A RESEARCH ON THE USE OF ENSET WOVEN FABRIC STRUCTURES FOR THE APPLICATIONS OF SOUND ABSORPTION AND BIODEGRADABLE COMPOSITE MATERIAL DEVELOPMENT

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PhD THESIS
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ÖZET

Doktora Tezi

ENSET DOKUMA KUMAŞ YAPILARININ SES YUTUM VE BİYOBOZUNUR KOMPOZİT MALZEME GELİŞTİRİLMESİ UYGULAMALARINDA KULLANIMI ÜZERİNE BİR ARAŞTIRMA

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Tekstil endüstrilerindeki yeni gelişmeler, boyutsal stabilite, su emicilik, nefes alabilirlik gibi tekstil kumaşları ve bitim özelliklerinin performansını geliştirmektedir. Tekstil liflerinin yapıları ve özellikleri; kompozitlerin yanı sıra iplikler, kumaşlardan oluşan malzemelerin mekanik özellikleri üzerinde büyük bir etkiye sahiptir. Uzun süredir metaller, en çok tercih edilen yapı malzemeleri olarak kullanılmıştır. Bununla beraber; insanların hızla büyüyen talepleri, araştırmacıları yüksek spesifik mukavemet ve elastisite modülüne sahip yeni kompozit malzemelerin geliştirilmesine itmiştir. Petrokimya ürünleri, sadece çevre dostu olmayan ürünler olmayıp aynı zamanda üretim, kullanım ve atıkların yok edilmesi süresince ciddi sağlık problemlerini oluşturmaktadır. Son zamanlarda araştırmacılar ve üreticiler, yeşil kompozit uygulamalar için yaprak, gövde ve meyvelerden özütü elde edilen doğal lif takviyeli kompozit malzemeler üzerinde araştırmalarını odaklamıştır. Doğal lifler, doğada bol miktarda bulunur, hafif, düşük maliyetli ve konvansiyonel lifler içerisinde iyi mekanik mukavemete sahip olan yenilenebilir doğal malzemelerdir. Doğal kaynaklardan elde edilen takviye ve matriks elemanları olarak kullanılan sentetik lifler ve reçinelerin yeri, kompozit malzeme sektörlerindeki ekonomiksel, sağlık sorunları ve çevresel problemleri önlemesine iyi alternatiftir. Bu doktora tezi çalışmasında, hafif nitelikli yapı uygulamaları için mekanik özelliklerinin geliştirilmesi ile yenilikçi tekstil kumaşları takviyeli yeşil kompozit malzemelerin karakterizasyonu ve araştırması üzerine odaklanılmıştır. Tekstil kumaşları, bu çalışmada takviye elemanları olarak kullanılmıştır. Yeni biyoreçine elemanları, 75:25, 70:30, 65:35, 60:40, 55:45 and 50:50 gibi çeşitli oranlarla Akasya tortillas ve Bosveliya papirifera karışımıyla hazırlanmıştır. Dokuma kumaş konstrüksiyonu ve çok katlı kumaş takviyeli yeşil kompozit malzemelerin, mekanik ve akustik performansları çalışılmıştır. Ayrıca nano lifler, α-amilaz enzim ön terbiyesi ve mekanik öğütmeyle üretilmiştir. Bu nanolifler; yüksek boyutsal stabilite, spesifik mukavemet, daha geniş yüzey alanı ve biyobozunabilir ürünler gibi benzersiz özelliklere sahip daha gelişmiş tekstil yapılarının üretilebilmesi için kullanılacaktır.

Anahtar Kelimeler: Akustik, biyobozunur, biyo reçine, enset nanolif, mekanik testler
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ABSTRACT

PhD Thesis

A RESEARCH ON THE USE OF ENSET WOVEN FABRIC STRUCTURES FOR THE APPLICATIONS OF SOUND ABSORPTION AND BIODEGRADABLE COMPOSITE MATERIAL DEVELOPMENT

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The rapid developments of technology in textile industries have been improving the performance of textile fabrics and finishing properties such as durability, water replants and breathability. The natures and properties of textile fibers have a major impact on the physical and mechanical properties of materials made from them such as yarns, fabrics as well as composites. For a long period of times, metals have been used as the most preferred structural materials. However the rapid growing and unlimited demands of human being have pushed researchers to innovate new materials called composite materials, having high specific strength and stiffness. Petrochemical based composite materials are not only non-ecofriendly products but also they bring a serious health problems during their manufacturing, usage and waste disposals. Recently, researchers and manufacturers have focused on natural fiber reinforced materials obtained from leaf, bast and fruit for green composite applications. Natural fibers are abundantly available, light weight, low cost and renewable with good mechanical strength. Substituting of commercially used synthetic fibers and resins by naturally existing resources as a reinforcing material and matrix are the best alternative to overcome economic, health hazard and environmental problems in composite manufacturing sectors. This Ph.D dissertation focuses on the investigation and characterization of novel textile fabric reinforced green composite materials and enhancing their mechanical properties for light weight structural as well as sound absorption applications. Enset woven fabrics were used as reinforcing materials for this study. A new bio resin material was prepared by mixing separately prepared acacia tortillas and frankincensepapyrifera bio resins at different ratios such as 75:25, 70:30, 65:35, 60:40, 55:45 and 50:50. Mechanical and sound absorption performance of enset fabric reinforced bio composites was studied with special reference to bio resin preparation ratio and number of fabric layer. Also, enset nano fibers were manufactured by α-amylase enzyme treatment and mechanical hammering using enset fibers and enset fabrics. These nano fibers would be used to produce more advanced textile structures having unique properties such as higher weight to strength ratio, large surface area and bio-degrability.

Key words: Acoustic, biodegradable, bio resin, enset nanofiber, mechanical strength
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SYMBOLS and ABBREVIATIONS

Symbols	Definition
^0C	Degree Celsius
^{0} F	Degrees Fahrenheit
%	Percentage
σ	Flexural Strength
α	Temperature Coefficient Resistance
μ	Viscosity
Al	Aluminum
b	Sample Width
cm	Centimeter
Co	Cobalt
E	Young's Modulus
gm	Gram
Gpa	Giga Pascal
g/cc (g/cm^3)	Gram per centimeter cubic
J/m	Joule per meter
Kg	Kilogram
KHz	Kilohertz
Ksi	Kilo-pound per square inch
kg/m^3	Kilogram per cubic meter
KJ/m^2	Kilojoule/square meter
l/d	Length per diameter
Mg	Magnesium
Mpa	Mega Pascal
m	Meter
mm	Millimeter
μm	Micro meter
nm	Nanometer
Ni	Nickel
P	Maximum test load
S	Dimension between load points
Sec	Second
t	Thickness
w/v	Weight per Volume

Abbreviation	Definition
2D	Two Dimensional
3D	Three Dimensional
AF	Aramid fibers
ASTM	American Society for Testing and Materials
BET	Brunauer–Emmett–Teller
BF	Basalt fibers

BUTAL	Bursa Test and Analysis Laboratory
BUU	Bursa Uludag University
CCM	Ceramic Composite Materials
CeF	Cellulose-based Fibers
CF	Carbon fibers,
CMC	Ceramic Matrix Composite
ENF	Enset Nano Fiber
FTIR	Fourier-Transform Infrared Spectroscopy
GF	Glass fibers
ISO	International Organization for Standardization
LDPE	Low Density Poly Ethylene
Max.	Maximum
MCM	Metallic Composite Materials
MMC	Metal Matrix Composite
MW	Molecular Weight
NASA	National Aeronautics and Space Administration
PAN-F	Polyacrylonitrile fibers
PC	Polycarbonate
PCM	Polymeric Composite Materials
PET-F	Polyethylene Terephthalate Fibers
PHA	Poly Hydroxyl Alkanoates
PLA	Polylactic Acid
PLLA	Poly-L-Lactic Acid
PMC	Polymer Matrix Composite Materials
PP	Polypropylene
PP-F	Polypropylene Fibers
RRIM	Reinforced Reaction Injection Molding
RT	Room Temperature
SEM	Scanning Electron Microscopy
SWR	Standing Wave Ratio
Temp	Temperature
TGA	Thermo Gravimetric Analysis

FIGURES

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1. INTRODUCTION

The metallic materials innovated approximately 5000 B.C are the most preferred structural materials in most industrial and engineering applications (National Research Council 1975, Sezgin 2018). In today's world, the drastically growing demand of new materials having low cost, abundantly available and high strength-to-weight ratio has attracted the researchers and manufacturers to realize new resources by mixing two or more existing materials, the so called composite (Hummel 2005, Pastuszak and Muc 2013). Around 3000 years ago, ancient Egyptians is considered to be producing the first composite material (Hummel 2005, Bhatt et al. 2017). It was formed from clay based materials and was used in construction sector. Composite is one of the most preferred materials as re-innovative product for their novel properties compared to commercially used materials such as metals and woods (Pastuszak and Muc 2013). Moreover, composites are becoming one of the critical structural materials which are being progressively improved their performance as well as functional properties (Hummel 2005, Pastuszak and Muc 2013, Sezgin 2018).

Commercially used reinforcements and matrices materials are obtained mostly from petrochemical products and mostly are not composted or degraded under standard ecological conditions for a long period of time (Zweben 2001, Mann and Singh 2018). Composite materials made from thermosetting resin materials might not be recycled or reprocessed. Conversely, a minor portion of these thermosetting composite materials has been crushed into small size particles, powder and dust form (Zweben 2001). Recently, the rapid growth of environment issues and economic concerns as well as finite nature of petrochemical resources have caused the rapid growth in the field of bio based polymers in the research centers and composite manufacturing industries (Mann and Singh 2018). The development of green composite materials that can be competitively replacing (economically) petroleum based polymer materials were becoming an attractive research area. Since 1960s, textiles such as fiber, yarn and/or fabric reinforced composites have been used in various engineering and industrial applications, revealing with abundantly available, higher strength-to-weight ratio, better fatigue performance and higher energy storage (Mann and Singh 2018, Sezgin 2018). Textile materials used as reinforcing materials in composite structures have contributed a significant share in all

type of composite structural materials (Khatkar et al. 2020). From the large family of textile materials: fiber, yarn, two dimensional (2D) and three dimensional (3D) fabrics are becoming the most series interesting field of study in green composite areas.

Green composite structures that have been derived from renewable resources bring very promising potential and provide benefits to manufacturer, environment and ecological conservation by decreasing the consumption of petrochemical resources (Mochane et al. 2019). The shift to more sustainable product fabrication for manufacturers are not only an initiative towards a more viable environment and cost efficiency but also a demand of European and most of the world's countries regulation (Zweben 2001). Development of advanced green composite materials having superior mechanical properties opened up new horizons in the engineering and material science.

Most of the composite materials at these times have used plant fibers as a reinforcing material to manufacture bio composite structures (Khatkar et al. 2020). Natural fiber reinforced composite structures (sometimes called bio-composites) are becoming a viable alternative materials to petrochemical and mineral fibers reinforced composites, especially in light weight engineered materials (Gholampour and Ozbakkaloglu 2020). The most attracting futures of natural fiber over petrochemical and mineral fibers are: abundantly availability, cheap, light weight, competitive specific mechanical strength, biodegradability and lower energy consumption. Also, natural fibers offer a possibility to developing countries to use their own natural resources in their processing industries and composite manufacturing sectors (Gholampour and Ozbakkaloglu 2020). Natural fibers, which traditionally were used as reinforcement for thermosets matrices, are becoming one of the fast developing alternative reinforcing materials for thermoplastic matrices. Bio based composite materials are dynamic and versatile field in which the bio polymers have been reached its final stages range from research level, initial market adaption and long term established performance.

Since the 1930's, it was observed from the previous studies on the bio-matrices based green composite made from vegetable oils like rape seed, soya, sun flower and linseed that they have been limited application area due to their inferior mechanical properties

and expensive production methods (Mochane et al. 2019). However, developments and innovation of new manufacturing technologies in research centers and composite manufacturing industries over the last few years have led to a number of promising technical improvements to substitute crude oil and petro-chemicals materials. Unfortunately, much of the research is still on going and it will take years even decades to produce a green composite materials with affordable prices (Mitra 2014, Koronis 2016).

In this dissertation work, renewable natural fibers such as enset fibers were composed with different natural gums like Acacia Tortilis (in Amharic language called girar mucha) and Frankincense (in Amharic language called etan mucha) in an attempt to prepare a new bio resin and high performance green composite materials. *Ensete Ventricosum* is the most drought tolerance and new alternative textile fiber, mostly grown in Ethiopia (Teli and Terega 2017). Enset fiber is an un-utilized agro waste fibers obtained from the pseudo stem (bast) and mid ribs of enset plant, which are morphologically resemblance with banana. Its abundantly availability, low cost, light weight and good specific strength are the major attractive characteristics of the fiber for potential application in technical textiles, especially lightweight green composite structures, geotextile as well as in packaging industries (Teli and Terega 2017). The reuse of agro waste fibers as a reinforcing material for bio degradable composite materials is a sustainable option for the global warming and environment concerns.

The primary aim of this thesis was to focuses on investigating and characterization of biodegradable composite material by using enset fibers (fabrics) as reinforcement and different gums obtained from plant secretion as bio-resin materials and also, fabrication of enset nano fiber (ENF) as nano materials, which are not manufactured and used before. In this perspective; this thesis was to seek out the possible solutions to enhancing the mechanical properties of textile fabric reinforced green composites for the lightweight industries. In order to achieve these objectives, a new bio resin was prepared by mixing acacia tortillas and frankincense with 6 different ratios such as 75:25, 70:30, 65:35, 60:40 55:45 and 50:50 and hand layup and spray up resin transfer technique was preferred as composite manufacturing method. Moreover, the physical and mechanical properties of

the prepared bio resin materials and textile reinforcement materials (enset fiber and enset fabric) were investigated separately and compared with those of most commercially used materials in composite industries. Furthermore, the effects of textile fabrics and prepared bio resins on the performance of green composites were studied by different test methods such as acoustic, tensile, flexural and impact strength tests. Also, this research works could contribute to development of new bio resins, enhancement of novel nanofiber fabrication, the reduction of petrochemical consumption, relative reduction of fossil fuel import dependence having a significant effect on greenhouse gas emission, reduction of harmful solid waste deposition, increase employment in agriculture sector and generate new income for poor farmers.

2. THEORETICAL BASICS and LITERATURE REVIEW

2.1. Introduction to Composite Material

The rapid growths of innovative manufacturing techniques have been improved the growth of materials and materials science, which are the basic inputs of industry (Hummel 2005). However, due to the inadequate nature of resources such as metal, polymer, ceramics etc., the materials and their properties could not stand with the development of technology (Hummel 2005, Nagavally 2017). Researchers have chosen the way to manufacture materials that are cost effective and appropriate to encounter the criteria of today's human needs in parallel with the growth of consistence manufacturing systems and innovation (Sapuan and Maleque 2005, Agarwal et al. 2014, Elanchezhian 2014, Raghavendra et al. 2015). Therefore, the innovation of new technology is intensifying on socio-economy and environmental benefits (Nagavally 2017, Mann and Singh 2018). Like this, the composite materials, which are formed by the combinations of two or more components with unique properties from its individual constituents are becoming more important in technical textile and most manufacturing sectors (Sapuan and Maleque 2005, Raghavendra et al. 2015). Traditionally used materials such as polymers, ceramics and metals have limitations of design flexibility. These limitations are overcome by combining two or more of them. In the material engineering and the growth of new technology, these materials play very significant roles on development of composite materials (Elanchezhian 2014, Nagavally 2017). Composite materials have unique characteristics in terms of their processing, functionality and structures as compared with commercially used materials like metal and wood products. Structural composite materials are manufactured for their mechanical performance while functional composite materials are fabricated for gaining the desired special function which does not exist in monolithic materials. Nowadays, researcher and manufacturers are trying to manufacture and commercialize these two properties in a single composite structures (Haruna et al. 2014). Composite materials are not new innovations. They exist naturally like wood and bone as well as fabricated by combing different materials from Paleolithic age (old stone age). Different scholars and book author's give different definitions for the term

composite material. Some of the definitions of composite material given by different scholars have been mentioned below in detail.

Composite materials are manufactured or naturally existing materials fabricated (made) by combining two or more components (Harris 1999, Nagavally 2017). The chemical and physical properties of the new material is different from their constituents (Campbell 2010, Al-Mosawi 2012, Hu 2012). The word composite is the composition of different monolithic materials to form single structural materials having a clear separate phase (interphase) and the properties are not found in any of the separate constituents. To say a material is composite, at least one the phase must be in solid form (Callister and Rethwisch 2006, Mallick 2007, Matthews and Rawlings 2009, Imanaka 2012). Composite materials are newly reinvented materials having surprising properties than the individual phases, obtained by combination of two or more materials. This combination helps to remove or minimize the limitations of traditionally used materials (Nagavally 2017). A composite material structure generally has 3 major parts; the primary phase is the matrix, which is used to attach the reinforcing materials, the secondary phase is reinforcement materials employed to give the desired mechanical strength and the third part is called interphase, which separates the matrix and reinforced material as shown in Figure 2.1 (Mallick 2007, Matthews and Rawlings 2009, Jose and George 2015).

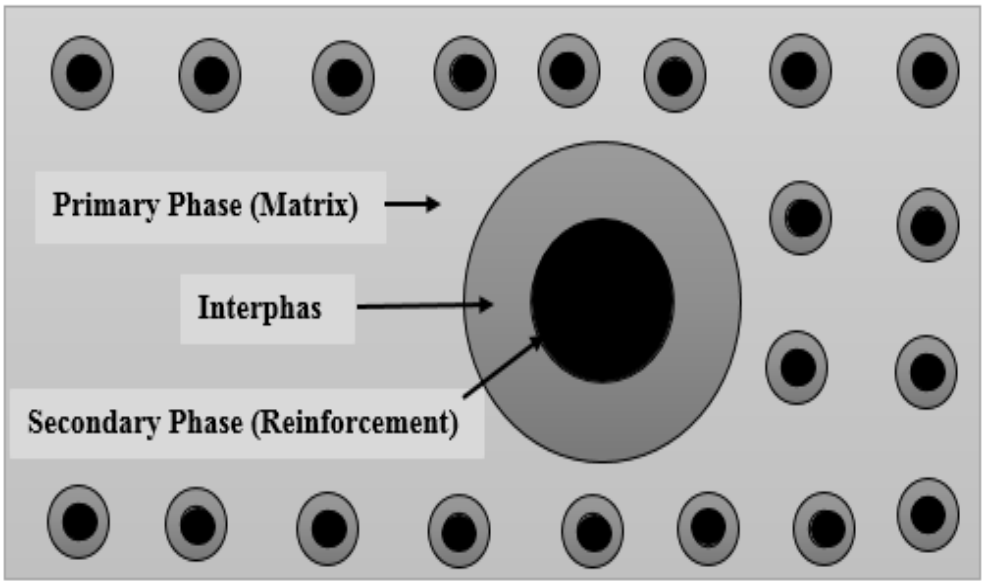


Figure 2.1. Basic constituents of composite material (Al-Mosawi 2012, Hu 2012)

The rapid development of composite materials and their manufacturing technologies have played a major role in the advancement of engineered biomaterials, which are used in our daily life (Harris 1999, Nagavally 2017). The significant reductions of weight of materials and design flexibility in composite materials have paradigm shift for automobile and aircraft industries. The reduction of weight of materials in transportation sectors plays a tangible reduction in the fuel consumption (Botelho and Silva 2006, Nagavally 2017).

2.1.1. Reinforcement material

Composite reinforcing materials provide the essential strength and stiffness to the composite structure (Dieringa and Kainer 2012). Different type and shape of reinforcing fibers are used in composite manufacturing. Based on their type and shapes, reinforcing phase can be classified in different ways. The mechanical performance and physical properties of the composite materials are very significantly affected by choosing the reinforcing material such as type, amount, geometry and distribution of reinforcement (Dieringa and Kainer 2012). The major types of composite reinforcing material forms are seen in Figure 2.2. Moreover, the composite reinforcing materials are also categorized based on their geometry (Botelho and Silva 2006, Matthews and Rawlings 2009, Dieringa and Kainer 2012, Imanaka 2012).

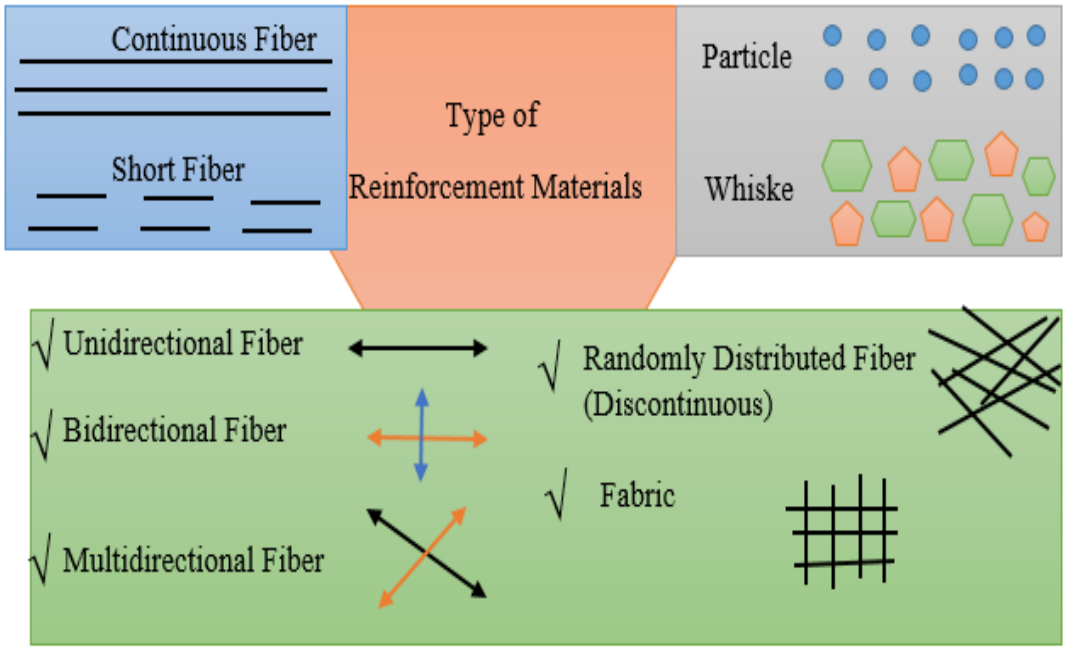


Figure 2.2. Classification of reinforcement material (Mria 2019)

Fiber Reinforcement: Fibers have been applicable as a composite reinforcing materials from a long period of time under different ways such as short or long fibers, continuous or discontinuous fibers, synthetic or natural fibers and so on (Maria 2019). They are reinforcing materials obtained in the form of either manmade or natural fiber. Fiber has longer length and very small diameter. Due to this, continuous fiber has higher aspect ratio than short fiber (length to diameter ratio, l/d) (Campbell 2010). Moreover, fibrous reinforced structure can be affected by the orientation of the fibers. Continuous fiber reinforced structures have preferred orientation while short fiber reinforced structures are randomly distributed (Imanaka 2012). Fibrous reinforced materials are predominantly used to enhance the mechanical properties of composite structures like strength, stiffness and reduced thermal expansion (Davoodi et al. 2010, Nagavally 2017).

Particle Reinforcement: Particles having any size, shape and configuration used as composite reinforcing materials are called particle reinforcement (Melby and Castro 1987, Dieringa and Kainer 2012). It may be a large particle, cermet (the combination of ceramics and metal), concrete as well as reinforced concrete. Particle materials used as a reinforcement mostly achieve to improve the strength, stiffness and toughness of composite structure (Dieringa and Kainer 2012). The mechanical and physical performance of the particle reinforced structure was significantly affected by type, size and shape of used particles (Davoodi et al. 2010, Dieringa and Kainer 2012). Particles used as composite reinforcing materials do not only enhance the mechanical performance of the composite structure but also used to improve the physicochemical properties of the materials like thermal resistance, electrical resistance, wear resistance, damping behavior, heat resistance, hardness etc. (Chen et al. 2020). Particle reinforced composite structure is less in strength and stiffer than fiber reinforced materials (Campbell 2010, Dieringa and Kainer 2012).

Skeletal Reinforcement: Skeletal reinforcement is a type of reinforcement in which the resin and reinforcing materials form a skeletal, which are manually penetrated (Loboda et al. 2020). The innovation involves the penetration of the resin into skeleton by a molten material (low-melting metal or polymer) which are solidified at the void and porous structure of the resin and form the armoring skeleton (Harris 1999, Loboda et al. 2020).

Whisker Reinforcement: Whiskers are a thin and needle shape crystal as well as mono crystal having an aspect ratio of approximately 10 and more (roughly, diameter of 1 μm) (Dieringa and Kainer 2012, Feng et al. 2020). Whiskers are formed by development from oversaturated gases elsewise electrolysis of liquid or solid materials. Because of the fabrication conditions, it has minimum defect on its density (Dieringa and Kainer 2012). Whiskers are very small and thin structures. This might create a health risks. It may be breathe in and not degraded in the lung, which can be a potential cause to carcinogenetic (Feng et al. 2020).

2.1.2. Matrix material

Matrix is mostly a homogeneous and monolithic material used to embed the reinforcing structure of a composite. The resin is fully in continuous phase. The matrix materials are used for binding and holding the reinforcements materials together to from solid structure. Moreover, the resin used as a protection of the reinforcing materials from external damage and assists the transferring of the load into reinforcement. The resin materials also help for finishing of composite materials such as texturing, coloring, resilience and functionality (Doyle 1989, Azom 2013, Andrew et al. 2019). The resin in composite structure has significant effect on the overall electrochemical properties of composites such as corrosion and oxidation (Doyle1989, Azom 2013). Polymer resin gives resistance from corrosion whereas ceramic resin has been providing excellent oxidation resistance. While the thermal resistance performance of composite is not only affected by reinforcing materials but also significantly affected by the matrix materials. The fabricating cost of the composite is significantly influenced by the matrix materials (Doyle 1989, Azom 2013, Andrew et al. 2019). Table 2.1, describes common properties of composites affected by matrix materials.

Table 2.1. Basic properties of composite materials controlled by matrix (Doyle 1989 & Azom 2013)

Type of matrix	Merits of matrices	Disadvantages of matrices
Thermoplastic Polymer	Can be re-formable and strong	High processing cost
Thermoset Polymer	Low processing cost	Hard (Brittle)
Ceramic	Strong resistance to temperature	High processing cost
Metal	Conductor and resistance to temperature	Form reaction with same reinforcing materials
Carbon	Resistance to temperature	High processing cost

Composite matrix materials are basically classified into three main categories such as polymer matrix material (PMM), metal matrix material (MMM) and carbon matrix material (CMM). The polymer matrix materials are also classified as thermoset and thermoplastic matrix materials based on the thermal behaviors, see in Figure 2.3 (Doyle 1989, Yi and Kumosa 2018).

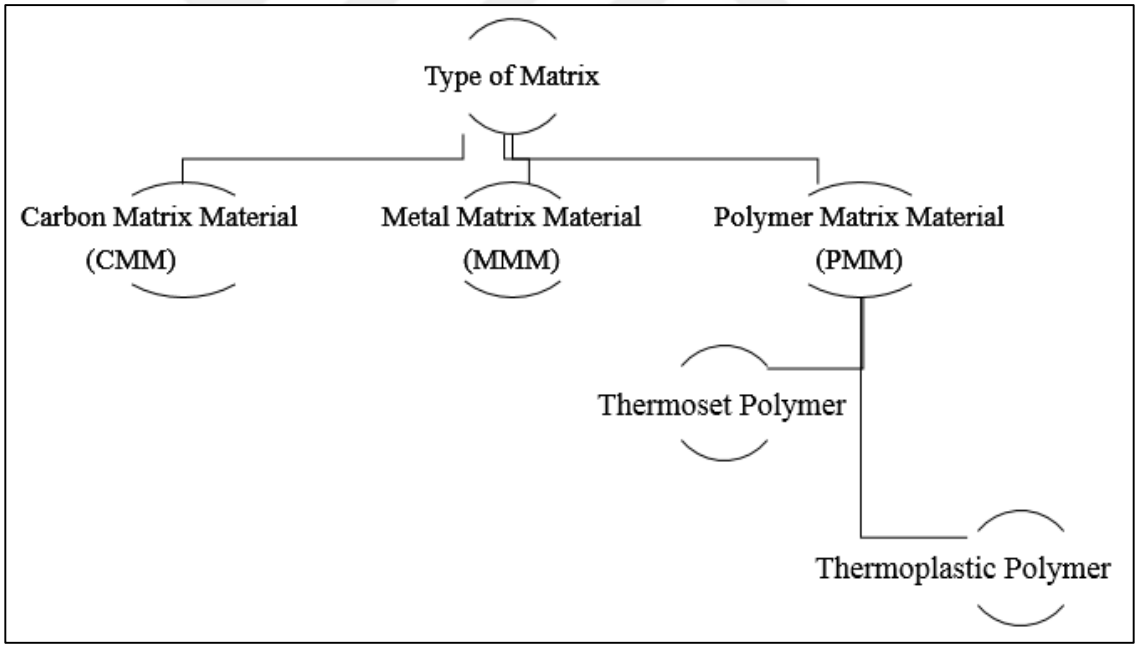
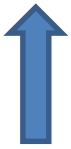
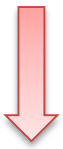
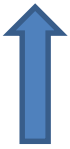


Figure 2.3. Type of matrix material (Yi and Kumosa 2018)

Polymer matrix material (PMM): Polymeric matrices are the well-known type of matrix material in composite manufacturing. The mechanical properties of polymer matrix was varied from one polymer to another polymer (Azom 2013, Lu et al. 2018). Polymer matrix materials have light weight, better strength and corrosion resistance than metal matrix materials. Moreover, the polymer matrix materials have lower thermal and electrical conductive properties without requiring farther surface treatment (Azom 2013). PMM is mostly used in light weigh design because of its poor thermal stability. Polymer is a macromolecule formed by the repeating of monomer structural units linked by covalent chemical bonds. PMM's have lower density than both metals and ceramics matrix. It also resists atmospheric effect and all type of corrosion and has superior resistance to electrical conductivity (Lu et al. 2018). Generally PMM are classified as the thermoset and thermoplastic matrix material. As their prefix "thermos" indicates, they need temperature during processing. Basic characteristics of thermoset and thermoplastic matrix materials are shown in Table 2.2 and Figure 2.3.

Table 2.2. General characteristics of thermoset and thermoplastic matrices (NASA-Langley research center 2019)

Descriptions			**Temperature**	**Process time**	**Toughness**	**Solvent resistance**
			Higher	**Higher**	**Higher**	**Higher**
Matrix types	Thermoset	-Normal resin -Hardened resin				
	Thermoplastic	-Lower cross-linked resin -Normal resin				
			Lower	**Lower**	**Lower**	**Lower**

Thermoset matrix: Thermoset matrix is a type of polymer matrix, in which the resin material is formed by cross-linked structure (irreversible reaction) in the polymer chain under chemical reaction during curing. The whole resin materials are connected together in 3-dimensional network. Once it reached the curing temperature, thermoset materials are not re-melted and re-shaped. Thermoset matrices are irreversibly changed their phase from liquid state into solid state by forming cross linked structure. The change of temperature highly affects the mechanical properties of thermoset matrices. This phenomenon gives the thermoset resin materials to have better dimensional stability and solvent resistance. Commonly the used thermosetting matrixes are epoxies, polyesters,

vinyl esters and polyamides. Thermosets resins are rigid and commonly stiffer, stronger and brittle than thermoplastic resin materials as indicated in Table 2.2 (Rudyak et al. 2019). Thermoset matrices are found in fluid form at room temperature. These give a basic advantage to process them with lower or moderate pressures. So, thermoset matrix is relatively low cost material. Unluckily their re-melting and re-usage problems are not completely solved yet. Nowadays, mostly thermoset matrices are used as high performance composite resin materials as seen in Table 2.3. Polyester and epoxy thermoset matrices are most widely employed in industrial applications as most known type of thermoset matrices (Joseph 2011, Rudyak et al. 2019).

Table 2.3. Properties of thermoset matrix (Stability and Gusakova 2015, Polymer degradation 2019)

Characteristic	**Type of thermoset resin**			
	Epoxy	**Cyanate-ester**	**Phenolic**	**Bismaleimide**
Density (g/cm^3)	1.11-1.41	1.10-1.3	1.23-1.31	1.1-2.8
Curing temperature (^0C)	RT[1]-179	181-217	152-193	219-295
Maximum temperature for continuous-use (^0C)	81-214	151-252	72-173	229-315
Modulus (MPa 10^{-3})	3.0-3.7	3.0-3.3	3.1-5.2	3.3-4.0
Degradation onset temperature (^0C)	255-330	405-421	290-355	361-405
Shrinkage of Mold (mm/mm)	0.0005	0.0041	0.00023	0.0071

Thermoplastic matrix: Thermoplastic (called engineering plastics, thermos-softening) matrices are polymeric matrix materials that are soften and molded at the elevation of temperature (heated) and then becoming solid when decreasing the temperature (harden upon cooling) with our affecting the physical properties (Soo-Jin 2011). General It is a ductile and stronger than thermoset matrix (Jin 2011). Commonly the used type of thermoplastic matrices are polyesters, polyphenylene sulfide, and polyether ether ketone (PEEK) and liquid crystal polymers (Jin 2011). Thermoplastic matrices can be pliable by heating and solidified upon cooling which helps them to frequent repeating the reforming and reshaping of the structure. Thermoplastic materials are flexible as compared with thermoset, due to lack of crosslinking. Thermoplastic materials are either amorphous or semi crystalline as shown in Table 2.4 and Table 2.5 (Congress 1988, Jin 2011). The

[1]RT =Room temperature

degree of crystallinity has a significant effect on the overall properties of the matrix materials (Congress 1988, Jin 2011). Thermoplastics have better resistance to impact and cracking than thermoset materials while it has lower resistance to high temperature (Congress 1988, Jin 2011). Nowadays, thermoplastic matrices are applicable with discontinuous types of fiber reinforcing materials such as glass, graphite and carbon. But thermoplastic matrices have a promising future in polymer composite manufacturing, due to their faster melting and easier cooling nature.

Table 2.4. Properties of commonly used thermoplastic matrices (Interface Science and Technology 2011)

Type of matrix	**Density (g/cm^3)**	**Tensile strength (MPa)**	**Modulus(GPa)**
Epoxy	1.21	68	1.53-3.35
Polyester	1.31–1.42	54–62	2.0–2.79
Polypropylene	0.91-1.23	25–38	1–1.4
Nylon	1.11	54–89	1.29–3.4
Poly carbonate	1.05–1.21	46–71	2.21–2.42
Polyether ether ketone	1.29–1.34	101	3.4–4.5
Poly ethylene	0.9–1.0	46–71	0.71–1.39
Polyetherimide	1.26	104	3.1
Polyphenylene sulfide	1.29–1.39	81	3.42

Thermoset or thermoplastic matrices: Some types of polymer matrix materials are available in the form of thermoplastic and thermoset matrices. Polyurethane, polyimides, polyester and epoxy matrices exist in both thermoset and thermoplastic form. The thermoset polymeric matrices crosslinking agent is broken down by the help of technology and can be used as thermoplastic matrix (Miller et al. 1998). Likewise, the thermoplastic matrices form, such as polyimide matrix freely releases the volatiles substances under the appropriated heat and pressure, which are producing parts in the structure with some voids (Jin 2011, Meola et al. 2016).

Table 2.5. Comparison of thermoplastic and thermoset matrix materials (Jin et al. 2011, Meola et al. 2016)

Characteristics of thermoset matrix	Characteristics of thermoplastic matrix
• Cross linked and non-recyclable via Standard techniques • Low molecular weight (MW) or solid • Low - medium viscosity requires cure • Liquid or solid • Low MW oligomer • Excellent environmental and solvent resistance • Long process cycle • Many structural components. • Excellent finishing • Resistance to heat and high pressure • Fatigue strength. • Not post-formable • Excellent thermal stability once polymerized	• Re-process able, recyclable via Standard techniques • High molecular weight solid • Stable materials • Amorphous or crystalline • Linear or branched polymer • Liquid solvent resistance • Short process cycle • Limited structural components. • Chemical resistance • Need to be heated above the melting point for processing purposes

Metal matrix material (MMM): Metal matrices are type of composite materials that encompass at least one component from the composite structure used as metal matrix. In order to reduce the weight of the composite structure mostly lighter metals are employed as matrix materials such as magnesium, aluminum and titanium. But for high temperature application cobalt-nickel alloy and cobalt matrix is mostly preferred. Metal matrices are used to improve the wear resistance and mechanical performance of the composite materials. Moreover, metal matrices have excellent creep and wear resistance as shown in Table 2.6 and Figure 2.5 (Azom 2013, Rawal 2016).

Table 2.6. Properties of commonly used type of metal matrix material (Rawal 2016)

Type of resin	E (10^6psi)	Density (g/cc)	$\alpha(10^{-6})$ (°F)	FTU (10^3psi)	εf (%)	FTY (10^3psi)	Melting temperature (°F)
Cobalt	31.0	8.85	6.82	109	21	47	2722
Nickel	30.1	8.88	8.61	51	51	17	2655
Aluminum	10.5	2.61	12.9	13.5	46	5.2	1193
Magnesium	6.51	1.81	14.1	45	7.11	33.3	952
Cobalt (Alloy)	35.2	8.68	9.32	139	34	72	2355
Nickel (Alloy)	29.8	8.14	5.55	120	8.2	105	2302
Titanium	15.56	4.52	5.52	34	26	26	2900

Ceramic matrix material (CMM): Ceramics mainly exist as crystalline and non-crystalline compound forms. Commonly ceramics are brittle materials but the strength of ceramic materials are governed by flaw size. The matrices were used to overcome the major disadvantages of ceramic such as lower fracture of toughness and their brittleness as seen in Table 2.7 and Figure 2.4. Ceramic matrices are used for both continuous (long) and discontinuous (short fiber) reinforcing fibers (Imanaka 2012).

Table 2.7. Commonly used ceramic matrix materials properties (Imanaka 2012)

Type of resin	Density (g/cc)	$\alpha(10^{-6})$ (° F)	Temp. (°F)	MOR (ksi)	E (10^6psi)	K_{IC}	ν
Titanium dioxide	4.24	5.21	3363	11	40	2.2	0.27
Aluminum dioxide	3.95	4.6	3720	68	48	3.1	0.25
Si_3N_4 SN	3.17	1.6	3395	71	44	5.0	0.23
Chromium(III) oxide	5.20	4.1	4413	37	14	3.4	-
Silicon dioxide	2.1	0.29	2925	-	10	0.6	0.15
Chromium carbide	6.6	5.6	3433	-	55	-	0.21

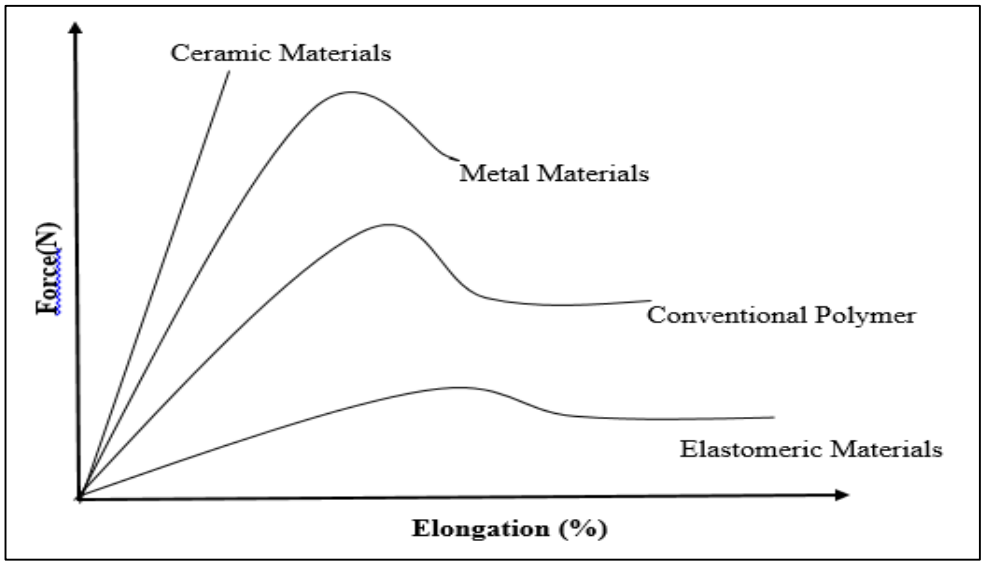


Figure 2.4. Stress-strain diagram of polymers, elastomer, metal and ceramic (Mitchell 2004, Raluca 2012)

2.1.3. Interphase

The interphase of composite structure is the region in which the coming loads from the external environment are transferred from matrices to the reinforcing structure, see Figure 2.1. The degree of interfacing between reinforcing materials and the matrices are significantly affected by strength of interaction, the size of the interface, aggregation, anisotropy of filler and orientation. The interface varies from stronger chemical bonding up to weaker frictional forces (Soo-Jin 2011). These variations can be controlled by proper distribution of the matrix materials into the reinforcing materials and using covenant manufacturing techniques (Naik 1994, Jin 2011). Normally, a strong chemical bonding between the reinforcing and the matrix materials makes the polymer composite structure becoming more rigid and brittle while a weak interaction bond between them will decrease stiffness of the composite structure by enhancing its toughness (Congress 1988). When the interaction bond between reinforcing and matrix materials is not as strong as the matrices, the deboning of the composite structure can ensued at the interphase region at lower loading conditions (Congress 1988, Jin 2011, Heredia 2016). The nature of the interferential bond also plays a major role in its prolonged existence and stability of the composite structure as shown in Figure 2.5 (Naik 1994, Jin 2011, Heredia 2016).

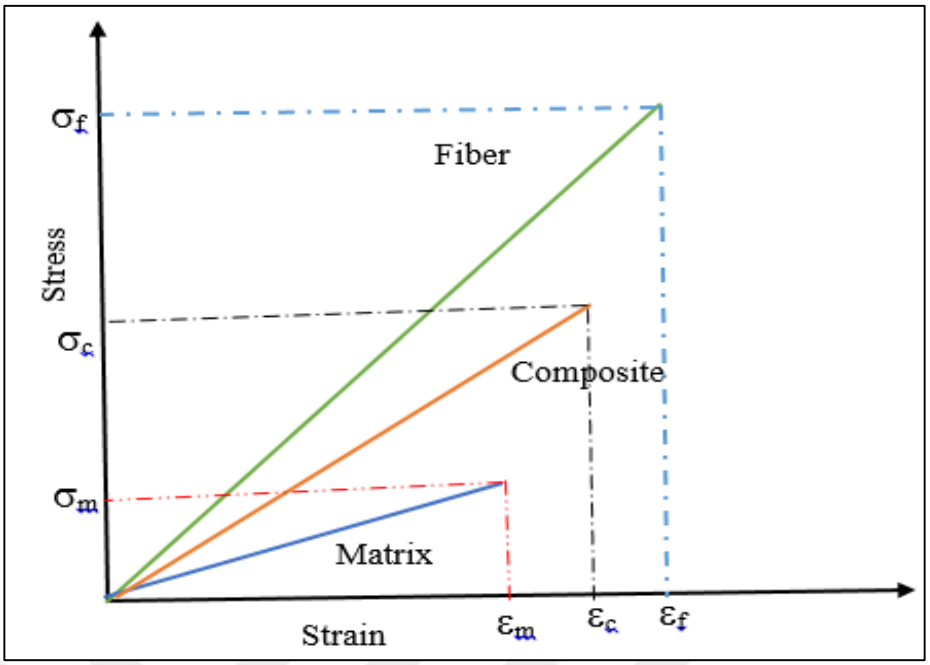


Figure 2.5. Stress strain diagram of fiber reinforcing, matrix and composite material (Heredia 2016)

2.2. General Characteristics of Composite Material

The rapid development of new manufacturing technology and the growth of composite materials are a vital change in the histories of material science and characterization of materials (Matthews and Rawlings 2009, Hu 2012). Composite materials are multifunctional structures having unique physical properties and mechanical performances which can be customized to satisfy the need of a specific applications (Kumar and Srivastava 2017). Composite materials are tremendously versatile structures. The unique characteristics of composite materials makes them different from conventional materials. They have low density, high strength, excellent resistance to fatigue, resistance to corrosion and wear, low coefficient of thermal expansion, and creep rupture. These distinctive characteristics give special engineering properties which cannot be obtained from conventional materials (monolithic-unreinforced structures). Moreover, composite materials are able to solve different major limitations of traditional materials such as mechanical and thermal shocks, integrating of different categories of monolithic solid materials such as plastic, ceramic and metal (due to their unique properties) in their structures. Recently, the problem of large and complex structural design fabrication was

solved by well adapting composite manufacturing technology with affordable cost (Lotfi and Li 2019). Polymers, carbon, ceramics and metals are monolithic solid materials found as reinforcement as well as matrix material. This phenomena opens a new era in structural engineering and industrial sectors by overcoming unique properties of a material and also obtaining a different properties which are not found from a single materials (Campbell 2010). There are different types of composite materials in the world. It may be natural or fabricated (manmade) composites. These different type of composite materials have different physical properties and mechanical performance. However, composite materials have some common properties and characteristics (Joseph 2012). The overall performance and properties of composite materials are characterized by the behavior of each composite constituent phases, numerous geometrical shapes and relative distribution of reinforcing materials, seen in Figure 2.6. The general characteristics of composites are affected by these properties. These general characteristics of composite materials are discussed briefly below in Figure 2.6 (Davoodi et al. 2010).

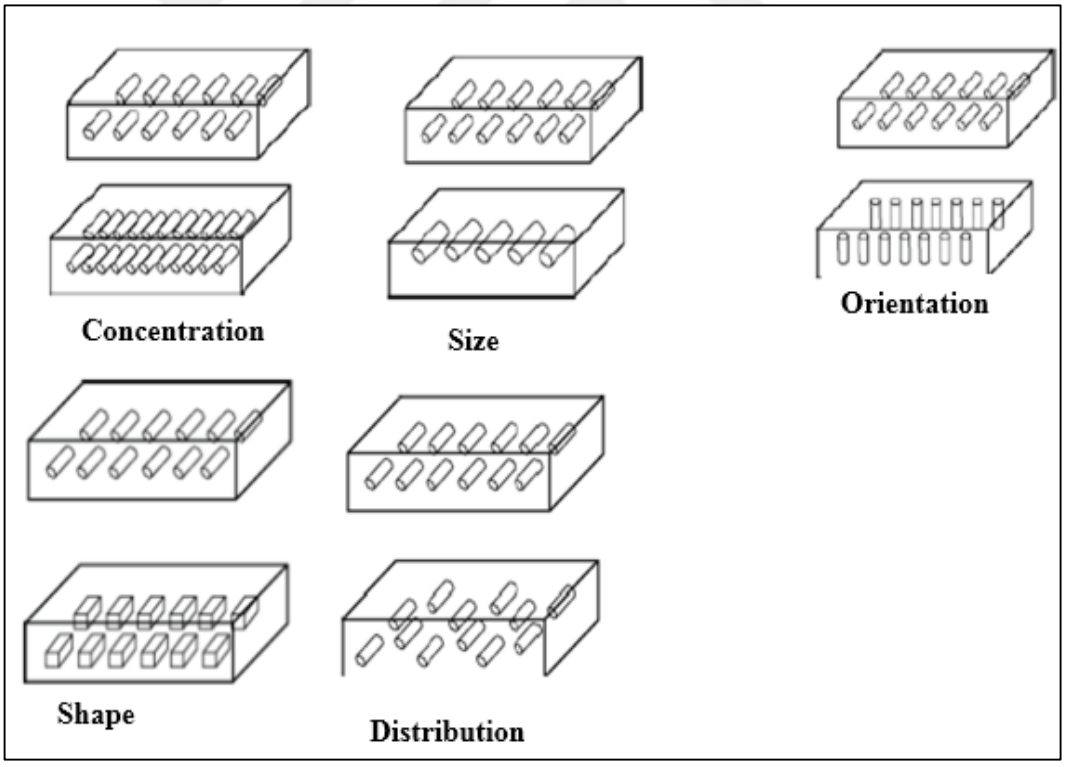


Figure 2.6. Schematic representation of different geometric shapes of reinforcing materials (Sophia and Berna 2012)

High strength and high stiffness to weight ratio: Fiber reinforced composite materials are highly strong for their weight. Composite materials have high specific strength to weight ratio and high stiffness than its individual constituents as well as most traditionally used monolithic materials. Because the density of the composite materials is normally lower than regularly used materials, this property gives a significant advantages to have lower specific strength and modulus (Unterweger et al. 2014).

Light weight: Composite materials are light weight multifunctional structures compared with commercially used most engineering materials such as wood, metal and ceramic. Their lightness play a significant role in vehicles and airplane industries. Lighter weight composite structures consume less energy and have better fuel efficiency. Automobile and aircraft designers greatly focuses on weight of structure in order to reduce the fuel consumption. Moreover, reduction of the weight of composite materials will increase the speed of automobiles as well as the airplanes. Based on the Australia composite (2019) report, currently most automobiles and airplanes are made from composite materials like Dreamliner, Boeing 787 (Campbell 2010, Unterweger et al. 2014).

Chemical and weather resistance: Composite materials can resist damage from most chemicals and environmental weathers. It can be used in harsh environmental conditions with a wide range of temperature change. Composite structures are not corroded, due to this tier handling and storage are not expensive like conventionally used structural engineering materials (Campbell 2010).

Design flexibility: Composite materials with intricate design manufacturing are not difficult as most engineering materials. Complex shapes and structures can easily mold with affordable cost. Easily molding of the desired form and shapes of composite structures gives freedom and flexibility for the designer to fabricate any products. Recently, recreational boats are manufactured from glass fiber. The fiber can easily be shaped into intricate structure and forms, which are enhancing the design of luxury boats by reducing the cost of fabrication. Also, surface finishing, texturing and smoothing of composite materials are easily achieved by the used matrices (Mohamed and Hosam 2018).

Durability: Composite materials have long shelf life and incredible durable structure. Most composite materials have been used for several half of a century without suffering like metal materials. They have outstanding fatigue resistance and tolerate to sever environmental weather conditions like moisture, extremely high temperature, damage from ultraviolet ray and chemical attack. Yet now, the life span of composite materials is not known because it did not come to end for natural composite materials like wood (Mohamed and Hosam 2018).

Radar transparent: Composite materials can easily pass the radar signals. It can be used in anywhere when radar signals exist. It also plays a significant role for enhancement of aircraft sector to fabricate nearly invisible from radar. B-2 stealth bomber of United State Air force is model examples of almost are not detected by radars. So, the development of composite materials will have a tangible effect in the growth and expansion of radar science having light weight structure with affordable cost (Mohamed and Hosam 2018).

Nonmagnetic and nonconductive: composite materials mostly do not consist of metal element unless metallic matrices are not used. Because of this, composite materials are nonconductors and nonmagnetic structures. Due to lack of conductivity and magnetic field interference, composite materials are used in sophisticated medical equipment like magnetic resonance imaging. Also, composite structures are used widely as electric circuit board and poles. When the conductivity of the materials are needed, it becomes easier to make them conductors (Mohamed and Hosam 2018, Composites-Australia 2019).

2.3. Classification of Composite Material

Recently, composite materials are becoming the most promising and multifunctional materials (Murr 2015). In the development of industrial technology, the customer needs a better replacement of commercially known materials with products having higher strength, lower density, better stiffness and affordable cost. Due to their outstanding properties such as high strength and light weight, natural as well as synthetic materials reinforced composite structures are becoming a very significant materials from industrial sector of airspace and construction (Dipen and Durgesh 2019). Composites are either

anisotropic or isotropic form as shown in Table 2.8 and Figure 2.2 and Figure 2.3 (Murr 2015). Composites are formed by the combination of two or more components having a superior properties from the individual materials used separately while in traditionally used metallic alloys, every one of the materials in the alloys maintained its isolated physical, mechanical as well as chemical properties (Maha 2017).

The broad classification of composite materials have been made based on their nature of formation i.e., traditional composite such as wood, bone and concrete or manufactured composite such as glass fiber reinforced composites. The properties of natural composite materials do not fulfill the unlimited need of human being. So, synthetic composite was manufactured in order to control and modify the properties as well as the structure of composite according our needs. The classifications of composite materials are based on their constituents i.e., based on their matrix and reinforcing materials. Based on the former, based on matrices, composite materials are classified as polymer matrix composite materials (PMC), ceramic matrix composite (CMC) and metal matrix composite (MMC) materials as shown in Figure 2.3 (Dipen and Durgesh 2019). While in latter case, based on reinforcing materials, composite materials are categorized as fiber reinforced composite, particle reinforced composite and structural composites. Furthermore, fiber reinforced composites are classified as continuous and discontinuous fiber reinforced composite materials. Like with the structural composite materials are further categorized as laminate composite and hybrid (sandwich) composites as shown in Table 2.8. (Maha 2017, Rahul 2017, Dipen and Durgesh 2019).

2.3.1. Classification of composite materials based on matrix

In composite materials manufacturing industries, there are three most commercially used type of matrices. These matrices are polymer, ceramic and metal with their alloys (Florea and Carcea 2012). The composite materials manufactured by the use of these matrices have been categorized as polymeric composite (PCM), ceramic composite (CMC) and metallic composite (MMC) materials as shown in Figure 2.8 (Maha 2017, Dipen and Durgesh 2019).

Polymer matrix composite material (PMC)

Recently, polymeric materials play a very significant role in our day today activities (Florea and Carcea 2012). Most of the materials used in our daily life are made of polymeric materials. These are because of their physical and mechanical properties such as lighter weight, easy for processing, non-corrosion and lower electrical conductivities (Voicu 2012). In 1980, polymer materials are stared to use as a matrix materials. It has been used in inorganic and textile fiber reinforcing composite manufacturing sector (Voicu 2012). Polymer matrices are a primary phase used to bind polymer, ceramic, metal and their alloys. Thermoplastic as well as thermoset type of polymeric matrices are most commonly used. Thermoplastic matrix is preferable due to its reusing and recyclability while thermoset matrix is favored because of its lower viscosity (Jose and George 2012, Youssef et al. 2015). Mostly the physical and mechanical properties of polymeric composite materials are significantly affected by environmental condition such as moisture and temperature. Same polymeric matrices are started to swell when the materials are exposed to moisture. This causes the delamination of reinforcing material at the interfacing regions (Voicu 2012). Moreover, polymer matrix composite materials are degraded by ultraviolet ray, due to the breakage of consecutive monomers connecting linkage in the polymeric structure (C-C bond) (Shokrieh and Omidi 2009, Youssef et al. 2015).

Metal matrix composite material (MMC)

Metals are used as matrix materials in modern composite manufacturing industries. The continuous phase of metallic matrix has better strength and stiffness with respect to their weight ratio. It has also a benefit of higher damage tolerance with a wide range of operating environment conditions as compared with ceramic and polymeric matrix composite materials. Mostly, titanium, aluminum, magnesium, copper and their alloys are used as a metallic matrix materials as shown in Figure 2.7. In order to get the desired benefit of metallic matrix composite materials, the selection of the type of reinforcing materials for metallic matrix materials is a vital criterion. Commonly used reinforcing materials are ceramic, tungsten and lead. These type of composite structures are mostly applicable when high thermal performance and wear resistance are needed (Adebisi et al. 2011, Rawal 2016).

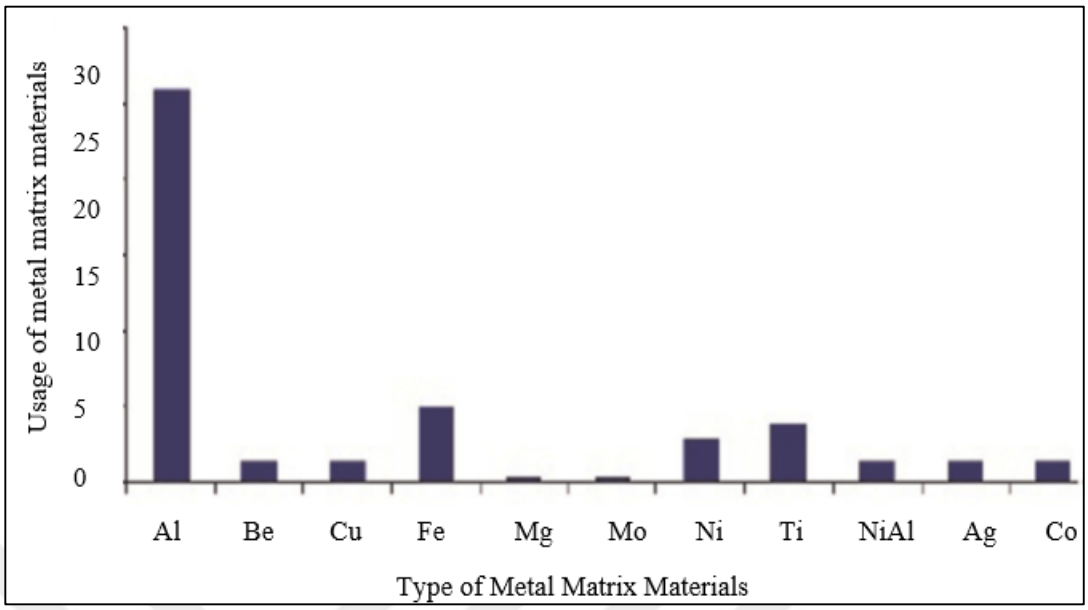


Figure 2.7. Metal matrix used in metal matrix composite material (Adebisi et al. 2011)

Ceramic matrix composite material (CMC)

Natural ceramic materials have poor flexural toughness. The cracking of matrices, delamination and poor mechanical toughness were solved by fabrication of ceramic matrix composite materials. Ceramic are used as both matrix and reinforcing materials. Ceramic matrix composite structures are formed by the combination of ceramic matrix with any type of ceramic oxides and carbides or with any type of reinforcing materials. It is characterized by low density, high thermal stability, extraordinary mechanical performance in higher temperature, better resistance for thermal shock with thermal stability, extraordinary stiffness and toughness, better resistance of corrosion at high temperatures. Ceramic reinforcing materials exist as a form of fibers, particles as well as whiskers. Ceramic matrix composite structures are becoming more popular due to their higher mechanical performance, resistance to cracking and higher thermal stability. Carbon and its matrix reinforced material are categorized under ceramic (Chawla 2011, Basutkar and Kolekar 2015, Singh et al. 2017, Rajak et al. 2019).

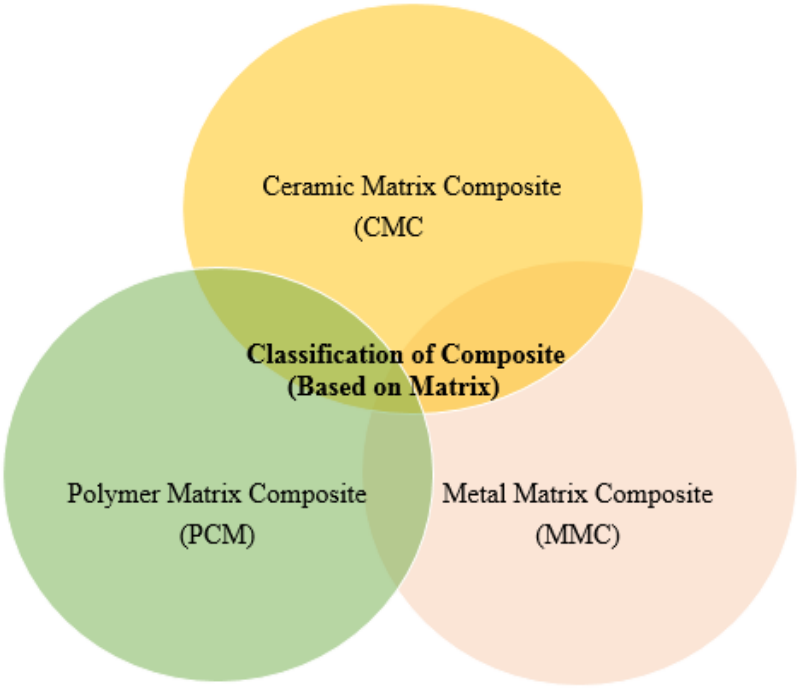


Figure 2.8. Classification of composite based on matrices material (Rajak et al. 2019)

2.3.2. Classification of composite materials based on reinforcement

The term reinforce is related to enhancing the mechanical strength of the materials when added to the existing materials. Different materials have shown different properties for applied load. For examples, concert material are poor resistance for tensile load when it was reinforced with metal, its mechanical performance was drastically enhanced. The physical and mechanical properties of composite materials are determined by the type, shape, distribution, orientation and concentration of reinforcing materials as shown Figure 2.2 and Figure 2.9. Composite materials are classified based on their reinforcing materials (discontinuous phase) such as particle/ whisker reinforcing composite, fiber reinforcing composite and hybrid composite materials (Chawla 2011, Basutkar and Kolekar 2015, Singh et al. 2017). These classification of reinforcing materials were explained in detail below.

Table 2.8. Type of reinforcing materials with formation of composite structure (Rajak et al. 2019)

Type of reinforcement	Formation of composite structure	properties
Particle	Particle reinforced	Isotropic
Short fiber	Random	Isotropic
	Aligned	Anisotropic
Continuous fiber	Aligned continuous fiber	Anisotropic
Laminate or layer	laminate	Anisotropic

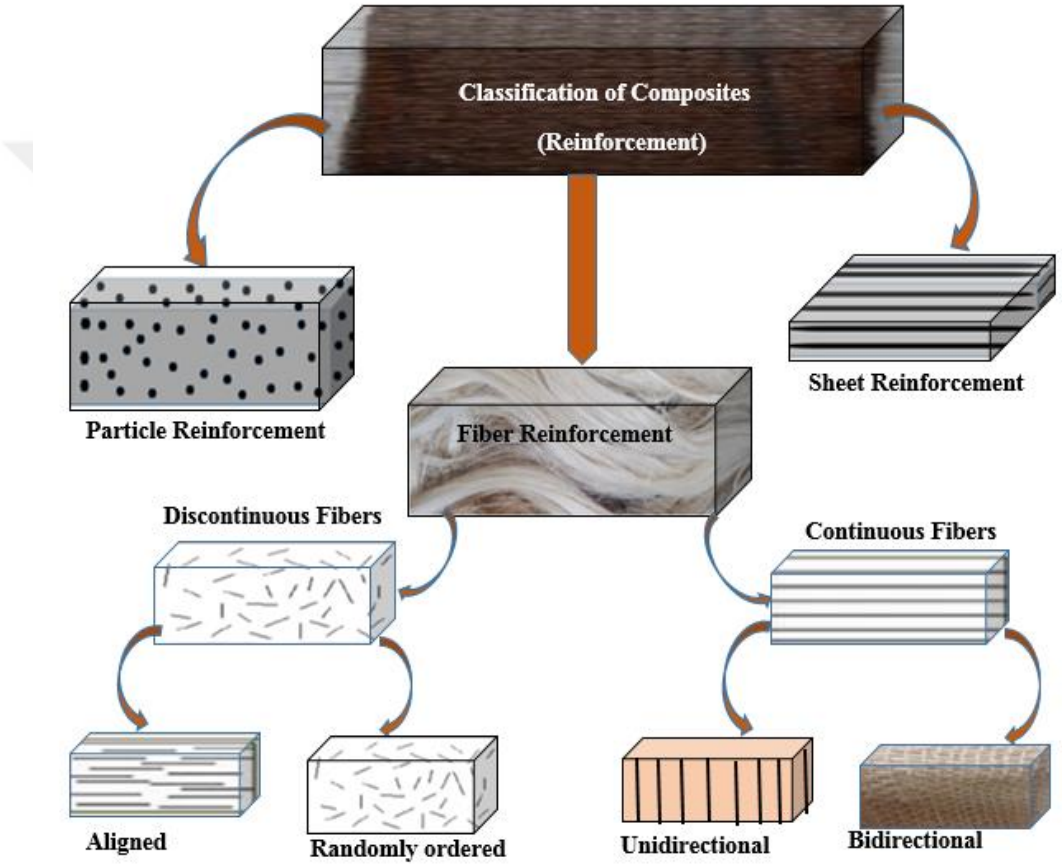


Figure 2.9. Classification of composite based on reinforcing material (Rajak et al. 2019)

Particle reinforced composite material

Different types, size and shapes of particles are used as reinforcing materials in composite manufacturing sectors as shown in Figure 2.10. Particle reinforcing is a simple and cost effect composite manufacturing techniques, which are providing isotropic behaviors and the same manufacturing mechanisms, are used like monolithic materials. It has poor strength, low deformation and inferior fracture resistance compared with fiber reinforced

composite structure. Mostly particle reinforced composite are used in the areas where high wear resistance materials are required such as road and civil structural materials (Maha 2017, Dipen and Durgesh 2019).

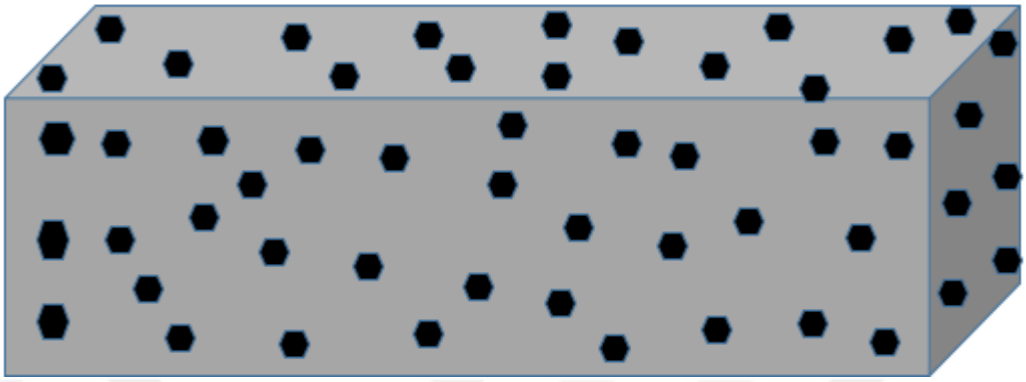


Figure 2.10. Particle reinforced composite material (Maha 2017)

Fiber reinforced composite material

In composite fabrication industries, if synthetic, natural fiber or the blend of them are used as reinforcing materials they are called fiber reinforcing materials as shown in Figure 2.11. Also, composite materials composed of continue fibers are known as fiber reinforcing composite. It may be either continuous or short fiber, based on fiber length. Fibers have high length to diameter ratio. Fiber reinforced composite materials have higher strength and stiffness than other type of reinforcing materials with their weight ratio (Scribante et al. 2018, Dipen and Durgesh 2019). The reinforcing material can be used as in the form of either unidirectional or bidirectional arrangements in composite fabrication processes. The arrangement and orientation of fibers have a significant effect on the mechanical properties of composite (Scribante et al. 2018, Dipen and Durgesh 2019).



Figure 2.11. Fiber reinforced composite material (Scribante et al. 2018)

Hybrid reinforced composite material

As the name indicates, in the case of hybrid reinforced composite materials, two or more type of reinforcing materials are combined together to form the composite structure as shown in Figure 2.12. Or, two or more type of matrices materials can also be used. In order to get the advantages and avoid the limitation of different type of reinforcing and matrix materials, composite manufacturers blend or mix reinforcing and matrix materials. Natural fibers are blend with synthetic fiber in order to achieve the benefit of bio degradable, light weight and cost benefits while synthetic fiber give good mechanical performance. Hybridization of composite material is a good strategy to solve the drawback of both reinforcing and matrix materials (Dipen and Durgesh 2019).

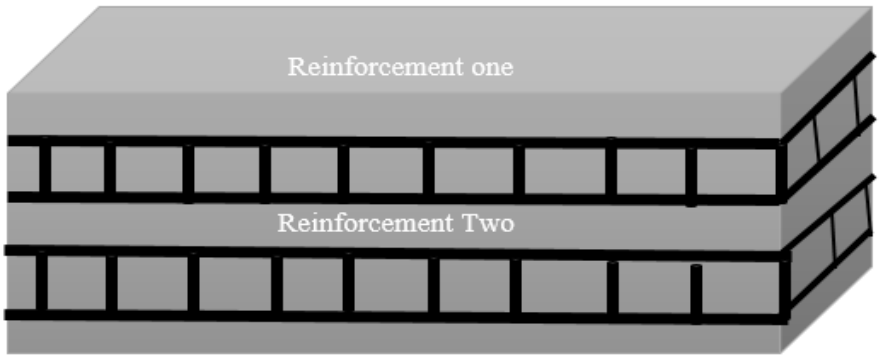


Figure 2.12. Model hybrid reinforced composite material (Dipen and Durgesh 2019)

2.4. Textile Reinforcing Composite Structure

The term textile comes from the Latin word “Texere” which means “to weave”. According to Textile Institute, textile materials are defined as “a materials made from either natural or synthetic fibers /yarns, having higher length to diameter (thickness) ratio, flexible and fine characteristics (Hossain 2018). Fiber is defined as long thin strand materials. Textile fiber is a very thin long structure having high length to diameter ratio. All type of fibers are not textile fiber but all textile fibers are fiber. The basic requirement of a fiber used as textile material is minimum of 5 mm length, enough strength and cohesion. Moreover, the fiber must have the desired fineness, durability and uniformity. Based on their source, textile fibers are classified as natural, synthetic and regenerated fibers as shown in Figure 2.14 (Wadje 2009).

Increasing demand of human being and the development of new technologies have created advanced multipurpose textile products. The finiteness of petrochemical resources and global warming have pushed researchers and manufacturers to find out sustainable composite products reinforced from textile materials. Textile materials such as synthetic fibers, natural fibers and their fabrics are used in composite industries as a reinforcing structure with different type of fabric constructions like woven, knitting, non-woven and braided (Adanur 2000, 2019). Textile materials reinforcing composite structures have a major benefits like weight, flexibility, higher strength and stiffness to weight ratio, adaptability and resistance to most chemicals (Aly 2017, Peled and Bentur 2017). Polymer reinforced composite materials are most commonly used in transportation areas whereas ceramic and metal reinforced composites are applicable when a special mechanical performance has been required. Textile materials reinforced products used in transportation and construction sectors have lower energy consumption, high durability and lower structural weight without influencing the mechanical properties (Lomov and Verpoest 2010, Aly 2017). The mechanical performance of textile reinforced composite structures are influenced by both fiber/yarn entanglement, orientation ($0, \pm 45, 90^0$), type of fabric construction and manufacturing technology (Aly 2017, Peled and Bentur 2017). The classification of composite structures reinforced with textile materials are demonstrated in Figure 2.13 (Aly 2017, Karaduman and Karaduman 2017, Shesan et al. 2019).

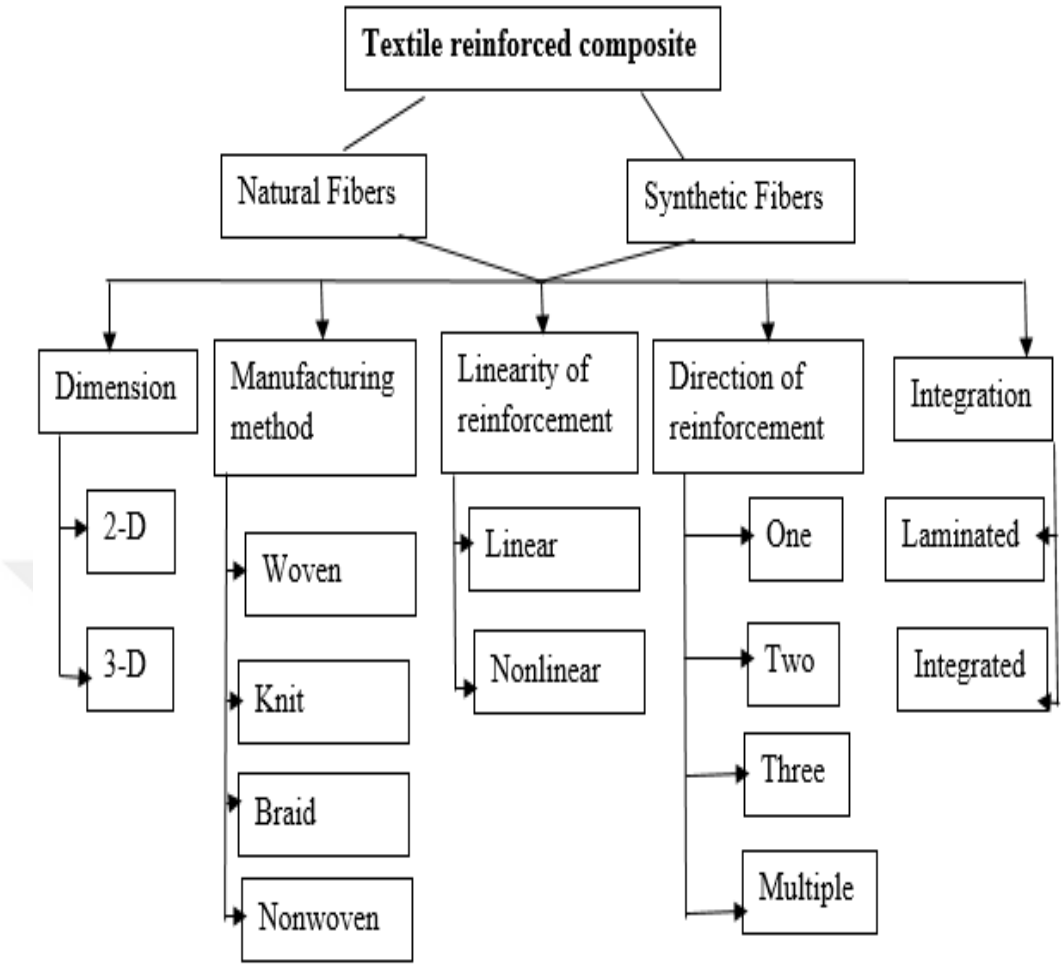


Figure 2.13. Classification of composite structures reinforced with textile material (Bhatfacharya 2017)

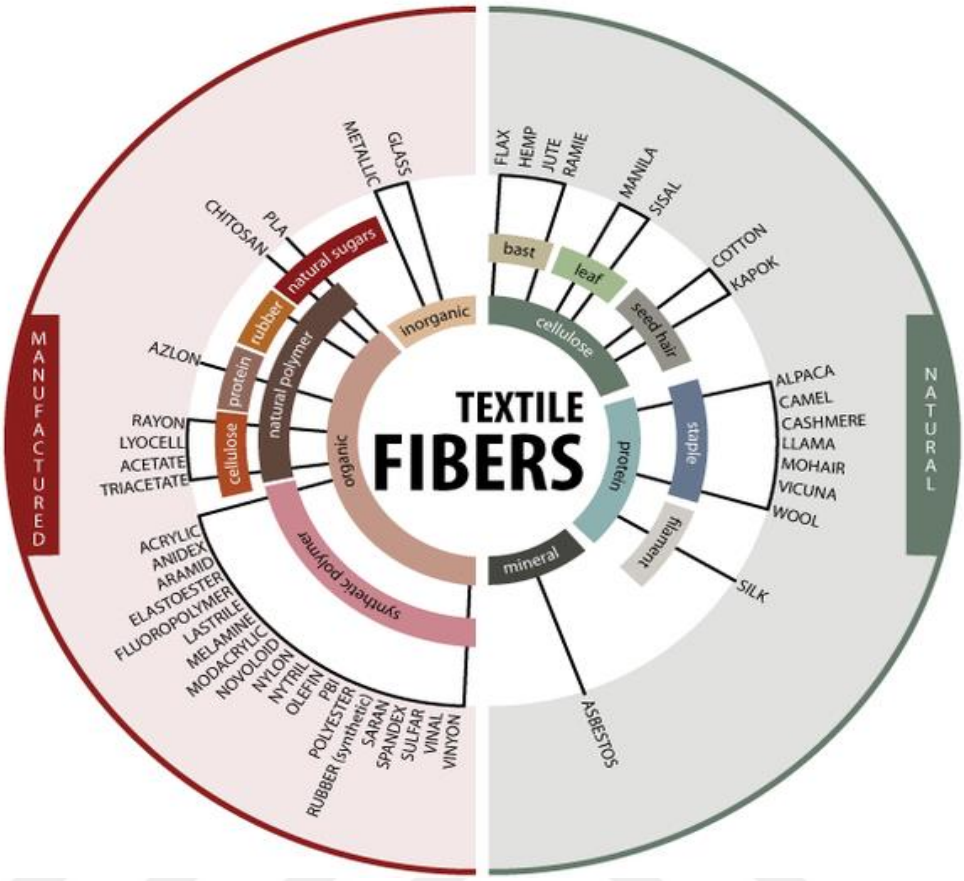


Figure 2.14. Classification of textile fiber (Textile Fiber Products Identification Act 2020)

2.4.1. Synthetic fiber reinforced composite material

Textile fibers, obtained from naturally or manufactured by chemical synthesis, are one of the most widely used reinforcing materials in composite manufacturing industries. Textile fibers manufactured by the interfering of human being with the help of technology and chemical synthesis. The fibers are produced from polymers of organic and inorganic sources. Synthetic fibers have better physical and mechanical performance than natural and regenerated fibers (Dipen and Durgesh 2019). Glass, carbon, polyester and Kevlar etc. are commercials available synthetic fibers used as filler or reinforcement in composite fabrication. The limitation of petrochemical resources and environmental issues are a major problem for synthetic fibers' use sustainably. Nowadays natural fibers are becoming popular in green composite manufacturing sectors. These fibers are found in the form of animal, plant as well as mineral fibers. Moreover, natural fibers have a very

wide diversity of resources (Karaduman and Karaduman 2017). Most commercially used natural plant fibers in composite manufacturing include flax, banana, sisal, jute etc. The mechanical performance of commonly used fibers are illustrated in Figure 2.13 (Unterweger et al. 2014). In this section, only composite materials reinforced by synthetic fibers were discussed in detail. Synthetic fiber reinforced structures have same basic benefits compared with natural fiber reinforced composite materials such as high mechanical performance, wide range of thermal stability, better process ability, higher impact and wear resistance as shown in Table 2.12 (Unterweger et al. 2014, Karaduman and Karaduman 2017, Shesan et al. 2019).

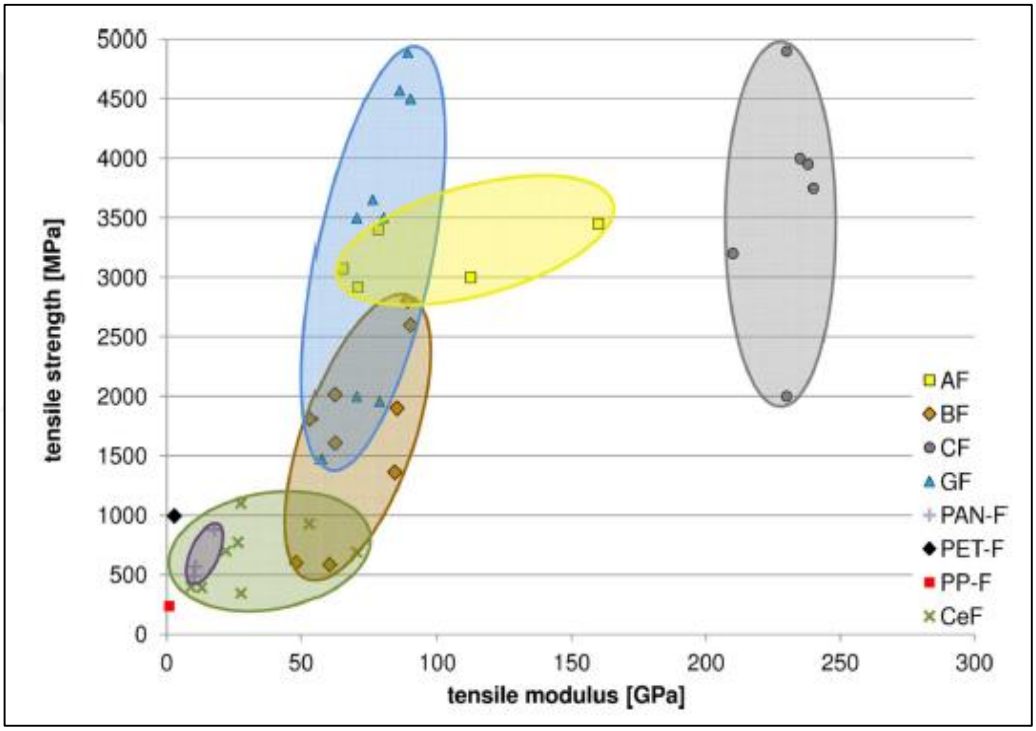


Figure 2.15. Tensile properties of commonly used fibers in composite manufacturing[2] (Wefer et al. 2012)

Glass fiber: Glass fiber is one of the most traditionally used reinforcing materials in composite histories. It is manufactured in the form of multi-filament bundles (Stickel and Nagarajan 2012). The fibers are characterized by their stability in higher temperature, durability, excellent wear/friction resistance, superior mechanical strength, better impact

[2] AF-Aramid fibers, BF-Basalt fibers, CF-Carbon fibers, GF-Glass fibers, PAN-F- Polyacrylonitrile fibers, PET-F Polyethylene terephthalate fibers, PP-F Polypropylene fibers, CeF Cellulose-based fibers

resistance and resistance to chemicals as shown in Figure 2.15 and Table 2.9. The major problem of glass fibers are their disposal of waste, difficulty in machining, high machine wears. Most usually used type of fiber in composite manufacturing industries are “E” and “S” type of glass fiber. E-glass fibers are cheaper and easily processed in machines as compared with other class of glass fibers while the “S “ type is preferable when high strength is required in the composite structure (Stickel and Nagarajan 2012).

Table 2.9. Frequency used reinforcing manmade fibers properties (Carl et al. 2015)

Type of reinforcing fiber	Density (g/cm^3)	Tensile strength (MPa)	Thermal conductivity (W/m·K)
E-glass	2.5	2020	0.91
Aramid	1.41	3100	0.039
High-density polyethylene	0.96	3010	-
S glass	2.6	4205	0.91
SM-Carbon -PAN	1.71	3210	9.1
Basalt	2.71	2800	1.69
UHM-Carbon -PAN	1.90	3750	17

Carbon and graphite fibers: Carbon and their derivatives like graphite are becoming best alternative reinforcing materials in aircraft industries due to their light weight and better mechanical properties. As seen from Figure 2.15 and Table 2.9, carbon fiber is lighter and more rigid than glass fiber. Carbon fibers sometime known as graphite fibers are found in different varieties of mechanical performance such as strength and modulus. It is characterized by low density, high strength and stiffness. Moreover, the fibers have excellent creep resistance, corrosion resistance, good fatigue resistance, better rupture and it is oxidised when the temperature is increased (Elanchezhian 2014). Graphenes are recently developed type of carbon fibers having superior electrical conductivity and mechanical strength. Most of its properties are enhanced. Due to this, it has better future potential to substitute traditionally used fibers in different application areas such as low density conductor wires, micro-motor and knittable capacitor (Dipen and Durgesh 2019). Polymeric fibers: Like aramids, polymer materials are frequently used as composite reinforcing materials. These kind of reinforcing materials are generally effective in polymer matrix material. The major limitations of polymeric fibers are their sensitivity for thermal and poor surface energy. Due to this, the fibers need basic surface modification before used as reinforced materials in high performance structures. Generally of fibers obtained from polymer sources have light weight, moderate to high

tensile strength, poor stiffness and degradation by ultraviolet radiation (degraded) (Joshi and Bhattacharyya 2011).

Basalt fiber: Nowadays, manufacturers focus on materials which are environmental friendly and biologically compostable as well as degradable. Basalt fiber is one of most recently used reinforcing materials. It has unique mechanical, chemical and physical property and economically effect fiber, see Table 2.9. It was characterized by its low cost and superior mechanical properties than glass and carbon fibers (Dipen and Durgesh 2019).

Table 2.10. Basic characteristics, advantages and limitation of natural and manmade fiber (Khubab et al. 2016, Sanjay et al. 2016)

Characteristics	Property	Manmade fibers	Natural fibers
Technical	Mechanical performance	High	Moderate
	Sensitivity of moisture	low	High
	Sensitivity of thermal	low	High
Ecological	Resource	Limited	Infinite
	Manufacture	High	Low
	Recyclability	Moderate	Good
Merits & Limitations	Merits	- Dimensional stable - Uniformity - Resistance to microorganism	- Low cost - Light weight - Biodegradable - No health effect
	Limitations	- Recycling problem - Expensive - Health hazard - Heavy	- Non uniformity - Dimensional problem - Affected by microorganism

2.4.2. Natural fiber reinforced composite material

Natural fibers obtained from plants have been used as composite reinforcing materials. This is not a new innovation, it was started from ancient times. The fibers are effectively utilized as one of the reinforcement material in house construction within the history of human civilization. In ancient times, a number of natural fibers were used to enhance the mechanical properties such as tensile strength, impact resistance and improve the stiffness of mud by straw in bricks fabrication processes (Fuqua et al. 2012). Nowadays,

increasing demand of human being and awareness of suitable products, manufacturers and researchers have focused on renewable natural materials like natural fibers and their derivate for replacing manmade fibers. Moreover, the limited resources of petrochemicals and the development of new environmental regulations play a significant role for the development of biodegradable composite materials (Lotfi and Li 2019). The growth of ruble agricultures, global warming and awareness of the society about ecofriendly products primarily initiated researchers to revisit natural products and found out novel natural fibers to substitute plastic and replacing old-fashioned metallic composite structures. Currently, the trend shows that the engineering and industrial composite materials are fabricated today more than before from natural fibers such as jute, sisal, banana, flax etc. as shown in Figure 2.16 (Lotfi and Li 2019).

Table 2.11. Commonly used natural and synthetic fibers reinforcement property (Stickel and Nagarajan 2012)

Type of fiber	Density (g/cm^3)	Moisture content (%)	Tensile strength (MPa)	Elongation (%)	Modulus (GPa)
Banana	1.34	8.6–12.5	300-500	1.4–8.5	12.2
Jute	1.32–1.5	12.4–13.6	350–750	1.1–1.7	8.2–75
Kenaf	1.42	9.1–12.2	225–940	1.4–2.6	15–52
Flax	1.41–1.51	8.5–12.5	340–1900	1.21–3.32	30–102
Cotton	1.51–1.60	7.8–8.4	290–750	3.2–9	6–12
Bamboo	0.7–1.11	9	145–810	2.4–3.8	15–31
Abaca	1.51	6–10.5	410–970	1.1–9	8–21
Hemp	1.39–1.50	7–12.5	300–850	1.5–3.4	25–92
Sisal	1.32–1.51	10.5–15	370–750	2.5–7.2	10–35
Coir	1.14–1.45	7.5	100–220	10–50	3–6.5
Aramid	1.41	-	3200	3.31–3.6	60–66
E-glass	2.5	-	3400	2.51–3.3	71
Carbon	1.6	-	3950	1.42–1.78	230
Enset[3]	1.1-1.2	8-10	340-500	2.5-3.6	12-38

Natural fibers are obtained from different parts of plants, animals as well as minerals. Sisal, banana and abaca are obtained from leaf part of the plants while jute and kenaf are extracted from the stems (bast) as shown in Figure 2.15 and Table 2.11 (Lotfi and Li 2019). The growth of composite materials reinforced with natural fibers have faced some challenges that are not commonly seen in synthetic fibers. These include the non-

[3] The results are the part of the Thesis

uniformity in their constituents, variation of properties due to geographical and climatic conditions, high moisture content, highly affected by micro-organisms and limitation of manufacturing technology as shown in Table 2.11 (Fuqua et al. 2012). On the other hand, natural fiber reinforced composite materials have a lot of advantages compared with manmade fiber reinforced structure such as, abundantly availability, low density, moderate mechanical strength, better acoustic performance and biologically composability or degradability (Lotfi and Li 2019). Manmade fiber reinforced composite materials have serious limitations such as non-biodegradability, expensive, consume high power, global warming and health problems (Unterweger et al. 2014, Lotfi and Li 2019).

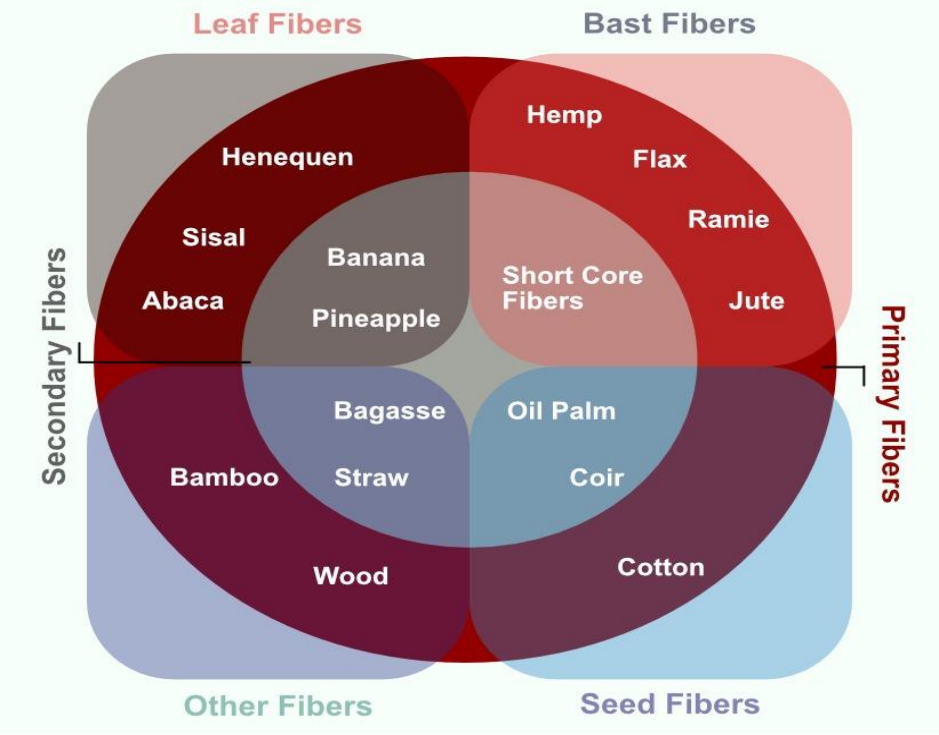


Figure 2.16. Commercially used reinforcing fiber obtained from plant (Shesan et al. 2019)

Jute fiber: The fibers are one the most abundantly available and cheapest natural fibers mostly grown in India and Bangladesh. Jute fibers are one highly commonly used natural fiber in technical textiles, obtained from the stem (bast) of jute plant. The extraction system of the fiber from its plant is simple and cost effective compared to other lead and seed fibers. The fibers are extracted by mechanical or using decortication machines. Raw jute fibers have harder and brittle nature, so the machinability of the fiber is very difficult without lubrication (emulsification). It is a lignocellulose fiber, which is composed of

cellulose, hemicellulos and lignin. The properties and chemical composition of jute fibers are depending on the geographical location and weather conditions (Kumar and Srivastava 2017). Generally, jute fiber is characterized by light weight (density of 1.3 to 1.5g/cm^3), moderate mechanical strength (tensile strength of 400 to 800 MPa and modulus of 10 to 30 GPa), elongation up to 1.8% and moisture content of 13% as shown in Table 2.11 and Table 2.12. The fiber is traditionally used in packaging and technical textile sector as rope, bag and mat. Due to their lightweight, cheap price, good mechanical performance and their biodegradability, researchers and manufactures have used it as an alternative of manmade fiber reinforcing materials in composites (Gupta 2015, Kumar and Srivastava 2017). The physical and mechanical properties of jute fiber reinforced composite materials are significantly influenced by length of the fiber, chemical constituents, fiber distribution, fiber orientation, size and shape of fibers. Moreover, the type of matrix used, composite fabrication and the strength of bond between the reinforcing and resin play a major roles on the mechanical performance of jute fiber reinforced composite structure like other plant fibers (Gupta 2015).

Table 2.12. Mechanical properties of jute fiber reinforced composite (Gupta et al. 2015)

Type of matrix	Nature of jute reinforcement	Tensile strength (MPa)	Flexural strength (MPa)	Impact strength (J/m)	Tensile modulus (GPa)	Flexural modulus (GPa)
Epoxy		15.54	79.25	-	0.2553	1.354
LDPE[4]	Jute fiber	16.4	21.6	-	1.2	0.7
PC[5]		62.4	86	50.38	-	4.2
		24.55-30.1	41.62	-	0.78-2.5	1.29-3.2
PP[6]	Jute fabric	51.5	60	22.31	1.05	3.26
	Jute fiber (Short)	31	37.5	64.5	0.84	1.67
polyester resin		63.5	24.5	-	6.15	-
	Jute fiber	68.67	93.08	-	6.18	5.92
Soy-protein	Jute strand Y-Direction	35.5	34.4	-	0.98	1.025
		36.8	37.8	10.8 (KJ/m^2)	1.030	1.11
Starch	Jute woven fabric (Untreated)	21.9	36.3	14.2 (KJ/m^2)	2.46	-
	Jute fabric (Nonwoven)	54	66	17	0.866	2.84
PLLA[7]	Jute woven fabric (Untreated)	81.2	82.2	12.97 (KJ/m^2)	1.11	4.2
	X-Direction	70	81.5	16.2 (KJ/m^2)	0.75	3.61

Banana fiber: The fibers are extracted from the bark and mid rib of banana plant under *Musaceae* family. The plant is cultivated for the production of banana fruits. The fibers are obtained from agricultural wastes because of this banana plant cultivation plays a significant role in poverty alleviation programs. The plant has pseudo stem (bast) growing up to 8meter height. Banana fibers have around 300 types of species in the world. From these, only 20 species are effectively consumed by human being. The best quality of banana fiber is obtained when the fibers are extracted at the flowering period before giving banana fruit (Pujari 2014). The major constituents of banana fibers are cellulose (60-65 %), hemicellulose (6-19 %) and lignin (5-10 %) like other plant fiber as shown in Table 2.11. Cellulose part of banana fibers are used as reinforcing materials for

[4]LDPE –Low Density Poly Ethylene
[5] PC-Polycarbonate
[6] PP- Polypropylene
[7] PLLA- Poly-L-Lactic Acid

hemicellulose and lignin. These special characteristics make the fibers by nature are composite material. Nowadays, banana fibers are widely used as a reinforcing material in composite industries. The maximum mechanical performance, especially tensile strength was obtained when banana fibers were mixed with glass fiber with fiber content of 40% (V/V) (Pujari 2014). Lignocellulose fibers like banana fibers are of excellent interaction with phenolic matrix than glass fiber. The fiber has good thermal stability (200-700 ^{0}C) compared with most plant source fibers (Pujari 2014).

Flax Fiber/ Linen fiber: Flax is one of the oldest seed cultivated in ancient human histories. Using of flax fibers are not a new innovation. The fibers have been used from 1000 years until today. Evidences showed that the flax fibers had been used as a form of yarn and fabric in Switzerland around 800 BC (Preisner et al. 2000). Flax fibers are one of the most known fibers under bast fibers family such as flax, jute, hemp and kenaf fibers. Due to the increasing of the awareness of sustainable products and light weight, flax fiber is used as reinforcing materials in composite manufacturing industries. The fiber has good mechanical performance and cost advantage compared with glass fiber. Also, the fibers are reinforced by most type of resin such as thermoset, thermoplastic and biological resin materials without affecting the mechanical properties (Preisner et al. 2000, Zhu et al. 2013). Flax fibers are composed of polysaccharides such as cellulose, hemicellulose and pectin with phenolic lignin. Flax fibers have excellent mechanical performance and biological advantage than other bast fibers as shown in Table 2.11. Due to the presence of phenolic substrates, it has an antioxidant behaviors and mostly used as a wound dresses for wound healing (Preisner et al. 2000).

Bamboo fiber: Bamboo plant is the second large forest resource in the world. It has around 1249 type of species ranging from 11cm to 39 meter height. The plant was highly grown in subtropical and tropical climatic conditions of the world (Imadi and Mahmood 2014). China is the leading country of cultivations bamboo plant. It covers around 7.1 million hectare, that is, approximately 35% of the world's forest was covered by bamboo plants. The plant needs a minimum of one year for commercial application. In Asia histories, bamboo plants are used in hand manufacturing of papers. This situation played a major role for the production of bamboo fibers (Imadi and Mahmood 2014). The fibers are extracted from bamboo plant by chemical treatment as well as mechanical powers as

shown in Table 2.11. Bamboo fibers are characterized by their better moisture absorption, antibacterial properties, and good protection of ultra violet radiation, bio degradable and hypoallergenic properties. Recently, bamboo fibers are becoming more popular in technical textiles, green composite manufacturing and researcher centers (Imadi and Mahmood 2014).

Sisal fiber: The fibers are extracted from the leaf (well cultivated single fiber contained approximately 200 leaves) part of the sisal plants by mechanical scrapping , decorticate machine or retting with water, invented from Mexico and currently mostly grown in East Africa, India, Brazil and Haiti. The fiber is categorized under hard fibers (Joseph et al. 1999). Sisal fibers are cultivated in a short period of time in all type of climatic conditions. The sisal leaves composed of 5 % of sisal fiber, 85 % of moisture, 1 % of cuticle and 9 % of other materials. A single sisal leaf contains approximately 990 fibers. Sisal fiber has been used as reinforcing materials with thermoset polymers from 1974 in composite manufacturing industries. The fiber has good physical and mechanical properties as shown in Table 2.11. Composite structure reinforced with sisal fibers have low density, cost effectiveness and bio degradability like other natural fibers (Joseph et al. 1999).

2.4.3. Textile fabric reinforced composite material

Textile fabrics (cloth) are a flexible manmade networked structure manufactured from natural and manmade textile fibers (yarn) or from their blend with the technology (mechanism) of weaving, knitting and nonwoven as shown in Figure 2.17. The braided fabrics are also manufactured for technical applications. The physical properties and mechanical performance of textile fibers are very significantly influenced by the fiber of yarns used for fabric construction and the type of technology used as shown in Table 2.9 and Table 2.10. Woven and knitted fabrics are most widely used textile fabrics. Briefly fabric manufacturing technology were descried below (Wadje 2009).

Woven fabric reinforced composite

Weaving is a technology of cloth manufacturing mechanism in which fabrics are formed by interlacing of weft and warp yarns at the desired angles by using weaving machine the so called loom, as shown in Figure 2.17a and Table 2.13. A textile fabric produced by

interlacing of weft and warp is characterized by their high strength developed in intra-laminar and inter-laminar structures of yarns and superior resistance for damage. There are different types of woven fabric constructions. The major type of woven fabrics are plain, twill or stain/sateen based on the inclination of yarn interlacing (angles) and floating of the yarns (constructions). Woven fabrics are manufactured in the form of two or three dimensional structure (Adanur 2000, Wadje 2009).

The uses of woven fabrics as reinforcing material has a long period of time in human history. Due to their fabric construction and constituent of continuous yarns (fibers), woven fabric reinforced composite structures provide higher resistance of fatigue, good mechanical strength, creep resistance and higher stiffness (Naik 1994, Sanjay et al. 2016). Woven fabric materials have superior biaxial strength, good orientation and better distribution. Woven fabric properties are affected by physical and mechanical properties of the yarn, the count and size of yarn, density of the fabrics and crimp of the yarn in woven fabrics. These properties have a significant influence and effect in their reinforced composite materials (Misnon 2016). Layup processes of woven fabric reinforcing materials are comparatively uniform and defect free from other fabric types reinforced composite structures (Taggart and Schwan 1987, Naik 1994, Sanjay et al. 2016). These advantages attracted researchers and manufacturers in light and heavy duty composite material manufacture using woven fabrics.

Nonwoven fabric reinforced composite

Nonwoven fabrics are a type of textile materials manufactured by inter-bonding of the fibers (webs) to form inter connected fibrous structure by the help of bonding agents such as mechanical force, heat or chemical treatment as shown in Figure 2.17-f and Table 2.13 (Wadje 2009). Nowadays, nonwoven fabrics are used in modern composite manufacturing industries, especially in automobile and aircraft as acoustic areas. Different type of fibers (webs) and unique characteristics of fibers are combined with inter-bonded mechanism to get the desired properties of composite materials. Nonwoven reinforced composite manufacturing mechanisms are the most cost effective and productive fabrication processes compared with other traditional fabric reinforced

composite manufacturing techniques. These processes are most effective in multilayer composite structure fabrication (Das and Pradhan 2012).

Table 2.13. Comparison between woven and nonwoven textile fabric (Raymond 2012)

Characteristics of woven fabrics	Characteristics of nonwoven fabrics
The structure is anisotropic	It is planar isotropic
Having high strength and modulus (in fiber orientation direction)	Lower strength and modulus (in all direction)
Having poor performance in off-axis direction	Have similar performance in all direction
Lower production rate	Higher production rate
Have higher fiber volume fraction	Have lower fiber volume fraction

Knitted fabric reinforced composite

It is the form of fabric manufacturing by interloping of a single yarn in the horizontal as well as vertical directions by using knitting machine. Based on the inter looping direction of the looped structure, knitted fabrics are classified as warp and weft knitted fabric as shown in Figure 2.17c and 2.17d. The mechanical performance of knitted fabric was significantly affected by loop density and geometries as shown in Table 2.14 and Table 2.15 (Wadje 2009). Until the recent time, knitted fabrics have been not effectively utilized as composite reinforcing materials. Mostly manufactures focused on using the woven and braiding textile materials in composite fabrications (Gommers 1998, Pamuk and Çeken 2008). There are two basic reasons why knitted fabrics did not attract researchers and manufacturers for a long period of time is that knitted fabric reinforced structures cannot carry heavy loads (lose their structure) and the required level of fiber contents are not achieved in the composite structures. But for the last 10 years, these two assumptions are disproved by various research work and investigators (Gommers 1998). Knitted fabric manufacturing is one of the most versatile techniques for the textile fabrics fabrication processes. This is one of the most desirable properties for textile materials used as reinforcing structures. Weft and warp kind of knitted fabric structures are used as a reinforcing materials as shown in Table 2.14. For knitted fabric reinforced composite, the knitted fabric structures are selected by three major criteria, the first criteria is knitted structure deformations, the second criteria is that, physical and mechanical properties of the knitted fabrics and the third selection criteria is the curl nature of knitted structure (Gommers 1998). Composite materials reinforced by knitted fabrics have special

properties and advantages: (i) knitted fabrics having super drapability and deformation properties help for the formation of complex and interacted shapes without creating of folds (ii) advanced knitting machines are used for manufacturing the desired shapes (iii) their versatility (Pamuk and Çeken 2008). These major properties have played a significant role for the overall properties of composite materials reinforced by knitted fabrics, to have better impact resistance, flexibility, excellent inter-laminar performance, lower fabrication periods and high fabrication rates (Gommers 1998, Pamuk and Çeken 2008).

Table 2.14. Characteristics of weft and warp knitting fabric (Hossen U. & Textile Fashion Study 2012)

Weft knitted fabrics	**Warp knitted fabrics**
Threads are run in vertical direction	Threads are runs in horizontal direction
Highly elongation	Less elongation
Required one yarn	One thread is required for each needle
The course are equal to the pattern	Higher course are need for a pattern
Elongated in width direction	Elongated lengthwise
Appropriate for thin fabric production	Appropriate for fabrication of course materials
Has a Problem of shrinkage	Good shrinkage resistance
Thread is feed from a single cone	Yarns are feed from beams

Braided fabric reinforced composite

It is the simplest way of fabric manufacturing without using weft insertion mechanism like weaving technology. A number of threads are interwoven one with the other in diagonal manner. Mostly braiding is used for the fabrication of narrow and circular shape structures. It has stable structural construction as compared with woven and knitted fabrics as shown in Table 2.15 and Figure 2.17-b (Wadje 2009). Braiding manufacture is an ancient type rope and carpet fabrication technique. The fabric manufacturing mechanism is very interesting and attracted researchers in composite industries because of unique characteristics such as excellent orientation of multi-axial, superior tolerance for damage, versatile and cost effectiveness. Braiding techniques are used to produce circular shape by using biaxial yarns Moreover, braiding structures are convenient for complex shape manufacturing with superior off axis balance in composite structures manufacturing (Pamuk and Çeken 2008).

Table 2.15. Characteristics of woven, knitted and braided textile fabric (Raymond 2012)

Properties	**Woven structure**	**Knitted structure**	**Braided structure**
Orientation of the fiber	Orthogonal	Varies	Varies
Dimensional stability	Good	Poor	Poor
Structural stability	Poor	Moderate	Good
Productivities	Higher for 2D Lowe for 3D	Higher	Higher for 2D Lowe for 3D

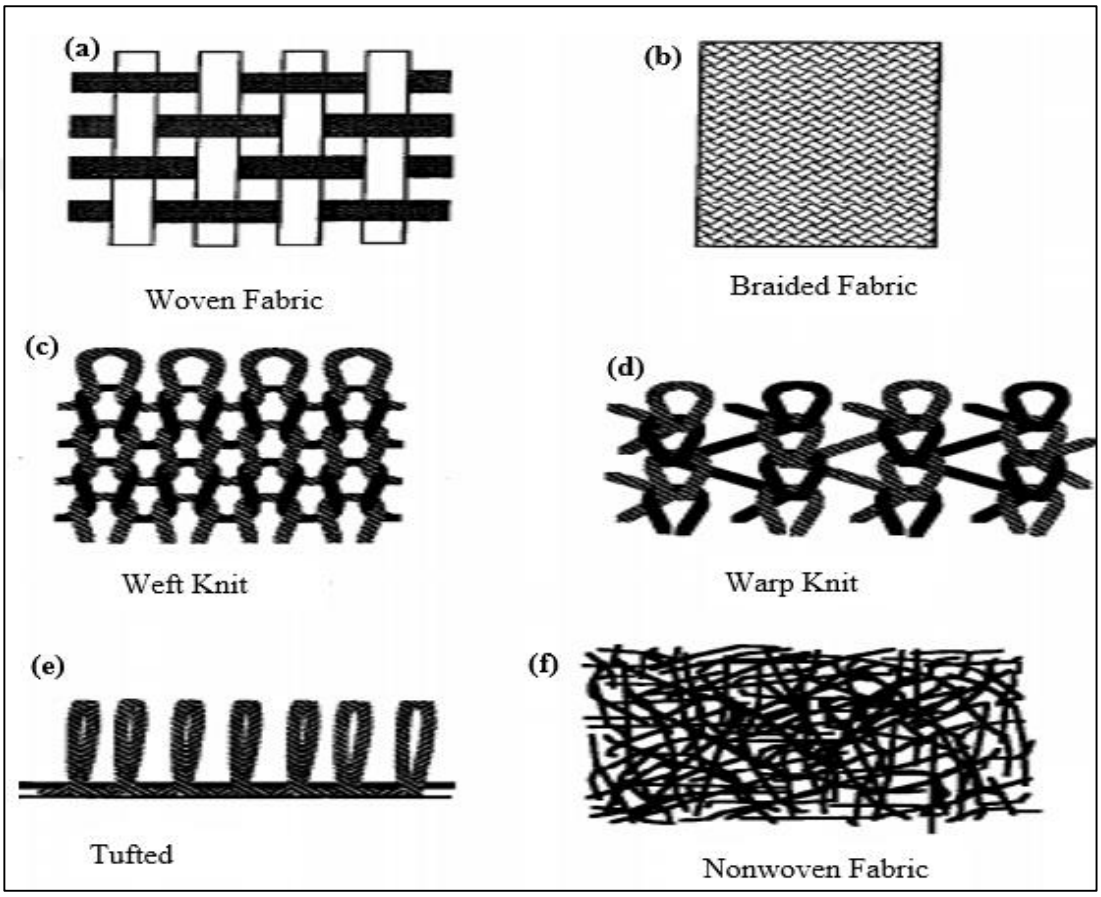


Figure 2. 17. Type of textile fabrics used in composite manufacturing (Sabit-Adanur 2000)[8]

Multilayer (3-Diamensional) fabric reinforced composite

The first textile fabric reinforced composite material was fabricated by layering of clothes, the so called 2D laminated composite structure. This type of composite manufacturing is wasting time, inefficient and limitation of mechanical performance in

[8] Hand book of weaving

the planar direction. Researchers and manufacturers have been tied to solve the limitations of 2D (two dimensional) composite by developing 3D (three dimensional) structure from textile materials as shown in Figure 2.18 (Fredrik 2016, Abbasali 2019).

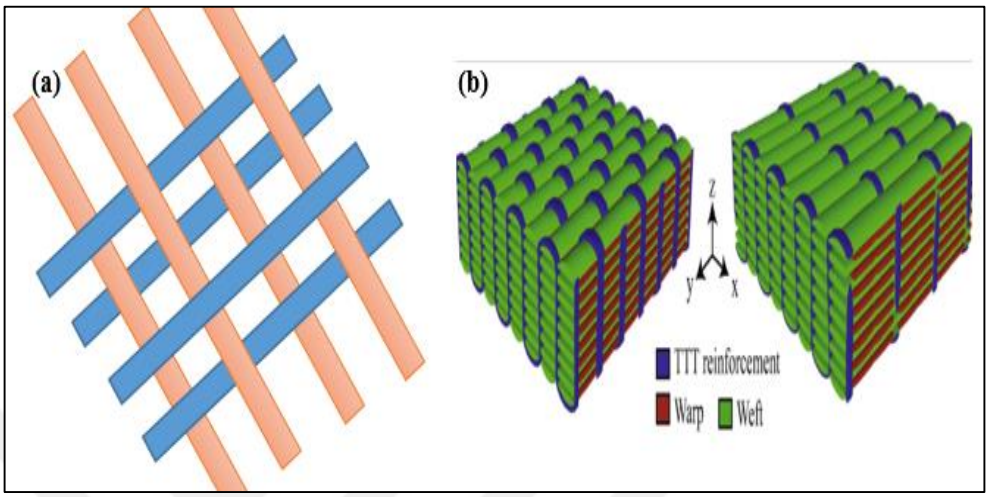


Figure 2.18. Fabrics structural molding a) 2D fabrics b) Multilayer fabric (Turmer et al. 2016)

3D textiles are materials having three dimensional structures with superior though thickness and in plane performance, which are manufactured in the form of woven, knitted, nonwoven and braiding structures (Mouritz 1997, Gopinath et al. 2014) as shown in Figure 2.19. The 3D textile materials are formed by mutual integrity in all the three directions of the fabric. This interaction gives higher strength and stiffness for the final products. The major driving factors for the development of three dimensional composite structures are: enhancing the mechanical performance through the thickness of composites, having neat shapes, minimize the manufacturing cost and enhancing the impact damage resistance. 3D textile materials reinforced composite was seriously studied by the National Aeronautics and Space Administration (NASA). Moreover other sectors such as automobile, marine and construction emphasized on the development of these areas (Fredrik 2016, Abbasali 2019). 3D textile structures are by themselves mold structure, so 3D materials can be used as beam without requiring mold materials, these characters are significantly reduces the cost and duration of composite structure manufacturing (Abbasali 2019).

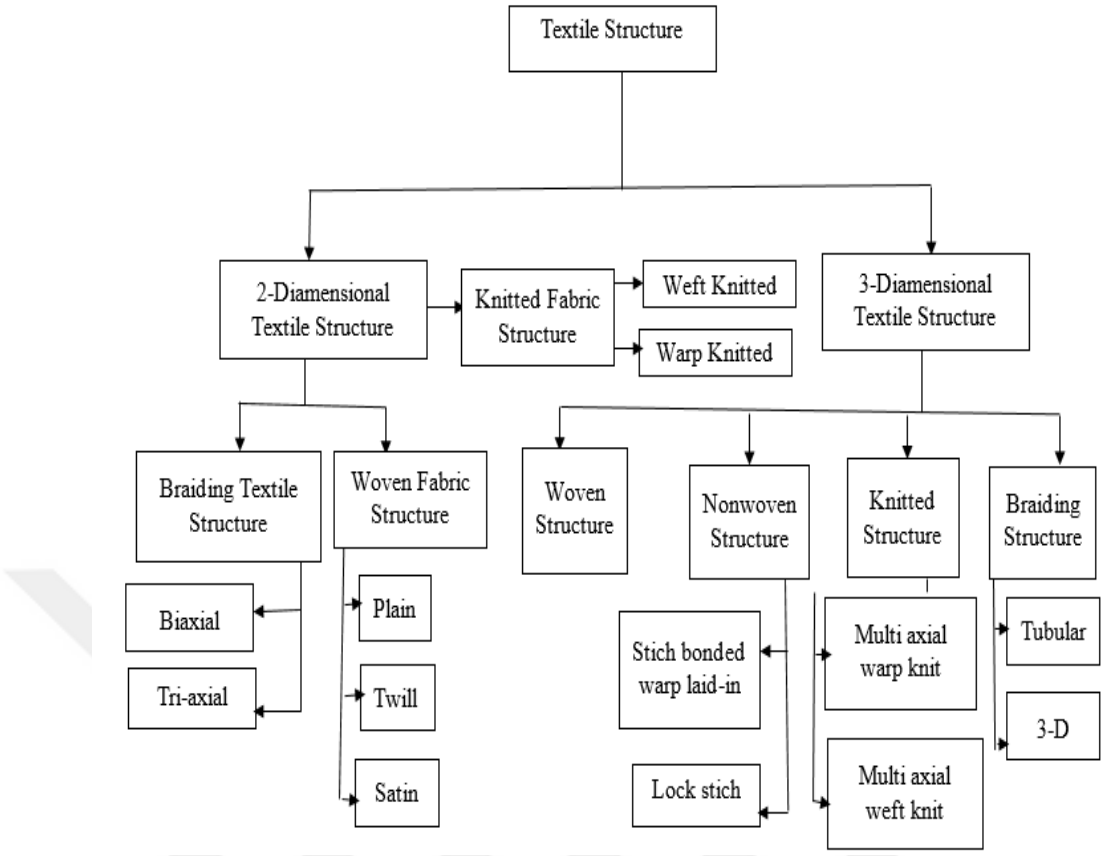


Figure 2.19. Major classification of textiles structure (Adapted from Horrocks 2000)

2.5. Merits and Limitation of Textile Materials Reinforced Composite

Composite materials have their own advantages and limitations like other traditional engineering materials. The basic aim of composite manufacturing is to reduce the weight of the composite materials. The lightening of composite structural elements has a very significant benefits in transportation, especially automobile and aircraft industries. Because, the reduction of weight of the vehicles as well as the planes results the reduction of fuel consumption (Jin 2011). For instance, when a vehicle incorporates composite in its structural elements, its weight is approximately reduced by 40 % compared with a car manufactured from Aluminum and by 60 % when it was compared with steal based fabricated cars (Gopinath et al. 2014). Moreover composite materials are not easily affected by chemicals and not corroded like other traditional engineering materials. Due to their design flexibility, composite materials manufacturing processes are cheap for mass production. The desired signals are transmitted through composite structures, this

help for future advancement of radar industries. For last few years, researchers and manufacturers have started to manufacture ecofriendly composite materials. This plays a very significant role in the reduction of global warming and the consumption of petrochemical products. In contrast, composite materials have their own limitations. The initial capital costs of composite manufacturing are very expensive. Also, when cracking or breakings of composite structures occurred, the inspection as well as maintenance of the composite material is very difficult and expensive. Mostly used matrix materials are sensitive to temperatures (working below 300 ^{0}C). Most commercially used matrices materials are not environmentally friendly, they affect human health during manufacturing, usage and their disposals (Piyoosh et al. 2013, Mitra 2014, Koronis 2016, Yıldızhan et al. 2018).

2.6. Bio Polymer Materials

Polymers which are biological degraded as well as compostable are categorized under a biopolymer materials. Biopolymers are either natural or manmade polymers. Generally, bio based polymers are obtained from three major sources such as cellulose, synthetic polymer and protein. The development and improving of the properties and performance of renewable bio polymers, especial in bio resin polymers manufacturing sectors needs a lot of research works (Mitra 2014, Koronis 2016, Yıldızhan et al. 2018). But in recently time, there are so many research works were actively conducted to enhance the mechanical performance and increase the production rate (massive production) of bio matrices with reasonable costs as shown in Table 2.16. Renewable matrices are manufactured by three major techniques:-

(i). By modifying natural existing resource like cellulose, the starch materials are modified into thermoplastic starch.

(ii). By polymerization of monomer obtained from bio based polymers like PLA.

(iii). Bio polymers are produced by genetic modification of plants or micro-organisms like PHA.

Generally bio based matrices play a very significant role for the development of chemical industries and give a merits of minimization of global warming, waste materials disposal,

reduce fuel consumption and additional income generate for poor people in agricultural areas, because waste materials are sold and used as industrial raw materials (Mitra 2014, Koronis 2016).

Table 2.16. Classification of renewable polymer materials based on their source (Mitra 2014)

Manufactured bio polymers	Source of natural bio-polymer	Natural bio polymer
PVA(polyvinyl acetate) Poly amide Polyvinyl alcohol Poly ester Polyethylene oxide	Animal source (Protein base)	Grains of proteins Gelatin of collagen Casein of silk
	Plant source(Polysaccharides)	Cellulose and their derivatives chitins starches Rubber
	Another polymer sources	Cashew nut liquid Lignin
	Manmade sources(poly ester)	Poly(hydroxylalkanoate)

2.7. Green Composite Material

The word green/ecofriendly/bio composite are the processes of manufacturing of composite materials by using entirely degraded as well as compostable reinforced and matrices materials (Koronis 2016). It is a composite structure manufactured from biodegradable reinforced materials (natural fiber) such as jute, banana, sisal, hemp etc. and bio resin such as starch, soya, PLA etc. (Mitra 2014, Koronis 2016, Yıldızhan et al. 2018). Considerable research works have been undertaken on green composites by using natural fibers as a reinforcing materials and bio based polymer materials obtained from PLA, starch, PCL, Poly-Hydroxy-Butyratehydroxyl-Valerate (PHBV). Green composite materials have lower mechanical strength (mostly they have a tensile strength of less than 100MPa) as compared with conventional composite and traditional materials (Mitra 2014). The word green/ bio / eco-friendly is generally used to include composite materials manufactured from at least one of the component bio degradable (bio polymer) such as:-

(i). Both of them are bio based polymers, i.e. the matrix element is obtained from bio based polymer like PLA as bio resin and natural fibers as a reinforcement.

(ii). When natural fibers are used as reinforcement and petroleum based resins are used as matrix such as epoxy and polyester.

(iii). When carbon and glass fibers are used as reinforcement and bio based resins are used as matrix (Mitra 2014).

Currently, there is a serious environmental problems and challenging issues on the consumption of petrochemical materials in world's manufacturing industries. The researchers and manufactures have tried to solve these problems and manufacturing sustainable products with affordable cost for the last few years. Green composite materials are characterized by their biodegradable as well as compostable features. The products have a promising future in bio material technology by substituting petrochemical base materials as shown in Table 2.17. Moreover, green composite materials have light weight, sustainability, moderate mechanical strength and eco friendliness during production usage as well as their disposal (Yıldızhan et al. 2018). However, biodegradable composites have certain limitations. The limitations of green composite materials are coming from their reinforcing natural fibers as well as bio matrices materials. The properties of natural fibers are affected by soil types, climatic conditions, season and geography. These factors have a significant influence on the variation of fiber properties and having non-uniformity with the fibers, even in a single fiber. Due to these, green composite materials have a major problem in uniformity of their structures. Natural fibers are hygroscopic in nature, especially plant fibers absorb moisture and chemicals easily, and these affect the interface region of the composite structure (weak interaction between reinforcing fibers and the matrix materials). Moreover the duet swelling of the fibers, the composite material show volumetric changes in its structure and result in crack formation of composite material under moisture conditions. Recently, researchers have tried to overcome the drawbacks of green composite materials by treating the fibers with alkali and other water replant materials before the composite structures have been manufactured (Mitra 2014, Koronis 2016, Yıldızhan et al. 2018). For the past 10 years, researchers have been studied widely PLA, protein and starch base biodegradable composite materials because of their biodegradability and comparatively simple process ability in composite manufacturing (Kargarzadeh et al. 2018). According to Wang and Drzal (2012) report, the bio-composite material manufactured by using cellulose

nanofibrils (CNF) as reinforcement and PLA as bio resins, at higher level of PLA loading rate (32 % wt.) caused the tensile strength and modulus of the composite structure to improve almost by 200 % and 60 % respectively (Wang and Drzal 2012). While Mehdi et al. (2010) was reported that increasing the amount of CNF by 5 wt. % in CNF-PLA composite was decreased the biocompsoites structure stain from 3.4 % to 2.7%. Likewise, Dhar et al (2016) reported the effect of non-compatibility between the CNC (cellulose nanocrystals) reinforcement and PLA bio resins on the tensile strength and modulus of the biodegradable composite materials. Polylactic acid is a synthesized biopolymer reins having same limitations such as moisture barrier, brittle and distortion with heat during composite manufacturing, which significant affects the mechanical strength of the composite structure and created unsuitable between the reinforcing and resins materials. Because of this limitation, it was limited on the fabrication of commodity goods. Moreover, due to the higher intermolecular hydrogen bond and agglomeration created in melt extrusion process of CNF and PLA resin, the manufacturing of composite materials by CNF/PLA are becoming difficult and a challenging manufacturing processes (Dhar et al. 2016). According the this study, the compatibility between the reinforcing (hydrophilic) and bio resin (hydrophobic) materials was reduced by adding a crosslinking agent such as Dicumyl Peroxide to enhance the grafting efficiency of PLA and improve the tensile strength and modulus of green composite materials by 40 and 490 % respectively (Dhar et al. 2016). According to Lu et al. (2005) report, the cotton base CNC reinforced green composite material via starch resin was studied. As stated by this report, using of starch bio resin (up to 40 wt. %) improved the tensile strength and modulus of the green composite material by about 200 and 700% respectively (Lu et al. 2005). Wang et al. (2006) and Li et al 2012 reported that most commonly used protein matrix materials such as soybean and silkworm fibroin (with the help of Polyethylene glycol, PEG) create a crosslinked network with the reinforcing structure, which help to develop intermolecular hydrogen bond between the protein matrix and the reinforcing materials (Wang et al. 2006, Li et al. 2012). According to the researchers cited above, adding 20 wt. % of reinforcing material in protein based matrix (soybean) improved the tensile strength from 5.7 MPa into 8.0 MPa and modulus 44MPa in to 130 MPa (Li et al. 2012). Nam et al. (2012) studied jute fiber reinforced green composites. The research work reported the effect of the amount of fiber loading (fiber contents) and poly butylene

succinate resin on the thermal and mechanical properties of the biodegradable composite (Nam et al. 2012). The tensile strength was improved from 66 to 157 MPa when the fiber content was increased from 10 to 50wt. %. Also, the flexural strength was also improved from 70 to 185 MPa. While adding the fiber content beyond the optimum level i.e. 60 wt. %, both the tensile and the flexural strength was decreased from 157 to 147 MPa and 185 to 150MPa (Nam et al. 2012).

Table 2.17. Properties of commonly used thermoset, thermoplastic and bio resins in green composite (Shah et al. 2016)

Type of resins		**Properties**				
		Density (g/cm^3)	**Tensile strength (MPa)**	**Young's modulus (GPa)**	**Elongation (%)**	**Reference**
	Polyester	10.3-10.61	24-68	1-2.4	3.9-5.1	Holbery and Houston 2006
Thermoplastic	PP	0.89-0.91	25-41	14-690	0.94-1.76	
	HDPE	0.93-0.95	14.4-37	2-125	0.4-1.4	
	Polyvinyl Chloride	1.41-1.80	13-51	19-39	1.1-3.6	MatWbe 2015
Thermoset	Polyester	1.2-1.49	30	2.0	3.11	Gere 2008
	Epoxy	1.11-1.39	34-100	1.1-6.1	2.9-5.9	Holbery and Houston 2006
Bio resins	PLA	1.25	24-29	1.79	2.33	Facca et al. 2007
	Elastomer	0.9-0.92	19-30	745-850	0.001-0.0049	Matbase 2015

2.8. Sound Absorption Characterization of Textile Materials

Human ears are not sensitive to all ranges of sound frequencies. Based on sound frequency and intensity, all the coming sound might not be audible to human ears. The audible sound by human being have a frequent range of 20 to 20000 Hz (Özdil et al. 2020). Excess interior noise have significantly affected human comfort and it might cause severe health problems (Jichun et al. 2018). These problems have been effectively solved by using sound absorbing structures. The fast development of urbanization and transport sectors have increased the demand and improvement of interior sound absorption materials (Leitao et al. 2018). Commonly, the noise absorption by using sound absorption materials could be done by two major approaches, i.e. by porous and resonant sound

absorption structures (Yonghua et al. 2014, Jichun et al. 2018, Leitao et al. 2018). Porous textile materials are materials which are composited of cracks, channels, pores, cavities or/and voids that permit the sound energy (waves) through their structures. In this mechanism, the coming sound waves are collapsed with the air molecules entrapped in the porous and void walls of textile materials developing frictions and then dissipated in the form of thermal loss as shown in Figure 2.20 (Xiao-Dan et al. 2016, Jichun et al. 2018, Leitao et al. 2018). While, resonant sound absorption mechanism are different from the sound absorption mechanism of porous materials. Resonant sound absorber materials are commonly found in two types: Helmholtz absorbers and panel/membrane absorbers. The working principles of resonance sound absorbers are based on the mechanism of internal resonance effects at lower frequency. In resonant sound absorber materials, the maximum sound absorptions are only influenced by the flow resistance of the sound absorber. It does not affected by the physical dimension of sound absorber such as diameter, depth of airgap and amount of open space (Xiao-Dan et al. 2016, Jichun et al. 2018, Leitao et al. 2018).

Recently, due to their porous and void structure of textiles like natural fibers, non-woven material, woven fabrics and textile base recycled materials have been extensively utilized for sound absorption and/or sound insulations (Özdil et al. 2020). The textile materials have been used as noise barrier, sound absorber, sound reflector and/or sound diffusers (Özdil et al. 2017).

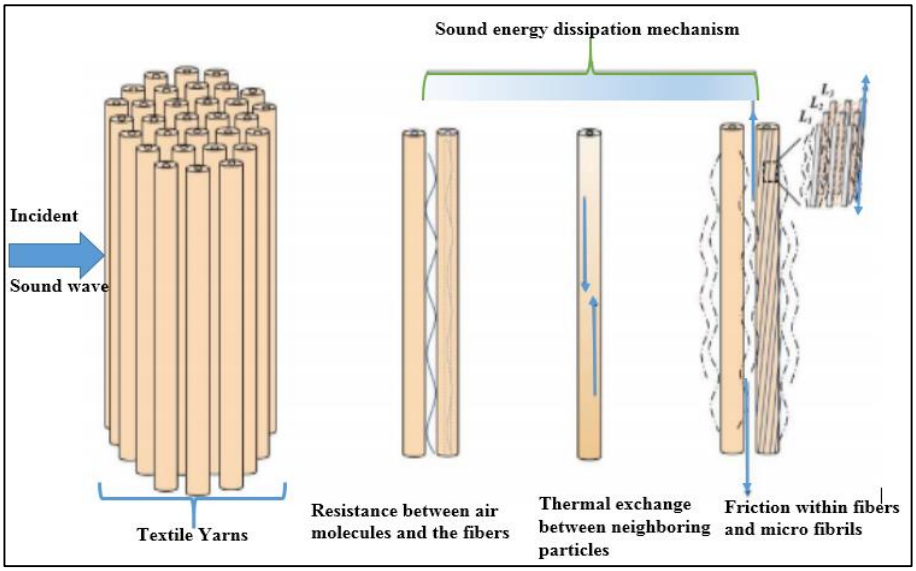


Figure 2.20. Sound energy dissipation mechanism of porous textile material (Jichun et al. 2018)

Natural plant fibers like flax, jute, sisal are ecofriendly multiscale microstructure materials widely used as sound absorber. Nowadays, these natural sound absorbing resources are extensively considered as a choice in most research centers and manufacturing sectors (Zheng et al. 2013, Leitao et al. 2018). According to Zheng and Li (2013), plant fiber reinforced composite sandwich materials have better sound absorption performance than glass fiber reinforced composites. Also, according to Prabhakaran et al. (2014) and Tang and Yan (2017) report, natural fiber reinforced composite materials have limited sound absorption properties but most researchers are studied and confirmed that plant fiber reinforced composite structures have better sound absorption coefficient than synthetic fiber reinforced composite material at a frequency of 1000-2000 Hz (Zheng et al. 2013).Because of the contribution of multiscale and pours structures on their morphology which are helping to attenuate and dissipate the coming sound energy (Zheng et al. 2013, Yonghua et al. 2014). Moreover, plant porous fiber based sound absorbing materials have a lot of advantages such as low cost, light weight and wide absorption range (Sargianis et al. 2013, Zheng et al. 2013, Yonghua et al. 2014, Leitao et al. 2018). The possibility of sound absorption of enset natural fiber is investigated as a part of this thesis as it has similar properties to other cellulosic natural fibers.

3. MATERIALS and METHODOLOGY

3.1 Materials

Enset fibers are extracted from mid ribs and bast of the plant by mechanically decortication and retting of the leaves/bast with continuously scratching by hand followed by washing, cleaning, fiber separation. Finally the fibers are converted into enset yarns by jute spinning machines in industry. Enset fabric used in this research was bought from Addis Ababa, Ethiopia. It has plain woven construction (the warp is 74 Tex and weft is 67 Tex). Enset woven fabric gives an advantage for the enset fabric reinforced composite structure in terms of dimensional stability and offers a good structural and mechanical strength. Enset woven fabrics are considered as reinforcing materials in this thesis because of their higher specific strength, flexibility, interlacing of yarns (give better strength for the structures than fibers) and formability properties (Salman et al. 2015). Acacia tortilis and frankincense natural gums were collected, cleaned and prepared as matrices materials. Enset fabric was used as reinforcing material for manufacturing of green composite structure. Acacia tortilis and frankincense bio resins were used as matrix in this thesis work.

3.1.1. Enset fibers as reinforcement material[9]

Enset is the name used by the Amharic language for Enset plant which is scientifically called *Ensete Ventricosum* under *Musaceae*. Enset plant is the most abundantly available pseudo stem plant in Ethiopia as shown in Figure 3.1. The variety of the enset plant distribution is characterized by a lot of factors related to climatic conditions, culture and geographical location. Around 12 million people use this plant as source of food in Ethiopia, especially in the southern and southwest part of Ethiopia. The fibers are obtained from the agricultural waste material by mechanically decortication and manual extraction of the food content from the plants. Fibers have an average equivalent diameter of 194-206 μm and a length of 0.5 to 4.5 m. These long technical fibers are generally

[9] Potential of Ethiopian Enset Fiber for Textile Application. 6th International Fiber and Polymer Research Symposium (6th IF&PRS), Book of Proceedings, 24-25 Journey 2020, Bursa, Pp. 63-66. www.uludag.edu.tr/ulpas.

characterized by hard, stiff and coarse in texture. Moreover, the fibers are also characterized by low cost, light weight, natural existence as composite fiber and good mechanical strength (seen in Table 3.1). The fibers (for example flax) can also be used as a source for micro and/or nano fiber generation by mechanical forcing after enzyme treatment (Fekadu and Ledin 1997, Birmeta 2004).



Figure 3.1. a) Enset plant b) Enset fiber

Morphological properties of enset fiber

Enset fibers have white to yellowish color with a hard touch morphological structure due to the existence of cellulose, hemi cellulose and lignin on their surface in addition to bark. The longitudinal streaks can also be observed in the structure of enset fiber which are characteristics of long plant fibers having consecutive layered structure with a lot of sectional regions as shown in Figure 3.2a and 3.2b. By examining these fibers in SEM, one can observe that the fiber is naturally a composite structured having consecutive layers which are connected by hemicellulose and lignin.

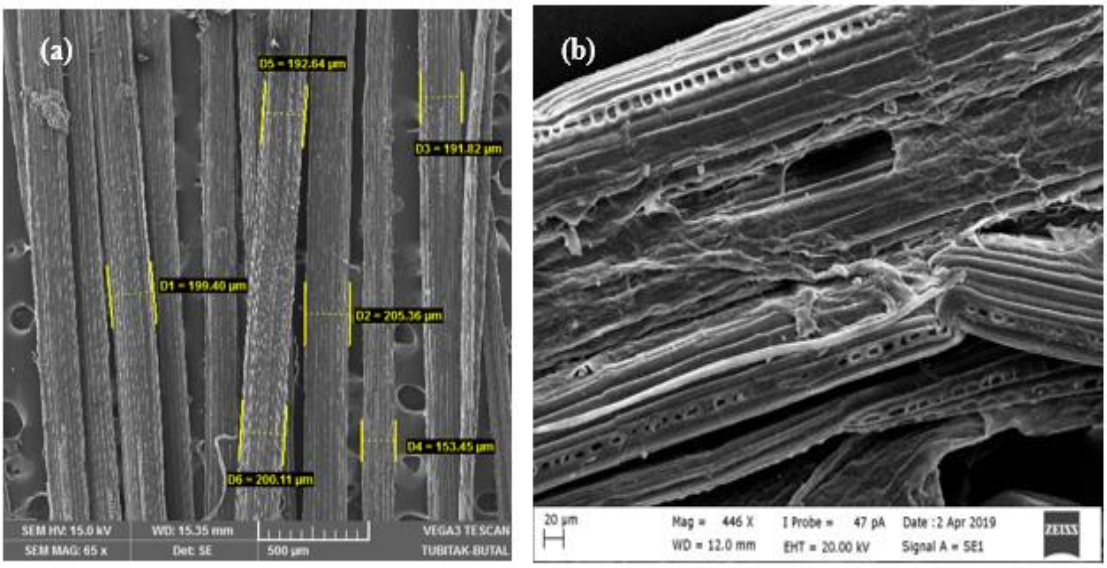


Figure 3.2. SEM view of enset fiber[10] a) Longitudinal view b) Naturally layered nature of single enset fiber (composite)

Chemical composition of enset fiber

The FTIR graph was used to characterize the chemical composition of the enset fibers. The major chemical compositions of enset fibers are cellulose, hemicellulose and lignin as shown in Table 3.2 and Figure 3.3. The percentage of cellulose, hemicellulose and lignin of enset fibers varies from place to place, season to season or in different geographical locations.

[10] Bursa Test and Analysis Laboratory (BUTAL) Part of this Thesis

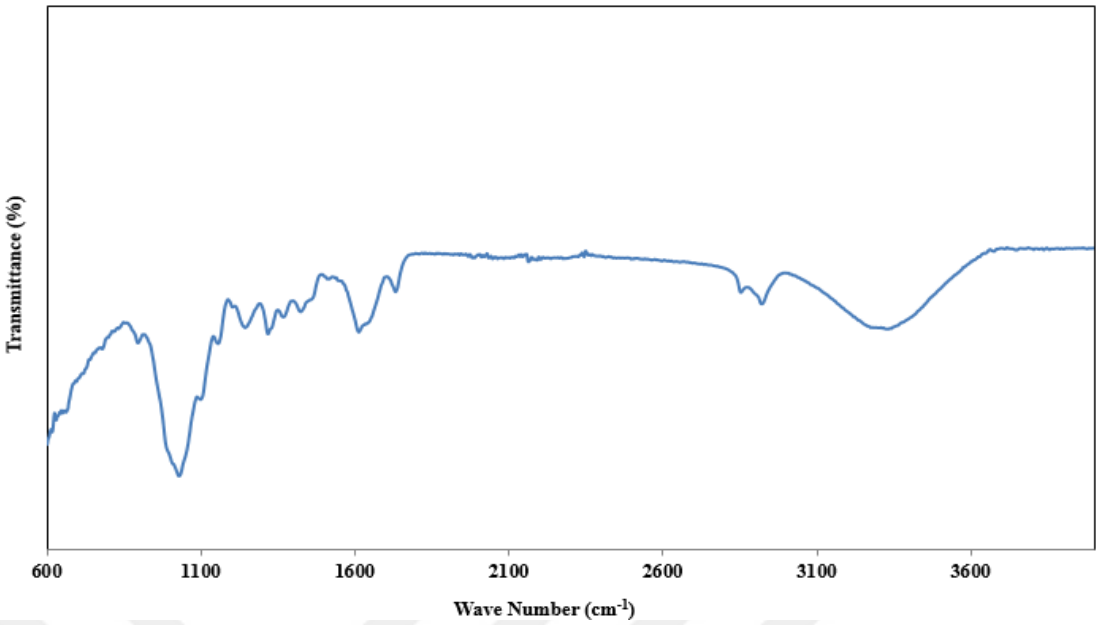


Figure 3.3. FTIR adsorption bands of enset fiber[11]

Mechanical properties of enset fiber

Like other lignocellulose fibers, mechanical and the physical performance of the fiber as well as the bio composite reinforced structure with the enset fiber will be significantly affected by the major constituents, especially the percentage of hemicellulose and lignin present in the enset fiber. Compared to other fibers from the same family, like banana fiber, enset fibers are light weight with a bulk density equal to 1099-1200 kg/m^3 measured at 20 °C ± 2 (Borrell et al. 2019). Also in comparison with other textile fibers, the enset fibers are more hydrophilic than cotton, flax, and other lignocellulose vegetable fibers with moisture regain up to 10%. At this point, they are comparable to jute and banana fibers. Due to the anisotropic nature of the fibers, the elongation of the fiber varies from 4 up to 12 %. This indicates that the mechanical and physical properties of enset fiber mostly deepened dependent on the lignocellulose content of the enset plants. Since enset fibers have good aspect ratio and it was the driving factor to use it as a new alternative reinforcement fiber for natural reinforced green composite structures. For a single fiber, the top and down tip of fiber have inferior quality of strength but the other middle parts have good mechanical performance.

[11] Bursa Technique University Laboratory (BIU) part of this Thesis

Table 3.1. Physical properties of the enset fibers[12]

Fiber	Dia.(μm)	Length (m)	Aspect ratio (l/d)	Bulk density (kg/m^3)	Tensile strength (MPa)	Max. elongation (mm)	Max. time fiber breakage (Sec)
Enset fiber	150 -206	1.5-3.5	170	1099-1200	350-500	4-12	1.6836

Comparison of enset and banana plant fiber

The physical structures of banana and enset plant fibers resemble each other but in reality as shown in Table 3.2 below, the physical as well as the mechanical properties of these fibers are different (Arun 2000, Yang et al. 2007). The lignocellulose content of enset plant was less than banana. Due to this, the enset fiber has high specific strength and modules than banana fiber. But lignin content is much greater in banana and because of this, the banana fiber has more amorphous area in its structure. The minimum moisture content (10-15.2 %)[13] and regain (12.15 %) [3] values of the banana fiber are mostly greater than the maximum moisture content (7.2-10.85 %) and regain values (10.5 %) of enset fiber. These moisture content differences have a significant effect on the overall mechanical performance of the fibers when used as reinforcement in composite structures. The enset had a significantly shorter and thicker pseudo stem structure compared to banana plant. Because of this, the enset plant gives large amount of fiber per single plant than banana (Arun 2000).

[12] Bursa Test and Analysis Laboratory (BUTAL), Bursa Technique University (BTU) and Bursa Uludag University (BUU).
[13] Part of this Thesis work.

Table 3.2. General properties of enset and banana fiber[14]

Parameters	Enset fiber	Banana fiber	Reference
Cellulose (%)	65- 69	61–66	
Hemicellulose (%)	8-18	5–14	
Lignin (%)	5-6	4–18	Arun 2000,
Ash (%)	Up to 3	5	Pujari 2014
Moisture content (%)	7 - 11 (11)[15]	11-15	
Moisture regain (%)	8-10 (10)[4]	12	
Flexural rigidity (Ncm^2)	1.2-2	3-5	
Tensile strength (N)	9-13[16]	8-10	
Elongation at break (%)	2-3.2 (3.2)	3	

3.1.2. Bio matrices[17]

Green matrix materials are made from renewable and naturally existing materials. The manufacturing of bio based polymeric matrices materials are either fully degradable or compostable though times. They are sustainable materials and play a very significant role for the reduction of global warning and ecological problems. However, polymer materials that have got ecologically acceptance are not necessary to be biologically degraded as well as compostable. If the polymer materials reduce the consumption of fuel during their resin production, low waste disposal and minimum greenhouse emission, these polymer materials are considered to be ecological acceptance polymers such as bio polymer matrices obtained from petrochemical materials (Nikola et al. 2012).

Acacia tortilis resins and their characterization

Acacia tortilis gum is naturally extracted plant secretion gum obtained from acacia tree under the leguminous families as shown in Figure 3.4, which are generally grown and

[14] TEMESGEN, A.G., EREN, R., AYKUT, Y. 2019, Investigation and Characterization of Fine Fiber from Enset Plant for Biodegradable Composites, 17th National 3rd International the Recent Progress Symposium on Textile Technology and Chemistry, 20-23 November, 2019, Bursa, Turkey, pp: 356-361. ISBN No.978-605-01-1312-9.
[15] Bursa Test and Analysis Laboratory (BUTAL).
[16] Bursa Uludag University (BUU).
[17] TEMESGEN, A.G. TURŞUCULAR, Ö.F., EREN, R., AYKUT, Y. 2020. Potential Application of Bio Resin in Green Composite. 7th International Fiber and Polymer Research Symposium, 25-26 September, 2020, Bursa, Turkey, pp: 72-77. (Book of Proceedings).www.uludag.edu.tr/ULPAS.

native in Africa, especially in Sudan and Ethiopia (Mariana and Montenegro 2012, Gashua 2016, Sarkar et al. 2018). The trees commonly have size of 5 to 13 meter height. Occasionally their high might reach to 18 and 21 meters in shrub or bush (Mariana and Montenegro 2012).

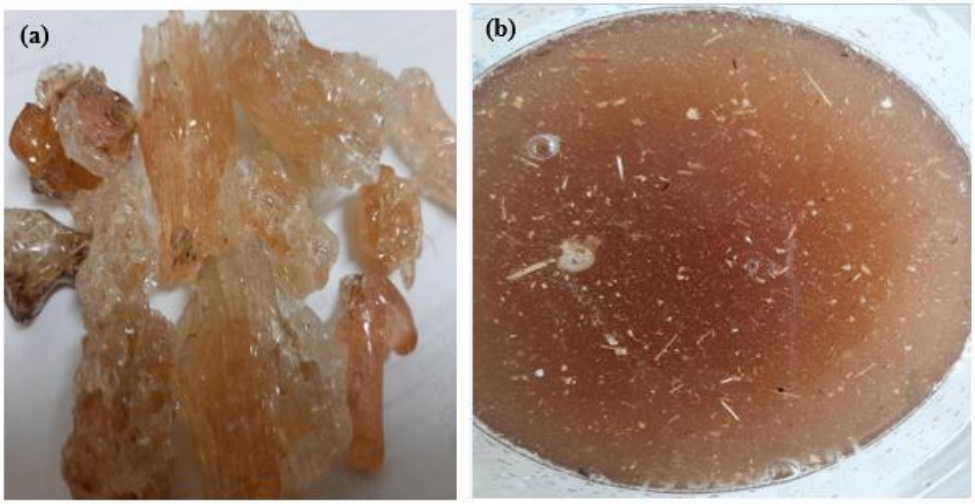


Figure 3.4. a) Granules of acacia tortilis gum b) Acacia gum's bio resin

The chemical composition of acacia gums is a mixture of different complex hetero-polysaccharides compositions and of different size of constituents such as L-arabinose, 1-3-galactopyranosyl and glucuronic acid (Mariana and Montenegro 2012, Patel and Goyal 2015). It contains around 97% of carbohydrate and 3% of protein (Islam et al. 1997). Naturally, it has slightly acidic properties (Patel and Goyal 2015). The composition of acacia gum is not affected by its climatic conditions, agricultural activities, age of the tree and processing like dying and spraying (Assaf et al. 2005, Hassan et al. 2005). The gum is a viscous polysaccharide (bio-polymer) material used as a traditional binder and emulsifier agents by ancient Egyptians before the 2^{nd} millennium BC. (Mariana and Montenegro 2012). Recently the gums have been widely used in most industrial application like textile, food and cosmetic (Mariana and Montenegro 2012). The FTIR spectra of acacia gum is shown in Figure 3.5. The graph has revealed that, the O-H groups and the sugar (galactose and rhamnose) were observed at absorption band of 3300 cm^{-1} and 2927 cm^{-1} respectively. The gums have also shown the aromatic (C=C stretch), aliphatic and amino acids with the wave length of 1300-1600 cm^{-1}. At the

absorption band of 1260 cm^{-1}, C-O stretching alcohol was observed (Sunita 2016, Patel and Goyal 2015).

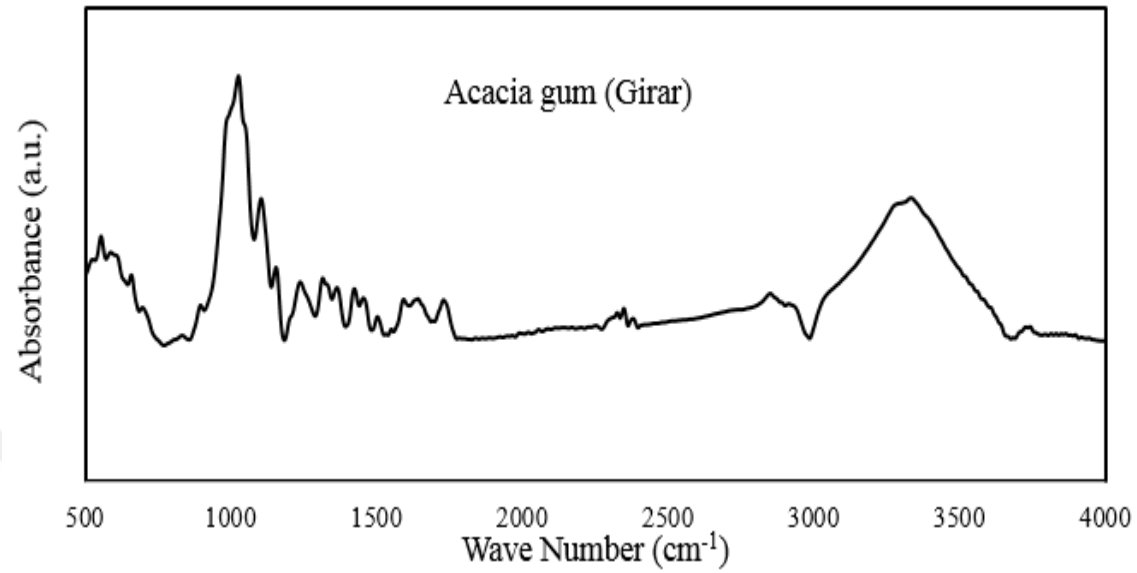


Figure 3.5. FTIR of acacia tortilis gum

Boswellia papyruses (Frankincense) resins and their characterization

Boswellia or frankincense is one of the natural gum under *Burseraceae* family, which is mostly used as an incense in regional ceremony and traditional medical applications (Moussaieff and Mechoulam 2009). The granules gum is extracted from the frankincense tree, which is grown in the dry region of most India and Africa such as Ethiopia, Sudan and Somalia as shown in Figure 3.6a (Dharmananda 2003, Moussaieff and Mechoulam 2009). The bio resin prepared from this gum is shown in Figure 3.6b. The basic constituents of frankincense are B-boswellic acid, acetate or oxide derivatives of incensole as shown in Figure 3.6c. The FTIR observation of Frankincense gum revealed that O-H stretching and C=O band was observed at 3440 cm^{-1} and 1650 cm^{-1} respectively. Likewise, the CH_3O and CH_3CO groups were observed at the peaks of 1730 cm^{-1} and 1460 cm^{-1} respectively. The presence of ester group in bio gum was revealed at the peak of 1030 cm^{-1} (Moussaieff and Mechoulam 2009, Rehman et al. 2020). Ethiopian frankincense is one of the most proper type of tropical frankincense with aromatic nature. Frankincense has been used from 11^{th} century by Persian in traditional medicine as antimicrobial materials (Moussaieff and Mechoulam 2009, De-Rapper et al. 2012).

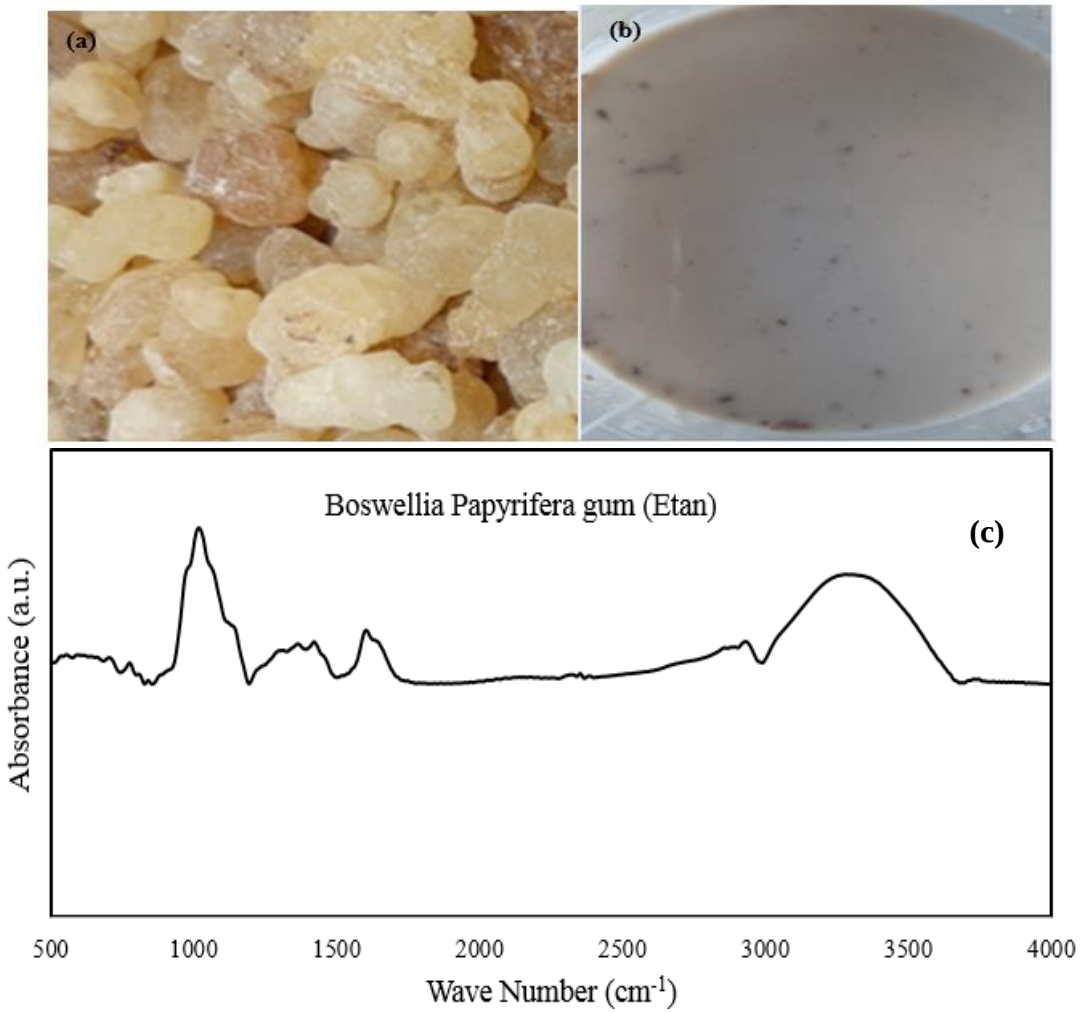


Figure 3.6. a) Granules of frankincense gum b) Frankincense gum's bio resin c) FTIR of frankincense gum

3.2. Methodology

3.2.1. Preparation of bio matrices from acacia and frankincense gum

In this thesis, green based matrices were prepared from the plant secretions gum of Acacia and Frankincense trees. Since these bio gums were directly used as a matrix materials for the manufacturing of green composite structures, the characterization of acacia and frankincense such as moisture content (Memmert-Modell 100-800, FAO 1990), solubility (T207om-93, ASTM D1110-84), viscosity (ASTM D 4603), Molecular weight (Mark 1938, Houwink 1940) and percentage of ash (ASTM D2584, D5630, ISO 3451) were a compulsory duties of this research work based on the international standard seen Table

3.3. The bio resin used for the fabrication of enset fabric reinforced green composite was prepared from acacia tortilis and frankincense gums in Bursa Uludag University laboratory. The bio resins were prepared with 1:20 material to liquor ratio of natural gums at a temperature of 70 ± 2 ^{0}C for 1 hour boiling as shown in Figure 3.4b and Figure 3.6b. The solutions were continuously agitated during 5 minutes period by SCILOGEX-0S40-S mixer with an interval of 20 minutes. The bio resins were prepared without adding any additional ingredients. Separately prepared acacia and frankincense resins were mixed with 6 different ratios (%) (75:25, 70:30, 65:35, 60:40, 55:45, and 50:50) to get the desired viscose resin as shown in Figure 3.7 and Table 3.3. The mixed acacia-frankincense bio resin preparation process was repeated by mixing separately prepared acacia resin and frankincense resin at a temperature of 200-250 ^{0}C for 30 minutes and finally it is left to cooling for certain time to obtain the final bio resin (desired lab made resin) solution as shown in Figure 3.7. Completely and easily solubility characteristics in distilled water is obtained from acacia granule gum while satisfactory plasticizer behavior is achieved with frankincense gum. Unless otherwise stated in the text, the bio resin was prepared with 50:50% mixing ratio because of lower cost of this lab made bio resin. The general properties of both acacia and frankincense and their mixed lab made bio resins were summarized in Table 3.3.

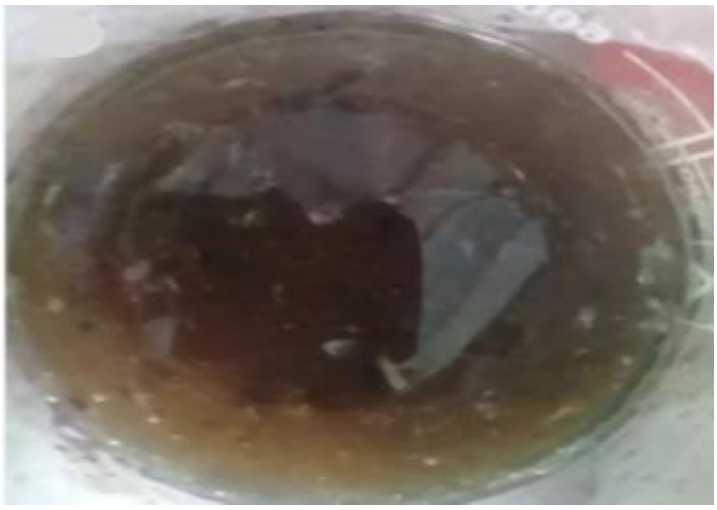


Figure 3.7. Lab made acacia-frankincense bio resin

Table 3.3. General properties of acacia and frankincense gum's bio resin[18]

Property	**Acacia tortilis**	**Frankincensepapyruses (frenkincense)**	
Moisture (%)	8.6	14.2	
Ash (%)	0.7	0.734	
Molecular Weight	157,400	153,000	
Solubility (%)	46.75 @ 60 Min. (Cold water)	32.3 @ 60 Min. (Cold water)	
	94.70 @ 60 Min. (Hot water)	56.5 @ 60 Min. (Hot water)	
Viscosity (cP)	1450.4	1236.6	
	Percentage of acacia (%)	**Percentage of frenkincense (%)**	**Viscosity (cP) of mixed resin**
Viscosity of mixed acacia and boswallia resin	75	25	1552.00
	70	30	1549.00
	65	35	1450.40
	60	40	1420.39
	55	45	1385.76
	50	50	1346.50

3.2.2. Composite manufacturing techniques

Composite materials have been manufactured by two major methods, namely called as open mold and close mold composite manufacturing techniques. In the former composite manufacturing techniques, the upper layers of the reinforcing and matrix materials are uncovered and exposed to the external atmosphere. These methods are cheap, comparatively simple, having high production rates and causing uncontrolled surface forms (Zin et al. 2016, Abdurhman 2018). Hand layup, spray up and filament winding are grouped under open mold composite manufacturing techniques (Zin et al. 2016) while closed mold (liquid composite mold) composite manufacturing is a technique in which the reinforcing materials are laid on a vacuum bag and the matrix materials are applied through the cavity of mold in order to fill the layers (laminates). These types of composite manufacturing methods are automatic and need unique equipment. Pultrusion, vacuum infusion, compression mold and resin transfer are commonly used type of closed mold methods. Brief summary of composite materials manufacturing techniques are described as shown in Table 3.4. The selection of composite material manufacturing techniques

[18] Part of Thesis results.

depend on the type of materials, mold complexity, matrix system, application and production cost.

Table 3.4. Summary of composite manufacturing technique (Zin et al. 2016)

Composite manufacturing	Type of techniques	Composite production volume		
		Low	Medium	High
Open mold Closed mold	-Filament winding -Hand lay up -Spray up -Pultrusion -Vacuum bag -Compression -Vacuum infusion -Resin transfer	-Vacuum bag -Vacuum infusion -Hand lay up -Spray up	-Compression -Centrifugal -Resin transfer -Filament winding	-RRIM[19] -Continues laminate -Pultrusion -Compression

3.2.3. Manufacturing of green composites by hand layup method

Hand layup composite manufacturing is one of the most commonly and widespread method of composite manufacturing (Bhatt et al. 2018). It is simple, needs a minimum infrastructure and has cheap set up cost as compared with other well-known composite manufacture methods (Zin et al. 2016, Abdurhman 2018). In hand layup method, the composite materials are produced by spreading the reinforcing materials on the mold and applying (spraying) the matrices (gels) into the laminating plies by using roller until the desired layers are achieved as shown in Figure 3.7. Polymeric sheets (polypropylene) and/or wax are spread on the upper and lower laminate plies to avoid sticking of composite materials and to have better surface finish (Abdurhman 2018). This technique is mostly convenient for thermoset polymers (Zin et al. 2016). The advantage and limitation of hand layup composite manufacturing was summarized in Table 3.5.

[19] Reinforced Reaction Injection Molding (RRIM)

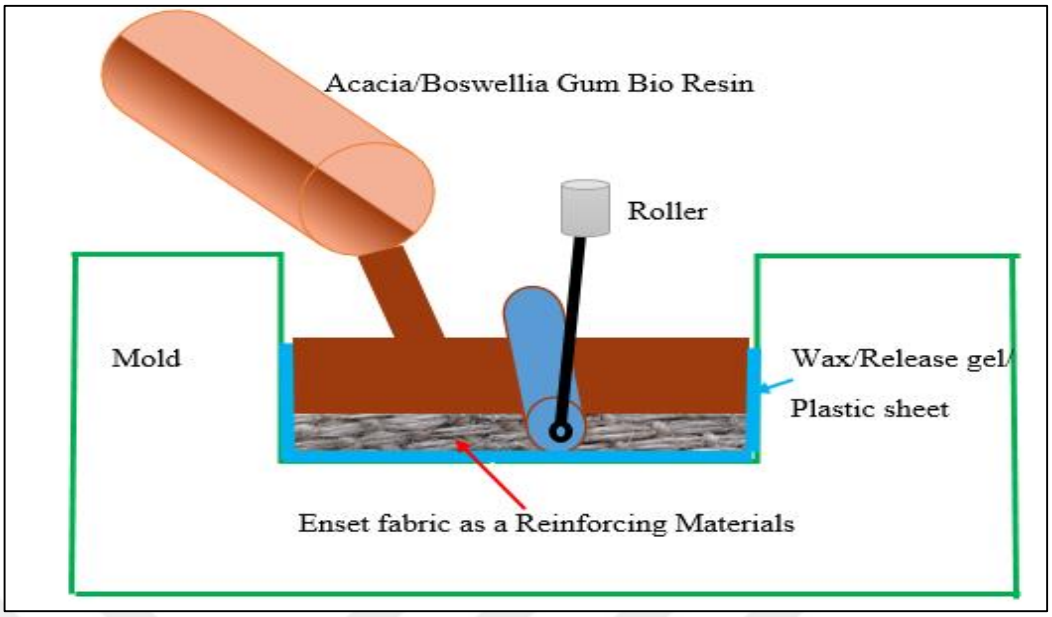


Figure 3.8. Hand layup composite manufacturing set up (Abdurhman 2018)

Table 3. 5. Summary on hand layup composite manufacturing methods (Zin et al. 2016, Bhatt et al. 2018)

Advantage of hand layup	**Limitation of hand layup**
Small initial capital	Need lower viscosity matrix
Higher volume of fibers	No health effect
Relatively simple processes	The quality of final product Depend on the operator skill
versatile	Higher curing time
Low cost of manufacture cost	

The first task of hand layup green composite manufacturing technique was waxing and/or spreading the plastic sheet (polypropylene) on the mold, then the enset woven fabric reinforcing plies were cut into the desired size (30 cm x 30 cm) and spread on the mold. Prepared bio matrices (obtained from acacia tortillas and frankincense) were released on the enset woven plies. The bio matrix was uniformly distributed into the woven fabrics by using roller (is used to remove excess bio resin and void space(air) between the enset woven fabrics) and a known load of 12 Kg was applied (loaded) for 12 hour at room temperature after the desired layers were achieved. Enset fabric reinforced green composites were cured at a temperature of 110 ^{0}C for 30 minutes by using RAPID-

TAYLAN drying device shown in Figure 3.8. Finally, the enset fabrics reinforced green composite materials were cut based on the ASTM standards for desired physical and mechanical tests such as tensile strength, flexural strength, impact strength, air permeability and acoustic property. Enset fabric reinforced green composite materials were prepared for sound measurements in two major types, single layers having a thickens of 2.5 mm and double layers having a thickness of 3.5 mm with 30 % of enset fiber (1.2 g/cm^3) and 70 % bio matrix mass fraction. For mechanical performance analysis, the bio composites were produced with each each ratio of acacia/frankincense gums and then their tensile strengths were measured. Later, bio composites were produced with 50%:50% bio resin ratio with single and double layer enset fabric mixture and



Figure 3.9. a) Enset fabric b) Acacia and frankincense gum bio resin c) Used hand layup set[20] and curing device d) Enset fabric reinforced green composite material

[20] Special hand layup set was design and prepared for this study.

3.2.4. Mechanical properties of green composite materials test and characterization

The mechanical properties and performance of green composite materials are mainly influenced by size, orientation, shape, distribution of reinforcing materials as well as the properties and types of the matrices materials (Bhatt et al. 2018). Moreover, composite manufacturing methods, volume of reinforcement-matrix fraction, nature of interface and nature of reinforced materials also have significant effect on composite mechanical properties (Salman et al. 2015, Ferreira and Capela 2016). One of the major mechanical property problems of green composite structures manufactured from lignocellulose fibers are the loss of their strength when the reinforcing materials are exposed to moistures (Salman et al. 2015). Enset woven fabrics are used as reinforcing materials because of their higher specific strength, flexibility, interlacing of yarns (give better strength for the structures than fibers) and formability properties (Yan et al. 2016).

The mechanical performance such as tensile strength, flexural strength, impact strength and stiffness of enset fabric reinforced composites were determined by using international standards like American Society for Testing and Materials (ASTM). The ductility nature and the strength of the composite materials were evaluated by documenting and demonstrating the level of damaging and cracking of the composite structures (Mobasher 2016). In composite materials, the rate of strain increases with the increment of load transferring capacity. Cracking of composite materials occurs because of stress developed within their structures and it results the delamination of the plies by forming protrudes of the fibers from their composite structures (Feih and Mouritz 2012, Ou et al. 2016). The test results obtained in the form of load-elongation is not directly interpreted because the results are significantly affected by the samples geometry, size, rate of loading and type test methods (Ou et al. 2016).

Tensile strength test

The tensile strength of composite materials refers to the capacity of composite structures which can handle the stress before the composite starts to crack, break and fail as well as deformed (Arumuga et al. 2014, Plateau 2017). It is the capability of composite materials to withstand against the tensile force (under pulling). It is expressed as force per unit areas (commonly used Mega Pascal for composite structures). The tensile strength of a

composite material is one of the most vital property used to explain its mechanical strength and performance (Plateau 2017). The fraction of reinforcing materials and their strength determine the mechanical strength (longitudinal) of composite structures (Arumuga et al. 2014). The tensile strength (breaking strength) of fiber reinforcing materials is very significantly higher than the strength of matrices. So the ultimate tensile strength of fiber reinforced composites is determined by their reinforcing materials (Feih and Mouritz 2012, Plateau 2017). Particularly, the tensile strength of newly manufactured composite materials are important for researchers and manufactures to expect the maximum load carrying capacity of the materials (Arumuga et al. 2014). These properties of composite materials are more imperative (significant) when brittle reinforcing materials are used than elastic ones. The tensile strength of composite structures can be expressed in three kinds such as yield strength (the structures are withstand the stress without everlasting deformation of the material is occurred), ultimate strength (withstand the ultimate stress) and breaking strength (the stress developed at rupture) of a composite materials. There are two major possibilities of composites material's failures, i.e. brittle failures (sudden and unexpected breakdown of the structure) and ductile failures (the structure failure after the formation of neck) (Plateau 2017).

Specimen Preparation: tensile strength test specimen preparation is depending on test method, aim of the test and specification. The samples mostly prepared and standardized as a form of round and flat shoulder are used for serrated griper and thread shoulder is used for thread griper. Flat shoulder having a hole is used for pinned gripper and butt end shoulder is used for split gripper in the universal strength testing equipment (Plateau 2017). Tensile strength testing of enset fabric reinforced composite materials were measured by Shimadzu test machine shown in Figure 3.9a based on ASTM D638-10 standard. The load cell had 5 KN capacity and the jaw moved at the rate of 6 mm/min during tests. For this thesis, flat shoulder specimens having 300 millimeter length and 50 millimeter width were used for tensile strength tester shown in Figure 3.9b.

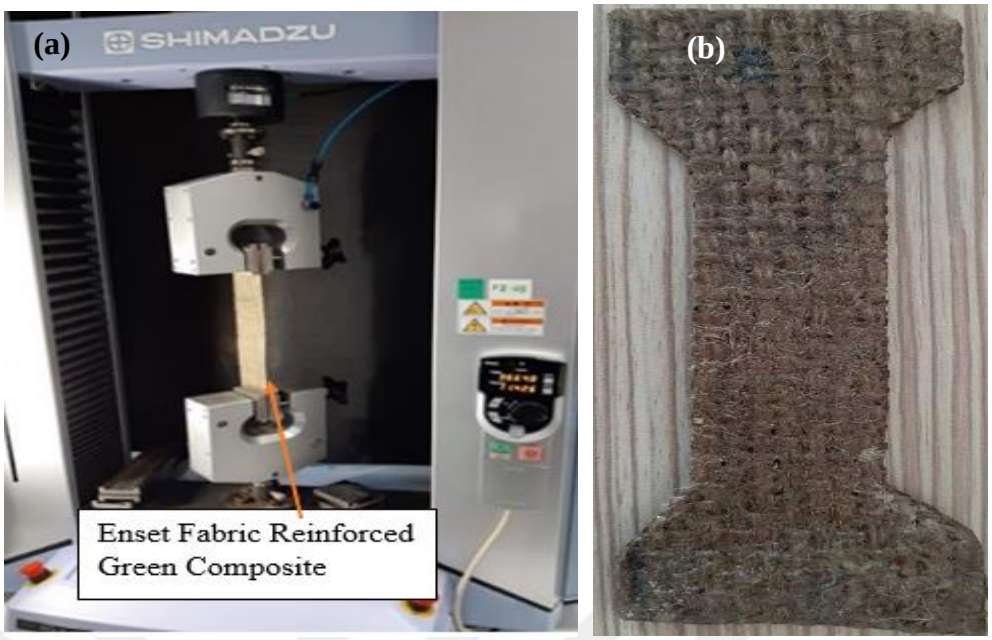


Figure 3.10. a) SHIMADZU stregth tester b) Speciemne for tensile stregnth test

Flexural strength test (3 Point bending / dynamic test)

Three points bending test is one of the most widely known test method used for evaluating the flexural properties of composite materials (Mallick 2007, Rokbi et al. 2011, Tiber and Balcıoğlu 2019). The flexural tests of enset fabric reinforced composite materials were done based on ASTM D-790 by using three point bending mechanisms. The specimens were prepared based on the ASTM standards having a dimension of 100 X 12 X 3 mm^3 as shown in Figure 3.10a. These specimen dimensions were cut with the length thickness ratio of 16:1 and width thickness ratio of 3:1. 5 samples were prepared for the test and their average test results were taken for the characterization of the flexural behavior of enset fabric reinforced composite materials (Tiber and Balcıoğlu 2019) as shown in Figure 3.10a and 3.10b. Equation 3.1 was used to calculate the flexural strength of the composite samples. The test was conducted at a temperature of 20 ± 2 ^{0}C and relative humidity of 65 ± 2 ^{0}C by loading (putting) the enset fabric reinforced composite samples on the 9280 Dynamic Data Acquisition System flexural strength and Shimadzu strength testing equipment testing equipment (as shown in Figure 3.10c and 3.10d) and exerting the load (for Dynamic Data Acquisition System flexural strength 5 and 10 bar pressure used to give force for the load cell) into the samples with a jaw speed of 10 mm/min till

the specimen was fractured (is a kind of sample damage which occurred once the material crack propagation is started) and broken as shown in Figure 3.10c and 3.10d. The flexural strength of enset fabric reinforced composite materials were expressed as the maximum stress developed on the outermost reinforcing materials (Al-Mosawi and Rijab 2013) . It was calculated on the convex side or tensioned portion of the specimens while the flexural modulus was obtained by calculating the slop of the flexural test curves (stress vs. deflection or load vs. time) (Al-Mosawi and Rijab 2013). All the test results were converted into numerical values and then analyzed.

$$\sigma = F\ X\ S = \frac{3PS}{2bt^2} \tag{3.1}$$

Where,

σ = Flexural strength (N/m^2)

P = Maximum test load (N)

S = Dimension between load points (mm)

b = Sample width (mm)

t = Temple thickness (mm)



Figure 3.11. a) Enset fabric reinforced composite specimens b) Schematic illustration of 3 point bending flexural test c) Dynamic flexural strength tester d) Shimadzu strength tester

Impact strength test

Impact strength of composite materials can be expressed as the ability of a materials or structures to resist the applied sudden load or force. Impact behaviors of the material normally tell the quantity of mechanical energy which are absorbed during the deformation of material under impact loading (Safri et al. 2014, Navaranjan and Neitzert 2017, Gholizadeh 2019). The impact strength of a material is defined as its capability to resist a sudden applied load or force. It is normally conveyed as the amount of mechanical energy absorbed in the process of deformation under the applied impact loading. Impact behavior of composite materials must be properly analyzed and evaluated because it has a very significant effect on the mechanical performance of the composite structures (Gholizadeh 2019). The effect of impact damage on the composite materials are analyzed in two ways (Gholizadeh 2019).

(i**)** Impact Damage Tolerance: It refers to the capability of a damaged composite structure or laminate to retain its original stiffness or strength. Mostly the damages are produced by drop weight impacting which are causing widespread internal damages. Such kind of damages are difficult to detect by visual inspections.

(ii) Impact Damage Resistance: it is associated with the ability of composite materials which are responding to applied impact.

The impact strength of natural fiber reinforced composite materials have been measured by using different testing methods (Navaranjan and Neitzert 2017). The choice of the test method and testing machine is dependent on the accessibility of the test machine, the awareness and interest of researcher and need of the industrial or business sectors. Each impact strength testing method has its own merit and limitations (Navaranjan and Neitzert 2017). Most commonly charpy and izod impact strength testing methods are used (Safri et al. 2014). The working principles of both methods are the same. The basic difference between izod and charpy impact strength testers are the supported position of the specimen as shown in Table 3.6. The specimen is supported as a beam in the case of charpy impact testing mechanism while the samples are supported as cantilever for izod testing method (Safri et al. 2014, Navaranjan and Neitzert 2017).

Table 3.6. CHARPY and IZOD impact strength test methods and their requirement (Navaranjan and Neitzert 2017)

Parameters	Charpy impact strength testing methods	Izod impact strength testing methods
Standards	ISO 148, EN 10045-1 and ASTM A370	Notched samples: ASTM D256 or ISO 180 Un notched samples: ASTM D4812 ISO 180
Type of notch	U or V type	Only V type
Loading mechanism of test sample	Placed horizontally	Placed vertically
Dimensions of test specimen	55mm x 10mm x 10mm	75mm x 10mm x 10mm
striking of hammer	At the point of notch (opposite direction)	At the upper tip of sample
Type of used hammer (as striker)	ball pin hammer	Farming hammer

The Charpy impact strength test of enset fabric reinforced green composites was conducted by using ASTM D256 standards having a specimen dimension of 55mm x 10mm x 10mm with v-shaped notched as shown in Figure 3.11a. The used impact velocity for this test was 10 mm/sec. The impact strength test of notched specimen of enset fabric reinforced composite was conducted as shown in Figure 3.11b and calculated from the potential energy difference between the starting position of the swinging pendulum and the first reversal point of the pendulum after kicking the specimen based on equation 3.2.

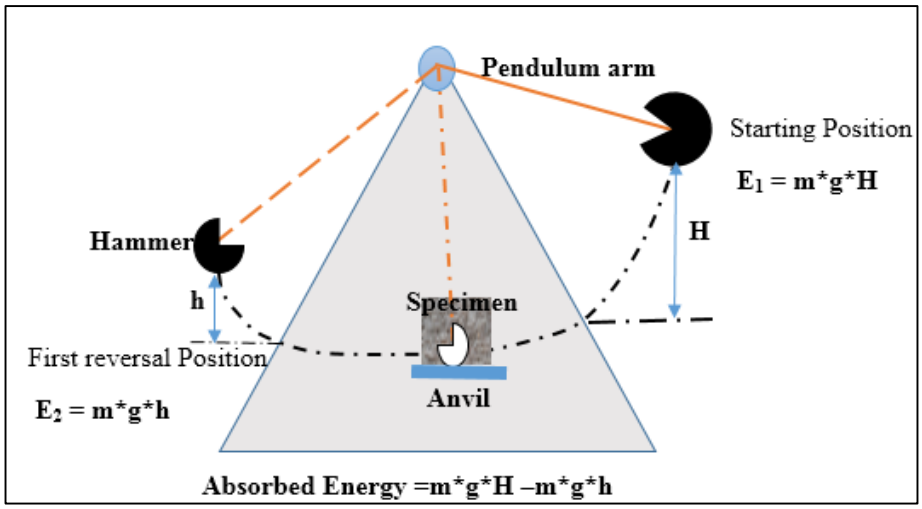


Figure 3.12. Mechanism of impact strength test

$$Kv = (m * g * H) - (m * g * h) \quad (3.2)$$

Where,

Kv= Notching impact energy (J)

m= mass

g= gravity

H= the height between the specimen at the anvil and starting position

h= the height between the specimen at the anvil and first reversal position

The impact strength of composite materials is fundamentally affected by the velocity of the impact test. Commonly the dynamic impact test is done by four type of velocity such as:-

- ✓ Low velocity (1-10 m/s)
- ✓ High velocity (10-100 m/s)
- ✓ Ballistic (50-1000 m/s). Damage caused by ballistic impact (>500 m/s)
- ✓ Hypervelocity (> 2000 m/s) (Razali, et al. 2014).

The toughness of enset fabric reinforced composites structures have been tested by Charpy impact testing mechanism. It was conducted by the ASTM-D-256 standards.

Impact test setup and testing procedures

Enset fabric reinforced composite test samples were prepared based on the ASTEM-D 256 standard. The specimens were grooved with V-shape notch and mounted on the lower part (anvil) of Charpy impact testing machine. After checking the center of notched specimen and turning the scale indicator into zero position, the test was began by releasing down the pendulums which are holding the dead weight (hammer). The dead weight of the pendulum broke the specimens and the scale indicators changed their position from zero (at the beginning the device has potential energy 1 at height "H") as shown in Figure 3.11. During this phenomenon, the pendulum arm did not reach the starting position (at the first reversal point, the device has potential energy 2 at height "h"). This height change

between the starting position of the pendulum arm and the first reversal position indicated that a certain amount of energy was absorbed by the test specimens and called notch impact energy. During the test, if the specimens were broken into two pieces, it indicated that the materials had brittle properties and absorbed small amount of notch impact energy. While the specimens were not broken into two pieces, it indicated that the samples absorbed much notching impact energy and the materials are tough.

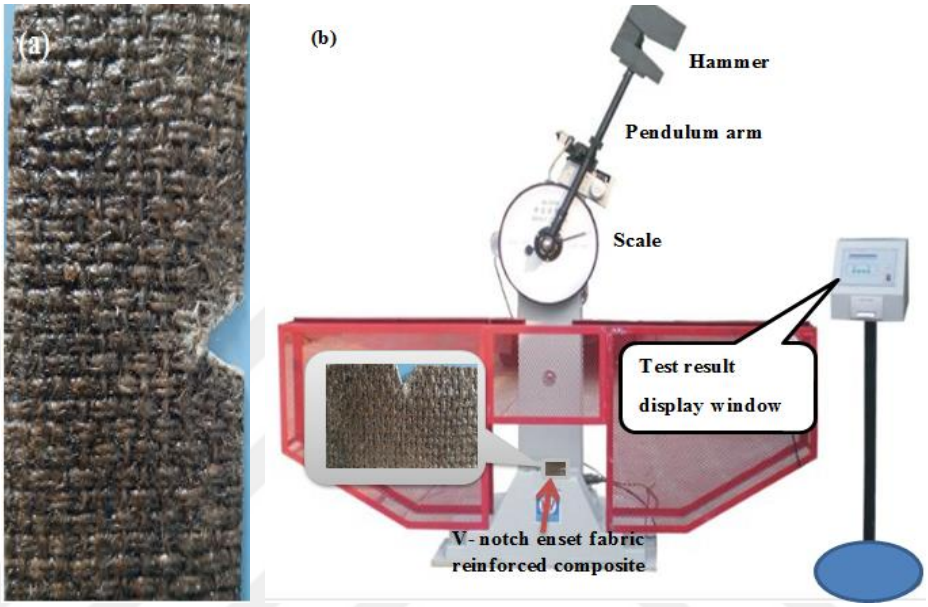


Figure 3.13. a) V- notch sample for impact strength testing b) JBW-300 computer display pendulum impact strength testing device

3.2.5. Green synthesis of enset nano fiber (ENF) via enzyme treatment and mechanical hammering

Different concentrations of α-amylase enzymes were used to defibrillate. HP-630 hot plate and SCILOGEX-0S40-S Mixer were used to prepare the homogenous enzymatic solution for enset nano fiber production. A hammer of 2.5 Kg was employed as a source of mechanical power for beating the enset fibers. EVO-40 Scanning Electron Microscopy (SEM) measurements were conducted for the examination of micro and nano structure of the treated and mechanically hammered enset fiber. BRUKER-TENSOR-37 FTIR was used for the characterization of untreated and treated enset fibers. BET measurement was conducted with the degassing temperature at 90 ^{0}C by using MICROMETER-TRISTAR II. Enset nano fiber was produced from enset fiber as well as enset fabric. Both enset fiber and enset plain woven fabric were used for this study. For easier mechanical hammering,

the enset fabric was found to be more convenient than enset fiber. There are three major types of nano fiber fabrication approaches: mechanical, chemical and biological nano fabrication (Kargarzadeh et al. 2017, Nohwar and Tuberosum 2017). This study was conducted on the technology of combined chemical treatment and mechanical hammering approaches for the reduction of fiber diameter.

Preparation of enset fabric samples: Enset fabric samples were cut into 20 X 20 cm^2 dimension for homogenous enzymatic treatment and mechanical hammering.

Enzymatic treatment and hammering: The logical approach for enset nanofiber fabrication was based on top-down nano materials manufacturing technique in which enset micro/nano fibers would be separated from each other by enzymatic treatment and mechanical hammering until the desired micro and nano enset fibers were extracted, by forming a lot of branched fine fibers as depicted in Figure 3.13. The prepared enset fabric samples were weighted and then treated with three concentrations of α-amylase enzyme (10% w/v, 15 % w/v and 20% w/v) for 1 hour at a temperature of 35 ± 2 ^{0}C with 30:1 material to liquid ratio. After the α-amylase enzyme application, the enset fiber started to swell and the lignocellulose parts in the fiber were appropriately digested to allow the transformation into micro/nano fibrous structure. The fibers were also subjected to mechanical destruction with hammer until the desired fine fibers were defibrillated from the macro scale enset fiber into micro/nano fibers at wet condition (wetted with α-amylase enzyme). The treated enset fibers were then cooled and left for 6 hours in the solution to give enough time for better swelling and digestion of hemicellulose 5 times. Mechanically extracted enset nano fibers were examined by using SEM analysis. Chemical characterization of untreated and treated enset fibers were examined with a FTIR spectroscopy. The thermal decomposition of enset and their fine fibers were examined with the help of TGA. BET was used for surface area analysis.

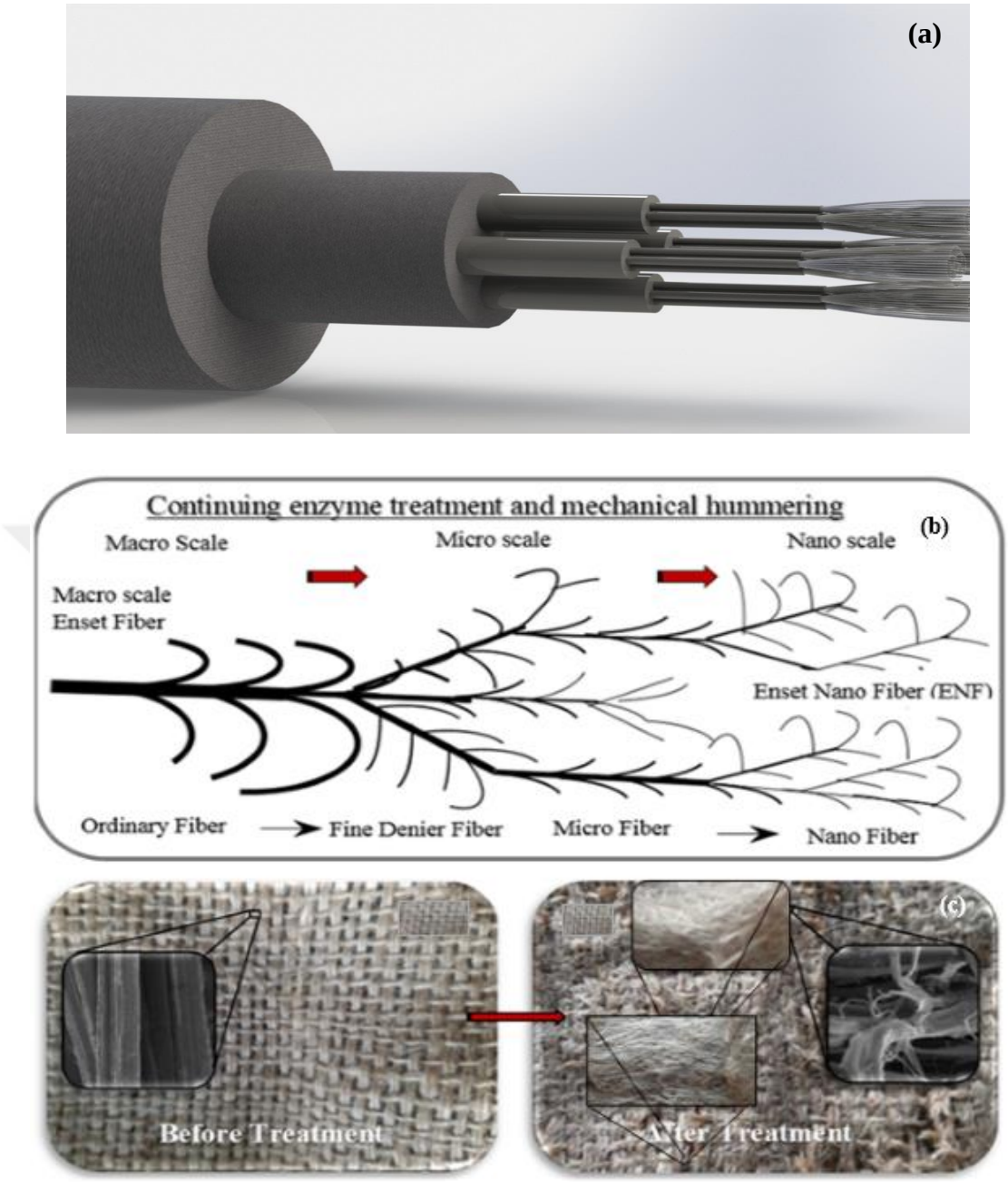


Figure 3.14. a) Auto CAD simulation b) Schematic illustration of micro and nano enset fiber preparation c) Micro and nano enset fiber preparation by α-amylase enzyme treatment and mechanical hammering

3.2.6. Acoustic and air permeability properties test of neat enset fabric, enset nano fiber and their green composite

Sound absorption and air permeability measurement of neat enset fabric and their green composite

The samples were prepared according to the TS-391-ISO9237 and ASTM 1050 standards for air permeability and sound absorption tests respectively as shown in Figure 3.14. The air permeability and sound absorption test results were obtained from the average of 5 samples.

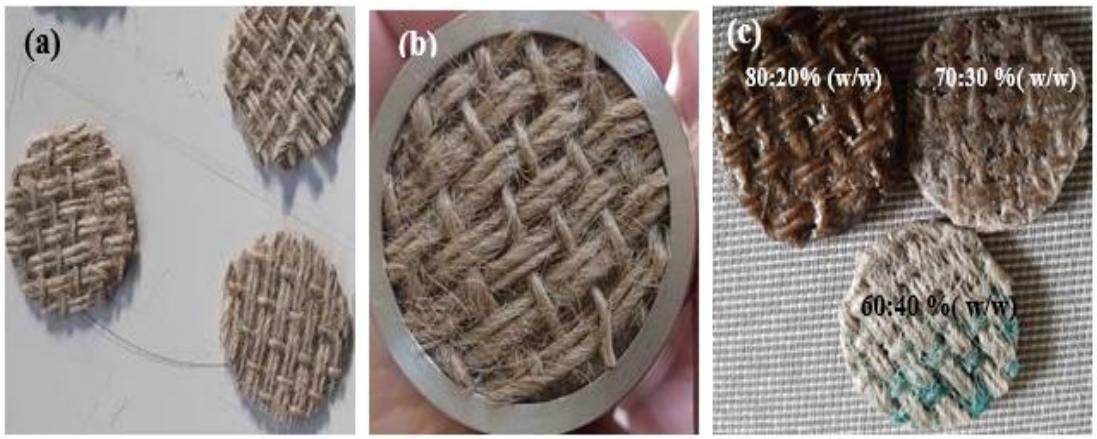


Figure 3.15. a) Enset fabric sample b) Test sample holder c) Green composite samples for air permeability and sound absorption measurement

Sound absorption Test: The sound absorption coefficient of enset reinforced composite samples was measured by using Impedance Tube Methods (ASTM 1050). Samples with 3 cm diameter of enset fabric and its composite were prepared as shown in Figure 3.14a. The test was conducted with polyurethane foam at the back side and enset fabric and also with only enset fabrics as shown in Figure 3.14b and Figure 3.14c. The sound wave was released and propagated from the load speaker to strike the enset samples at the other side of the equipment and the test result was recorded. The measurements were done by 3 different thicknesses of 1.5 mm, 2.5 mm and 4.5 mm at frequencies between 1 KHz and 6 KHz. Table 3.7 shows testing materials and their specifications for sound absorption measurements. Fig.3.15a shows schematic view of working principle of Impedance Tube Method of sound absorption measurement device.

Table 3.7. Sound absorption test specification

Sample type	No. of fabric layer (Reinforcement)	Thickness (mm)	Sample area (cm^2)	Weight (gm.)
Enset fabric	1	1.5	3	0.43
Single layer composite	1	2.5	3	1.0
Double layer composite	2	4.5	3	1.88

Calculation of Sound absorption coefficient: The microphone is used to detect the maximum and minimum amplitude of sound pressure variation (Tran 2017). The ratio of maximum sound pressure to the minimum is called standing wave ratio (SWR) (Lee et al. 2017). The values of sound absorption coefficient changes between zero and one (Lee et al. 2017) . The sound absorption coefficient is expressed by equation 3.3.

$$Sound\ absorption\ coefficient\ = 1 - (SWR - 1)\text{^}2/(SWR + 1)\text{^}2 \quad (3.3)$$

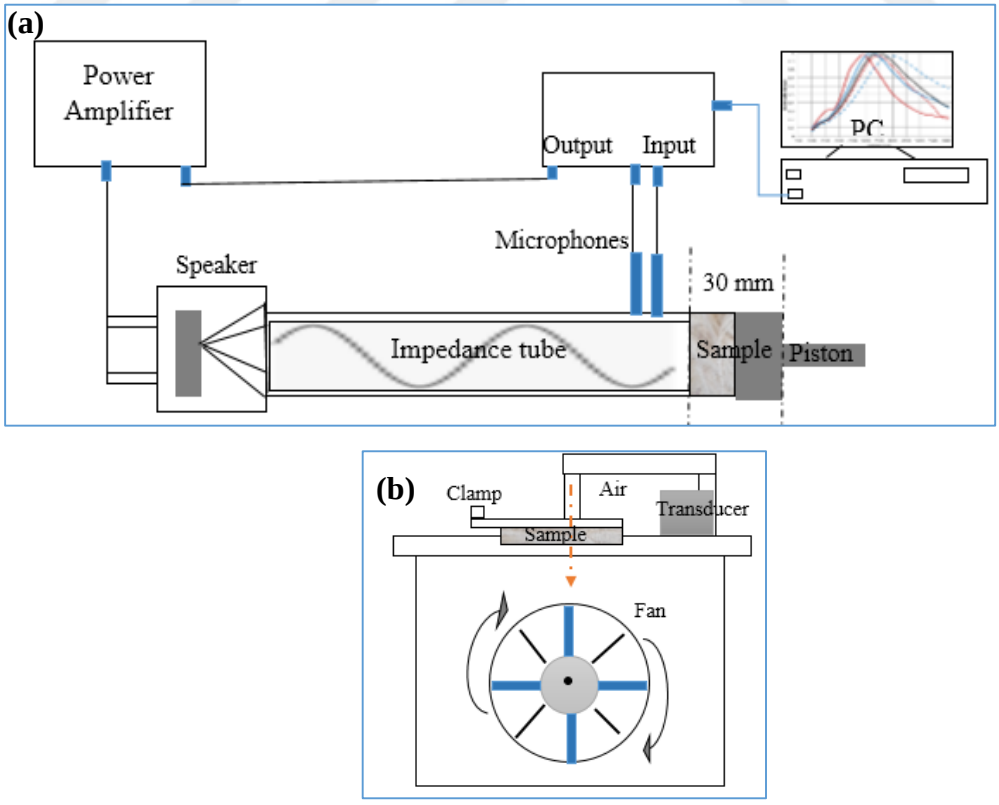


Figure 3.16. a) Impedance tube sound absorption tester b) Air permeability tester

Air permeability Test: SDLATLAS-MO21A air permeability tester was used for measuring the air permeability of enset fabric and its composites. According to the test standard, 5 cm^2 samples of enset fabric and its composites were prepared and the test was conducted as shown in Figure 3.15b and Table 3.8. Firstly, the air permeability tester was calibrated and then the test area and air pressure used were set (200 Pa). The samples were loaded and the test was conducted by pressing the pressure arm shown in Figure 3.15b. When the testing equipment showed a green light, the test result was recorded and their average values were determined and used as air permeability value.

Table 3.8. Air permeability test specification

Sample type	No. of fabric layer (Reinforcement)	Thickness (mm)	Sample area (cm^2)	Weight (gm.)
Enset fabric	1	1.5	5	0.8
Single layer composite	1	2.5		2.19
Double layer composite	2	4.5	5	3.45

Sound absorption and air permeability measurement of neat enset fabric and enset nanofiber web materials

The sound absorption coefficient of neat enset fabric and enset nano fiber web was measured by sing Impedance Tube Method. The samples were prepared base on ASTM E1050 standard with a diameter of 30 mm and fixed to the instrument as seen in Table 3.9 and Figure 3.15a, 3.16a and 3.16b. The measurements were done with 3 different enset nano fibers which were synthesized by three different concentrations of amylase enzyme, i.e. 10 %, 15% and 20% w/v. The samples had an average thicknesses of 11.5 mm. The sound wave was released and propagated from the load speaker to strike enset nano fiber samples at the other side of the equipment and the test results were recorded at the frequency interval of 1000-6000 Hz. MO21A air permeability tester was used for measuring the air permeability of enset fabrics and enset nano fibers. Samples were prepared based on ASTM D737 standard. According to the test standard areas, 5 cm^2 from enset nano fiber structures were cut as shown in Figure 3.15b and 3.16c. Firstly, MO21A air permeability tester was calibrated and then the air pressure was set to 200 Pa. The samples were loaded and the tests were conducted by pressing the pressure arm. A

suction fan forces the air to flow perpendicularly through the enset nano fiber and enset fabric samples. The flow pressure are applied on the fabrics and enset nano fibers. The air flow is adjusted gradually until the required pressure drop is achieved across the test region. The transducer is used to measure the volumetric air flow rate in l/m^2 /sec as illustrated in Figure 3.15b. These values are divided by the samples area (5 cm^2) to get the air flow rate. Finally the test results were recorded in l/m^2/sec after the green light turned on.

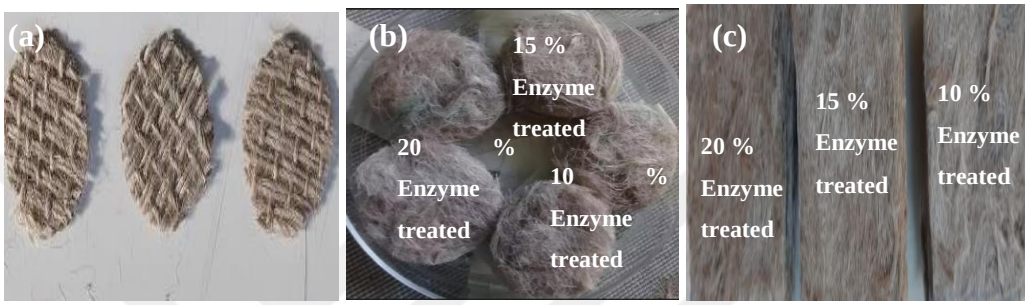


Figure 3.17. Samples for test: a) Neat enset fabric. Cellulosic enset nanofiber**:** b) Sound absorption c) Air permeability

Table 3.9. Main properties of the materials in this studied

Characteristics of Textile Material	**Mean Thickness (mm)**	**Acoustic Measurement**		**Air Permeability Measurement**	
		Weight (gm)	**Diameter (mm)**	**Weight (gm)**	**Area (mm^2)**
Neat Fabric	10-13	0.89	30	2.90	500
Enset Nanofiber treated by 10 % amylase Enzyme	10-13	0.89	30	2.886	500
Enset Nanofiber treated by 15 % amylase Enzyme	10-13	0.88	30	2.884	500
Enset Nanofiber treated by 20 % amylase Enzyme	10-13	0.89	30	2.888	500

3.2.7. Morphological analysis of enset fabric reinforced composite

The morphological analysis of enset fabric reinforced composite structures were analyzed by using optical microscopic and scanning electron microscope as shown in Figure 3.17a. Gold was used to coat the specimens to enhance the surface conductivity as shown in Figure 3.17b.

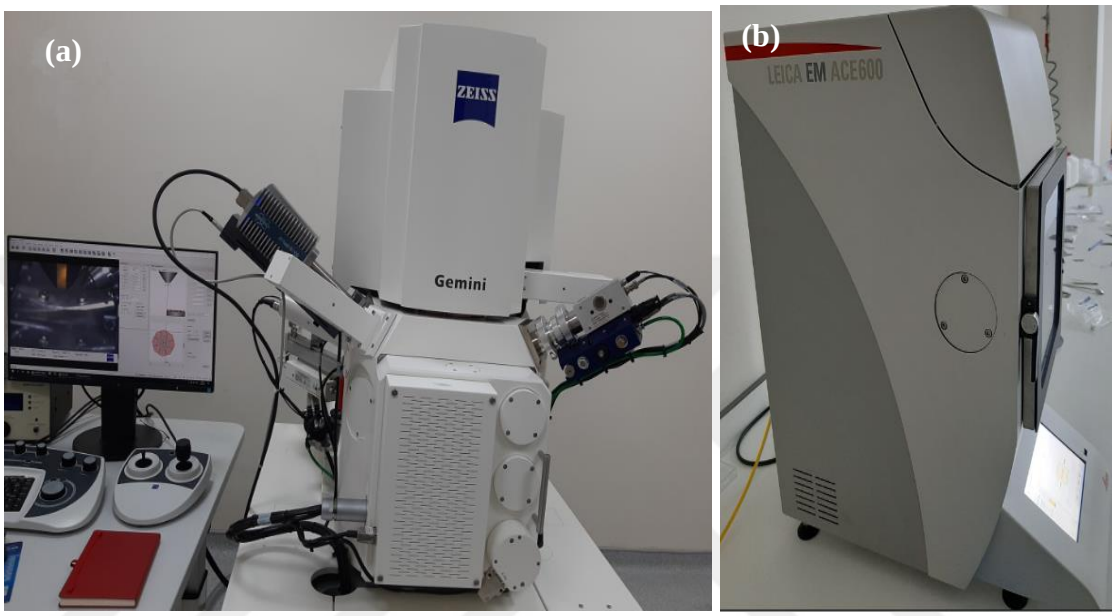


Figure 3.18. a) Scanning electron microscope (SEM) b) Specimens gold coating equipment

4. RESULTS and DISCUSSIONS

The properties and performance of textile fibers have a significant effect on the physical and mechanical behaviors of yarns as well as fabrics. Advanced fiber modification and their physical treatment must be taken in to account on its final end use and environmental effects. Enset nano fibers would be used to produce more advanced yarn, fabric and composite having unique properties such as high durability, higher weight to strength ratio, large surface area and bio-degradable products.

4.1. Green Synthesis of Enset Nano Fiber (ENF) Production

The fine fiber formation occurred by the reduction of its hierarchical structure at hemicellulose and lignin parts (Poddar et al. 2015, Hanieh 2017). Enset fibers contained a number of layers in a single macroscale structure. So, enzymatic treatment and mechanical hammering separated the layered structures into a number of branched fibrils and formed new fine fibers having the same properties like the virgin enset fiber. Because of its microspore structure, the defibrillation of the fiber into micro and nano scale structures was carried out with simple mechanical hammering.

4.1.1. Scanning electron microscope analysis of enset fiber

The microscopic images of enset fiber indicated that the fibers were defibrillated from the multicellular structures into fine fibrils having a lot of helical-hollow shaped and convoluted cross sectional areas as shown in Figure 4.1a to Figure 4.1d. Moreover, Figure 4.1b and 4.1d showed that single macroscale enset fibers contained a number of consecutive layers in its structure. Because of its microspore structure as shown in Figure 4.1a and Figure 4.1d, the defibrillation of the fiber into micro and nano scale structures was achieved by simple mechanical hammering enzyme treatment. The fine fiber formation occurred by the reduction of its hierarchical structure of hemicellulose and lignin parts of raw enset fibers. The defibrillation of enset fiber from the upper surface of the woven fabric created a lot of branched fine fibers as shown in Figure 4.1c. So, enzymatic treatment and mechanical hammering was used for the separation of layered

structures into a number of branched fibrils and formed new fine fibers having the same properties like the virgin enset fiber as shown in Figure 4.1c.

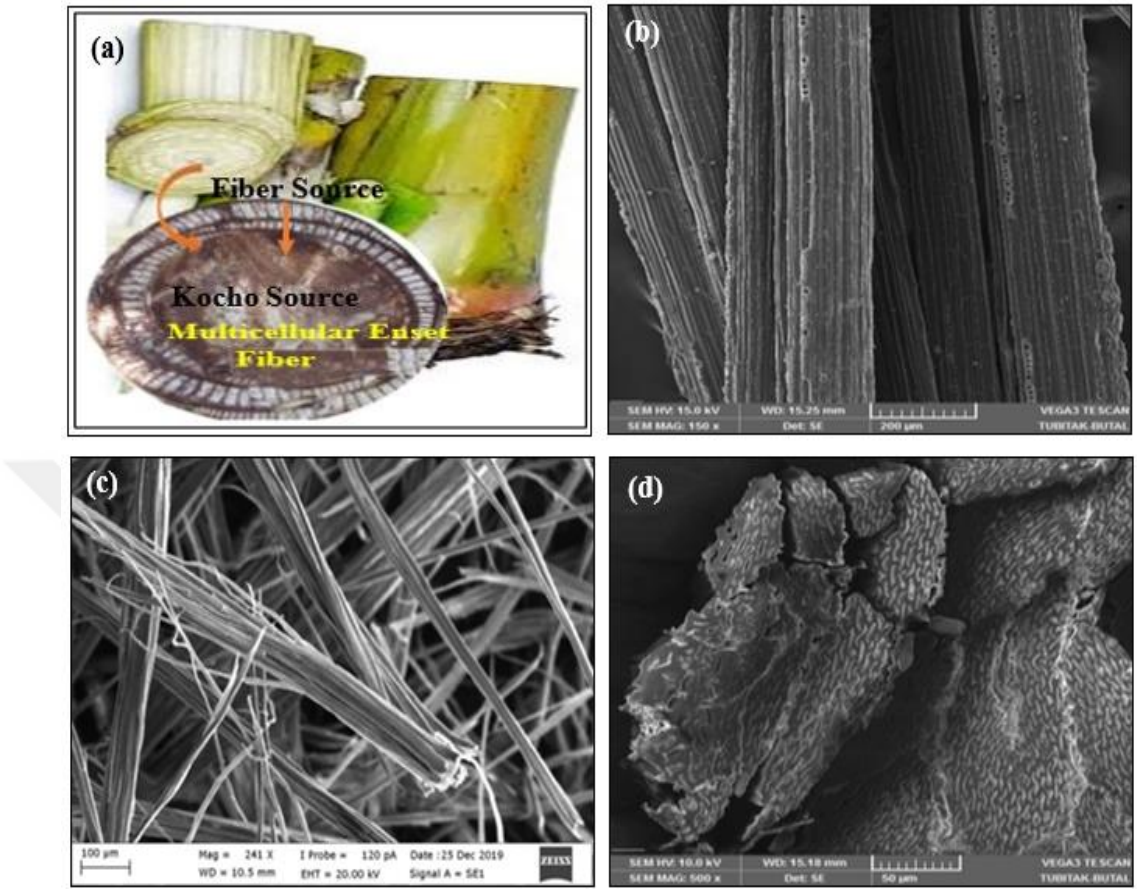


Figure 4.1. a) Hallow morphologic view of enset plant, cell phone photograph (25Mp); SEM. b) Natural composite structure of single enset fiber c) Multicellular view of enset fiber d) Micro pores and cross sectional view of enset fiber

The applied treatment changed the morphology of enset fiber in terms of its size (diameter) from 2μm to 63 nm. Figure 4.2a indicated that the enset fiber was clustered because of ineffective removal of lignin which joined cellulose and hemicellulose to form the macroscale enset fiber while Figure 4.2b indicated that the lignin was effectively removed and naturally connected layers started to be defibrillated into two and more. Successive enzymatic treatment and mechanical hammering continued the defibrillation of enset fiber structure until the desired nano fibers were obtained (Figure 4.2c and 4.2d).

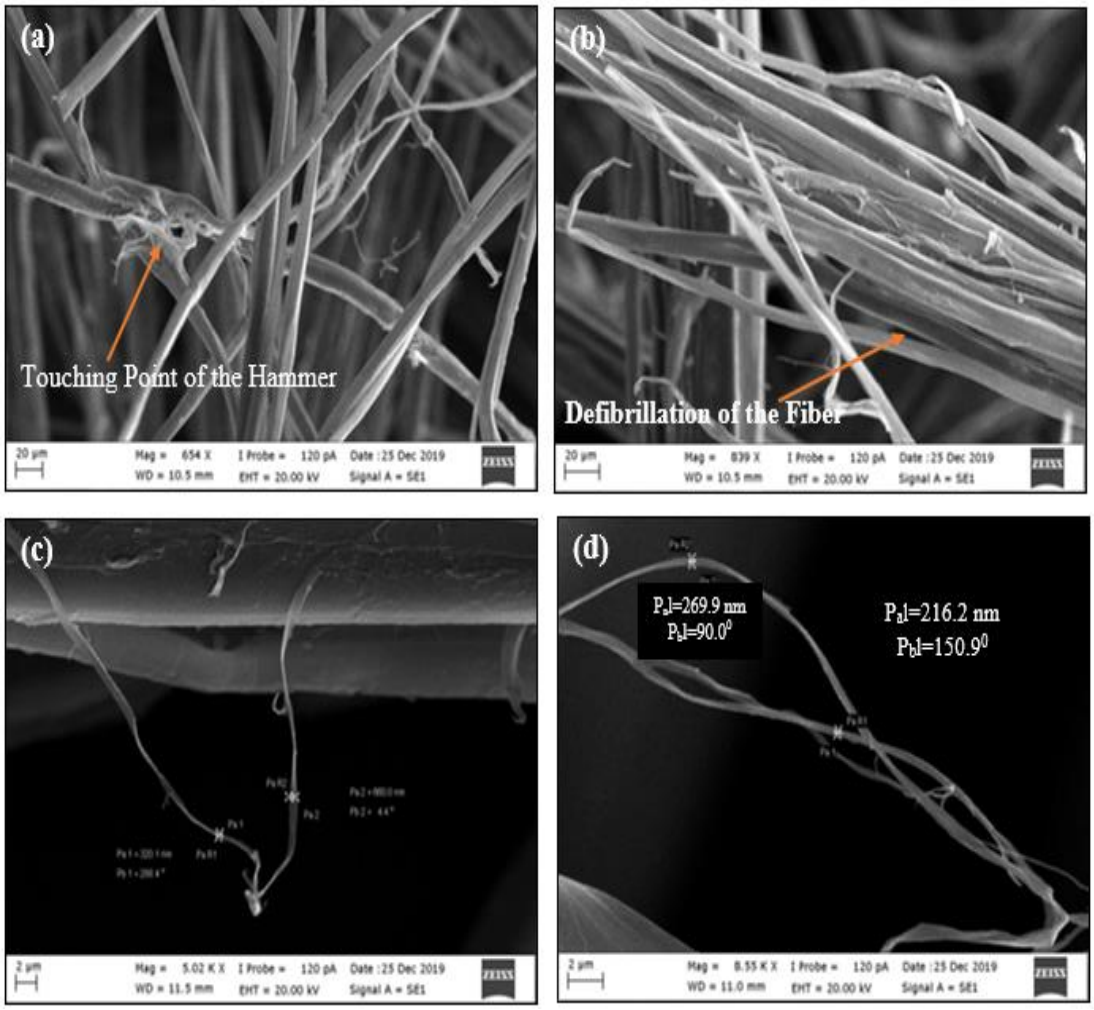


Figure 4.2. SEM view of α-amylase enzyme treated and mechanical hammered enset fiber a) Clustered enset fiber b) 10% (w/v) α-amylase enzyme concentration. (micro enset fiber) c) 15% (w/v) α-amylase enzyme concentration (nano enset fiber) d) 20% (w/v) α-amylase enzyme concentration (nano enset fiber)

In a preliminary study, up to 5 % (w/v) concentration of α-amylase enzymatic treatment, the enset fiber fibrillation was not clearly observed as seen in Figure 4.3a and 4.3b. When the concentration of α-amylase was increased to 10 % (w/v), micro fibrils having a size of 2 to 20 μm were formed and additionally started to protrude from the upper surface of enset woven fabric (sequentially shown in points 1, 2 and 3, in Figure 4.3a). Likewise when the concentration of α-amylase enzyme was further increased from 10 % (w/v) to 15-20 % (w/v), macroscale fiber was further defibrillated and fine sized enset nano fibers started to be formed depending on the applied mechanical power. At 15% (w/v) concentration of α-amylase enzyme, the nano fibers were observed at a large size of

diameter of 133 nm – 660 nm. This can be seen at point 4 in Figure 4.3a. When the concentration of α-amylase enzyme was increased to 20% (w/v), the fibers were additionally further fibrillated into very fine scaled nano fibers having diameter between 85.07 - 63.19 nm shown at point 5 in Figure 4.3a. The hierarchical structure of enset fibers was further defibrillated with the continuation of α-amylase enzyme treatment and mechanical hammering until lower size diameter of micro and nano fibrils were obtained. Observations made in Figure 4.3a and Figure 4.3b indicated that the virgin enset fiber was swollen and immature hemicellulose structure was began to split out from the macro scale structure and form sub macro and micro structures of enset fiber. The removal of deposited film was done by mechanical hammering till the desired fine fiber structure was obtained (Figure 4.3a).

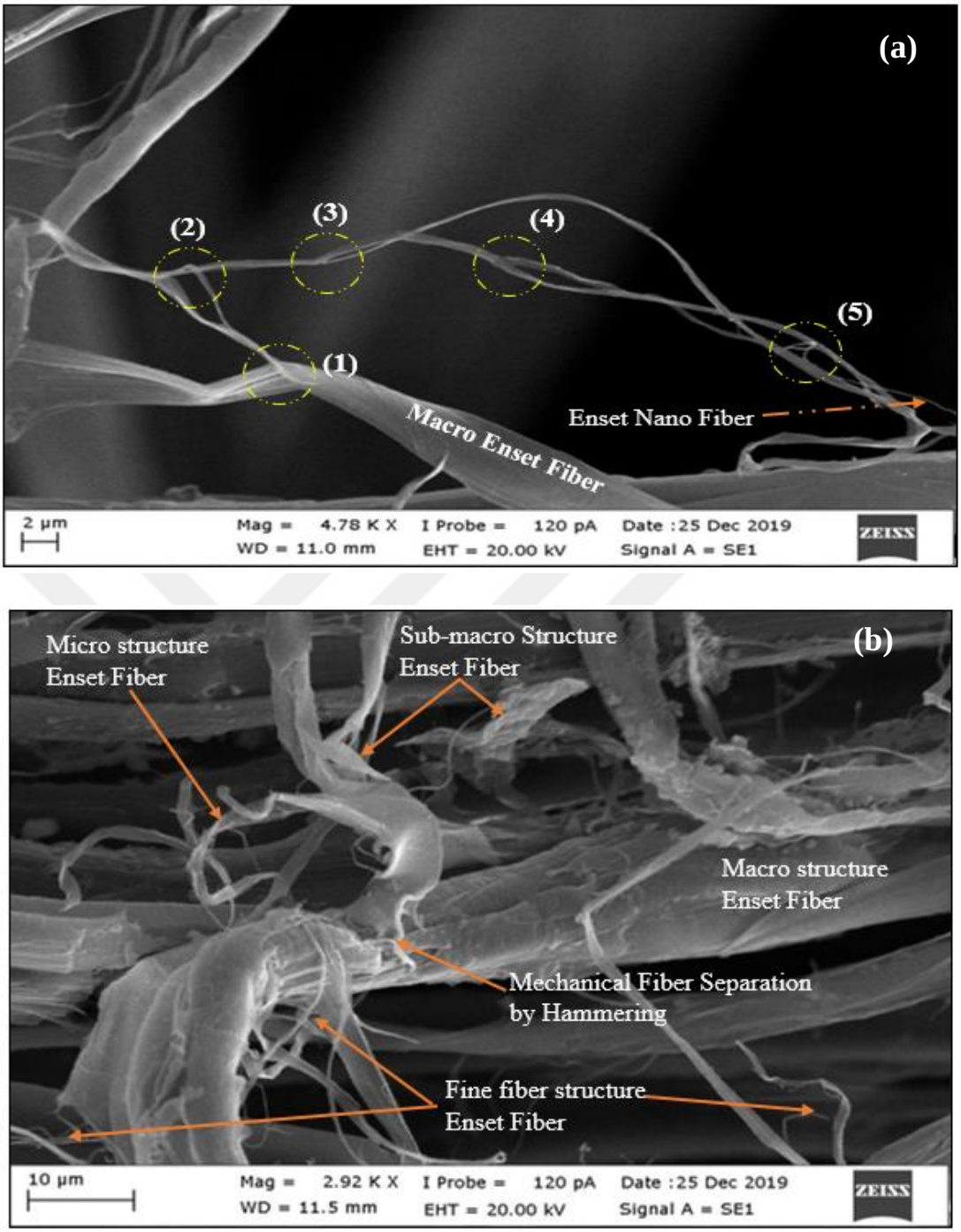


Figure 4.3. SEM views: a) Hierarchical defibrillation of macro-scale enset fiber into enset nano fiber b) Gradually removal of hemicellulose and lignin from the macro structure of enset fiber by mechanical hammering

4.1.2. Surface analysis (BET) of enzyme treated and mechanically hammered enset fiber

The specific surface area of the amylase treated and mechanically hammered enset fibers were investigated using nitrogen gas adsorption method. Figure 4.4a, 4.4b and 4.4c demonstrated the isotherms of the treated enset fibers. A type-IV isotherm and H3-type hysteresis loop was observed from the plot, and the results revealed that the fibers contained mesoporous structure (Tan et al. 2012). The sharp rise in the loading at the lower values of P/Po of treated enset fibers indicated a high surface area, the abrupt loading transition with in the P/Po range of 0.1 - 0.4 revealed a uni-modal pore size and the presence of hysteresis confirmed the existence of micro and mesoporous since unrestricted monolayer-multilayer adsorption occurred at high P/Po. The measured BET surface areas were 4.271 m^2/gr, 4.596 m^2/gr and 11.60 m^2/gr for 10 %, 15 % and 20 % w/v concentration of α-amylase enzyme treated samples respectively. The surface area of treated fine enset fiber was improved by 270 % when the concentration of used α-amylase enzyme was changed from 10 to 20 % (w/v). The surface area could be increased with transforming more macro fiber to micro and nanofibers by increasing mechanical hammering and concentration of α-amylase enzyme used.

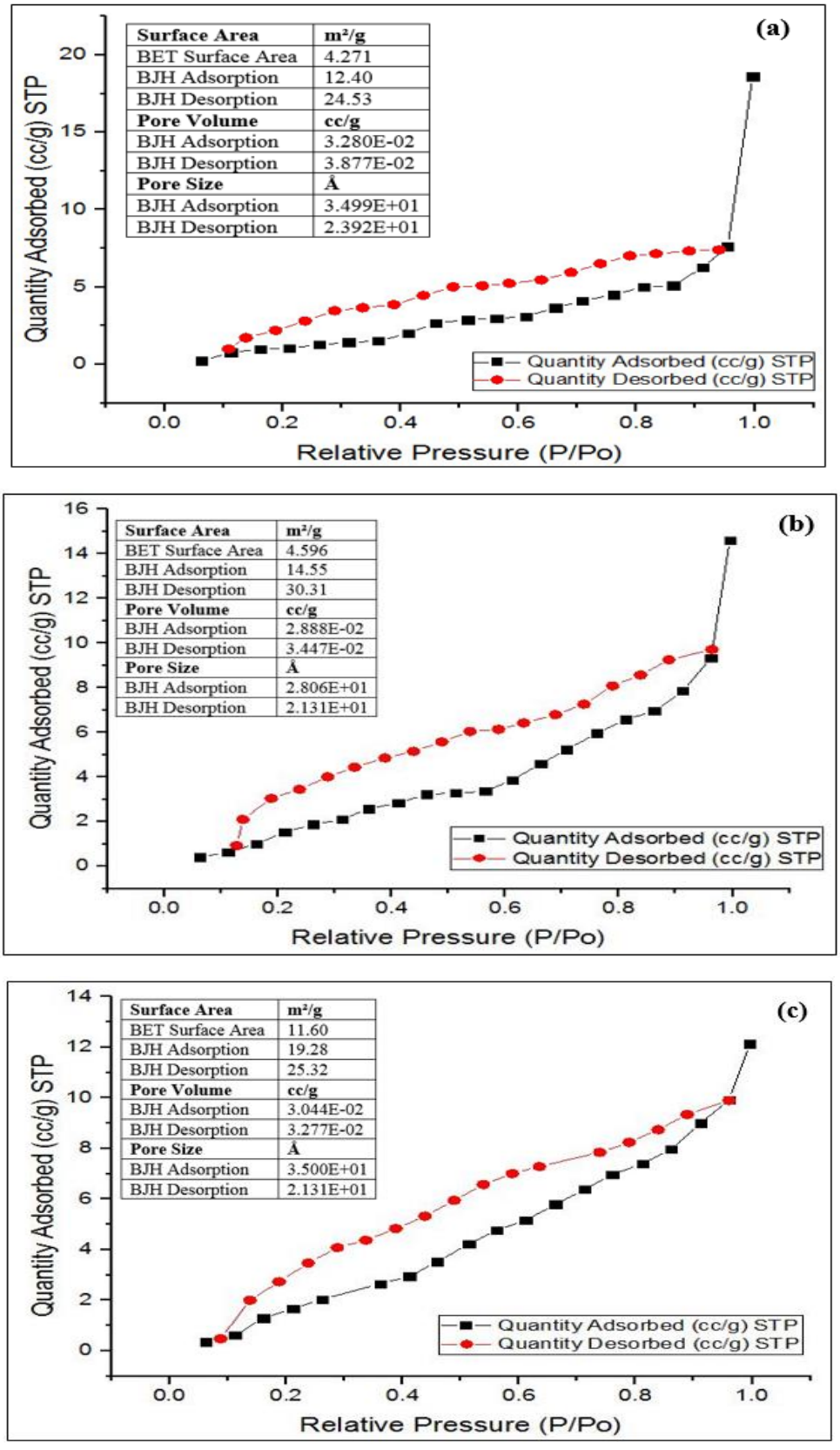


Figure 4.4. BET curve-surface area analysis of treated enset fiber a) 10 % w/v b) 15% w/v and c) 20% w/v concentration of α-amylase enzyme

4.1.3. Chemical analysis of the neat and treated enset fiber with FTIR spectroscopy

The FTIR results were used to prove the chemical modification and defibrillation of enset fiber treated with α-amylase enzyme. The FTIR plots of treated samples were compared with those of untreated enset fiber and given in Figure 4.5. The peaks in the band of FTIR spectrum of 3331 cm^{-1} revealed that enset fiber had O-H group in its structure (Sarkar and Ray 2001, Yang et al. 2007). With the peak at 2917cm^{-1} wavelength, C-H aliphatic and aromatic stretching groups were shown and this indicates the presence of cellulose, hemicellulose and lignin (Teli and Jadhav 2015, Teli and Terega 2017). Likewise, the peaks at 1730 cm^{-1} and 1665 cm^{-1} of enset fiber indicated the presences of C=O stretching group of hemicellulose and C=O stretching conjugate aromatic ring respectively. The absorption bands between 1500 cm^{-1} and 600 cm^{-1} indicated the fingerprint region of the fiber in its infrared spectrum. The peaks at 1422 cm^{-1} revealed aromatic ring vibration in cellulose and hemicellulose. The absorption spectra at the peak of 1242 cm^{-1} and 1100 cm^{1} exhibited C=C and C-O stretching ester group of hemicellulose and lignin groups in cellulose structure respectively. Moreover, the absorption at 1028 cm^{-1}, C-O, C=O, C-C-O stretching were due to the presence of cellulose, hemicellulose and lignin in the enset fiber (Bilba and Moroi 2007). Observation shown in Figure 4.5 described a major difference in peak heights and shapes between untreated and treated enset fibers at a wave number range of 1500 to 1000 cm^{-1}. The hemicellulose and excess lignin part of enset fibers began to defibrillate from the fiber structure. Under the FTIR absorption band ranging from 1500 cm^{-1} to 1000 cm^{-1} wave number, the prominent peaks seen in Figure 4.5 at the point “a”, “b” and “c” disappeared. Moreover, the major peak shapes as well as their height gradually wiped out for the treated enset fiber depending on the amount of α-amylase enzyme used. The shape of the FTIR peaks encircled with red color -A pointed at “a”, “b” and “c” in Figure 4.5 revealed that the removal of hemicellulose from macro structure of enset fibers started (Teli and Jadhav 2015) and the enset fibers began to defibrillate to produce micro and nano enset fibers.

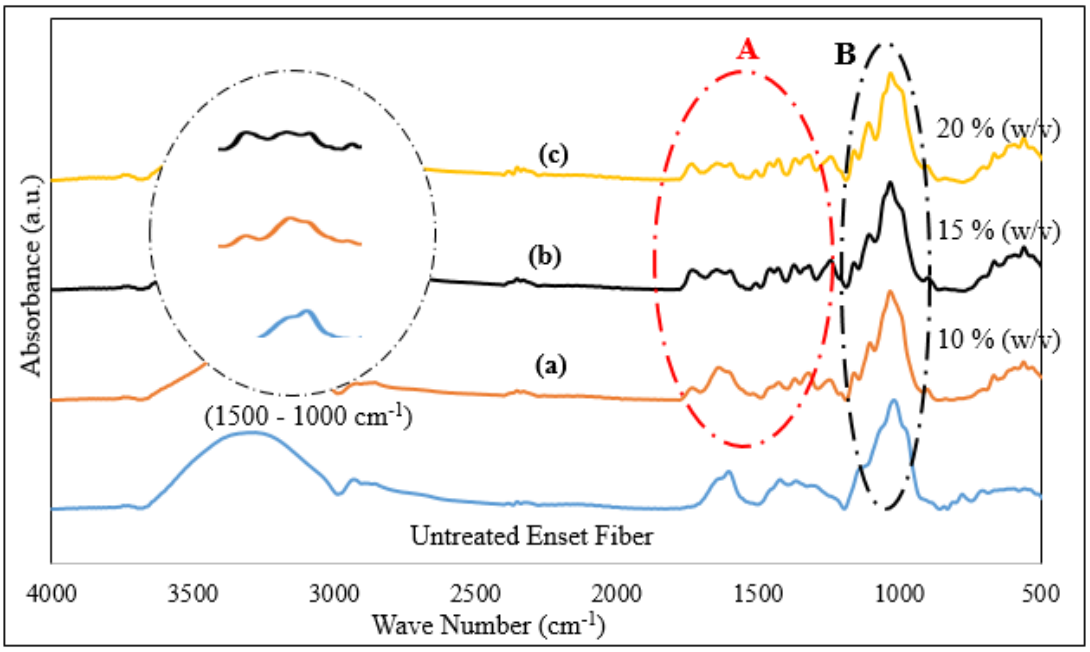


Figure 4.5. FTIR gradual wiped-out of hemicellulose and lignin from enset fiber with α-amylase enzyme treatment and mechanical hammering a) 10% (w/v) α-amylase enzyme treated b) 15% (w/v) α-amylase enzyme treated c) 20% (w/v) α-amylase enzyme treated enset fiber

The fine fibers began to defibrillate from the cellulose part of the fiber at the concentration of 10% (w/v) α-amylase enzyme (seen at point "a") encircled with black in Figure 4.5. Mostly the fine fibers were obtained up to 2μm. The peak of 1100 cm^{-1}, corresponding to C-O stretching conjugate (lignin) and 1321 cm^{-1}, ring condensed stretching (lignin) revealed the removal of excess lignin from enset fibers (Das et al. 2000). Moreover, there was a clear peak shape difference between untreated and treated enset fiber at wave number of 1100 cm^{-1}. These phenomena proved that the treatment of enset fiber with enzyme helped for easy defibrillation of macro fiber into fine structure (encircled with black color (B) in Figure 4.5. At the concentration of 15 and 20 % (w/v) of α-amylase enzyme (shown at point "b" and "c", encircled with black in Figure 4.5), the micro fibers were fabricated up to 2-200 μm and enset nano fibers with 63-85 nm size respectively.

4.1.4. Thermal characterization of enset nano fiber (TGA)

The weight loss of enset fiber and enset fine fibers were measured and analyzed by Thermo-Gravimetric-Analysis (TGA) with respect to the decomposition temperature

(Wielage et al. 1999). The thermal analysis was conducted between 30 and 800 ^{0}C shown in Figure 4.6 (Wielage et al. 1999, Johar 2012). The TGA curve revealed that enset fiber remained thermally stable up to the decomposition temperature reaching to 227 ^{0}C while this temperature raised to 264 ^{0}C with medium and highly treated enset fibers. At lower decomposition temperature ranging from 38 -115 ^{0}C of raw enset and treated fine enset fibers, only the moisture present on their components were evaporated (Bismarck et al. 2002, Almeida et al. 2008). The water loss of raw, medium and highly treated enset fibers wase realized around 9 % at the initial decomposition temperature of the fibers due to evaporation of water shown in Table 4.1. The major thermal degradation of raw, medium and highly treated enset fibers was observed at a temperature of 240 ^{0}C and 285 ^{0}C respectively. A drastical thermal degradation occurred due to the breaking of glycoside linkage of cellulose, rupture of lignin and decomposition of hemicellulose (Nair et al. 2001). The major thermal degradation of raw enset fiber was observed in between 230 and 346 ^{0}C with a mass loss of 74 %. Whereas medium and highly treated enset fibers had a weight loss of 76 % and 89 % at a temperature of 260-390 ^{0}C respectively. The overall residuals of raw, medium and highly treated enset fibers after the whole thermal decomposition ensued were 24 %, 17 % and 13 % at a temperature of 800 ^{0}C respectively. The TGA graph approved that medium and highly treated enset fibers had better thermal stability than raw enset fibers shown in Figure 4.6.

Table 4.1. Cumulative weight loss (%) of raw enset fiber and treated fine fibers

Type of fiber and weight loss (%)	**Temperature (^{0}C)**							
	100	200	300	400	500	600	700	800
Raw enset fiber	9	10	30	70	73	75	75	75
Medium treated Enset fiber	9	10	20	78	81	82	83	83
Highly treated Enset fiber	9	10	20	82	85	86	87	87

Enzymatic treatment and mechanical hammering of enset fibers resulted weight loss and fiber damage due to the hydrolysis and uncontrolled fiber breakage respectively. The concentration of enzyme and applied mechanical hammering power had a direct effect on the possibility of micro/nano fiber formation and fiber weight loss. The test result proved that, when the concentration of α-amylase enzymes were used beyond 20 % (w/v), the enset fibers were degraded as well as partially destroyed the main cellulose components

of the fibers rather than fine fiber formation. So in order to solve such kind of problem and to get a better quality fine fiber, the applied mechanical power and concentration of enzyme must be determined in an optimum way.

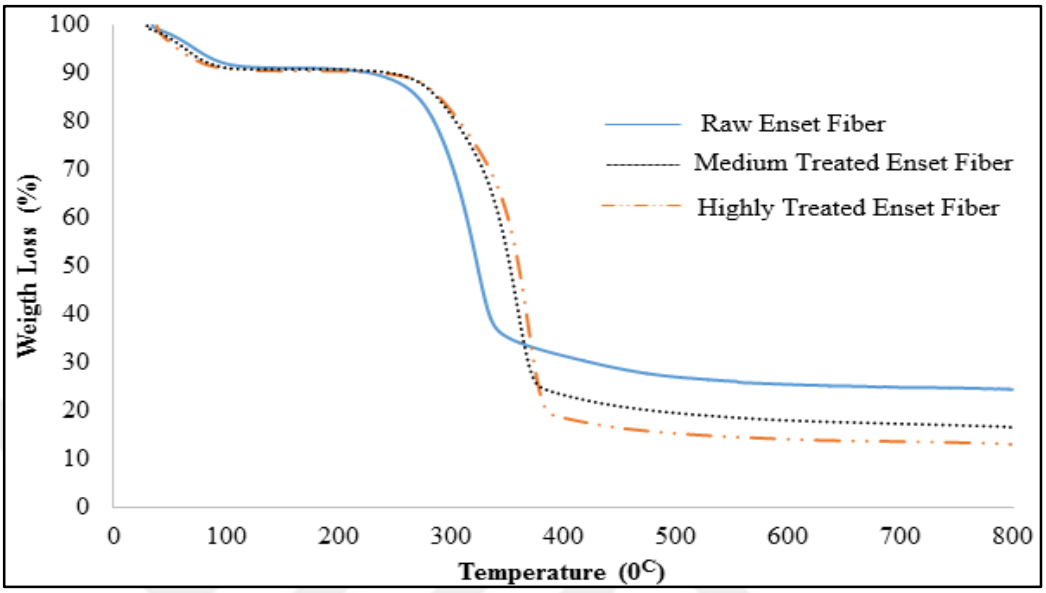


Figure 4. 6. TGA curves of raw, medium and highly treated enset fibers

4.2. Mechanical Performance Analysis of Enset Fabric Reinforced Green Composite

Researchers and composite materials manufacturers have focused more on the development and enhancement of natural fiber reinforced green composite structures for light weight duties, automotive and construction sectors. Generally the mechanical properties of natural fibers are poorer than petrochemical based fibers but their eco friendliness, biodegradability, no health hazard and lower cost (abundantly available) are mostly preferred compared to synthetic fibers. The mechanical performance and properties of enset fabric reinforced green composite such as tensile strength, bending strength (flexural strength) and impact strengths have been studied by using a newly developed bio resin and enset woven fabric. The effect of mixture ratio of acacia tortilis and frankincense on bio resin performance was investigated by conducting tensile tests the composite produced with each ratio. Then, a detailed mechanical performance analysis was continued with 1:1 (or 50%:5 0%) mixture ration bio resin. Results are presented below.

4.2.1. Tensile strength result analysis

Enset fibers have high lignocellulose content and its immature cellulose materials create a barrier in the internal polymer chains of enset fiber and make it dissociated from its structure. This natural gift property provides the enset fibers to have a good tensile strength (~500 MPa) which is better than the most commercially known natural fibers like sisal (400 MPa), bamboo (140-230 MPa) and coir (106 -270 MPa) fibers. Table 4.2 describes that the elongation of the enset fiber at break (2.5-3.4 %) is near to flax (2.7-3.2 %), bamboo (2.9-3.3 %) and jute (2.6 - 4.5 %) fibers. This promising property suggests that the un-utilized enset fiber would be employed in light weight composite industries and can make a contribution in reducing the dependence on finite petrochemical fibers.

Table 4.2. Tensile strength and modulus of elasticity of natural fiber mostly used in the composites

Type of fiber	Tensile strength (MPa)	Modulus of elasticity (GPa)	Elongation at break (%)	Reference
Enset [21]	350-500	10-22.4	2.5-3.4	-
Sisal	400-700	9-20	4.5	Mallick 2007,
Jute	550-900	17-26	2.6-4.5	Sanjay et al.
Bamboo	140-230	7.7-32	2.9-3.3	2016,
Coir	106-270	3-6	-	Plateau
Flax	1100	100	2.7-3.2	2017

The test results showed that the tensile strength and percentage elongation of enset yarn had average values of 53.23 N and 3.04 % respectively as shown in Table 4.3 and in Figure 4.7a. Likewise, it was also shown that enset woven fabric (there are 8 warp yarns in its effective width) had a tensile strength and percentage elongation of 528.83 N and 5.70 % respectively as indicated in Table 4.4 and Figure 4.7b. Based on the experimental values of a single enset yarn strength, the calculated tensile strength of enset fabric having 8 warp yarns in the sample width would be 425.84 N. But the experimental test result of woven enset fabric's tensile strength was measured as 528.83 N. The difference between the calculated and experimental test results was due to inter and intra friction developed between the yarns in the woven fabric structure. This phenomenon affects the tensile

[21] Part of this dissertation studies

strength and crimp affects elongation of enset fabric as yarns are flattened in the fabric with decreasing crimp under load. As a result, the fabric's tensile strength increase over the tensile strength of total 8 warp (from 425.84 to 528.83 N) was caused by the friction developed between warp and warp yarns at interlacing points. In the same manner, the free movement developed between the warp and weft yarns and flattening trend of warp yarns in the fabric structure increased the elongation of fabric almost twice (5.70%) compared to enset yarn breaking elongation (3.04%).

Table 4.3. Enset yarn tensile test results

Max. Force (N)	Max. Percentage elongation (%)
49.26	2.55
53.52	3.24
51.98	4.12
54.26	3.23
57.32	2.05
Average = 53.23	**Average = 3.04**

Table 4.4. Enset woven fabric tensile test results (8 warps per sample)

Max. Force(N)	Max. Percentage elongation (%)
545.26	5.52
454.59	6.86
537.56	6.09
601.65	6.00
505.04	4.02
545.26	5.52
Average = 528.82	**Average = 5.69**

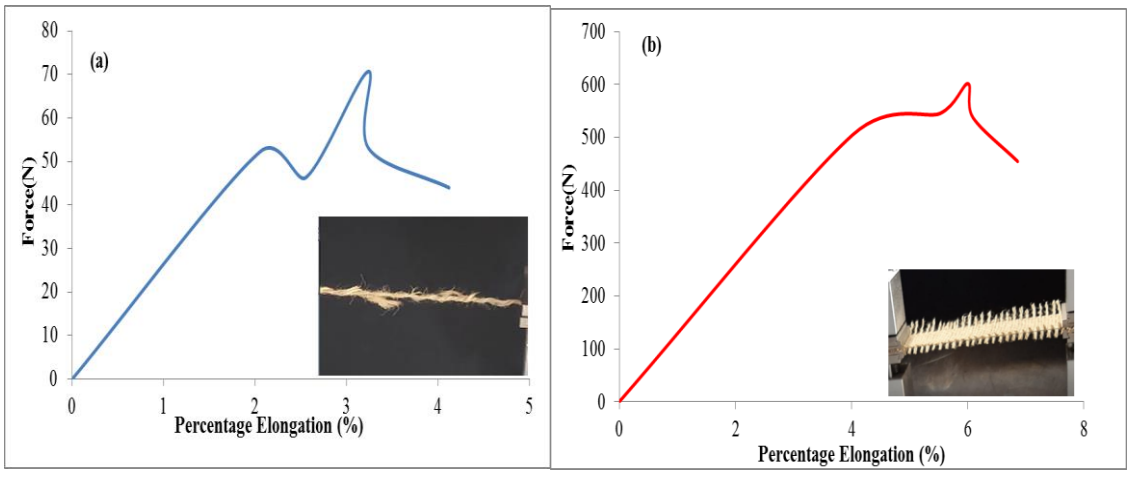


Figure 4.7. Tensile strength results a) Enset yarn and b) Enset woven fabric (8 warps per sample)

To analyses the performance of bio degradable composite produced by the developed bio resin and enset fabric, single layer composites were produced with different bio resin mixing ratios and their average breaking tensile strength and percent elongations were measured as presented in Table 4.5. Also, load elongation curves are given in Figure 4.8a to g for each bio resin mixing ratio. The data in Table 4.5 and curves in Figure 4.8 have revealed that the mechanical strength of bio composites was very significantly affected when the percentage of acacia resin was increased. As seen from Table 3.3, the viscosity of the bio resin increased 1346.5 to 1552 cP when the acacia resin ratio increased from 50 to 75% in the mixed bio resin. This increased tensile strength of the bio composite from 2356.5 N to 2851.6 N. At the same time, percent breaking elongation of bio composites also increased with increasing the amount of acacia ratio in the mixed bio resin. It increased from 5.7 to 9.08% with the increase in acacia ratio from 50 to 75% in the mixed bio resin. Because, naturally acacia gums contains highly viscose gel solutions and gums can easily soluble and form viscos solution with water at lower temperature and mixing forces (string) as compared with frankincense gums. Further research regarding tensile analysis of the bio composite continued with 50:50% ratio as the cost of mixed bio resin increased with increasing acacia percent and 5.70 % breaking elongation was found sufficient with the bio composite. Tensile strength and percent elongation of the bio resin given in Table 4.5 for 50:50% ratio mixed bio resin is the average of 5 different measurements presented in Table 4.6. Among 5 measurements, there are around 100 N variation in tensile strength and 0.6% variation in breaking elongation. As the properties of natural fibers are not strictly homogenous, these deviations will occur and suitability of a bio composite for a specific application should be decided by the requirement of the applications.

Single layer enset fabric reinforced bio composites had a tensile strength of 2356.50 N as shown in Table 4.5 and Table 4.6. The average tensile strength and percentage of breaking elongation of enset fabrics having the same number of warps and wefts for the same samples dimensions had 528.82 N and 5.69 % respectively. Converting a single layer enset fabric sample of 5cm x 20cm to bio composite using the developed bio resin (50:50% ratio) increased the tensile strength to 2356.5 N while the percent elongation

remained almost the same. Around 445% increase in tensile strength showed a great effect of the developed bio resin on the tensile strength of the composite. This result approved that the developed bio resin enhanced the mechanical performance of enset fabric reinforced green composites and have a promising potential to be used in green composite manufacturing with some improvements. It should be mentioned here that the bio composites used in the tests were produced with 30%:70% enset fabric to bio resin material weight ratio.

Table 4.5. Single layer enset fabric reinforced green composite tensile test results (8 warps per sample)

Acacia to frankincense bio resin mixed ratio (%)	**Force (N) (Average value)**	**Elongation (%) (Average value)**
50 : 50	***2356.50***	***5.70***
55 : 45	2416.91	6.35
60 : 40	2486.71	7.07
65 : 35	2574.4	7.86
70 : 30	2675.2	8.35
75 : 25	2851.6	9.08

Table 4.6. Single layer enset fabric reinforced green composite (8 warps per sample)

Max. Force (N)	**Max. Percentage elongation (%)**
2292.5	5.38
2421.33	5.78
2315.43	6.07
2350.04	6.02
2405.26	5.52
Average = 2356.50	**Average = 5.70**

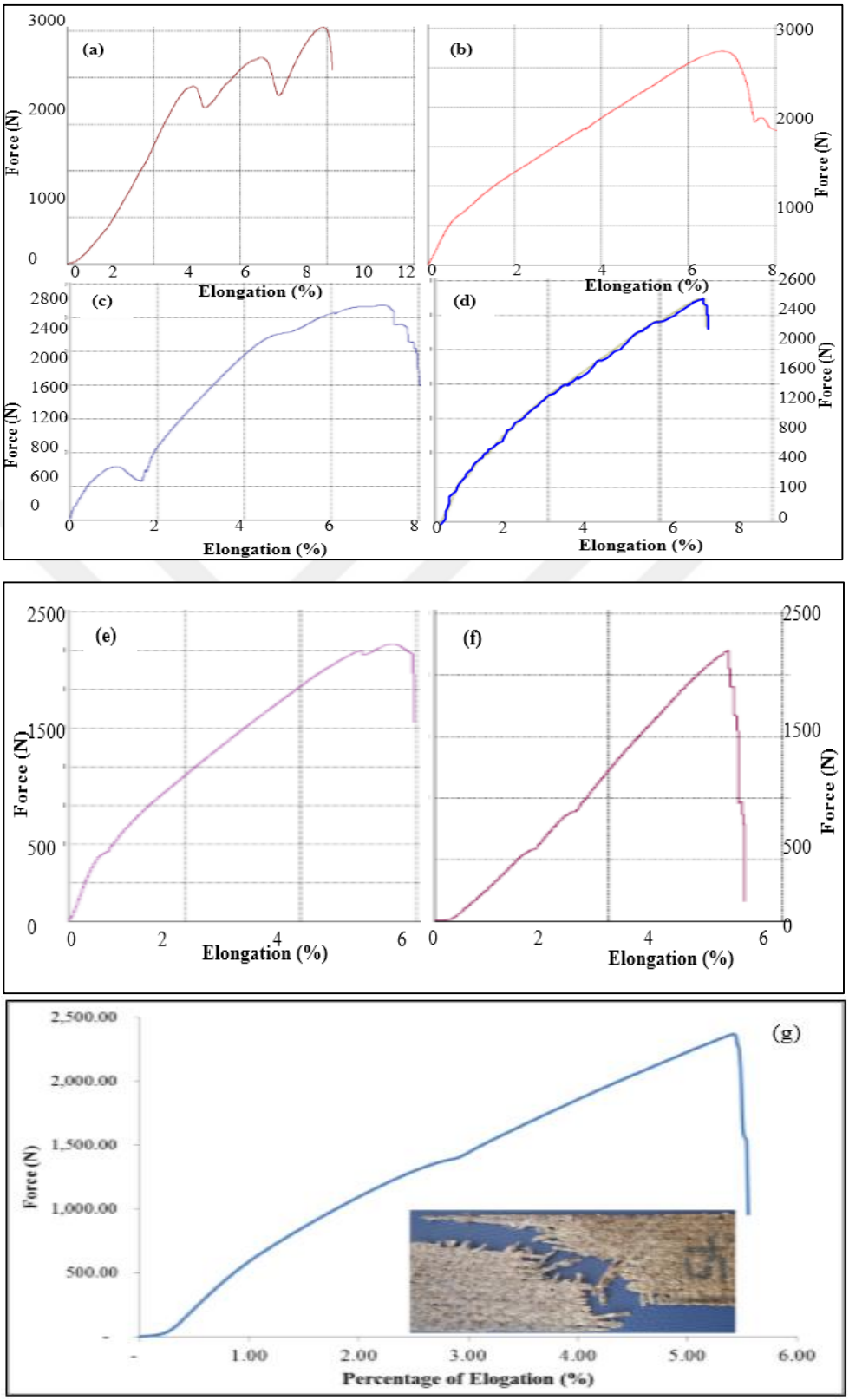


Figure 4.8. Single layer enset fabric reinforced green composite via acacia to frankincense bio resin mixing ratio (%) a) 75:25 b) 70:30 c) 65: 35 d) 60:40 e) 55:45 f and g) 50:50

The bio composite tensile strength and elongation were further investigated by producing double layer composites with again the same material to resin ratio (30%:70%). Bio resin was also prepared with 50%:50% mixing ratio as was explained above. Tensile test results are presented in Table 4.7 and Figure 4.9. The average tensile strength of double layered enset fabric reinforced composites increased to 4671.53 N which corresponds to almost the twice that of single layer composite. Hence, double layer composite tensile strength become 500% higher compared to that of double layer enset fabric fabric. During the tests, the damage occurred in the fabrics and they were separated into two pieces together with the bio resin matrix. No delamination was observed in the double layer composite during tensile tests. This revealed that there was a good interface between the bio resin and enset fabrics and also good match between the prepared bio resin and enset fabric. Breaking shape of the double layer composite was the same as that of single layer one. But, percent elongation at break increased very small amount to 6.05%. Absence of delamination, having the same breaking mechanism with both single and double layer composites and very close breaking elongations and almost twice of single layer strength with double layer composite have indicated a good match between enset fabric and the developed new bio resin to manufacture a new green composite material. From the above discussion, it can be said that agro-based resin matrix and enset fabric would have a potential and promising future for the manufacturing of eco-friendly light weight composite materials.

Table 4.7. Double layer enset fiber fabric reinforced green composite (8 warps per sample)

Max. Force(N)	**Max. Percentage elongation (%)**
4708.59	6.78
4667.09	5.59
4679.27	5.40
4657.05	7.12
4646.46	5.52
Average = 4671.53	**Average = 6.05**

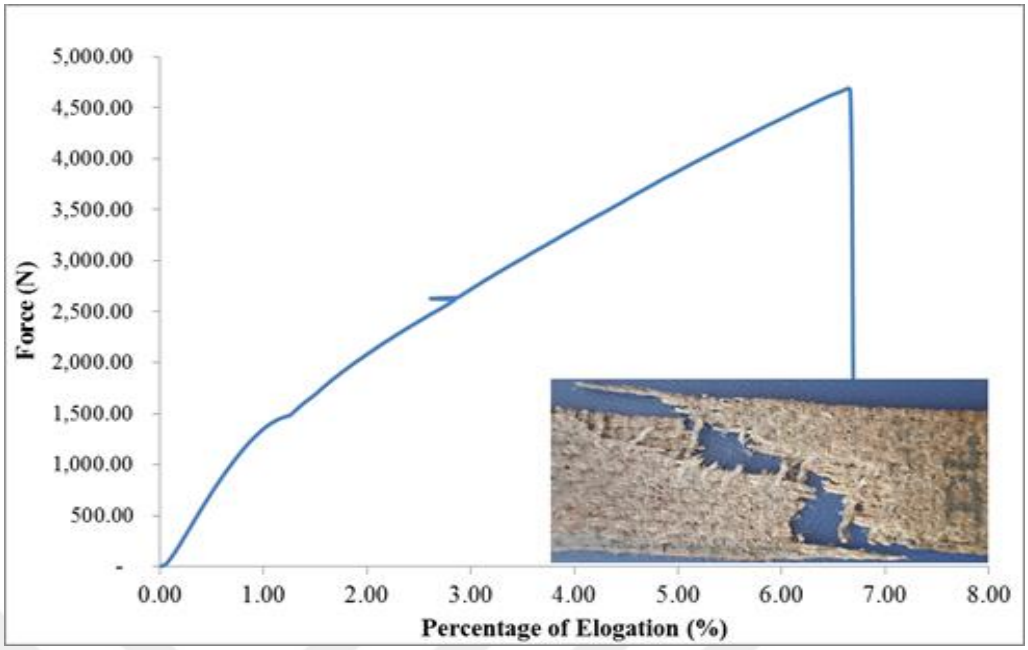


Figure 4.9. Double layer enset fabric reinforced green composite (2x8 warp ends)

4.2.2. Flexural strength result analysis

Enset fabric reinforced composite specimens were prepared based on the ASTM-D-256 standards (65X 12X 3 mm^3). The test was conducted by using universal testing machine with 3-point flexural testing method and the flexural characteristics of enset fabric reinforced green composite structures were analyzed and evaluated at standard atmospheric conditions (temperature of 20 ± 2 ^{0}C and RH of 65 ± 2 %). The force was applied by the universal testing machine until the enset fabric reinforced green composite specimens were broken and fractured. The flexural test of enset woven fabric composite was conducted by using 5 and 10 bar pressures as shown in Table 4.8 and Figure 4.10a and 4.10b. The maximum flexural strength of woven fabric reinforced composite structure was found as 76.43 N/mm^2 and 151.19 N/mm^2 for 5 and 10 bar tests respectively. At 5 bar of applied load, the green composite specimens were not broken, rather the two layers of the composite materials started to separate from each other (delamination) for double layered structure as shown in Figure 5b. When the pressure was increased to 10 bar, the specimens were broken into two pieces as shown in Figure 4.10b. Also, Figure 4.10a and 4.10b clearly showed that enset woven fabric reinforced green

composite specimens were yet carrying the applied load after the force-elongation curve deviated from linearity (Hook's Law) for both 5 bar and 10 bar applied pressures during 2 and almost 3 second respectively. When the crack propagation reached its critical length, the composite materials started to lose their load carrying capability and it resulted with damage in the composite structures without showing breakage. This phenomenon indicated that the green composite formed by using enset woven fabric and bio resin materials showed rapid damage without an excessive elongation due to enset fabrics and plastic nature of used bio resin as shown in Figure 4.10a and 4.10b.

Table 4.8. Average result of the three point bending test

Description		**Thickness (mm)**	**Maximum force (N)**	**Maximum flexural strength (N/m^2)**
Average Values	5 bar pressure	3.5	115.24	76.43
	10 bar pressure	3.5	227.95	151.19

According to Pupure et al. (2015) report, composite materials reinforced with regenerate cellulose based fibers exhibit high nonlinearity while bio resin shows relatively lower nonlinearity during mechanical tests. The nonlinearity behavior of bio based resins gave information about the stiffness and the strength of resin materials. For example, epoxy and tribest resin material show higher nonlinearity in their flexural test result curves after reaching a strain of 2% than bio based resins (Pupure et al. 2015). The limitation of natural fibers and bio resin materials are solved by treating the reinforcing materials by different type of treatments such as alkalization (Benkhelladi et al. 2020). Surface modification treatments of fibers have a significant effect on the topography of the fiber, which affects the flexural strength of the fiber reinforced composite structure by enhancing the interface adhesion between the fiber and matrix (Alawar et al. 2009, Zhou et al. 2014).

As shown in Figure 4.10a, the enset fabric reinforced green composite specimens were yet carrying the applied load after the force-elongation curve left the linearity (Hook's Law). When the cracking started and propagated in the specimens until the critical length, the enset fabric reinforced composite materials started to lose their force (load) carrying ability (capacity) and resulted with damage of composite structures without showing

breaking. Moreover, in the second portion of the Figure 4.10a, after the maximum load was applied on enset fabric reinforced green composite materials; there were a clear indication of failure (rapid decline) in the curves. This phenomenon indicated that green composite material structure made by using enset fabric and bio resin materials showed both rapid damage without excessive elongation due to enset fabrics and plastic nature due to used bio resin obtained from plan gums. Due to relative motions of interlaced warp and weft yarns (weaving structure), enset fabric reinforced green composite materials indicated extra ductile nature (behavior) than enset fiber reinforced or laminated green bio degradable composite materials during flexural strength tests.

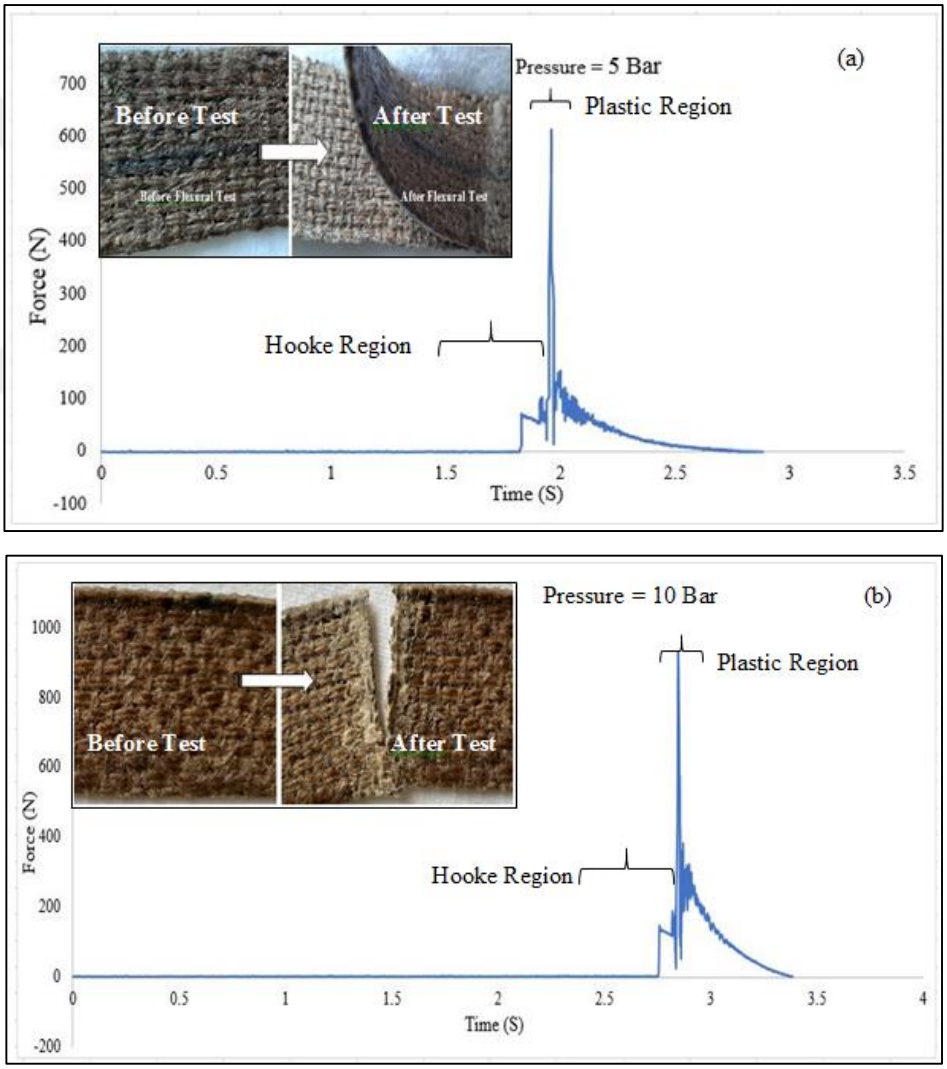


Figure 4.10. Curves of the flexural force versus the displacement enset woven matt reinforced Composite with specimens after flexural test a) 5 Bar b) 10 Bar Pressure

4.2.3. Impact Strength Result Analysis

Impact strength of the developed bio composites was investigated with 3 different enset fabric to bio resin ratios, which are 20%:80%, 30%:70% and 40%:60%. The impact energy was dissipated by delimiting of enset fabric and bio resin material during enset breakage and enset yarn pull out as shown in Figure 4.11a. The impact strength of enset fabric reinforced composite structures decreased with the increase in enset fabric ratio as indicated in Figure 4.11b. This test result indicated that the increased content of enset fabrics beyond the desired quantities during composite manufacturing was ineffective to enhance or improve the brittleness of bio resin materials. The impact strength variation was observed due to the difference in impact respond between enset fabric and bio rein materials. In addition, it was an indication for inferior adhesions between the reinforcing fabric and resin materials during green composite manufacturing process. From the three fabrics to bio resin compositions of composite structures, 20 % fabric and 80 % bio resin composite structure had a better impact absorption as shown in Table 4.9. This approved that 20% of enset fabric and 80% of bio resin reinforced composite structure had better interfacial bond within enset fabrics and bio resin materials.

Table 4.9. Average of impact strength of enset fabric reinforced composite structures

Fabric to bio resin material composition	**Thickness (mm)**	**Width (mm)**	**area(A) (mm^2)**	**Actual energy absorbed, K (J)**	**Calculated impact strength (J/mm^2)=(K/A)**
20 % fabric and 80% bio resin	3.5	10	35	68.5	1.96
30 % fabric and 70% bio resin	3.5	10	35	53.6	1.53
40 % fabric and 60% bio resin	3.5	10	35	52.6	1.50

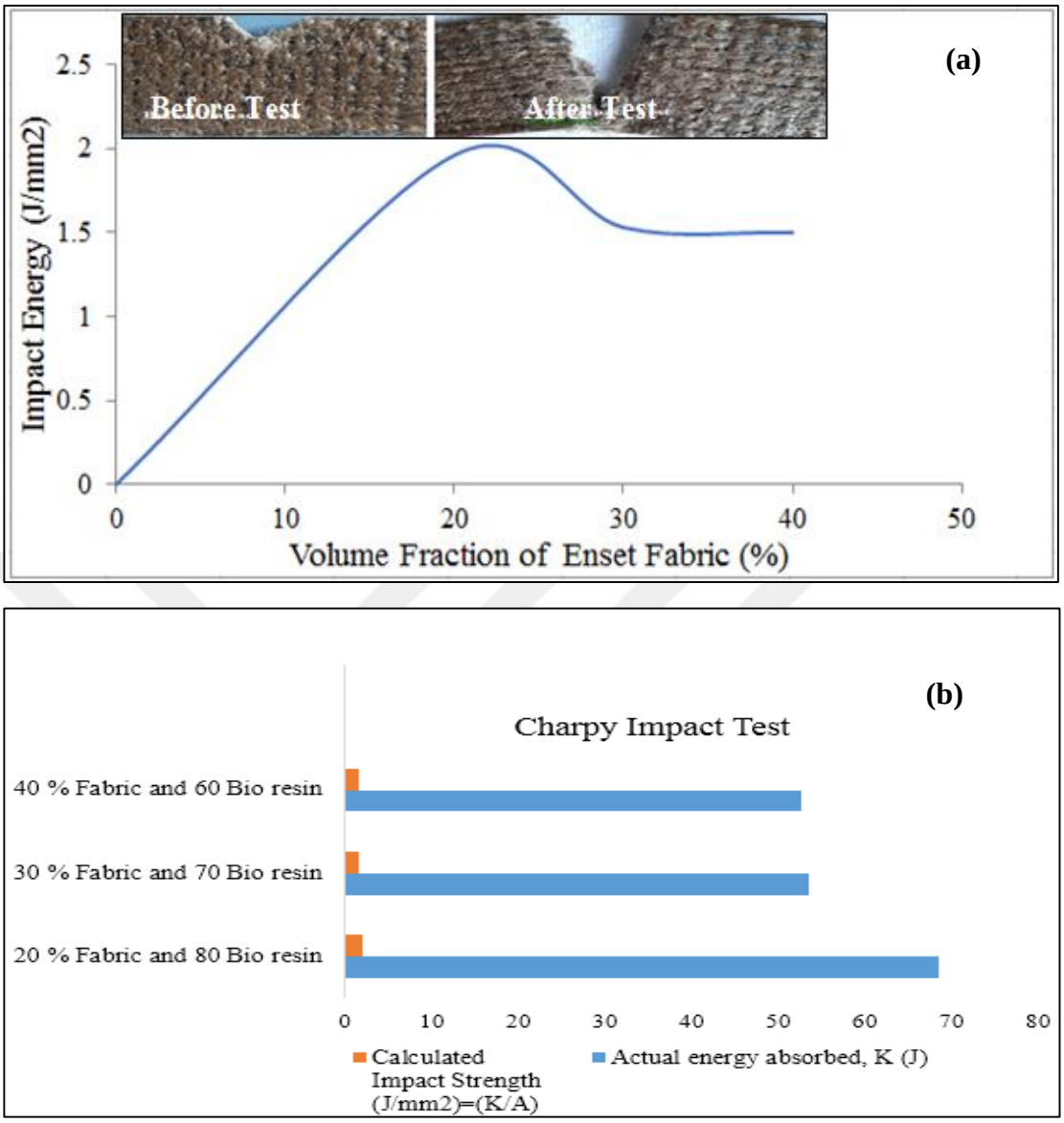


Figure 4.11. Flexural strength test results a) Composite sample after impact test b) Effect of percentage of fabric content on impact absorption properties

It is understood from the analysis of mechanical tests that the enset fabric reinforced bio degradable composite show different performances for tensile, bending and impact forcing depending on the ratio of bio resin components as well as enset fabric to bio resin ratio in the composite. The composite can be designed with best parameters depending on which application it is to be used.

4.3. Morphological Analysis of Enset Fabric Reinforced Green composite

The morphological view of enset fabric reinforced composite materials illustrated in Figure 4.12 revealed that the interaction and interfacing bond between enset woven fabrics and bio resins have been a promising futures for acacia and frankincense gums to be used together as the biological degradable matrix materials. These bio resins also give relatively lower level fiber protruding (pull out), better resistance to delamination, good bonding and low fiber breakage compared with biopolymer matrix materials as shown Figure 4.12a. Moreover, the SEM results illustrated in Figure 4.12b to 4.12d of enset fabric reinforced composite materials indicated that the enset fibers protruded from the composite structures during different mechanical test, i.e. tensile, flexural and impact test respectively. This phenomenon revealed that the enset fibers were broken and then the bio matrix started to get fallen. The broken enset fibers indicated that the fibers were start to fall after enduring a moderate mechanical load and the composite materials will be broken after the maximum load was revived by the structures.

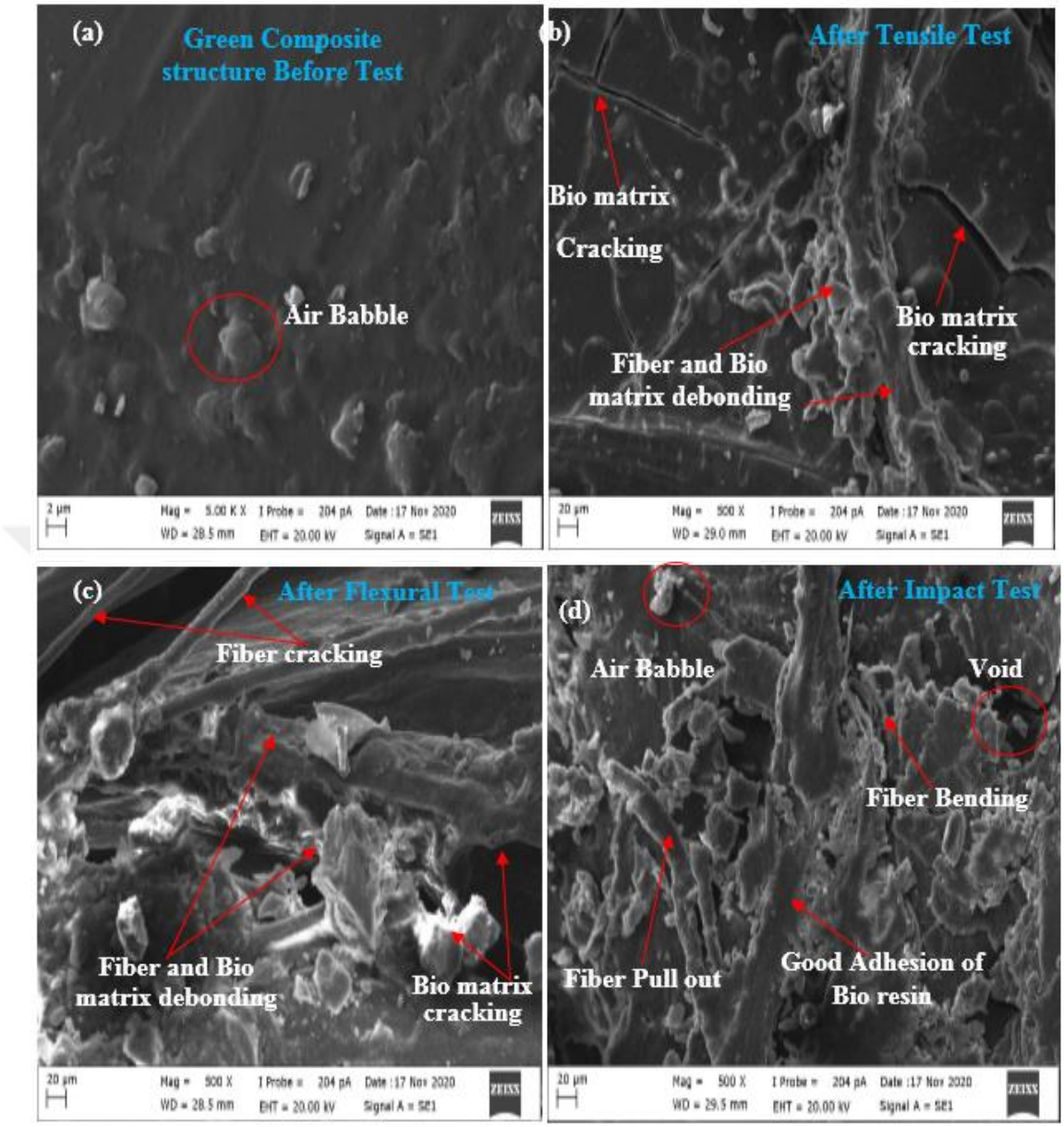


Figure 4.12. SEM: Morphological view of bio resin reinforced green composite structure during mechanical testing a) Composite structure before test b) Tensile strength c) Flexural strength d) Impact strength test

The observation made from Figure 4.13a and 4.13b have shown that the image of surface fracture and cracking propagation of enset fabric reinforced composite structures are seen and analyzed by using optical microscope and SEM after the applying different type of loads or strength tests such as tensile, impact and flexural strength. The optical microscopic view confirmed that, the propagation of the crack through enset fabric reinforced material into bio resin interface of the samples for different types of tests as

shown in Figure 4.13a at point A, C and E. These situations were confirmed and used as an evidence of start of crack propagation of the composite structures and the deboning of enset fabrics from the bio resin matrix would happen through the crack path. The scanning electronic microscopic morphological analysis of enset fabric reinforced composite structures has revealed that there were a lot of smooth grooves (porosity) in the green composite structures and the cracks started and would be propagated to enset fabrics- bio resins deboning at these grooves as shown in Figure 4.13b.

Also, the bio matrix started to crack and the majority of enset yarn in the woven fabrics was broken in the warps direction. As seen from Figure 4.13a and 4.13b at a point of A, B, and D, the weak bonding interactions were observed between the bio matrix and enset fabric (due to higher voids areas). On contrary, the rest part of the composite showed tight packed portions (more or less there is no void areas in the structures). All the above phenomena have been a significant effect on the overall mechanical performance of enset fabric reinforced composite materials. So, good mechanical properties of enset fabric reinforced composite was obtained by rescuing the percentage of void ratio (porous space occupied in the composite structure) and using the optimum amount of reinforcing materials, i.e.70: 30 % of bio resin to enset fabric ratio.

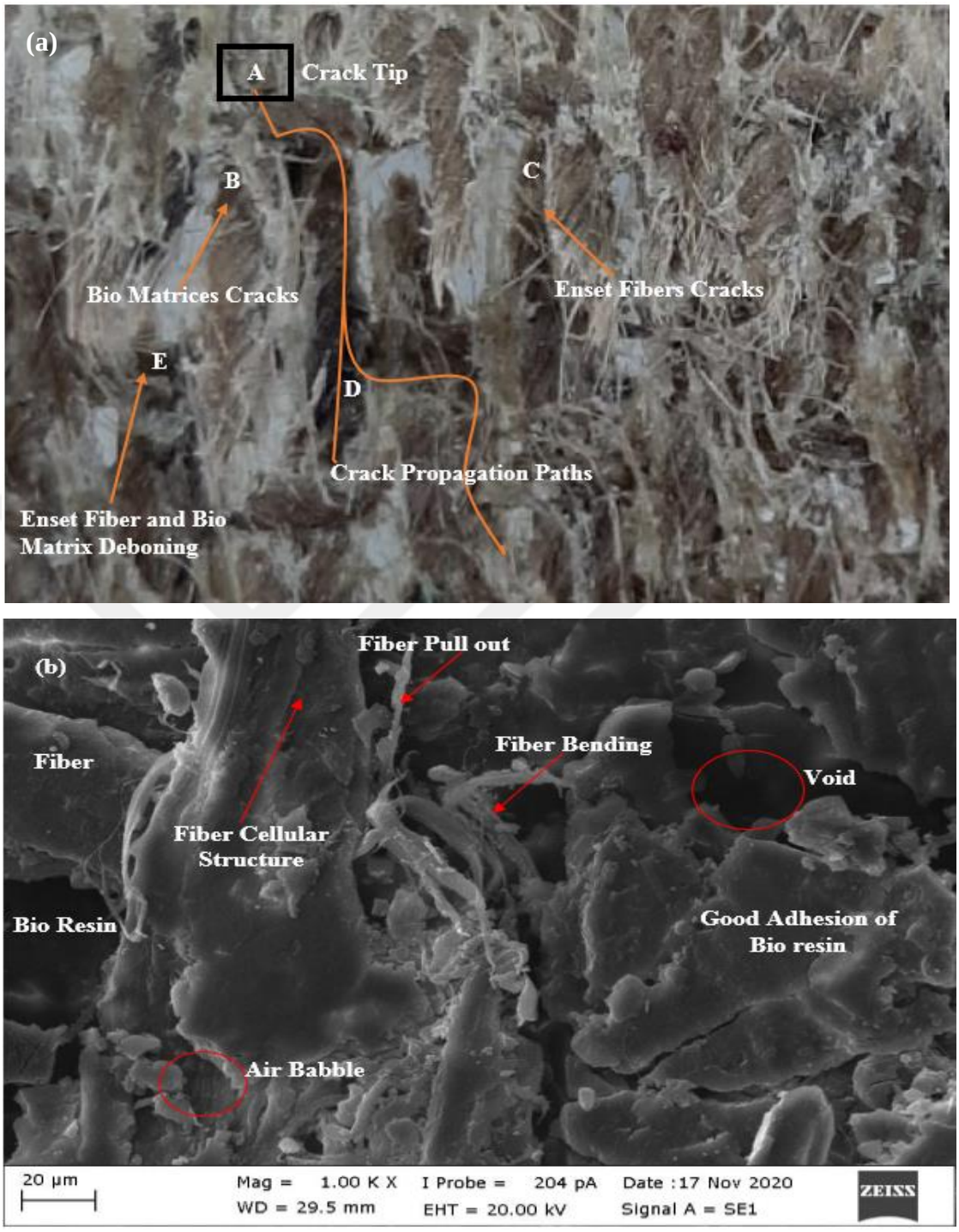


Figure 4.13. Crack propagations analysis of enset fabric reinforced composite structures during mechanical testing view a) Optical microscope b) SEM

4.4. Acoustic Properties of Enset Fabric Reinforced Composite Structure

In the literature, most of the studies have focused on the effect of porous and void structure on sound absorption as well as sound insulation characteristics of textile materials such as non-woven, different natural and synthetic fiber reinforced composites at lower, medium and higher frequencies regions (Lee et al. 2017, Leitao et al. 2018, Özdil et al. 2020, Vikas and Samrat 2020). Mannning and Panneton (2013) studied the sound absorption properties of thermal bonded, resin bonded and needle punched textile materials made from industrial recycled fibers. The study revealed that at lower frequency ranging from 0 to 1000 Hz, the sound absorption coefficient value was obtained as 0.20 (Mannning and Panneton 2013, Özdil et al. 2020). Likewise, Patnaik et al. (2015) studied the sound absorption properties of needle punched nonwoven textile waste materials by mixing wool and PET fiber with a ratio of 50/50. According to this report, a good acoustic properties were obtained at lower, medium and higher frequency regions from 50 Hz to 5700 Hz. For all samples, at lower frequency ranging from 1000 Hz to 2000 Hz, the sound absorption was obtained as 0.61 while at higher frequency ranging from 2000 Hz to 5700 Hz, it increased to 0.75 (Patnaik et al. 2015, Özdil et al. 2020). Özdil et al. (2020) reported the sound absorption characteristics of recycled textile materials. According to the study, the sound absorber made from recycled textile materials was used as a sound insulator in lower frequency region from 100 Hz to 400 Hz and it could substitute conventional fibers used in acoustic applications. At medium frequency ranging from 400 Hz to 1600 Hz, the sound absorption coefficient values of PP and PES fabrics were greater than 0.5 (Özdil et al. 2020). In this study, the sound absorption properties of enset fabrics and their reinforced bio composite structures at lower, medium and higher frequencies were explained in detail.

4.4.1. Morphological analysis of enset fiber for sound absorption

As seen in Figure 4.16a and 4.16b, the enset fibers have a lot of porous and void structure in the longitudinal direction of the fiber axis. The porosities and voids present on the fiber structure could have a significant advantage in the sound proof and acoustic applications by dissipating sound energy (wave) through air molecules entrapped in the wall of the fibers (see in Figure 4.16c). The friction and collapsing of air molecules in the wall of the

enset structure would result sound energy loss as a form of heat loss caused by the heat exchange between air molecules through porous structure of the fiber (Mariellen 2018, Vikas and Samrat 2020). This effect can be contributed by manufacturing of multilayer and sandwich textile structures. The main factors affecting sound absorption properties of fabric reinforced composite structures were not only fiber diameter and fabric bulk density but also the lumen's diameter (see Figure 4.16d) (Zhange et al. 2018, Vikas and Samrat 2020). Hollow lumen structure of fibers and their arbitrary distribution is expected to support for the better acoustic properties of enset fiber structures.

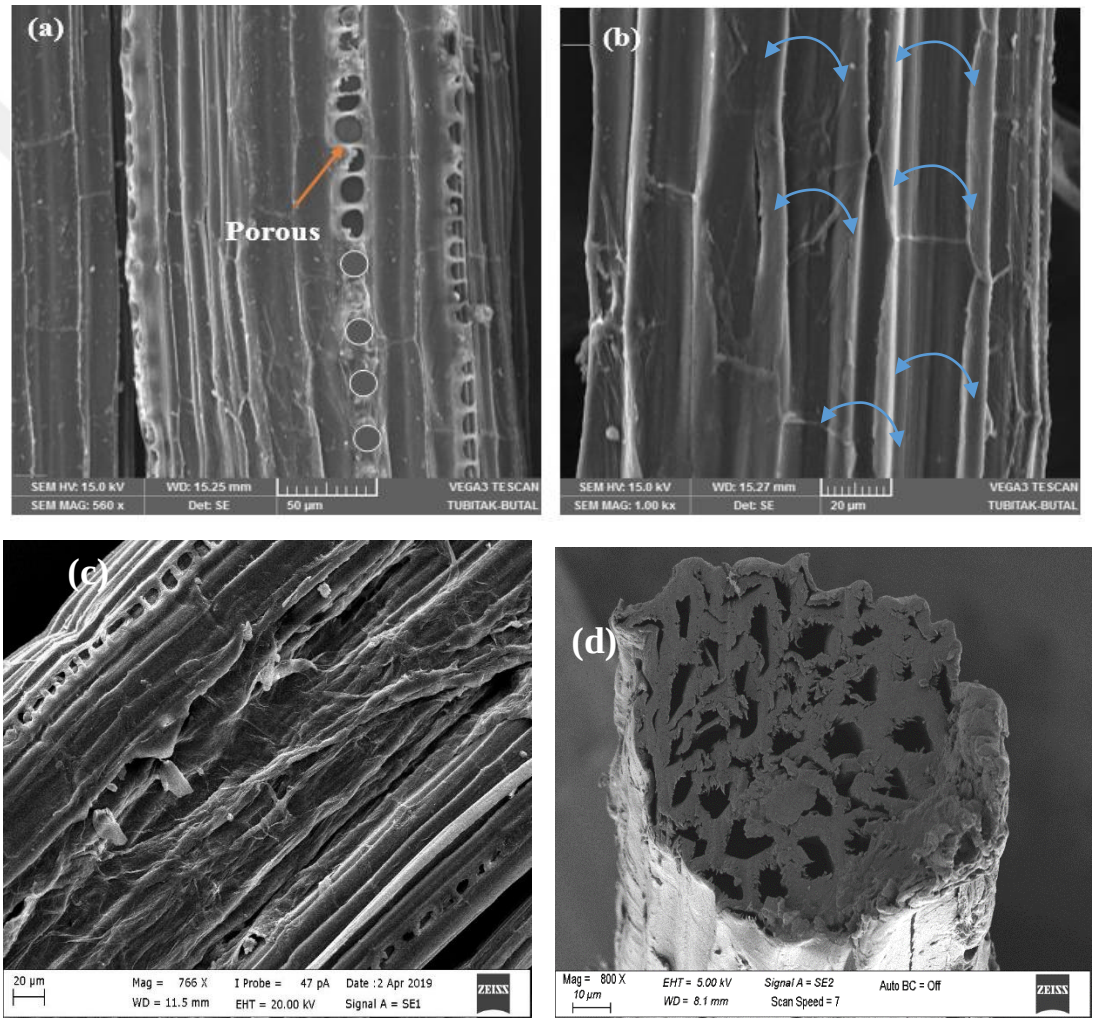


Figure 4.14. SEM of enset fiber a) Micro porous structure on the fiber b) Longitudinal and tangential porous structure of enset fiber c) Multilayer void structure of enset fiber d) Enset fiber cross sectional view

4.4.2. Acoustic properties of enset fabric and its composite structures

The absorption of sound in pore and void region of the enset fiber would be a key factor that influences the noise insulation performance of the enset fabrics and enset textile structures. This might make the fiber having great attractive properties for interior and exterior applications. Enset fabric and its composite have a number of porous space in their structure. The propagation of sound wave through enset fabric reinforced composite is restricted by air molecules entrapped within the porous structures, creating a friction between them and dissipating the sound energy (wave) in the form of thermal loss. Figure 4.17a shows sound absorption coefficient of single layer enset fabric backed by polyurethane foam of 6mm thickness. The curve below belongs to the polyurethane foam. As seen from the curve, using enset fabric with backing foam increased sound absorption coefficient at all frequencies and it reached around 0.55 at 5500 Hz. Used foam thickness for the sound absorption in Figure 4.17a was 6 mm. When the foam is substituted with an enset fabric having the same thickness, the maximum sound absorption increased to around 0.8 (it has the same value as 4 layer enset fabric) as shown in Figure 4.17b. Enset fabric might be used as an alternative for sound absorber applications at high frequencies with low cost of manufacturing, abundantly availability, low bulk density and eco friendliness.

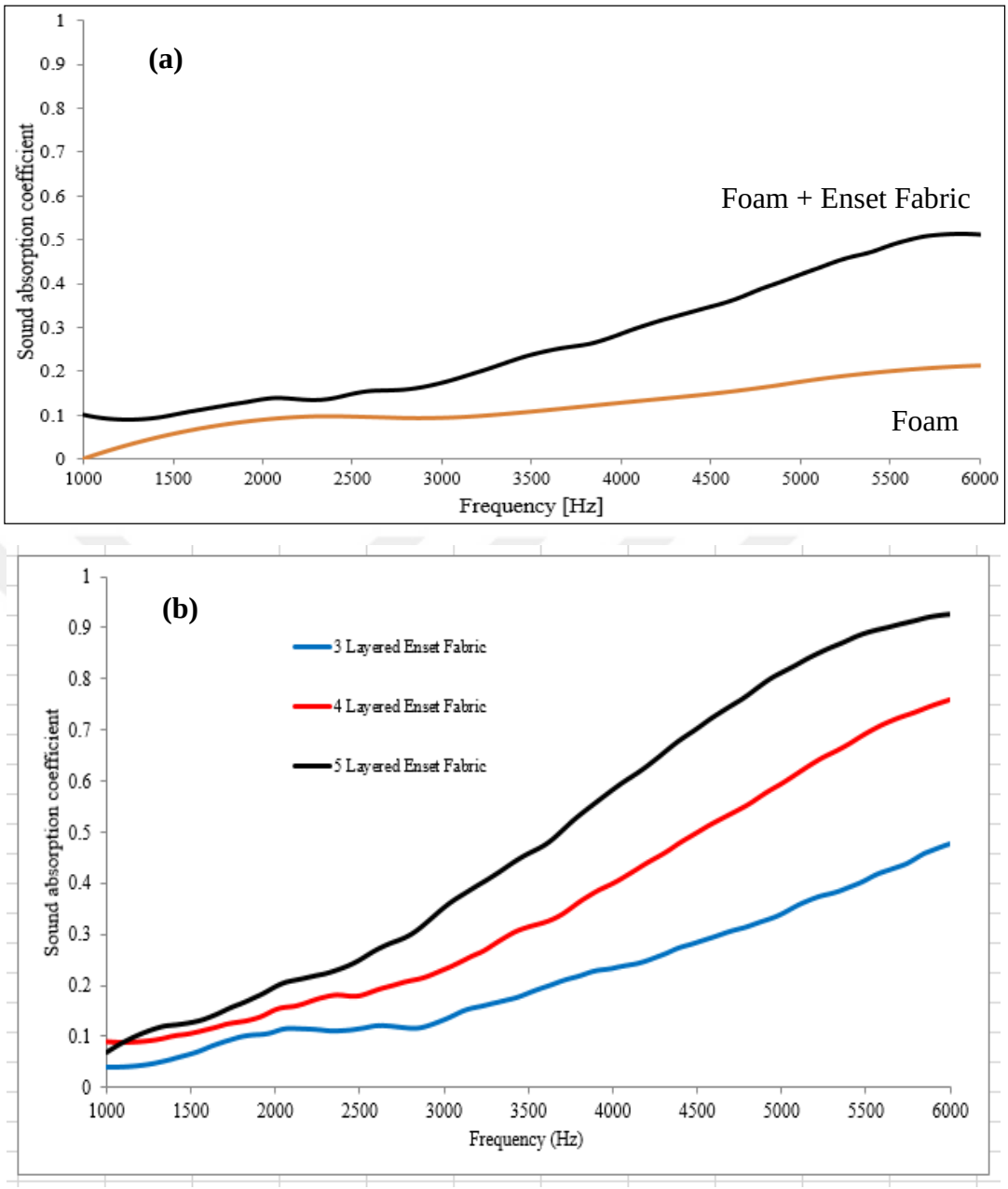


Figure 4.15. Average sound absorption coefficient of a) Enset fabric (2 mm thickness) with foam b) Multilayer enset fabric without foam

Figure 4.18 shows the sound absorption coefficient change with respect to the sound frequency for single layer enset fabric composite with backing polyurethane foam. As seen from the figure, using enset fabric composite improved sound absorption significantly. Composite enset fabric structure not only improved the sound absorption level but also shifted the sound absorption to mid frequency range. This absorption mechanism of enset fabric composite can be explained by the structure of the composite.

In the enset fabric composite production, hand laying technique was used and the porosities and voids in the fabric structures were not fully filled by the bio matrix materials. It is thought that the remaining porosity and voids in the composite structure contributed significantly the sound absorption. The sound absorption coefficient started with near to 0.2 at 1000 Hz and tended to increase at a slower rate up to around 1750 Hz. It reached to around 0.6 at 3000 Hz and 0.9 at 4000 Hz. The maximum sound absorption was obtained as 0.96 at 4500 Hz. Considering Figure 4.17a and Figure 4.17b reveals that layering of enset fabric structures one with another significantly improved the sound absorptions coefficient at medium and higher frequency regions in comparison to enset fabric and foam combinations at the same sample thickness. Large porosities and voids between yarns in enset fabric can be seen as a reason for this difference. Bio resin in composite production blocked these voids and improved the sound absorption. This can be explained by air permeability results presented in Figure 4.19.

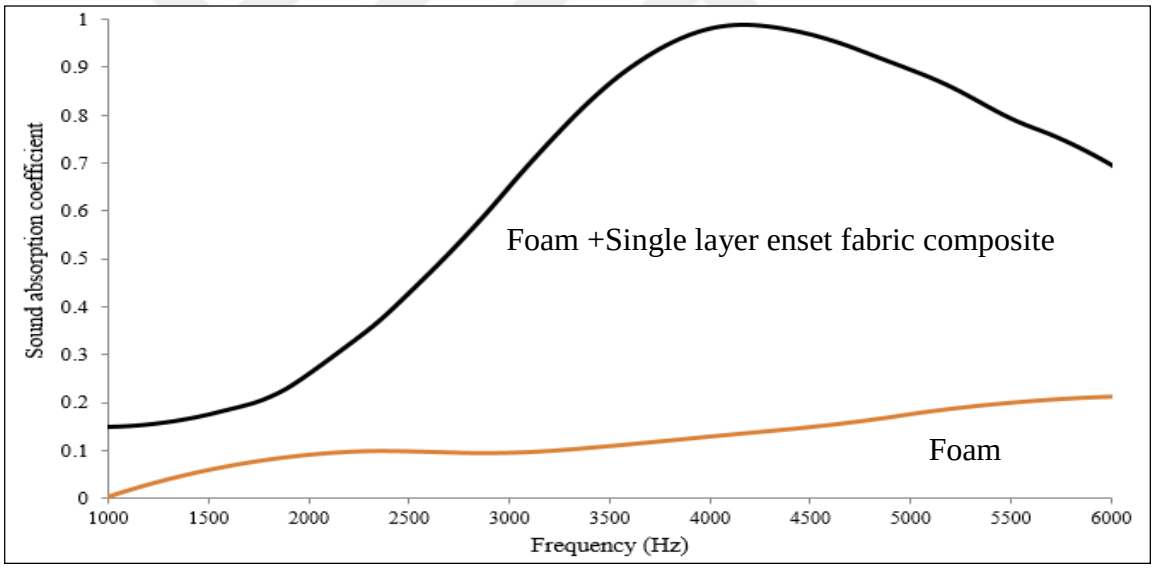


Figure 4.16. Average sound absorption coefficient of single layer green composite (2.5 mm thickness)

The intra and inter porosity of the fabric highly affects the transportation of air through the composite and enset fabric structures. Analysis of the data in Figure 4.19 indicates that the air permeability of enset fabric was significantly affected by the structure. The average air permeability of enset fabric was 3046 l/m^2/sec while the value of air permeability drastically decreased by single and double layer enset fabric reinforced composite structures. Single layer and double layer enset fabric reinforced composite

structure had air permeability values of 1846 l/m^2/sec and 1164 l/m^2/sec respectively. These results indicate that the porous and void structure of enset fabric was highly affected by the used bio-matrix. Also, the amount of bio-matrix on the composite structure had tangible effect on the air permeability characteristics of the composite structure. The air permeability results confirmed that the porosities formed in the composite structure had a significant effect on the acoustic properties by reducing the sound energy due to friction between oscillating air molecules in the pores (Suvari et al. 2016, Lee et al. 2017).

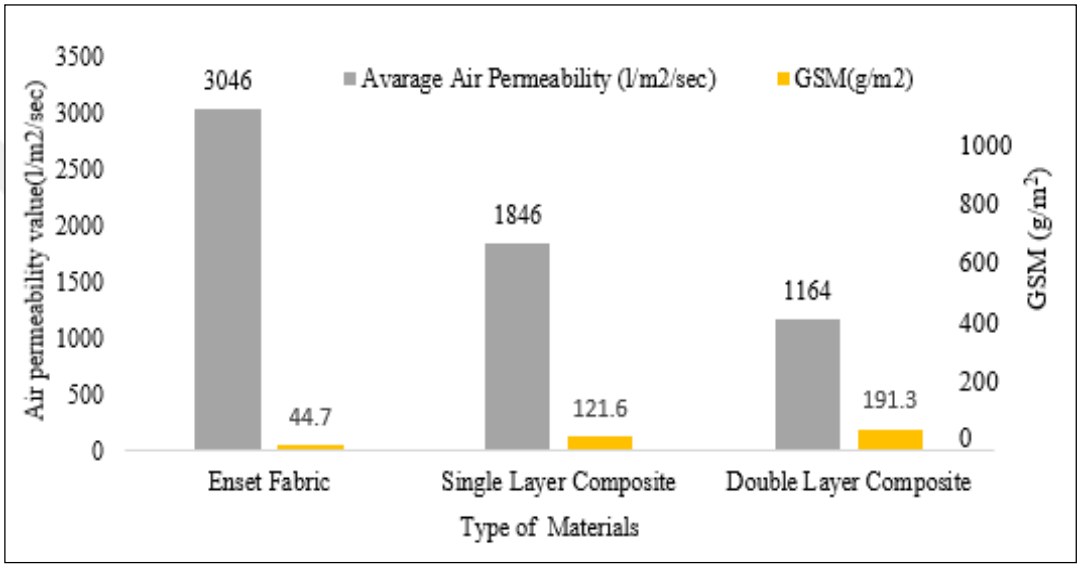


Figure 4.17. Air permeability of enset fabric and its green composite

4.4.3. Effect of fabric layer on acoustic properties of enset reinforced composite structure

The effect of number of fabric layer on the sound absorption was investigated by 5 different fabric layer sound absorption measurements. In the measurements only enset fabrics were used. Polyurethane foam was not included. Number of fabric layers was taken as 1, 2, 3, 4 and 5 in the measurement. The results are shown in Figure 4.20. As seen from the curves in the figure, the sound absorption coefficient increased very significantly with increasing number of fabric layers. Increase in the sound absorption became more pronounced after 2500 Hz. With single layer fabric, the sound absorption showed only a small increase reaching to 0.1 at 5000 Hz whereas it became 0.5 at 3500 Hz and 0.81 at 5000 Hz in 5 layer enset fabric. The sound absorption increased between

these values as the fabric layer changed from 2 to 5. But increase in fabric layers increased the sound absorption at the expense of increasing fabric thickness. Five layers in the experimental fabric sample corresponded to around to 10 mm. This and large thickness fabrics can be used in applications where there is enough space available. Sound absorption of polyurethane foam plus single layer fabric amounts to 0.55 at 5500 Hz whereas it became 0.69 at the same frequency with four layers of fabrics. Considering the same thickness of both structures, four layer fabrics becomes advantageous with higher sound absorption and biodegradable properties (Mariellen 2018).

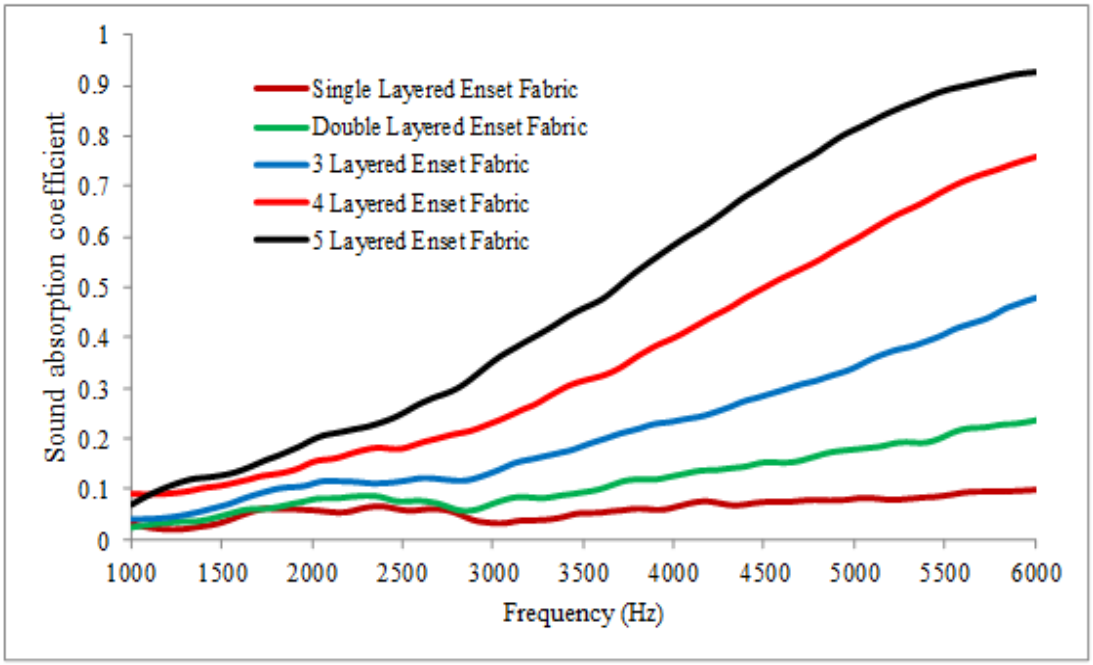


Figure 4.18. Effect of enset fabric number of layers on sound absorption properties

To investigate the sound absorption properties of multilayer composite structures, the measurements were conducted by using 2, 3, 4 and 5 layers composite samples without polyurethane foam. Composites samples were produced with 70:30 wt. % resin to enset fabric weight ratio. The results are presented with respect to sound wave frequency in Figure 4.21. The sound absorption curves with composite structures differed from sound absorption curves of multilayer fabrics as in Figure 4.21 in that the maximum absorption values were reached at lower frequencies with composites. The maximum sound absorption was obtained at 4250 Hz as 0.95 with five layers (around 10 mm thickness) while it was reached at 5000 Hz as 0.95 with four layer samples. For two and three layers,

the sound absorption coefficient is measured as 0.34 and 0.83 respectively at 6000 Hz. With five layers composite a sound absorption coefficient remains over 0.5 at a large frequency interval of 2830 and 6000 Hz. This result can be attributed to fibrous and porous structure of enset fabric reinforced composite. A similar trend was observed in sound absorption curve of four layers composite. The results in Figure 4.21 suggests that the sound absorption frequency interval can be drawn to lower frequency range by increasing number of layer of enset fabric bio-composite.

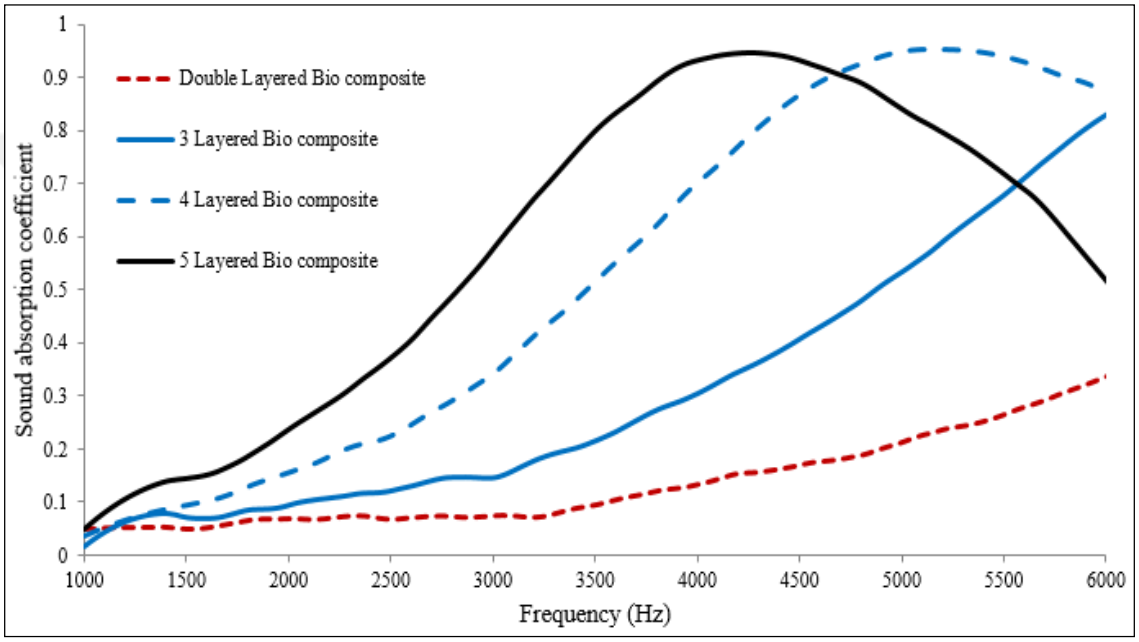


Figure 4.19. Effect of green composite layering on peak shifting of sound absorption region

4.4.4. Effect of bio matrix weight ratio on acoustic performance of green composite

As a better sound absorption performance was obtained with enset fabric bio-composite, it was decided to investigate the effect of matrix bulk density on acoustic performance of the composite. For this purpose 80:20 wt. % and 70:30 wt. % enset fabric reinforced bio-composites were produced and sound absorption measurement were conducted with 2, 3, 4 and 5 layers of composites without polyurethane foam. The results are shown in Figure 4.22. As Figure 4.20 shows multilayer sound absorption curves for only enset fabrics without bio matrix materials, they will be used in this part for comparison purpose. The sound absorption results of 70:30 wt. % bulk density composite are presented in Figure

4.21 and they will also be here for comparison. Putting Figure 4.20, Figure 4.21 and Figure 4.22 side by side shows a very explanatory change about the effect of bio matrix material on sound absorption behavior. Without bio matrix material, the sound absorption of only enset fabrics increased with increasing number of layers at high frequency range towards 6000 Hz (Figure 4.20). In all layers the sound absorption coefficient continuously increased up to 6000 Hz. As seen from Figure 4.21, when the enset fabric was converted to composite structure with 70:30wt. % bulk density the sound absorption occurred at lower frequencies with higher absorption coefficient. Increasing bio matrix ratio in composite structure to 80:20 wt. % bulk density produced a significant result of sound absorption. According to Figure 4.22, the sound absorption curve shifted towards lower frequency range. Also sound absorption interval of 0.5 to 1 corresponds to lower frequency interval between 2000 and 3800 Hz for five layers. This interval was lower compared to 70:30 wt. % bulk density for the same layer. Result of four layer composites also indicates a similar trend. This is thought to be due to the shift of the composite structure from fibrous and porous nature to more solid structure with increasing matrix bulk density.

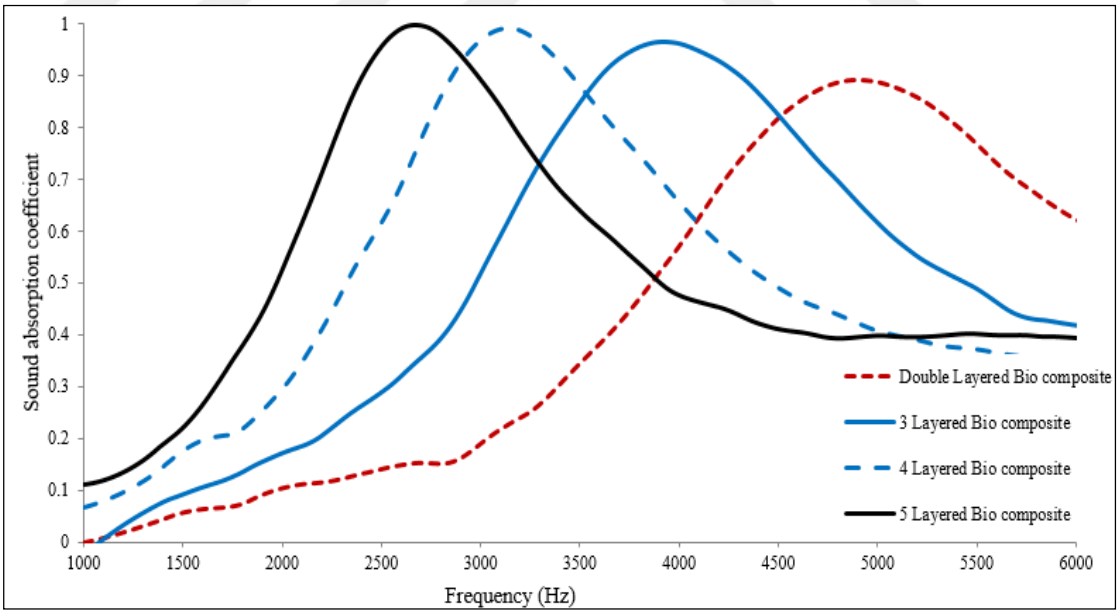


Figure 4.20. Sound absorption coefficient of enset fabric reinforced composite with 80: 20 wt. % bio matrix –fiber combination

4.5. Acoustic Properties of Green Synthesis Cellulose Enset Nano Fibers

4.5.1. Influence of enset nanofiber structure on sound absorption performance

The applied enzyme treatment and mechanical hammering defibrillated the macroscale enset into fine fibers from the surface of woven fabric and changed the morphology of enset fiber in terms of its size (diameter) from 2µm to 63 nm as shown in Figure 4.23. Enset micro fiber was formed when 10 % w/v concentration of α-amylase enzymatic treatment was applied while enset nano fibers were formed when the enzyme concentration was increased to 15% - 20 % w/v. The SEM image also confirmed the formation of the enset nano fibers as shown in Figure 4.23.

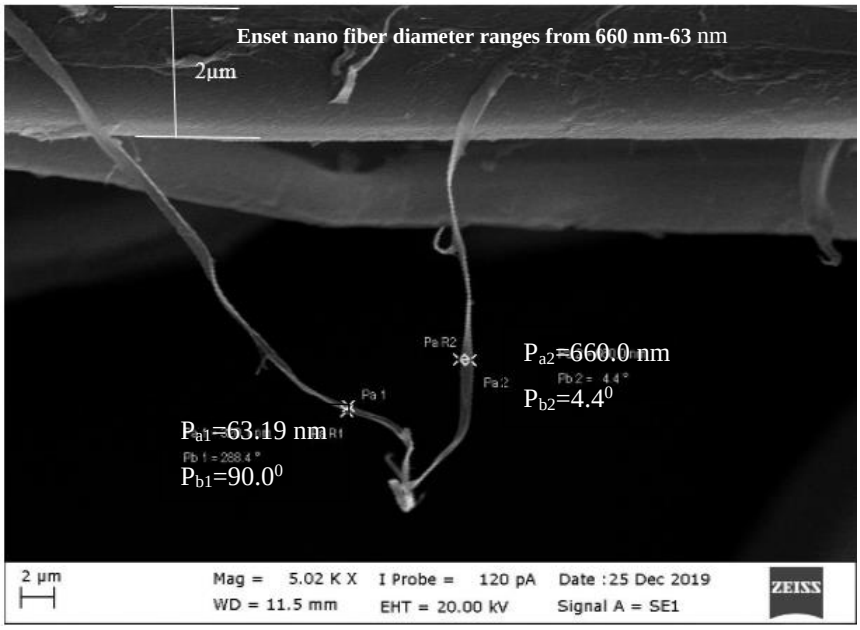


Figure 4.21. SEM: Enset nanofiber syntheses via enzyme treatment and mechanical hammering

Green synthesis of micro and nano fibers from the surface of enset woven fabrics via enzyme treatments and mechanical hammering increased the surface area of synthesized fine fibers. The higher amounts of nano fibers with increased surface area increased the friction developed between the enset nanofibers and air molecules. When sound waves entered into micro and nano enset fiber structures, its amplitude was decreased by friction as the waves try to move through the tortuous passage and dissipated in the form of heat energy (Hur et al. 2005, Ozturk et al. 2017). So, the difference between macro scale, micro and nano enset fibers in sound absorption mechanisms were observed to be quite

remarkable as shown in Figure 4.24. Also, Figure 4.24 confirmed that sound absorption performance of enset fibers could be improved when the sound absorbers were fabricated with small fiber diameters such as micro and nanometer. For the sound absorption values of enset fabric up to 3000 Hz, it is clearly seen that enset fiber (fabrics) is not efficient to trap sound wave compared with micro and nano enset fibers. The average sound absorption of enset fabrics having the same thickness (11.5 mm) with fine fibers reached the peak value of 0.62 up to 3000 Hz. But micro and nano scale enset fines fibers have sound absorption coefficients values 0.51 around 2500 Hz. and 0.73 at 2000 Hz. respectively. So, green synthesis micro and nano fibers improved the acoustic properties of enset fibers with 70% and 150% ratios respectively. Sound absorption of enset fabrics at macro scale having the same thickness with fine fibers reached its maximum peak value of 0.62 at around 4900 Hz. Whereas, the sound absorption of micro scale enset fiber which was treated with 10 % w/v concentration of α-amylase reached a peak value of 0.62 at 3500 Hz and to its maximum value of 0.85 at 4900 Hz. Similarly, the sound absorption coefficient value of enset nano fibers reached 0.96 at around 4900 Hz. The general observations made in Figure 4.24 after 3000 Hz frequency, the maximum sound absorption value of macro scale, micro scale and nano scale enset fibers were 0.62, 0.88 and 0.98 respectively. These test results revealed that green synthesis of fine fibers from the surface of woven fabric enhanced the maximum acoustic values by 43% (micro scale) and 59% (nano fiber) compared with macro scale enset fibers.

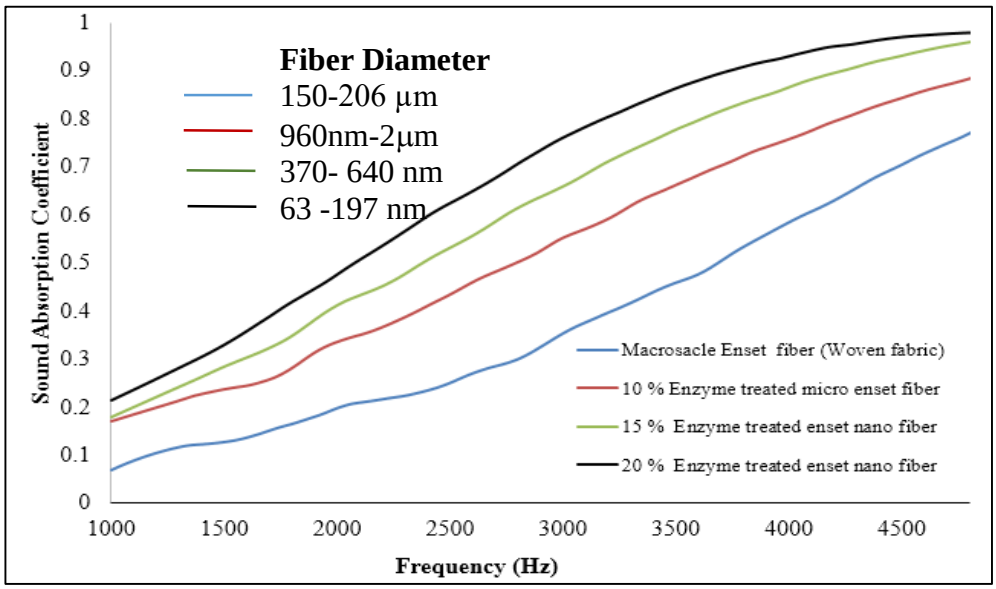


Figure 4.22. Effect of surface area on sound absorption coefficient of enset fabric, micro and nano enset fiber

4.5.2. Effect of nanofiber layer thickness on sound absorption coefficient

To study the relation between the sound absorption properties and thickness of enset nano fibers, 4 different samples treated with 3 different concentration of amylase enzymes were selected as test materials. Under similar conditions of density (354.98 kg/m^3) and with thicknesses of 10, 11, 12 and 13 mm, the sound absorption coefficients under different frequencies are shown in Figure 4.25. Based on the experimental data, all enset nano fiber samples showed an increasing sound absorption value with respect to sound frequency. All samples gave a high sound absorption coefficient at a frequency between 4000 Hz and 6000 Hz. At the frequency of 4925 Hz, enset nano fiber formed by 20% enzyme treatment with a thickness of 10 mm have shown a highest sound absorption coefficient (0.98) compared to enset nanofiber formed by 10 and 15% enzyme treatment (0.90 and 0.89 respectively) as shown in Figure 4.25a. Similarly at the frequency of 5404 Hz, enset nano fiber formed by 15 and 20% enzyme treatment with a thickness of 11 mm have shown a higher sound absorption coefficient of 0.99 compared to enset nanofiber formed by 10% enzyme treatment (0.94) as shown in Figure 4.25b. The sound absorption coefficient of the sample treated with 15 and 20% was recorded as 0.99 while for 10% treatment, it was obtained as 0.95 at 4375 Hz. For 12 mm thickness of enset nanofiber samples, the effect of enzyme treatment on sound absorption was significantly seen up to 4375 Hz (seen in Figure 4.25c). At the frequency of 4073 Hz, enset nanofiber formed by 20% enzyme treatment with a thickness of 13 mm have shown a highest sound absorption coefficient (0.99) compared to enset nanofiber formed by 10 and 15% enzyme treatment (0.95 and 0.97 respectively). For 13 mm thickness of enset nanofiber samples, the effect of enzyme treatment on sound absorption was significantly seen at lower and medium frequency as shown in Figure 4.25d. The test result revealed that increasing the thickness of the samples not only enhanced the acoustic performance of enset nanofiber, it also shifted the highest peak value from higher to lower and medium peak regions.

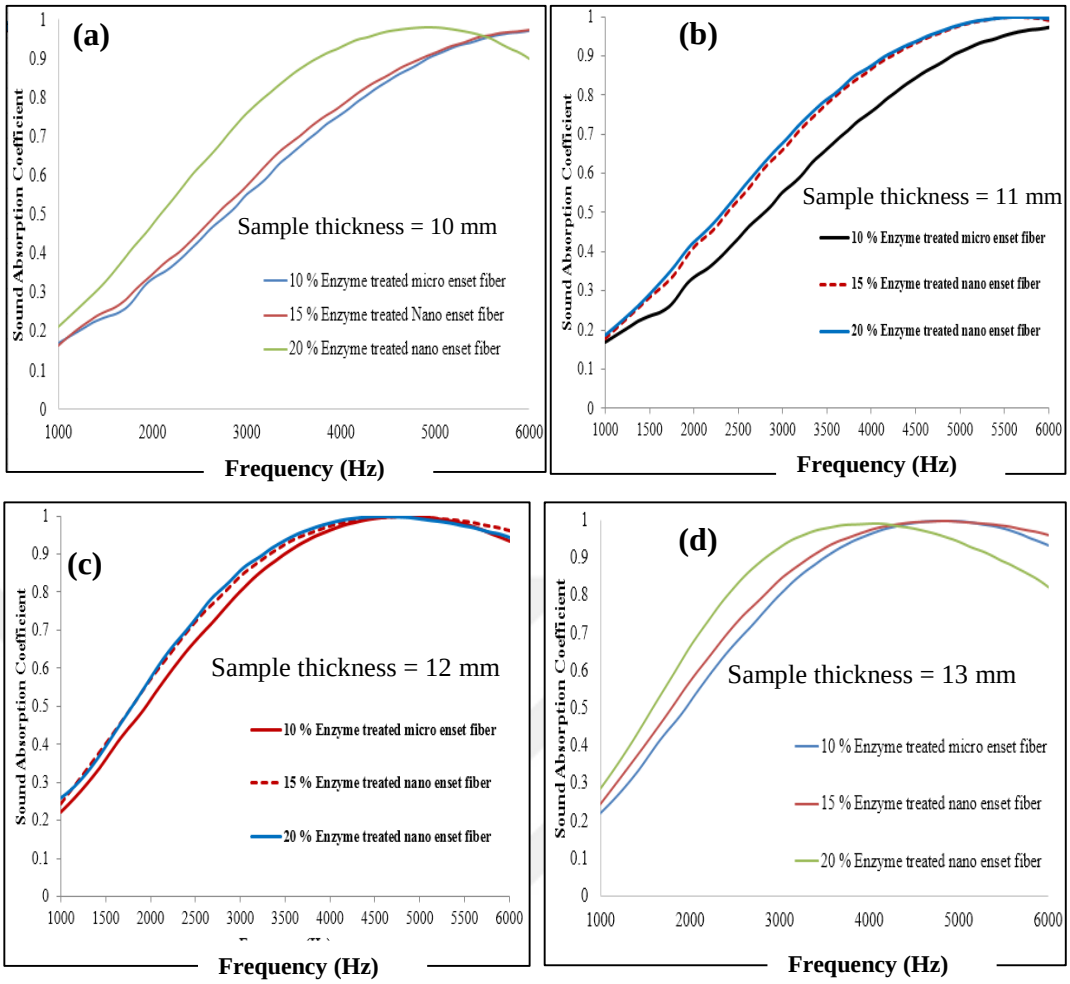


Figure 4.23. Effect of structure parameters (thickness): (a) 10 mm sample, (a) 11 mm sample, (b) 12 mm sample, and (d) 13 mm sample

4.6. Biodegradability of Fiber, Fabric and Fiber Reinforced Green Composite

The biodegradability of enset fiber reinforced green composite materials was studied in Bursa Uludağ University Görükle Campus area (soils) for one year period between 25/12/2019-25/12/2020). ÖZSOY and AKSOY (2004) reported that, Bursa Uludağ University's soils mainly contained lime, neogene clay and sometimes also contained quaternary alluvium. The investigation stated that the soils were composed of weak alkali and acidic properties with cation exchange of 4.8 - 65 cmol.kg^{-1} and occupied 90 % of Ca^{+2} and Mg^{+2} (Özsoy and Aksoy 2004). For biodegradable study, 2 different types of reinforcing materials such as enset fibers and fabrics were used for the manufacturing of enset fiber/fabric reinforced green composite via bio resins. The green composite, enset fiber and enset fabric samples were left under the soils (the soils have 10 cm depth) for

one year period as shown in Figure 4.26a, 4.26c and 4.26e. 240 gram initial weight of enset fibers, enset fabric and enset fiber reinforced green composites were used. The view of the test material after one year period is shown in Figure 4.26b, 4.26d and 4.26f. After one year, the samples were picked from the soil and their weights and mechanical strengths were measured. No bio resin was left in the composite and it was 100% biodegraded as shown in Figure 4.26a. The enset fiber, enset fiber reinforced composites and enset fabric lost their weight by 73.27%, 57.98% and 38.41%. Likewise, both enset fibers and enset fiber reinforced green composites completely lost their mechanical strength. Due to their high mechanical strength loss, it was difficult to test the mechanical strength of the samples using testing equipment as shown in Table 4.11. The strength of enset fabric was found to get less affected, although it was significant, compared to the enset fiber and enset fiber reinforced composite because of the more compact structure of the enset fiber in the fabric.

Table 4.10. Weight loss of fiber, fabric and fabric reinforced green composite within one year

Type of textile material	**Initial weight(g)**	**Weight after 6 months (g)**	**Weight after one year (g)**	**Weight loss (%)**
Enset Fiber	240	127.20	64.15	73.27
Fiber reinforced Green composite	240	176.4	100.85	57.98
Enset fabric	240	194.8	147.82	38.41



Figure 4.24. Biodegradability test of enset textile materials kept in in soil for one year a) Neat enset fiber b) Enset fiber after one year c) Fiber reinforced green composite d) Green composite after one year e) Neat enset fabric f) Enset fabric after one year

5. CONCLUSION

Utilization of agro waste products such as enset fiber and natural gum bio resin materials may play a significant role on decreasing of global warming by substituting petrochemical fiber reinforcing composite materials in light weight structural applications without affecting the desired mechanical properties like tensile strength, flexural strength, impact strength and tearing strength. In this dissertation work, the enset fiber and enset fabric were investigated to be used as a reinforcing material in biodegradable composite production and as a sound absorbing material. A new bio resin was also developed using 2 different natural gums and green composites were manufactured with different ratios such as 80:20, 70:30 and 60:40 of lab made bio resin as a matrix and enset woven fabric as reinforcing material respectively. Following conclusions can be drawn from the conducted research work.

Enset fiber has mechanical properties similar to most of the natural fibers like jute, banana, bamboo etc. Therefore enset fibers and enset fabrics can be used in composite material production as reinforcing material at the same or better quality with other natural cellulosic fibers. This provides a good opportunity for agro waste enset fibers to gain value and bring some income to local farmers. SEM view analysis of enset fibers revealed porous regions on the enset fiber through its axis. As the porous regions are filled with air, the textile structures produced with enset fibers were tested for sound absorption properties. On the other hand, it was observed from cross section SEM views of an enset fiber, which is naturally exist as a composite structure like wood, bones and has around fiber diameter of 200 μm that enset fiber consisted of so many nanofibers surrounded by immature hemicellulose, lignin and waxes. Hence, an attempt was made to produce enset nanofibers for developing further enset fabric structures.

Enset nanofibers were produced by treating enset fabric with α-amylases enzyme of different concentrations (10, 15 and 20%) and then by applying mechanical hammering. Nanofibers were obtained at different diameters. Enset fiber were broken into smaller lengths because of mechanical hammering. Finest nanofibers were obtained with 20% α-amylases enzyme concentration at 63-85 nm (nanometer) interval. Although nanofibers were obtained with all α-amylases enzyme concentrations, the more and finest nanofibers

were attained with higher concentrations. More research is needed to produce finest nanofibers with longer lengths for use in more advanced applications like sound absorption.

As the aim was to develop a fully biodegradable enset fabric reinforced composite with acceptable mechanical properties for industrial applications, a new bio-resin was developed using the mixture of acacia tortilis and frankincense bio-resins produced from their gums. Composite performance of new bio-resin was investigated by changing mixture percent ratio of acacia to frankincense bio-resins between 75:25 percent to 50:50 %. Both breaking strength and elongation increased with increasing acacia bio-resin ratio. Further research was continued with 50:50% mixture ratio as the bio composite of this ratio had almost the same breaking elongation with that of neat fabric. Bio degradable composites were produced for mechanical tests with 70:30 % bio-resin to enset fabric ratio. Additionally, 80:20 % and 60:40 % ratio bio degradable composites were produced for impact tests. Enset woven fabric sample (with 8 warp threads in its effective width) had a tensile strength and percentage elongation of 528.83 N and 5.70 % respectively. The average tensile strength of single and double layered enset fabric reinforced composites were measured as 2356.50 N and 4671.53 N respectively without a significant variation in their percentage of elongation. This corresponds to more than 4 times of neat fabric's tensile strength and can be counted as a very significant increase. Similarly, the maximum flexural strength of woven fabric reinforced composite structure was found as 76.43 N/mm^2 and 151.19 N/mm^2 for 5 and 10 bar tests respectively. The impact properties of enset fabric reinforced composite structure was significantly affected by grammage (the weight of fabrics per unit areas). The impact test results indicated that green composite structure made from 20 % of enset woven fabric to 80 % of bio resin ratio had a better impact absorption performance than other compositions such as 30:70% and 40:60 %. Mechanical test results suggest that the bio degradable enset fabric reinforced composite can find applications in packaging, housing, automotive panels, product casing etc. where not high loads are involved. But some further research is recommended for improvement of bio-resin by using also some bio degradable additives.

The morphological analyses of textile fabric reinforced composite structures indicated that there were a lot of smooth grooves (porosity) in the green composite structures and

the cracks started and would be propagated to fabrics. Surface fracture and cracking propagation of textile fabric reinforced composite structures were analyzed by the optical microscope and SEM view after applying different type of loads or strength tests. The SEM and optical microscopic view confirmed that there was a propagation of crack through textile fabric reinforced material into bio-resin interface of the samples. This situation was confirmed and used as an evidence that the crack propagation of the composite structures was started and the deboning of textile fabric from the bio-resins (mixed acacia-frankincense) would have happened through the crack path. The bio-resins deboning at these grooves also revealed that the bio matrix was started to crack and the majority of yarns in the woven fabrics were broken in the warps direction. All the above phenomena have a significant effect on the overall mechanical performance of textile fabric reinforced composite materials.

The morphological study of enset fiber indicated that the porosity and void structure of the fibers could help enset fabrics to show a good sound absorption. Single and multilayer enset fabric and enset fabric reinforced composites with differing bio resin ratios (80 %, 70 % and 60 % w/v) were produced and their sound absorption performances were measured. The test results indicated that enset fabrics showed good sound absorption at high frequency range (over 4000 Hz) with 4 and 5 layer structures. Composite structures managed to draw sound absorption frequency to medium frequency range (2000-4000 Hz). Increasing bio-resin ratio to 80% shifted sound absorption frequency range to lower values but also narrowed absorption interval because of a decrease in the fibrous structure due to increasing resin amount. Structure tended to behave like a solid structure with increasing resin amount. No effective sound absorption was achieved at lower frequency range up to 1000 Hz with current enset fabric and enset fabric reinforced composite structures. It is thought that sound absorption capability of enset fabrics could be improved by developing special enset fabric constructions and by including enset nanofiber structures.

Finally, laboratory made biodegradability test of enset fiber, enset fabric and enset fabric reinforced green composite materials revealed that the laboratory prepared bio resin was completely degraded in one year period while enset fiber, enset fiber reinforced composite and enset fabric lost their weight by 73.27 %, 57.98 % and 38.41 % respectively. No

mechanical test was conducted on the samples as both enset fibers and enset fiber reinforced green composite completely lost their mechanical strength.

As a further research, an optimization work on bio-resin development is recommended by also including additional bio degradable materials to strengthen the resin for more demanding applications. Some research should also be done on nanofiber production with longer nanofiber length from enset fiber as this might open new application areas for enset fibers.

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APPENDIX

Appendix 1: Chemical constituents of commonly used textile fibers obtained from plant

Appendix 2: List of textile fibers commonly used as a reinforcing materials with their properties and origin

Appendix 1: Chemical constituents of commonly used textile fibers obtained from plant

Type of Fiber	**Cellulose (%)**	**Lignin (%)**	**Hemi-cellulose (%)**	**Pectin (%)**	**Wax (%)**	**Micro Fibril angle (^{0})**
Banana	62-82	6	10-15	-	10	10-12
Abaca	52-64	8-10	20-24	-	4	21-24
cotton	82-90	-	4	0.7	9-10	-
Bamboo	25-42	5-30	25	-	9	-
Flax	65-71	5-20	10-15	1.7-2.5	-	5-11
Jute	60-70	10-15	20-22	0.6	-	9
Hemp	65-75	3-6	10	0.9	1.3-6	2-6.3
Kenaf	44-56	20	9-14	0.7	0.7	2.2-6.3
Sisal	77	7	10	-	2.5	-
Ramie	70-90	0.5-0.7	6-14	2	-	70-82
Coir	35-42	0.26	40-44	3.5	-	31-50

Appendix 2: List of textile fibers commonly used as a reinforcing materials with their properties and origin[22]

Type of Fiber	Yearly production (10^6)	Obtained from	Density (g/cm^3)	Moisture content (%)	Tensile strength (MPa)	Elongation (%)
Banana	2.6	Bast(Leaf)	1.34	11-12	530-900	2.5-6
Abaca	0.071	Leaf	1.49	13.5	420-800	3-9
cotton	0.018	Seed	1.5	6.6-8.5	350-400	3.5
Bamboo	101	Grass	1.26	10	300	-
Flax	0.8	Stem	1.45	8	850-1400	1.5
Jute	0.26	Plant	1.5	11	400	1.7
Hemp	-	Stem	1.5	8	600	1.5-4
Kenaf	0.8	Stem	1.5	6.5	300-900	1.7
Sisal	0.3	Leaf	1.4	11	400	2.5
Ramie	0.1	Bast(stem)	1.4	13-16	450	2.1
Coir	Abundant	Fruit	1.26	11	200	15
Basalt	-	Mineral	2.7-2.8	-	2850	2.7
Aramid	-	-	1.44	-	3000	2.4
S-Glass	-	-	2.5	-	2000-3500	3.1
Carbon	-	-	1.4	-	4900	1.44-1.81
Silicon	-	-	3.16	-	350-430	-

[22] *M. J. Mochane, T. C. Mokhena, T. H. Mokhothu*4, *A. Mtibe, E. R. Sadiku, S. S. Ray, I. D. Ibrahim*6, *O. O. Daramola*, Recent progress on natural fiber hybrid composites for advanced applications: A review. eXPRESS Polymer Letters Vol.13, No.2 (2019) 159–198.