Investigating the Mechanical, Physical, Machining and Tribological Behaviors of Enset and Sisal Fiber Hybrid Reinforced Polyester Composite

by

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Department of Mechanical Engineering

School of Mechanical, Chemical and Material Engineering

A Dissertation

Presented in fulfillment of the requirements for the degree of Doctor of Philosophy in Manufacturing Engineering

Office of Graduate Studies

Adama Science and Technology University

May, 2023

Adama, Ethiopia

Investigating the Mechanical, Physical, Machining and Tribological Behaviors of Enset and Sisal Fiber Hybrid Reinforced Polyester Composite

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Declaration

I hereby declare that this Dissertation entitled ***"****Investigating the Mechanical, Physical, Machining and Tribological Behaviors of Enset and Sisal Fiber Hybrid Reinforced Polyester Composite****"*** is my original work. That is, it has not been submitted for the award of any academic degree, diploma or certificate in any other university. All sources of materials used for this thesis have been duly acknowledged through appropriate citations.

Abera Endesha Bekele	_______________________	__________________
Name of student	Signature	Date

Recommendation

We, the supervisor(s) Professor Hirpa G. Lemu and Dr. Moera Gutu Jiru of this dissertation, hereby certify that we have read and revised the dissertation entitled "*Investigating the Mechanical, Physical, Machining and Tribological Behaviors of Enset and Sisal Fiber Hybrid Reinforced Polyester Composite*" prepared under our guidance by Abera Endesha Bekele submitted in partial fulfillment of the requirements for the degree of Doctor of Philosophy in Manufacturing Engineering.

Therefore, we recommend the submission of the final version of the dissertation to the department following applicable procedure.

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Co-supervisor	Signature	Date

APPROVAL PAGE

We, the undersigned, members of the Board of Examiners of the dissertation open defense by Abera Endesha Bekele have read and evaluated the dissertation entitled *"Investigating the Mechanical, Physical, Machining and Tribological Behaviors of Enset and Sisal Fiber Hybrid Reinforced Polyester Composite"* and examined the candidate during open defense. This is, therefore, to certify that the dissertation is accepted for partial fulfillment of the requirement of the degree of Doctor of Philosophy in Manufacturing Engineering.

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ABSTRACT

In the manufacturing process of innovative fiber-based composite materials, natural fibers are among the most commonly employed reinforcements. The properties of composites depend on type and properties of the fiber, fiber structure and treatment. In this study, enset and sisal fibers were used to develop the composite, and the properties of both the fibers and the composite are investigated. Furthermore, the effects of NaOH treatment on the fibers and the composite were explored. Understanding that mechanical properties of composites are affected by several parameters, the effects of volume ratio and fiber orientation on mechanical, physical, machinability and tribological properties of the developed hybrid composites, which were fabricated by hand lay-up, were investigated. Both unidirectional and woven orientations with 5% and 10% NaOH treatment, as well as 100/0, 75/25, 50/50, 25/75, and 0/100 fiber volume ratios with a constant polyester resin were considered. To conduct the experimental studies, diverse tools such as optical microscopy, Fourier transform infrared spectroscopy (FTIR), Universal Test Machine (UTM), Scanning Electron Microscopy (SEM), 3D optical surface profiles as well as Taguchi design and ANOVA tools were used. The results indicated that the 5% and 10% NaOH treatment significantly improved the moisture absorption and diameters of the fibers. In particular, both treatments improved the moisture absorption of enset fibers by approx. 33%, while the same treatments on sisal fibers improved the moisture absorption by 30% and 40% respectively. The 5% NaOH treated enset and sisal fibers showed better tenacity, which is an indication of the tensile strength of single fiber, than the untreated and the 10% NaOH treated fibers. As expected, treated and woven fiber orientation hybrid composites exhibited better mechanical properties than untreated and unidirectional hybrid composites. The tests on tensile, flexural and impact strengths indicated that the 5% NaOH-treated composites improved by 5.21%, 9.25%, and 5.98%, respectively, over untreated enset/sisal hybrid composites. The enhanced mechanical properties of the composites can be attributed to the improved moisture content, diameter, as well as the lignin and hemicellulose contents of the fibers. From studies on drilled hole surface, it has been observed that the feed rate parameter had the highest percentage contribution, while the tribological properties of woven type of composite had a lower weight loss than unidirectional composites. In general, the results of the study showed that 5% NaOH treated, woven and 50/50 enset/sisal hybrid composites have optimum mechanical properties and water absorption properties.

Keywords: Alkali treatment, Hybrid composite, Mechanical properties, Tribological machinability, Taguchi method,

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ABBREVIATIONS

ANOVA	Analysis of variance
ASTM	American Society for Testing and Materials
ASTU	Adama Science and Technology University
CNC	Computer numerical control
cN	Cent newton
DF	Degree of freedom
D_{max}	Maximum diameter drilled hole
D_{nom}	Nominal diameter drilled hole
E/S	Enset/Sisal
E_o	Untreated enset fiber
$E_{5\%}$	Five percent alkali treated enset fiber
$E_{10\%}$	Ten percent alkali treated enset fiber
F_d	Delamination factor
F_f	Friction force
F_{max}	Maximum load
FTIR	Fourier transformation infrared spectroscopy
HSS	High stainless steel
KN	Kilo newton
mm/min	Millimeter per minute
MPa	Mega Pascal
m/s	Meter per second
N	Newton
NaOH	Sodium hydroxide
NF	Natural Fibers
NFRCs	Natural Fiber Reinforced Composites
OM	Optical Microscopy
POD	Pin –On –Disk
rpm	Revolution per minute
S_o	Untreated sisal fiber
$S_{5\%}$	Five percent alkali treated sisal fiber
$10_{\%}$	Ten percent alkali treated sisal fiber
SEM	Scanning electron microscopy
Sec.	Second
SR	Surface roughness
S/R	Signal to noise ratio
UF_d	Unidirectional delamination factor
UPE	Unsaturated Polyester
USR	Unidirectional surface roughness
UTM	Universal Test Machine
VARTM	Vacuum Assisted Resin Transfer moulding
V_c	Volume of composite
Vo.%	Volume percentages
V_f	Fiber volume
WA	Water absorption
WFd	Woven delamination factor
WSR	Woven surface roughness
Wt. %	Weight Percentages

LIST OF PUBLICATIONS

Journal Publication

Paper I: Abera E. Bekele, Hirpa G. Lemu and Moera G. Jiru (2021), Experimental Study of Physical, Chemical and Mechanical Properties of Enset and Sisal Fibers, *Polymer Testing* (ELSEVIER), vol. 106, 107453

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Paper III Abera E. Bekele, Hirpa G. Lemu and Moera G. Jiru (2022), Study of Machinability in Drilling Operation of Enset–Sisal Hybrid Polyester Composite. *Journal of Composites Science (MDPI Journal),* vol. 6, 205.

Paper IV Abera E. Bekele, Moera G. Jiru and Hirpa G. Lemu (2023), Study of the Effects of Alkali Treatment and Fiber Orientation on Mechanical Properties of Enset/Sisal Polymer Hybrid Composite. *Journal of Composites Science (MDPI Journal),* vol. 7, 37.

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CHAPTER-ONE

1. Introduction

1.1. Background

Natural fiber composites (NFCs) are alternative materials for replacing environmentally harmful synthetic materials. These materials have several advantages including reduced pollution effects, biodegradability, light weight and they have low energy consumption (Sanjay et al., 2018). Nowadays, natural fibers as alternative reinforcements in polymer composites have got high attention in research and industries due to their advantages over glass and carbon fibers because they have low environment impact, low cost, , in abundance availability having wide range of applications.

The properties of fibers mainly depend on their chemical composition and their structure, which are related to the type of fibers used, method of extraction, harvesting period, growing conditions and chemical treatment (Saba et al., 2015) (Pickering et al., 2016b)**.** Fiber treatment, fiber volume ratio, fiber orientation and fiber hybridization are the main parameter that affects the mechanical, physical, machinability and tribological properties of natural fiber reinforced composites (NFRCs).

Fiber treatment is one way of reducing its hydrophilic tendency for improving compatibility with the matrix materials (Stana-kleinschek et al., 2003)**.** Chemically fiber was treated by using Alkali (NaOH), Acetic acid, Silane, Benzoyl peroxide, Isocyanate treatment, etc. For the improvement of physical, mechanical, chemical, morphological and thermal properties of natural fibers and its composites (Niaz et al., 2017)**,** (Kabir et al., 2012a)**,** (Mishra et al., 2001)**,** (Malkapuram et al., 2009)**,** (Joseph, 1997)**,** (Jayaramudu et al., 2011). Chemically fiber treatments are the common and best way to remove the impurities from the fibers. Alkali (mercerization) treatment is the most common chemically fiber treatment method using Sodium hydroxide (NaOH), which is widely employed to modify the crystal structure modifying the cellulosic molecular structure, by removing weak components like lignin and hemicelluloses, wax and oil cover the parts of the fiber surface to increase the roughness of the surface of the fibers (Arthanarieswaran et al., 2015), (Kabir et al., 2012a).

Alkali treatment is used improve the mechanical properties of natural fiber by modifying crystal structure and removing of impurity materials depending on the concentration of NaOH with soaking time which directly related with the final properties of composite (Cruz & Fangueiro, 2016a), (Hossain et al., 2011). The used concentrations range from 1 to 15 wt% , immersing time ½ hr to 3 hrs and dry time from 1 day to 5 days (Cai et al., 2016), (Dawit et al., 2019). If the alkali concentration is less, the impurities are not sufficiently removed, while higher alkali concentration above 10% can lead to damage of fibers (Kabir et al., 2012a). The effects of immersion (soaking) time were also studied for coir fiber. The result showed that 2 hrs immersion time is better tensile strength than immersing for less hours (1 hr) or longer hours (3 - 5 hrs) (Musanif & Thomas, 2015). Alkali treatment remove the outer (lignin) and the inner (hemicellulose) contents from the fibers (Hossain et al., 2014).

Commonly, natural fibers have hydrophilic properties that are vulnerable to moisture. Fiber moisture absorption properties were improved by alkali treatment by reducing the hydrophilic hydroxyl groups of the fibers (Shehu et al., 2017). If the treatment parameters are not optimized, the NaOH can cause fiber defibrillation, pore formation, and fiber embrittlement (Ouarhim et al., 2018). Alkali treated Palmyra Palm Leaf Stalk Fiber (PPLSF) and jute fibers with unsaturated polyester matric hybrid composite properties i.e tensile and flexural were studied and both mechanical properties were enhanced. But the impact strength of pure PPLSF polyester composite is higher than that of hybrid and pure jute polyester composites and this shows properties drawback of single fiber composites improved by hybridizing with fiber treatments (Shanmugam & Thiruchitrambalam, 2013a).

The properties and structure of fiber base composite were defined by its fiber orientations (Rajak et al., 2019). Rather than fiber volume ratio, fiber orientation has more influence on the physical properties of fiber composites (Ramesh et al., 2016). In addition to fiber treatment, fiber volume and fiber orientation a hybrid of two or more different fibers improves the mechanical properties of its composites. Some of issues related to synthetic fibers hybrid composites are environmental issues, recyclability, biodegradability, and reusability (Shubhra et al., 2010; Yang et al., 2011). But some of the advantages associated with natural fibers are low-cost manufacturing, renewable nature, eco-friendliness, light weight, and optimum mechanical properties that alternatively used instead of synthetic fibers. Due to all these advantages, hybrid natural fiber composites are currently being used in number of applications (Automotive, aerospace, construction and furniture) industries. Hybridization of natural fibers is the best option to improve the single natural fiber (Cavalcanti et al., 2019a). When two natural fibers are hybridized, then the attention is often on getting a better balance in mechanical, chemical and physical properties, rather than on optimizing the hybrid effect (Mochane et al., 2019), (Srinivasan et al., 2014), (Swolfs et al., 2014).

The most common thermosetting polymer composite fabrication methods are hand Layup (Chaudhary, 2017), resin transfer molding (Hahn, 2000; Rouison et al., 2002, 2004) and Pultrusion, (Peng et al., 2011; Santulli, 2010). Hand lay-up is the simplest method of processing the thermosets-based fibers composite. Advantages of hand lay-up are the relatively lower mold costs, simplicity, low processing cost, ability to manufacturing complex design the tools required for production are readily available and the molds are easy to maintain, while the process is also known for having a long processing time and being labor intensive, which are the few disadvantages associated with the process (Stringer, 1989), (Yuhazri & Sihombing, 2010), (Madhukiran et al., 2013).

Moisture absorption is a chief hindrance that makes Poor fiber–matrix adhesion, a decrease in interfacial bonding, and a decrease in mechanical properties incorporate into any micro cracks and void spaces present in a natural fiber composite (Vlaev et al., 2009).

Enset (E) plant cultivated in East Africa and widely in high land of Ethiopia (Negawo et al., 2019)**.** Enset plant Figure 1.1 (a) containing leave, pseudostem and corn parts. Leaf and pseudostem parts having full of fibers. Pseudostem parts contain cellulosic semisolid flush which is reinforced by the fibers. These solid parts were extracted and used as a food (kocho) and the fibers. In rural areas, traditionally used for making rope, carpet, mats, cordage, bags, construction materials (such as tying materials that can be used in place of nails), and sieves and the remaining waste. So, converting this waste byproduct is economical and environmentally friendly product using as reinforcement material in polymer-based composite. However, only a few studies were yet performed to investigate its use in the fabrication of green polymer composites ((Simoons, 1965), (Negawo et al., 2019). About 4.5 million tons per year sisal (S) fibers are extracted from Agava Sisalana plant Figure 1.1 (b) throughout the world mostly in tropical nations of Africa, the West Indies, and the Far East (Mishra et al., 2004). The sisal fiber is traditionally used for rope and twine and has many other uses, including rope, cloth, footwear, hats, bags, carpets, and dartboards ((Mishra et al., 2003). Sisal fiber has good impact properties than jute fibers. Sisal fiber has satisfactory mechanical properties, abundantly available, biodegradable and the processing costs to manufacture it as a composite material is low. In Ethiopia sisal plant used for fancing and its fibers are wasted whithout any usage. Mechanical decortication is the most common extraction process to get a fibers from the leaf part sisal plant and Pseudostem parts of enset plants among others (Bous et al., 2018). Both fibers are economical, naturally available, renewable and environmentally friend. Among other natural fibers enset and sisal fibers are chosen for the study due to potentially availability of the resources in the study target i.e., South-West Ethiopia.

The properties of plant fibers naturally depend on the climate in which the plant grows. Therefore, this study aims to characterize the fibers of enset and sisal by experimental investigation the effects of alkali treatment on the physical, chemical composition and mechanical properties as a probe for reinforced polymer composites. After characterizing enset-sisal fibers, the hybrid composite was fabricated from (unidirectional and woven orientation), (untreated, 5% and 10% NaOH treated) with varying weight ratios of enset/sisal (100/0, 75/25. 50/50, 75/25 and 0/100) with having constant polyester resin. The total fibers volume ration 30% and the total volume of polyester ration 70% in all types of hybrid composite. The mechanical, water absorption, machinability and tribological properties are investigated. The fractured surfaces of the composites were analyzed using scanning electron microscopy (SEM) and 3D optical profilometer. The results are expected to provide guide to get the optimum NaOH concentration and weight ratio, better mechanical, machinability and tribological properties, lower water absorption properties to develop enset–sisal hybrid polyester composite.

(a) (b)

Figure 1. 1 (a) Enset (Ventricosum) plant and (b) Sisal Plant (Agava Sisalana)

1.2. Problem Statement

Since most of the plastics and synthetic materials are harmful to our environment, several research are trying to find a solution for these problems by developing environmentally friendly materials (i.e., natural fiber composites). Different researches indicate that sisal fiber, whose plant is available in Africa, West India and Far East (Mishra et al., 2004) has satisfactory mechanical properties, abundantly available, biodegradable and the processing costs to manufacture it as a composite material is low. Sisal fiber-based polymer composites are mostly applicable in automotive, construction, marine, and aircraft structures. Enset ventricosum plant which is found in Ethiopia is mainly used for food after the fiber has been extracted (Negawo et al., 2019). Enset fiber is a byproduct that can be extracted from Pseudostem part of enset plant by scrapping or machine decorticator. However, the amount of enset fiber extracted is much more in volume but, very less amount of fiber is used traditionally for some manufacturing goods (sacks, ropes, mats, etc.) locally. Earlier research have been conducted on enset fiber and sisal polymer separately, while no significant research has been reported out on enset/sisal hybrid polymer composites. Investigating the effect of fiber volume, fiber treatment and fiber orientations on mechanical (flexural, tensile and impact), machinability, moisture and tribological properties of enset/sisal hybrid composite are thus significant. Therefore, to take the advantages of one material over the other, it is necessary to study the hybridization effect of enset fiber with sisal fiber composite for interior part applications.

1.3. Research objectives

1.3.1. Main objective

The main objective of this research is investigating the mechanical, physical, machining and tribological Behaviors of enset and sisal fiber hybrid reinforced polyester composite.

1.3.2. Specific objectives

1. To characterize the physical, chemical and mechanical properties of untreated and 5% and 10% NaOH treated enset and sisal fibers.
2. To study the effects of alkali treatment, fiber volume ratio, and fiber orientation on mechanical properties of enset/sisal hybrid Polyester composite.
3. To study the machinability in drill hole surface of unidirectional and woven enset/sisal hybrid polyester composite.
4. To study the tribological behavior of unidirectional and woven of enset/sisal hybrid Polyester composite.

1.4. Motivation

The motivation to conduct this research is the observation that natural fiber plants can be used to develop composites that are potentially new alternatives, cost-effective and environment friendly products compared with synthetic based composites. Producing composites from enset and sisal fibers provides good opportunities because:

- ✓ Fibers are abundantly and easily available
- ✓ Minimum costs and waste byproducts
- ✓ Create job opportunity (i.e. cultivation, harvesting and extraction, packaging, fiber processing composite fabrication and marketing the product etc.).
- ✓ Enhanced mechanical properties when hybridized.

1.5. Expected outcomes of the study

- ➢ Enset/sisal (E/S) hybrid composite will have better mechanical and moisture properties than individual enset and sisal fiber composite.
- ➢ Alkali treatment will improve the mechanical and moisture properties of E/S hybrid composite.
- ➢ Drilled hole surface and tribological properties of treated E/S hybrid composite will be better for optimum fiber volume.

1.6. Significance of the Study

Natural fiber composite is replacing the synthetic fiber ones due to more availability, eco-friendly, good mechanical properties and wider applications. So enset and Sisal fiber are abundantly available in the sub-saharan countries and good mechanical properties the same as other natural fibers. This study focuses on Ethiopian enset and sisal fibers due to abundant availability in the southern and south-west parts of the country. Both natural fibers collected from the Oromia region (South-West Zone). Sisal fiber are very common, and most researchers have study the mechanical properties of this fiber but, enset is indigenes plant in Ethiopia and limited studies are done before and existing literature showed wider promising application area. In this study we have investigated mechanical, physical, and chemical enset and sisal fibers and the mechanical, physical, machining and tribological properties characterization of enset/sisal hybrid composites.

1.7. Scope of the study

The scope of this research is to characterize of untreated and treated enset and sisal fibers, and fabricating individual and hybrid by varying orientation, volume and treatment with polyester composites. Study the mechanical, physical, chemical, machinability and tribological properties of fabricated composite. Compare the result of untreated and treated fiber, composite, individual and hybrid, unidirectional and woven fiber orientation composite. Finally compare our result with the existing literature and recommend for some applications.

1.8. Structure of the dissertation

This dissertation is divided into four main chapters.

Chapter-1 Introduction, discuses about the background of the study, problem statements, objectives, motivation, expected outcomes, significant, scope of the study.

Chapter- 2 Literature Review, gives overview of the existing literature regard to natural fiber hybrid, reinforced composites. The gap and advantage of treated and fiber volume, orientation, hybridization of the natural fiber hybrid composite is briefly explained, and finally key finding of literature will be summarized.

Chapter-3 Material and Methods, under this chapter, we develop the whole plan of the study that we follow throughout our study. We explain the material (chemical, equipment, tools, machine and other equipment) and detailed approach for each and every part of the experiment.

Chapter -4 Results and Discussion, under this section the summary of result assembled from the appended publications. Finally, *Conclusion and Recommendations* of the study as well as its potential contribution are presented in Chapter 5.

CHAPTER-TWO

2. Literature Review

2.1. Introduction

This chapter covers review of previous literature related to natural fiber reinforced hybrid composites. It also reports assessments done on the factor affecting natural fibers and hybrid composite. The effects of those factors on mechanical, machining and tribological behavior of natural fiber based hybrid composite are discussed and finally key findings of literature and the literature gaps are summarized.

2.2. Overview of natural fibers

The shape, size, and strength of the natural plant fibers may vary on the source, growth climate and processing techniques and age (Gopinath et al., 2014). Fiber structure and strength influence the mechanical properties of the natural fiber-reinforced plastic (Ochi, 2008)**.** Plant fibers tend to exhibit better mechanical properties than animal-based fibers. The applications include interior paneling for aircraft and automobiles, household tables and chairs, window frames (Bavan & Kumar, 2010). Classification of natural fiber was tabulated on Figure 2.1(Athijayamani et al., 2009).

The shape, size, and strength of natural plant fibers may vary widely depending on the environment, region of origin, and other characteristics. In turn, these features of the natural fibers are likely to influence the mechanical properties of the natural fiber-reinforced plastics (Ochi, 2008).

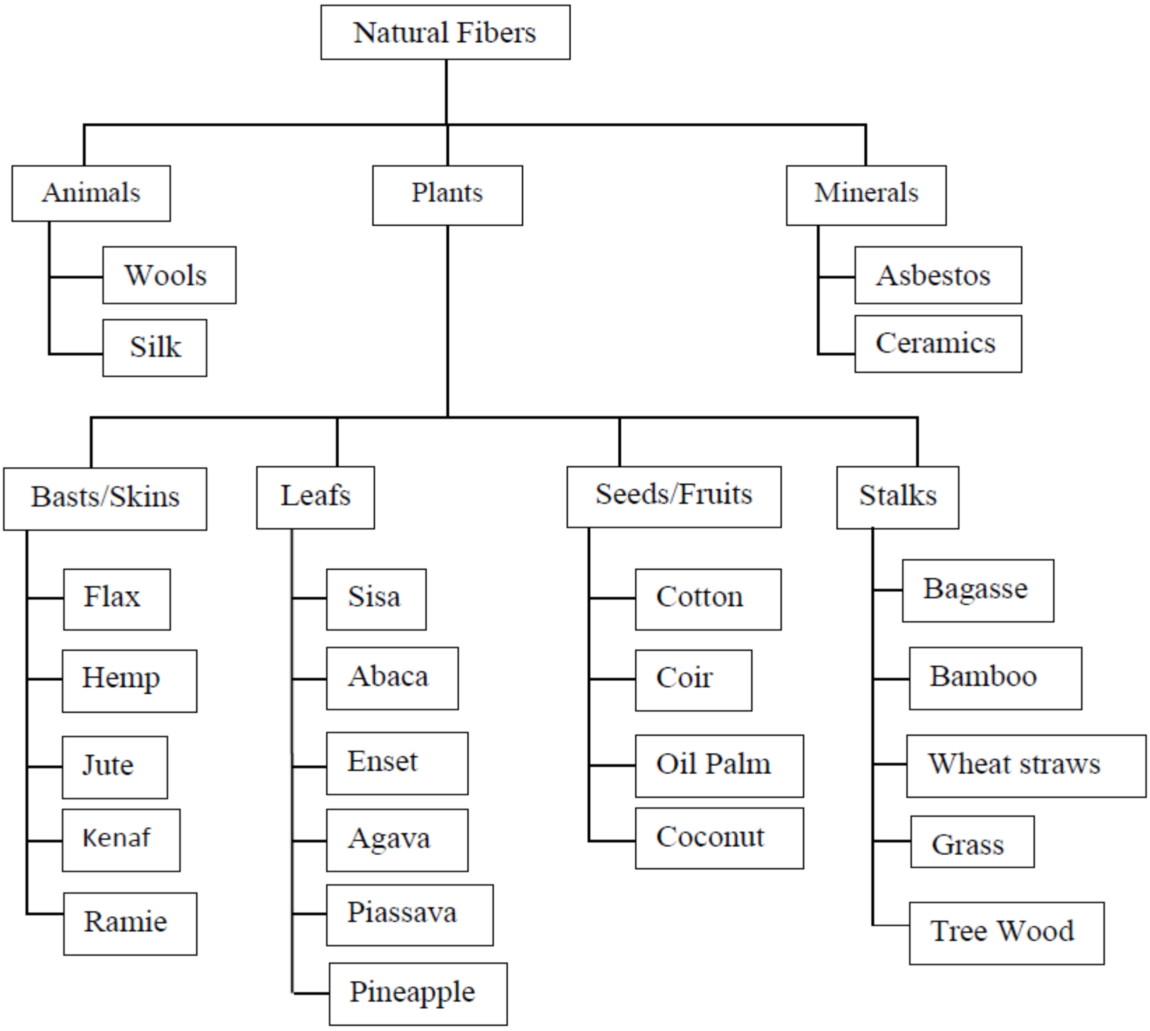


Figure 2. 1 Natural fiber classifications

2.3. Composite material

Composites are a structural material that formed from a combination of two or more constituents which, when combined, retain their identities and properties. Typically, a composite would consist of reinforcing fiber which is the hardest, strongest, and stiffest component, embedded in a continuous matrix. The matrix phase materials are generally continuous. The main functions of the matrix are to transmit externally applied loads to the reinforcement and to protect the latter from external mechanical damage (William D. Callister, 2001).

2.4. Hybrid composite

Hybrid composites consist of a combination of two or more fibers in a polymer matrix. The possible combinations of hybrid composites include artificial–artificial, natural–artificial and natural–natural fiber types (Cavalcanti et al., 2019b). Fiber hybridization is a promising strategy, where two or more types of fibers are combined in a matrix of composite material to mitigate the drawback of the type of fiber, keeping benefits of others. The main factors affecting the mechanical properties of hybrid composites are the fiber volume ratio, stacking sequence of the fiber layers, treatment of fibers and effect of environmental conditions (Swolfs et al., 2014), (Codispoti et al., 2015).

2.5. Factor affecting natural fiber composites

2.5.1. Chemical composition of fibers

The main factors affecting mechanical properties of NF composites are fiber selection (types, chemical composition, harvesting, extraction methods, aspect ratio, treatment and fiber content) and matrix selection, interfacial strength, fiber dispersion, fiber orientation and fabrication techniques (Pickering et al., 2016a). The chemical components are dispersed throughout the cell wall, which is composed of primary and secondary wall layers. Chemical composition differs from plant to plant, and within different parts of the same plant, from different geographic locations, ages, climate, and soil conditions. The chemical composition of the fiber influences its properties and the fibers themselves could be considered as fibrous composite materials (Han & Rowell, 2005). Cellulose is the main component in the fiber. The strength and stiffness of the fibers are provided by cellulose components via hydrogen bonds and other linkage. Hemicellulose is composed of cellulose chains, but it is much shorter in length and is built up of a mixture of polysaccharides of relatively low molecular weight. Hemicellulose is responsible for biodegradation, moisture absorption, and thermal degradation of the fibers. The lignin content influences the fiber structure, properties, and morphology, and it has a strength 100 times compared with hemicellulose in moisture of 70% (Thygesen et al., 2012). Also lignin is thermally stable, but responsible for the Ultraviolet (UV) degradation of the fibers (Akil et al., 2011). Table 2.1 shows the basic properties (physical and chemical) of common natural and synthetic fibers. The mechanical properties of natural and synthetic fiber as shown in Table 2.2.

Table 2. 1 Physical properties and chemical composition of common natural fibers (NF)

Properties	Cellulose (%)	Length (m)	Hemi cellulose (%)	Lignin (%)	Moisture Contents (%)	Density (kg/cm^3)	Ash
Enset fiber	56.05	1-3	24.04	2.21	11.8	1.53	-
Sisal fiber	67	1-2.3	16	12	16	1.45	-
Pineapple	81	-	16-19	4.6-12	11-12	1.44	-
Banana	63-64	-	6-19	5-10	10-12	1.35	-
Abaca fiber	56-63	-	15-17	7-9	-	-	3
Hemp	57-77	-	14-22	3-13	-	-	0.8
Jute fiber	45-63	-	4-10	12-25	-	-	1-2
Kenaf fiber	31-57	-	21-23	15-19	-	-	2-5
E-glass						2.55	
S-glass						2.50	

Source: (Negawo et al., 2019), (Gupta and Srivastava, 2015), (Martin et al., 2013), (Madhukiran et al., 2013)

Table 2.2 Mechanical properties of common natural fibers

Properties	Tensile strength (MPa)	Tensile modulus (GPa)	Elongation (%)
Enset fiber	513	26.7	1.92
Sisal fiber	400-700	9-20	2-3
Jute	393-800	10-30	
Pineapple	413-1627	34.5-82.5	
Kenaf	250	4.3	
Abaca leaf fiber	418-486	12-13.8	
Spider silk	873-972	11-13	
Flax	345-1500	27.6-80	
Hemp	550-900	70	
Banana	529-759	8.2	
Coir	108-252	4-6	
Bamboo	503	35.9	
Palm	377	2.75	
E-glass	2000-3400	70-71	0.5
S-glass	4570	86	2.2-2.8

Source: (Negawo et al., 2019), (Gupta and Srivastava, 2015), (Martin et al., 2013), (Madhukiran et al., 2013)

2.5.2. Fiber extraction methods

Decortication is the common fiber extraction method by combination of shear stress and impact force that from stem leaf or fruit to reduce long processing time. Retting is of the existence of the bacteria and moisture in the plants allows breaking down large parts from cellular tissues and its adhesive substances that surrounds the fibers, enabling the separation of individual fibers from the plant (Bous et al., 2018), (Gurukarthik et al., 2018), (Hyness et al., 2017). The most common fiber bundles extractions from the leaf and bast of fiber plants are decortication and retting techniques (Pickering et al., 2016b). Fiber extraction techniques are developed and classified to improve fiber properties for getting quality fibers based on nature of plant as shown on Figure 2.2 (Sanjay et al., 2019), (Butkut et al., 2015), (Pickering et al., 2007) (Sisti et al., 2018), (Om et al., 2015), (Scheliga et al., 2018).

Sisal fibers are extracted by microbiological retting, by hand scraping or by using a raspador machine. Leaves cut from sisal plant are decorticated by mechanical means by scraping action of the blade which removes cellulosic material. Decorticated fibers are then washed and dried, either in the sun or by hot air. This is done to obtain high quality fibers. Superior quality fibers in terms of luster, flexibility and strength are obtained after a repeated process (Sood et al., 1987).

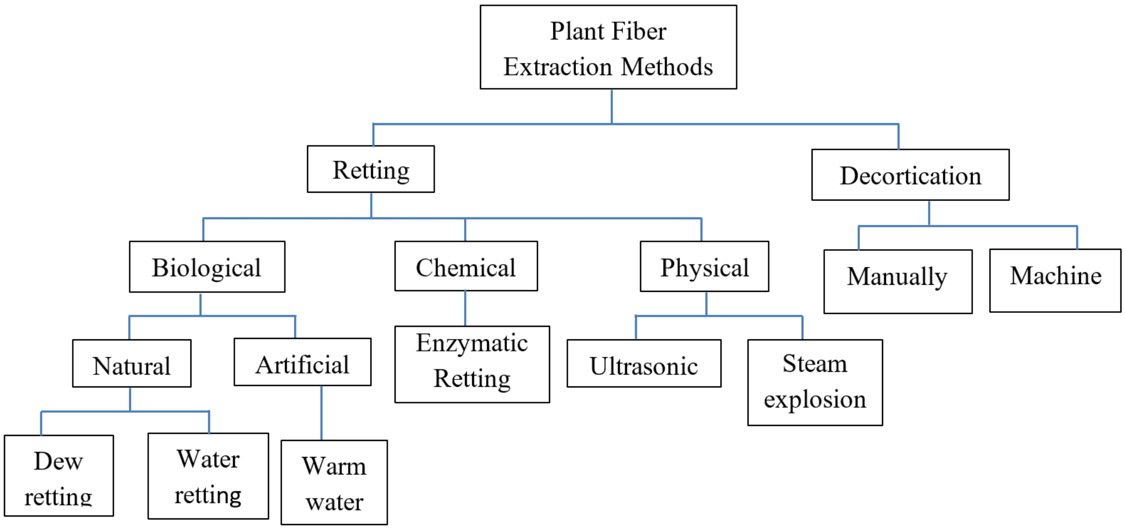


Figure 2. 2 Classification of fiber extraction techniques.

The fibers are extracted using a primitive machine popularly known as a 'periquita'(Mukhopadhyay et al., 2008), (Satyanarayana & Deme, 2007). Sisal fiber is extracted by retting followed by scrapping and mechanical means using decorticator smart art (Thomas et al., 2011)**.** Figure 2.3 (Zhi et al., 2001) shows the steps of extraction of sisal fiber i.e (a) cutting leaf from sisal plant, (b) decortication, (c) drying in the sunlight to remove impurities and then (d) making ready to use for different applications (Zhi et al., 2001).

Enset is usually larger than banana, with the largest plants up to ten meters tall and with a pseudostem up to one meter in diameter. The pseudomstem part tall two to three meters full of edible pulp and quality fiber. Similar to other cellulosic natural fibers, Enset fiber is chemically composed of cellulose, hemicellulose, lignin, pectin, moisture, wax and oils and manually decortication methods is the most common extracting fiber from pseduestem part of enset (Negawo et al., 2019).

2.5.3. Fiber treatment

Modifications have been major topics in natural fiber reinforced composites to improve the interfacial adhesion and in turn the overall properties of the composite product. Fiber treatment is one way of reducing its hydrophilic tendency for improving compatibility with the matrix materials. Physically, chemically, and biologically fiber treatment methods are used to modify the properties of fibers and their composites (Stana-kleinschek et al., 2003). (Kabir et al., 2012a), (Li et al., 2007), (Sanjay et al., 2019). There different types of chemical treatment as shown in Table 2.3. Chemically fiber treatments are the common and best way to remove the impurities from the fibers. Alkali (mercerization) treatment is the most common fiber chemical treatment method using Sodium hydroxide (NaOH), which is widely employed to modify the cellulosic molecular structure by removing amorphous contents like lignin, wax and oils covering the external surface of the fiber for making strong link between fiber and matrix (Arthanarieswaran et al., 2015), (Kabir et al., 2012a), (Analysis et al., 2008) (Arthanarieswaran et al., 2015), (Kabir et al., 2012a).

2.5.3.1. Effects of alkali treatment on natural fibers

Alkali treatment is used improve the mechanical properties of natural fiber by modifying crystal structure and by removing weak components like hemicelluloses and ligning depending on the concentration of NaOH with soaking time which leads to fiber surface becoming rough that directly related with the final properties of composite (Cruz & Fangueiro, 2016a), (Hossain et al., 2011), (Cruz & Fangueiro, 2016b) and (Inder et al., 2017). Rough surface promotes a strong link of interfacial bond between the fibers and the matrix composites (Kabir et al., 2012b).

Table 2.3 Types of chemical treatment of natural fibers

Name of treatment	Chemical used	Chemical structure	Reference
Alkali treatment	Sodium Hydroxide	$NaOH$	(Hemsri et al., 2012)
Acetic acid	Acetic acid	CH_3COOH	(Kabir et al., 2012b)
Benzoylation treatment	Benzotyle Chloride	C_7H_5ClO	(Li et al., 2007)
Isocyanate treatment	Carbon Tetrachloride	CCl_4	(George et al., 2001)
Peroxide treatment	Benzoyl Peroxide	$C_{14}H_{10}O_4$	(Joseph. et al., 2003)
Permananganate treatment	Potassium Permanaganate	$KMnO_4$	(Rahman, 2007)
Aceylation treatments	Siliane	SiH_4	(Singh et al., 1996)
Sulfaric acid treatment	Sulfuaric acid	H_2SO_4	(Sartika et al., 2019)
Stearic acid treatment	Stearic acid	$C_{18}H_{30}O_2$	(Annie et al., 2010)

Alkali sensitive hydroxyl (OH) groups present among the molecules are broken down, which then react with water molecules (H-OH) and move out from the fiber structure. The remaining reactive molecules form fiber–cell–O–Na groups between the cellulose molecular chains. Due to this, hydrophilic hydroxyl groups are reduced and leads to increased fiber's moisture resistance property. In addition, this also makes fiber surface clean and reduces fiber diameter to improve the length–diameter aspect ratio. The main modification in alkali treatment is the removal of hydrogen bonding in the network structure (Li et al., 2007)(Agrawal et al., 2000).

Fiber- cell- OH + NaOH $\longrightarrow$ Fiber cell - $O^{-}Na^{+}$ + H_2O + impurities

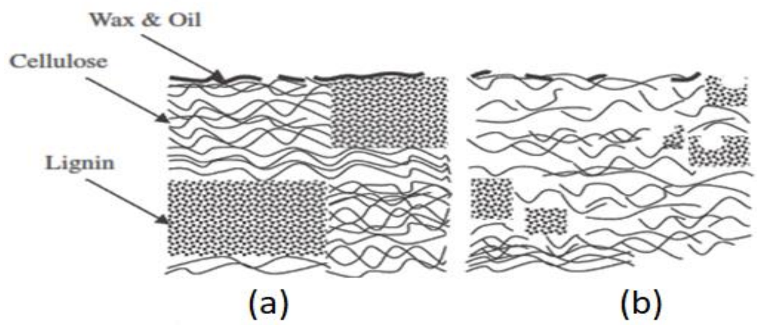


Figure 2. 3 Typical structure of (a) untreated and (b) alkalized cellulose fiber

As illustrated in Figure. 2.3. (a), untreated natural fiber contains more impurities like lignin, cellulose, wax and oil than alkali treated fibers Figure.2.3 (b). This affects the mechanical properties of fibers and its composite (Kabir et al., 2012b). The main parameters that will be considered during alkali treatments are concentration, immersing time, drying methods, and drying time. while alkali treatment of fiber, we use either constant concentration for constant period of time, different concentration for constant period of time, constant concentration for different period of time or different concentration for different period of time as shown in Table 2.4**.** Abaca with 5–15 wt.% NaOH solutions concentration for 2 hrs results showed that 5 wt.% NaOH high cellulose crystallinity and minimal fibrillation and displayed excellent interfacial adhesion with epoxy resin. Higher NaOH concentrations decreased the cellulose crystallinity causes weak adhesion with the epoxy resin and low concentration better for surface properties improvement for advance composite applications (Cai et al., 2016). Treated flax fiber was studied by varying treating time (2, 4, 6 and 8 hrs) and the results showed that average treating time leads to maximum flexural but, untreated flax fiber showed higher breaking energy than treated fiber (Ray et al., 2001).

Table 2. 4 Existing literatures on fiber alkali treatment (alkali concentration, immersing time and drying time.

Alkali Concentrations	Immersing time	Dried (hr)	Neutralized	Reference
5,10 & 15 wt.%	0.5 hr at room	For 24 hr at room temperature	By 2.1 % HCl solution	(Boopathi et al., 2012)
2 wt.%	heat at 40 ^{o}C for 20 hr	For 20 hr in an oven at 50 ^{o}C		(Shehu et al., 2017)
5 wt.%	At 20 ^{o}C for 1/2 hr	For 24 hrs at room temperature and 50 ^{o}C in an oven	Fiber Sealed in plastic bag	(Yan et al., 2016)
5 wt.%	30 minutes in Ambient	65 ^{o}C in an oven for 12 hrs		(Sawpan et al., 2011a)
5,10 & 15 wt.%	In 100 ml. For 2 hrs under	85 ^{o}C in vacuum oven for 2 hrs	NaOH Solution 100 ml	(Cai et al., 2016)
10 and 20 wt.%	Alkali for 3 hrs.	Dried in an open air for five days.	NaOH solution*	(Dawit et al., 2019)

*Note: The NaOH solution is stirred for 15 min in unidirectional and soaked required fiber

So, for kenaf fiber treatment higher amount of alkali concentrations are not recommended (Mahjoub et al., 2014). The mechanical properties i.e tensile and mean elongation of untreated and 5 to 15 % alkali treated borassus fruit fiber was determined and result indicates that treated fiber better than untreated, while 5 % alkali concentration more better than all in case of tensile strength (Boopathi et al., 2012)**.** The effects of alkali treatment on mechanical properties of kenaf and pineapple leaf fibers was studied and the result showed that the diameter, tensile strength, tensile module and the interfacial stress strength of alkali treated of kenaf fiber and pineapple fiber higher than that of untreated fibers (Asim et al., 2016a). The effects of alkali treatment on mechanical properties on enset fiber was studied and the result shows that linear density, tensile strength and young's modulus improved while comparing with untreated fiber (Negawo et al., 2019).

Diameter of treated sugar fiber less than untreated fiber. This is due to reducing the amount of amorphous material like hemicellulose and lignin from the fiber by treatment of the fiber. The mechanical properties treated fiber better than untreated. The maximum improvement in mechanical properties was obtained in the fibers treated with 5% alkali solution for sugar fiber (Hossain et al., 2014). Table 2.5 shows the effects of treatment on mechanical properties of natural fiber and result shows that the treated fiber higher tensile strength than untreated fiber. Also the diameter of treated fiber smaller than untreated fiber due to remove the impurities from the fiber.

Table 2. 5 Mechanical properties of alkali treated and untreated fiber

	Fibers	Fiber diameter (mm)	Tensile strength (MPa)	Strain to failure (%)	References
Pineapple leaf fiber	Untreated		613.75	6.63	(Yusof et al., 2015)
	Treated		763.7	3.6	
Enset	Untreated		552	1.9	(Negawo et al., 2019)
	2.5% NaOH		742.5	1.63	
	7.5% NaOH		1358	2.19	
Kenaf	Untreated	83.5	282.6		(Asim et al., 2016b)
	NaOH treated	46.4	432.01		
PALF	Untreated	78.8	290.61		
	NaOH treated	47.4	455.74		
Sugar fiber	Untreated	0.26	169.51	6.25	(Hossain et al., 2014)
	1 % NaOH	0.18	204.5	7.80	
	5 % NaOH	0.16	350.6	7.87	
Hemp fiber	Untreated	31.5 µm	538		(Sawpan et al., 2011b)
	Alkali treated	25.8 µm	598		

2.5.3.2. Effects of alkali treatment on composites

Alkali treatment is not only improving the properties of fiber it also enhances the mechanical properties of its composites. The main modification in alkali treatment is the removal of hydrogen bonding in the network structure and improving the fiber and matrix interfacial interactions (Li et al., 2007)(Agrawal et al., 2000). The effects of treated on mechanical properties of natural fiber hybrid polymer composites was tabulated on Table 2.6, which shows that the tensile strength, flexural strength and impact strength of NaOH treated fibers hybrid composites better than untreated natural fibers hybrid composites.

Table 2. 6 Effects of alkali treatment on mechanical behaviors of natural fiber reinforced hybrid composites.

Composites	Tensile strength (MPa)	Flexural strength (MPa)	Reference
Untreated			
Jute/Sisal composite	70.39	67.56	(Cavalcanti et al., 2019a)
Jute/Curaua composite	66.77	97.67	(Cavalcanti et al., 2019a)
Sisal/Jute composite	42.45	39.8	(Perremans et al., 2018)
Bagasse aliphatic polyester composite	23.47	43.87	(Cao et al., 2006)
Abaca epoxy composite	717	-	(Cai et al., 2016)
Treated			
Jute/Sisal composite	74.78	54.67	(Cavalcanti et al., 2019a)
Jute/Curaua composite	63.9	80.86	(Cavalcanti et al., 2019a)
Sisal/Jute composite	53.7 4	44.58	(Perremans et al., 2018)
Bagasse aliphatic polyester	26.77	50.86	(Cao et al., 2006)
Abaca epoxy composite	773	-	(Cai et al., 2016)

Alkali treated Palmyra Palm Leaf Stalk Fiber (PPLSF) and jute fibers with unsaturated polyester matric hybrid composite properties i.e tensile and flexural were studied and both mechanical properties were enhanced (Shanmugam & Thiruchitrambalam, 2013a). 5 % NaOH treated coir fiber reinforced composite showed increases tensile strength, tensile module, flexural strength module and flexural module which alkali treatment attributed the interfacial adhesion but decrease the tensile strain, flexural strain and damping ratio (Yan et al., 2016). Kenaf and bamboo fibers were treated by 6 % NaOH fibers improve the composite flexural strength by 25 % and 36 % respectively, which NaOH improve the interfacial adhesion of fibers and matrix (resin) (Edeerozey et al., 2007) and (Yousif et al., 2012). The mechanical properties of treated flax, hemp, jute and sisal fibers are very good, which makes them capable of competing with glass fibers as regards strength and modulus (Velde & Kiekens, 2002).

The mechanical properties (tensile strength, flexural strength and impact strength) of alkali treated Bagasse aliphatic polyester composite higher than untreated (Cao et al., 2006). The mechanical properties i.e. tensile strength and flexural of 5 % NaOH alkali-treated jute, sisal and banana fiber reinforced epoxy composites increase by 50% when compare with non-treated fiber composite and significantly increase the flexural strength (Inder et al., 2017)**.** Alkali treated kenaf fiber composites showed less moisture absorption of 3.85 %, compared to untreated fiber composites of 6.38 % correspondingly (Azwa & Yousif, 2013)**.** The properties of flexural and impact properties of sisal reinforced epoxy composites by varying the fiber lengths (5, 10, 15 and 20) mm with a constant 30 % of sisal fiber were investigated. The results reveal that 15 mm and 20 mm sisal fiber reinforced composite shows significant improvement in the flexural and impacted property (Doan et al., 2012). The treated woven mat sisal fiber composites improved three times compared to the untreated. The tensile force of 5% NaOH-treated agave Americana fiber was higher than raw and 10% NaOH-treated fibers (Mylsamy & Rajendran, 2011), (Cavalcanti et al., 2019a).

The impact strength of treated jute/curaua hybrid composite decreased by 25%, also decrease the tensile strength over untreated. This shows that treating all types of fiber not get always enhanced result and it depends on the nature of the individual fiber. If the composite result shows high impact value (high amount of energy absorbed) is directly related to the properties of interfacial bonding between fiber and matrix that low interfacial bonding (Yue et al., 1995).The effects of chemical treatment on water absorption properties of was studied for nano SiO_2 filled rice husk polypropylene composite and the result showed that reduces the water absorption tendency of the composite was reduced due to the removal of free hydroxyl group present in the surface of the fiber (Nourbakhsh et al., 2011). The effects of NaOH on mechanical and water absorption of almond shell-sugarcane leaves hybrid composite was evaluated and NaOH treated fiber composite shows an improved mechanical and water absorption properties than untreated (Mechanics et al., 2016).

2.5.4. Fiber volume effects

Fiber volume plays a significant role in determining the physical and mechanical properties of composites. This means that reinforcing fibers have weakened the composite rather than strengthened it, and the composite strength is even lower than that of the matrix alone. On the other hand, if the content of fiber exceeds the, fibers failure results in composite failure, since the polymer matrix is not able to carry an additional load transmitted to the matrix from the fibers.

It has been reported that the mechanical properties of composites improve with certain increases in fiber volume (V_f) (Brahim & Cheikh, 2007). Therefore, the proper selection of a fiber volume is of paramount importance for strengthening the composites. The tensile strengths of the natural fiber reinforced polymer composites increase with fiber content, up to a maximum or optimum value, the value will then drop. However, the Young's modulus of the natural fiber reinforced polymer composites increase with increasing fiber loading (Phiri & Sadiku, 2014). When fiber loading was increased, the composites became stiffer and harder leading to a decrease in elongation at break of the composites. With increasing fiber loading, tear strength of the composites increased due to fiber to the tear path more difficult of crack propagations (Wongsorat & Suppakarn, 2014)**.** Flax fiber was studied by varying of fiber loading (8, 15, 23, 30 and 35%) and the result showed that maximum fiber loading having maximum flexural and breaking energy (Ray et al., 2001).

Oil palm empty fruit bunch (EFB)/jute fiber reinforced epoxy bi-layer hybrid composites fabricated by hand lay-up technique having total fiber loading of 40% by weight (1:4) and the effect of jute the effect of jute fiber loading on tensile, storage modulus, loss modulus and damping properties were studied by comparing with pure EFB and jute composite. The result showed that, storage modulus of EFB and jute (4:1) hybrid composite is lowest. It shows that when jute fiber loading is increased, the effectiveness of stress-transfer is increased. Hybrid composite materials exhibits unique properties as compared with Oil palm empty fruit bunch (EFB) and jute composite, which can explore potential applications of oil palm–epoxy composite with jute fibers (Jawaid et al., 2015)**.** The fiber loading up to certain limit mostly improves the mechanical properties of natural fiber reinforced polymer composites (NFRPCs) (Brandt, 1997)**.** The effects of treatment and fiber volume was studied on jute/sisal hybrid composite and result showed that as the fiber volume of untreated and treated jute increase the tensile, flexural, impact strength and hardness was increased (Premnath, 2018). The effects of fiber volume on impact strength of Jute/Sisal fiber hybrid epoxy composites were studied and the result showed that impact energy increasing with increasing weight fraction of sisal fiber up to 35 wt.% (Sivakandhan et al., 2019). Maximum stress can be seen in composites with 1:1 ratio of banana and sisal. As fiber loading increases, flexural strength also increases up to 40% and then decreases. The fiber/matrix interaction is improved up to 40%, and on further loading, the fiber-to-fiber contact increases and fiber agglomeration results which lead to a decrease in stress transfer between the matrixes (Idicula et al., 2010).

2.5.5. Fiber orientation effect

The other main factor that affects the mechanical properties of fiber-based composite is the way the fiber will be oriented. The properties and structure of fiber base composite were defined by its fiber orientations (Rajak et al., 2019). Rather than fiber volume ratio, fiber orientation has more influence on the physical properties of fiber composites (Ramesh et al., 2016).

2.5.6. Hybridization effects

Hybridizing is a promising strategy to improve the mechanical properties of its composites. The properties of individual fiber composite were improved by hybrid with other fibers to mitigate the disadvantage of one fiber by others, keeping advantages of others (Chaudhary et al., 2017) (Rajak et al., 2019). The impact strength of pure Palmyra Palm Leaf Stalk Fiber (PPLSF) polyester composite is higher than that of hybrid and pure jute polyester composites and this shows properties drawback of individual fiber composites improved by hybridizing with fiber treatments (Shanmugam & Thiruchitrambalam, 2013a).

The mechanical properties of jute/sisal hybrid epoxy composite with 0/35,10/25,20/15, 25/10 and 35/0 volume ratio are investigated and the result shows that sisal fiber composite has better impact strength and triaxial tensile strength but jute fiber composite have higher flexural strength and co-axial tensile strength. Generally, Jute/Sisal hybrid epoxy composite result shows that increasing jute fiber improve the flexural properties and increasing sisal fiber improve impact and tensile strength of hybrid composites (Sivakandhan et al., 2019). Dynamic mechanical properties and water absorption characteristics of hybrid sisal and jute fiber reinforced epoxy composites were investigated. from their work, they concluded that natural fibers are a better replacement of synthetic fibers due to its acceptable mechanical properties ((Gupta and Srivastava, 2015). The variation in mechanical properties such as tensile, flexural and impact strength of hybridized kenaf and pineapple leaf fiber reinforced high-density polyethylene (HDPE) composites has been studied. Hybridization effects of Kenaf/PALF was studied that pineapple leaf fiber improve the tensile and flexural strength properties whilst kenaf improved impact and water absorption properties (Aji et al., 2012)**.**

Evaluation of the effect of hybridization on the mechanical performance of short banana/sisal hybrid fiber reinforced polyester composites found that tensile properties of NFCs were improved with the addition of banana fibers (Idicula et al., 2010). The maximum tensile strength (58 MPa) was obtained for composites having a ratio of banana: sisal of 3:1 at 67 vol.% fiber content. The results were explained as being due to the smaller diameters of banana fibers compared to sisal fibers and better stress transfer in unit area of banana/polyester composite. The hybridization of jute/ sisal and jute/ Curaua impact properties are analyzed and the result showed that in all hybrid composites better when compared to pure jute (Cavalcanti et al., 2019a).

In (Idicula et al., 2010), evaluation of the effect of hybridization on the mechanical performance of short banana/sisal hybrid fiber reinforced polyester composites was conducted and found that tensile properties of the natural fiber composites (NFCs) were improved with the addition of banana fiber. The mechanical properties of manually operated hot compression mold banana/jute fabricated composite were studied (Gowda, 2017) and the result indicated that tensile and flexural strength enhanced up to a certain level of fiber loading and impact strength was improved as the fiber volume increased. In the same study, the mechanical properties of the hybrid composite increased slightly with the increased thickness of the composite**.** The mechanical and moisture absorption of banana epoxy composite and banana and sisal hybrid composite was studied (Venkateshwaran et al., 2011) and the results showed that hybrid composite has better mechanical properties than banana epoxy composite.

The hybridization of jute/ sisal and jute/ Curaua impact properties are analyzed and the results showed that the properties in all hybrid composites are better when compared to pure jute (Cavalcanti et al., 2019a). Previous studies (Elanchezhian et al., 2018) also show that the impact strength of the fiber-reinforced polymeric composites depends on the nature of the fiber, polymer, and fiber-matrix interfacial bonding.. The reduction in water uptake is not entirely dependent on the natural fiber but the compatibility and synergistic behavior of fiber and matrix that increased their adhesion to the matrix improved their covalent bonding, which resulted in reduced overall water uptake by the hybridized composite (Athijayamani et al., 2009).

2.5.7. Composite fabrication methods

Molding is the best and widely used for the fabrication and processing of thermosets by most of researchers. The most common thermosetting polymer composite fabrication methods are open mold like hand Layup and filament winding (Chaudhary, 2017), closed mold likes resin transfer molding, compression molding and injection molding and Pultrusion,(Hahn, 2000; Rouison et al., 2002, 2004), (Athijayamani et al., 2009),and (Peng et al., 2011; Santulli, 2010). Brief details of these methods are presented in the following Table 2.8. The detail matrix hardener ratio with composite fabrication size of NF hybrid composite was tabulated in Table 2.7. The advantages and disadvantages of different NFRC fabrication methods were tabulated on Table 2.8 (Lotfi et al., 2019). A hand lay-up technique is the most common fabrication of natural fiber hybrid composite as mold and layer of sequence for woven types of composite. Most thermoset composites are fabricated by manual layup and 10:1 ratio of polymer hardener ratio is widely used (Naheed Saba et al., 2015). (Yahaya et al., 2015)

Table 2. 7 Ratio of polymer and hardener used in hybrid natural fiber reinforced composites

Type of fiber used	Type of polymer	Hardener types	Polymer hardener ratio	Fabrication methods	Mold size	Reference
Kenaf, Aloe Vera, Jute	LY556	HY951	10:1	Compression moulding	27 cm × 27 cm	(Athijayamani et al., 2009)
Banana/ Sisal	LY556 density 1.15–1.20 g/cm^3	HY951 density 0.97–0.99 g/cm^3	10:1	hand lay-up technique	300 mm × 300 mm × 3 mm	(Venkateshwaran et al., 2011)
Banana/ pineapple	LY556 density 1.15–1.20 g/cm^3	HY951 density 0.97–0.99 g/cm^3	10:1	hand lay-up technique	200 mm × 200 mm × 3 mm	(Madhukiran et al., 2013)
Bamboo/ Sisal/glass	LY951	HY 556	10:2	hand lay-up technique		(Balachandar et al., 2019)
Sisal epoxy composite	AY-105	HY-951	10:1	*	**	(Gupta and Srivastava, 2015)

* Composite load 30 kg for 24 hrs before removing from mold and cured for 24 hrs after removing from mould.
** Stainless steel mold dimension 500 mm × 300mm × 50 mm Composite was made of dimension 400 mm× 200 mm × 3 mm

Hand lay-up is the simplest method of processing the thermosets-based fibers composite. Advantages of hand lay-up are the relatively lower mold costs, the tools required for production are readily available and the molds are easy to maintain. Most of natural fiber hybrid composite were used to fabricate the composite bamboo/sisal/glass, banana/pineapple, Banana/Sisal (Venkateshwaran et al., 2011)**,** (Madhukiran et al., 2013)**,** (Balachandar et al., 2019)**.** In hand lay-up technique, thin plastic sheets are used on bottom of mold and then a releasing agent is sprayed on it to avoid the sticking of polymer to the surface of mold and to get good surface finish of product. Enset fibers as a reinforcement placed at the upper surface of mold.

Table 2. 8 Advantages, disadvantages, and production volume of various composite fabrication techniques.

Process	Advantages	Disadvantages	Production
Hand Layup	• Simple principles • Versatility • Low cost tooling • Suited for large size • Great choose of material	- Labor intensive - Health and safety - Not economical - Low	Low volume
Compression molding	• Low cost at high volume product. • High surface quality • Improved impact strength & uniform shrinkages • High dimensional accuracy	- High equipment cost - Not suitable for structural parts. - Limited to the size of parts	High volume
Resin transfer molding	• Good surface finish on both side • Array combination of reinforcement and orientation • Uniform thickness of composite parts • Low material wastage	- Limited size of composite - Complex mold design - High tooling cost - Limitation on reinforcing material contents	High volume
Extrusion	• Fast and economical processes • High structural properties of laminate • High fiber volume fractions can be obtained • Dimensionally repeatable	- Limited to constant or near constant cross-sectional parts - Limited to part size due to amount of fibers	Medium Volume
Automated fiber placement	• Less labor intensive • Less waste of material • Repeatability of result • Seamless transition between fabrication and design • Can produce large unique structures	- High capital cost - Head geometry limits - Laps and gaps issues - Distortion in thermoplastic composite processing	Medium volume

2.6. Sisal fiber and its composites

Sisal plant has the potential to grow in arid regions and hot climates that are often unsuitable for other plants. It can grow in almost all types of soil except clay. The cultivation of sisal plants is simple, and it is resilient to diseases. It can produce 120 to 240 leaves, depending on the location, rainfall, and altitude. The weight of a normal leaf is around 600 g and each leaf contains about 1000 fibers longitudinally. (Sreekumar et al., 2007) studied the detail investigation of sisal fiber and impact of various chemical treatment on mechanical properties. Decortication is the most common method for extracting sisal fiber. In this process, the leaves are crushed between blunt knives and moisture and the fleshy pulp are removed from the fiber. Water is used to clean debris that is present in the leaves. The sisal fiber that is obtained is dried in the hot sun. Several studies have reported that sisal fiber can act as a potential reinforcement of polymer composites can be used effectively in automobiles, such as their internal engine covers, door panels, hat racks, package trays, sun visors, seat backs, and exterior or under-floor paneling. They have been used in interior paneling in the aerospace and aircraft industries. Thermosetting polymer are widely used for plant fiber and hand layup followed by hot compression molding is a simple and efficient fabrication methods (Li et al., 2006). Sisal fiber (SF)-based composites are frequently being used for automobile interiors and upholstery in furniture due to their good tribological properties (Rajak et al., 2019).

Compared with polyester, sisal fiber-reinforced polyester composites have three times higher impact strength and up to 40% volume ratio the tensile strength increased. Chemical treatment of sisal fiber resulted in better mechanical properties. The chemical treatments improved the tensile, flexural and impact strength of the hybrid sisal banana fiber-reinforced PLA composites (Asaithambi et al., 2013). (Gupta & Rao, 2018) studied the behavior of hybrid sisal- hemp hybrid that having low-cost bio-reinforcement in polymer composites with a higher strength to weight ratio. Hybridization of sisal fiber in the banana epoxy composites up to 50 wt% improved the mechanical properties and decreased moisture absorption were studied by (Venkateshwaran et al., 2011).

2.7. Enset fiber and its composites

Tensile, flexural, compression strength of Enset and Banana fiber gypsum composites were studied. The fiber orientation, matrix types and thickness of fibers are the main factors to determine the mechanical properties of both composites. the result shows that diagonally oriented and higher fiber thickness Enset fiber-reinforced composite better tensile and flexural strength than banana composite (Tadasse et al., 2018). Ensete ventricosum is widely cultivated in East Africa and mostly used for food crops. Though fiber extraction from Enset stem is economical and as a byproduct it is abundant, Enset fiber at present is a waste product of enset ventricosum cultivation.

The pseudostem maybe two to three meters tall and contains an edible pulp and quality fiber. Enset provides fiber as a byproduct of decorticating the leaf sheaths. In rural areas the enset fiber is used to make sacks, bags, ropes, cordage, mats, construction materials (such as tying materials that can be used in place of nails), and sieves, however, only a few more studies were yet performed to investigate its use in the fabrication of green polymer composites. Moreover, without any additional cost input, enset fiber can be obtained in bulk quantity. Ensete ventricosum the only known wild species in Ethiopia, abundantly concentrated in southern highlands and southeast part of Ethiopian. Enset, however, is usually larger than a banana, with the largest plants up to 10 meters tall and with a pseudostem up to one meter in diameter. Similar to other cellulosic natural fibers, Enset fiber is chemically composed of cellulose, hemicellulose, lignin, pectin, moisture, wax and oils (Brandt, 1997). The ASTM standard size for mechanical testing of natural fiber hybrid composite tabulated on Table 2.9.

Table 2. 9 The existing literature of natural fiber hybrid composite sample standards for mechanical testing.

No	Composite	mold size	Standards	Sample size	Test	Reference
1	Sisal/jute	(300 × 300 × 3) mm (l × w × t)	ASTM D790	(13 × 127 × 3) mm (l × w × t)	Flexural	(Sivakandhan et al., 2019)
			ASTM D256	(13 × 66 × 13) mm	Impact	
			ASTM D3039	(250 × 25 × 3) mm	Tensile	
2	Palm Leaf Stalk Fiber/jute		ASTM: D790	(127 × 12.7 × 3) mm	Flexural	(Shanmugam & Thiruchitrambalam, 2013b)
			ASTM: D256	(64 × 13× 3) mm	Impact	
3	Jute/Hemp /Flax		ASTM G99-95	Sliding speed (1-5 m/s) Applied load (10-15N)	Wear	(Chaudhary et al., 2018a)
4	banana/sisal	(300 × 300 × 3) mm	ASTM D 638–	Test speed of 5 mm/min	Tensile	(Venkateshwaran et al., 2011)
			ASTM D 790	Test speed 1.3 and 1.5 mm/min.	Flaxural	
			ASTM D 256	(65×13×3) mm with 1.2 depth notch	Impact	
5	banana and palm		ASTM D638	Each specimen has area 187.2mm2 and width 12.62mm, gauge length 50mm	Tensile	(Dinesh et al., 2019)
6	jute and sisal		ASTM –D 638	165×19×2×60mm(length	Tensile	(Cavalcanti et al., 2019a)
			ASTM	144×15×3×120mm	Flexural	
			ASTM	110×15×3×40mm	Impact	
			ASTM D 570	(75 × 75 × 5) mm	Water absorption	
7	Kenaf/Aloe Vera/Jute	(27 × 27) cm	ASTM	(250 × 25 × 3) mm (120 × 13 × 3) mm (66 × 13×3) mm (Ø15 × 500)mm	Tensile Flexural Impact Wear	(Arunkumar & Saravanan, 2018)
8	sugarcane bagasse/coir		ASTM D638	300× 50 × 5	Tensile	(Jayaraman, 2015)
			ASTM D 570	75 × 75 × 5	Moisture Absorption	
9	Bamboo/Sisal/ Glass		ASTM D638-0	250 × 25 × 3	Tensile	(Balachandar et al., 2019)
			ASTM D790	66 × 13 × 3	Flexural	
			ASTM	127 × 13 × 3	Impact	
			ASTM D638	165 × 20 × 3 with 53 mm gauge length	Tensile	

Where: l = length w = width and t = thickness

2.8. Machining of natural fiber polymer composite

Applications of composites may be impossible without some amount of machining involved during the assembly of the components. The surface quality of the machined composite component is very important in the assembly of mechanical parts and is an indicator of exactness of the manufacturing process. Drilling holes on a fiber-reinforced composite is an indispensable and inevitable operation that is required for assembly and maintenance operations. The drilled metal surfaces are smoother and more regular than the drilled composite surfaces under the same conditions (Nasir, 2015), due to the abrasive nature, heterogeneity and anisotropy of fiber reinforced composite (FRC) materials (Ramulu et al., 2001). Number of approaches have been used for making holes in composites, conventional radial drilling and CNC machining are widely used drilling methods till date (Bajpai et al., 2015), (Packiara and Kumar, 2009). During assembly operation processes, the drilled hole delamination factors lead to account the rejection of the composite products. In drilling, many factors affect the machinability; some of the important machining parameters are the spindle speed, the feed rate, and the drill diameter (Palanikumar et al., 2014). From drilling parameters feed rate and cutting speed affects delamination and surface roughness of natural fiber reinforced composites. The quality and performance of a composite product is highly dependent on the surface condition created by machining.

Surface roughness (Ra) defined as the average mean of the deviation of the roughness profile from the average line within the estimated length. In drilling, surface roughness is the indication of irregularity of circumferential drilled hole surface, the reason for the development of high wear, fatigue and corrosion mechanisms (Lotfi et al., 2019). Therefore, several researchers studied the effect of machining factors like feed rate, speed, and drill diameter to optimize the machining process in order to obtain a superior surface roughness. Surface roughness increase with decrease feed rate. Increase the cutting speed cause decrease the surface roughness. This behavior is mainly due to the decrease in cutting forces. Better surface finish can be achieved at higher cutting speed and lower feed rate. The experimental results also indicate that at low feed, the values of Ra increase with the increase in diameter of the drill (Lotfi et al., 2019).

Delamination is the most critical one among the defects that associate with machining. Damage during drilling happens both at the entrance and at the exit surface of the work piece. The damage that occurs around a drilled hole is known as damage factor or delamination factor or deface (Vinayagamoorthy, 2018).Delamination in a composite material occurs whereby reinforced fiber plies separate, either by peel up or push-out phenomenon (Oluwarotimi et al., 2016). Delamination is mainly an inter-ply failure phenomenon induced by an exterior force, such as drilling, which results in separation of different layers of reinforcement or plies. Delamination has been found as a major concern during drilling of composite laminates as it can result in a drastic reduction of structural integrity, assembly tolerance, surface finish quality, and bearing strength. The “peel-up” delamination which occurs around the periphery of the drill entrance and “push-out” delamination which occurs at the exit of the drilled holes periphery (Karnik et al., 2008), (Oluwarotimi et al., 2016) (Ismail et al., 2016), (Ramesh M. & Gopinath A., 2016). This defect was improved by proper selection of cutting condition like feed, speed, tool material and tool geometry, poor surface roughness of the hole wall and fiber/resin pull-out and among the above issues associated with drilling, delamination appears to be the most critical (Mudhukrishnan et al., 2019).

The delamination and surface roughness of drilled surface aloevera and woven sisal fiber reinforced with polymer composite was analyzed and the result showed that less delamination occurs at high speed and high feed rate. The same for the surface roughness (Sekaran & Kumar, 2019). The effects of drilling parameters with fiber ratio on delamination and surface roughness of hemp fiber-reinforced polycaprolactone was studied and the result showed that the delamination and surface roughness reduced with increasing the cutting speed, whereas delamination and surface roughness increase with increasing the feed rate (Ismail et al., 2016). The drill hole quality of Hemb fiber-reinforced polymer composite was studied and a smooth drill hole appeared with low feed rate, the moderate-high cutting speed with a small diameter of drill bits. Also, Continuous and coiled chip was formed during drilling of hemp fiber-reinforced polymer (HFRP) composite while discontinues chip in carbon fiber reinforced polymer (CFRP) composite. Drill hole damage like burrs and uncut-fiber are common in natural fiber reinforced polymer (NFRP) composite (Ismail et al., 2016**).** woven sisal and Aloe vera NFP composite surface roughness and delamination are main two-element studied by using carbide tip drill bit with having 6,8and 10 mm diameter and the result shows less delamination and surface roughness at high speed and high feed (Sekaran & Kumar, 2019).

Drilling hole damage was studied in case of sisal fiber-reinforced polylactic acid and Grewia optiva fiber reinforced polylactic acid and result showed that drilling-induced damage decreasing with cutting speed increase and decrease feed rate. The drill geometry and feed rate are critical parameters in drilling of green composite laminates for generating damage-free holes. Therefore, several research has been carried out to optimize the process parameters in order to achieve a desired surface roughness (Bajpai et al., 2015). The composites with 30 % roselle and sisal hybrid fiber content and 8h alkali treatment of fibers resulted in a better dimensional accuracy than other fiber volume fraction and treatment times during drilling (Athijayamani et al., 2009). The delamination minimum during drilling of hemp fiber reinforced composite than jute fiber reinforced composite (Jalumedi et al., 2015). Machining parameters value are chosen based on different literature. As literature shows that cutting speeds from 20 to 60 m/min, feed from 0.1-0.3mm/rev and 6-12 mm drill bit diameter were frequently used. Higher cutting speeds develop the temperature and makes the polymer based composite material is soften. Higher value of feed rate and drill diameter increase the damage around drilled hole and mostly not recommended by different researchers (Jayabal & Natarajan, 2011), (Nassar et al., 2017). A study on drilling of coir-reinforced composite reported that a lowest drill size of 6 mm, a spindle speed of 600 rev/min, and a highest feed rate of 0.3 mm/rev are found to be the optimum conditions for drilling (Jayabal et al., 2013). In on work the three variables namely, speed (v), feed (f) and drill bit diameter (*D*) which are category in three levels.

The L9 orthogonal array was developed for analyzing three variables and three levels in each variable(Jayabal & Natarajan, 2011):

Regression models play an important role in statistical methods in machining process. For analyzing the responses, regression models are developed. The regression line formulate the correlation between the cutting parameters with regards to the responses (delamination and surface roughness). The regression equation can be represented as (Mudhukrishnan et al., 2019);

Response = constant + coefficient (predictor) + coefficient (predictor)

or

$$Y = \beta_0 + \beta_1 X_1 + \beta_2 X_2 + \beta_3 X_3 + \cdots + \beta_n X_n \qquad (2.5)$$

Where the Response (Y) can represent either delamination or surface roughness, β_0 is constant value when the predictor variables such as speed, feed and drill diameter is zero (0). β_1 , β_2 …. are the coefficients. In our investigation, delamination and surface roughness are used as response variables. The model considered consists of the first order, the interaction and square effects. The responses with respect to the cutting parameters are analyzed and the statistical regression models are developed.

2.9. Tribological study of natural fiber reinforced polymer composite

Tribological behaviors are the main cause for mechanical failure leads to heavy cost of the component parts in the industries (Bajpai et al., 2013). Pin on disc technique was used for experimentation and other parameters considered were 20-40 N applied loads, 0-30 Km sliding distances and at a constant sliding speed of 0.5-4 m/s. Authors concluded that specific wear rate was sensitive to the change in applied loads and sliding distances (Yousif et al., 2012).

Maximum wear studies available are on either on unidirectional mat or randomly oriented mat based composites. Wear resistance of (jute, hemp and flax) hybrid epoxy polymer composite was investigated that reinforcement of different combination of natural fibers has substantially enhanced the wear performance of epoxy. Wear analysis confirmed that incorporation of natural fibers into epoxy polymer matrix significantly improved the wear behavior of the developed NFRP composites in comparison to neat epoxy polymer. When the sliding speed increases the interfacial temperature increase and applied load increases. Debonding, fiber pull-out and tearing of fibers were the major factors for wear of the developed composites as depicted by SEM micrographs (Chaudhary et al., 2018a). Natural fibers have been found to be beneficial for their mechanical properties and wear resistance of polymers because, the main objectives of designing good tribological properties of natural fiber polymer composites (NFPCs) are to reduce the wear volume loss or wear rate and coefficient of friction. In wear test, the composite specimen with alkali-treated fiber content gives a better wear resistance than untreated fiber content composite (Wong et al., 2010). Loading, sliding distance, fiber orientation, fiber volume fraction, are the main parameters that considered by different researchers for the analysis of wear rate and friction of coefficient for NFRCs (Yousif et al., 2009).

2.10. Classification of polymers

The polymer can be classified into two classes, thermoplastics and thermosetting. The most used thermoplastics are polypropylene (PP), polyethylene, and polyvinyl chloride (PVC); while phenolic, epoxy and polyester resins are thermosetting matrices. Mostly thermosetting types of polymers can be bonded to nearly all materials like wood, glass, natural fibers, and metal. They exhibit little or no shrinkage after curing (Sawpan, 2009). Most of thermosetting polymer are decomposing on heating, low strains to failure, definite shelf life, cannot reprocessed, low fabrication temperature but thermoplastics polymers are not (Kaw, 2006) The advantage and disadvantages of thermoset and thermoplastic polymers are tabulated on Table 2.10 (Joseph. et al., 2003), while their mechanical properties are tabulated on Table 2.11 (Kaw, 2006).

Table 2. 10 Advantages and disadvantages of thermoset matrix polymers

Polymers	Advantage	Disadvantages
Thermoset	- Low resin viscosity - Good fiber wetting - Excellent thermal stability once polymerized -Chemically resistant	-Brittle -Non-recyclable -Standard techniques -Not post-formable

Table 2. 11 Mechanical properties of thermoset matrix

Types of thermoset resin	density (g/cm3)	tensile strength (MPa)	Young's Modulus (GPa)	Elongations (%)
Phenolic	1.2	40-50	3	1-2
Polyester	1.2	50-65	3	2-3
Epoxy	1.1.-1.4	50-90	3	2-8
Vinylester	1.15	70-80	3.5	4-6
Polyimide	1.42	70-150	2.5	8-70

A polyester resin is an unsaturated (reactive) polyester solid dissolved in a polymerisable monomer. Unsaturated polyester (UPE) resin is a condensation polymer formed by the reaction between a glycol and an unsaturated dibasic acid (maleic or fumaric of polyol and polycarboxylic acid (Sawpan, 2009). Unsaturated polyesters are long chain, linear polymers containing a number of carbon double bonds(Njoku et al., 2011).A schematic chemical structure for an unsaturated polyester resin is presented in Figure. 2.4 (Njoku et al., 2011).

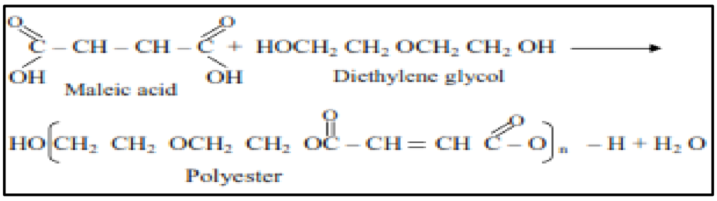


Figure 2. 4 Schematic chemical structure of UPE.

Polyester resin is a feasible polymer, which has effective strength, good toughness, appreciable resilience, good resistance to moisture and chemical attack and also found in more than 20 grade to meet specific property (Sawpan, 2009).

2.11. Applications area of natural fibers

Today in modern industries, natural fiber-based polymer composites have potential applications to replace synthetic fibers-based polymer composites. This is due to natural fibers being extensively available in the environment, low cost, renewable, and environment friendly (Mohammed et al., 2015), (Rajak et al., 2019)**.** The most application area of natural fiber composites are building and construction, furniture, electronics, automotive and everyday appliances, etc. (Elanchezhian et al., 2018). Enset plant and Sisal plant widely available that having full of fiber that promising for different applications. In rural areas, the Enset fiber is used to make sacks, bags, ropes, cordage, mats, construction materials (such as tying materials that can be used in place of nails), and sieves etc. (Brandt, 1997). In advance the enset fiber composite promising for the fabrication of automotive components like parcel shelves, dashboard, seat cushion, door trim panel, backrests and cabin linings, for non-structural applications like in packaging industries (egg shelves), consumer products and sports items (racket handle, bicycle frames) (Negawo et al., 2019).

The sisal fiber is traditionally used for rope and twine and has many other uses, including paper, cloth, footwear, hats, bags, carpets, and dartboards (Mylsamy & Rajendran, 2011)**.** Natural fiber-based composites used for potential automotive applications due to light in weight and environmentally friendly materials. Studies indicate that natural fiber composites can contribute to a cost reduction of 20% and a weight reduction of 30% of the automotive part. This reduction of weight leads to lower fuel consumption, reduction in the waste disposal and greenhouse emissions. Today natural fiber used for interior parts of automotive parts such as dashboards, door panels, parcel shelves, seat cushions, backrests and cabin linings seat backs, front and rear door linens, and truck linens. But for exterior applications using natural fiber composite is very limited (Mohammed et al., 2015), (Bavan & Kumar, 2010), (Lotfi et al., 2019) (Carus et al., 2015).

There are other industries which gradually branched out to products segment made of natural fiber and composites as well, such as furniture, medical, sports, among others. Most of Automotive parts are fabricated from natural fiber composite as shown in Figure.2.5 (Mohammed et al., 2015).

Figure 2. 5 Automobile components made of natural fiber composites

Therefore, in our country Ethiopia enset and sisal fibers are abundantly available resource among other natural fiber resources. So, investigating the fiber mechanical, physical and chemical property is very important before developing composite and its product. Also, study the mechanical, physical, machining and tribological properties and enset/sisal hybrid composite is mandatory before converting those abundantly available resources. While converting this research work to the real applications having multi-advantages to the economy, environment, and job opportunity for sustainable development etc.

2.12. Observed literature gaps

Even the natural fiber-based polymer composite is replacing the synthetic fiber composite materials still their mechanical performance is not sufficient. However, the mechanical performance of a composite material depends on the fiber types and matrix, fibers loading, fiber/matrix adhesion, fiber treatment, fiber length, fiber orientation, and hybridization (Dong, 2018), (Graupner et al., 2016). Sisal/epoxy composite are the most common studied and commercialized in interior part applications. However, few studied are present on sisal/polyester and enset/polyester composites last 2 years. But, the effects of hybridization, for sisal and enset are not studied before. In this research the effects of fiber volume, treatment and fiber orientation on enset/polyester, sisal/polyester and enset/sisal hybrid polyester composites will be studied for mechanical performance, environmental resistance, machinability and tribological behavior. Figure 2.6 shows existing and gap of literature on enset and sisal fiber polymer composites.

As the overview of the research in the area indicates, various researchers have conducted study of the effects of alkali treatment on different natural fibers. The properties of plant fibers naturally depend on the climate in which the plant grows. The study of enset and sisal fibers in this region of Ethiopia that this study targets is so far not reported. Therefore, this study aims to characterize the fibers of enset and sisal from South-West

Ethiopia by experimental investigation of the effects of alkali treatment on the physical, chemical composition and mechanical properties as a probe for reinforced polymer composites.

From the above literature, fiber treatment, fiber volume ratio and fiber orientation are the main parameter that affects the mechanical properties of natural fiber-based hybrid composites. In the present study, untreated, 5% and 10% NaOH treated enset and sisal hybrid composite were fabricated in the orientation of unidirectional and woven types.

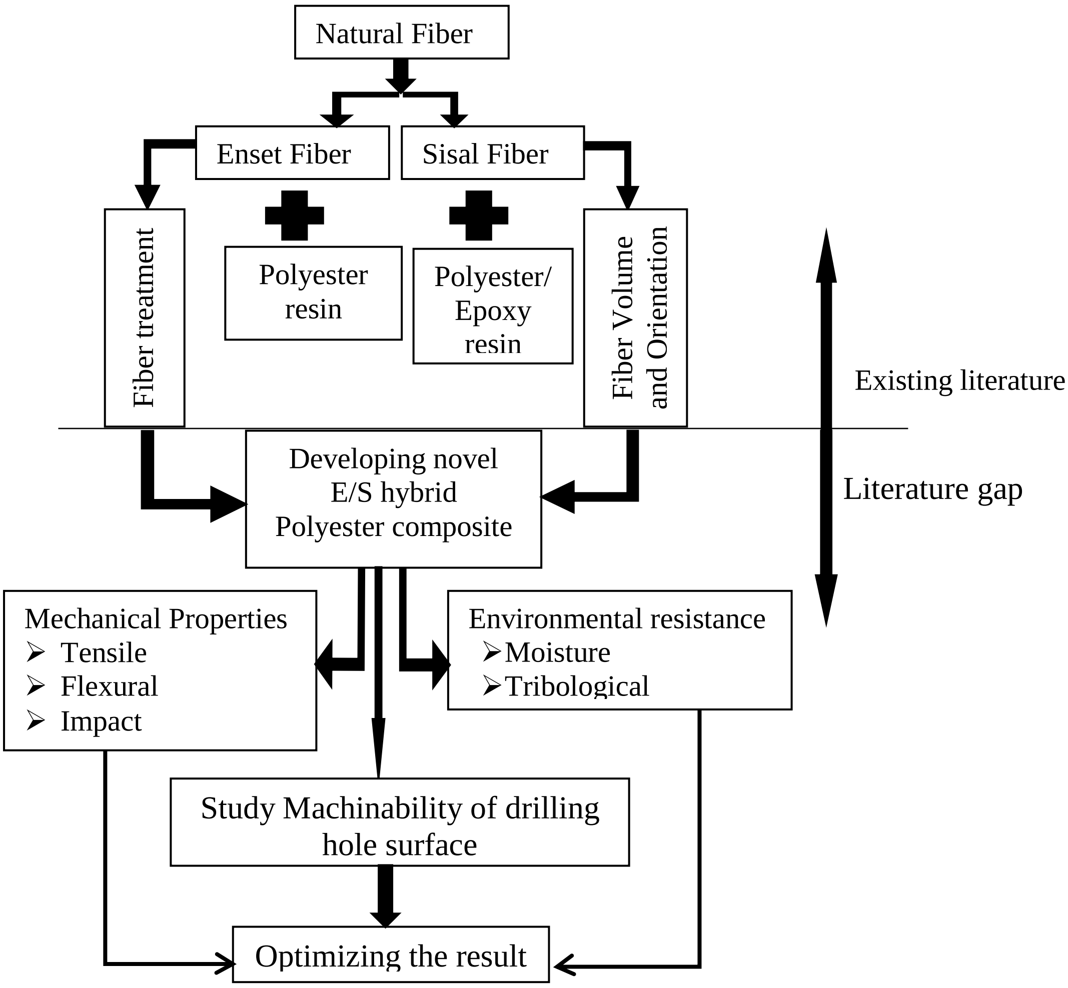


Figure 2. 6 Existing and gap of literature on enset and sisal fiber composite

The effects of treatment on enset and sisal fibers were characterized, and again the effects of treatment, volume ratio and orientation on mechanical and physical properties of enset/sisal hybrid reinforced polyester composites were analyzed. By choosing the optimum volume ratio and treatment based on the result of mechanical and physical properties of enset/sisal hybrid composite. After selecting the optimum value, study was done on the machinability and tribological properties of woven and unidirectional E/S hybrid polyester composites. The fractured surfaces of the composites were being analyzed using scanning electron microscopy (SEM) and 3D optical profilometer.

CHAPTER-THREE

3. Materials and Methods

3.1. Introduction

In this research, enset and sisal fibers were used to produce polyester matrix hybrid composites, employing both unidirectional and woven fibers. Both fibers were treated with 5% NaOH and 10% NaOH was investigated to fibers/matrix interfacial and mechanical properties of composites. A set of physical, mechanical and thermal composition for the fibers and composites was adopted to obtain a thorough understanding of the composites behavior to help identify the possible ways in which performance might be improved. This chapter describes the materials and approaches including fiber treatment methods and fiber characterization techniques, composite processing and mechanical testing methods, composite machining study approach and tribology study methods.

3.2. Materials and equipment

Enset and sisal fibers for this study were collected from Oromia region around Woliso, South-West Ethiopia due to availability of fiber resources. Alkali (NaOH) was used to remove amorphous materials from the fibers and to determine hemicellulose contents of the fibers, and sulfuric acid (72%H_2SO_4 solution) was used to determine fiber lignin contents and ethanol- Tolune (1:2) ratio, which was prepared to use for determination of wax contents, and HCl was used to neutralize the 5%NaOH during determination of hemicellulose percentages of the fibers. All these chemicals were purchased from local market. To determine the fiber chemical properties, laboratory equipment such as soxhlet apparatus, sensitive balance, oven, furnace and water bath were used. In addition, optical microscopy (OM) and Fourier transform infrared spectroscopy (FTIR) were used to determine the diameter and chemical compositions of the fibers respectively. Fibers mechanical properties were determined using AUTODYN-II machine yarn (bundle) tensile test. Mechanical properties of the composite were determined using Universal testing machine (UTM) and Charpy test machine, while composite tribological properties were determined using Pin On Disc (POD) and morphology characterization were used Scanning electro-microscopy (SEM), for machining (drilling) Computer numerical control (CNC) machine, surface roughness of drilled hole surface using 3D optical surface profiler, analysis software using SSP, “ImageJ” and MINITAB18.

3.3. Methods

3.3.1. *Fiber extraction methods*

The matured enset was cut and the fiber was extracted from pseudostem part using decortication methods based on the existing literature explained under Section 2.4.2. The fiber was dried in sunlight for 8hr. The sisal fiber was extracted from leaf parts of Agava sisilana plant using manually extraction methods and dried in sun light for 8hr. The dried fibers were washed by fresh water followed by distilled water for several time and then combing with same sized comb to remove fragments, dusts and impurities and dried at sunlight for 8 hr (Yan et al., 2016) that is shown Figures. 3.1 (a) and (b). Enset and sisal fiber were immersed in prepared 5wt% and 10wt% NaOH concentrations for about 2 hr at room temperature. Then washed fibers several times with fresh and distilled water to take away any NaOH sticking on the fibers surface and dried at room temperature for 24hr (Mishra et al., 2001). Treatment of fibers were used to break the hydrogen bonding in the network structure that modified the cellulosic molecular structure by removing the amorphous for making strong link between fiber and matrix.

Enset/Sisal fiber- cell- OH + NaOH $\longrightarrow$ Enset/Sisal fiber cell - O^-Na^+ + H_2O + impurities

The enset and sisal plant and their traditional fiber extraction methods are shown in Figure 3.1(a) and Figure. 3.1(b) respectively.

3.3.2. *Treatment of fibers*

The enset and sisal fibers were immersed in 5wt.% and 10 wt.% NaOH concentrations for about 2 hrs at room temperature. Then, fibers were washed several times with fresh water then followed by distilled water to take away any NaOH sticking on the fibers surface and dried at room temperature for 24 hrs. The 5% and 10% NaOH alkali treated enset and sisal fibers are shown in Figures 3.2 (c) – (f) respectively. Those parameters are chosen based on the existing literature explained under Section 2.5.3.1 in Table 2.4.

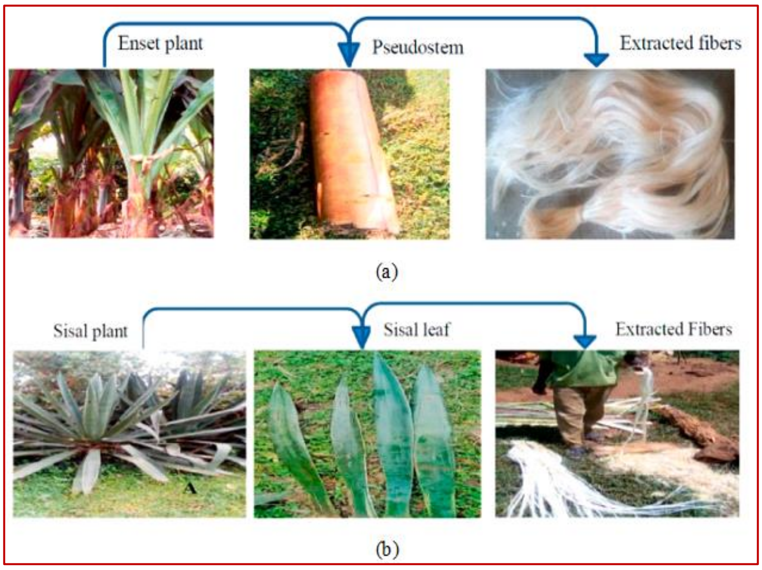


Figure 3. 1 Fiber extraction (a) enset fiber and (b) sisal fiber

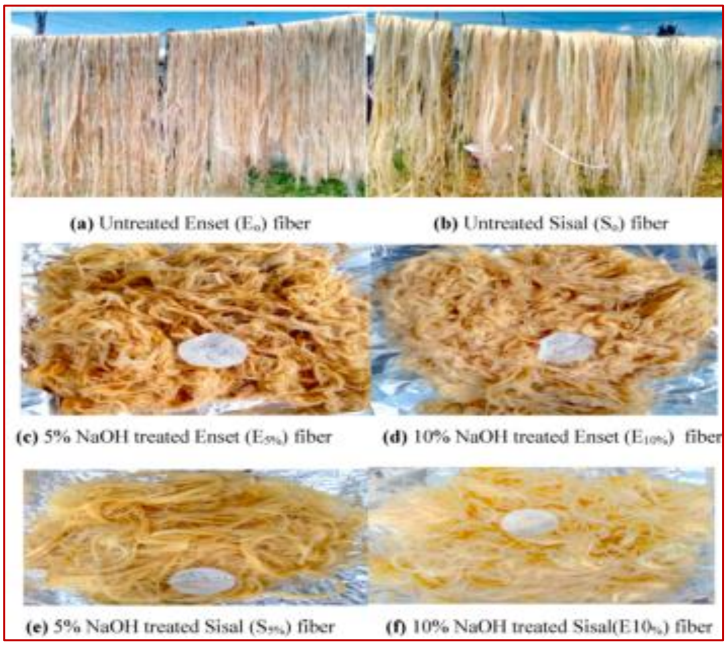


Figure 3. 2 Untreated and treated with 5% and 10% NaOH of enset and sisal fibers

Figure 3.3 shows the detail information on composite sample preparation in which 5 samples (i.e. 2 individual fiber volume + 3 hybrid fiber volume × **a** (untreated, 5% and 10% NaOH treated fibers) × **b** (unidirectional and woven fiber orientation) composite were fabricated. In total 30 samples (i.e. 5 different fiber Vols. × 3 treatments × 2 fiber orientations) were produced.

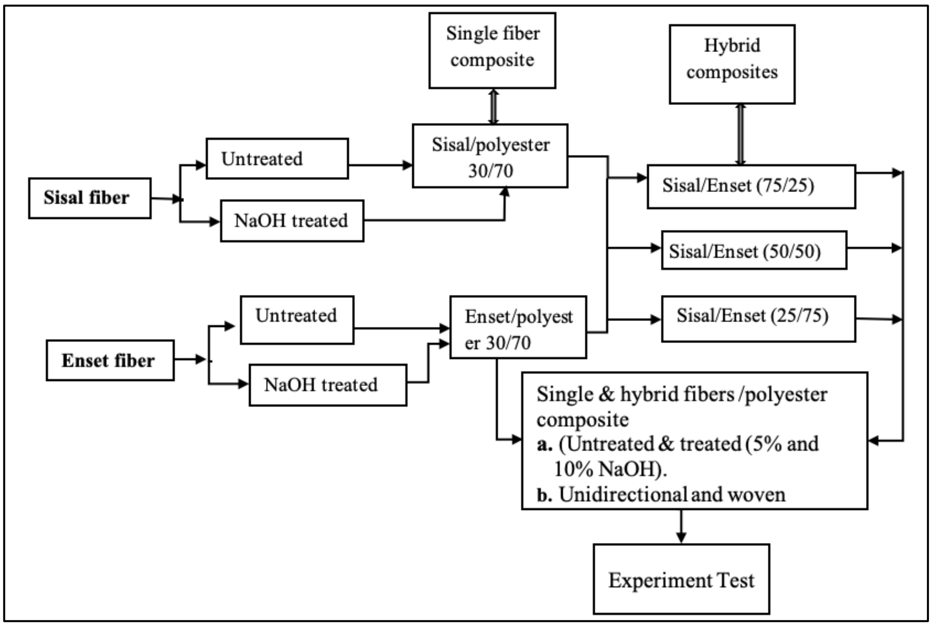


Figure 3. 3 Manufacturing setup of E/S hybrid composites

Figure 3.4. shows the detail approach of laboratory work for the work procedure to achieve the research work, in which the extraction, treatment techniques, fabrication and test were summarized.

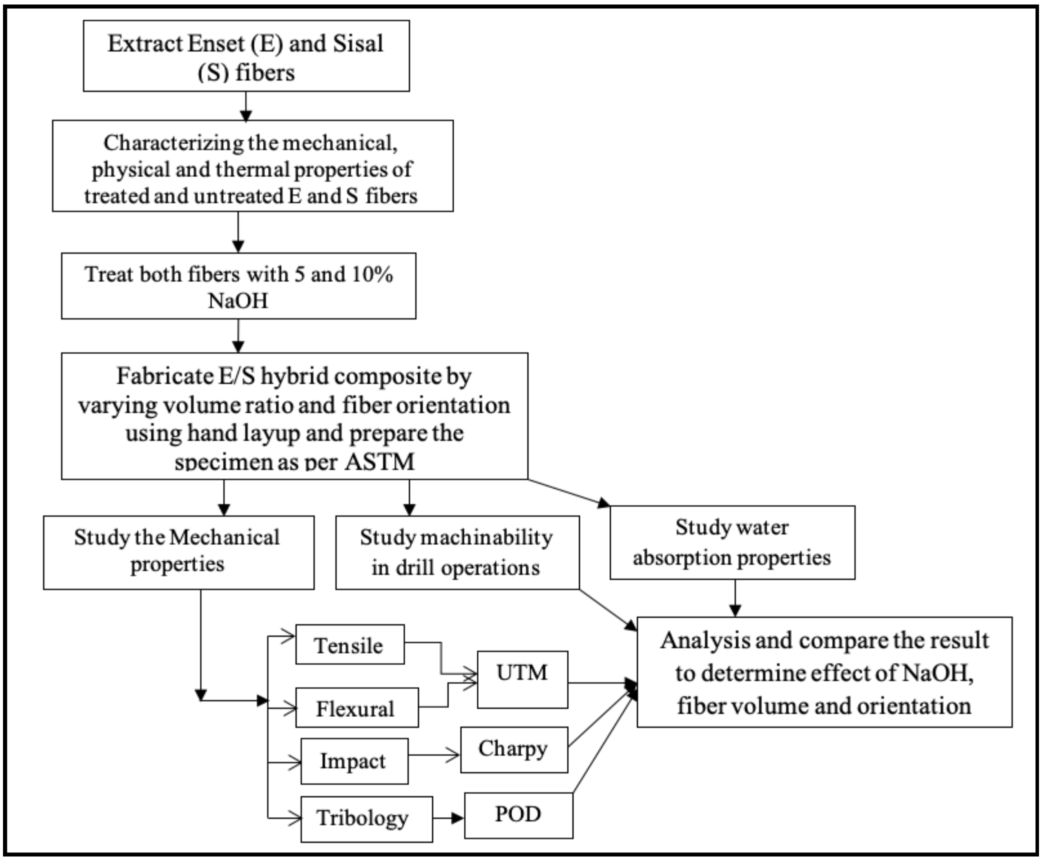


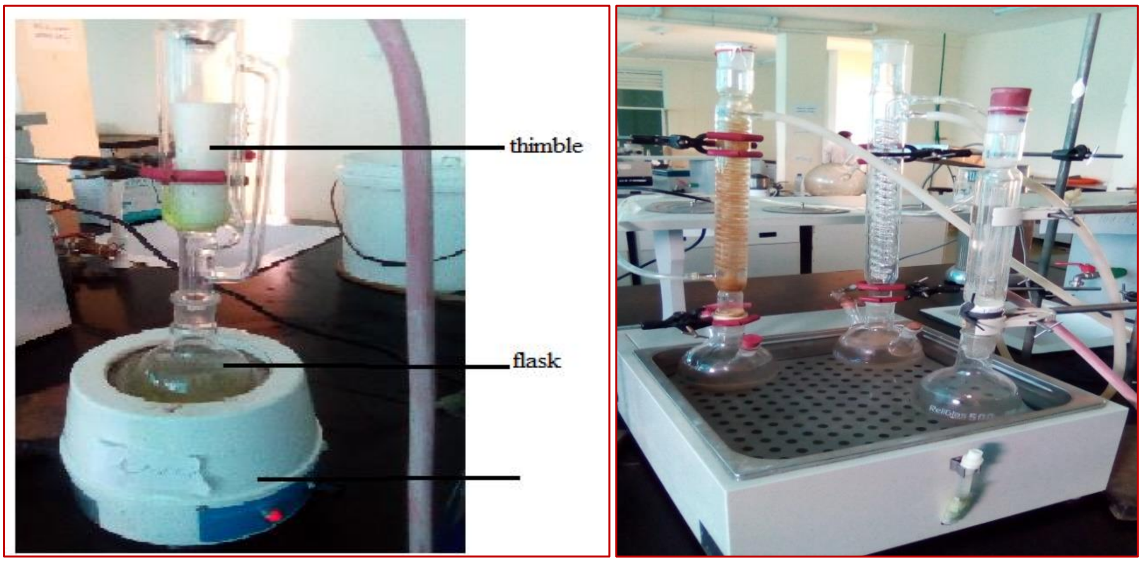
Figure 3. 4 Methodology

3.3.3. *Chemical composition of fibers*

The chemical composition of the fiber influences its properties and the fibers themselves could be considered as fibrous composite materials (Han & Rowell, 2005). Cellulose is the main component in the fiber. Hemicellulose is composed of cellulose chains, but it is much shorter in length and is built up of a mixture of polysaccharides of relatively low molecular weight. It is also responsible for biodegradation, moisture absorption, and thermal degradation of the fibers. The lignin content influences the fiber structure, properties, morphology, strength and moisture absorption when compared with hemicellulose (Thygesen et al., 2012). Also lignin is thermally stable, but responsible for the ultraviolet degradation of the fibers (Edeerozey et al., 2007). Chemical compositions of fibers were determined using soxhlet apparatus as shown in Figure 3.5. A gravimetric method was used to determine the contents of pectin, lignin, hemicellulose and cellulose in fibers ((Mooney et al., 2018), (Ioelovich, 2015), (Willis et al., 1988). To know the percentages of lignin, cellulose and hemicellulose contents of sisal and enset using gravimetric methods as per ASTM D1106 standard.

3.3.3.1. Wax contents determination

The wax content was measured with the help of soxhlet apparatus as per D 1107 – 96 (Han & Rowell, 2005). The ethanol-toluene solution (1:2) ratio was prepared. Two grams of treated and untreated enset and sisal fibers were extracted for 8 hrs. Then, after refluxing for 1 hr, the fiber sample was dried in an oven and it was weighed and the weight difference confirmed the wax content.



(a) Soxhlet apparatus (b) water bath

Figure 3. 5 Extraction equipments used for the determination chemical compositions

3.3.3.2. Lignin contents of fibers

Manually extracted untreated and NaOH treated enset and sisal fibers were determined by gravimetric methods as per ASTM D1106 standard recommendation. The prepared 1 gram of treated and untreated enset and sisal fibers were placed in beaker, 72%H_2SO_4 was added slowly and stirred for 2 hrs and then after diluting the H_2SO_4 to 3% and refluxed for 3 hrs. Filter residue and washed by hot water several times and then after oven dried at 105 ^{0}C for 1 hr and weighed (w_2). The residue weight was noted as the lignin content was determined using Equation (3.1).

$$\% \text{ ligning} = \frac{w_2 - w_1}{w_s} \times 100 \qquad (3.1)$$

Where w_1 = weight of as sample w_2 = weight of oven dried sample w_s = initial weight of dried sample.

3.3.3.3. Cellulose contents of fibers

A one-gram sample was placed into 250 ml round bottom flask and reflux for an average of 1 hr in a mixture of concentrated nitric acid in ethyl alcohol. At the end of the extraction process, which lasted one hour each, the flask containing the extractive nitric acid solution was transferred into known weight of gauche crucible for proceed filtration process used under vacuum suction. The extraction materials were washed several times by distilled water. Then the acid mixture extractives were dried in oven at 105^{o}C for an average of 1 hr, cooled in desiccators, and weighed until a constant weight was obtain. The cellulose contents were determined by using Equation (3.2).

$$\% \, cellulose = \frac{w_2}{w_1} \times 100 \qquad (3.2)$$

Where w_1 = the amount of extract is free samples taken for analysis and w_1 = is the residual mass of cellulose

3.3.3.4. Hemicellulose contents of fibers

Hemicellulose is composed of cellulose chains, but it is much shorter in length and is built up of a mixture of polysaccharides of relatively low molecular weight. Hemicellulose is responsible for biodegradation, moisture absorption, and thermal degradation of the fibers. The dry fibers treated and untreated fiber of weighed quantity was immersed in a mixture of 5% NaOH solution at room temperature for ½ hr and neutralized by HCl and dry the residua and weighed. The weight difference accounts for the presence of hemicellulose content as shown by Equation (3.3).

$$\% \text{ Hemicellulose} = \frac{w_A}{w_2} \times 100 \qquad (3.3)$$

Where: $\mathrm{w_A}$ = Dried weight of precipitated hemicellulose, $\mathrm{w_2}$ = Dried weight of fiber sample

3.3.3.5. Ash contents of fibers

For the determination of fiber ash contents about 2 g of sample was put on pre-weighted crucibles and placed in the furnace at 575 ^{0}C for 3 hrs and cooled the crucibles in desiccator and weighed until the difference of two successive weights were less than 1 mg (Atalie & Rotich, 2018). Percentage of ash contents was calculated using Equation (3.4).

$$\%\ \text{of Ash content} = \frac{\text{Initial sample weight}}{\text{Final ash weigth}} \times 100\% \tag{3.4}$$

3.3.4. *Physical properties of sisal and enset fiber*

3.3.4.1. Moisture contents

To determine the moisture content of the untreated, 5% and 10% NaOH enset and sisal fibers, the sisal sample was dried in an oven at constant temperature for certain times and weighed in an electronic weighing machine. The percentage of moisture present per unit weight of each extracted fiber was evaluated as shown in Equation (3.5).

$$\%\ \text{MC} = \frac{W_{fb}-W_{fa}}{W_{fb}} - \times 100 \tag{3.5}$$

Where: MC is moisture contents of fibers, W_{fb} (gram) is weight of fiber before putting in an oven and W_{fb} (gram) is weight of fiber after putting in an oven.

3.3.4.2. Fiber diameters

Measuring the diameter of individual natural fiber was one of the major challenges. Generally, while observing the individual natural fiber, the cross-sectional area has irregular profile and not the same in throughout the length. Even though the cross-section is not uniform along the length of the fiber, it is assumed to be a circular cross-section during the calculation of the tensile strength of the fibers. The sample of individual fiber was cut into small splices, placed on to a microscopy slide and projected into screen through a microscopy. The enset and sisal fibers' diameter was measured using the optical microscopy and the image is shown in Figure 3.6. Randomly selected 10 samples were measured at 5 locations along the length and the average diameter value was reported.

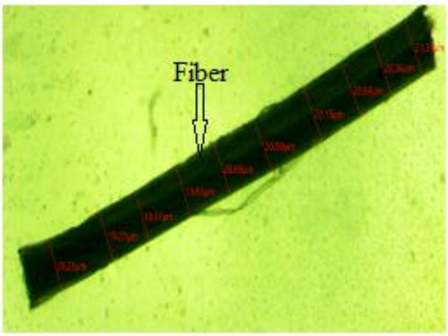


Figure 3. 6 Diameter of fiber measured using Optical microscopy

3.3.4.3. Fiber densities

The sample, a bundle of treated and untreated enset and sisal fibers, was weighed in air and then in a distilled water that was used to wet the sample and was smaller in density than the sample. The difference of the two media was the buoyancy force. The weighing process was conducted on an analytical balance with a resolution of 0.001g, and was adapted for suspension weighing through a stainless steel suspension wire approximately 0.4 mm in diameter. The buoyancy force was converted into sample volume by dividing the liquid density. The sample density ρ_f was acquired by dividing sample weight in air with sample volume as per ASTM-D3800–99, 2005 standard (Truong et al., 2009)**.** The density of fibers was calculated by using Equation (3.6).

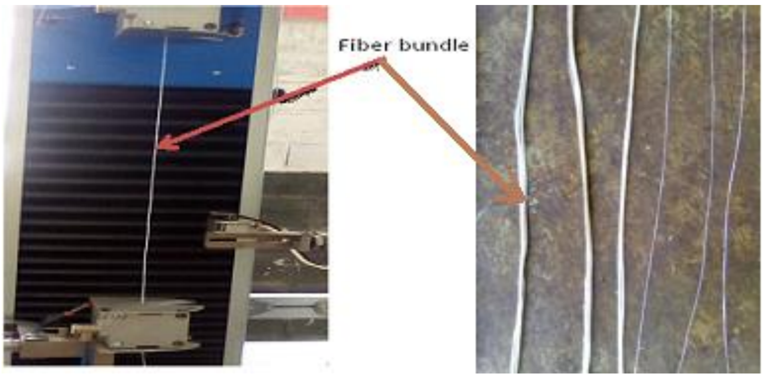
$$\rho_F = \left(\frac{m_3 - m_1}{(m_3 - m_1)\ (m_4 - m_2)}\right)\rho_w \tag{3.6}$$

where ρ_F is density of fiber in (g/cm^3), m_1=weight of suspension wire in air m_2 =weight of suspension wire in liquid m_3 = weight suspension wire plus sample weight in air and m_4 weigth of suspension wire plus sample weight in liquid in (gram) and ρ_w standard density of distilled water i.e 0.998 (g/cm^3). All densities were measured at 23 ± 0.2^{0}C using Metller Toledo density measurement kit and a known standard density block (Amiri et al., 2017).

3.3.5. *Mechanical testing of fibers*

The strength tester, which was used mainly in research environments is the Mantis individual fiber tensile tester (Patelke, 1992). This tester is capable of testing single fiber strength using a pair of clamps that grip the fiber without damaging the fibers. An electro optical system is used for measuring the projected ribbon width of each fiber prior to breaking. The test was done at textile industry development institute in Addis Ababa, Ethiopia, according to ASTM-D4605 standard. The tensile strength of untreated, 5% and 10% NaOH treated sisal and enset fibers was investigated using tensile tester. Ten specimens of untreated and treated fibers were tested with 100 N load cell having gauge length of 200 mm at room temperature.

The fibers were conditioned for 24 hrs at room temperature. The samples were randomly selected from the batch and its count was determined by indirect methods using measuring the length by the fiber ruler and mass by digital balance having 0.001g accuracy. The test was determined under ambient temperature. Ten samples each of untreated, 5% and 10% NaOH treated enset and the same for sisal fibers were tested and the average values were noted. Before conducting the tensile test, the fiber diameter was measured and checked for uniformity by using OM. To avoid the diameter variation effects, the specimen was compared by indirect counting using the ratio of breaking force to length per unit mass. Length per unit mass is called "tex". The tensile strength was tested using single or bundle fiber strength tester as per the ISO 5079 standard. The fibers were clamped at each end and a force was applied as showed on Figure 3.7 (Tarabi et al., 2016).



(a) (b)

Figure 3. 7 Fiber specimen for the tensile test using (a) Autodny-II Machine (b) Sample of single fiber and yarn (bundle) tensile test

3.3.6. *Fabrication methods of composites*

Hand layup is a simple technique which has been employed to make thermoset composites Because of its simplicity; hand layup has been widely used for making hybrid composites as per the literature showed under Section 2.4.7. The mold size used to prepare the composite prepared from wood having 300 mm × 300 mm × 5 mm dimension (Jayaraman, 2015). Hand layup method mostly recommended for the fabrication of hybrid composite. Unidirectional and woven oriented sample with having untreated, 5% and 10% NaOH treated and volume ratio of 100/0, 75/25, 50/50, 25/75 and 0/100 of enset/sisal composite sample were prepared. The steps of composite preparation were tabulated in Figure 3.8

shows the process flow of composite fabrication fiber hybrid composites were discussed (Jawaid & Khalil, 2015).

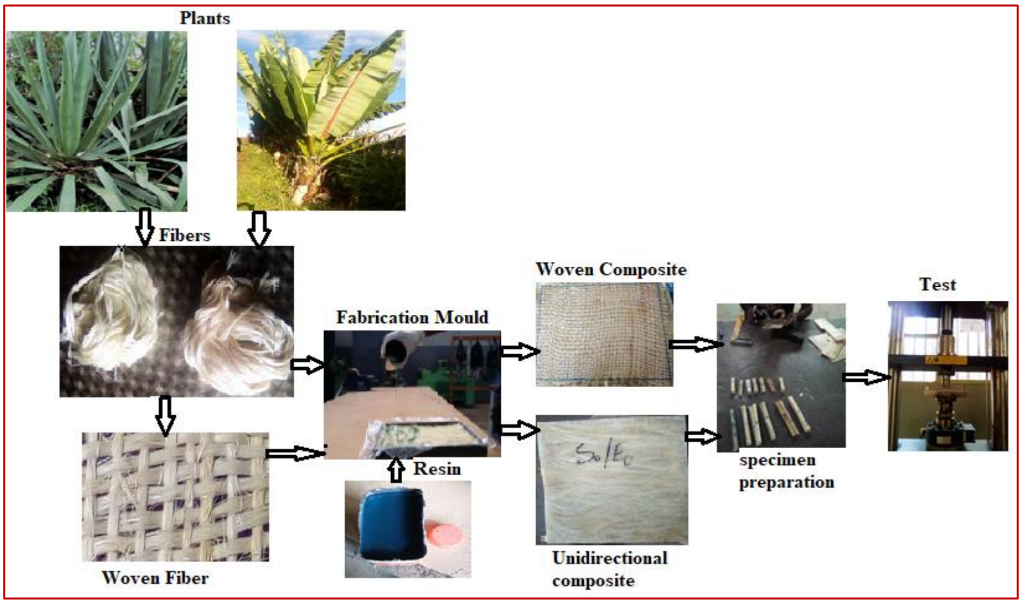
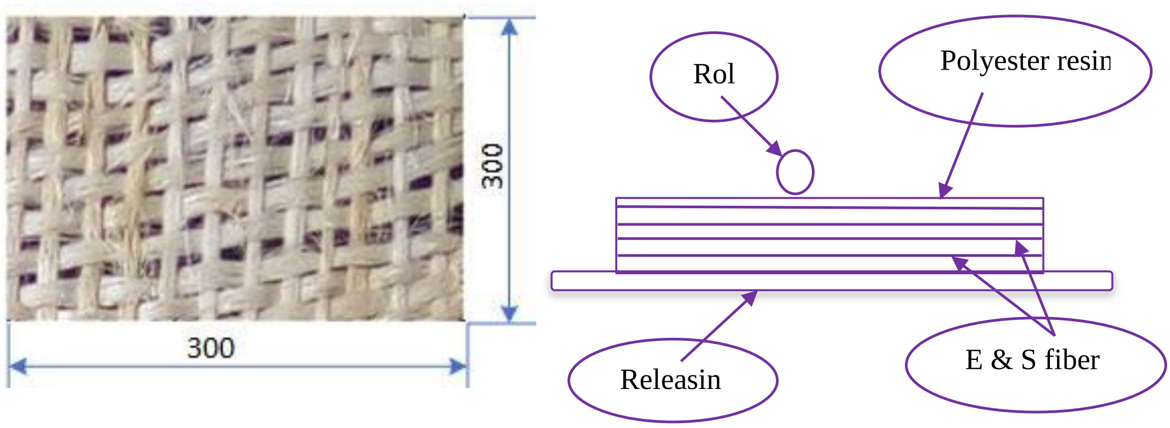


Figure 3. 8 Composite preparation procedure

The surfaces of the mold and the walls are coated with wax to prevent sticking of fibers and allowed to dry. The resin was prepared in 10:1 with hardener. After orienting the measured enset and sisal fibers in the orientation of ($0^E/0^S/0^E/0^S/0^E/0^S/0^E$) for continuous unidirectional composite. Both enset and sisal woven mat is prepared separately. The woven fabric fiber and mold sketch map were shown on Figure 3.9 (a) and (b) respectively and a roller used to distribute the resin. The functions of top and bottom plates are to cover, compress the fiber after the polyester is applied, and also to avoid the debris from entering into the composite parts during the curing time. To strength the composites, the sample was cured for 24-48 hr. After curing process, test specimen was prepared as recommended ASTM standards show on Table 2.9.

3.3.6.1. Volume fraction of materials

The volume fraction of reinforcements and matrix material used for the development of hybrid composites were considered according to the hybridization concept. It is defined as the fabrication of composites with two or more different fibers under the same polyester resin matrix volume (70% with the addition of hardener) with 30% total fiber loading. In this research, a total of five different composites were fabricated in two different fiber orientation and the weight proportions of the Enset/Sisal hybrid samples are 100/0, 75/25, 50/50, 25/75, and 0/100 volume ratio.



(a) (b)

Figure 3. 9 (a) Individual layer of woven fibers and (b) molding sketch map

3.3.6.2. Mass and volume fraction of hybrid composite

The Density of untreated, 5% NaOH and 10%NaOH treated enset and sisal fiber were calculated by using equation 3.5 and summarized in Table 3.1. The composition, volume and mass of each composite were calculated using the rule of composite mixture and summarized in Table 3.2.

In all types of composites, constant volume and mass of polyester were used. To calculate the volume of composite (V_c), the volume of die was used, where V_c = Volume of die.

Table 3. 1 Data of enset and sisal fibers and polyester density.

S.No	Density(g/cm)[3]		
	Untreated	5%NaOH	10% NaOH
Enset	1.66	2.02	2.23
Sisal	1.52	2.31	2.51
Polyester		1.20	

Table 3. 2 Deign specimen composition, volume and mass for the study

Sample	Composition (%)			Volume (m^3)		Mass (kg)	
	Polyester	Enset	sisal	Enset	sisal	Enset	sisal
Untreated							
E_{100}/S_0	70	30	0	1.35×10^{-4}	0	0.224	0
E_{75}/S_{25}	70	22.5	7.5	1.01×10^{-4}	0.03×10^{-4}	0.051	0.167
E_{50}/S_{50}	70	15	15	0.67×10^{-4}	0.67×10^{-4}	0.112	0.1026
E_{25}/S_{75}	70	7.5	22.5	0.03×10^{-4}	1.01×10^{-4}	0.167	0.0515
E_0/S_{100}	70	0	30	0	1.35×10^{-4}	0	0.205
5% NaOH treated							
E_{100}/S_0	70	30	0	3.39×10^{-4}	0	0.331	0
E_{75}/S_{25}	70	22.5	7.5	1.01×10^{-4}	0.03×10^{-4}	0.23	0.068

E_{50}/S_{50}	70	15	15	0.67×10^{-4}	0.67×10^{-4}	0.155	0.136
E_{25}/S_{75}	70	7.5	22.5	0.03×10^{-4}	1.01×10^{-4}	0.068	0.234
E_{0}/S_{100}	70	0	30	0	3.39×10^{-4}	0	0.273
10% NaOH treated							
E_{100}/S_{0}	70	30	0	3.39×10^{-4}	0	0.338	0
E_{75}/S_{25}	70	22.5	7.5	1.01×10^{-4}	0.03×10^{-4}	0.254	0.075
E_{50}/S_{50}	70	15	15	0.67×10^{-4}	0.67×10^{-4}	0.169	0.151
E_{25}/S_{75}	70	7.5	22.5	0.03×10^{-4}	1.01×10^{-4}	0.753	0.254
E_{0}/S_{100}	70	0	30	0	3.39×10^{-4}	0	0.301

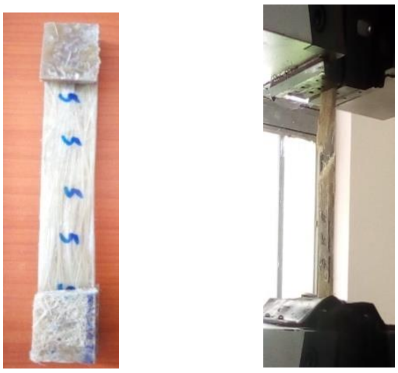
3.3.7. Composite mechanical testing

The test specimens were prepared and undergone different mechanical tastings namely tensile test, flexural, and impact test and moisture absorption as per the ASTM standards. The specimen test standard for hybrid natural fiber composite was sumarized in chapter 2 (Table 2.9). Hybridizing sisal fiber with other natural and synthetic fibers significantly enhances their physical and mechanical behaviors by taking the advantage of their individual constituents. The mechanical behaviors (tensile, flexural and impact strength) of untreated and NaOH treated sisal-jute fiber hybrid epoxy composite was studied and the result showed that treated fiber composite improved in all case of mechanical properties (Perremans et al., 2018).The effects of alkali treated fiber was tabulated under Chapter 2 in Table 2.6.

The test specimens were prepared and experienced different mechanical tests namely tensile test, flexural test, and impact test as per the ASTM standards tabulated in Table 2.9 under Section 2.4.9. The specimen size and shape for corresponding tests are described in the following sections. Five specimens were tested for each set of samples and the average value was taken for analysis.

3.3.7.1. Tensile strength

Tensile testing is used to measure the force required to break the test specimen and the extent to which the specimen stretches or elongates to that breaking point. The better mechanical properties of the composite can be obtained when the fiber orientation is aligned in the load direction. The specimen dimension used for the tensile tests is 250 mm × 25 mm × 5 mm (length × width × thickness) as per ASTM D3039 standards. The test was carried out on (bairoe, shanghai, China universal test machine) having 50 kN capacity with the test speed of 5 mm/min crosshead speed and 150 mm gauge length. A sample specimen for the tensile test is given in Figure 3.10.



(a) *(b)*

Figure 3. 10 (a) Specimen of tensile test before test (b) Specimen after test on UTM

3.3.7.2. Flexural strength

Flexural strength is carried out to find the ability of the material to resist the deformation under the load. The cross-section of the test specimens is rectangular, as they are directly molded or cut from molded parts. This is a three-point bend test, which is used to promote failure by inter-laminar shear. This test was conducted as per ASTM D790 standard on UTM (WP 310 universal material tester, gunt, German) (Sivakandhan et al., 2019) with a test speed maintained between 0.5 and 1 mm/min was used. The specimen dimension is 127×13×5 (mm). Figure 3.11 (a-c) indicates, the flexural specimen before, during and after test.

(a) (b) (c)

Figure 3. 11 (a) flexural specimen before test (b) during test (c) specimen after test

3.3.7.3. Impact strength

The impact test is a single-point test measuring the resistance of a material against impact load from swinging pendulum on the specimen until fracture. The specimen dimension for this test is 65 × 13 × 5 (mm) and the impact strength was determined using charpy impact tester (Ceast Torino, Italy) as per ASTM D256 standard (Shanmugam & Thiruchitrambalam, 2013b) was used. The specimen of impact was prepared as shown in Figure 3.12.

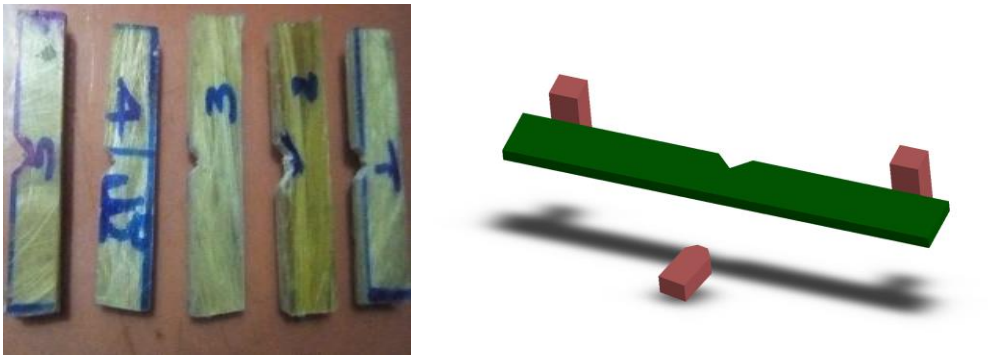


Figure 3. 12 Specimens before the impact test

3.3.7.4. Water absorption

Water absorption properties of enset/sisal hybrid composites in water at room temperature were recommended. A water absorption test was carried out as per ASTM D570 standard (Venkateshwaran et al., 2011) To study the water uptake, the specimens were immersed in water at room temperature. The samples were taken out periodically and weighed immediately, followed by cleaning the surfaces of the samples with a dry cloth. Then, the samples were weighed using a precise 4-digit balance to find out the content of water absorbed (Alshammari et al., 2019). All the samples were dried in an oven until constant weight is reached before immersing again in the water. The percentage of water absorption was determined using the formula in equation (3.7).

$$WA(\%) = \frac{m_2 - m_1}{m_2} \qquad (3.7)$$

Where: WA = Water absorption, m_1 and m_2 are the weight of dry and wet samples of composites respectively.

3.3.8. *Machinability of enset/sisal hybrid composite*

Post processes such as machining or other finishing operation is still requires in order to meet assembly and dimensional of the composite product. The most machining operation used to facilitate assembly operation is drill or making hole. To determine the delamination caused by drilling operation both on the upper surface (peel-up delamination) and lower surface (push-down delamination), the following equation is applied as showed on Equation 3.8. *(Vinayagamoorthy, 2018)* (Nassar et al., 2016).

$$\text{Delamination factor } F_d = \frac{D_{max}}{D_{nom}} \quad (3.8)$$

D_{max} = maximum damage diameter D_{nom}=nominal or actual dia.

There are various techniques used for measuring the diameter or radius used in the equations for assessment of delamination as shown in Figure 3.13. (Nassar et al., 2016), (Lotfi et al., 2019). Among these techniques, profile projector, microscopy, and image processing using a scanner are the most easily accessible and cost effect techniques for measuring the diameter and radius.

The HSS drill bit used to drill the woven and unidirectional E/S hybrid composite on CNC drill machine (XH7145, Shandong Schuler CNC Machinery CO.,Ltd.,Tengzhou, China).

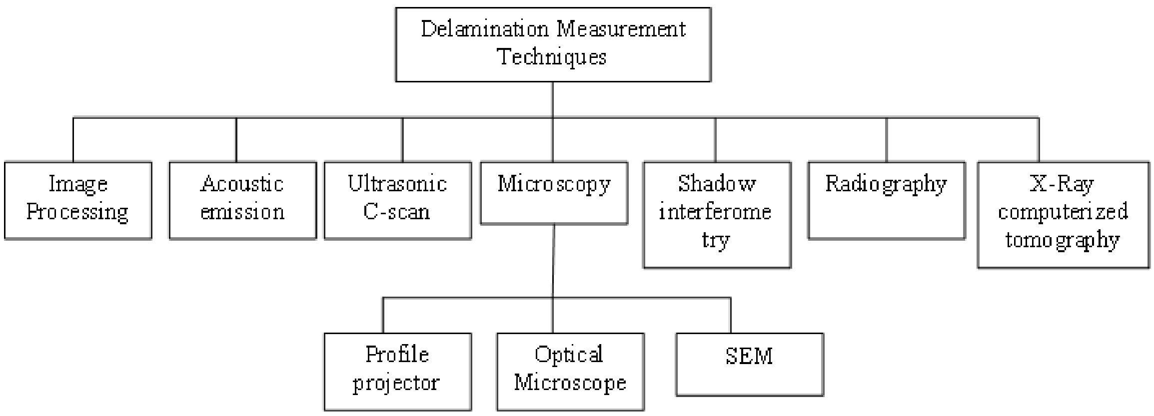


Figure 3. 13 Techniques for measuring various delamination parameters

The composite specimen was held in a rigid fixture attached to the machine table and support at the back of specimen by flat wood to avoid the damage during drilling operation. Figure 3.14 below indicates that composite samples unidirection (top), woven (bottom), HSS drill bit, CNC machine and drilled hole composite. The machining condition for drilling operation in this work is dry condition, due to having low abrasive, less wear and liquid absorption properties (hydrophilic) nature of natural fiber (Oluwarotimi et al., 2016). Less expensive alternatives would be a better choice for NFRCs.

Measuring the damage (delamination) of the NFRCs can be carried out either directly or indirectly. Direct measurements can be implemented using parameters such as delamination factor, damage width, surface roughness, and chip type produced. Indirect measurements involve assessment of the damage due to the thrust force, torque, or power

Unidirectional

generated during the machining operation. A number of approaches have been used for making holes in composites, conventional radial drilling and CNC machining are widely used drilling methods till date (Bajpai et al., 2015), (Packiara and Kumar, 2009).

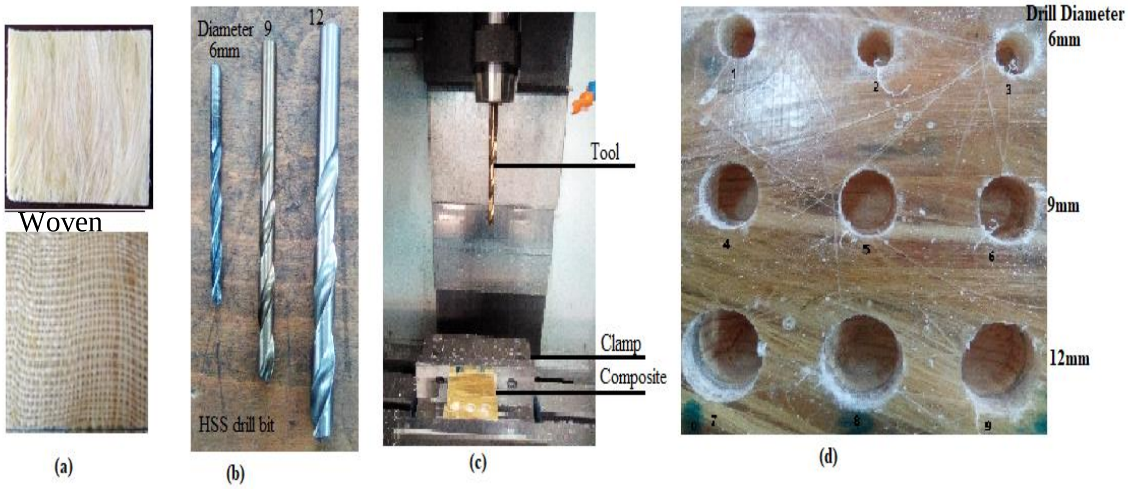


Figure 3. 14. (a) Unidirectional (top) and woven (bottom) E/S hybrid composite (b) high speed steel (HSS) drill bit (c) CNC machine (d) drilled composite part

3.3.8.1. Drilling parameters

The three parameters, speed (A), feed (B) and drill bit diameter (C) and three levels in each category was considered in this work. The L_9 orthogonal array was developed for analyzing 3 variables and 3 levels in each variable (Jayabal & Natarajan, 2011). The parameters and levels for the study selected based on the existing literature (Babu et al., 2013). The analysis will be done by using MINITAB18 software (Ismail et al., 2016).

The percentage of error was calculated using the Equation 3.9.

$$\% \text{ of error} = \frac{\text{Experimental value} - \text{Predicted value}}{\text{Experimental value}} * 100 \quad (3.9)$$

3.3.8.2. Design of experiment

The design tools play a vital role for determining the machinability parameters. There are several tools have been utilized by the researchers for the optimization of machining parameters. Full factorial, fractional factorial and OrthogTaguchi design are the most experimental design used in the manufacturing companies for improving the product quality and process efficiency. Choice of experimental design depends on number of factors, resource available, time needed and cost required for experiment. Full factorial design uses full level and factors (2^k) and needs more time, resource and budget. But fractional and Taguchi experimental design are robust methods that drastically reduce the required number of tests to find the optimum results that help to save cost, resource and time. Taguchi method is used to determine the desired optimum cutting parameter and drill bit diameter for minimized appearance of delamination and surface roughness in drilled enset/sisal hybrid woven and unidirectional orientation natural fiber-reinforced composites. The experimental results are transformed into a *Signal-to- noise ratios(S/N)* that uses as a quality characteristic deviation from or nearing to the desired value. There are three main categories of quality characteristic in the analysis of S/N ratio. That is higher the better, nominal the better and lower the better (Packiara and Kumar, 2009). The smaller the better-quality characteristic Equation 3.10 is used for surface roughness and delamination. The formula for nominal and higher quality characteristic are written in Equation (3.11 and 3.12).

- For smaller the better of quality characteristic

$$N/_S \text{ ratio } (\eta) = -10\log_{10} \frac{1}{n} \sum_{i=1}^{n} y_i^2 \quad (3.10)$$

Where y_i^2 observed response value and η number of replications.

- For nominal the better of quality characteristic

$$N/_S \text{ ratio } (\eta) = -10\log_{10} \frac{\mu^2}{\sigma^2} \quad (3.11)$$

Where = μ^2 mean and σ^2 = variance.

- For higher the better of quality characteristic

$$N/_S \text{ ratio } (\eta) = -10\log_{10} \frac{1}{n} \sum_{i=1}^{n} \frac{1}{y_i^2}$$

Most of the time, a key result of ANOVA is determined by p-value (denoted by alpha (α)) or by comparing the F-value and F_{crit}. value. While most people use 0.05 as a traditional cutoff for p-value, i.e. α-value, a significance level of 0.05 indicates a 5% risk of concluding that there is no actual difference. Also the factor is statistically significant if F greater than F_{crit}. If the p value is less than or equal to the difference between sum, then the means are statistically significant, and the null hypothesis can be rejected and concluded that not all population means are equal. If the p value is greater than the difference between sum, then the mean are not statistically significant. You do not have enough evidence to reject the null hypothesis that the population means are equal. If the p-value higher than α. We should have to improve the power hypothesis test. (a) By using higher significance level- using of a higher significance level increase the probability that we reject the null hypothesis (while rejecting a null hypothesis that is true (0.05) is called type I error). (b) By using more replication – using of more replication is the most practical way to increase power of hypothesis test.

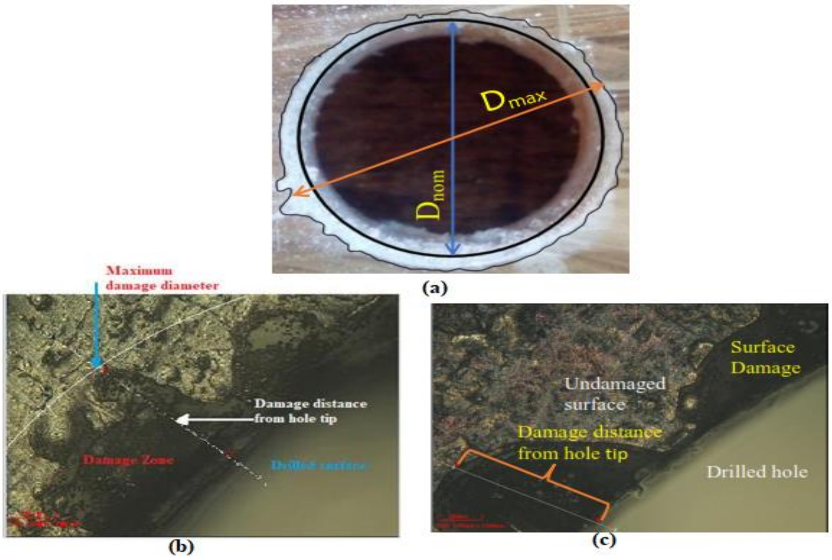


Figure 3.15 Delamination of (a) drill hole (b) unidirectional and (c) woven orientation E/S hybrid composite by 3D Optical surface profile.

3.3.8.3. Delamination and surface roughness methods

After drilling operation, the surface was analyzed using 3D optical surface profiler (ZETA) for measuring the drill hole damage and surface roughness of sectioned hole. The surface roughness measure the finish of the drilled hole with the factors of cutting parameter, drill bit diameter and fiber orientation. The delamination defect around the drilled holes, known as delamination damage zone Figure.15 (a) of the samples is a microscopic phenomenon of unidirectional and woven types which tabulated on Figure 3.15 (b) and (c). Therefore, it is observed and measured using a ZETA INSTRUMENT optical microscope operated on a 5X magnification.

3.3.9. *Tribology of enset/sisal hybrid composites*

A pin-on-disc (POD) rotating type friction and wear tribometer (TR-20-MICRO) was used to evaluate the friction and wear characteristics of the developed composites as showed on Figure. 3.16 (a). Sliding occurs between a stationary pin clammed in the specimen holder and the rotating disc. Normal load, sliding speed and wear track diameter can be varied to suit the test requirements(Kumar et al., 2013).The coefficient of friction was calculated using recorded of frictional force and by using equation (3.13).

$$\text{Coeffeicient of friction } (\mu) = \frac{\text{Frictionl force } (F_f)}{\text{Applied Load (P)}} \qquad (3.13)$$

Were F_f is the frictional force (μ), and P is normal load (N). First measuring the initial weight of test specimen and measured after wear test is done. The difference in the weight measured before and after the test gives the weight loss of the composite specimen. Normal load, sliding speed and wear track diameter can be varied to suit the test requirements.

Normal load, sliding speed and wear track diameter can be varied to suit the test requirements. The weight of sample before and after each experiment was measured using digital electronic balance having 0.0001g precision. The worn-out material was measured by using the equation. (3.14) (Patel & Sahu, 2020).

$$\text{Weigth loss in sample } (\Delta w) = (W_b - W_a) \tag{3.14}$$

where: ΔW=Weigth loss, W_b= Weight sample before wear W_a=weight sample after wear

The wear rate was calculated by taking the weight difference before and after test of sample. Many researchers calculate the wear rate by using weight loss methods (El-Tayeb, 2008), (Goriparthi et al., 2012). Unwearied sample and wearied sample are shown in Figure (a) and (b) respectively. The counter face is a ground hardened steel disc having of maximum wear diameter of 60 mm and thickness 8 mm as specimen prepared as per ASTM standard for pin-on-disc test methods. The two different fiber orientation samples of composite wear characterized by varying normal load and sliding speed to suite the test requirements. All testing was performed at a laboratory ambient temperature of about 22^{o}C and under dry conditions.

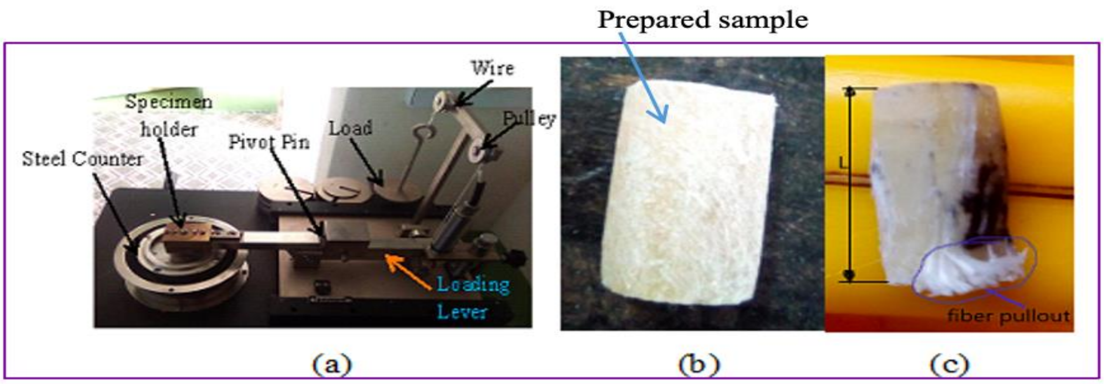


Figure 3. 16 (a) pin on disc instrument (b) sample before test (c) sample after wear

3.3.10. *Fourier transformation infrared spectroscopy machine*

The Fourier transform infrared spectroscopy (FTIR) analysis used to detect characteristic functional groups in the fibers. The infrared light was passed through the fibers sample. Alkali was used to remove the hemicellulose, lignin, and other impurities from the fiber surface and change of fiber chemical composition was analyzed using FTIR (Suryanto et al., 2013). Each spectrum was recorded by co-adding 32 scans at 8 cm^{-1}resolution within the range of 4000–600 cm^{-1} range (Hossain et al., 2014)**.** The FT-IR spectrum was made for untreated, 5% and 10% alkali treated of sisal and enset fibers. Table 3.3 below shows that FTIR peak positions and corresponding chemical stretching mode vibrations on the natural fiber (Alawar et al., 2009; Arthanarieswaran et al., 2015; Fortunati et al., 2010;

Maepa et al., 2015). The allocations of the wave length that used to determine the chemical compositions of the fibers shown on the Table (3.3).

3.3.11. *Composites morphology*

SEM is a type of electron microscope that produces images of a sample by scanning it with a focused beam of electrons. To conduct the morphological characterization of the composite fracture surface, the scanning electron microscope of (Model JEOL, Kyoto, Japan) was used. The composite samples were adequately cleaned; air-dried and are coated with 100A^o thick platinum in JEOL sputter ion coater and observed in SEM at 10 kV. The fracture surface morphology of the composite specimens was observed. The drilled hole damage and surface roughness were used. The damaged area of around drill hole was also observed in detailed using 3D optical surface profiler.

Table 3. 3 The FTIR wavelength and their allocations

Wavelength (cm^{-1})	Allocations
3700-3500	O-H stretching of α cellulose
3500-3300	N-H stretching (amine)
3500	N-H stretching (amide)
3100-3000	$C = C - H$ stretching
2960-2850	$C - H$ stretching
2590-2540	$S - H$ stretching
1740-1720	$C = O$ stretching of hemicellulose
1685-1655 &1600	$\alpha, \beta -$ unsaturated stretching
1685-1650	$N = O$ stretching
1650-1630	OH (Absorbed water)
1600	$C = O$ stretching (Amide)
1600,1585,1500 &1450	$C = C$ stretching
1450-1400	CH_2 symmetric bending
1567,1380	NO_2 stretching
Above 1500	$C = O, CH_2, NH, C = C, C = N$ functional group stretching
Below 1500	Single bonds and bending vibrations
1395-1385	t-butyl stretching
1365	CH bending (deformation)
1350-1260	C-O stretching
1150-1070	C-O-C stretching
1050-1020	Symmetric C-OH stretching of ligning
815-750	N-O Stretching
800-600	C-S stretching
770-730	C-C deformation
520-420	S-S stretching

CHAPTER-FOUR

4. Discussion of Results

4.1. Introduction

In this chapter, the results obtained for each specific objective of the research work are discussed. Parts of these results are also published in Scopus indexed international journals (Paper I – Paper V, as listed on page xiii). Copies of the articles are appended at the end of this dissertation.

4.2. Experimental study of physical, chemical and mechanical properties of Enset and Sisal fibers

The physical, chemical and mechanical properties of natural fibers are essential and the main criteria for designing and developing industrial products. Chemical composition, mechanical properties and physical properties are differs from plant to plant, and within different parts of the same plant, from different geographic locations, ages, climate, and soil conditions. To develop better property products, studying the effects of alkali treatment on physical, chemical and mechanical properties of natural fibers is very important. Natural fibers are rich of cellulose, hemicelluloses, lignin, and pectins, all of which are hydroxyl groups, which are usually hydrophilic sources and strong polar whilst polymers show considerable hydrophobicity. It is also responsible for biodegradation, moisture absorption, and thermal degradation of the fibers. Fiber alkali treatment reduces hemicellulose, lignin, and wax content and increasing surface roughness for better interlocking with the matrix materials to enhance the properties of the composites. The properties of fibers mainly depend on their chemical composition and their structure, which are related to the type of fibers used, method of extraction, harvesting period, growing conditions and chemical treatment. Sisal fiber extracted from leave parts of sisal plants can be used for making the variety of products.

This subtopic addresses the first specific objective of the research and part of the work has been published in Paper I (Bekele et al., 2022a) and covers the following results:

- Influence of NaOH concentrations on the physical, mechanical and chemical composition of enset and sisal fibers.
- The effects of treatment on physical properties (moisture, diameter and density) of enset and sisal fibers.

- The effects of treatment on mechanical properties (tensile strength, tensile module and elongation) of enset and sisal fibers.
- The effects of treatment on chemical composition (wax, cellulose, hemicellulose, lining and ash contents) of enset and sisal fibers, and
- FTIR analysis of (untreated and treated (5% and 10% NaOH)) treated of enset and sisal fibers.

4.2.1. Physical properties of fibers

4.2.1.1. Moisture content

The moisture content value of untreated and NaOH treated (5% and 10%) enset sisal fibers were depicted in Figure. 4.1. The moisture of both treated and untreated enset and sisal fibers were measured according to methods described in Section 3.3.4.1. As the result in plots shows, NaOH treatment reduced the moisture content of enset and sisal fiber. It has been noticed that there is higher moisture absorption of untreated enset and sisal fibers than that of treated fibers. The reason for this moisture absorption property is the presence of hydroxyl groups. The hydrophilic nature of fiber is also one of the main reasons for poor adhesion. In the structure of fibers, the hydrogen bond is broken down due to alkali treatment and improves the fiber moisture absorption properties by reducing the hydrophilic hydroxyl groups of the fibers for better interlocking with the matrix. Previous studies (Shehu et al., 2017), (Shanmugam & Thiruchitrambalam, 2013a) also show that fiber moisture absorption properties improve by alkali treatment by reducing the hydrophilic hydroxyl groups of the fibers.

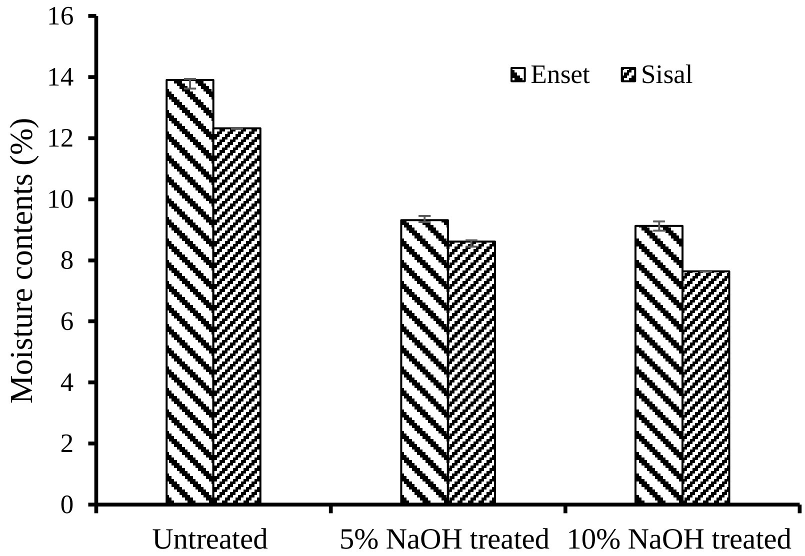


Figure 4. 1 Moisture absorption properties of untreated and treated fibers

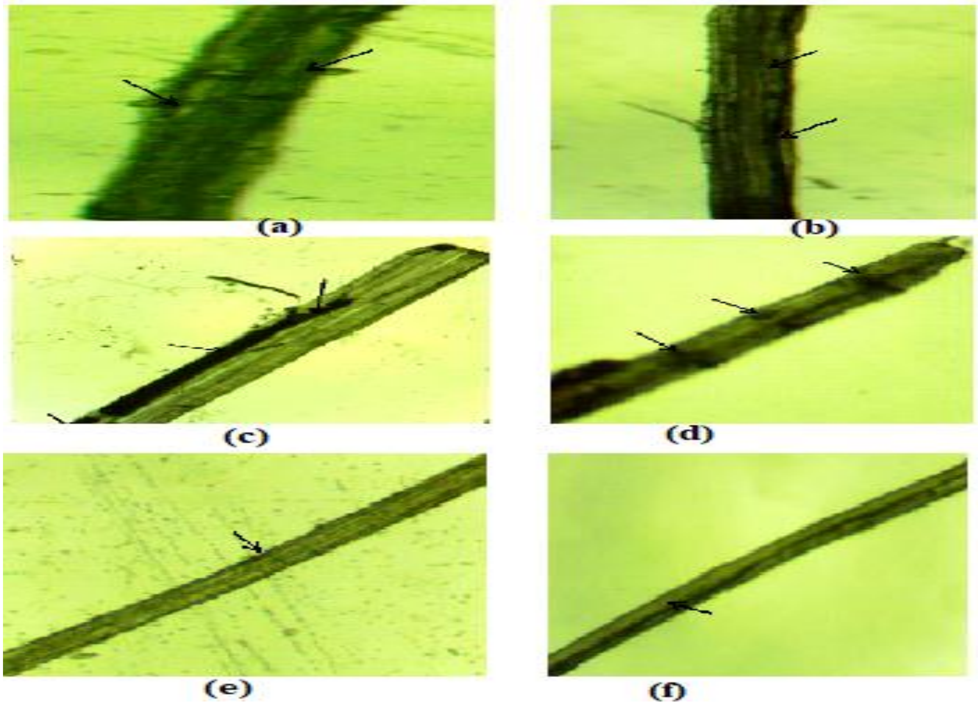


Figure 4. 2 Optical Microscopy image of defects in untreated (a) enset (b) sisal, 5% NaOH treated (c) enset and (d) sisal fiber and 10% NaOH treated enset and (f) sisal fibers.

Figure 4.2. (a-f) shows the images of all types of fibers considered in this study i.e., untreated, 5% and 10% NaOH treated enset and sisal fibers. The average diameters of the fibers were calculated using optical microscopy as shown in Figure 3.7, and the average value was taken as the fiber size.

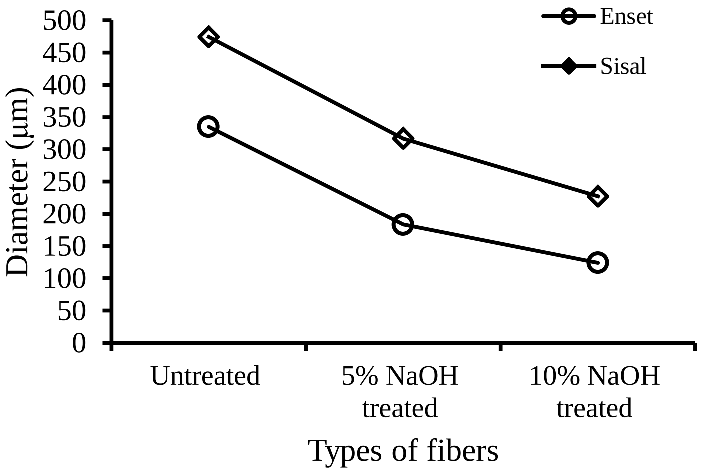


Figure 4. 3 The effects of treatment on fiber diameter

4.2.1.2. Fiber density and diameter

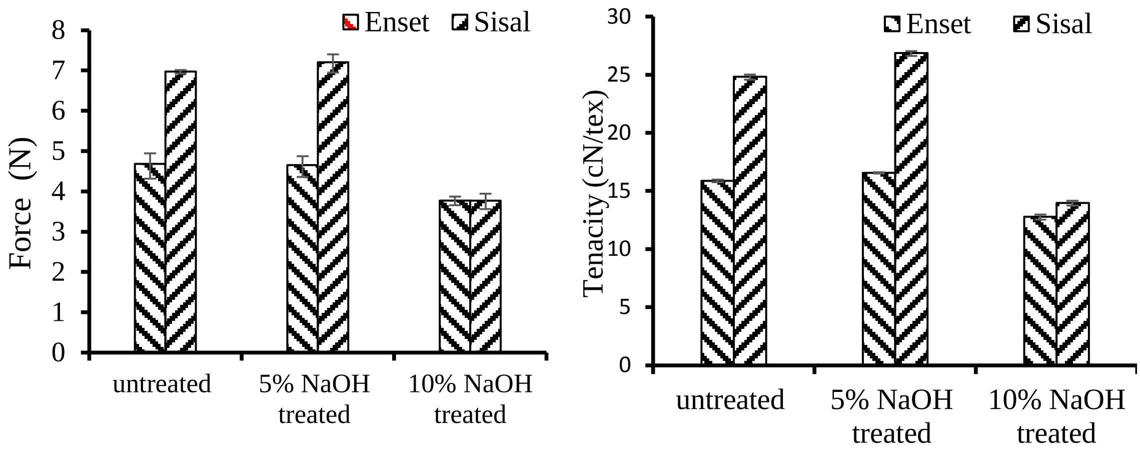
The density of the untreated and treated enset and sisal fiber were measured according to the method described in Section 3.3.4.3 and Equation 3.6. As observed from the optical microscopy image shown in Figure 4.2 (a- f) the dark image was observed on untreated enset and sisal fiber (Figure (a) and (b)). The dark regions on the fiber indicate the impurities left on the fiber during extraction process. While observing the image shown in Figure 4.2 (c) and (d), small amount of dark were seen and in Figure (e) and (f) almost dark region was removed and clean image was observed. The measured results given in Figure 4.3 show that the diameter reduced by 45.2% and 33.3% for 5% NaOH treated and 62.8% and 52.1% for 10% NaOH treated enset and sisal fibers than untreated fibers respectively. This is due to removal of impurities from the inner and outer fiber surfaces.

The density of both untreated fibers is found to be lower than the treated fibers. The densities increased due to the removal of the lower denser regions such as lignin and hemicellulose. Higher density indicates that the fibers have higher cellulose contents, while alkaline treatment increases the density of fibers by removing non cellulosic components (hemicellulose and lignin), which are less dense. The result similar with the result given on (Sanjay et al., 2018).The density of treated fiber is observed to be higher due to the removal of alkali soluble materials which caused cell wall densification of the fibers.

4.1.2. Mechanical properties of Enset and Sisal fibers

4.1.2.1. Tensile strength

After treating by 5% NaOH, the tensile strength of enset and sisal fibers increased by 51.5% and 31.67% respectively, as shown on Figure 4.4 (a). The methods to measure the tensile strength of bundle fiber was described in Figure 3.7. Tenacity measures the strength of a fiber expressed by maximum tensile force (breaking force) divided by the linear density of the fibers. The unit of tenacity is cN/tex. Tenacity of 5% NaOH treated individual fiber is higher than the untreated and the 10% NaOH treated. Higher concentration of NaOH damages the fibers and decreases tenacity value as the result in Figure 4.4 (b) shows. Similar observation is reported in (Khan et al., 2012) and (Sasser et al., 1991).



(a) (b)

Figure 4. 4. (a) Tensile force and (b) Tenacity of treated and untreated enset and sisal

4.1.2.2. Fiber elongations

As shown in Figure 4.5, bundle fiber of untreated enset and sisal fiber were experimentally characterized using Autodyn-II machine (Figure 3.7). Five repetitions were done to take the average value of each sample, whose average value of elongation for all types of fiber is given in Figure 4.6.

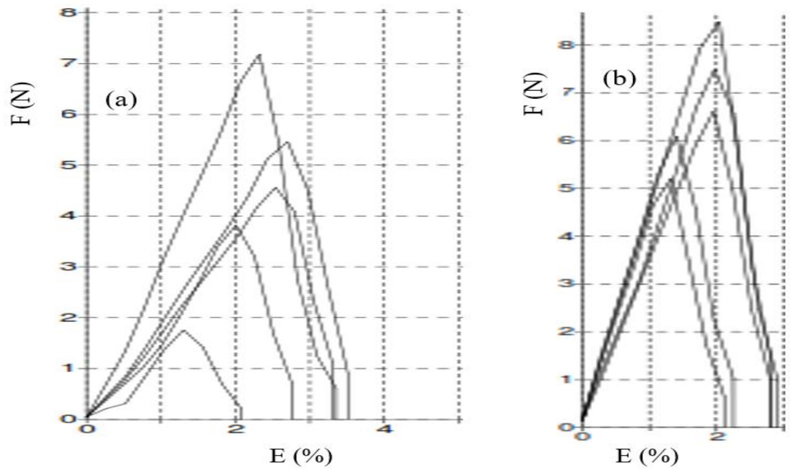


Figure 4. 5 Maximum force vs. elongation diagram of untreated (a) enset and (b) sisal fibers.

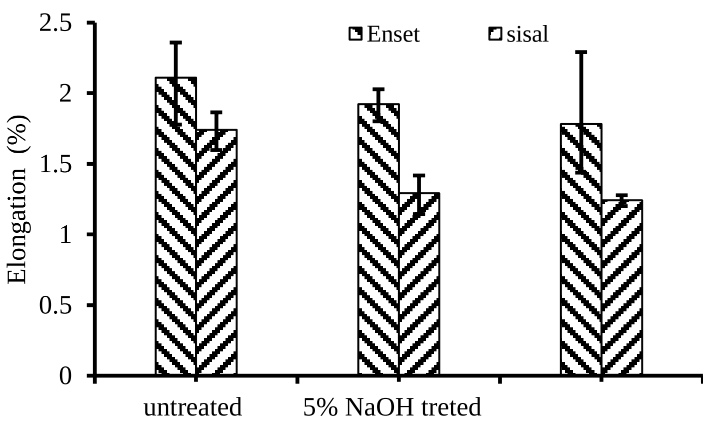


Figure 4. 6 Average value of elongation for untreated, 5% and 10% NaOH treated enset and sisal fibers

4.1.3. Chemical properties of fibers

Many researchers have used alkali concentration, immersing time and drying time as main parameters to determine the chemical properties of fibers. The used concentrations range from 1 to 15% , immersing time ½ hr to 3 hrs and dry time from 1 day to 5 days (Cai et al., 2016) (Dawit et al., 2019)). If the alkali concentration is less, the impurities are not sufficiently removed, while higher alkali concentration above 10% can lead to damage of fibers (Kabir et al., 2012b). The effects of immersion (soaking) time were also studied for coir fiber. The result showed that 2 hrs immersion time leads to better tensile strength than immersing for less hours (1 hr) or longer hours (3 - 5 hrs) (Musanif & Thomas, 2015). The methods are described under Section 3.3.3 and Equation 3.1 - 3.4 for the determination of lignin, cellulose, hemicellulose and ash content respectively. The effects of alkali on diameter of sugarcane fiber was studied and the results confirm that alkali treatment fiber has less diameter than untreated fibers. This shows that alkali treatment removes the outer (lignin) and the inner (hemicellulose) contents from the fibers. Commonly, natural fibers have hydrophilic properties that are vulnerable to moisture. The cellulose, lignin, hemicellulose and wax are the most common fiber chemical compositions that affect the physical, chemical and mechanical properties of the fibers. Table 4.1 shows the results of the chemical compositions for untreated and 5% and 10% NaOH treated enset and sisal fibers. The change in fiber chemical properties was observed due to NaOH treatment. The NaOH treatment caused the cellulosic fiber to swell and it removed the hemicellulose, lignin and other impurities from the fiber surface.

Table 4. 1 Chemical properties of untreated and NaOH treated enste and sisal fibers.

Sample	Wax	Cellulose	Hemicellulose	Lignin	Ash contents
E_0	5.13 ± 0.09	80.88 ± 0.09	10.90 ± 0.09	10.43 ± 0.05	2.67 ± 0.1
$E_{5\%}$	1.80 ± 0.69	82.97 ± 0.28	6.33 ± 0.08	3.00 ± 0.01	3.17 ± 0.05
$E_{10\%}$	0.09 ± 0.11	85.19 ± 0.46	3.01 ± 0.46	3.33 ± 0.13	5.17 ± 14
S_o	1.10 ± 0.77	73.53± 0.36	10.02 ± 0.46	8.00 ± 0.23	1.50 ± 0.13
$S_{5\%}$	1.37 ± 0.73	85.58± 0.24	5.00 ± 0.34	5.20 ± 0.12	2.00 ± 0.15
$S_{10\%}$	2.47 ± 0.48	90.66 ± 0.18	2.67 ± 0.18	3.67 ± 0.25	3.33 ± 0.21

Higher cellulose content ensures good mechanical properties, while high levels of hemicellulose decreases the fiber strength by causing disintegration of cellulosic microfibrils. Fiber treatment by NaOH removes most of the lignin content, which affects the fiber structure, properties and morphology. The lignin contents of 5% NaOH treated fiber reduced by 71.1% and 54.1% than untreated enset and sisal fibers respectively. Natural fibers in nature have more amount of cellulose than hemicellulose, lignin and others. NaOH treatment increases cellulose contents and ash contents as the values in Table 4.1 demonstrate.

4.1.4. Fourier Transform Infrared Spectrometry (FTIR)

The fibers are mixtures of cellulose, hemicellulose, lignin, wax and other ingredient, where the NaOH treatment was used to remove the hemicellulose, lignin, and other impurities from the fiber surface. The effects NaOH treatment on fiber chemical compositions was analyzed using FTIR. The infrared whole pattern of the untreated, 5 % and 10 % NaOH treated sisal fibers are shown in Figure 4.7 and enset fibers are shown in Figure 4.8

All spectra were recorded in the range from 4000 cm^{-1} to 400 cm^{-1}. The broadband at around 3000 – 4000 cm^{-1} was referred to alcohol compound or hydrogen-bonded OH stretching from cellulose, hemicellulose, and lignin. The broadband at 3400 – 2850 cm^{-1} was attributed to C–H bond frequently observed in alkane groups. The intense peaks for C=O groups in the ketone and carbonyl groups were shown as peaks at 1740 – 1720 cm^{-1} and 1450-1650 cm^{-1} referred to the hemicellulose and lignin compound (Subramanian et al., 2019).

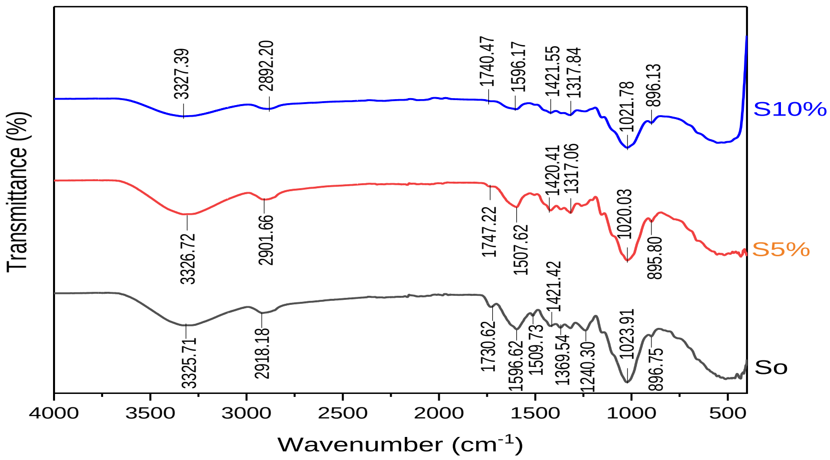


Figure 4. 7 FTIR Spectrum of sisal fiber

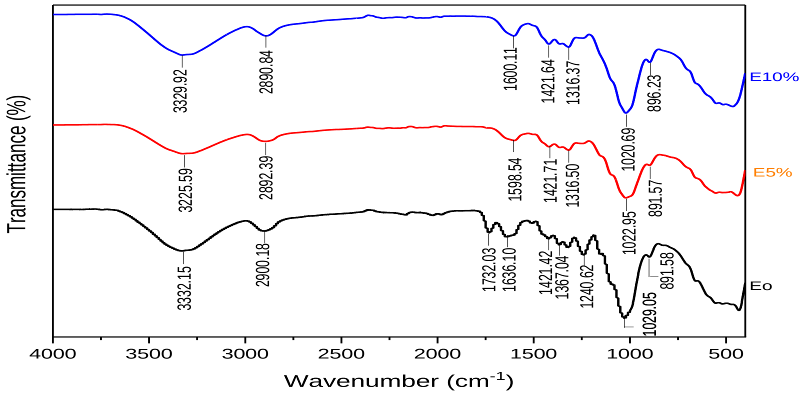


Figure 4.8 FTIR Spectrum of ensaet fiber

The spectrum display O-H absorption for enset is around 3332.15 cm^{-1}, 3325.59 cm^{-1} and 3330.35 cm^{-1} for untreated and 5% and 10% NaOH treated fibers respectively, while the corresponding values for sisal are 325.71 cm^{-1}, 3326.72 cm^{-1} and 3329.75 cm^{-1}. Similarly, the C-H stretching of untreated, 5% and 10% NaOH treated enset are around 2900 cm^{-1}, 2892.32 cm^{-1} and 2899.15 cm^{-1}; while the corresponding values for sisal fibers are around 2918.18 cm^{-1}, 2901.66 cm^{-1} and 2891.90 cm^{-1} are respectively α-cellulose (Maheshwaran et al., 2017).

The peaks at 1732.32 cm^{-1} and 1730.02 cm^{-1} were attributed to C=O stretching vibration of acetyl groups in hemicellulose compounds for both untreated sisal and enset fibers as shown in Figure 4.7 and Figure 4.8. The intensity of those peaks was not seen (removed) in the case of 5% and 10% NaOH treatment of sisal and enset fibers. These shows that NaOH treatment reduces or removes the hemicellulose compounds from the fibers.

The peak at 1450 -1650 cm^{-1} refers to aromatic structure of lignin present in the fiber. The peak 1596.62 and 1636.1 cm^{-1} appear in untreated sisal and enset fiber respectively. In case of NaOH treatment, the decreased intensity of those peaks are proof that lignin reduced by NaOH treatment as the concentration of treatment increases from 5% to 10%. The peak at 1300 - 1500 cm^{-1} shows that C-H bending of hemicellulose, O-H stretch of cellulose and C-O stretching of NaOH group. The peak at 1240.3 cm^{-1} and 1240.62 cm^{-1} is attributed to C-O stretching of acetyl (lignin) and its intensive removal by 5% and 10% respectively. Peaks at 1369.54 cm^{-1} and 1367.04 cm^{-1}can be attributed to C-H bending (deformation) of hemicellulose compound and their intensity were removed by NaOH treatment. Peaks at 1029.05 and 1023.91 cm^{-1} can be attributed to the C-H stretching of hydroxyl group in cellulose and 891.58 and 896.30 cm^{-1} existence of b-glyosidic linkage between the monosachorides on untreated enset and sisal fibers. Similarly, those compounds do exist on treated fibers indicating that treatment does not minimize or remove those compounds from the fibers.

Finally, from tabulated result under subtopic 4.1, we conclude that, treated enset and sisal fiber showed less moisture absorbance than that of untreated fibers. This is due to removal of non cellulose components like hemicellulose and lignin. The chemical composition of enset and sisal fiber was improved by NaOH treatment for the better interfacial surface with the matrix materials. Increasing NaOH concentration removes the impurities from the fibers leading to reduced diameter of the fibers. FTIR spectroscopy data also indicates that there is a reduction of hemicellulose and lignin with 5% and 10% NaOH treatments. Tenacity results indicated that tensile strength of individual fiber at 5% NaOH treated enset and sisal fiber got better tenacity than untreated and 10% NaOH treated fibers.

4.3. Effects of fiber volume ratio, alkali treatment and fiber orientation on mechanical properties

Hybrid composites consist of a combination of two or more fibers in a polymer matrix. Fiber hybridization is a promising strategy, where two or more types of fibers are combined in a matrix of composite materials to mitigate the drawback of the type of fiber, and keep the benefits obtained from the others. The main factors affecting the mechanical properties of hybrid composites are the fiber volume ratio, stacking sequence of the fiber layers, treatment of fibers, and effect of environmental conditions. In addition to fiber treatment, a hybrid of two or more different fibers improves the mechanical properties of its composites. Hybridization of fibers is the best option to improve the mechanical performance of natural fiber polymer composites**.** The properties of individual fiber composites can be improved by hybridizing with other fibers, where double or multiple reinforcing phases with a individual matrix phase or individual reinforcing phase with double or multiple matrix phases are used. Weight fraction or fiber volume is the other main factors that affect the mechanical properties of the hybrid composites. Many researchers conducted the study by varying the fiber volume fraction and analysis the positive and negative impacts on mechanical properties of the hybrid composite based on that variation.

Hybridizing, orientation and chemical treatment are the main parameters that were to have effects the properties of natural fiber composites. Hybridizing is a promising strategy to improve the mechanical properties of its composites. The properties of individual fiber composites were improved by hybriding with other fibers to mitigate the disadvantage of one fiber by others, keeping advantages of others. When two natural fibers are hybridized, then the attention is often on getting a better balance in mechanical, chemical and physical properties, rather than on optimizing the hybrid effect. The other main factor that affects the mechanical properties of fiber-based composite is the way the fiber is oriented. The properties and structures of fiber based composites were defined by its fiber orientations. Rather than fiber volume ratio, fiber orientation has more influence on the physical properties of fiber composites. The hydrophilic character of natural fibers makes too poor interfacial interaction with hydrophobic polymeric materials that limit the stress transfer between the composite components. It is known that poor adhesion between fiber and matrix affected the mechanical properties of composites. Modifications have been major topics in natural fiber reinforced composites to improve the interfacial adhesion and in turn the overall properties of the composite product.

The composite was fabricated from enset and sisal and (Enset/Sisal) hybrid by varying weight ratios (100/0, 75/25. 50/50, 75/25 and 0/100) with constant polyester resin. The effects of fiber volume and treatment on mechanical and water absorption properties are evaluated for individual enset and sisal polyester composite and enset/ sisal fiber hybrid composites. The fractured surfaces of the composites were analyzed using scanning electron microscopy (SEM). Result guide to getting the optimum weight ratio and optimum treatment concentration to develop enset/sisal hybrid polyester composite.

This subtopic addresses the second specific objective of the research and part of the work has been published in Paper II (Bekele et al., 2022b) and paper IV (Bekele, et al., 2022, in review) covers the following results:

- Influence of fiber volume on mechanical properties of enset- sisal individual composite and enset/sisal hybrid composites.
- Influence of fiber volume percentage (100/0, 75/25, 50/50, 25/75, 0/100) of enset/sisal fibers on tensile properties, impact strength, flexural properties of hybrid composites.
- The effects of alkali treatment (untreated, 5%NaOH, 10%NaOH) of enset/sisal fibers on tensile, flexural and impact properties of hybrid composite.
- The effects of fiber orientation (unidirectional and woven) on mechanical (tensile, flexural and impact) properties of enset/sisal fiber hybrid composites.
- The effects of fiber volume percentages, treatment and fiber orientation on water absorption properties of enset/sisal hybrid composites.

4.2.1. Effects of fiber volume ratio on E/S hybrid composites

4.2.1.1. Tensile test

The tensile testing to measure the force required to break the test specimen and the extent to which the specimen stretches or elongates to the breaking point. After fabrication of the composite as described in Section 3.3.7.1. in Figure 3.10 (a) and (b) for woven and unidirectional respectively and test samples with dimensions of (250 x 25 x 5) mm length, width, and thickness respectively were prepared per ASTM D3039 standard recommendations.

With the same ratio of 100/0 and 0/100 E/S composite, i.e., a composite of pure enset fiber and pure sisal fiber respectively, the tensile strength of pure sisal fiber (0/100) is higher than that of enset (100/0) composites. This is probably due to the loss of the integrity of the matrix and insufficient wetting between fiber and matrix in composites. When sisal fiber was added to the composite, the tensile strength of the composite was improved. Since sisal fibers are more compatible with the polyester resin and the tensile strength of a composite material mainly dependent on the strength and modulus of its fibers.

From the experimental work results of the tensile strength of fabricated enset, sisal and enset/sisal hybrid composites materials, the following results were obtained. The load was applied in the fiber direction for unidirectional composites. For woven types of composite, the load was applied in the fiber direction. As the results in Figure 4.9. show, sisal polyester composites have higher tensile strength (152.3 MPa) than enset and enset/sisal hybrid composites. The enset polyester composite shows lower tensile strength (69.7 MPa) and 56.25 MPa than sisal and enset/sisal hybrid composite for unidirectional and woven types of orientations respectively. As can be observed from the results, the tensile strength of enset fiber composite was improved by hybridizing with sisal fiber resulting in better tensile strength. The tensile strength of 50/50 volume ratio of untreated unidirectional and woven enset/sisal polyester composite was improved by 43.5% and 51.3% respectively, when compare with 100/0 enset/sisal fiber composites. The tensile strength of 25/75 volume ratio of untreated unidirectional and woven enset/sisal polyester composite was improved by 47.8% and 55.3% respectively, when compare with 100/0 enset/sisal fiber composites. As the volume percentage of sisal fiber increases in the hybrid composite, the tensile strength was improved.

The increase in tensile strength of the hybrid composites is also due to the higher tensile properties of the fibers (Negawo et al., 2019), particularly that of sisal fibers ranging from 600-700 MPa (Venkateshwaran et al., 2011). The optimum tensile strength value for both types of fiber is 50/50 volume ratio. When the enset fiber volume percentage was increased, tensile and flexural strength properties decrease, but the impact strength was improved. The Tensile strength, tensile modulus, flexural strength, and flexural modulus showed a positive hybrid effect when the volume ratio of the fiber was varied in the hybrid composites at each fiber loading and similar result was observed (Idicula et al., 2010). Tensile strength is higher when fibers of greater strength are oriented longitudinally so as to bear the tensile load along the direction of the fibers. The force elongation of 50/50 volume ratio of enset/sisal fiber for 5%NaOH treated composite was tabulated on Figure 4.10 (a) and (b) for unidirectional and woven types of composites respectively.

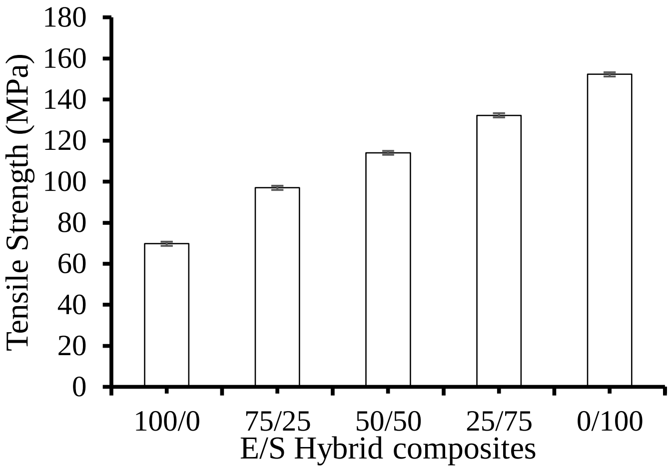
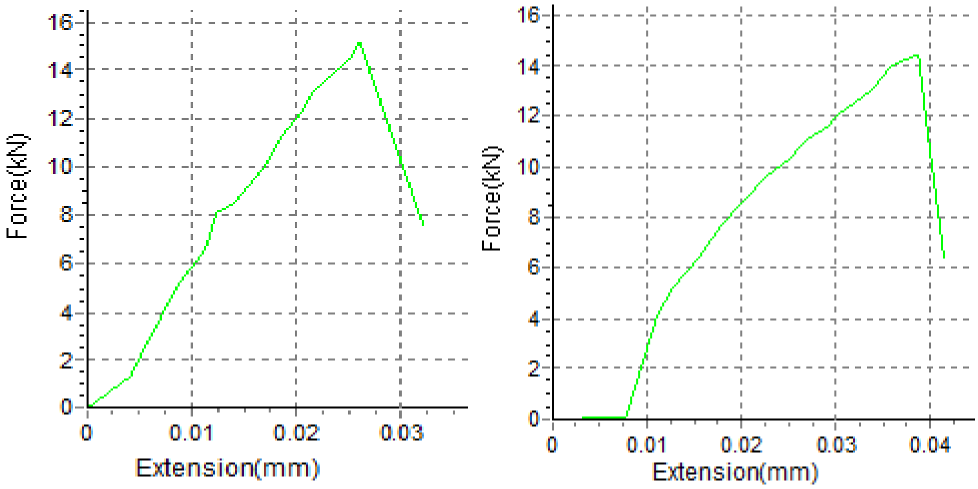


Figure 4. 9 Tensile strength test results



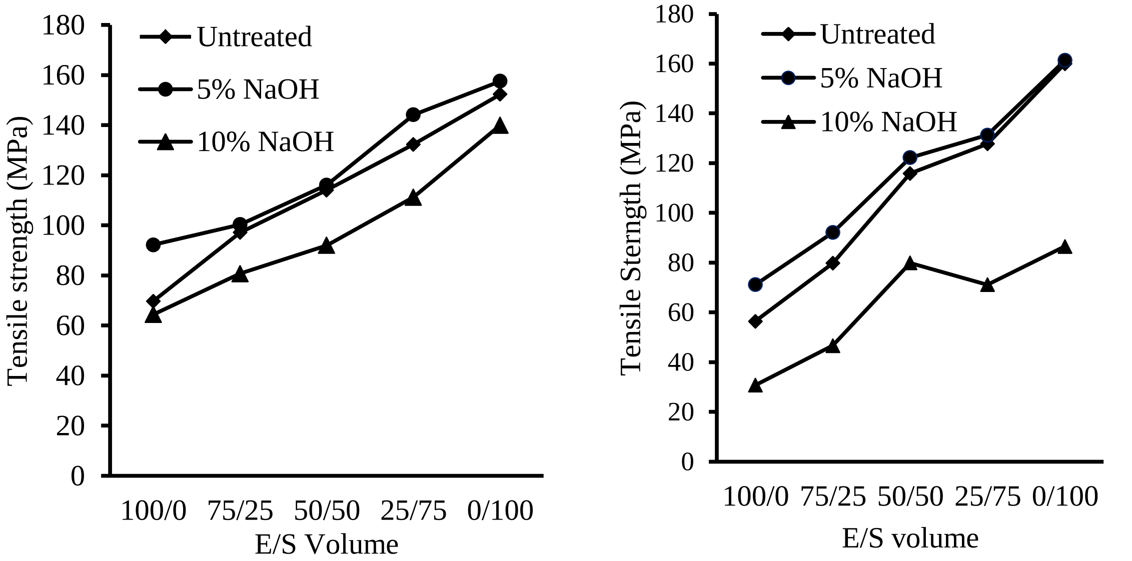
(a) (b)

Figure 4. 10 Forces verses elongation of 5% NaOH treatment with having 50/50 volume ratio (a) woven and (b) types of composites

Also, higher lignin contents affect the interfacial bond between the fiber and the polymers materials and makes the composite to have weak strength. Due to this reason, many researchers used fiber treatment to reduce the lignin content to improve the interfacial bond for better mechanical properties of the composite. As can be observed from Table 4.1, sisal fibers have less lignin contents than enset fibers. Due to this reason, sisal fiber composite has better mechanical properties than enset fiber composite and while increase sisal fiber in the hybrid composite is used to improve the tensile and flexural strength of the composite. It can be observed from Figure 4.11 that a 5% NaOH treated fiber hybrid composite has higher tensile strength than an untreated and a 10% treated fiber hybrid composite. This is because impurities were not fully removed from the fibers, which causes weak fiber matrix interfacial and a greater optimal condition for delignification, leading to weakening and damage of fibers (Kabir et al., 2012b). This is because the untreated fiber contains more lignin and hemicellulose contents than the treated fibers and 10% treated is high concentration of NaOH leads to damage the fiber. 5% NaOH result demonstrates optimum tensile strength for both fiber orientation composites. The optimal setting for eliminating impurities from fibers is a 5% NaOH treatment. Table 4.2 shows that the mechanical properties of both types of composite with untreated, 5% and 10% NaOH treated composite was tabulated.

Table 4. 2 Tensile, flexural, impact and water absorption properties of enset/sisal fiber hybrid composite

Hybrid composite	UTS			WTS			UFS			WFS			UIS			WIS			UWA			WWA		
	Untreated	5% NaOH	10% NaOH	Untreated	5% NaOH	10% NaOH	Untreated	5% NaOH	10% NaOH	Untreated	5% NaOH	10% NaOH	Untreated	5% NaOH	10% NaOH	Untreated	5% NaOH	10% NaOH	Untreated	5% NaOH	10% NaOH	Untreated	5% NaOH	10% NaOH
100/0	69,73	92,21	64,39	56,42	71,15	30,72	53,69	50,62	27,91	30,3	41,17	32,31	27,43	26,61	19,03	23,09	25,11	16,41	7,98	7,38	6,34	7,54	6,52	6,74
75/25	97,11	100,3	80,67	79,77	92,09	46,62	58,66	55,21	31,32	55,92	60,92	56,92	24,13	24,42	19,11	23,54	24,76	19,99	7,15	6,73	6,27	7,49	6,13	6,23
50/50	114,02	116,1	92,02	115,84	122,21	79,89	61,47	60,92	46,15	105,54	116,3	55,38	22,21	23,33	21,03	22,77	24,11	22,17	7,99	6,35	6,19	6,52	5,85	6,11
25/75	132,31	144,1	111,24	127,77	131,31	71,14	63,32	61,02	54,27	107,19	121,22	67,54	20,17	23,07	18,52	15,24	23,62	12,05	7,47	6,29	6,11	6,78	5,61	6,23
0/100	152,32	157,5	139,99	159,94	161,33	86,51	83,26	89,71	76,36	112,55	124,55	77,97	18,53	22,67	20,21	15,31	22,59	22,11	7,35	6,17	6,09	6,57	5,36	6,12
		Where: UTS-Unidirectional Tensile strength						UFS=Unidirectional Tensile strength					UIS-Unidirectional Impact strength						UWA=Unidirectional Water Absorption					
		WTS=Woven Tensile Strength						WFS=Woven Tensile Strength					WIS=Woven Impact Strength						WWA=Woven Water Absorption					



(a) (b)

Figure 4. 11 The tensile strength of (a) unidirectional (b) woven types of composites

The tensile strength of 100/0, 75/25, 50/50, 25/75 and 0/100 enset/sisal hybrid composite for unidirectional and woven composite are plotted in Figure 4.11(a) and (b) respectively. Those data values can be seen in Table 4.2. The result shows that increasing sisal fiber contents in the composite resulted in increased tensile strength. The tensile strength of untreated enset/sisal of 100/0 is 67.92 MPa and 0/100 is 152.32 MPa for unidirectional composites due to sisal fiber having higher tensile strength and better interfacial with the polyester materials. The tensile strength of untreated enset/sisal of

100/0 is 56.42 MPa and 0/100 is 159.94 MPa for woven composites. Untreated unidirectional enset polyester shows better tensile strength, but in case of sisal polyester composite woven types it is higher. It shows that enset fiber has better interfacial at a unidirectional fiber orientations and sisal has better interfacial with polyester matrix in woven types of fiber orientation in the field applications.

In both fiber orientation sisal polyester composite has higher tensile strength than enset polyester composite due higher tensile strength of sisal fibers and good adhesion with polyester matrix materials. While observing the tensile strength of untreated enset/sisal hybrid composite having 75/25, 50/50 and 25/75 volume ratio, 25/75 enset/sisal volume ratio the tensile strength was 132.31 MPa which is higher than polyester. In enset polyester composite the tensile strength enset polyester composite has been improved by hybridizing with sisal fiber in composites. So hybridization of sisal in enset shows positive response on tensile strength. The tensile strength of untreated, 5% and 10% alkali treated enset/sisal hybrid composites are shown in Figure 4.11, in which a positive hybrid effect was observed for the composites. It can be noted that increasing the sisal fiber content in the composite resulted in increased tensile strength of the composites. The tensile strength increased by 28%, 38% and 47%, 8.06%, 20.58% and 36.02%, 20.18%, 30.02% and 42.11% for unidirectional and 29.27%, 51.29% and 55.84% , 29.43%, 41% and 45.81% , 34%, 61.54% and 56.81% for woven types of enset/sisal hybrid of 75/25, 50/50 and 25/75 composites respectively compared to the 100/0 composites. Hybridizing high strength sisal fibers with enset and usage of treated fibers as reinforcement attributed to this enhancement in properties of both types of fiber orientation hybrid composite. An increase in properties of the treated fiber composites in comparison to untreated composites was observed due to alkali treatment.

Good fiber matrix adhesion resulting from physical modification like formation of rough surfaces, and defibrillation that occur on the fiber surfaces during alkali treatment improved the properties of enset composites. Figure 4.12 (a), (b) and (c) shows the SEM images of the tensile fractured surfaces of the enset/sisal hybrid unidirectional composites with having 100/0, 0/100 and 50/50 volume percentages respectively. The ruptured surfaces of the fabricated composites after tensile testing were evaluated using SEM. We observe that, the SEM image of 50/50 enset/sisal shows that better bonding was observed enset/ sisal hybrid composite.

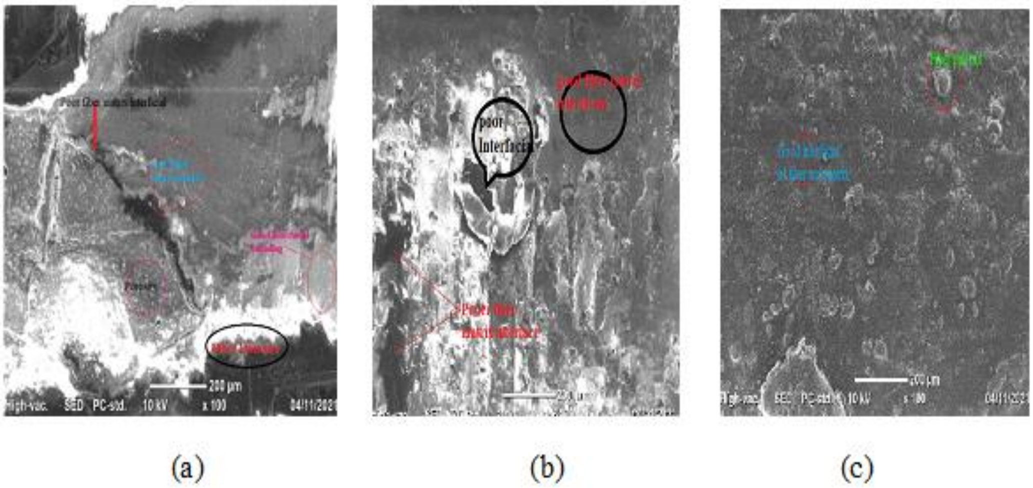


Figure 4. 12 SEM image of enset/ sisal hybrid composite (a) 100/0, (b) 0/100 and (c) 50/50

The hybrid composite (50/50) volume percentage was better adhesion between the fiber and matrix than enset composite and sisal composites. As the tensile strength value optimum for enset/sisal is 50/50 volume ratio, the effects of treatment were observed in Figure 4.11(a) and (b).

As shown in Figure 4.12 the woven types of composite higher flexural strength than unidirectional composites. The flexural strength of 5% NaOH treated was improved by 9.2%, for woven comparing with untreated composites. The adhesion between woven types of enset/sisal hybrid composite is better than that of unidirectional types of hybrid composites. As shown in Figure 4.13 the 5% NaOH treated fiber hybrid composite has a higher tensile strength than an untreated and a 10% treated fiber hybrid composite. This is due to the lack of removal of impurities from the fibers, which causes weak fiber matrix interfacial and a greater optimal condition for delignification, which causes weakening and damage of fibers. The 5% NaOH result demonstrates better tensile strength in both fiber orientation composites. The optimal setting for eliminating impurities from fibers is a 5% NaOH treatment. The woven types of fiber orientation composite have higher tensile strength than unidirectional fiber orientation composite in case of untreated, 5% and 10% NaOH treated. The tensile strength of 5% NaOH treated woven was improved by 5.2% comparing with untreated enset/sisal composites. Low tensile strength due to poor adhesion between matrix and the reinforcement.

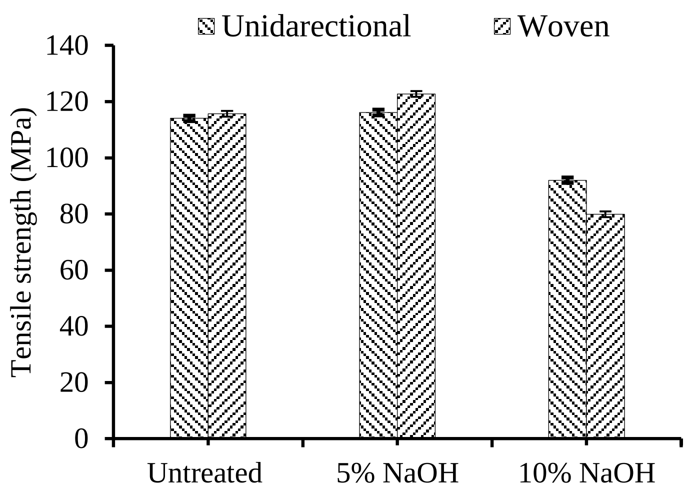


Figure 4. 13 The effects of treatment and orientation for 50/50 enset/sisal hybrid composites

The mechanical and wear properties of jute/hemp/flax hybrid composites was investigated and the result showed that tensile strength, tensile modulus and impact strength of jute/hemp/flax reinforced epoxy composite has improved whereas flexural strength and flexural modulus of some hybrid composite has reduced (Chaudhary et al., 2018a). Similar trends are observed in the results of the research reported in this thesis.

4.2.1.2. Flexural test

The flexural strength was measured according to methods described under Section 3.3.7.2 on Figure 3.11. The flexural strength of untreated, 5% and 10%NaOH treated unidirectional and woven enset/sisal fiber hybrid composite were in Figure 4.14 (a) and (b). It was observed that in all the hybrid composites the flexural strength increased with increasing sisal fiber volume percentages. The flexural strength increased by 8.47%, 12.65% and 15.20%, 8.31%, 10.88%, 39.52 and 48% for 75/25,50/50 and 27/75 enset/sisal hybrid composites respectively when compare with 100/0 enset/sisal composite. Similarly, the flexural modulus of the hybrid composites increased when sisal content was increased, but only marginal. In flexural properties are the presence of high modulus sisal and reinforcement using alkali treated fibers. Improved interfacial adhesion due to treatments has reduced defragmentation of fiber from the matrix increasing the properties of the hybrid composites (Yousif et al., 2012). Besides, from earlier researches (Peijs et al., 1990), it is known that flexural properties of hybrid composites are also influenced by the composition and adhesion levels of the fibers.

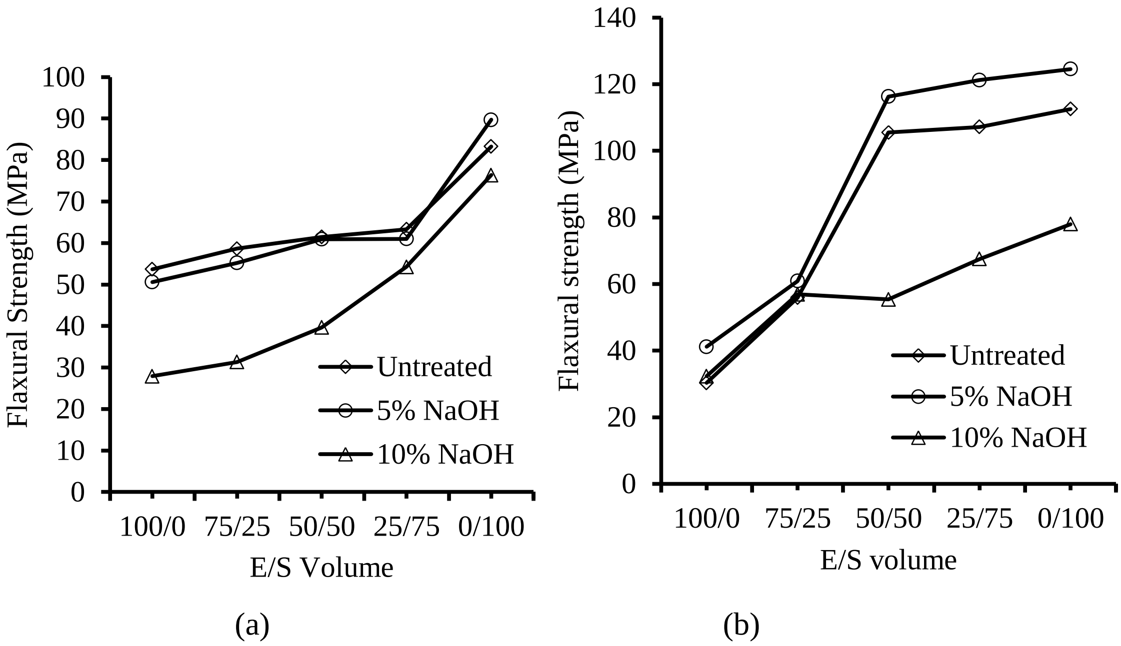


Figure 4. 14 The flexural strength of (a) unidirectional (b) woven types of composites

Weak fiber/matrix interfacial bonding contributes to poor flexural properties. The flexural strength of enset/sisal volume ratio (50/50) hybrid polyester composite is 114.02 MPa and 116.11 MPa for untreated and 5% NaOH treated for unidirectional and 116.11 MPa and 122.21 MPa for untreated and 5% NaOH treated composites.

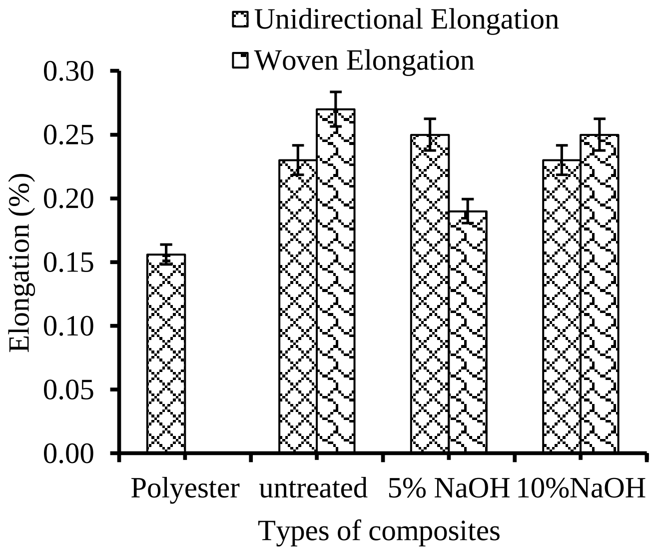


Figure 4. 15 (a) Elongation versus –types of composites for enset/sisal hybrid composite

Figure 4.15 indicates the effect of fiber volume ratio of enset and sisal on flexural strength in the hybrid composites. Sisal polyester composite flexural strength is higher than enset and enset/sisal hybrid polyester composites. This indicates that hybridazation of sisal fiber contributed in improving the flexural strength of enset polyester composite. Flexural strength of the hybrid composites is higher than enset polyester composites at each fiber loading. A positive hybrid effect is observed. In the hybrid composite, the flexural strength maximum at 50/50 enset/sisal fiber ratio than untreated and 5% NaOH treated fiber composites. Flexural strength is maximum when the relative fiber volume ratio of enset and sisal in the composite is 50/50.but in case of 10% NaOH treated fiber composites minimum. As explained earlier, higher compatibility as well as dispersion in hybrid composites is achieved, which lead to a better stress transfer ability in composites.

4.2.1.3. Impact tests

Impact resistance of a composite is measure of total energy dissipated in the material before final failure occurs. The test procedure was described in Figure 3.12. The cause for impact failure occurs, when matrix fracture, fiber/matrix deboning and fiber fracture are for composite materials. Among these fibers, pull–out is found to be an important energy dissipation mechanism in fiber reinforced composites. Fracture occurs when the stress level exceeds the fiber strength. The fractured fibers may be pulled out of the matrix, which involves energy dissipation. The effect of hybridization for unidirectional composite was depicted in Figure 4.16.

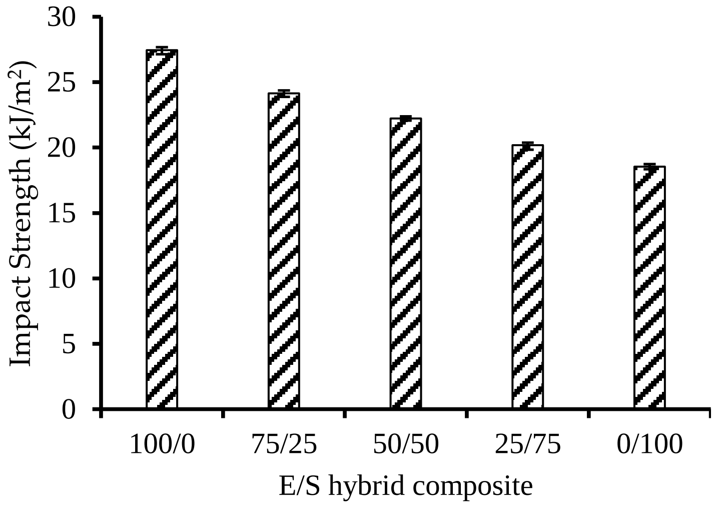


Figure 4. 16 Impact Strength test result of enset/sisal hybrid composites

The effect of fiber volume ratio on impact properties of the composites at untreated, 5% NaOH and 10% NaOH for enset/sisal polyester for unidirectional and woven composites. Enset reinforced polyester composites have demonstrated a significant increase in the values of impact strength for unidirectional and woven types of composite as compared with sisal reinforced and enset/sisal hybrid composite which has shown an impact strength of 27.43 kJ/m^2 and 26.61 kJ/m^2 respectively. But the sisal reinforced composite has less impact strength and improved as enset fiber is added to the composite. As can be observed from the results, 50/50 enset/sisal reinforced fibers have the optimum (the average of maximum and minimum), value (22.21 kJ/m^2). These optimum values of the composite were improved by 16.57% compared with the sisal reinforced polyester composite. The high impact strength of enset fiber is due to the high porous nature of it due to the higher lumen size and high spiral angle. Also, better computability will decrease the impact strength due to the least possibility fiber pull out in the hybrid composite that discus on flexural and tensile strength section.

The effects of treatment on impact strength were shown on Figure 4.17. Though there is a need to guaranty the formation of strong bond to achieve materials for interior and structural application, weak interfacial link provides increased toughness through the promotion of pullout eff ects needed in energy-absorption i.e., for impact strength purposes. Due to this reason the enset polyester composite has better impact strength than other hybrid composites.

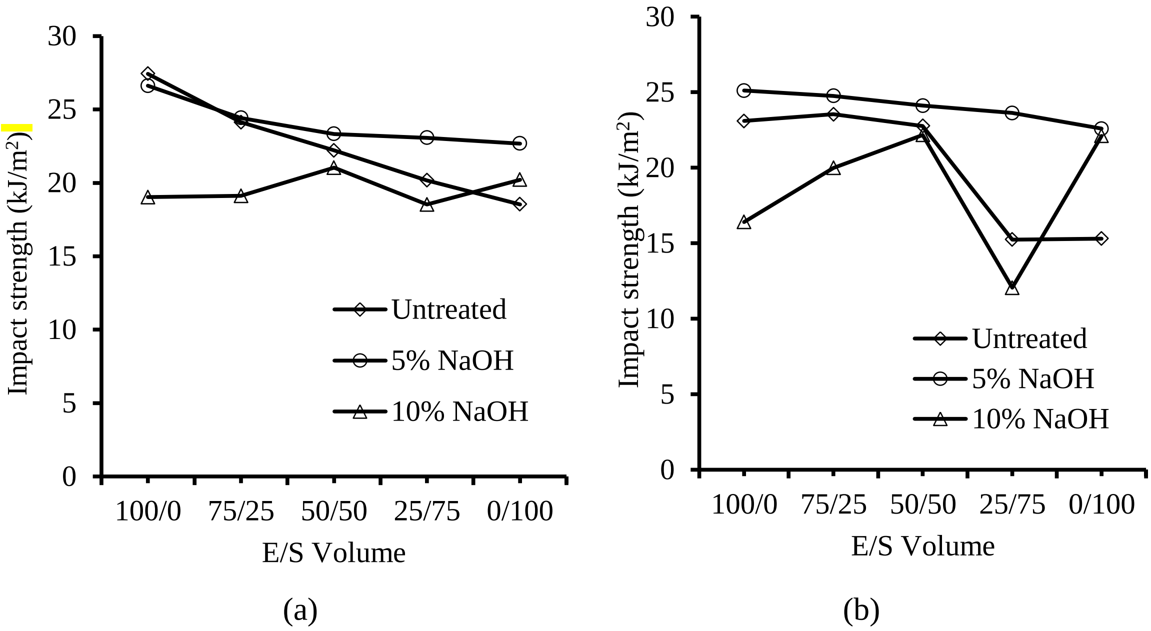


Figure 4.17 The impact strength of (a) unidirectional (b) woven types of composites

4.2.1.4. Moisture absorption

Representative curves of the moisture absorption composites of individual and hybrid composites over time are shown in Figure 4.18**.** As observed from the results, enset fiber composite (100/0) has higher water absorption than sisal (0/100) and enset/sisal hybrid polyester composite. At 50/50 enset/sisal hybrid reinforced composite ratio we observe the water absorption in between 100/0 and 0/100. The water absorption properties of all composites are the same that means for the first 12 hrs, the range of water absorption varied from 5% to 6% and after 48 hrs and 72 hrs, the water absorption increased from

6.5% to 9% and stayed somewhat constant after 84 hrs. As a reinforcement in composite materials, expansion of the fiber after water absorption would lead to micro-cracks in the composite material, thus affecting its mechanical properties (Inder et al., 2017). One can only conclude that types of fiber, fiber volume ration, fiber orientation and fiber treatment, compatibility between fiber and matrix can help reduce water uptake in natural fiber composite as shown in Figure 4.18(a) and (b).

For polymer composites water absorption test is very critical aspect especially for bio-composites incorporating various natural fibers which is the case for the present study. As natural fibers are hydrophilic in nature, so these are having affinity to water. The moisture uptake capacity depends upon the basic constituents of natural fibers.

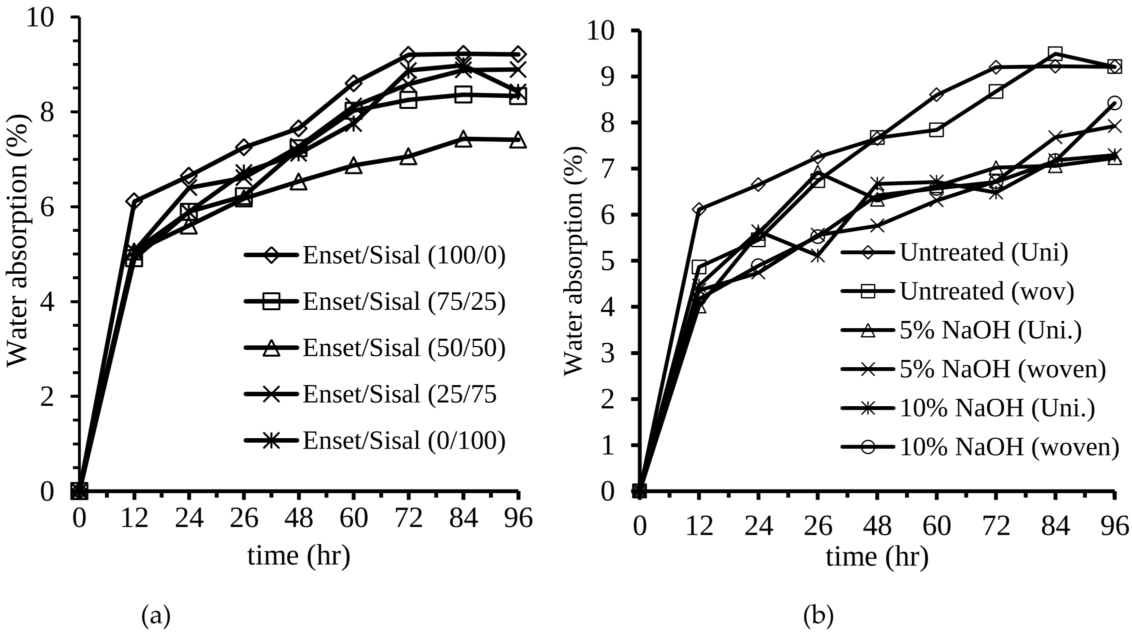
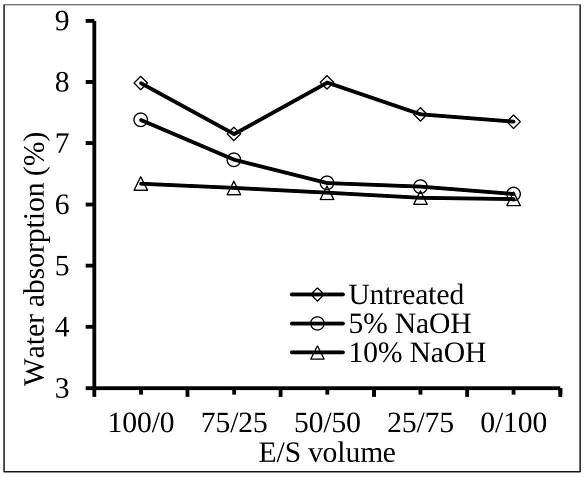


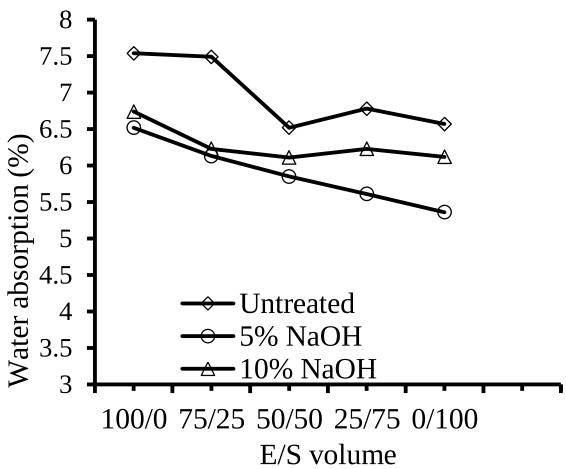
Figure 4.18 Moisture absorption test result of hybrid enset/sisal hybrid composites

The water absorption test reveals that how the developed hybrid composites behave against moisture in practical applications where moisture exposure is almost inevitable situation (Chaudhary et al., 2018b). The enset/sisal hybrid composite percentage weight gain of water as calculated using Equation (3.7) has been plotted with respect to time. As the time of immersion increases the percentages of water absorption also increases in all types of composites. Untreated unidirectional and woven composite have higher water absorption properties than treated unidirectional and woven types of composites. As observed from the result in Figure (4.18), woven types of enset/sisal hybrid composites absorbed maximum 9.5% water in 84hr. Unidirectional enset/sisal hybrid composites absorbed minimum 7.06% water in 84 hr. This is since untreated fiber contains higher lignin and hemicellulose contents than treated fibers as shown on Table 4.2. The minimum water absorption was shown at 5%NaOH treatment for both types of fiber orientation composites. The better interfacial of fiber and polymer was leads to improve the mechanical and water absorption properties of composites.

Figure 4.19 (a) and (b) indicates the effects of fiber volume for untreated, 5% and 10%NaOH treated composites and the result showed that as the volume ratio of enset increased, more water is absorbed in the hybrid composites. This indicates that hybridization of sisal fiber in the composite is used to improve the properties of enset polyester composite. In all types of composites, a woven type of composite was absorbing less water than unidirectional composite. The sisal/bamboo hybrid composite of moisture composite was analyzed and the result showed that for all ratios of composites, moisture absorption becomes stable after 55 hrs and from hybrid composite of 50/50 volume percentage has the lowest water uptake and permeability coefficient (Venkatesh & Ramanathan, 2016). Similar trends are also observed in the results obtained in this research.



(a)



(b)

Figure 4. 19 Water absorption of (a) unidirectional and (b) woven types of composites

The types of fibers, fiber orientations, fiber length, fiber weight ratio and composite thickness, and types of matrix used are the main factors that affect the mechanical properties of natural fiber hybrid composites (Mechanics et al., 2016). Table 4.3 shows that untreated enset/sisal fiber hybrid composite with having 50/50 volume ratio. The result indicates that mechanical properties are higher in tensile strength and similar result in flexural strength with the existing literature. Higher tensile strength may due to composite thickness and enset and sisal fiber weight ration and better interfacial bonding of two fibers in the polymer resin. The impact strength of the study is similar with the existing literature.

Table 4. 3 The existing and current study and existing literature of mechanical properties of natural fiber hybrid composites

Hybrid composites	Tensile strength (MPa)	Flexural strength (MPa)	Impact (kJ/m^2)	Reference
Sisal/Enset/polyster	114.02	61.21	22.21	***Current Study***
Jute/Sisal/ epoxy	66.77	114.01	32.25	(Cavalcanti et al., 2019a)
Banana/Jute/polyester	25.83	182.34	20.1	(Adhikari, et al.,2017)
Sisal/Jut/epoxy	42.45	39.8	-	(Premnath, 2018)
sugarcane leaves/	12.86	39.91	-	(Mechanics et al., 2016)
Hemp/Jute/Polyester	50.83	73.39	-	(Sowmya et al., 2018)
Palmyra Palm Leaf Stalk Fibers/Jute	64.3	145.6	27.01	(Shanmugam & Thiruchitrambalam, 2013b)
Hemp/Flax/epoxy	44.17	44.61	4.18	(Chaudhary et al., 2017)
Sisal/epoxy	153.8	-	-	(Sivakandhan et al., 2019)

Table 4.4. shows comparison the mechanical properties (tensile, flexural and impact) of untreated and treated various natural fiber hybrid polyester composites. As we understood from the literature, treated hybrid composite better mechanical properties than untreated hybrid composites, treatment has improved the mechanical properties to a large extent.

Table 4. 4 Effects of alkali treatment on mechanical behaviors of natural fiber reinforced hybrid composites.

Composites	Tensile strength (MPa)		Flexural strength (MPa)		Impact (kJ/m^2)		Reference
	Unt.	Trt.	Unt.	Trt.	Unt.	Trt.	
Enset/Sisal unidirectional	90.23	116.12	38.77	60.92	22.21	23.33	***Current Study***
Enset/Sisal Woven	115.67	122.56	105.23	116.3	22.77	24.11	***Current Study***
Jute/Sisal	70.39	74.78	67.56	54.67	-	-	(Cavalcanti et al., 2019a)
Jute/Curaua	66.77	63.9	97.67	80.86	-	-	(Cavalcanti et al., 2019a)
Sisal/Jut	42.45	53.7 4	39.8	44.58	19.5	22.25	(Perremans et al., 2018)
Bagasse aliphatic	23.47	26.77	43.87	50.86	8.82	11.27	(Cao et al., 2006)
sugarcane leaves/ almond shell	12.86	17.16	39.91	40.68	-	-	(Mechanics et al., 2016)
sisal fiber epoxy composite unidirectional	132.73	-	288.6	-	-	-	(M.K. Gupta, 2014)

where: Unt = Untreated, Trt = Treated

The tensile, flexural and impact strength of 5% NaOH treated at 50/50 volume ratio of was improved by 22%, 36%, and 4% respectively for unidirectional and 5%, 9%, and 5% respectively for woven types of composites while comparing with untreated 50/50 volume ratio of composites. In this study similarly result can be seen that all the other hybrid composites tabulated in Table 4.4.

Finally, from the result under subtopic 4.2, it can be concluded that the enset/sisal fiber was successfully fabricated hybrid composite at 30% of fibers and 70% of the resin. The tensile and flexural strength of enset polyester composites is lower than sisal polyester composite at a similar fiber weight ratio. The addition of sisal fiber increases the tensile and flexural strength and decreases the impact strength of the hybrid composite. The tensile strength of enset polyester composite was improved by 38.8 % and 12.6% of tensile strength and flexural strength at 50/50 weight ratio. The impact strength of enset polyester composite better than sisal polyester composite. The addition of enset fiber improved the load absorption capacity by 16.6% at 50/50 weight ratio. The water absorption minimum for 50/50 enset/sisal hybrid composite than enset and sisal polyester composite. The recommended weight percentage of enset/sisal hybrid composite is 50/50 to get adequate mechanical and water absorption properties. Result guide to getting the optimum weight ratio to develop enset/sisal hybrid polyester composite of different applications.

The treated fiber composite has better mechanical properties than untreated fiber hybrid composites, while woven types of fiber orientation have better mechanical properties than unidirectional fiber orientation in both untreated and treated hybrid composites. In case of unidirectional fiber orientation, the result of tensile and flexural strength of 10%NaOH composite similar with untreated fibers but, less in woven fiber orientation composites. The higher tensile and flexural strength was obtained for 5%NaOH in both unidirectional and woven types of fiber orientation composites when compare with untreated and 10% NaOH treated composites. Water absorption properties of treated E/S hybrid composite less than untreated hybrid composite. The analysis of SEM images for unidirectional and woven types of composites mode of failure of natural fibers and matrix during the tensile test was analyzed and the better surface interference was appeared in woven fiber orientation composite with a having 5% NaOH treatment. The E/S hybrid polyster composite was successfully fabricated and the 5% NaOH treatment is used to improve the composite properties for the wider applications.

4.2.1.5. Two factor ANOVA for tensile, flexural, impact and water absorption

Tables 4.5 to 4.12 show summary of the ANOVA study conducted on tensile, flexural and impact strength as well as the water absorption of both woven and unidirectional fibers.. From these results, it is observed that treatment factor is statistically significant for tensile, flexural, impact strengths and water absorption properties for both types of composite samples. Hybridization factor is statistically significant for tensile and flexural strength of both composite samples and for woven water absorption properties. Hybridization factor is not statistically significant on the impact strength of unidirectional and woven samples, and on the water absorption properties of the unidirectional composite samples. However, there remains a lot of uncertainty about whether any of the averages are different regarding the p-value of both samples in impact strength and water absorption. Furthermore, there’s a lot of uncertainty about whether any one average is larger than another. In tensile and flexural strength, hybrid factor shows the higher percentage of contribution for unidirectional and woven composite samples. Similar situation is observed in woven impact strength. In both composite samples of water absorption properties and unidirectional impact strength, treatment factor shows the highest percentage contribution.

Table 4. 5 ANOVA for tensile strength for unidirectional fiber based composite

Source of Variation	SS	DF	MS	F	P-value	F crit	%Contr.
Hybridization	10383.43	4	2595.85	99.42	7.44E-07	3.837	85.71
Treatment	1522.194	2	761.09	29.151	0.000212	4.458	12.56
Error	208.8683	8	26.10				1.72
Total	12114.5	14					

Table 4. 6 ANOVA for tensile strength for woven fiber based composite

Source of Variation	SS	DF	MS	F	P-value	F crit	%Contr.
Hybridization	12891.09	4	3222.77	30.64	6.7E-05	3.83	59.079
Treatment	8087.48	2	4043.74	38.45	7.88E-05	4.45	37.06
Error	841.21	8	105.151				3.85
Total	21819.78	14					

Table 4. 7 ANOVA for flexural strength for unidirectional fiber based composite

Source of Variation	SS	DF	MS	F	P-value	F crit	%Contr.
Hybridization	2766.07	4	691.51	26.78	0.00011	3.83	71.09
Treatment	917.83	2	458.91	17.77	0.001139	4.45	23.59

Error	206.57	8	25.82				5.30
Total	3890.48	14					

Table 4. 8 ANOVA for flexural strength for woven fiber based composite

Source of Variation	SS	DF	MS	F	P-value	F crit	%Contr.
Hybridization	10953.55	4	2738.38	12.6716	0.00153	3.837	69.025
Treatment	3186.407	2	1593.20	7.37238	0.015305	4.458	20.07
Error	1728.835	8	216.10				10.89
Total	15868.79	14					

Table 4. 9 ANOVA for impact strength for unidirectional fiber based composite

Source of Variation	SS	DF	MS	F	P-value	F crit	%Contr.
Hybridization	30.68897	4	7.672243	1.932424	0.198425	3.83	27.07
Treatment	50.88945	2	25.44473	6.408818	0.021809	4.45	44.89
Error	31.76215	8	3.970268				28.02
Total	113.3406	14					

Table 4. 10 ANOVA for impact strength for woven fiber based composite

Source of Variation	SS	DF	MS	F	P-value	F crit	%Contr.
Hybrid	73.79317	4	18.44	1.862	0.210	3.83	31.52
Treatment	81.05584	2	40.52	4.091	0.059	4.45	34.62
Error	79.24083	8	9.905				33.85
Total	234.0898	14					

Table 4. 11 ANOVA for water absorption for unidirectional fiber based composite

Source of Variation	SS	Df	MS	F	P-value	F crit	%Contr.
Hybrid	0.89016	4	0.222	2.543	0.121	3.837	13.23
Treatment	5.136693	2	2.568	29.359	0.0002	4.458	76.36
Error	0.69984	8	0.087				10.40
Total	6.726693	14					

Table 4. 12 ANOVA for Water absorption for woven fiber based composite

Source of Variation	SS	df	MS	F	P-value	F crit	%Contr.
Hybrid	1.717267	4	0.4293	9.4778	0.0039	3.837	33.64
Treatment	3.024493	2	1.5122	33.385	0.0001	4.458	59.25

Error	0.362373	8	0.0452	7.09
Total	5.104133	14		

4.4. Machinability in drilling operation of E/S hybrid polyester composites

Machining of natural fiber reinforced composites is complicated task due to the mechanically anisotropic and inhomogeneous structure. After the fabrication of natural fiber reinforced composites, post processing operation is required. Machining operation is one of the mostly used post processing operation to facilitate the parts for the assembly. The most machining operation used to facilitate assembly operation is drilling or making holes. During assembly operation processes, the drilled hole delamination factors lead to account the rejection of the composite products. The damage that occurs around a drilled hole is known as damage factor or delamination factor. Surface roughness (Ra) is defined as the average mean of the deviation of the roughness profile from the average line within the estimated length. In drilling, many factors affect the machinability; some of the important machining parameters are the spindle speed, the feed rate, and the drill diameter. The quality and performance of a composite product is highly dependent on the surface condition created by machining. This defect was improved by proper selection of cutting condition like feed, speed, tool material and tool geometry, poor surface roughness of the hole wall and fiber/resin pull-out and among the above issues associated with drilling, delamination appears to be the most critical. In drilling, surface roughness is the indication of irregularity of circumferential drilled hole surface, the reason for the development of high wear, fatigue and corrosion mechanisms (Lotfi et al., 2019).

This subtopic addresses the third specific objective of the research and part of the work has been published in Paper III (Bekele et al., 2022c) covers results for the:

- Influence of fiber orientation on the surface roughness and delamination factor of 5% NaOH treated enset/sisal hybrid composites.
- The effects of drill parameter (feed, speed and drill diameter) for the surface roughness and delamination factors of 5% NaOH treated enset/sisal hybrid composites.
- Analysis the effects of 5%NaOH treated enset/sisal for (unidirectional and woven) types of fiber orientation using Taguchi methods techniques. Parameters using treated of enset and sisal fibers.

The data in Table 4.13 show the factors to be studied. The Taguchi design of experiment methods and result are analyzed using analysis of variance (ANOVA) techniques to understand the effects of each parameter on delamination and surface roughness of the drilled hole surface. MINITAB 18 statistical analysis software were used for the design and analysis the data in the experiments. In drilling of enset/sisal hybrid composite process, the parts were rejected due to defects such as tearing along the in the entry and exit and poor surface finish. It was identified that delamination factors and surface roughness have been the causes for the defects.

Table 4. 13 Levels of the variables used in the experiment.

Parameters	Levels		
	1	2	3
Speed, rpm	600	1200	1800
Feed rate, mm/rev	0.10	0.20	0.3
Drill diameter, mm	6	9	12

Taguchi design for this experiment was tabulated and the experiment results are given in Table 4.6. As given, there are two responses; (1)the delamination factor (DF) and (2) the Surface Roughness (SR). For both F_d and SR, are smaller the better type of quality characteristic and analysis of S/N ratio was used using Equation (3.10). L_9 orthogonal array was used as shown in drilled hole samples in Figure 4.20.

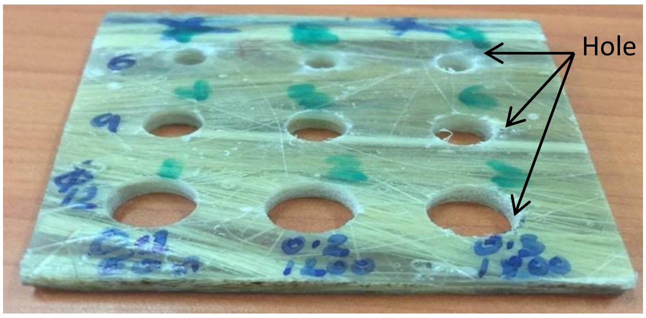


Figure 4. 20 L9 Drilled hole designed by Taguchi

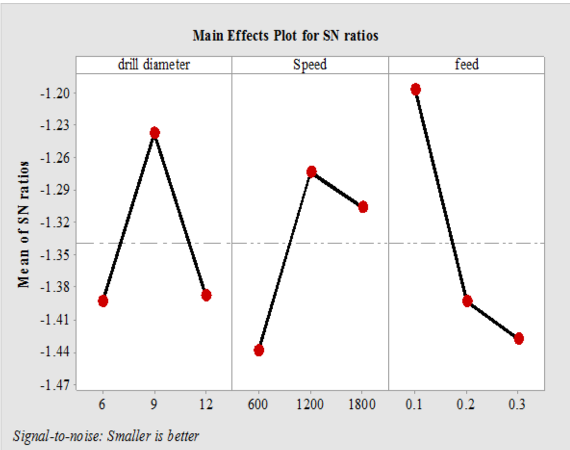
As shown in Table 4.14, the delamination factor increases with an increasing feed rate and speed. As feed rate increases, the thrust force also increases leading to increasing delamination. Similar results were obtained in this study. Also, lower feed rate reduces the tool wear result in reducing the cutting force which helps to reduce the delamination and surface roughness. Smaller diameter helps to reduce the cutting force and wear which helps to minimize the delamination. This could be because higher speed reduces the cutting force and torque and also results in continuous chip that makes drilled hole free of crack and sub crack. Higher fiber content leads to increased the thrust force and torque which increase the delamination.

Minimum delamination takes place when fiber is optimum in volume and higher treated time. S/N ratio of the speed (rpm), feed (mm/rev) and drill bit diameter (mm) for the delamination and surface roughness of the hole drilled by 1200 rpm speed , 0.1 mm/rev feed and 9 mm drill bit diameter were optimum as shown on Figure 4.22 (a and b) and Figure 4.23 (a and b) because this combination has the higher signal to noise ratio and similar result was obtained (Krishnaraj et al., 2012).

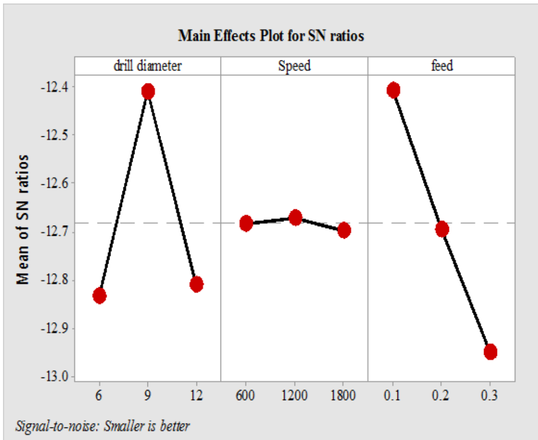
Table 4. 14 L_9 orthogonal array and the desired parameter valued

Trial	Parameters			Delamination factor (Fd)		Surface roughness (SR) (µm)		Signal to Noise (S/N)			
	Drill diam. (mm)	Speed (rpm)	Feed rate (mm/rev)	UFd	WFd	USR	WSR	UFd	WFd	USR	WSR
1	6	600	0.1	1.15	1.165	3.45	4.15	-1.252	-1.33	-10.8	-
2	6	1200	0.2	1.16	1.176	3.79	4.49	-1.309	-1.41	-11.7	-
3	6	1800	0.3	1.15	1.181	3.53	4.51	-1.268	-1.63	-11	-
4	9	600	0.2	1.16	1.173	3.58	4.16	-1.44	-1.48	-11.1	-12.4
5	9	1200	0.3	1.14	1.153	3.05	4.20	-1.261	-1.24	-9.83	-12.6
6	9	1800	0.1	1.12	1.133	2.95	4.14	-1.057	-1.11	-9.46	-
7	12	600	0.3	1.19	1.202	3.72	4.61	-1.578	-1.6	-11.6	-
8	12	1200	0.1	1.13	1.145	3.12	4.21	-1.111	-1.21	-10.2	-
9	12	1800	0.2	1.15	1.173	3.51	4.28	-1.283	-1.39	-11	-

UF_d = unidirectional delamination factor WFd = woven delamination factor
USR = unidirection Surface roughness WSR = Woven Surface roughness

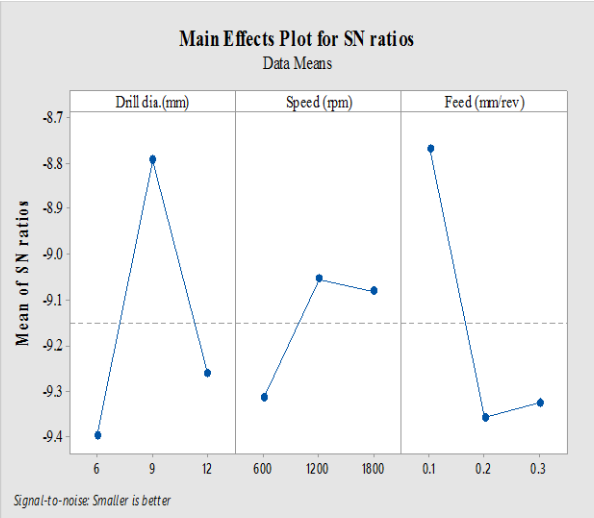


(a)



(b)

Figure 4. 21 Main effects plot for S/N ratio of (a) WF_d and (b) WSR types of composite



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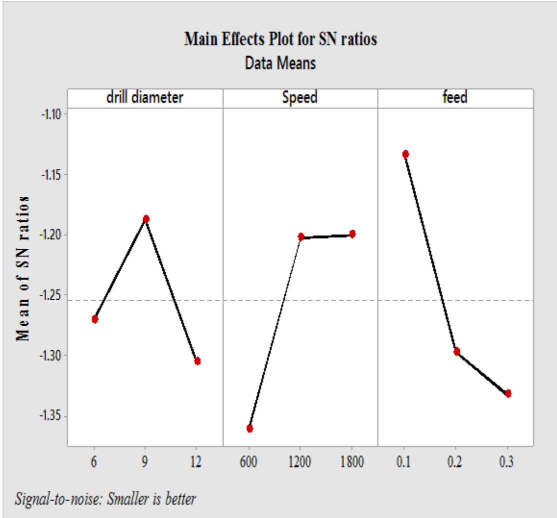


(b)

Figure 4.22. Main effects plot for S/N ratio of (a) USR and (b) UF_d types of composite

As shown on Table 4.15 WFd the optimum process parameters on the surface roughness are obtained as feed at level 1 (0.1 mm/rev), speed at level 2 (1200 rpm), drill diameter (12 mm). Table 4.16 WSR the optimum process parameters on the surface roughness are obtained as feed at level 1 (0.1 mm/rev), speed at level 2 (1800 rpm), drill diameter (9 mm). Table 4.17 UFd the optimum process parameters on the surface roughness are obtained as feed at level 1 (0.1 mm/rev), speed at level 2 (1200 rpm), drill diameter (12 mm).

Table 4. 15 Response WF_d for S/N ratio

Level	Drill diam. (mm)	Speed (rpm)	Feed rate (mm/rev)
1	−11.10	−11.09	−10.02
2	−10.07	−10.40	−11.20
3	−10.75	−10.43	−10.70
Delta	1.03	0.69	1.18
Rank	2	3	1

Table 4. 16 Response WSR for S/N ratio

Level	Drill diam. (mm)	Speed (rpm)	Feed rate (mm/rev)
1	−1.271	−1.361	−1.134
2	−1.187	−1.203	−1.297
3	−1.306	−1.200	−1.333
Delta	0.118	0.161	0.199
Rank	3	2	1

Table 4. 17 Response UFd for S/N ratio

Level	Drill Diam. (mm)	Speed (rpm)	Feed Rate (mm/rev)
1	−12.83	−12.68	−12.41
2	−12.41	−12.67	−12.69
3	−12.81	−12.70	−12.95
Delta	0.43	0.03	0.54
Rank	2	3	1

Table 4. 18 Response for S/N USR ratio

Level	Drill diam. (mm)	Speed (rpm)	Feed rate (mm/rev)
1	−1.393	−1.439	−1.198
2	−1.238	−1.274	−1.393
3	−1.388	−1.306	−1.428
Delta	0.155	0.166	0.231
Rank	3	2	1

As can be obseved from S/N ratios in Table 4.7-4.10, the feed is the main parameter for both types of composites (unidirectional and woven) and both responses (delamination factor and surface roughness). But for speed is the second main parameter ifluence the surface roughness of unidirectional and wove types of composite and drill diameter is the second parameter influrence the delamination of unidirectional and woven types of composite. It means that, feed is the critical parameter affects the delamination and surface roughness of enset/sisal fiber hybrid polyester composites.for both types of rresponse i.e delamination and surface roughness 0.1mm/rev is the optimum value used to produce best delamination and surface roughness in both types of composites. Delamination of unidirectional and woven types of composite, in addition to 0.1mm/rev feed, the 1200 rpm speed and 12 mm drill diameters level values are the optimum for both types of composites (unidirectional and woven).But for surface roughness 1800rpm speed and 9mm drill diamter optimum values that used to get the best surface of unidirectional and woven types of enset/sisal hybrid polyester composites.the result similar to the result of (Packiara and Kumar, 2009).

Analysis of variance (ANOVA) has been used to statistically analyze the effect input process parameters individually and their interaction on delamination and surface roughness for both unidirectional and woven types of enset/sisal fiber composites. The ANOVA data are shown in Table (4.19-22). From Table 4.19, it is found that feed rate is the major parameter contribution (44.93%) for UF_d followed by speed and drill bit diameter (34.31%) and (14.90%) respectively. From Table 4.20, it is found that feed rate is the major parameter contribution (43.48 %) for USR followed by drill bit diameter and speed (33.56 %) and (18.34%) respectively. Similarly, it is observed from Table 4.21 that feed rate is the major parameter contribution (48.98%) for WF_d followed by speed and drill bit diameter (24.65%) and (24.44%) respectively. Table 4.22 also shows that feed rate is the major parameter contribution (42.73%) for WSR followed by drill bit diameter and speed (32.93%) and (0.11%) respectively. It implies that the feed rate is the main parameter influencing both types of composites (unidirectional and woven) and both responses (delamination factor and surface roughness). On the other hand, speed is the second main parameter ifluencing the surface roughness of unidirectional and wove types of composites and drill diameter is the second parameter infulencing the delamination of unidirectional and woven types of composites. It means that, speed and drill diamter are less influencial on delamination and surface roughnesss, respectively. From the result presented above, the feed is seen to make the largest contribution to the delamination and surface roughness. That my result similar with the (Oluwarotimi et al., 2016).In general, as the speed increase and feed decrease, the delamination and surface roughness become reduced in both types of composite surface. That my result similar with the (Babu et al., 2013)

Higher delamination is observed on both unidirectional and woven fiber types of orientation composite at 1200 rpm. It is also observed from the data that the effect of feed rate on the delamination and surface roughness decreases. In general, as the speed increases and feed rate decreases, the delamination and surface roughness is reduced in both types of composite surfaces.

Table 4. 19 ANOVA for the UF_d

Source	DF	Seq SS	%Contribution	Adj SS	Adj MS	F-Value	P-Value
Drill diameter (d)	2	0.000397	14.90	0.000397	0.000199	2.54	0.282
Speed (v)	2	0.000915	34.31	0.000915	0.000458	5.85	0.146
Feed (f)	2	0.001199	44.93	0.001199	0.000599	7.67	0.115
Error	2	0.000156	5.86	0.000156	0.000078		
Total	8	0.002667	100.00				

DF-degrees of freedom, SS-sum of squares, MS-mean squares, F and P-values

Table 4. 20 ANOVA for the USR

Source	DF	Seq SS	Contribution	Adj SS	Adj MS	F-Value	P-Value
drill diameter(d)	2	0.24119	33.56%	0.24119	0.12059	7.25	0.121
Speed(v)	2	0.13179	18.34%	0.13179	0.06589	3.96	0.202
Feed(f)	2	0.31250	43.48%	0.31250	0.15625	9.40	0.096
Error	2	0.03326	4.63%	0.03326	0.01663		
Total	8	0.71874	100.00%				

DF-degrees of freedom, SS-sum of squares, MS-mean squares, F and P-values

Table 4. 21 ANOVA for the WFd

Source	DF	Seq SS	%Contribution	Adj SS	Adj MS	F-Value	P-Value
drill diameter(d)	2	0.000831	24.44	0.000831	0.000415	12.64	0.073
Speed(v)	2	0.000838	24.65	0.000838	0.000419	12.75	0.073
Feed(f)	2	0.001665	48.98	0.001665	0.000833	25.33	0.038
Error	2	0.000066	1.93	0.000066	0.000033		
Total	8	0.003400	100.00				

DF-degrees of freedom, SS-sum of squares, MS-mean squares, F and P-values

Table 4. 22 ANOVA for the WSR

Source	DF	SS	%Contribution	Adj SS	Adj MS	F-Value	P-Value
drill diameter(d)	2	0.085429	32.93	0.085429	0.042715	1.36	0.424
Speed (v)	2	0.000276	0.11	0.000276	0.000138	0.00	0.996
Feed(f)	2	0.110878	42.73	0.110878	0.055439	1.76	0.362
Error	2	0.062881	24.24	0.062881	0.031441		
Total	8	0.259464	100.00				

DF-degrees of freedom, SS-sum of squares, MS-mean squares, F and P-values

4.4.1. Regression analysis

The correlation equations have been developed to calculate delamination and surface roughness for in terms of speed feed rate and drill bit. Mathematical regression equations for unidirectional delamination and surface roughness and woven delamination and surface roughness for surface roughness have been developed with the help of ANOVA, which are given in Table (4.19-4.22).The correlation between the factors v, f and d and the responses of the unidirectional and woven delamination factor (UF_d & WF_d) and the surface roughness (USR and WSR) fiber orientation enset/sisal fiber hybrid polyster composite were determined by multiple linear regression using Minitab18 software. The linear regression models as functions of the factors v, f and d for the above-mentioned parameters are described by the following equations in Table 4.23. The effectiveness of the developed model is measured by "R-*sq*" values. The R-*sq* indicates the closeness of the developed model with respect to real experimental values. When the value equal to 1 represents the model is exactly the same with the experimental result that is 100%. Two models show R-*sq* values greater than 0.95, which indicates the models are very effective in predicting the responses and one model R-*sq* values is 0.9414 which indicate the model is effective in the prediction and one model R-*sq* values is 0.7576 which not effective with respect to the machining variables. Similar result was obtained for milled natural fiber reinforced composite (Babu et al., 2013). In general, the correlation between the factors (v, f and d) responses unidirectional and woven delamination factor (UF_d & WF_d) and the surface roughness (USR and WSR) fiber orientation enset/sisal fiber hybrid polyster composite were determined by multiple linear regressions using Minitab18 software.

Table 4. 23 Regression models for predicting delamination and surface roughness

Response		Regression Equations	$R\text{-}_{Sq}$
delamination (UF_d)	=	1.1432 - 0.000018v + 0.1328f + 0.00081d	R = 0.9414%
delamination (WF_d)	=	1.1432 - 0.000018v + 0.1328f + 0.00081d	R = 0.9807%
Surface Roughness (USR)	=	3.609 - 0.000210v + 1.3f- 0.0226d	R = 0.9537%
Surface Roughness (WSR)	=	4.053 + 0.000004v+ 1.359f- 0.0021d	R = 0.7576%

4.4.2. *Validation of regression model*

The predicated or theoretical values of various parameters obtained using regression equations are shown in Table 4.15. The comparison plot for experimental and predicted values of unidirectional and woven delamination factors and unidirectional and woven surface roughness of L9 drilled enset/sisal fiber reinforced polyester composites are shown in the Figure 4.23 (a-d).

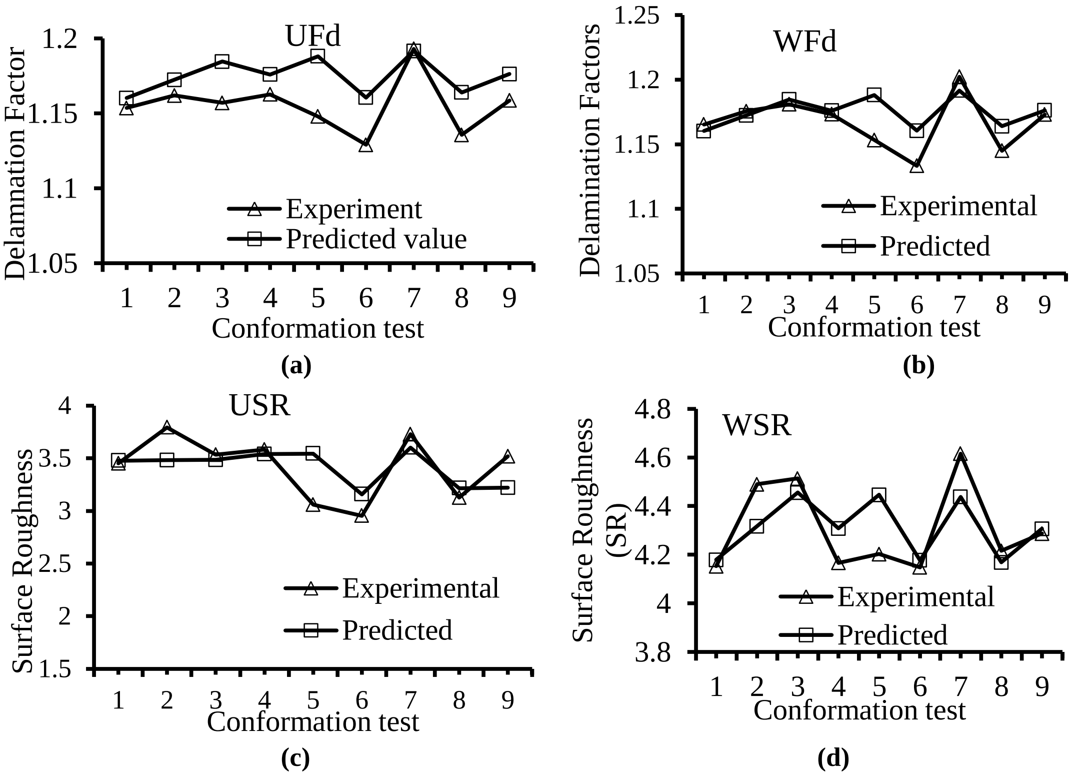


Figure 4.23. Comparation between experimental results and predicted for (a) UFd (b) WFd (c) USR and (d) WSR

The average absolute percentage of error for UFd, WFd, USR and WSR factors of enset/sisal fiber reinforced polyester composites were tabulated on ANOVA Table (4.19-4.22) are 5.86, 1.93, 4.63 and 24.24 respectively. The low values of error percentages witnessed a close prediction of the Taguchi analysis. For drilled hole delamination factors of carbon fiber reinforced polymer composite was obtained the same trend (Krishnaraj et al., 2012). The predicted value mostly above the experimental result (Venkateshwaran et al., 2012). Similar result was obtained in this study as shown in Figure 4.23 (a-d). This work will be useful for industries while the selection of process parameters in the drilling of natural fiber reinforced composite materials, to improve the quality of the drilled holes by reducing the delamination and surface roughness.

4.4.3. Chips formation morphology and characterization

Chips are generated immediately when the composite materials undergo a plastic deformation, generally at the drill-material slipping interface within a shearing region. During the chip formation, neither a discontinues nor a very long continuous chips is encouraged during drilling operations. The discontinues chips in enset/sisal hybrid composite are not easily evacuated from the drill flutes, causing a chip clogging due to high friction and forces developed, but in continuous chips often results into a serious chip evacuation predicaments (Oluwarotimi et al., 2016). These problems have a high propensity of causing decrease in drilled holes surface, damage around hole and drill breakage, if they are not properly managed. The chips formation increased during drilling of both samples at an increased feed rate and cutting speed, which was well prominent at 1200rpm and 0.1m/min feed and 9mm drill diameter respectively. Continuous chips formed during the drilling of low speed, low feed and moderate drill diameter. While the speed and feed increase the chip formed become discontinues due to the properties of their fibers and matrices. The higher the feed rate and cutting speed, the wider, longer, more ribbon-like in enset/sisal hybrid composite as shown in Figure 4.24.

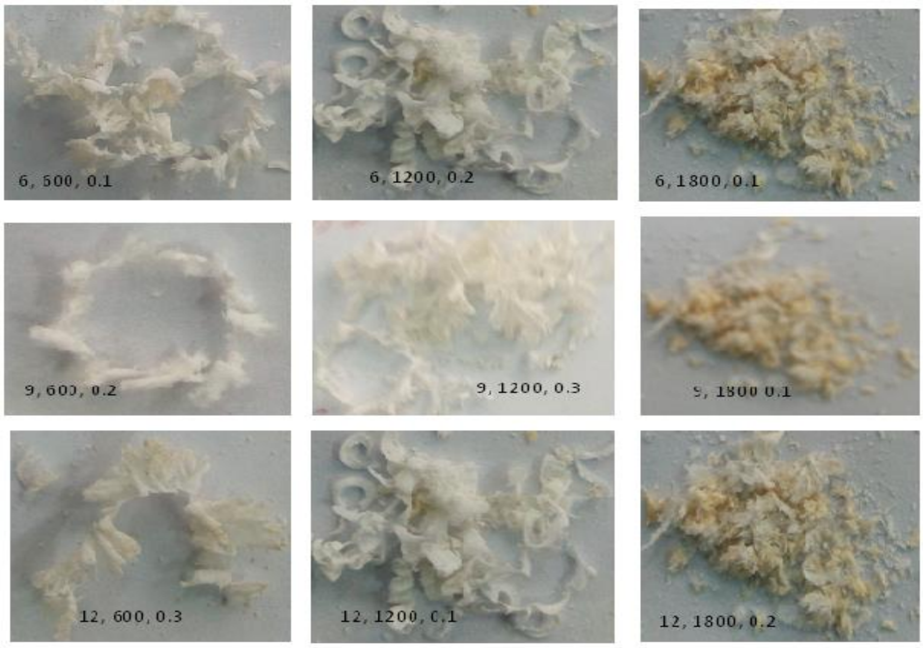


Figure 4.24. Photograph of chip type as per L9 orthogonal array

These types of chips formation produced a lower surface roughness and samples, delamination in the enset/sisal hybrid composite samples. Conclusively, the type of chips formation also affected the drilled surface quality and damage on both samples and the drill bit used and close result was achieved for both samples of composites. This chip was produced according to material properties and the operating condition used for produced the drilled holes. This also used for the optimization of the operating conditions. The drilling operations are performed, and material is prepared, so the operating conditions may be optimized easily. In HSS tool drill bit, continuous and discontinuous chip formation was produced for minimum cutting forces and tool wear due to low cutting speed and high feed rate which similar to (Jayabal & Natarajan, 2011).

Finally, the summaries of the result of subtopic 4.4. shows that visual and microscopic analyses of the drilled hole cut sections showed that the higher cutting speed together with higher feed rate should be avoided because it gives a poor surface finish. The ANOVA result showed that feed rate and speed are the most significant influencing factors of the delamination and feed rate, and drill diameter is the most significant factor for surface finish of both unidirectional and woven types of enset/sisal fiber hybrid composite. Regression analysis of data shown that feed rate is the most influential control parameter that affects the surface roughness and delamination. Lastly, based on the ANOVA and other statistical results obtained, the unidirectional enset/sisal hybrid polyester composite sample has a lower surface roughness (better surface finish) and lower delamination drilling-induced damage. Both samples are considered and analyzed within the same drilling conditions and parameters. Hence, the choice of their engineering applications should depend on their responses to this damage.

4.5. Tribological behavior of E/S hybrid polyester composite

The properties of hybrid composites governed by the fiber length, orientation, fiber/matrix interfacial bonding, fiber content, extent of intermingling of fibers, and arrangement of both of the fibers. Several researchers developed hybrid composite by combining natural fibers with polymeric matrices (Saba et al., 2015).Microstructure study of the wear tracks are carried out to analyze the wear mechanism that the composites undergo during tribological testing (Ghosh et al., 2012). Tribology is the study of friction, wear, and lubrication between two rotating elements. Tribology being the vigorous criteria for material selection in industry and stringent environmental guidelines. Tribological behaviors are the main cause for mechanical failure leads to heavy cost of the component parts in the industries (Bajpai et al., 2013). Natural fibers have been found to be beneficial for their mechanical properties and the wear resistance of polymers because, NFPCs in an attempt to investigate the potential of applications concerning wear and friction. The main objectives of designing good tribological properties of NFPCs are to reduce the wear volume loss or wear rate and coefficient of friction. The orientation of natural fiber in the composite also influences the tribological behavior as seen in the traditional composites (Chin & Yousif, 2009).The wear rate was calculated by taking the weight difference before and after test of sample. Many researchers calculate the wear rate by using weight loss methods (El-Tayeb, 2008; Goriparthi et al., 2012; Hashmi et al., 2006).

This topic addresses the fourth specific objective of the research and part of the work has been presented in a national conference Paper -V (not appended) and covers results for the:

- The influence of time and friction force on the woven and unidirectional composite of 5% NaOH treated enset/sisal hybrid composites.
- The effects of the applied load and coefficient of friction force on the woven and unidirectional composite of 5% NaOH treated enset/sisal hybrid composites.
- The influence weight loss and applied load on the woven and unidirectional of 5% NaOH treated enset/sisal hybrid composites.
- Study the wearied surface of the using woven and unidirectional composite the woven and unidirectional composite of 5% NaOH treated enset/sisal hybrid composites.

4.5.1. Friction force of enset/sisal hybrid composite

The experimental set up was tabulated in Table 4.24 show the factors to be studied.

Table 4. 24 Parameters of the specimens and sliding conditions.

Parameters	Levels		
	1	2	3
Load (N)	10	20	30
Sliding speed (rpm)	0.10	0.20	0.3

As shown by the results the friction force increased in all cases. Friction force and hence coefficient of friction depends on interaction between composite rubbing surface and counterface. It has been established that adhesion and deformation are prevailing mechanism governing friction. Deformation is dominating on rough surface while adhesion has its impact on smooth surface. In our case rubbing surface of specimen composite is smooth after sliding of some times. This was due to accumulation of thin film of polymer on rubbing surface of composite. This deposition of polymer film was accountable for reducing coefficient of friction after some time of rubbing. Also, asperities on the sliding surface of composites deform during sliding which reduces coefficient of friction and Fluctuations in coefficient of friction may be attributed to the fact that deposited polymer film on composite sliding surface breaks and transfers to the counter surface. The result was similar with (Chaudhary et al., 2018a). Each test was repeated for three times and the average of the measurements was determined at given load with speed tabulated in Table 4.25. The friction force was then recorded periodically after every 150 s during the tribological-testing for unidirectional and woven types of composites specimen.

Table 4. 25 Coefficient of friction force with respect to time (sec.)

time	10 N at 0.5m/s		20 N at 1m/s		30 N at 1.5 m/s	
(sec.)	Unidirectional	Woven	Unidirectional	Woven	Unidirectional	Woven
0	-0.001	0.002	-0.0001	0.0002	0.000433	0.005467
150	0.4975	0.225	0.4542	0.59825	0.367433	0.330833
300	0.7117	0.3505	0.66625	0.6065	0.537867	0.685067
450	0.8052	0.5076	0.78615	0.63925	0.6025	0.656533
600	0.8955	0.6565	0.85725	0.6765	0.659533	0.424633
750	0.9527	0.7205	0.93825	0.70125	0.710833	0.764
900	0.9985	0.7975	0.9788	0.76065	0.734333	0.8231

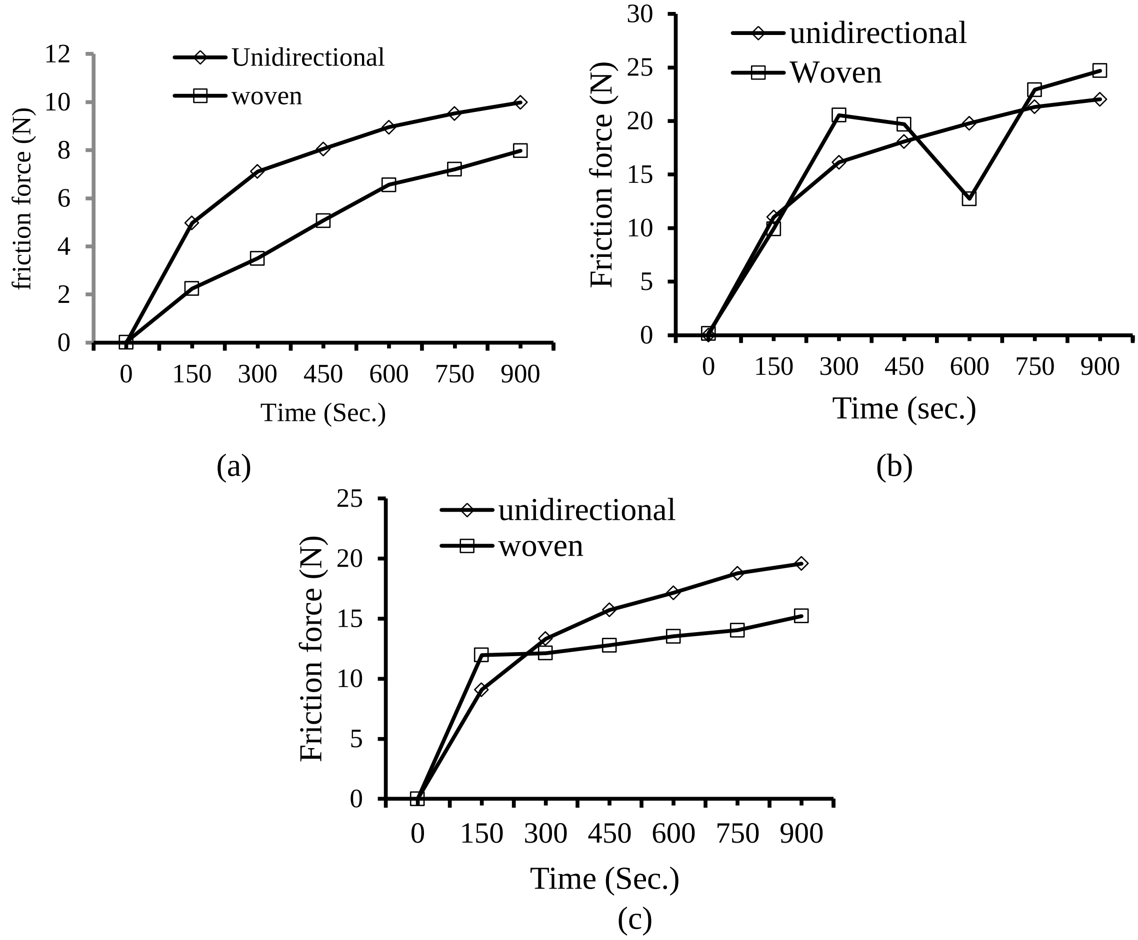
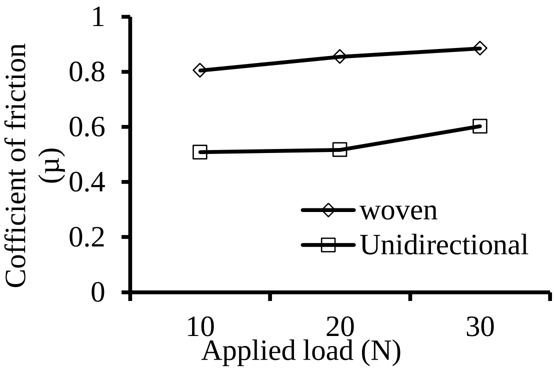


Figure 4.25. Friction forces versus time for unidirectional and woven composite at (a) 10 N, (b) 20 N and (c) 30 N load

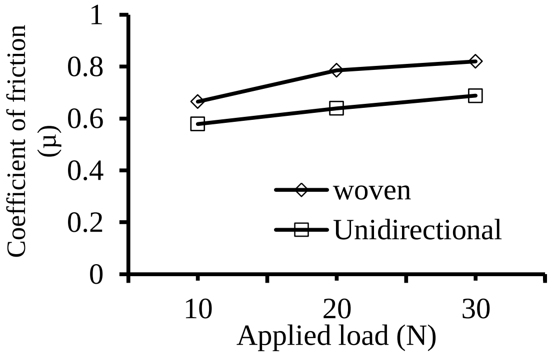
As can be observed from the coefficient of friction results shown in Figures 4.25 (a-c), it has been found that coefficient of friction decreases as applied load decrease for both types of composites. At minimum load the coefficient of friction also minimum for as the load applied increase the friction force also increase for both types of composites. The result similar with (Bajpai et al., 2013). Nature of coefficient friction was (increase, decrease or constant) depends upon the interfacial of polymer and nature of fibers at the interface. Initially, at low load, increased value of friction coefficient is due to mechanical interlocking of at the interface. As the load increases, polymer remains deform plastically and a thin layer of polymer adhere to the composite interface covering the fiber cross-section.

4.5.2. *Coefficient of friction in enset/sisal hybrid composite*

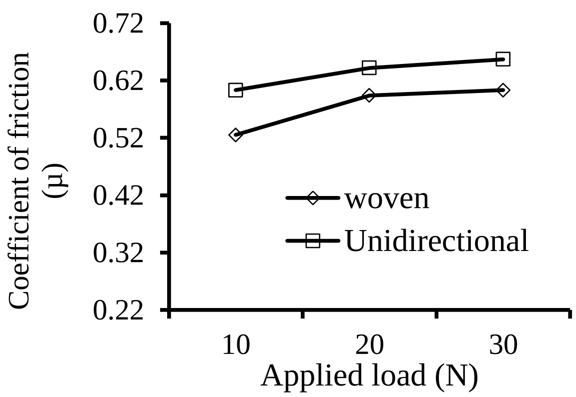
The orientation of natural fiber in the composite also influences the tribological behavior as observed in the traditional composites studied the influence of fiber orientation on wear behavior of sisal fiber reinforced epoxy composites. The variation of coefficient of friction with fiber orientation is shown in Figure 4.26 (a-c). Compared with asbestos and mineral/steel fiber, sisal/enset hybrid reinforced friction composites show better performance with respect to coefficient of friction at different load conditions. The coefficient of friction of woven fiber orientation composite less than unidirectional types of fiber composite. In general, observing the friction coefficient results in has been found that friction coefficient increased as applied load increased for both types of composites. The friction coefficient of woven types of composites was less in all case of speeds (0.5, 1, and 1.5) m/s. For woven composites, a friction coefficient was observed the same result as the load is increase upto 20 N increase the load further little amount increase. At 1.5 m/s speeds, variation of friction coefficient at 20N both unidirectional and woven nearly similar values and when the load – increased beyond 20 N almost insignificant. For all the composite showed almost same friction coefficient at 10 N applied load for 0.5, 1 and 1.5 m/s speeds and then followed increased trend as applied load increased up to 20N and almost constant value of coefficient of frictions.



(a)



(b)



(c)

Figure 4.26.Coefficient of friction versus applied load for E/S hybrid unidirectional and woven types of composites at (a) 0.5 m/s, (b)1 m/s and (c)1.5 m/s speed.

4.5.3. Weight loss of enset/sisal hybrid composite

Woven types of composite observed weight loss than unidirectional composite in all types of loads applied as shown in Figure 4.27 (a-c). At lower load i.e., 0.5 m/s the weight loss was maximum and minimum weight loss at the lower and higher load. As the sliding speed increase the weight loss of the sample was also increase. The result of our work in weight loss similar with the 0.039 gram (weight loss) (Goriparthi et al., 2012) 0.014 gram (weight loss) (Hashmi et al., 2006), 0.098, 0.06 g (weight loss) (El-Tayeb, 2008).

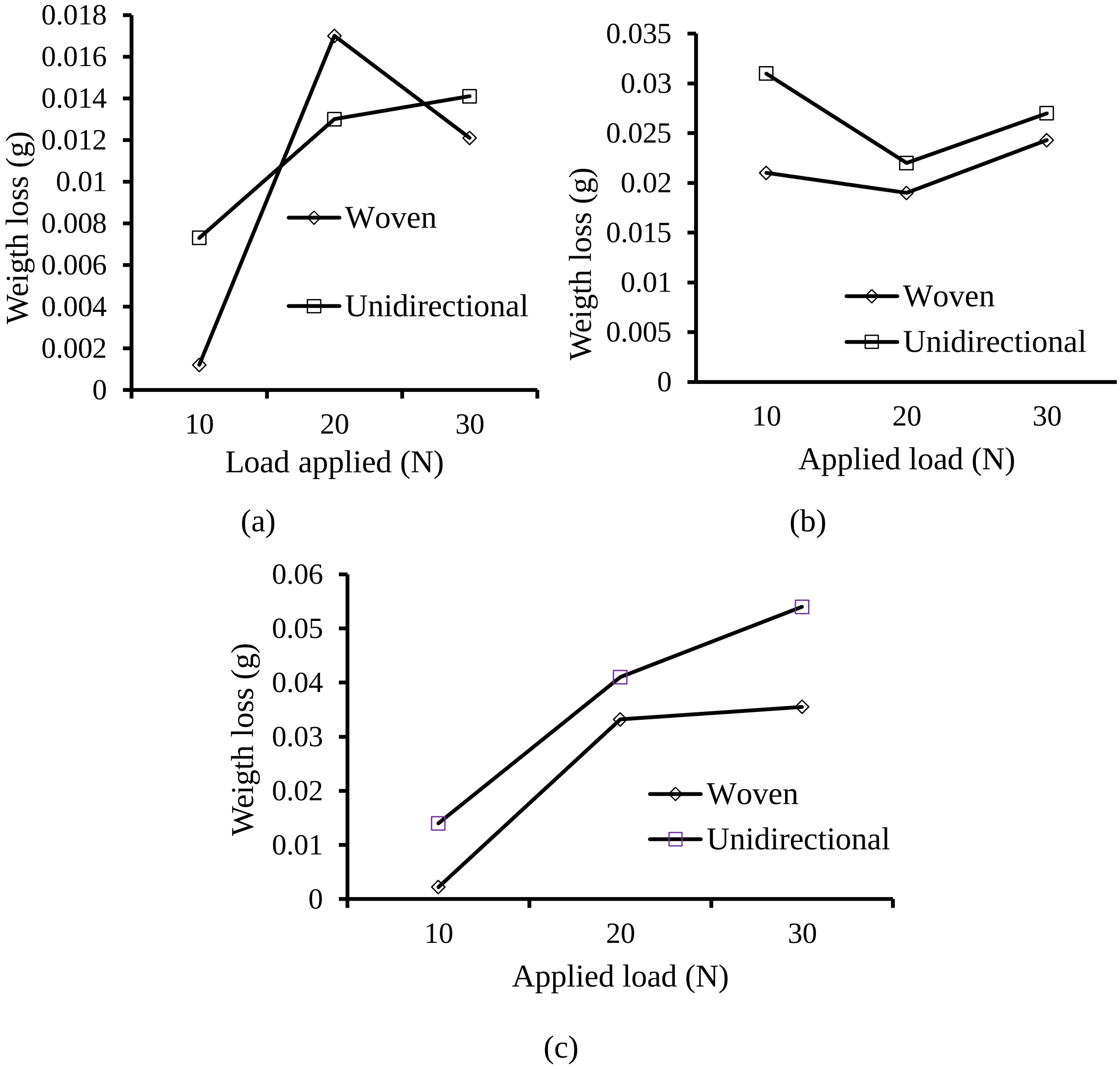
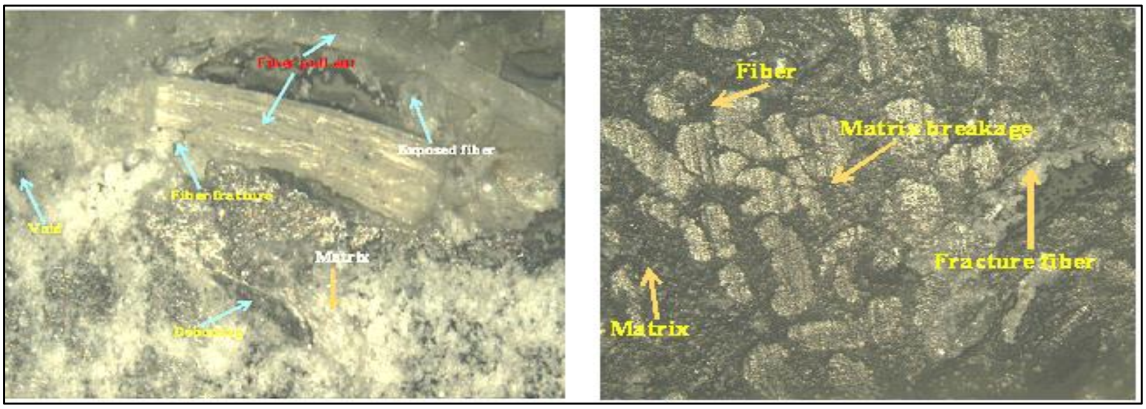
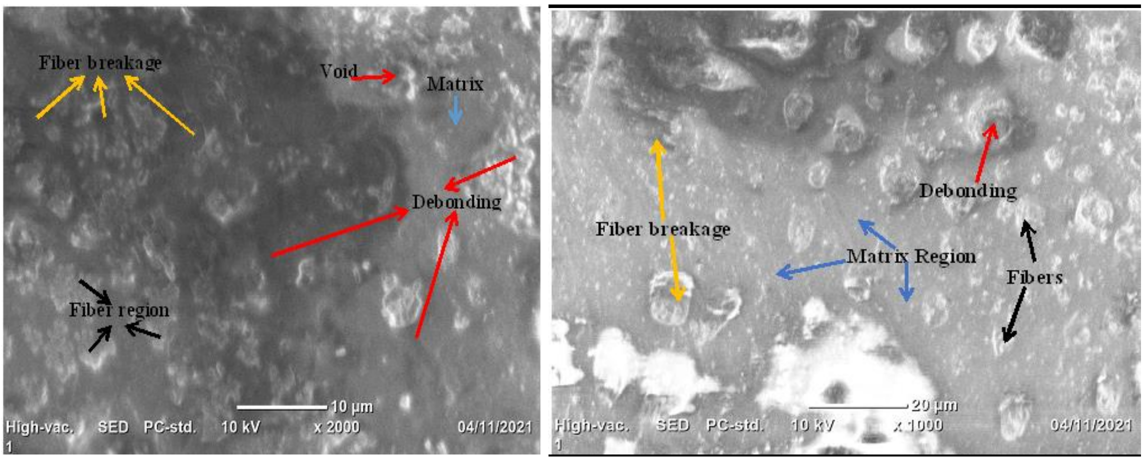


Figure 4.27. Weight loss versus applied load for E/S hybrid unidirectional and woven types of composites at (a) 0.5m/s, (b)1m/s and (c)1.5m/s speed.



(a) (b)

Figure 4.28. 3D microscopy image of (a) Unidirectional composite (b) Woven composite at 1 m/s speed and 20 N applied load.



(a) (b)

Figure 4.29. SEM micrograph image of (a) Unidirectional composite (b) Woven composite at 1 m/s speed and 20 N applied load.

At medium load (20N) and medium sliding speed (1 m/s) are shown on Figure 4.28 (a) and (b) are examined for unidirectional and woven types of samples composites using 3D micro profilometer and SEM instrument respectively. As shown in Figure 4.29(a) and (b) the material removal from the composite surface and hence lower weight loss. During SEM observation of enset/sisal polyester composite after wear testing, it was observed that debonding, fiber fracture and voids were predominately present in both types of composite orientation but fiber ends were still in good condition, protecting the resinous regions (Bajpai et al., 2013). Debonding is greatly observed in all the unidirectional specimens. As shown on Figure. 4.5.4 (a) shows that the higher fiber pull out, fiber breakage, debonding and void are exist in the composite. The SEM micrographs of woven types of composite with the same parameter show that the fibers are still in good condition and no detachment and fiber pull out was observed, this indicates that better bonding between fiber matrix leads to high load carrying capacity of the composite than unidirectional enset/sisal hybrid composite. The dissimilar wear behavior of two composites with fiber orientation can be due to the fiber matrix interfacial.

Finally, summarized result of subtopic 4.5. Shows that, Influence of fiber orientation unidirectional and woven) types of composites, for various loads and speed on friction and weight loss are studied. Natural fiber reinforced composites can be effectively used as friction materials in the brake applications. Natural fibers improve tribological performance and hence can be used for friction materials. Making hybrid of natural fibers used further to improve the wear properties of composite. Natural composites can also be used as anti-friction materials. The wear performance of developed composites more sensitive to the load variation than sliding speed. The experimental endeavor establishes the woven types of enset/sisal fiber orientation better than unidirectional types of composites for tribological applications. Debonding was significantly observed in 3D optical profilometer micrographs of composite specimens after sliding wear testing. The SEM images also showed plowing in the resinous region confirming the presence of abrasive type of wear mechanism.

4.6. *Promising applications of enset/sisal hybrid composites*

In comparison to other hybrid fiber reinforced polymer composites, the 50/50 volume ratio with 5% alkali treated enset/sisal hybrid composites have comparable, acceptable, and satisfactory mechanical and physical, machinability, and tribological properties that govern some promising and potential applications with the advantages of the environment, renewable resources, availability, and sustainability. Automotive, construction, aerospace, packaging, furniture, shipping pallets, electronic, medical, sporting goods, etc. are some of the most likely industrial applications. Unusually, the enset/sisal fiber hybrid composite with 5% alkali treatment can be utilized to make truck linens, front and back door linens, dashboards, parcel shelves, seat backs, backrests, and cabin linings for automobiles. For partition, window frame, and moulded panel components in the building industry with low maintenance costs. It can also be used in non-structural applications like the furniture industry (tables, chairs, etc.), the packaging industry (egg shelves), and sports equipment (helmet, racket handle, bicycle frames etc.). The use of composite materials made from natural fibers was encouraged by concerns about cost, weight, and the environment.

CHAPTER-FIVE

5. Conclusions and Recommendations for Future Work

5.1. Conclusions

Natural fibers that were collected from the pseudostem enset and leaves of sisal plants were characterized. For enset and sisal fibers with such conditions (untreated, 5% and 10% treated NaOH solution), the effects of NaOH treatment on physical, mechanical, and chemical properties were examined. On the mechanical (tensile, flexural, and impact) and physical (water absorption) properties of the enset/sisal hybrid composite, the effects of fiber volume ratio (100/0, 75/25, 50/50, 25/75, and 0/100 E/S), treatments (untreated, 5% and 10% treated NaOH), and fiber orientations (unidirectional and woven) were investigated. For both the unidirectional and the woven enset/sisal hybrid polyester composites, the ideal value was found at 5% NaOH treatment and a 50/50 volume ratio. After obtaining the ideal value, the composite's tribological behavior and machinability during drilling operations were examined.

The results of this investigation allowed us to generally reach the following conclusions:

1. The physical, chemical and mechanical properties of enset and the sisal fiber were improved significantly after alkali treatment.
2. Fiber lignin contents affect the interfacial between fiber and polymers. FTIR spectroscopy data also indicates that there is a reduction of impurities (hemicellulose and lignin) with 5% and 10% NaOH.
3. The adequate mechanical properties and minimum water absorption was obtained at 50/50 volume of E/S hybrid polyester composite.
4. The mechanical properties (tensile, flexural) and impact strength of E/S fiber hybrid polyester composites were improved by increasing the sisal volume and enset volume ratio.
5. The water absorption and mechanical properties of E/S fiber hybrid polyester composites were improved significantly after alkali treatment. 5% NaOH treatment has minimum water absorption and optimum mechanical properties than untreated and 10% NaOH treated E/S fibers hybrid composites.
6. Woven types of fiber orientation gave better mechanical properties than unidirectional fiber orientation in both untreated and treated hybrid composites.

7. Highest tensile and flexural strength was obtained for 5%NaOH in both unidirectional and woven types of fiber orientation composites when compared with untreated and 10% NaOH treated composites.
8. The failure mode analysis was done using SEM and better surface interference was observed in 5% alkali treated samples that approved the result of mechanical properties and fiber matrix interfacial bonding of E/S hybrid composite.
9. Based on the ANOVA statistical results, lower surface roughness (better surface finish) and delamination factors were observed in unidirectional samples than those in woven enset/sisal hybrid polyester composites.
10. Regression analysis of data has shown that feed rate is the most influential control parameter that affects the surface roughness and delamination.
11. The tribological performance of developed composites was observed to be more sensitive to the load variation than sliding speed.
12. The experimental endeavor establishes the woven types of enset/sisal fiber orientation better than unidirectional types of composites for tribological applications.

5.2. Recommendations for future work

Though 5%NaOH treated E/S hybrid reinforced polyester composite achieved better mechanical properties result in this study, more future works can be done focusing on other properties (such as degradability, thermal, electrical and fracture), and other parameters (such as fiber length, fiber random orientation and sliding distance) using both numerical approach and validation with experimental research work. The future works can include:

1. To get uniform diameter of fibers throughout the length, fibers extraction methods required better extraction technology.
2. In this study, fibers were treated with only alkali as a means of improving fiber/matrix interfacial strength. Further investigation could be carried out using different or hybridizing chemical to improve the compatibility between the fibers and the polyester.
3. Unsaturated polyester is very brittle; thus plasticizers could be used to improve the toughness of the unsaturated polyester based composites.
4. The enset and sisal nonwoven mats and unsaturated polyester are commercial products/formulations; however, the technology of making the fibers woven need improvement.

5. Polyester curing formation could be further optimized in hardener types, water content, so as to improve the material performance for structural applications and environmental conditioning.
6. Conducting feasibility study to manufacture a complex geometry product from unidirectional and woven types of the composites.
7. Building proper material-level (mainly mechanical and environmental degradation properties) models for the enset /sisal reinforced hybrid polyester composites.
8. Since natural composites, especially for enset/sisal hybrid composite, behave differently from normal synthetic hybrid composites, for the best validation of a modified model/theory is needed. Such models can be used to product properly estimate the performance of real products and to further accelerate the whole progress of research work on this topic.

5.3. Scientific contribution of the study

Several studies have been conducted on natural fiber hybrid composite in order to obtain the better properties, maximizing the applications and producing the environmental friendly materials. This study aims to contribute further knowledge on properties characterization of enset and sisal fibers and their hybrid composites. Alkali treatment (NaOH), volume ratio and orientation are the main parameters considered to evaluate the mechanical, physical, machining and tribological properties.

As shown from the E/S hybrid composite result, the optimum alkali concentration, volume ratio and fiber orientation were developed. Thorough characterization has been accomplished, that can inspire for the potential applications, among others, in automotive, structural and non-structural manufacturing industries, especially for interior part applications. Existing knowledge indicates that use of natural fibers for automotive part applications reduces 20% cost and 30% weight of the automobiles. The result of newly developed E/S hybrid composite material leads to further savings in economical, energy and environment as well as support the green economy and sustainable development.

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Appended Articles