor eco-friendly, and their presence poses serious impacts for the user and the environment. Natural fibers have found their usage in composite applications over the past few decades (Kaushik et al., 2019).

Substantial pressure on standing forest resources as a result of growing demand in the forest industry due to the growing population and new application area has encouraged investigators to find solutions. One of the solutions is to employ other alternatives including agricultural residues that are excellent materials to substitute wood and synthetic fibers because they are abundant, widespread, and easily accessible; besides, utilization of agricultural residues advantages is beneficial economically, environmentally, and technologically (Mohseni Tabar et al., 2015).

Nowadays polymer composite with natural fiber is mainly used for different engineering application, predominantly for the development of the interior panel of automobiles. Common fiber-reinforced composites are composed of fibers and a matrix. Fibers are the reinforcement and the main source of strength whereas matrix glues all the fibers together in shape and transfers stress between the reinforcing fibers. Sometimes, filler might be added to smooth the manufacturing process, impact special properties to the composites, and/or decrease the product cost (Arpitha et al., 2014).

Natural fibers reinforced polymer composites highly fascinated designers by their several advantages such as low weight, low cost, high availability, easy productivity, they are friendly to environment and high specific mechanical performance. Natural fibers confirm advanced mechanical properties like stiffness, flexibility, and modulus composite to glass fibers (Y. Li et al., 2008). Their availability, renewability, low density, and price, as well as acceptable mechanical properties, make natural fibers more attractive ecological alternatives to glass, carbon, and manmade fibers used for the manufacturing of composites (Alawar et al., 2009).

Natural fiber composites can be very cost-effective for the following applications: Building and construction industry: panels for partition and false ceiling, partition boards, wall, floor, window

and door frames, roof tiles, mobile or pre-fabricated buildings which can be used in times of natural calamities such as floods, cyclones, earthquakes, etc.Furniture: chair, table, shower, bath units, etc. Electrical appliances, Transportation: automobile and railway coach interior, boat, etc.

Impressive enhancement of material properties achieved with the inclusion of submicron-size fillers in elastomers has stimulated active research. Clay Nano-composites, especially nano-clay/polymer composites, exhibit dramatic increases in modulus, strength, barrier properties, flammability resistance, and heat resistance compared with conventional composites(Lei et al., 2007). Enhancing the physical properties of composites could expand their acceptance in load-bearing structural applications(Faruk & Matuana, 2008).

Proper engagement of the nanoparticles in composite systems depends strongly on the ability to homogeneously disperse them throughout the matrix without destroying their integrity. The properties of the matrix, the distribution, and properties of the filler as well as the nature of their interface control the behavior of typical composite material. most of the scientific work has been focusing on the synthesis of polymer nanocomposites and the study of their physical and mechanical properties. The primary aim is to improve the deficiencies of existing composites or to extend their functional limits(Njuguna et al., 2011). Nanosilica has high surface area due to its nanosize, which is capable to seal the fibre’s pores. nanosilica in order to reduce the water uptake of the resulting composite (Bajuri et al., 2018).

Agro wastes

The agro waste materials fulfill the low cost materials that can improve profit margins, and improve recycling of obtainable agro wastes that can reduce environmentalpollution (Sinha et al., 2019). Agro-wastes plastic composites (APCs) produced from agro-residues (AR) and plastics resins are greatly popular. It is used widely in residential construction, windows, doors, automotive, marine, and aeronautics industries (H. Wu et al., 2016). Coffee, grown in about 80 countries, (Campos-Vega et al., 2015a) which is one of the most consumed beverages and the second most traded product in the world after petroleum, so that the coffee industry generates a lot of waste (García-García et al., 2015a). The coffee husks, peel, and pulp, comprising nearly 45% of the cherry, are the main byproducts of the coffee agro-industry and can be a valuable material for several purposes, including caffeine and polyphenols extraction. Coffee husks and

skins are traded as crops and livestock products with export and import (Campos-Vega et al., 2015a). Production of coffee is the industrial process of converting the raw fruit of the coffee plant into the finished coffee. These processes generate a lot of solid wastes (by-products) and wastewater. An estimated 3.5 billion cups of coffee are consumed worldwide every day (Blinová et al., 2017).

Coffee wastes

Coffee is one of the most important agricultural commodities in the world. Coffee contains over 1500 chemical substances, 850 volatile and 700 soluble. When coffee is extracted in water, most of the hydrophobic compounds, including oils, lipids, triglycerides, and fatty acids remain in the grounds, as do insoluble carbohydrates like cellulose and various indigestible sugars. Production of coffee is the industrial process of converting the raw fruit of the coffee plant into the finished coffee. These processes generate a lot of solid wastes (by-products) and waste water. The main solid by-products from cultivation and preparation of coffee are spent coffee grounds, by-products of coffee fruit (coffee cherry) and bean processing (coffee husks, peel, pulp). Huge amount of contaminated waters are also produced in several washing steps, with a high carbon load, and thus with high impact on the environment (Blinová et al., 2017)

1000 kg of fresh berry gives about 400 kg of wet waste pulp. The coffee husk contains caffeine, tannins, polyphenols, and organic solid residues. More than 50% of the harvest coffee berry is green and not used for commercialization. These green berries are discarded as a by-product during processing (Ochoa et al., 2017). Coffee waste has toxic nature and thus has not been utilized beneficially. For this reason, efforts have been made to develop methods for coffee waste treatment and management, also its utilization as a raw material to produce feeds, beverages, vinegar, biogas, caffeine, pectin, pectic enzymes, protein, and compost (Blinová et al., 2017). In Ethiopia, 192000 metric tons of coffee husk is cast adrift as a byproduct per year (Sime et al., 2017). Hulling the dry parchment coffee leads to what is called hulls. Exocarp plus mesocarp and endocarp represent 60% of the dried fruitmass. Therefore, a considerable volume of husk and hulls are produced when coffee fruit processed (Bekalo & Reinhardt, 2010).

As a cellulose-rich material, coffee hull can be considered a potential source of reinforcing phase in thermoplastic matrix composites (Wang et al., 2019). The mechanical, and thermal properties

of coffee silver skin with loading rate (10, 20, 30 wt%) withbio-plyethylene was studied, and was found the elastic modulus,and strain at maximum stress was improved (Dominici et al., 2019). The researchers compared nano silicon dioxide and coffee husk as organic filler in linear low density polyethylene; morphological and mechanical properties were studied. The addition of polymer matrix improved the tensile modulus and tensile strength. Some relevant modifications of melting temperature and crystallinity degree for coffee husk composites were observed, but not observed for inorganic fillers composites.The properties of the composites prepared with coffee husk were comparable to those obtained with inorganic fillers, demonstrating that this coffee residue can be used as filler for obtaining composites for many possible applications (Yepes et al., 2017). These researchers studied coffee husk fiber as cost effective thermoplastic bio-composite; their experimental results showed that increasing the coffee husk in HDPE matrix improved modulus and thermal properties of composites, but resulted in poor water resistance (Huang et al., 2018). Another researchers had fabricated polybutylene adipate terephthalate composites with different concentration of coffee husk loading using silane treatment. The result showed that silane treated coffee husk more hydropobic than raw coffee husk, improved its compatibility with PBAT. The tensile strength and Young's moduli improved, as well the cost of the composite decreased by 32% (Lule & Kim, 2021).

1.2 Statement of the problem

During the process producing green coffee, the husk, pulp, parchment, and mucilage are removed, and they account for 12%, 29%, 12%, and 5%, respectively, of the dry weight of the cherry (Zarrinbakhsh et al., 2016). During coffee processing, the coffee husk is disposed to the environment which is not consumed by animals and it also contaminates the land. Since the coffee price is increasing in the world the production also increases, this increase the waste proportionally (Campos-Vega et al., 2015a). Composite manufacturers in Ethiopia, use the imported synthetic fiber reinforcers which are costly and not biodegradable.

Removing the coffee waste is a pollutant since it goes to burning or landfill. Water pollution is caused by the presence of organic, inorganic, biological, and physical substances in the water that tends to degrade its quality (Blinová et al., 2017). These substances affect the lives of animals and the water becomes not potable. The coffee shell waste is not well studied to use it for productive purpose. As a result, this byproduct is not utilized at all in Ethiopia. Finally

enhancing the property of waste coffee is crucial in terms of producing a good quality product at a lower cost also it keeps our environment clean by reusing the coffee husk as composite reinforcement. So by using coffee husk as composite reinforcement for unsaturated polyester resin the waste can be eliminated or decreased from coffee wastes.

1.3 Objectives

1.3.1 General objectives

The general objective of this study is to study the mechanical properties of the composite made from the coffee husk and polyester resin, by adding Nano-silicon-dioxide and using NaOH chemical treatment of the coffee husk.

1.3.2 Specific objectives

- ✓ To study the physical properties of coffee husk powder and polyester resin composite
- ✓ To fabricate the coffee husk and polyester resin composite;
- ✓ To conduct tensile strength test, impact test, hardness test, and microstructure test;
- ✓ To investigate the effect of the Nano silicon dioxide on the mechanical properties of the coffee husk polyester composite and the effect of chemical treatment on mechanical properties of the composite, and to recommend the best rsult.

1.4 Significance of the study

The outcome of this study has significance for composite, and coffee manufacturers, and waste management of coffee processing firms, for using the coffee shell, which may reduce cost and better waste management. The result of this project can be a useful reference for individuals, researchers, and manufacturers who are engaged in developing new green composites. The country and the society may benefit from the cleanness of the environment at the same time using the waste for useful products. The society benifited from the removal of the coffee husk which is waste. This may recognize coffee tree growers to be beneficial by selling coffee shells for composite production purposes.

1.5 Scope and limitation of the study

This study was delimited to the mechanical property testing of coffee husk and unsaturated polyester resin composite with nano silicon dioxide as a filler and NaOH as alkaline treatment. During conducting this research the major limitation was the laboratories were closed as well materials was not available due to the outbreak of COVID-19 pandemic. So the research obliged to postponed and using optional laboratories with costly payment made possible for the finalization of this research.

1.6 Organization of the study

The thesis will be organized with the following chapters:

Chapter One: Introduces the background of natural fiber, agro-waste composite materials, and this project's objectives, Problem Statement, Scope, and limitations.

Chapter Two: Reviewed all relevant research papers regarding natural fiber, agro-waste composite materials, ranging from polymer types, fiber types, and composite's chemical treatment, mechanical properties.

Chapter Three: this chapter explains every procedure used during the processing of the composite, sample preparation, and testing.

Chapter four: this chapter explains the results obtained from testing. It includes characterization of a composite including tensile strength, impact, and water absorption.

Chapter five: this chapter is dedicated to the conclusion and future works of this research.

CHAPTER TWO

2. LITERATURE REVIEW

The literature review consists the past works a bout biocomposites, natural fibers, nano materials, treatment methods and processing techniques. It also includes why the researcher chooses the materials used in the research work.

2.1 Composite materials

A composite material is a material system composed of a mixture or combination of two or more micro or macro constituents that differ in form and chemical composition and are essentially insoluble in each other (Smith, 1986). Composites are generally used because they have desirable properties that cannot be achieved by any of the constituent materials acting alone (Gibson, 2013).

Wood is the oldest composite: cellulose fibers in a lignin matrix. The cellulose fibers have high tensile strength but are very flexible (i.e., low stiffness), while the lignin matrix joins the fibers and furnishes the stiffness. Bone is yet another example of a natural composite that supports the weight of various members of the body. Such composite material systems result in a performance unattainable by the individual constituents, and they offer the great advantage of a flexible design; that is, one can, in principle, tailor-make the material as per specifications of an optimum design (Krishan Chawla, 2013).

Technological progress is associated with continuous improvement of existing material properties, as well as with the expansion of structural material classes and types. Usually, new materials emerge due to the need to improve structural efficiency and performance. Also, as a rule, new materials themselves in turn provide new opportunities to develop updated structures and technology, which then challenge materials science with new problems and tasks (Vasiliev, 2013). Natural fibers are biologically occurring materials which have two main sources (a) agriculture production and (b) production residue of crops when they are processed for the primary uses. Typically known natural fibers are wood, silk, ramie, jute, hemp, kenaf, sisal, coir, flax, bamboo, and fruit fibers. Natural fibers are majorly used with thermoplastics and thermoset plastics (Lalit et al., 2018). Classification of composite materials is shown in Fig. 2.1.

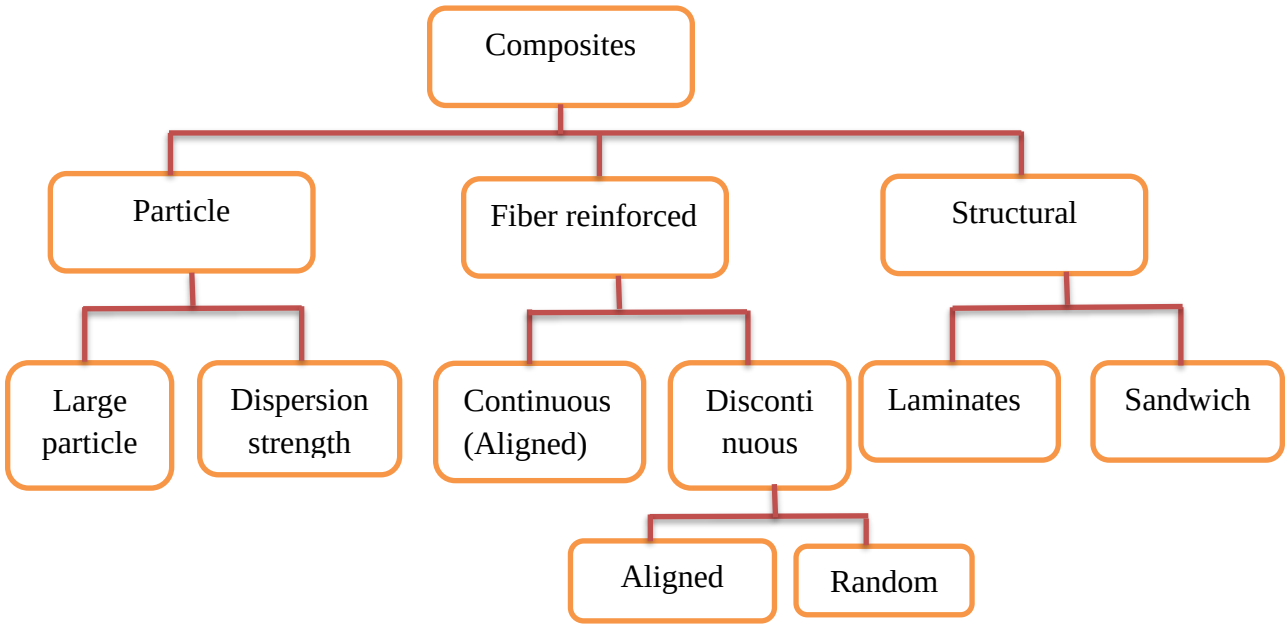


Figure 2.1 Composite classification (Otani et al., 2014)

2.2 Matrices

Many composite materials are composed of just two phases; one is termed the matrix, which is continuous and surrounds the other phase, often called the dispersed phase. The properties of composites are a function of the properties of the constituent phases, their relative amounts, and the geometry of the dispersed phase (William D. Callister, 2007). The matrix holds the fibers together in a structural unit and protects them from external damage, transfers and distributes the applied loads to the fibers, and, in many cases, contributes some needed property such as ductility, toughness, or electrical insulation. A strong interface bond between the fiber and matrix is desirable, and so the matrix must be capable of developing a mechanical or chemical bond with the fiber. The fiber and matrix materials should also be chemically compatible, so that undesirable reactions do not take place at the interface (Gibson, 2013). Thermoplastics and thermoset plastics both are non-bio degradable but thermoplastics are recycled in an easy manner as compared to thermoset. The use of natural fiber with the above said plastics does not result in fully biodegradable composite but it decreases the amount of non biodegradable waste (Lalit et al., 2018). Classification of matrices shown in Fig. 2.2.

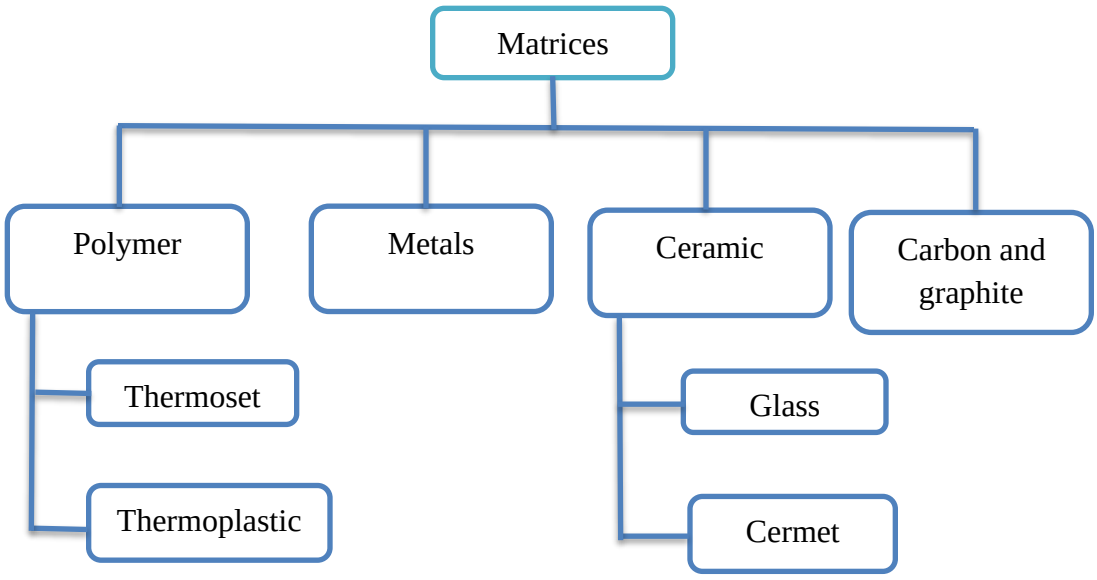


Fig 2.2 Classification of matrices (Stokke et al., 2013)

2.2.1 Polymer matrices

Polymers are structurally much more complex than metals or ceramics. They are cheap and can be easily processed. On the other hand, polymers have lower strength and modulus and lower use temperature limits. Because of predominantly covalent bonding, polymers are generally poor conductors of heat and electricity. Polymers, however, are generally more resistant to chemicals than are metals. Thermosetting polymers decompose on heating. Cross-linking makes sliding of molecules past one another difficult, making the polymer strong and rigid. (Krishan K. Chawla, 2013). The most used matrix materials are polymeric. The reason for this is twofold. In general, the mechanical properties of polymers are inadequate for many structural purposes. Their strength and stiffness are low compared to metals and ceramics. These difficulties are overcome by reinforcing other materials with polymers (Chandramohan & Marimuthu, 1996).

Thermoset matrices

Like the reinforcement, the matrix is also an important element and a continuous phase in the composite. There are two categories of polymer matrices: thermosets and thermo- plastics. The main characteristic of these thermoset composites is their irreversible cycle. This makes it possible to consider that this type of composite undergoes an exothermic transformation. It is characterized by its thermal stability, chemical resistance at low viscosity, as well as the creep,

and stress resistance. Among the common polymer matrices used with continuous fibers are polyester and epoxy resins.

Unsaturated Polyester resin

Unsaturated polyester is a thermoset resin commonly used as a matrix material in polymer composites. The polyester resin is a viscous liquid resin resulting from the condensation reaction between a glycol and an unsaturated dibasic acid. It is classified as one of the most versatile synthetic copolymers (Ouarhim et al., 2018). To polymerize this resin, it is mixed with its proper catalyst. A catalyst (commonly, methyl ethyl ketone peroxide, MEKP) is used to start the curing reaction. The cured thermoset is amorphous in structure. Moreover, they are characterized by their performance, low cost, and extreme processing versatility (Krishan K. Chawla, 2013).

Epoxy resin

Epoxy resin is a viscous liquid resin classified in the group of synthetic resin which contains an epoxide group in its chemical structure. It is characterized by its high adhesive properties (good adhesion with reinforcement), high chemical resistance, better moisture resistance, excellent mechanical properties, good fatigue resistance, low shrinkage, strong durability at low and high temperatures, and high electrical resistance, which justifies its numerous uses in modern industries. The epoxy resin having a transparent color is mixed with its suitable hardener (darker color) to polymerize and get an irreversible material (Ouarhim et al., 2018).

2.3 Reinforcements

The role of the reinforcement in a composite material is fundamentally one of increasing the mechanical properties of the neat resin system. Reinforcements need not necessarily be in the form of long fibers. One can have them in the form of particles, flakes, whiskers, short fibers, continuous fibers, or sheets (Krishan Chawla, 2013). All the different fibers used in composites have different properties and so affect the properties of the composite in different ways. Fiber reinforcements can be classified as synthetic and natural fibers. Synthetic fibers include glass, aramid, and carbon. Natural fibers are the organic fiber that is found in plants and animals. Natural fibers are considered the main constituent type in the natural fiber reinforced polymer composites.

2.3.1 Natural fibers

Natural fibers are biologically occurring materials that have two main sources agricultural production and production residue of crops when they are processed for the primary uses. Natural fibers are majorly used with thermoplastics and thermoset plastics. Fiber is the reinforcement used to bear the load transferred through the matrix. Plant fibers are the most abundant fiber among all the natural fiber. Naturally occurring fibers can be classified into three main categories i.e. animal fiber, mineral fiber, and plant fiber (Lalit et al., 2018). Classification of fibers is shown in Fig.2.3.

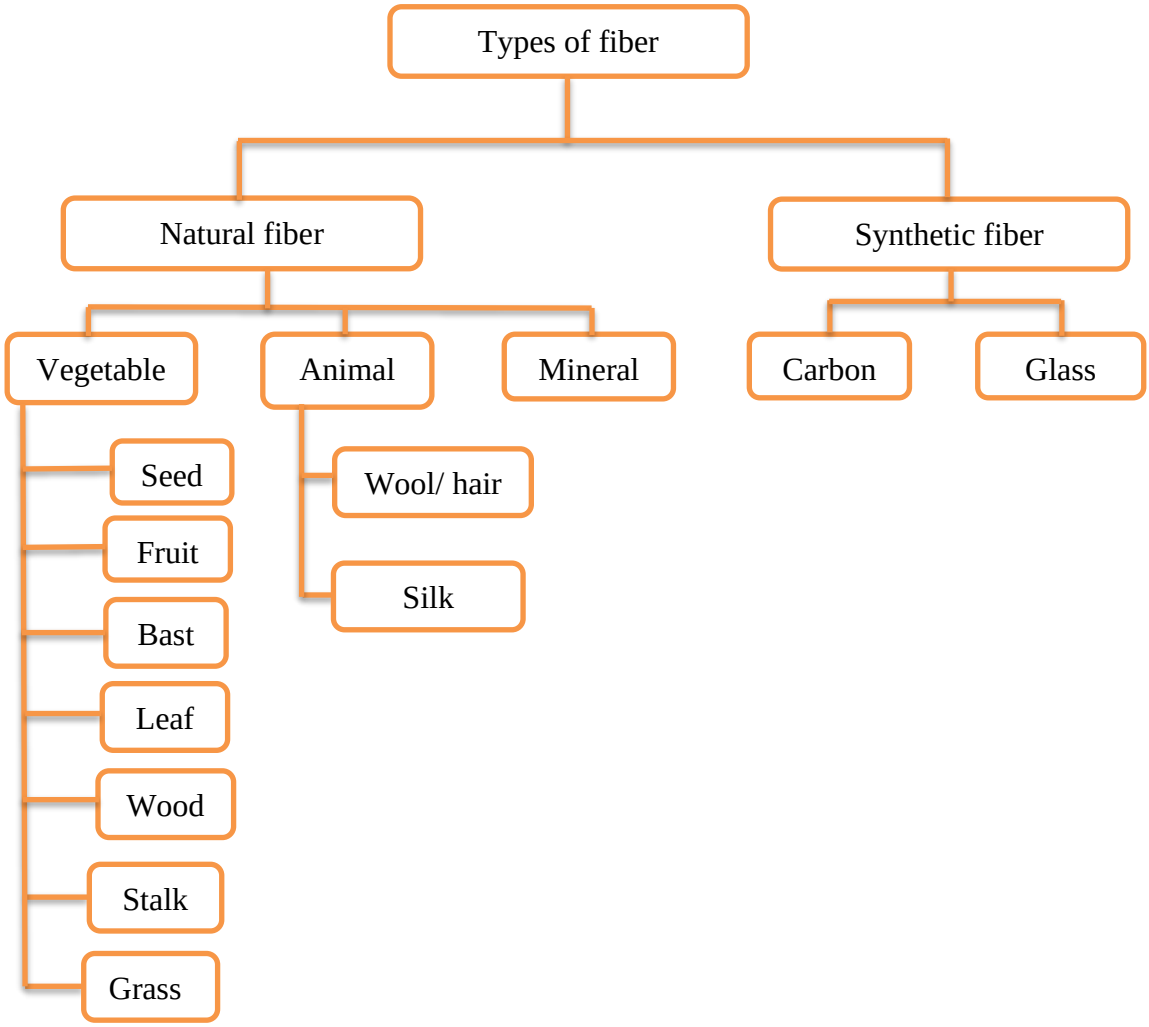


Fig. 2.3 Classification of fibers (Lalit et al., 2018)

2.3.2 Chemical composition of natural fibers

Plant-based natural fibers are lignocellulosic and are composed of cellulose, hemicelluloses, lignin, pectin, and waxy substances (Dominici et al., 2019a). Coffee husk represents an interesting source of cellulosic reinforcing materials. The coffee husk contains 57% cellulosic and 22% lignin. Natural cellulose acquired from these kinds of biomasses can be changed into micro-and nano-scale materials, yielding products, such as microcrystalline cellulose, micro febrile cellulose, or cellulose nano-crystals with very good properties as reinforces, while this permits for a superior utilization of lingo-cellulosic residues (Blinová et al., 2017).

The properties of natural fibers are affected by many factors such as variety, climate, harvest, maturity, retting degree, decortications, disintegration (mechanical, steam explosion treatment), fiber modification, textile and technical processes (spinning and carding) (Essabir et al., 2018).

Cellulose: is a glucan polymer of D-glucopyranose units, which might be linked collectively by using β-(1-4)-glycosidic bonds. The constructing block for cellulose is cellobiose because the repeating unit in cellulose is a two-sugar unit (Rowell, 1996).

Hemicelluloses: In nature cellulose in no way takes place in pure form. The main additives found collectively with cellulose are hemicellulose, lignin, and pectin. In general, the hemicellulose fraction of flora consists of a collection of polysaccharide polymers with a lower degree of polymerization than cellulose and containing specifically sugars.

Lignin: Lignin is amorphous, incredibly complex, particularly fragrant, polymers of phenylpropane units. All plant lignin consists especially of 3 basic building blocks of guaiacol, syringe, and p-hydroxyphenyl moieties, even though other fragrant type devices additionally exist in lots of different types of plants about 70% of the lignin is located inside the secondary wall (Han & Rowell, 2008).

Extractives: The extractives are a group of cellular wall chemical compounds mainly which include fats, fatty acids, fatty alcohols, phenols, terpenes, steroids, resin acids, rosin, waxes, etc. These chemicals exist as monomers, dimers, and polymers. They derive their name utilizing being chemical compounds that are eliminated by way of one of the numerous extraction procedures (Han & Rowell, 2008).

2.3.3 Coffee

Coffee is a crucial plantation crop belonging to the *Rubiaceae*, subfamily *Cinchonoideae*, and tribe *Coffeae*. Coffee trees grow in tropical regions, between the tropic of Cancer and Capricorn, that have abundant rainfall, year-round temperature averaging 70 ˚F and no frost. They grow at altitudes starting from sea level to 6500 feet and even above (Murthy & Madhava Naidu, 2012). A typical coffee tree and coffee cherry fruit wastes are shown in Fig. 2.4.

(a) (b)

Fig. 2.4 (a) Atypical coffee tree picture (b) coffee cherry fruit wastes(Campos-Vega et al., 2015)

Depending on the method chosen for coffee processing, different by-products can be produced:

Pre-roasting coffee by-products

- Dry processing: coffee husks,
- Semi-dry and wet processing: coffee pulp,

Post-roasting coffee by-products: coffee silver skin, spent coffee grounds. Major coffee wastes are shown in Fig. 2.5.

Fig. 2.5 Major coffee by-products a – Husks, b – Pulp, c – Silverskin, d – Spent coffee ground (Campos-Vega et al., 2015)

Coffee husk

Coffee husks are the main solid residues from the processing of coffee, that there are not any current profitable uses, and their adequate disposal constitutes a serious environmental problem (Sime et al., 2017b). Coffee pulp or husk is a fibrous mucilaginous material (sub-product) obtained during the processing of coffee cherries by wet or dry process, respectively. Coffee pulp/husk contains some amount of caffeine and tannins, which makes it toxic, leading to the disposal problem (Pandey et al., 2000). The main by-product from the dry method (also termed as "unwashed") is the coffee cherry husk (Fig. 2.5 a) which is composed of dried skin, pulp, and parchment. It representing about 12 % of the berry on a dry-weight basis (Blinová et al., 2017). The chemical composition of coffee husk and pulp is given in Table 2.1.

Table 2.1 Chemical composition of coffee husk and pulp (Oliveira & Franca, 2015)

Content	Coffee husk (dry-processed) in %wt	Coffee pulp (wet-processed) in %wt
Protein	8-11	4-12
Lipids	0.5-3	1-2
Minerals	3-7	6-10
Carbohydrates	58-85	45-89
Caffeine	1	1
Tannins	5	1-9

Coffee pulp

Coffee pulp is the first by-product obtained during wet or semi-dry processing and represents 29% dry weight of the whole cherry. Coffee pulp comprises the exocarp (outer skin) as well as the mesocarp (fleshy portion) (Blinová et al., 2017). The main composition of coffee husk and pulp is given in Table 2.2.

Coffee silver skin

Coffee silverskin frequently known as chaff is the first coffee industry residue produced in consuming countries since it is released during roasting if the beans were not polished before shipping (Blinová et al., 2017).

Table 2.2 Main composition of coffee husk and pulp (Bekalo & Reinhardt, 2010)

Type	Coffee residue	
	Coffee husk (%wt)	Coffee pulp (%wt)
Cellulose	19-26	40-49
Hemicellulose	24-45	25-32
Lignin	18-30	33-35
Ash	6-7	0.5-1

2.4 Nanomaterials (Nanofillers)

The improvement of the efficiency of composite materials for engineering applications has led to the utilization of nano-sized fillers like nano clay, nano-silica, nano-graphene, carbon nanotubes. Nanofillers are often considered as additives in solid form to upgrade different properties of original materials without increasing the density of materials. Nano silica particle is one of the common nanoparticles utilized in literary studies to enhance behaviors of fiber-reinforced composite materials. Its better mechanical properties, nano-silica inclusion in polymer composite has been utilized in many areas like the automotive, electronics, aerospace industries (Bozkurt et al., 2017).

Attempts are made to extend the compatibility between the polymer matrix the natural fibers with the assistance of varied surface treatment strategies like alkylation, acetylation, malleated coupling, etc. Nanoparticles or nanofillers have tremendous potential to be used as filler material that can improve the properties of polymer composites. Nano SiO_2 has been proven very useful for the improvement within the mechanical properties of those fiber-reinforced composites (Devnani & Sinha, 2019). A polymer matrix with reinforcing short fiber and nano-silica can help

achieve enhanced mechanical properties of composites without a significant increase in cost and environmental degradation (Assaedi et al., 2019).

However, natural fibers tend to absorb moisture either from the air or from rainwater, which will affect the properties of the overall composite. The hygroscopic nature of natural plant fiber is contributed by its microscopic like tubes with cell walls surrounding the central lumen. The lumen contributes to water absorption behavior due to the transport of water molecules via capillary action, which also involves the pores of the fibers. Furthermore, natural plant fibers structure composition contains a high amount of cellulose and hemicellulose, which produce the polar hydroxyl group that makes them hydrophilic, as matrix materials have a tendency of being hydrophobic, hydrophilic natural fibers will repel them and making it difficult to achieve good fiber wettability during production.

There are three types of mechanism in which moisture will be absorbed into the composite as listed below:
a) Diffusion of the water molecules into the microbubbles between polymeric chains. The micro bubbles are produced during the mixing and fabrication procedure.

b) Via capillary transport into the gaps and flaws in between the interfaces of fiber and matrix which occurs due to improper fiber wettability and poor fiber-matrix interaction.
c) Microcracks in the matrix produced due to the swelling of fibers. This mostly happens in the case of natural fiber-reinforced composites.

During the addition of nano-silica into the fiber composite system, the blending technique needs to be high speed and low time which results in low water absorption. When fabricating composite, the idea behind the impregnation process is to fill the empty spaces of the natural fiber (lumen cavity and cell wall) with polymer resin. By blocking the water path into the fiber, it's anticipated that the fiber will reduce its water uptake. Nano silica is expected to be able to impregnate the fibers' lumen to reduce the fiber water absorption rate (Bajuri et al., 2018a). Tensile properties of nano-silica-filled sisal composites were found 1.5 and 1.08 times higher than that of exclusive nano-silica-filled composite (Prajapati & Gupta, 2019).

2.5 Treatment

Mechanical properties of natural fiber and their compatibility with the polymer can be increased with the help of physical and chemical methods. In physical methods like corona discharge, sputtering, calendaring, low-temperature plasma, stretching, thermal treatment, etc., the structural properties of natural fibers are altered which results in increasing the mechanical bonding between fiber and polymer. Chemical methods include some popularly known treatments such as graft copolymerization, silane treatments, alkali swelling, cyanate treatment, impregnation of fibers, etc. which results in enhancement of the adhesion between the fiber and polymer (Zhuang & Chen, 2019). Alkalization (KOH and NAOH treatment) eliminate open hydroxyl group that tend to bond with water molecule and also dissolve hemicelluslose (Lalit et al., 2018).

Alkali treatment

Alkaline treatment or mercerization is one of the most used chemical treatment of natural fibers when used to reinforce thermoplastics and thermosets. The important modification done by alkaline treatment is the disruption of hydrogen bonding in the network structure, thereby increasing surface roughness. This treatment removes a certain amount of lignin, wax, and oils covering the external surface of the fiber cell wall, depolymerizes cellulose, and exposes the short length crystallites. Alkali treatment led to an increase in amorphous cellulose content at the expense of crystalline cellulose. It is reported that alkaline treatment has two effects on the fiber: (1) it increases surface roughness resulting in better mechanical interlocking; and (2) it increases the amount of cellulose exposed on the fiber surface, thus increasing the number of possible reaction sites. Consequently, alkaline treatment has a lasting effect on the mechanical behavior of flax fibers, especially on fiber strength and stiffness (X. Li et al., 2007). The alkaline treatment gave up to a 30% increase in tensile properties (both strength and modulus) for flax fiber–epoxy composites and coincided with the removal of pectin. Alkaline treatment also significantly improved the mechanical, impact fatigue, and dynamic mechanical behaviors of fiber-reinforced composites. The effect of NaOH concentration (0.5, 1, 2, 4, and 10%) for treating sisal fiber-reinforced composites and concluded that maximum tensile strength resulted from the 4% NaOH treatment at room temperature (Ray et al., 2001). 5%NaOH treated sisal fiber-reinforced polyester composite had better tensile strength than 10% NaOH treated

composites. This is because, at higher alkali concentration, excess de-lignification of natural fiber occurs resulting in a weaker or damaged fiber. The tensile strength of the composite decreased drastically after a certain optimum NaOH concentration (Mishra et al., 2003).

2.6 Fabrication method

There are different types of a fabrication methods for composite materials. Some common composite fabrication techniques are described below.

(i) **Spray layup:** is an open mold method that could produce complex parts greater economically than hand lay-up. Chopped fiberglass reinforcement and catalyzed resin, and in a few cases, filler materials, are deposited on the mold surface from a combination chopper/spray gun. Rollers or squeegees are used to manually castoff entrapped air and work the resin into the reinforcements. Woven fabric or woven roving is frequently delivered in precise regions for more strength. General purpose, room temperature remedy polyesters are normally used to produce such elements as truck camper shells and tub & bathe stalls. As in hand lay-up, gel coats are used to provide a high-quality colored part surface (Thomas et al., 2012). The spray layup technique is as shown in Fig. 2.6.

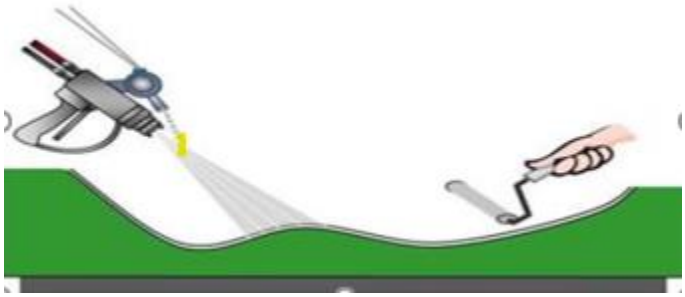


Fig. 2.6 The spray layup technique (Thomas et al., 2012)

(ii) **Compression molding:** Compression molding is a closed mold system finished with matched steel molds utilizing. Sheet molding compound (SMC) and bulk molding compound (BMC) is broadly used thermosetting-based molding compounds. Typical thermoset resins used in compression molded components are polyesters, vinyl esters, epoxies, and phenolic. Compression-molded merchandise varies from dinnerware,

trays, buttons, appliance housings, huge containers, to leisure vehicle frame panels which include snowmobiles, and jet skis (Thomas et al., 2012).

(iii) **Injection Molding**: Material granules for the element are fed through a hopper right into a heated barrel, melted the use of heater bands, and the frictional motion of a reciprocating screw barrel. The plastic is then injected via a nozzle proper into a mold hole area in which it cools and hardens to the configuration of the cavity.

(iv) **Hand Lay-Up Technique:** Hand lay-up is the oldest simplest and the most generally used approach for the manufacture of each small and big reinforced material. A flat surface, a hollow space, or a positive-formed mold, made from wood, metal, plastic, or a combination of those substances might also use for the hand lay-up method. A mold release agent is applied onto the molding floor for ease of removing completed composite merchandise. The laying-up of a component starts with making use of a gel coat to the mold surface. A roller brush is used to consolidate the composites and the composites are made layer by layer. The system is repeated with more layers until the desired thickness is obtained. Then the composites are cured at room temperature or in an oven. Thermosetting resins like unsaturated polyester, vinyl ester, and epoxy are several of the most generally used resins (Thomas et al., 2012). The hand layup technique is shown in Fig. 2.7.

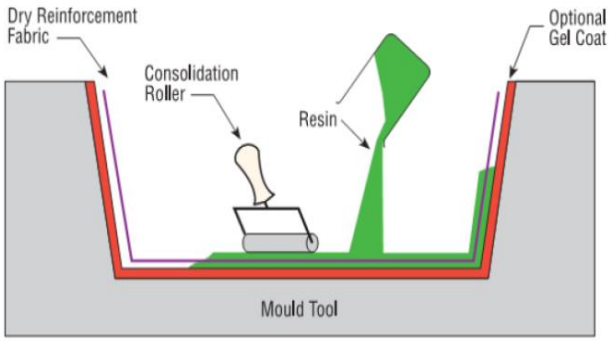


Fig. 2.7 Hand layup technique (Thomas et al., 2012)

2.7 Previous research works

In the literature, there are many investigations devoted to incorporating coffee husk particles into polymers. Some of the previous investigations are given below:

Researchers found that when coffee silver skin is introduced in bulk enhances elastic modulus while reducing strain. They used CSS treated and untreated and polyethylene as matrix and modified polyethylene as a binding agent. Treated coffee silver skin decreases water uptake and improves the bonding (Dominici et al., 2019a). Some studied the effect of coffee parchment and coffee husk content on thermal-mechanical and microstructural properties of composites. They found it increases thermal stability and decreases crystallinity with little effect on mechanical properties (Devnani & Sinha, 2019). Other researchers studied the effects of alkali treatment relative to the tensile and flexural mechanical properties of Colombian coffee silver skin fiber using manual layup manufacturing. Alkali treatment increases surface roughness which leads to better mechanical coupling (Ochoa et al., 2017). Some studied the outcome of water-hating treatment on expended coffee ground powder is compared with conventional salinization treatment and use of bonding copolymer in relationships of mechanical properties, morphology, and water uptake (García-García et al., 2015b). Researchers studied the structure of pectin/coffee hybrid film and they showed that coffee particles distributed uniformly but the mechanical properties become worse (Cataldo et al., 2017). Other researchers studied acrylic acid-grafted used to enhance the necessary properties of membranes since water resistance increased (C. S. Wu, 2017). Researchers found the removal of oil improved the water-resistance of composites. The physical properties of spent coffee grounds polypropylene composites were enhanced since oil removal enhances interfacial adhesion and compatibility between filler and matrix (H. Wu et al., 2016). Other scholars obtained a substantial reduction of thermal conductivity when coffee grounds increase which can be used in the thermal lining in constructions so lining wall from inside saves a lot of energy in cold weather (Lachheb et al., 2019). Studied the effects of deviations maleic anhydride and spent coffee ground in terms of strength and physical parameters they use soya bean oil as a matrix (Tarazona et al., 2017). Other researchers found mechanical strength of organic composites reduces with growing natural filler. However, mechanical, and thermal properties were enhanced by adding of coupling agent (Baek et al., 2013).

Researchers got experimental results that showed that increasing the CHF loading in the HDPE matrix increased the modulus and thermal properties of the composites, but resulted in poor water resistance (Huang et al., 2018). Scholars studied the effect of copolymer and homopolymer using spent coffee ground and maleic anhydride polypropylene, the result showed increased

mechanical properties except impact strength to co-polymeric composite with compatibilizer (Chitra et al., 2014).

Some other researchers used roast coffee shells using cellulose nanocrystals and polylactic acid. The tensile strength and elastic modulus increased by 1-3%. Water permeability decreased and it becomes an enhanced biopolymer (Sung et al., 2017). Researchers investigated the effects of silane treatment on the spent coffee ground which shows better bonding as a result tensile and torsion properties are improved (Essabir et al., 2018). Researchers studied the anti-ultra-violet property of spent coffee ground and it shows better characteristics including high thermal comfort performance and mechanical properties were also improved (R. X. Li et al., 2016).

Among natural organic fillers that have been studied are wood fibers and sawdust, flax, sisal, kenaf, jute, eucalyptus, starch, and oil palm empty fruit bunches and rice husk ashes, coffee grounds, and coffee silver cover (Zarrinbakhsh et al., 2016).

Organic fillers other than wood are desirable because of the decreasing availability of natural resources (La Mantia et al., 2005). Tensile, flexural, and impact properties are the most studied mechanical properties of natural fiber plastic composites (Faruk et al., 2014) the weak point is impact strength. Recent interest in environmentally friendly materials has led to the use of agricultural residues as raw material to produce particleboard from coconut chips, bagasse, and waste tea leaves. Further, the coffee shell can be used to produce a value-added product such as particleboard or building insulation. The two most grown varieties are the highly regarded Coffee Arabica and Coffee canephora (Toldy et al., 2011) coffee Arabica is produced in Ethiopia. During coffee processing, the husk, pulp, parchment, and mucilage are removed, and they account for 12%, 29%, 12%, and 5% respectively, of dry weight (Zarrinbakhsh et al., 2016). The silver skin floated during roasting. Seventy percent of the coffee exported from Ethiopia is sun-dried and dry-processed while the rest (30%) is wet-processed coffee. Dry processed coffee husk has moistures ranging from 7% to 13% which affects further processing (Oliveira & Franca, 2015).

The use of agricultural materials like a coffee husk and hulls not only provides a renewable material source, but also generates a non-food source of economic development for farming and rural areas (Bekalo & Reinhardt, 2010). Epoxy resins are widely used as matrices in advanced

composites due to their good impregnation and adhesion to fiber reinforcement, resulting in excellent mechanical performance; chemical and electrical resistance, and low shrinkage on the cure (Yan et al., 2012).

Researchers studied influence of coffee bean reinforcement (5, 10, 15, 20, 25, and 30 %wt) on mechanical properties of epoxy composite. The maximum mechanical properties were observed for a 25% mass fraction of coffee bean natural filler composite. The fracture toughness increases with the incorporation of coffee bean filler, due to the uniform distribution of coffee bean in an epoxy matrix, and stress was disseminated equally. It also indicates that bonding between filler and matrix is better and 30% mass fraction filler reveals reduced wettability with the epoxy matrix in the composite (Muniappan et al., 2020). Others investigated spent coffee ground as filler for epoxy composite. Spent coffee ground was chemically treated with alkaline to decrease water absorption. Treated spent coffee ground can be used as the reinforcement in polymeric material matrix. The composites prepared with a molar ratio of maleic anhydride 1: 1.2 and SCG mass fraction of 35% were those that presented the best mechanical properties (Tarazona et al., 2017).

The researchers used coffee husk fiber as reinforcement in thermoplastic based composite. The researchers studied the chemical composition and thermal properties of CHF and WF; their result showed that both have similar properties. The use of coffee husk fiber improved the addition of coffee husk fiber into HDPE matrix improved the modulus and thermal properties of composite, but it decreased water resistance (Huang et al., 2018). Other researcher studied PBAT with different coffee husk composition with silane treatment. Their result showed that silane treated coffee husk is more compatible and had lower water absorption. The addition of 40% wt coffee husk improved the tensile strength and modulus of the composite (Lule & Kim, 2021). Researchers incorporated the coffee ground into polypropylene (20, 30, and 40 %wt) with maleated compatibilizer using rotating screw extruder and subsequent injection molding. The 40% wt coffee husk increase flexural strength, flexural modulus, tensile modulus, and heat distortion temperature. However, strain at break, impact, and tensile strength decreased by around 95, 40, and 20%, respectively (Leal et al., 2020). Other researchers studied coffee husk as organic filler in low density polyethylene matrix. The properties of this composite compared to the other inorganic fillers (micro calcium carbonate, and nano-SiO_2); the fillers improved tensile

modulus and tesnsile strength. Some relevant modifications of melting temperature and crystallinity degree for coffee husk composites were observed, but not observed for inorganic fillers composites (Urrego Yepes et al., 2017).

2.8 Research Gap

As above shown researchers have done researches on composites using agricultural waste materials including coffee wastes. The researches focused on spent coffee grounds to change into usable products like particle boards. The coffee husk composite was investigated with thermoplastic matrix composite while there is no research done on thermoset matrix composite using coffee husk. So, this is the gap to be studied. So, based on this research gap the researcher decided to research by using coffee husk with nano silicon dioxide reinforced unsaturated polyester composite. Studying coffee husk as reinforcement in thermosets can be used in home and small scale companies because itcan be processed easily unlike thermoplastics. The usage of coffee husk in composites decrease the coffee husk waste.

CHAPTER THREE

3. MATERIALS AND METHODS

In this chapter the methosds, and materials used during research briefly explained. The over all flow of the coffee husk polyester composite preparation and testing shown in Fig.3.1.

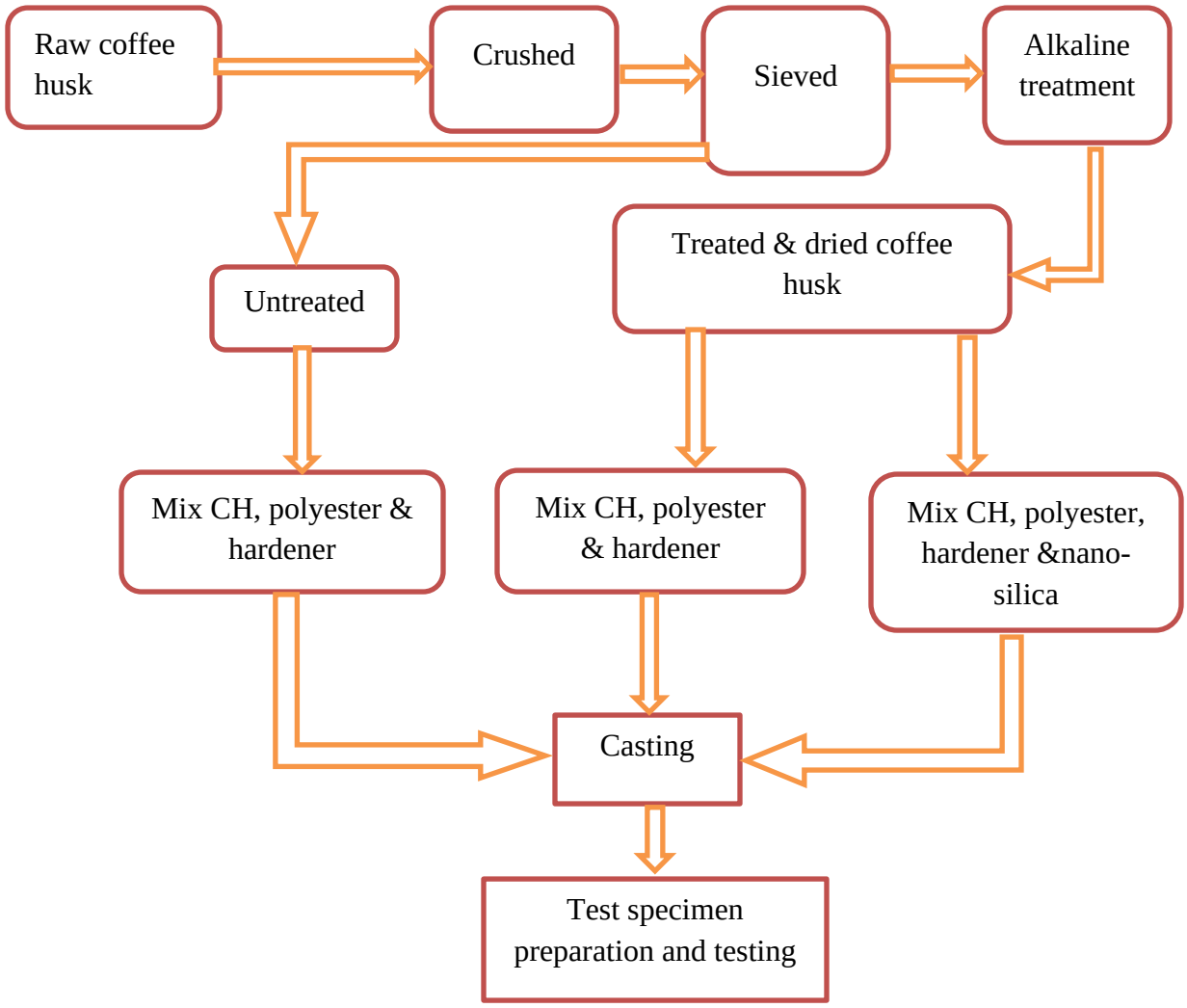


Fig. 3.1 Flow chart of the coffee husk polyester composite

The coffee husk plyester composite samples were prepared in nine composition sets because the coffee composition must be below 30% to get enough wetting, so the researcher chose three composition sets namely (15, 20, and 25 wt%). The other factor was NaOH treatment of coffee husk so both treated and untreated was evaluated. The nano-silica was used to improve the

mechanical properties of the coffee husk polyester composite, the nano-silica composition must not exceed 5%. Since the researcher had limited nano-silica, it was decided to improve the property of 80/20 wt% composite by compromising the cost and strength.

3.1 Materials

Unsaturated polyester resin

For this project work researcher used polyester resin as a matrix with a brand name of TOPAZ-1110 TP, which is a medium reactive unsaturated polyester resin based on phthalic anhydride, with excellent laminating properties, which is purchased from the local world fiberglass supplier in Addis Ababa, Ethiopia. Polyester is a commonly used resin in the manufacturing of composites with different reinforcing materials. Their extensive use is due to low-cost good performance and ease of processing.

Hardener

Unsaturated polyester resin is cured by adding a catalyst, which causes a chemical reaction. The catalyst initiates the chemical reaction of the polyester resin and monomer ingredient from liquid to solid-state. Therefore, the hardener (curing agent) used for this specific project work is a hardener with a brand name methyl ethyl ketone peroxide (MEKP) hardener, which is purchased from world fiberglass Addis Ababa, Ethiopia.

Mold release

Release wax is a chemical agent used to stop the bonding of the molding material with the mold. Mold release is essential for preventing the polyester from sticking to the mold when the composite is apart. Even though, there are several types of mold release used depending on the mold material and desired characteristics of the finished part, the most common type and used for this thesis work is HONEY WAX 250 purchased from world fiberglass Addis Ababa, Ethiopia.

Coffee husk preparation

The coffee husk used in this project purchased from Wolaita zone, Ethiopia which is dry processed, and fresh. Raw coffee husk crushed with the grinder and sieved, after that, it will be treated with sodium hydroxide as shown in Fig 3.2.

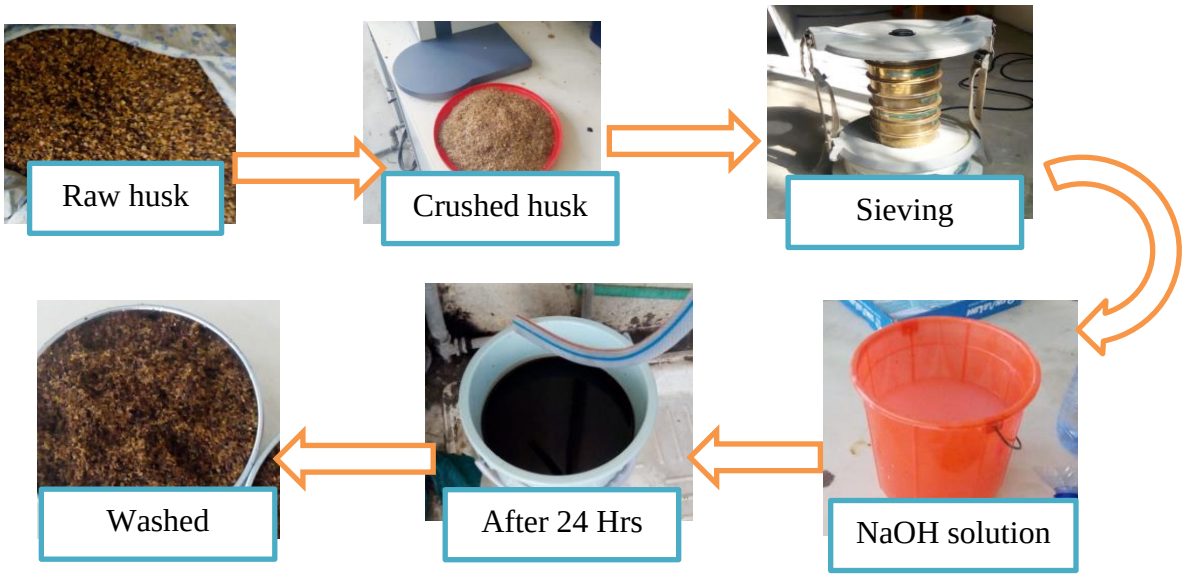


Fig. 3.2 Coffee husk preparation processes (photo taken ASTU chemical laboratory)

The treated coffee husk had clear and rough surface compared to the untreated coffee husk. After the coffee husk preparation process the coffee is dried naturally for three days, as shown in Fig. 3.3.

(a) **(b)**

Fig. 3.3 a) untreated coffee husk b) treated coffee husk (photo taken ASTU chemical laboratory)

Nano silicon dioxide

Nano-SiO_2 is a white fluffy powder composed of high purity amorphous silica powder. Because of its small particle size, nano-SiO_2 had the advantages of large specific surface area to volume ratio, strong surface adsorption, large surface energy, high chemical purity, and good dispersion. This material is purchased from India mart produced by Intelligent Materials Pvt. Ltd. The nano silicon dioxide is as shown in Fig. 3.4.

Stock No: NS6130-03-344
CAS: 7631-86-9
Purity: 99.9%
APS: 60-70nm
Molecular Formula : SiO_2
Molecular Weight : 60.08 g/mol

Form: Powder
Color: White
Density : 2.2 g/cm³
Melting Point: 1610 °C
Boiling Point: 2230 °C
Solubility: Insoluble in Water

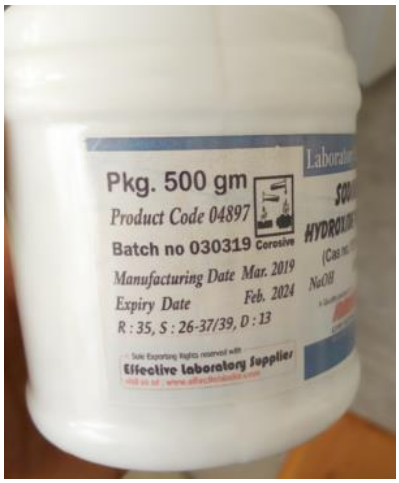


Fig. 3.4 Nano SiO2 (purchased product)

Fig. 3.5 Sodium hydroxide (purchased product)

Sodium hydroxide

Sodium hydroxide, also known as lye or caustic soda, has the molecular formula NaOH and is a highly caustic metallic base. Pure sodium hydroxide is a whitish solid, which is available in pellets, flakes, granules, and as a 50% saturated solution form. Sodium hydroxide is used in

many industries, mostly as a strong chemical base in the manufacture of pulp and paper, textiles, drinking water, soaps, and detergents. In this work, I used NaOH in pellets form, purchased from a local supplier (Sida general trading) with the product code of, 04897. At room temperature, sodium hydroxide is a white crystalline odorless solid that absorbs moisture from the air, if it is not sealed. Sodium hydroxide solution appears as a colorless liquid denser than water. Contact may severely irritate skin, eyes, and mucous membranes. Toxic by ingestion, corrosive to metals and tissue. The sodium hydroxide is as shown in Fig. 3.5.

Mold

The mold is made up of wood with 400x300x5mm and it contains the cover frame. The cover and Base surfaces of the mold and the walls are coated with mold release wax and allowed to dry. The functions of the cover plate are to cover, compress the fiber after the polyester mixture is poured, and avoid the debris from entering the composite parts during the curing time.

3.2 Methods for sample preparation

The coffee husk was crushed using PX-MFC 90D to the grit size of 0.25 – 2 mm. The coffee husk fiber separated in size of fiber length using a sieve. After that, some of the coffee husk fiber treated with 5%wt composition sodium hydroxide for 24 hours and washed with tap water. The coffee husk fiber dried using natural sunlight for three days.

Alkali treatment

Alkali treatment is the simplest method of chemical treatment of fibers; it leads to an increase in the amount of amorphous cellulose at the expense of crystalline cellulose. The important modification occurring here is the removal of hydrogen bonding in the network structure. The following reaction takes place as a result of alkali treatment. According to the literature, alkali solution has a good effect on treating natural fibers. In this study, a 5% NaOH solution was used to treat the raw coffee husk fiber, to improve the adhesion between the fibers and matrix, fibers were subjected to surface treatments. Sodium hydroxide is the most used chemical for cleaning/bleaching the surface of cellulosic fibers. Crushed coffee husk fibers were soaked in 5wt% NaOH solution for 24 hours. The treated fibers were washed in tap water to neutralize the excess of NaOH. This treated coffee husk fibers were dried in sunlight for three days before using as reinforcement in the synthesis of composite. The coffee husk powder soaked in the

sodium hydroxide solution for 24 hours at room temperature, then washed using tap water to avoid excess sodium hydroxide. The treated coffee husk fiber dried with natural sunlight.

Weight fraction and volume fraction of the fiber and the matrix content of the composite

The volume of the composite has been defined by the length, width, and depth of the mold, which is prepared for molding the composite material and the total volume of the composite is also the sum of the volume coffee husk and polyester resin.

$Vc = L * W * D 400 * 300 * 5 = 600{,}000mm^3 = 600cm^3$

$Vc = Vh + Vp$

Where: Vc is the volume of mold/composite Vh is the volume of coffee husk.

$Vp -$ is the volume of polyester resin, L- is the length of the mold, W- is the width of mold D- is the depth of the mold

Density of composite

The density of composite calculated as follows

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

Where; ρ_c is the density of the composite

m_c is mass of composite

V_c is the volume of composite

We can define the density of the composite from the above density equation:

$$\frac{m_c}{V_c} = \frac{m_h}{V_h} + \frac{m_p}{V_p}$$

Where; m_h is mass of coffee husk V_h is the volume of coffee husk

m_p is a mass of the polyester resin V_p is the volume of polyester resin

We can define the volume of the composite from the above density equation:

$$\frac{m_c}{\rho_c} = \frac{m_h}{\rho_h} + \frac{m_p}{\rho_p}$$

Where; $m_h -$ is mass of coffee husk $m_p -$ is the mass of the polyester resin

$\rho_h -$ is the density of coffee husk $\rho_p -$ is the density of polyester

The mass composition of the composite is the percent mass composition of the coffee husk fiber plus the percent mass composition of polyester resin.

$m_c = x\% * m_h + y\% * m_p$

Where x is the mass fraction of coffee husk fiber, y is the mass fraction of polyester resin.

For 85% polyester resin and 15% coffee husk fiber

$m_p = \rho_p * V * \%w = 1.13 * 600 * 0.85 = 576.3g$

$m_h = \rho_h * V * \%w = 0.6 * 600 * 0.15 = 54g$

$m_{ns} = \%w * m_t = 6.225g$

$m_{hr} = \%w * m_p = 1.5 * 576.3 = 8.644\ g$, similarly others are calculated and tabulated below

Mass composition

For different fiber- matrix ratios the mass composition of the composite material has been summarized in Table 3.1.

Table 3.1 fiber-matrix mass composition

Designation	Composition (%wt)				Mass (gram)				
	Polyester	Coffee husk fiber	Nano SiO_2	MEKP	Polyester	CH fiber	Nano SiO_2	MEKP	Total
S1	85	15	-	1.5	576.3	54	0	8.644	638.944
S2	80	20	-	1.5	542.4	72	0	8.136	622.536
S3	75	25	-	1.5	508.5	90	0	7.627	606.127
S4	85	15	-	1.5	576.3	54	0	8.644	638.944
S5	80	20	-	1.5	542.4	72	0	8.136	622.536
S6	75	25	-	1.5	508.5	90	0	7.627	606.127
S7	79	20	1	1.5	535.62	54	6.225	8.644	604.489
S8	77	20	3	1.5	522.06	72	18.676	8.136	618.872
S9	75	20	5	1.5	508.5	90	31.126	7.627	637.253

Note: **S1-S3** is untreated coffee husk reinforced polyester composite **S4-S5** treated coffee husk reinforced polyester composite **S7-S9** treated coffee husk reinforced polyester composite with nano-silica.

Volume fraction

The volume fraction of the constituents calculated by dividing the mass of the constituent by the density of constituent. The volume of fiber is equal to the weight of the fiber divided by the density of fiber

The volume of the matrix calculated as follows

$V_c=V_f+V_m$

Volume composition for sample one

$V_f = \frac{m_f}{\rho_f} = \frac{54}{0.6} = 90\text{cm}^3$

$V_m = \frac{M_m}{\rho_m} = \frac{576.3}{1.13} = 509.73\text{cm}^3$

similarly, the volume composition of constituents calculated and tabulated in table 3.2.

Table 3.2 Volume composition of the composite

Designation	Composition (%wt)				Volume (cm^3)				
	Polyester	Coffee husk fiber	Nano-SiO_2	MEKP	Polyester	Coffee husk fiber	Nano-SiO_2	MEKP	Total
S1	85	15	0	1.5	509.73	90	0	7.38	607.11
S2	80	20	0	1.5	479.64	120	0	6.95	606.59
S3	75	25	0	1.5	450	150	0	6.51	606.51
S4	85	15	0	1.5	509.73	90	0	7.38	607.11
S5	80	20	0	1.5	479.64	120	0	6.95	606.59
S6	75	25	0	1.5	450	150	0	6.51	606.51
S7	79	20	1	1.5	474	120	2.28	7.38	603.66
S8	77	20	3	1.5	462	120	8.48	6.95	597.43
S9	75	20	5	1.5	450	120	14.14	6.51	584.14

3.3 Preparing samples

3.3.1 Preparation of polyester and hardener:

The coffee husk powder, polyester resin, hardener, and nano-silica are mixed homogeneously in different proportions. Then the mix was poured from the mixing bowl and reacted in a mold in

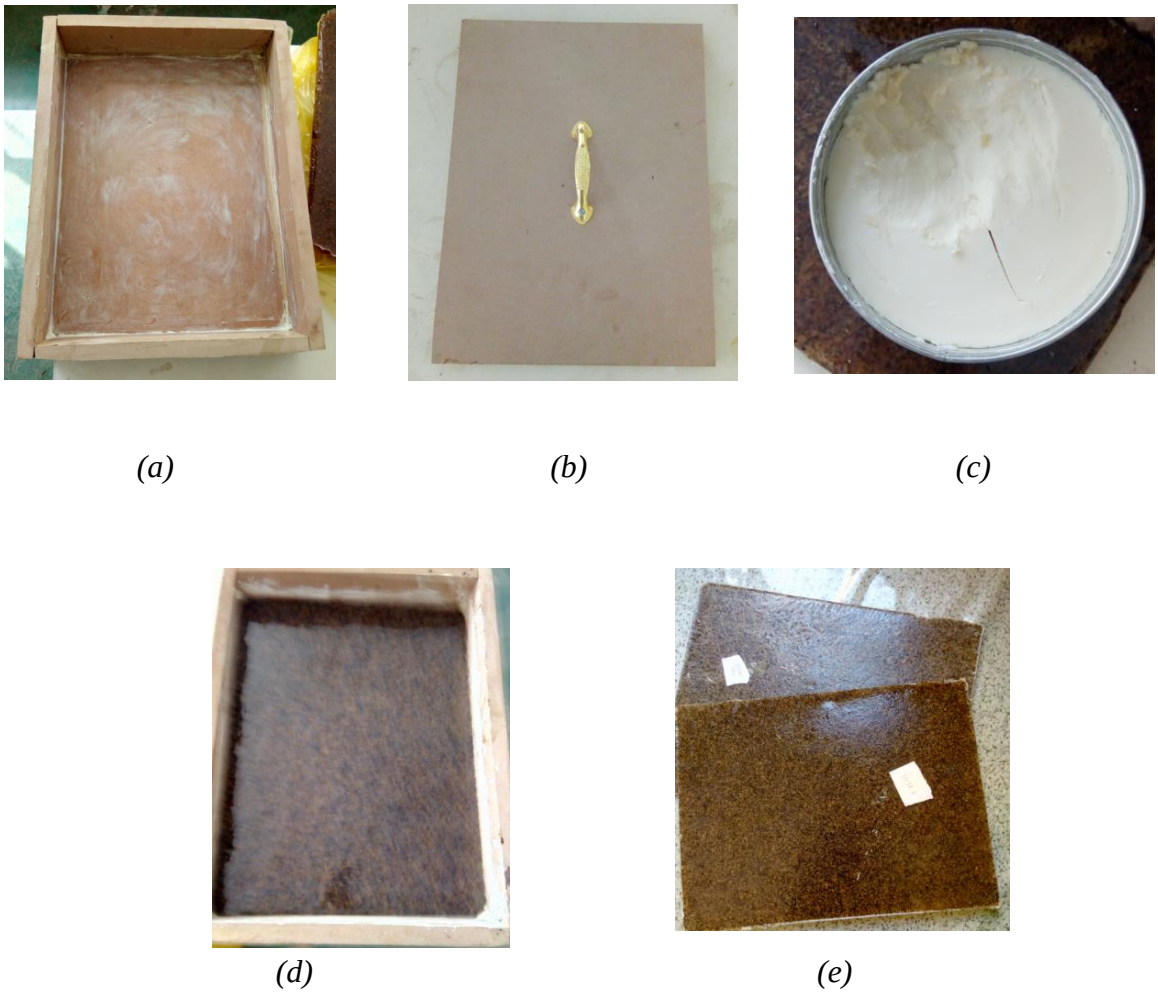
different proportions of coffee shell and polyester resin as well as nano silicon dioxide. Finally, the composite was ejected from the mold after 24 hours.

The coffee husk powder and the unsaturated polyester resin was mixed thoroughly for 25 minutes to get better wetting and uniform mixture. Then unsaturated polyester of TOPAZ-1110 TP resin and coffee husk powder mixture was mixed with hardener MEKP for about 5 minutes. The weight ratio for mixing hardener is 1-2 percent. The researcher used 1.5% of the hardener recommended by the manufacturer. The mixing was performed in the mixing containers (Bowl), the bowl is made of nickel to prevent melting of the bowl during the exothermic reaction of unsaturated polyester resin and hardener, the mixing was done slowly to not entrain any excess air bubbles in the resin. The mixing of coffee husk and unsaturated polyester and unsaturated polyester resin is as shown in Fig. 3.6.

Fig. 3.6 a) mixing coffee husk and polyester b) unsaturated polyester resin

3.3.2 Fabrication

The hand-lay-up method was adopted to fill up the prepared mold with an appropriate amount of unsaturated polyester resin and coffee husk fiber mixture. The process was conducted at room temperature. The prepared MDF mold was first covered with mold release wax. After the mixture was poured into the mold cavity, the mold was covered and applied some weight to remove dirt and the distortion of the final product. The curing process takes 24 hours and it was ready for test after a week. The fabrication process of the composite is as shown in Fig. 3.7.



(a) *(b)* *(c)*

(d) *(e)*

Fig. 3.7 (a) mold (b) mold cover (c) mold release wax (d) molding (e) some of the produced samples

3.3.3 Experimental procedures and steps

ASTM standard was used for preparing the sample to evaluate the mechanical properties of coffee husk fiber reinforced unsaturated polyester resin composite material. For each of the tests researcher used three specimens from each the samples. The specimens were prepared using a grinder then ground manually for better dimensional accuracy.

Dimensions for test specimens

The appropriate American Society of Testing Materials (ASTM) standard, ASTM D-3039, was followed while preparing the tensile test specimens for coffee husk fiber reinforced unsaturated polyester composite test and these samples are illustrated in Figure 3.8.

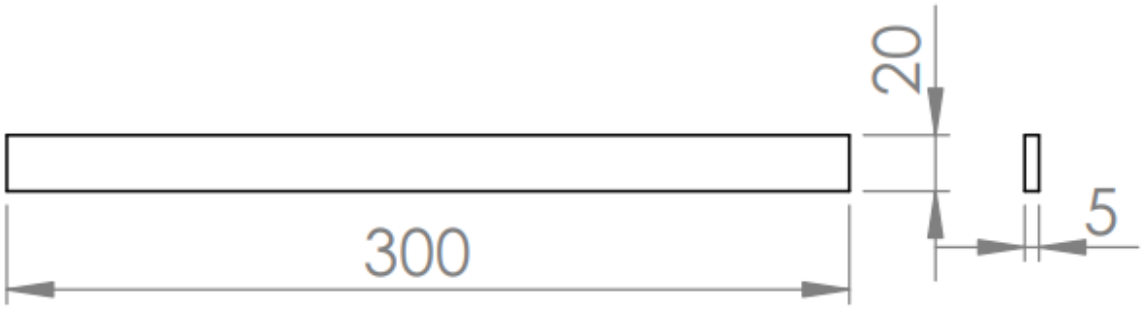


Fig. 3.8 Sample size of the tensile test specimen

The appropriate American Society of Testing Materials (ASTM) standard, ASTM D-256, was followed while preparing the impact test specimens for coffee husk fiber reinforced unsaturated polyester composite test and these samples are illustrated in the Figure 3.9.

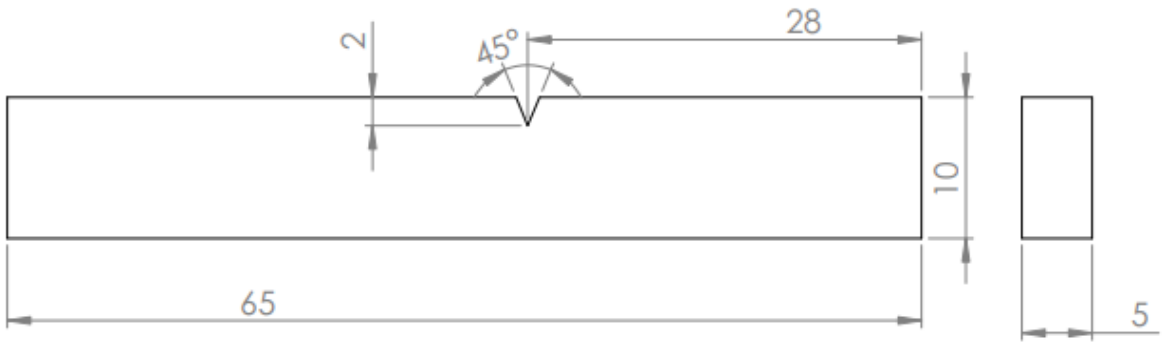


Fig. 3.9 Sample size of the impact test sample

The appropriate American Society of Testing Materials (ASTM) standard, ASTM D-785, was followed while preparing the hardness test specimens.

3.4 Introduction of Test Apparatus

A universal testing machine (UTM) was used to test the mechanical properties (tension, compression, etc.) of a given test specimen by exerting tensile, compressive, or transverse

stresses. This machine is known as a universal testing machine, because of the wide range of tests it can perform.

A universal testing machine consists of two main parts: a loading unit and a control unit. The arrangement of the test specimen and the exertion of the load was held in the loading unit. The variations in the application of the load and the corresponding test result were obtained from the control unit. The universal testing machine and its components are as shown in Fig. 3.10.

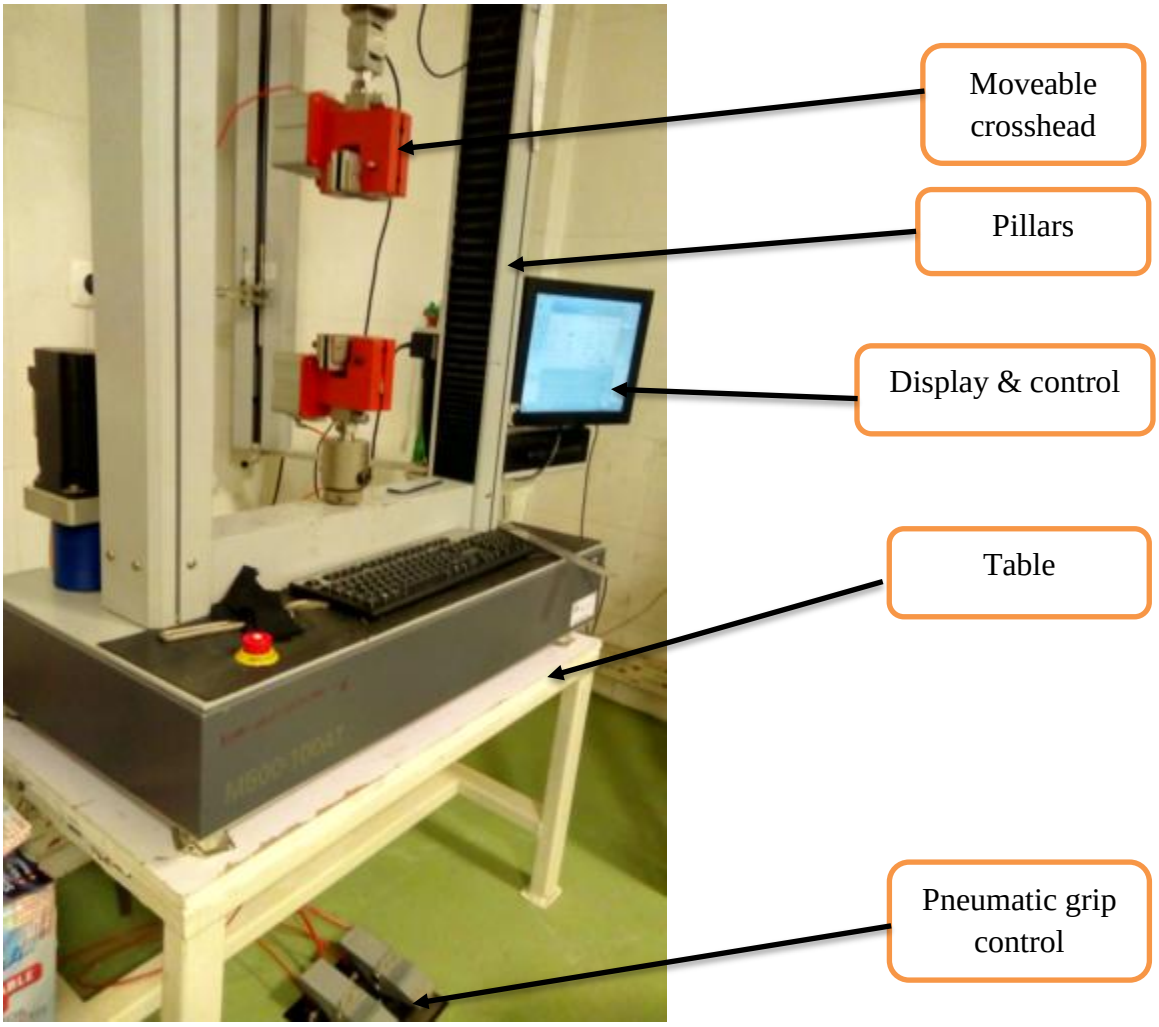


Fig. 3.10 universal testing machine and its major components (Ethiopian conformity assessment enterprise)

Tensile Strength Test (ASTM D3039)

The test specimen was in a controlled environment at a temperature of 20 °C and humidity of 65%. This test was used to determine the plane strength of the composite material. For each

sample (there were 9 samples) three specimens were tested, and the sample dimensions were 300x20x5 mm. Prepared samples under a controlled environment at a temperature of 22^0C and humidity of 65% as shown in Fig 3.11.

Fig. 3.11 Prepared tensile samples under controlled environment

During the test the specimens were gripped by the UTM crosshead, the bottom was fixed while the upper was movable, then the specimen tensioned by using the tensile force until fracture of the specimen. The crosshead was moving at a rate of 5mm/min and the gauge length was 200mm while the other 100mm for gripping. Force in newtons and elongation were obtained from the machine including stress at the peak, force at the peak, and strain at the peak. The typical specimen under the tensile strength test and some of the failed samples are shown in Figure 3.12.

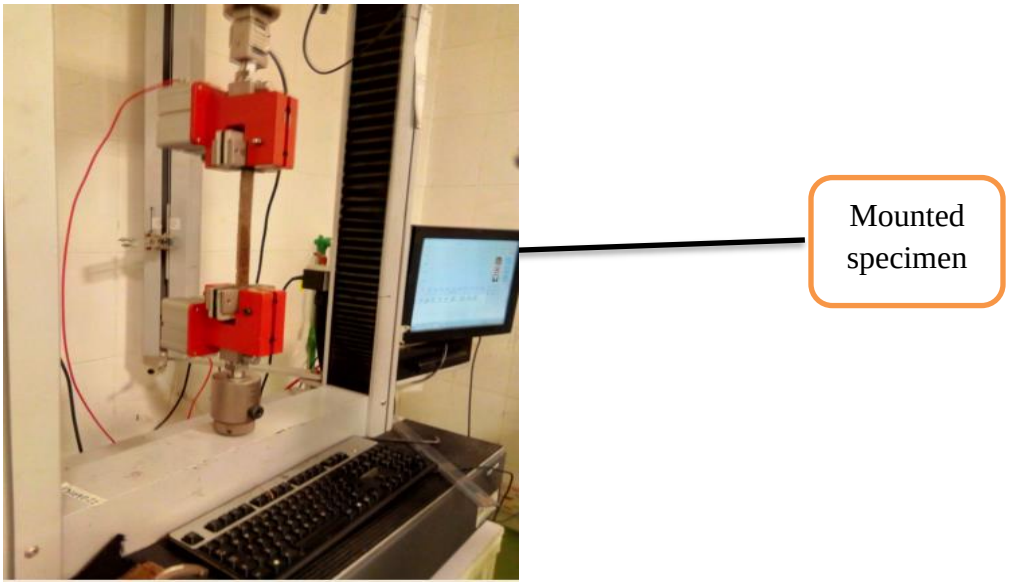


Fig. 3.12 Some of the failed specimens during tensile test and Tensile strength test machine respectively (Ethiopian conformity assessment enterprise)

Pendulum impact test machine

The Izod impact test, also known as the Izod V-notch test, is a high strain rate test that involves striking a standard notched specimen with a controlled weight pendulum swung from a set height. The impact test helps measure the amount of energy absorbed by the specimen during fracture. The impact test machine is shown in Fig. 3.13.

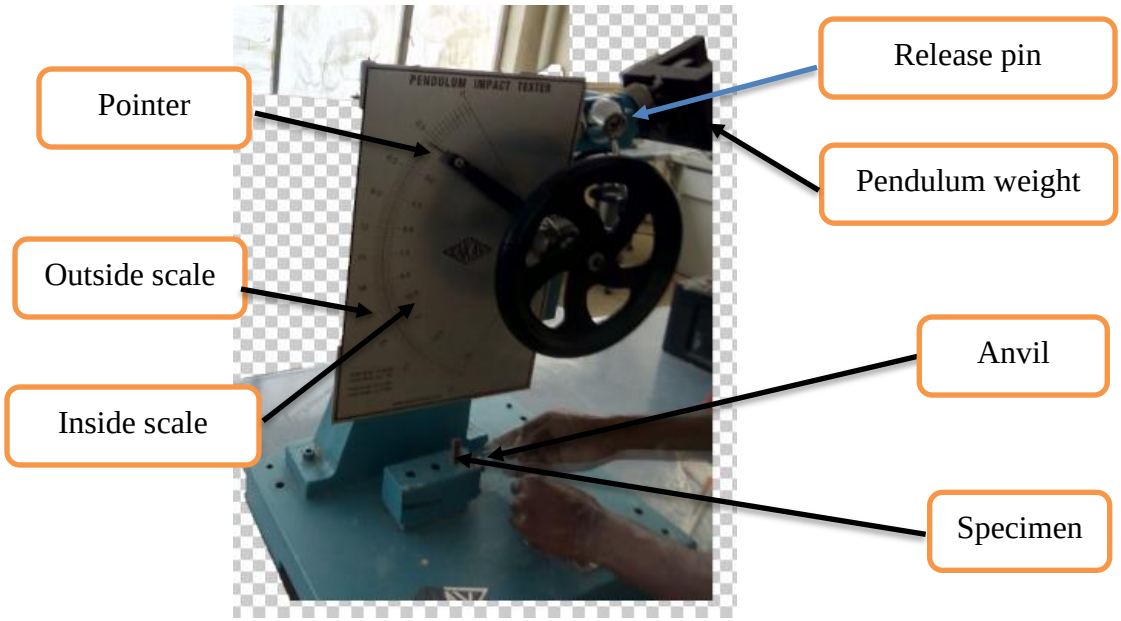


Fig. 3.13 Impact test machine

The notch guide tool was used to prepare the samples notch at a 45-degree angle before testing. The prepared impact test specimens and notch guide tool as shown in Figure 3.14.

Fig. 3.14 Prepared impact samples and 45-degree notch guide tool

Impact test (ASTM D256)

The impact test was used to determine the material's ability to withstand a high strain rate, the energy absorbed to fracture the material indicates the toughness of the material. For each sample (there were 9 samples) three specimens were tested, and the sample dimensions were 65x10x5 mm. During the test, the sample was gripped at one side and the other side was free, then the weight was released to strike the specimen, the result was displayed by the mechanical pointer. Some of the fractured specimens during the impact test is as shown in Fig. 3.15.

Fig. 3.15 Some of the failed specimens during an impact test

Water absorption test

Samples were prepared in dimension (30x25x5) mm, the specimens were kept dry at room temperature, then the dry weight was measured and inserted in tap water. It was covered to remove the entrance of dirt and other substances; it was measured continuously in 24 hours difference for three consecutive days. The water absorption test samples and water absorption test are shown in Fig. 3.16.

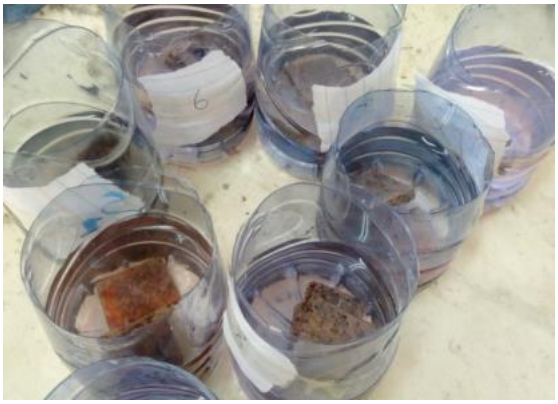



Fig. 3.16 water absorption test samples and water absorption test respectively

Water absorption expressed as

$$W(g) = \frac{m_f - m_i}{m_i} * 100$$

Where, $W(g)$ weight gained in percent m_f- final mass m_i- initial mass

Hardness test

Hardness is a measure of a material's resistance to localized plastic deformation. A small indenter is forced into the surface of a material to be tested, under controlled conditions of load and rate of application. The depth or size of the resulting indentation is measured, which in turn is related to a hardness number; the softer the material, the larger and deeper the indentation, and the lower the hardness index number. In this case hardness test is conducted using Vickers hardness test, Vickers (sometimes also called diamond pyramid). For each test a very small diamond indenter having pyramidal geometry is forced into the surface of the specimen. The resulting impression is observed under a microscope and measured; this measurement is then converted into a hardness number (William D. Callister, 2007). To conduct this test ASTM D570

followed. The test was conducted in a materials engineering laboratory using an HVS50 hardness testing machine. The hardness testing machine is as shown in Fig. 3.17.

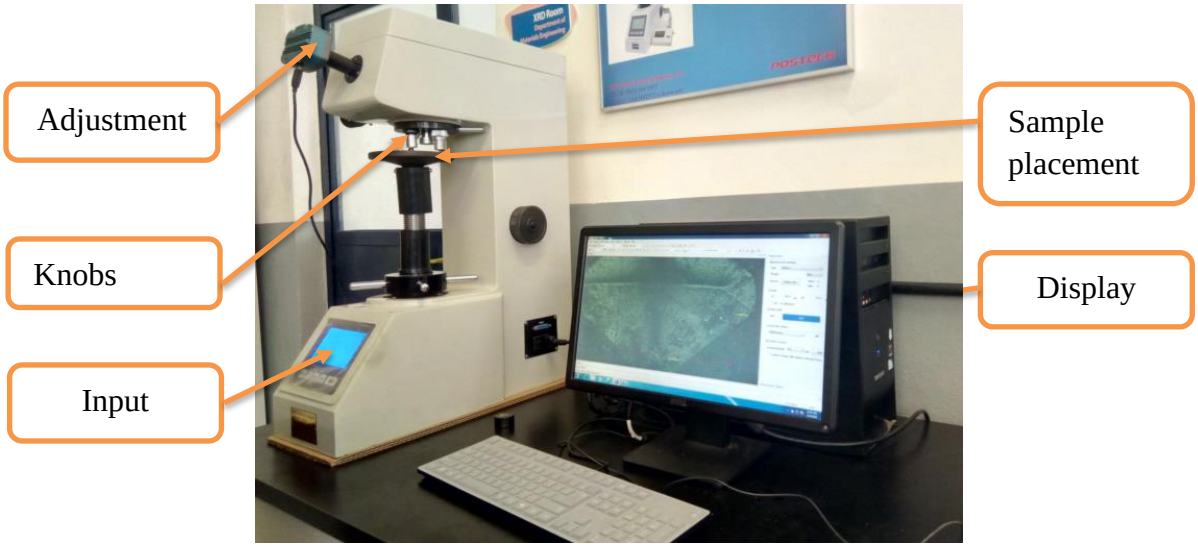


Fig. 3.17 HVS50 hardness testing machine (ASTU material laboratory)

Microstructure observation

The microstructure observation was conducted using scanning electron microscopy in the Biology laboratory, using JCM-6000Plus scanning electron microscopy. The specimen preparation considers the sample's size, shape, state, and conductive properties before sample preparation. The samples were prepared with 20 mm diameter and 5 mm thickness. The samples were cleaned and dried (to avoid water vapor) without changing the texture of the sample. Then the samples were mounted and observed. SEM is as shown in Fig. 3.18.

Fig. 3.18 JCM-6000Plus scanning electron microscope (ASTU biology laboratory)

CHAPTER FOUR

4 RESULT AND DISCUSSION

4.1 Experimental results of tensile strength

Tensile strength is the ability to withstand pulling force, the researcher used to conduct the test using the universal testing machine in Ethiopian Conformity Assessment Enterprise. The gauge length was 200mm and the crosshead was moving at the speed of 5mm/min until fracture. The specimen size has been discussed in the previous chapter, for each sample three specimens were tested and the average was taken. All the specimen's failure was due to brittle fracture since unsaturated polyester is highly brittle.

The tensile strength of coffee husk polyester nano SiO_2 hybrid composite presented in Table 4.1, the sample four with composition 15% treated coffee husk was the strongest. The maximum force resisted was 1867.9 N and the maximum tensile strength was 18.679 N/mm^2 while the maximum elongation was 1.851% at sample 2. Based on the composition of the coffee husk, polyester resin, and nano SiO_2 the properties also vary. The maximum Young's modulus was obtained at sample nine which contains 5% nano-silica. The average tensile forces and average stresses for nine samples are shown in Figures 4.1 and 4.2.

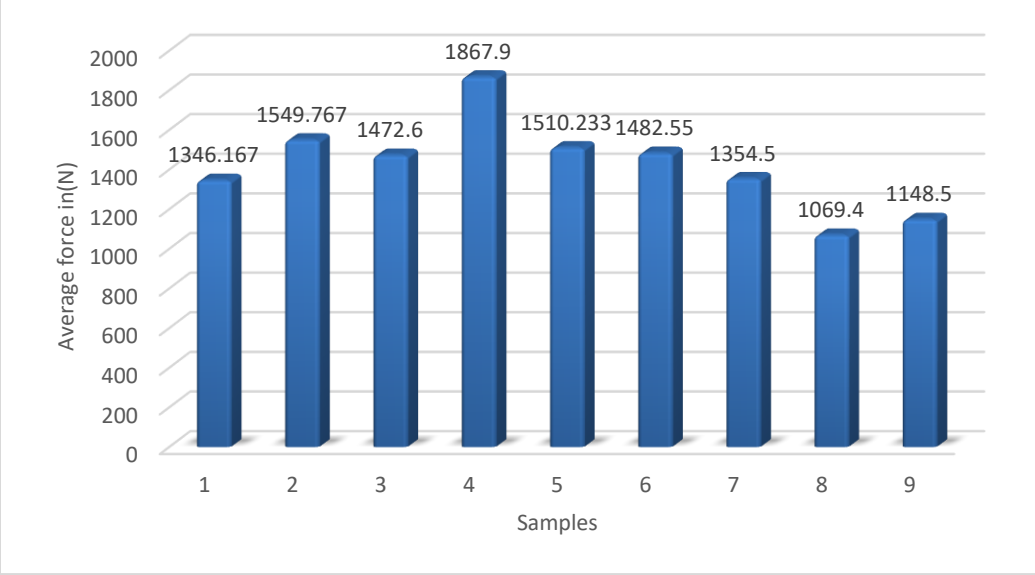


Fig. 4.1 Average force resisted during the experiment for sample 1-9

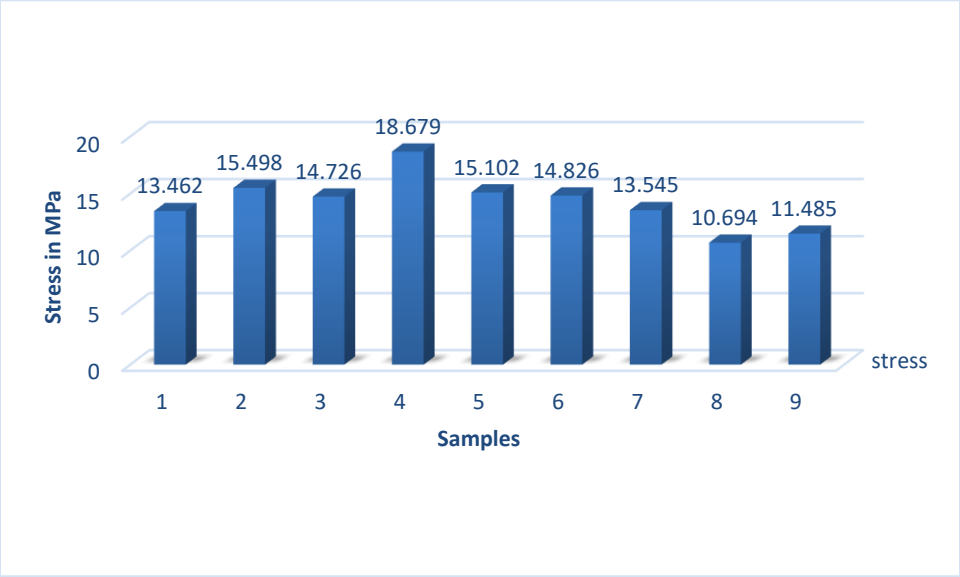


Fig. 4.2 Average stresses for sample 1-9

The average strain, stress, force, and Young's modulus for samples 1-9 are shown in Table 4.1.

Table 4.1 Average strain, stress force, and Young's modulus of each sample (see detail on appendix)

Samples	Strain @ Break (%)	Stress @ Break (N/mm²)	Force @ Break (N)	Youngs Modulus (N/mm²)
S1	1.625	13.462	1346.17	1472.38
S2	1.851	15.498	1549.77	1467.804
S3	1.523	14.726	1472.6	860.378
S4	1.381	18.679	1867.9	1469.777
S5	1.486	15.102	1510.23	1044.054
S6	1.528	14.826	1482.55	1148.308
S7	1.137	13.545	1354.5	1646.28
S8	0.975	10.694	1069.4	1530.174
S9	0.979	11.485	1148.5	2216.951

4.2 Experimental results of impact strength

The specimen was notched at 45 degrees to the depth of 2mm. The energy needed to fracture the specimen is displayed on the manual display board. The tester has two types of scale called inner

and outer scale researcher used the outer scale the numbers after decimal point should be multiplied by 0.3. The impact test result is tabulated in Table 4.2.

Table 4.2 Impact test results

Designation	Outer reading	Inner reading	Interpretation	Toughness (Nm)
S1	3	2	3+2*0.3	3.6
	3	2	3+2*0.3	3.6
	3	1	3+1*0.3	3.3
Average				**3.5**
	3	1	3+1*0.3	3.3
S2	3	1	3+1*0.3	3.3
	3	1	3+1*0.3	3.3
Average				**3.3**
S3	3	1.5	3+1.5*0.3	3.45
	3	1.5	3+1.5*0.3	3.45
	3	1.5	3+1.5*0.3	3.45
Average				**3.45**
S4	3	2	3+2*0.3	3.6
	3	2	3+2*0.3	3.6
	3	2	3+2*0.3	3.6
Average				**3.6**
S5	3	2	3+2*0.3	3.6
	3	2	3+2*0.3	3.6
	3	2	3+2*0.3	3.6
Average				**3.6**
S6	3	2	3+2*0.3	3.6
	3	2	3+2*0.3	3.6
	3	1	3+1*0.3	3.3
Average				**3.5**
S7	3	3	3+3*.3	3.9

	3	2	3+2*.3	3.6
	3	3	3+3*0.3	3.9
Average				**3.8**
S8	3	4	3+4*0.3	4.2
	3	3	3+3*0.3	3.9
	3	3.5	3+3.5*0.3	4.05
Average				**4.05**
S9	3	5	3+5*0.3	4.5
	3	4	3+4*0.3	4.2
	3	3	3+3*0.3	3.9
Average				**4.2**

Discussion

As shown in the Figure 4.3 above the highest impact strength was obtained at sample nine which have compositions of 20% treated coffee husk, 75% unsaturated polyester resin, and 5% nano-silica with an impact energy of 4.2Nm, the next was sample eight with a toughness of 4.05Nm, and sample four was the fourth in the rank with impact strength 3.6Nm. The average impact energy graph is as shown in Fig. 4.3.

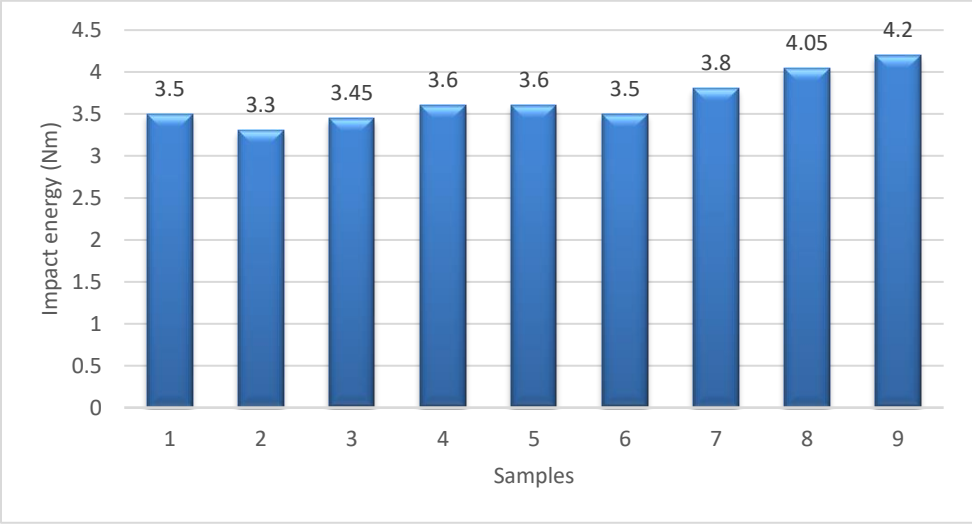


Fig. 4.3 Average impact energy absorbed

4.3 Water absorption test result

When the specimen kept in water specimen absorbed water, the weight of the specimen increased due to absorption of water. The weight gained due to water absorption is measured and tabulated in Table 4.3.

Table 4.3 Water absorption test results

Samples	Day 0	Final weight 24hrs	Weight gain (%wt)	Final weight 48hrs	Weight gain (%wt)	Final weight 72hrs	Weight gain (%wt)
S1	4.531	4.565	0.75	4.565	0.75	4.565	0.75
	4.439	4.515	1.71	4.501	1.39	4.508	1.55
	4.405	4.465	1.36	4.469	1.4	4.464	1.33
	Average		**1.27**		**1.18**		**1.21**
S2	5.568	5.626	1.04	5.609	0.73	5.605	0.66
	5.699	5.745	0.8	5.736	0.64	5.731	0.56
	5.673	5.717	0.77	5.711	0.66	5.711	0.66
	Average		**0.87**		**0.67**		**0.62**
S3	4.29	4.344	1.25	4.335	1.04	4.332	0.97
	4.628	4.683	1.18	4.677	1.05	4.676	1.03
	5.581	5.632	0.91	5.629	0.86	5.624	0.77
	Average		**1.11**		**0.98**		**0.92**
S4	5.798	5.847	0.84	5.84	0.72	5.842	0.75
	6.111	6.144	0.54	6.147	0.58	6.156	0.73
	6.338	6.373	0.55	6.381	0.67	6.396	0.91
	Average		**0.64**		**0.65**		**0.79**
S5	5.702	5.746	0.77	5.757	0.96	5.762	1.05
	4.481	4.53	1.09	4.534	1.18	4.532	1.13
	5.041	5.089	0.95	5.091	0.99	5.096	1.09
	Average		**0.93**		**1.04**		**1.09**
S6	6.068	6.134	1.08	6.134	1.08	6.143	1.23

	5.739	5.816	1.34	5.809	1.21	5.822	1.44
	5.805	5.877	1.24	5.882	1.32	5.888	1.42
	Average		**1.22**		**1.2**		**0.89**
S7	6.592	6.631	0.59	6.637	0.68	6.647	0.83
	6.035	6.081	0.76	6.091	0.92	6.104	1.14
	6.233	6.27	0.59	6.279	0.73	6.291	0.93
	Average		**0.64**		**0.77**		**0.96**
S8	6.436	6.471	0.54	6.478	0.65	6.504	1.05
	6.57	6.604	0.51	6.613	0.65	6.63	0.91
	6.875	6.904	0.42	6.915	0.58	6.937	0.9
	Average		**0.49**		**0.62**		**0.95**
S9	6.553	6.571	0.27	6.575	0.33	6.591	0.57
	6.479	6.515	0.55	6.529	0.77	6.534	0.84
	6.886	6.915	0.42	6.928	0.6	6.937	0.74
	Average		**0.41**		**0.56**		**0.71**

Discussion

The water absorption of the composite is shown in Figure 4.4. The lowest water absorption is better so sample two had the lowest water absorption with 0.62%wt, followed by sample nine with 0.71%wt water absorption value, sample four was the third with a water absorption value of 0.79%wt. Water absorption of coffee husk unsaturated polyester composite is shown in Fig.4.4.

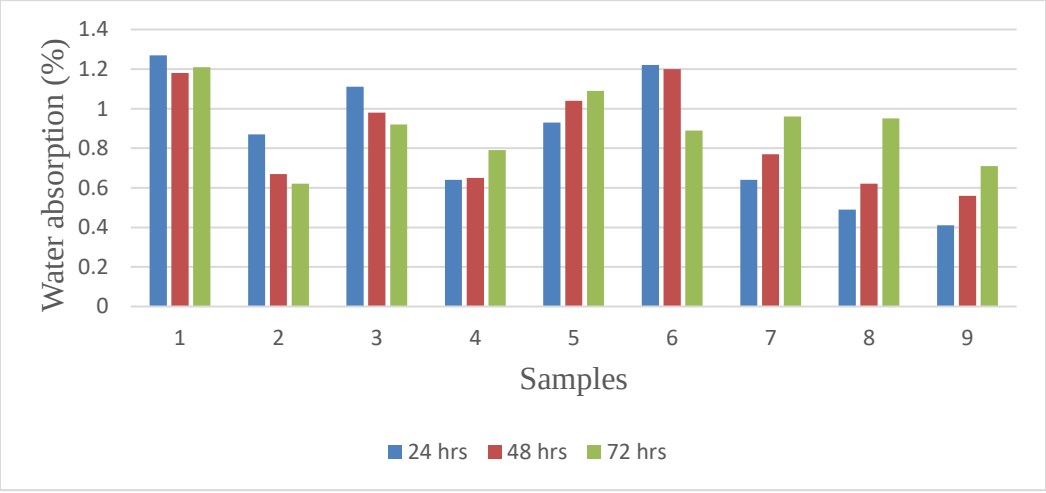


Fig. 4.4 Water absorption of the coffee husk reinforced polyester composite samples.

4.4 Experimental results of hardiness test

The hardness was measured using Vickers hardness testing machine which displays the diamond shape indentation, diagonals, and the result. So, the results are as shown in Table 4.4. The Vickers hardness graph is as shown in Fig. 4.5.

Table 4.4 Vickers hardness value

Samples	S1	S2	S3	S4	S5	S6	S7	S8	S9
Hardness value	4.7	4.0	4.8	6.7	6.5	5.9	6.6	5.8	6.2

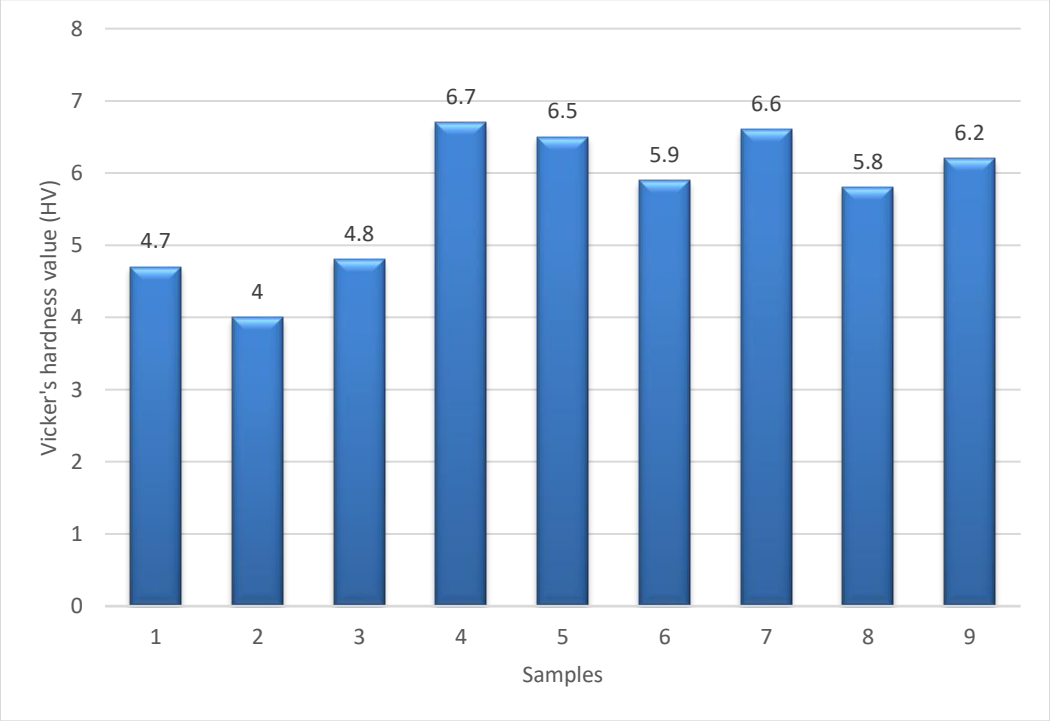
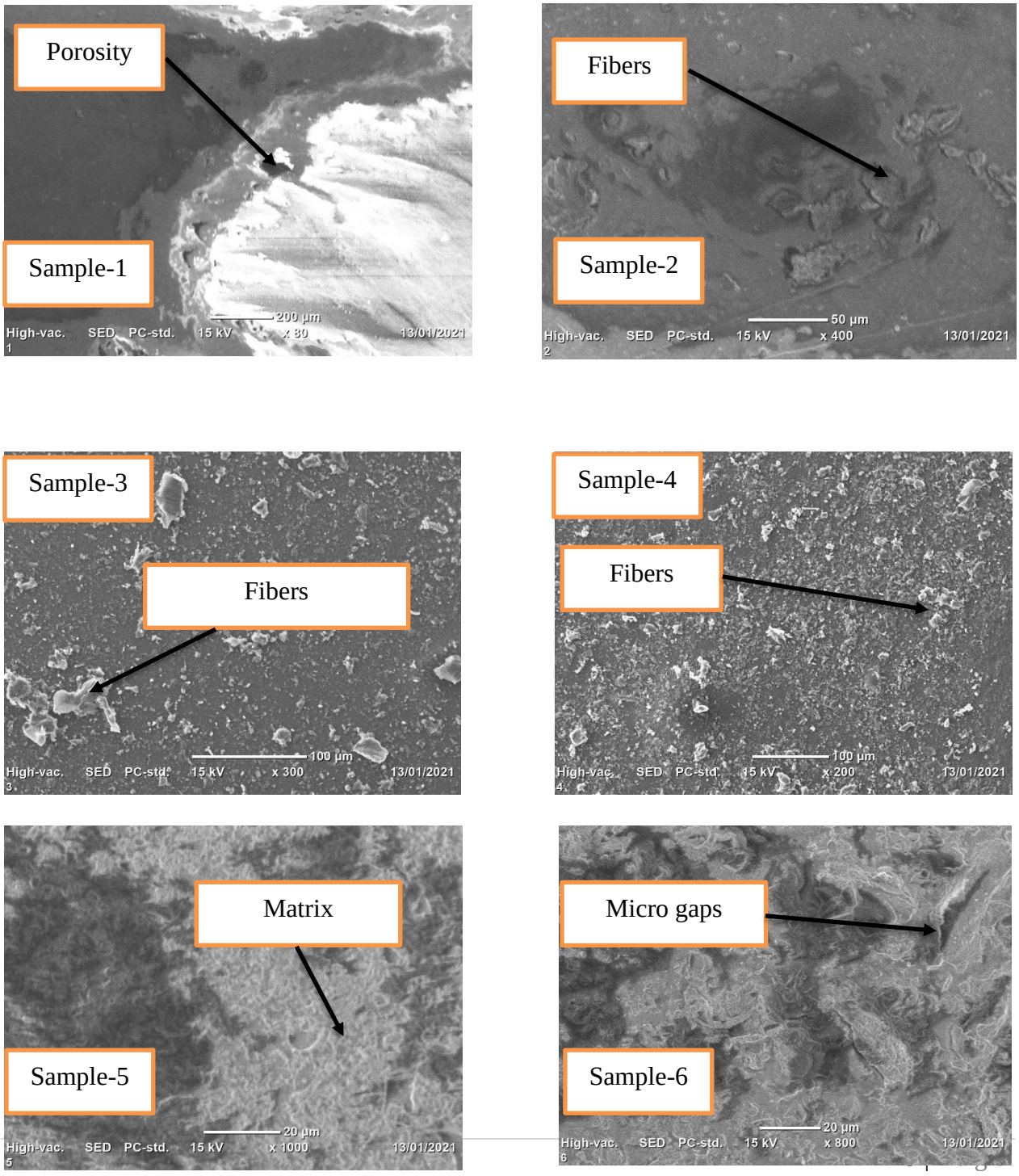


Fig. 4.5 Vickers's hardness value of samples

It was observed from Fig. 4.5 that the highest Vickers hardness number is 6.7 from sample four followed by samples seven, five, and nine with Vickers hardness values of 6.6, 6.5, and 6.2 respectively. This indicates that hardness sample four is higher than other samples. Generally the NaOH treatment and and nano-silica increased the hardness of the composite.

4.5 Microstructure results

The microstructure of the coffee husk polyester composite has been seen using SEM. The images were used to observe the distribution of matrix and substrate powder in the composite, cluster formation, voids, and surface cracks as shown in Fig. 4.6.



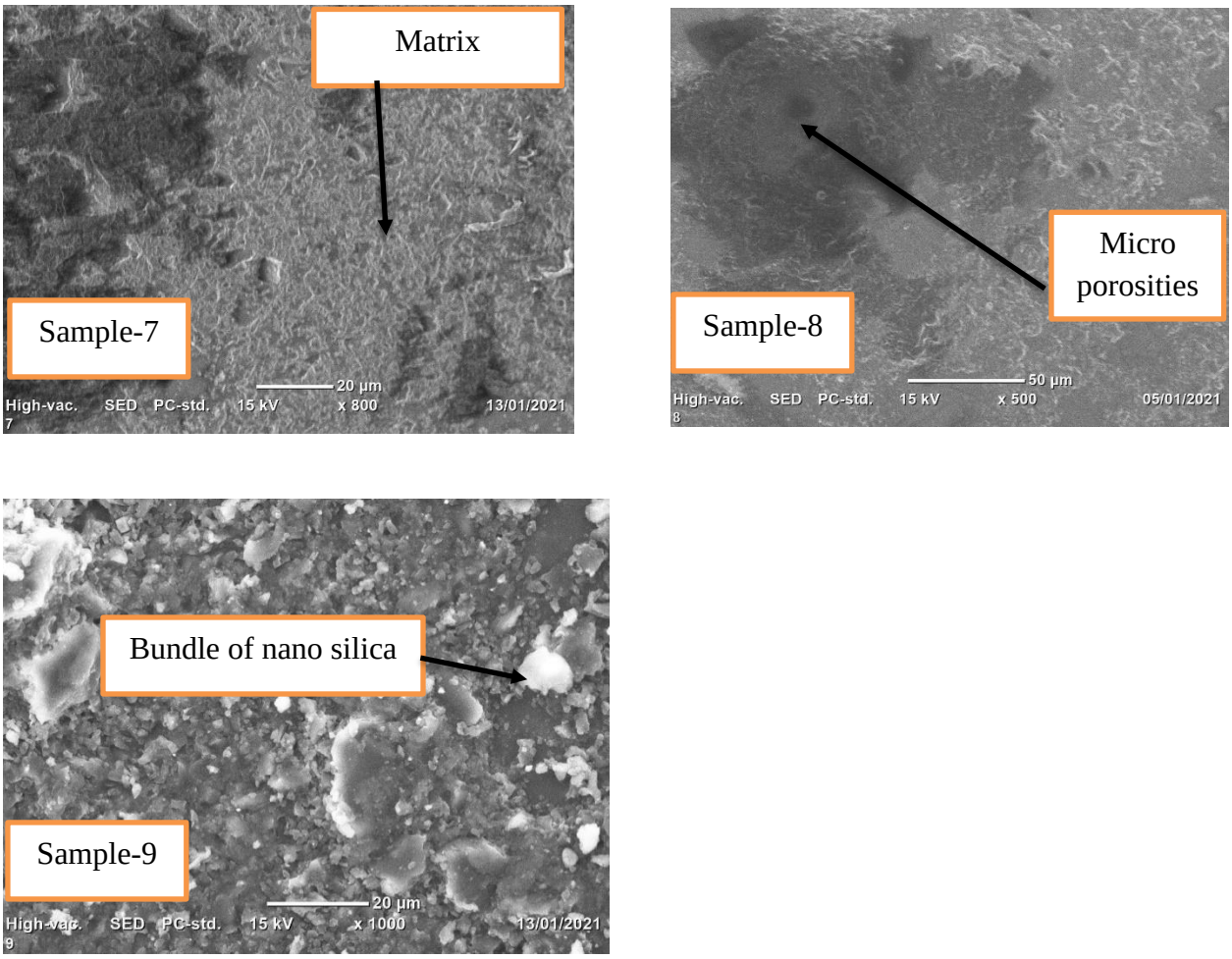


Fig. 4.6 SEM images of sample 1, 2, 3,4, 5, 6, 7, 8, and 9 respectively

4.6 Comparison with the similar previous works

The researchers used pectin and spent coffee ground for reinforcement with weight composition of 85/15%, found the tensile strength of 17Mpa, Young's modulus of 505Mpa (Mendes et al., 2019). When it compared with the present work, the present work showed 1.09x improvement in tensile strength and it showed 2.938x improvement in Young's modulus. Another scholars studied LLDPE and treated coffee husk in weight percentage of 80/20% and found the tensile strength of 15.5 Mpa and Young's modulus of 300Mpa (Yepes et al., 2017). The current work improved by a factor of, tensile strength 1.2x and Young's modulus 4.89x. Another researchers

studied PLA and coffee husk and tea leaves found tensile strength of 19.6 Mpa which is higher than current research, while impact strength is 0.081kJ/m^2 which is too small compared to current work 72kJ/m^2. Generally the current work is much strong than the previous works.The comparison with the previous similar research work is as shown in Table 4.5.

Table 4.5 Comparison with the previous works

Matrix	Reinforcement	%wt	Process	Tensile (Mpa)	Youngs modulus (MPa)	Impact (kJ/m^2)	Reference
Polyester	**Treated CH**	**85/15**	**Casting**	**18.679**	**1469**	**72**	**Current work**
Pectin	SCG	85/15	Continuous casting	17	505	------	(Mendes et al., 2019)
LLDPE	Treated CH	80/20	Injection molding	15.5	300	--------	(Yepes et al., 2017)
PLA	SCG &tea leave	70/30	--------	19.6	------	0.081	(Songtipya et al., 2019)
HDPE	CH	85/15	Compression casting	18.99	1413	----	(Wang et al., 2019)

CHAPTER FIVE

5 CONCLUSION AND FUTURE WORK

5.1 Conclusion

Natural fibers and agro-wastes are attracting attention due to their availability, environmentally friendly, cheap, and lightweight. Previous works in this area show the way of incorporation of natural fibers, like jute, sisal, banana, and so on using different matrices like epoxy and polyester. They showed the use of agricultural wastes such as rice husk, coconut husk, stalks, and spent coffee ground as filler and reinforcement. In this work, the researcher investigated the performance of coffee husk and unsaturated polyester resin matrix composite. The coffee husk was purchased from a local supplier and processed to be used in the composite. Then the composite manufactured on nine different composition sets namely, untreated coffee husk reinforcement and unsaturated polyester matrix (15/85, 20/80, 25/75) %wt, with treated coffee husk (15/85, 20/80, 25/75) %wt, and finally treated coffee husk and nano-silica (20/79/1, 20/77/3, 20/75/5) %wt. These sets of samples were experimented with, and finally treated coffee husk (15/85) %wt was found best. But the composites which contain nano-silica has better impact strength compared to other samples. Based on data analysis in the previous chapter confirm that treated coffee husk has superior performance. The addition of nano-silica improved water absorption and impact strength while decreased tensile strength.

Finally, the results confirmed that coffee husk can be used as reinforcement in the unsaturated polyester matrix by using a combination of other natural or synthetic fibers. Sample four is best at tensile strength and water absorption properties. The treated 15/85wt% have been found that 18.67Mpa tensile strength, 6.7 Vickers hardness value, 0.79%wt water absorption, and 3.6Nm impact strength. In terms of micro-structure also sample four is best with uniform distribution of coffee husk fiber without porosity. From all the results and comparisons, the researcher can conclude that the fabricated coffee husk reinforced polyester composite has a good mechanical property and it is recommended to use it for light-load applications including the production of the internal door panel. These composites can be used for indoor applications like false ceilings, windows, bathroom, and furniture.

Genarlly the treated coffee husk polyester composite is stronger comparative to the thermoplastic matrix composites. So, the treated coffee husk polyester composite is recommended to use in manufacturing composite that can decrease the cost of composite, for indoor application.

5.2 Future work

This work mainly addressed the performance of coffee husk reinforced unsaturated polyester composite. But there are different areas to improve the mechanical properties of the coffee husk reinforced polyester composite. Here the following topics are suggested for further studies, such:

- ✓ Characterization of the coffee husk fine powder type;
- ✓ Investigate the mechanical property of coffee husk in combination with other types of natural and synthetic fibers;
- ✓ Using different technique for the treatment of coffee husk;
- ✓ Using coffee cellulose as nano reinforcement;
- ✓ Investigate the coffee husk fiber with other types of the polymer matrix.

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Appendix

Tensile test results

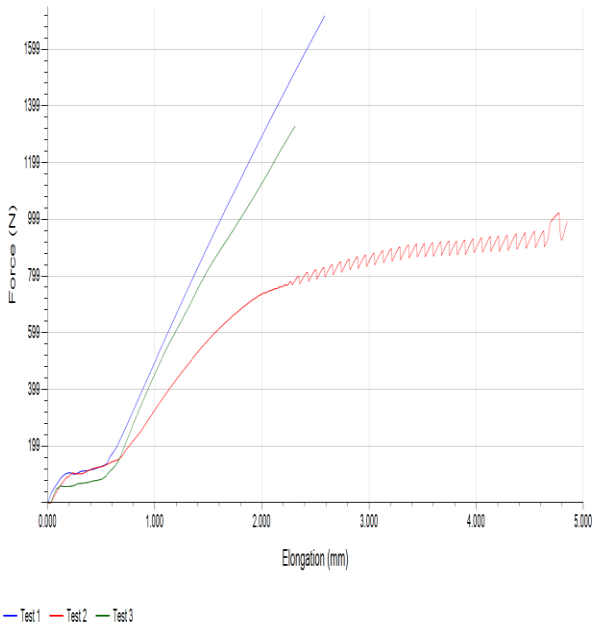
TEST REPORT

SAMPLE TYPE : composite	**Test Name : composite**
LDS : 1309022	**Test Type : Tensile**
CUSTOMER : Engdasew	**Test Date : 31/12/2020 10:07**
ANALYST : Mengistu	**Test Speed : 5.000 mm/min**
STANDARD : As per customer request	
	Width : 20.000 mm
	Thickness : 5.000 mm
	Sample Length : 200.000 mm

Comments :

Test No.	**Strain @ Break (%)**	**Stress @ Break (N/mm²)**	**Force @ Break (N)**	**Elong. @ Break (mm)**	**Force @ Peak (N)**	**Youngs Modulus (N/mm²)**	**Tensile Strength (N/mm)**
1	1.294	17.172	1717.2	2.587	1717.2	1693.265	85.86
2	2.425	9.928	992.8	4.85	1022.5	944.632	51.125
3	1.155	13.285	1328.5	2.31	1328.5	1779.243	66.425
Mean	1.625	13.462	1346.167	3.249	1356.067	1472.38	67.803
Max	2.425	17.172	1717.2	4.85	1717.2	1779.243	85.86
S.D.	0.697	3.625	362.523	1.393	348.169	459.061	17.408
C. of V.	42.887	26.93	26.93	42.887	25.675	31.178	25.675

Test No.	Elong. @ Peak (mm)	Stress @ Yield (N/mm^2)
1	2.587	17.172
2	4.772	7.692
3	2.31	13.285
Mean	3.223	12.716
Max	4.772	17.172
S.D.	1.349	4.766
C. of V.	41.843	37.476



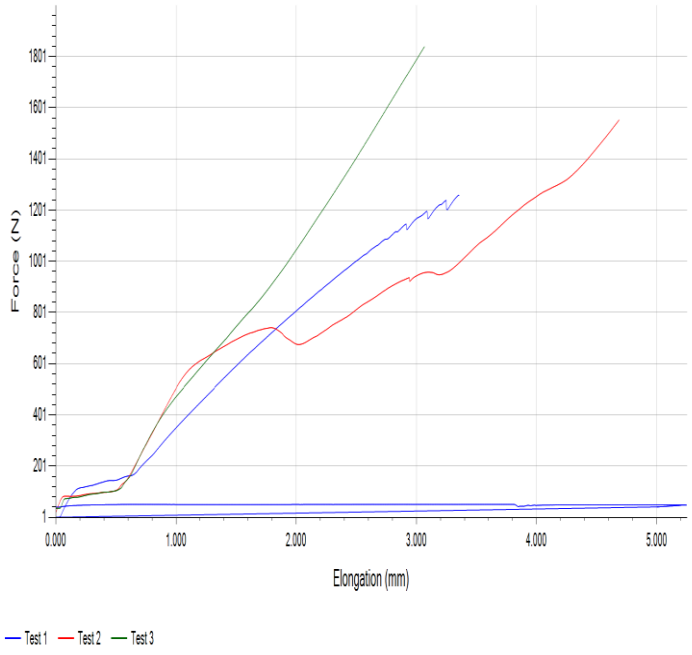
TEST REPORT

SAMPLE TYPE : composite	**Test Name : composite**
LDS : 1309023	**Test Type : Tensile**
CUSTOMER : Engdasew	**Test Date : 31/12/2020 10:27**
ANALYST : Mengistu	**Test Speed : 5.000 mm/min**
STANDARD : As per customer request	**Pretension : Off**
	Width : 20.000 mm
	Thickness : 5.000 mm
	Sample Length : 200.000 mm

Comments :

Test No	Strain @ Break (%)	Stress @ Break (N/mm²)	Force @ Break (N)	Elong. @ Break (mm)	Force @ Peak (N)	Youngs Modulus (N/mm²)	Tensile Strength (N/mm)
1	1.678	12.573	1257.3	3.355	1258.7	1138.403	62.935
2	2.343	15.531	1553.1	4.686	1553.1	1794.93	77.655
3	1.534	18.389	1838.9	3.067	1838.9	1470.079	91.945
Mean	1.851	15.498	1549.767	3.703	1550.233	1467.804	77.512
Max	2.343	18.389	1838.9	4.686	1838.9	1794.93	91.945
S.D.	0.432	2.908	290.814	0.864	290.111	328.269	14.506
C. of V.	23.326	18.765	18.765	23.326	18.714	22.365	18.714

Test No.	Elong. @ Peak (mm)	Stress @ Yield (N/mm²)
1	3.351	11.457
2	4.686	7.41
3	3.067	18.389
Mean	3.701	12.419
Max	4.686	18.389
S.D.	0.864	5.552
C. of V.	23.356	44.709



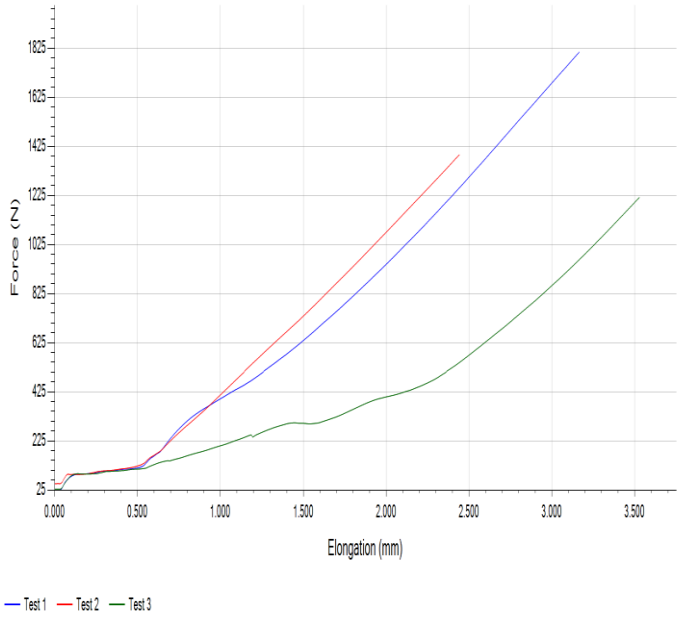
TEST REPORT

SAMPLE TYPE : composite	**Test Name : composite**
LDS : 1309024	**Test Type : Tensile**
CUSTOMER : Engdasew	**Test Date : 31/12/2020 11:12**
ANALYST : Mengistu	**Test Speed : 5.000 mm/min**
STANDARD : As per customer request	**Pretension : Off**
	Width : 20.000 mm
	Thickness : 5.000 mm
	Sample Length : 200.000 mm

Comments :

Test No.	Strain @ Break (%)	Stress @ Break (N/mm^2)	Force @ Break (N)	Elong. @ Break (mm)	Force @ Peak (N)	Youngs Modulus (N/mm^2)	Tensile Strength (N/mm)
1	1.583	18.097	1809.7	3.165	1809.7	816.347	90.485
2	1.221	13.908	1390.8	2.442	1390.8	1285.833	69.54
3	1.764	12.173	1217.3	3.528	1217.3	478.955	60.865
Mean	1.523	14.726	1472.6	3.045	1472.6	860.378	73.63
Max	1.764	18.097	1809.7	3.528	1809.7	1285.833	90.485
S.D.	0.276	3.046	304.554	0.553	304.554	405.237	15.228
C. of V.	18.156	20.681	20.681	18.156	20.681	47.1	20.681

Test No.	Elong. @ Peak (mm)	Stress @ Yield (N/mm²)
1	3.165	18.097
2	2.442	13.908
3	3.528	2.496
Mean	3.045	11.5
Max	3.528	18.097
S.D.	0.553	8.074
C. of V.	18.156	70.206



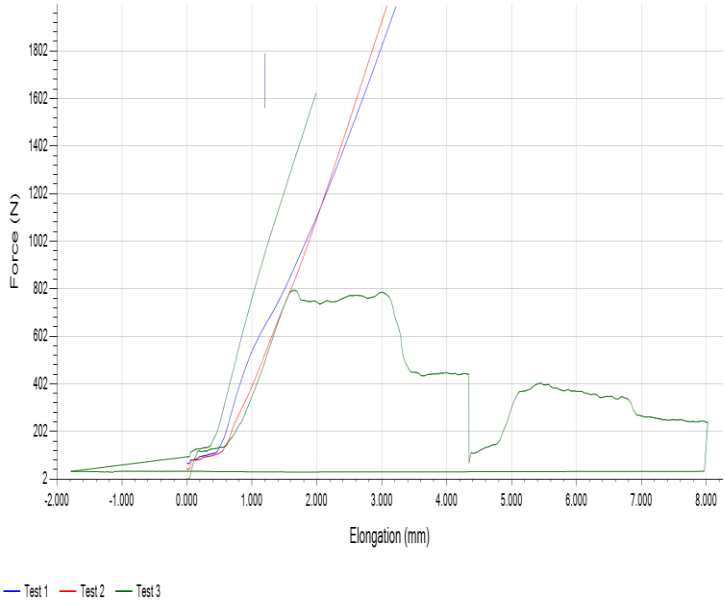
TEST REPORT

SAMPLE TYPE : composite	**Test Name : composite**
LDS : 1309025	**Test Type : Tensile**
CUSTOMER : Engdasew	**Test Date : 31/12/2020 11:20**
ANALYST : Mengistu	**Test Speed : 5.000 mm/min**
STANDARD : As per customer request	**Pretension : Off**
	Width : 20.000 mm
	Thickness : 5.000 mm
	Sample Length : 200.000 mm

Comments :

Test No.	Strain @ Break (%)	Stress @ Break (N/mm²)	Force @ Break (N)	Elong. @ Break (mm)	Force @ Peak (N)	Youngs Modulus (N/mm²)	Tensile Strength (N/mm)
1	1.608	19.88	1988.0	3.216	1988.0	1624.111	99.4
2	1.54	19.915	1991.5	3.08	1991.5	1387.886	99.575
3	0.994	16.242	1624.2	1.987	1624.2	1397.334	81.21
Mean	1.381	18.679	1867.9	2.761	1867.9	1469.777	93.395
Max	1.608	19.915	1991.5	3.216	1991.5	1624.111	99.575
S.D.	0.337	2.111	211.058	0.674	211.058	133.741	10.553
C. of V.	24.402	11.299	11.299	24.402	11.299	9.099	11.299

Test No.	Elong. @ Peak (mm)	Stress @ Yield (N/mm²)
1	3.216	19.88
2	3.08	19.915
3	1.987	7.957
Mean	2.761	15.917
Max	3.216	19.915
S.D.	0.674	6.894
C. of V.	24.402	43.31



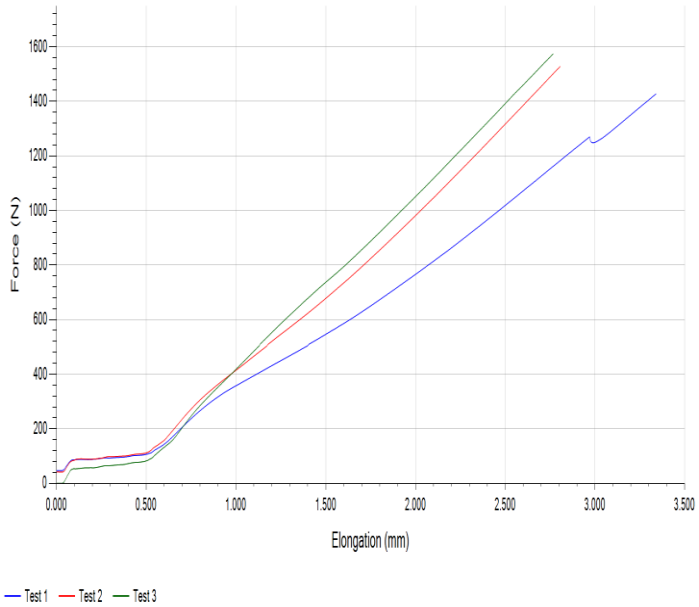
TEST REPORT

SAMPLE TYPE : composite	**Test Name : composite**
LDS : 1309026	**Test Type : Tensile**
CUSTOMER : Engdasew	**Test Date : 31/12/2020 11:39**
ANALYST : Mengistu	**Test Speed : 5.000 mm/min**
STANDARD : As per customer request	**Pretension : Off**
	Width : 20.000 mm
	Thickness : 5.000 mm
	Sample Length : 200.000 mm

Comments :

Test No.	**Strain @ Break (%)**	**Stress @ Break (N/mm²)**	**Force @ Break (N)**	**Elong. @ Break (mm)**	**Force @ Peak (N)**	**Youngs Modulus (N/mm²)**	**Tensile Strength (N/mm)**
1	1.67	14.279	1427.9	3.34	1427.9	764.399	71.395
2	1.403	15.282	1528.2	2.806	1528.2	1050.403	76.41
3	1.384	15.746	1574.6	2.767	1574.6	1317.36	78.73
Mean	1.486	15.102	1510.233	2.971	1510.233	1044.054	75.512
Max	1.67	15.746	1574.6	3.34	1574.6	1317.36	78.73
S.D.	0.16	0.75	74.982	0.32	74.982	276.535	3.749
C. of V.	10.776	4.965	4.965	10.776	4.965	26.487	4.965

Test No.	Elong. @ Peak (mm)	Stress @ Yield (N/mm^2)
1	3.34	12.701
2	2.806	15.282
3	2.767	15.746
Mean	2.971	14.576
Max	3.34	15.746
S.D.	0.32	1.641
C. of V.	10.776	11.255



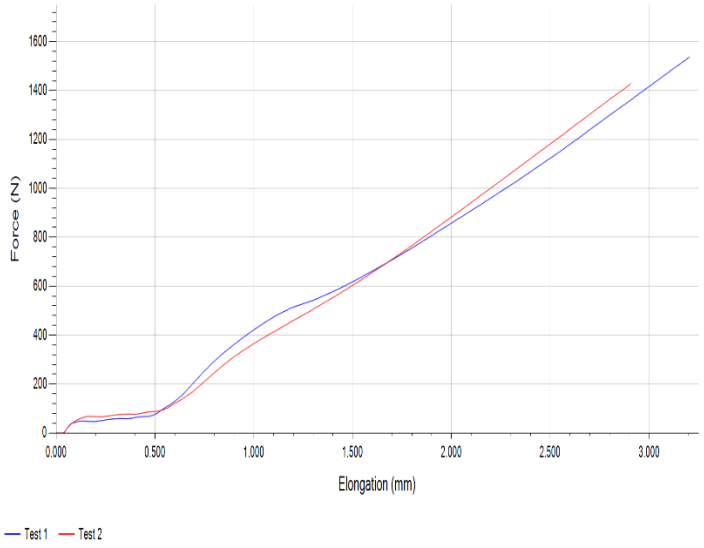
TEST REPORT

SAMPLE TYPE : composite	**Test Name : composite**
LDS : 1309027	**Test Type : Tensile**
CUSTOMER : Engdasew	**Test Date : 31/12/2020 11:45**
ANALYST : Mengistu	**Test Speed : 5.000 mm/min**
STANDARD : As per customer request	**Pretension : Off**
	Width : 20.000 mm
	Thickness : 5.000 mm
	Sample Length : 200.000 mm

Comments :

Test No.	Strain @ Break (%)	Stress @ Break (N/mm²)	Force @ Break (N)	Elong. @ Break (mm)	Force @ Peak (N)	Youngs Modulus (N/mm²)	Tensile Strength (N/mm)
1	1.603	15.378	1537.8	3.206	1537.8	1329.269	76.89
2	1.454	14.273	1427.3	2.907	1427.3	967.347	71.365
Mean	1.528	14.826	1482.55	3.057	1482.55	1148.308	74.128
Max	1.603	15.378	1537.8	3.206	1537.8	1329.269	76.89
S.D.	0.106	0.781	78.135	0.211	78.135	255.918	3.907
C. of V.	6.917	5.27	5.27	6.917	5.27	22.287	5.27

Test No.	Elong. @ Peak (mm)	Stress @ Yield (N/mm²)
1	3.206	15.378
2	2.907	14.273
Mean	3.057	14.826
Max	3.206	15.378
S.D.	0.211	0.781
C. of V.	6.917	5.27



TEST REPORT

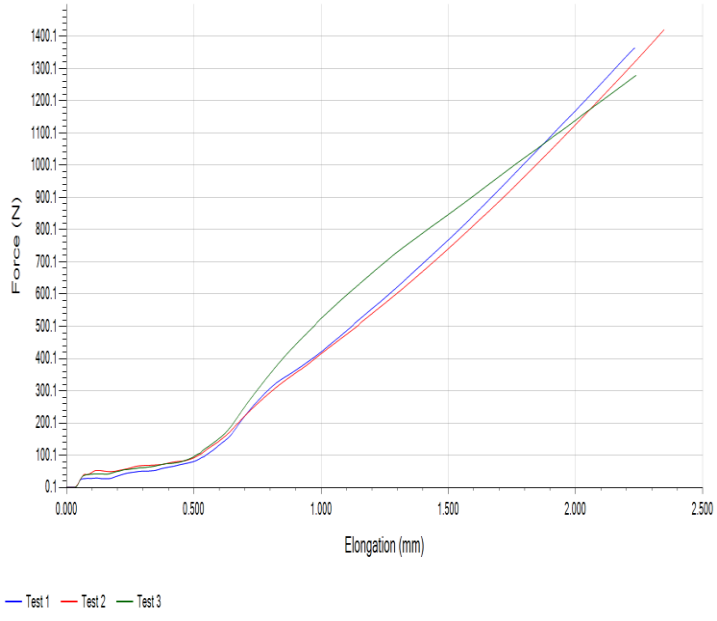
SAMPLE TYPE : composite	**Test Name : composite ISO 6892**
LDS : 1309027	**Test Type : Tensile**
CUSTOMER : Engdasew	**Test Date : 31/12/2020 11:51**
ANALYST : Mengistu	**Test Speed : 5.000 mm/min**
STANDARD : As per customer request	**Pretension : Off**

	Width : 20.000 mm
	Thickness : 5.000 mm
	Sample Length : 200.000 mm

Comments :

Test No.	**Strain @ Break (%)**	**Stress @ Break (N/mm²)**	**Force @ Break (N)**	**Elong. @ Break (mm)**	**Force @ Peak (N)**	**Youngs Modulus (N/mm²)**	**Tensile Strength (N/mm)**
1	1.117	13.636	1363.6	2.233	1363.6	1688.076	68.18
2	1.174	14.209	1420.9	2.348	1420.9	1195.085	71.045
3	1.119	12.79	1279.0	2.238	1279.0	2055.678	63.95
Mean	1.137	13.545	1354.5	2.273	1354.5	1646.28	67.725
Max	1.174	14.209	1420.9	2.348	1420.9	2055.678	71.045
S.D.	0.032	0.714	71.386	0.065	71.386	431.816	3.569
C. of V.	2.86	5.27	5.27	2.86	5.27	26.23	5.27

Test No.	**Elong. @ Peak (mm)**	**Stress @ Yield (N/mm²)**
1	2.233	13.636
2	2.348	14.209
3	2.238	12.79
Mean	2.273	13.545
Max	2.348	14.209
S.D.	0.065	0.714
C. of V.	2.86	5.27



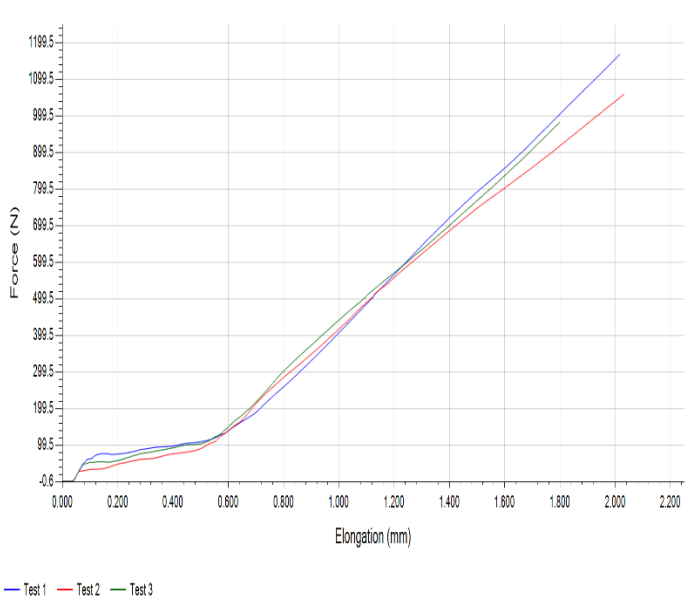
TEST REPORT

SAMPLE TYPE : composite	**Test Name : composite**
LDS : 1309028	**Test Type : Tensile**
CUSTOMER : Engdasew	**Test Date : 31/12/2020 11:59**
ANALYST : Mengistu	**Test Speed : 5.000 mm/min**
STANDARD : As per customer request	**Pretension : Off**
	Width : 20.000 mm
	Thickness : 5.000 mm
	Sample Length : 200.000 mm

Comments :

Test No.	Strain @ Break (%)	Stress @ Break (N/mm^2)	Force @ Break (N)	Elong. @ Break (mm)	Force @ Peak (N)	Youngs Modulus (N/mm^2)	Tensile Strength (N/mm)
1	1.009	11.679	1167.9	2.017	1167.9	1549.916	58.395
2	1.016	10.583	1058.3	2.032	1058.3	1364.65	52.915
3	0.9	9.82	982.0	1.799	982.0	1675.955	49.1
Mean	0.975	10.694	1069.4	1.949	1069.4	1530.174	53.47
Max	1.016	11.679	1167.9	2.032	1167.9	1675.955	58.395
S.D.	0.065	0.934	93.446	0.13	93.446	156.589	4.672
C. of V.	6.69	8.738	8.738	6.69	8.738	10.233	8.738

Test No.	Elong. @ Peak (mm)	Stress @ Yield (N/mm^2)
1	2.017	11.679
2	2.032	10.583
3	1.799	9.82
Mean	1.949	10.694
Max	2.032	11.679
S.D.	0.13	0.934
C. of V.	6.69	8.738



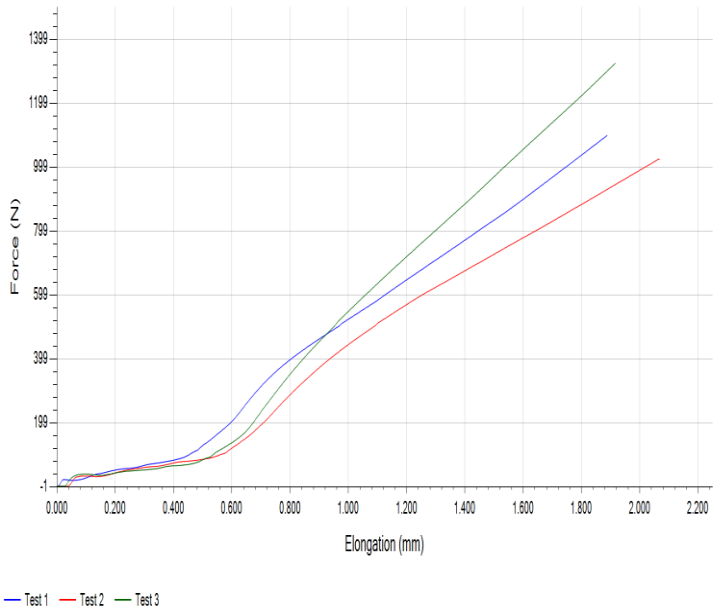
TEST REPORT

SAMPLE TYPE : composite	**Test Name : composite**
LDS : 1309030	**Test Type : Tensile**
CUSTOMER : Engdasew	**Test Date : 31/12/2020 12:06**
ANALYST : Mengistu	**Test Speed : 5.000 mm/min**
STANDARD : As per customer request	**Pretension : Off**
	Width : 20.000 mm
	Thickness : 5.000 mm
	Sample Length : 200.000 mm

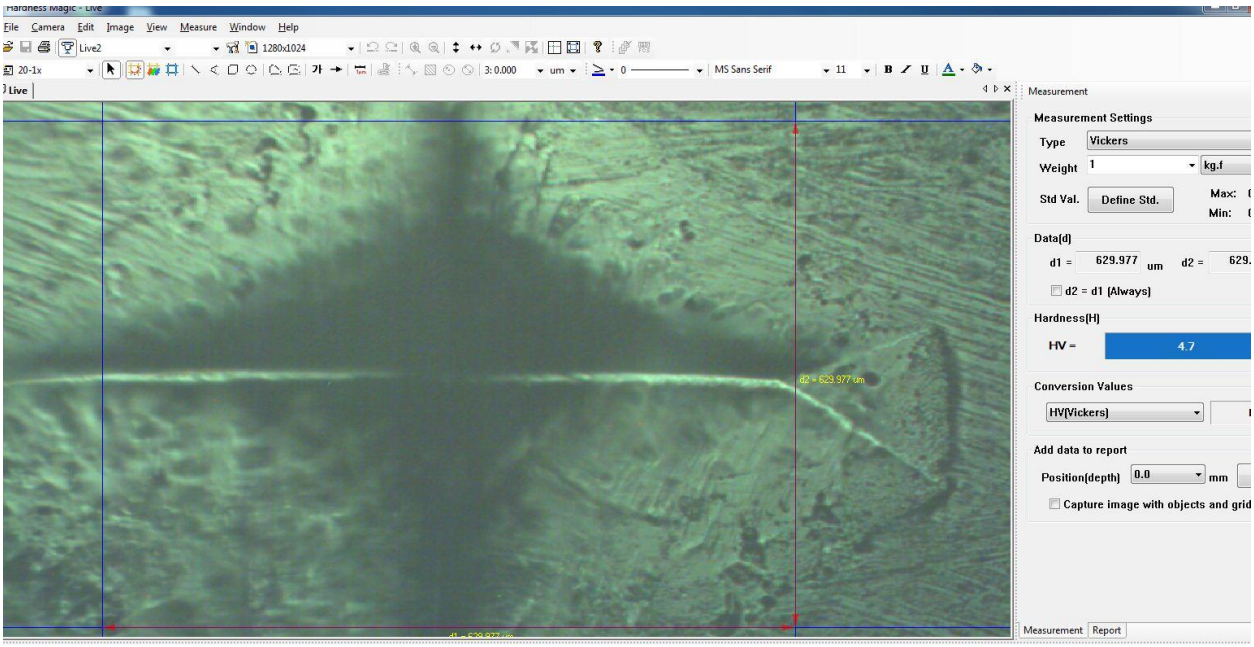
Comments :

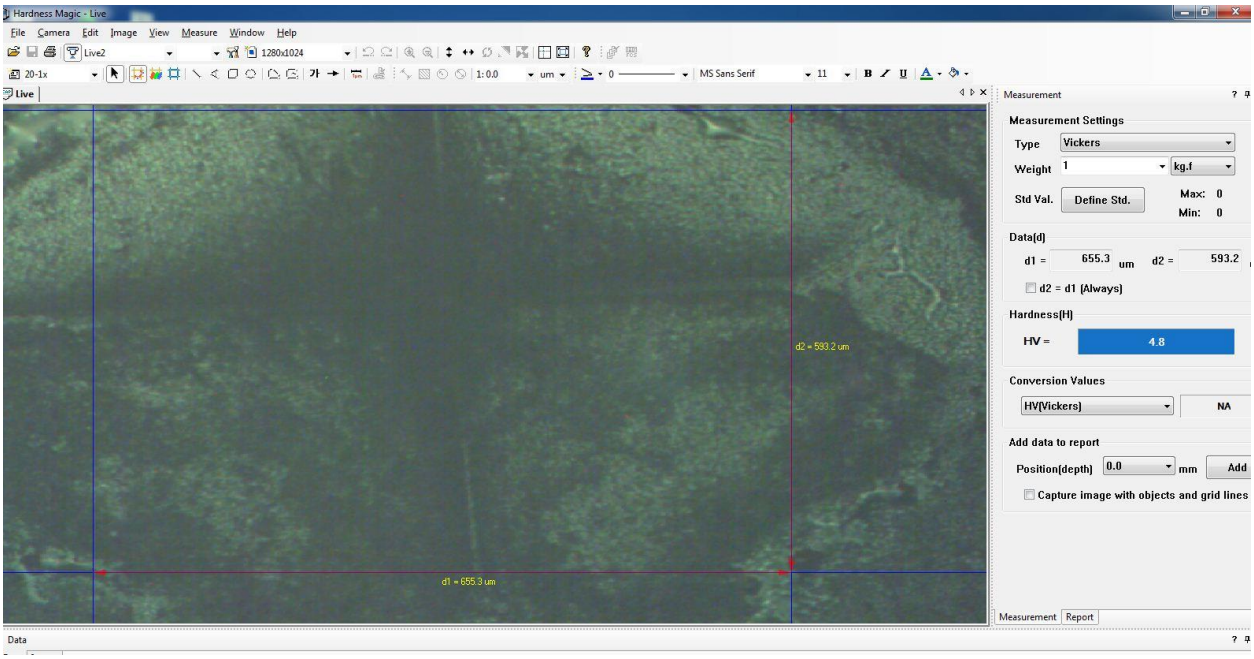
Test No.	Strain @ Break (%)	Stress @ Break (N/mm²)	Force @ Break (N)	Elong. @ Break (mm)	Force @ Peak (N)	Youngs Modulus (N/mm²)	Tensile Strength (N/mm)
1	0.944	10.981	1098.1	1.888	1098.1	2332.587	54.905
2	1.034	10.232	1023.2	2.068	1023.9	1977.331	51.195
3	0.958	13.242	1324.2	1.916	1324.2	2340.935	66.21
Mean	0.979	11.485	1148.5	1.957	1148.733	2216.951	57.437
Max	1.034	13.242	1324.2	2.068	1324.2	2340.935	66.21
S.D.	0.048	1.567	156.702	0.097	156.422	207.559	7.821
C. of V.	4.948	13.644	13.644	4.948	13.617	9.362	13.617

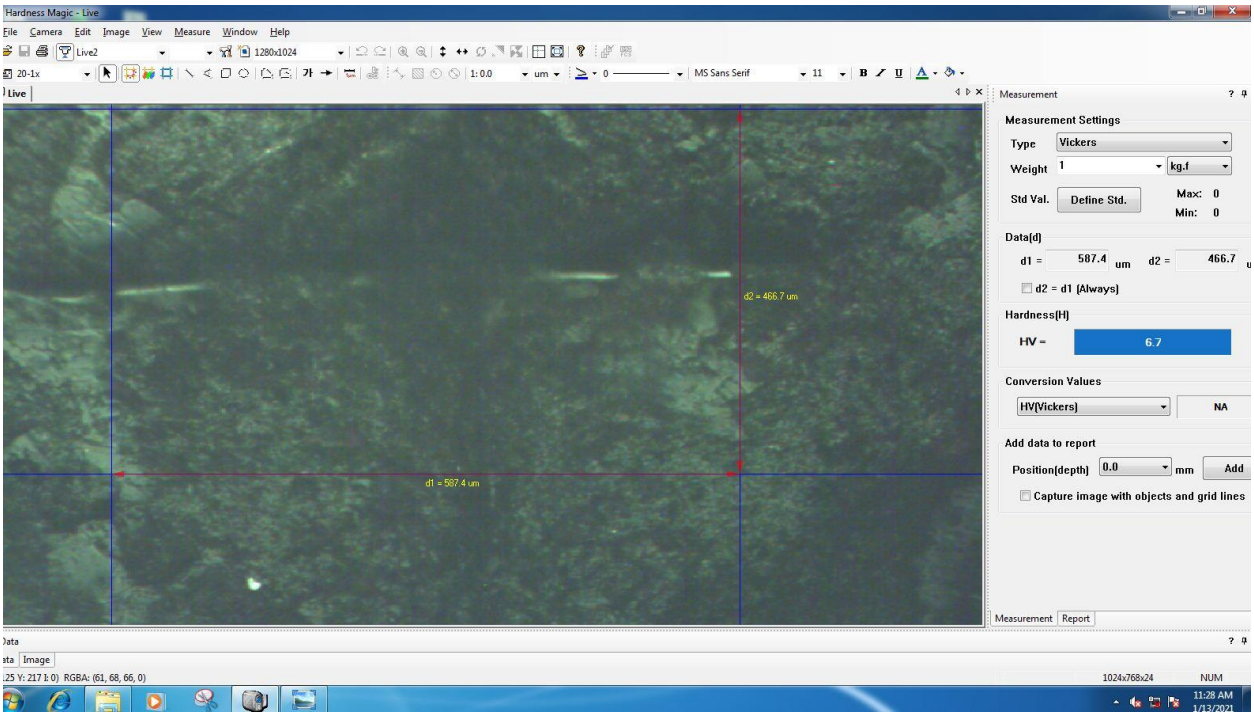
Test No.	Elong. @ Peak (mm)	Stress @ Yield (N/mm²)
1	1.888	10.981
2	2.064	10.239
3	1.916	13.242
Mean	1.956	11.487
Max	2.064	13.242
S.D.	0.095	1.564
C. of V.	4.835	13.617

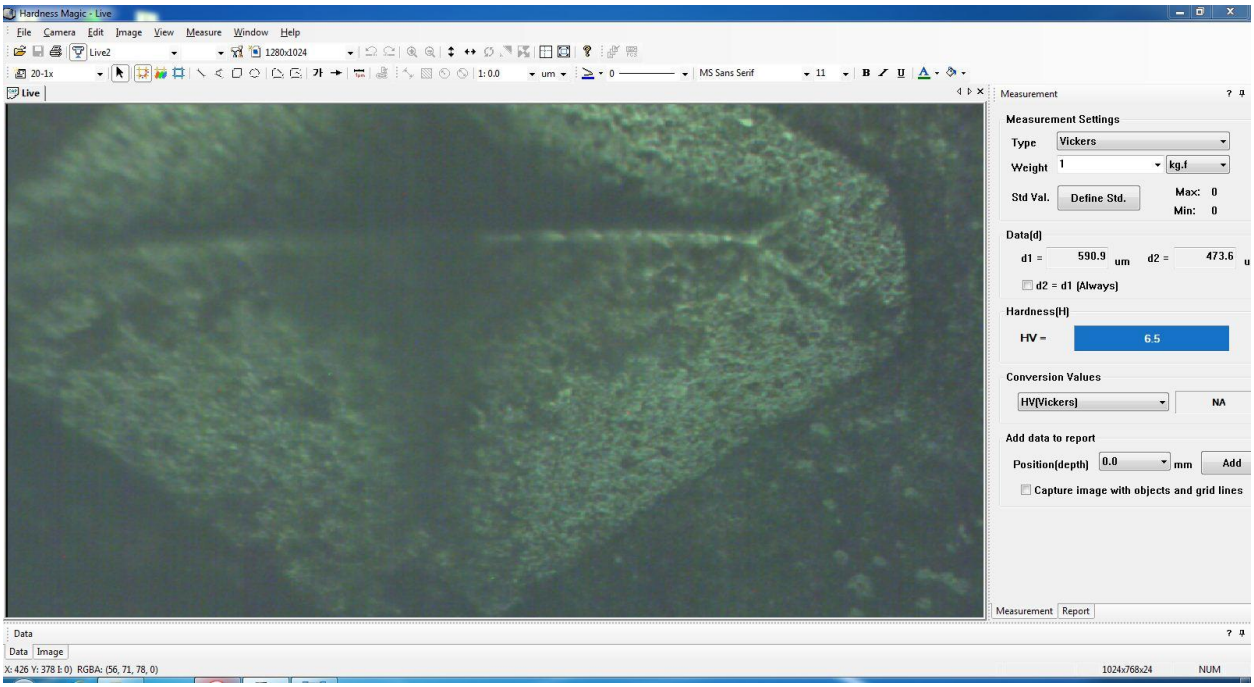


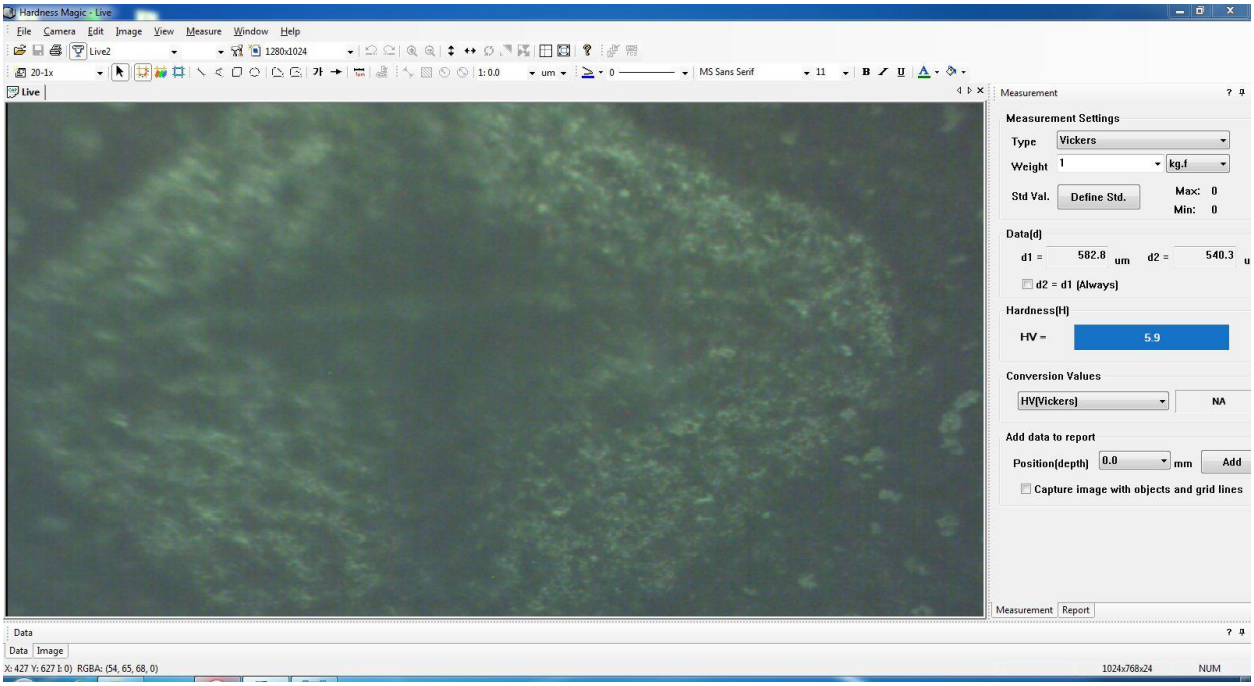
Hardness test results starting from sample one to sample nine in order**.**

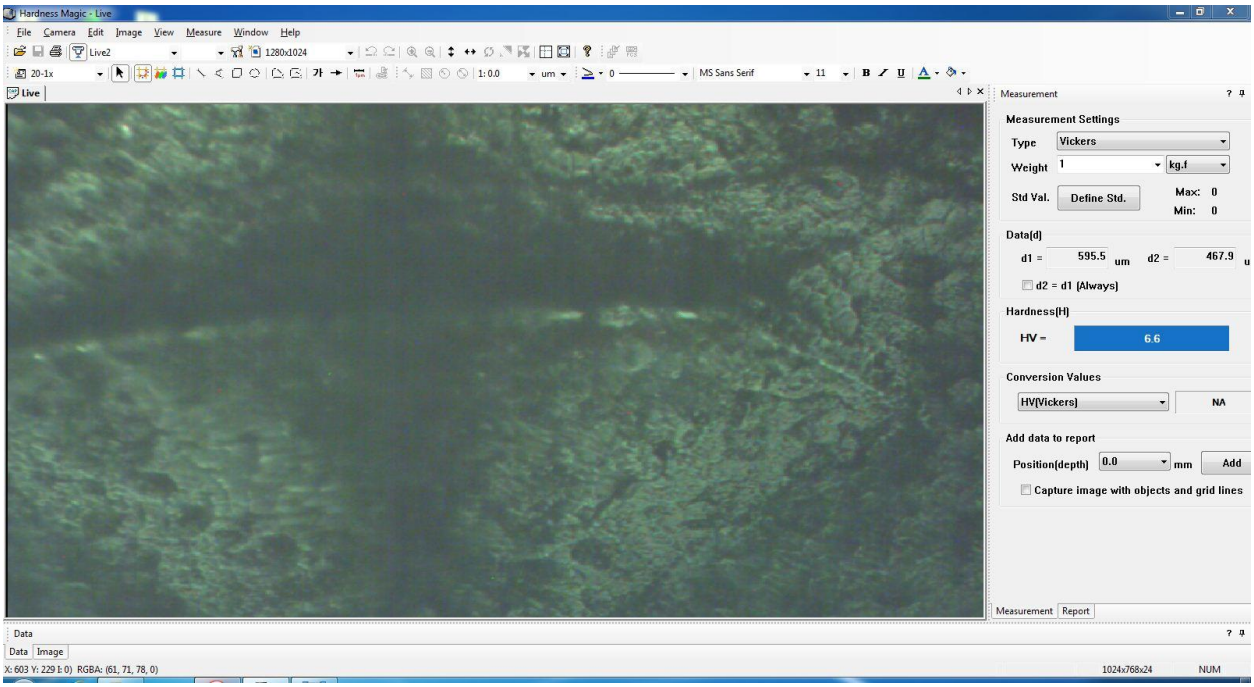


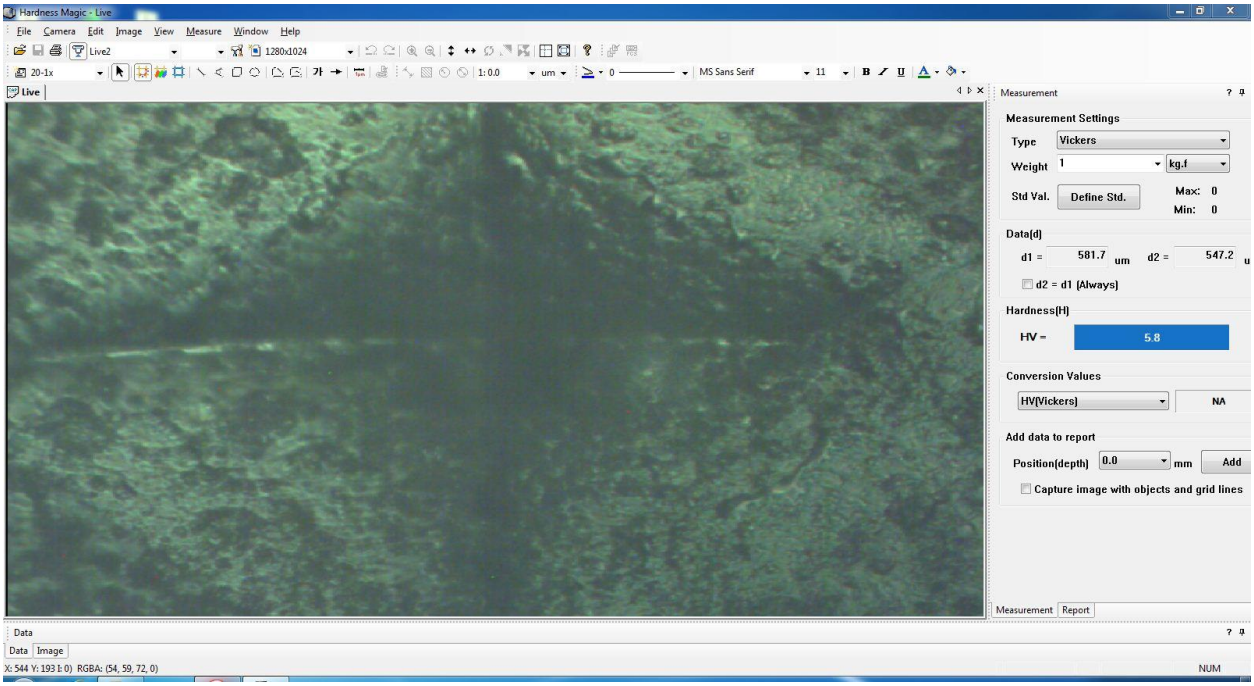

Hardness Magic - Live
File Camera Edit Image View Measure Window Help
Live
d2 = 593.2 um
d1 = 655.3 um
Measurement
Measurement Settings
Type Vickers
Weight 1 kg.f
Std Val. Define Std.
Max: 0
Min: 0
Data[d]
d1 = 655.3 um d2 = 593.2
d2 = d1 (Always)
Hardness[H]
HV = 4.8
Conversion Values
HV(Vickers) NA
Add data to report
Position(depth) 0.0 mm Add
Capture image with objects and grid lines
Measurement Report
Data

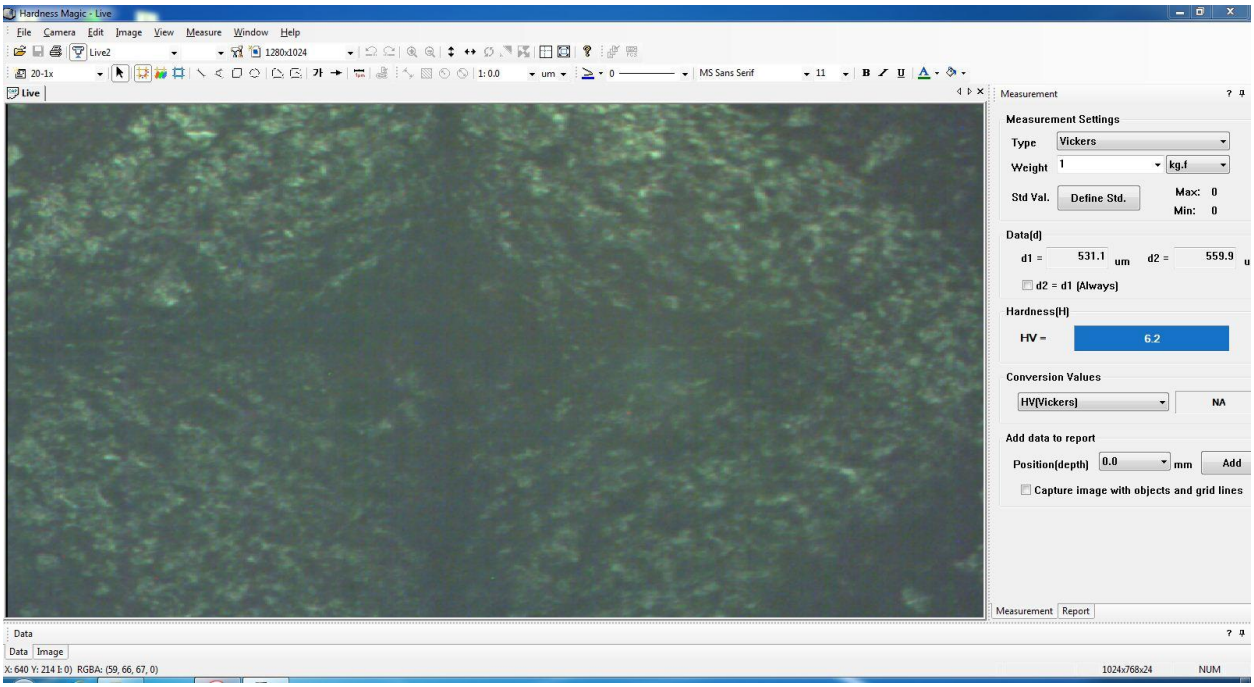

Hardness Magic - Live
File Camera Edit Image View Measure Window Help
Live
d2 = 466.7 um
d1 = 587.4 um
Measurement
Measurement Settings
Type Vickers
Weight 1 kg.f
Std Val. Define Std.
Max: 0
Min: 0
Data[d]
d1 = 587.4 um d2 = 466.7
d2 = d1 (Always)
Hardness[H]
HV = 6.7
Conversion Values
HV(Vickers) NA
Add data to report
Position(depth) 0.0 mm Add
Capture image with objects and grid lines
Measurement Report
Data
Image
1024x768x24
NUM
11:28 AM
1/13/2021


Hardness Magic - Live
File Camera Edit Image View Measure Window Help
Live
Measurement
Measurement Settings
Type Vickers
Weight 1 kg.f
Std Val. Define Std.
Max: 0
Min: 0
Data(d)
d1 = 590.9 um
d2 = 473.6
d2 = d1 (Always)
Hardness(H)
HV = 6.5
Conversion Values
HV(Vickers) NA
Add data to report
Position(depth) 0.0 mm Add
Capture image with objects and grid lines
Measurement Report
Data
Data Image
X: 426 Y: 378 I: 0) RGBA: (56, 71, 78, 0)
1024x768x24
NUM


Hardness Magic - Live
File Camera Edit Image View Measure Window Help
Live
Measurement
Measurement Settings
Type Vickers
Weight 1 kg.f
Std Val. Define Std.
Max: 0
Min: 0
Data(d)
d1 = 582.8 um
d2 = 540.3
d2 = d1 (Always)
Hardness(H)
HV = 5.9
Conversion Values
HV(Vickers) NA
Add data to report
Position(depth) 0.0 mm Add
Capture image with objects and grid lines
Measurement Report
Data
Data Image
X: 427 Y: 627 I: 0) RGBA: (54, 65, 68, 0)
1024x768x24
NUM


Hardness Magic - Live
File Camera Edit Image View Measure Window Help
Live
Measurement
Measurement Settings
Type Vickers
Weight 1 kg.f
Std Val. Define Std.
Max: 0
Min: 0
Data(d)
d1 = 595.5 um
d2 = 467.9
d2 = d1 (Always)
Hardness(H)
HV = 6.6
Conversion Values
HV(Vickers) NA
Add data to report
Position(depth) 0.0 mm Add
Capture image with objects and grid lines
Measurement Report
Data
Data Image
X: 603 Y: 229 I: 0) RGBA: (61, 71, 78, 0)
1024x768x24
NUM


Hardness Magic - Live
File Camera Edit Image View Measure Window Help
Live
Measurement
Measurement Settings
Type Vickers
Weight 1 kg.f
Std Val. Define Std.
Max: 0
Min: 0
Data(d)
d1 = 581.7 um
d2 = 547.2
d2 = d1 (Always)
Hardness(H)
HV = 5.8
Conversion Values
HV(Vickers) NA
Add data to report
Position(depth) 0.0 mm Add
Capture image with objects and grid lines
Measurement Report
Data
Data Image
X: 544 Y: 193 I: 0) RGBA: (54, 59, 72, 0)
NUM


Hardness Magic - Live
File Camera Edit Image View Measure Window Help
Live
Measurement
Measurement Settings
Type Vickers
Weight 1 kg.f
Std Val. Define Std.
Max: 0
Min: 0
Data(d)
d1 = 531.1 um
d2 = 559.9
d2 = d1 (Always)
Hardness(H)
HV = 6.2
Conversion Values
HV(Vickers) NA
Add data to report
Position(depth) 0.0 mm Add
Capture image with objects and grid lines
Measurement Report
Data
Data Image
X: 640 Y: 214 I: 0) RGBA: (59, 66, 67, 0)
1024x768x24
NUM