EXPERIMENTAL INVESTIGATION AND ANALYSIS OF MAIZE NATURAL FIBRE COMPOSITES FOR STRUCTURAL APPLICATIONS

Thesis

Submitted in partial fulfillment of the requirements for the degree of

DOCTOR OF PHILOSOPHY

by

SARAVANA BAVAN D

DEPARTMENT OF MECHANICAL ENGINEERING

NATIONAL INSTITUTE OF TECHNOLOGY KARNATAKA,
SURATHKAL, MANGALORE – 575025

MARCH, 2014

DECLARATION

by the Ph.D. Research Scholar

I hereby *declare* that the Research Thesis entitled "**Experimental Investigation and Analysis of Maize Natural Fibre Composites for Structural Applications"** which is being submitted to the **National Institute of Technology Karnataka, Surathkal** in partial fulfillment of the requirements for the award of the Degree of **Doctor of Philosophy** in Mechanical Engineering is a *bonafide report of the research work carried out by me*. The material contained in this Research Thesis has not been submitted to any University or Institution for the award of any degree.

SARAVANA BAVAN D
Register No. **081046ME08F02**
Department of Mechanical Engineering

Place: NITK Surathkal, Srinivasnagar

Date:

CERTIFICATE

This is to *certify* that the Research Thesis entitled "**Experimental Investigation and Analysis of Maize Natural Fibre Composites for Structural Applications**" submitted by **Saravana Bavan D** (Register Number: **081046ME08F02**) as the record of the research work carried out by him, is *accepted as the Research Thesis submission* in partial fulfillment of the requirements for the award of degree of **Doctor of Philosophy**.

Research Guide	Head
Dr. G. C. Mohan Kumar	**Dr. Prasad Krishna**
Professor	**Professor**
Department of Mechanical Engineering	Department of Mechanical Engineering
(Signature with Date and Seal)	(Signature with Date and Seal)

Dr. Vijay Desai
Professor
Chairman – DRPC
(Signature with Date and Seal)

With Blessings of God,

I Dedicate This Work to My Whole Family, Teachers and Friends

ACKNOWLEDGEMENT

I take this opportunity to express my deepest gratitude to my Research Supervisor **Prof. G. C. Mohan Kumar**, Department of Mechanical Engineering, NITK, Surathkal for providing this great opportunity and giving their valuable suggestions and support during my research work.

I am grateful to **Dr. Prasad Krishna,** Head, Department of Mechanical Engineering, for providing facilities for my Ph.D. Research work in the Department of Mechanical Engineering, NITK, Surathkal.

I would like to extend my thanks to the RPAC Members **Prof. Prasad Krishna**, Department of Mechanical Engineering and **Prof. Udaya Kumar R. Y** (Dean-Students Welfare) Department of Electrical and Electronics Engg, NITK for their apt suggestions and valuable discussions during the research progress phase.

I am extremely obliged to **Prof. Sabu Thomas,** Mahatma Gandhi University, India and **Prof. Amar K Mohanty,** University of Guelph, Canada, for sharing their experience in this research field. Their appreciation at every level of the progress of the research has been a motivating factor. I also extend my deepest gratitude to **Dr. Gopalan**, Executive Director, Society for Development of Composites- Bangalore, for the initial support that helped me in taking up this work.

I am grateful to **Dr. Nagaraj Upadhaya, Dr. Kishore Ginjupalle,** Manipal Institute of Dental Sciences, Manipal, **Dr. Keshav Prabhu**, Konspec – Konkan Speciality Polyproducts Private Limited, Mangalore and **Mr. M. G. Ananda Kumar**, Material Technological Division, Central Power Research Institute (CPRI), Bangalore and for providing the necessary support in terms of facilities and equipments for executing this research work.

I am grateful to Ministry of Human Resource Development (**MHRD**), Government of India, for Ph.D. Research fellowship. Science and Engineering Research Board

(**SERB**), Department of Science and Technology (**DST**), Government of India, New Delhi, Council of Scientific and Industrial Research (**CSIR**), New Delhi, Centre for International Co-operation in Science (**CICS**), Chennai and **NITK Alumni Association**, Surathkal, for their kind support for availing travelling grant fellowship for attending and presenting papers at International Conferences at abroad.

I extend my sincere thanks to University of Agricultural Sciences, Bangalore, Composites Technological Park, Bangalore, Department of Chemical Engineering, Department of Materials and Metallurgy Engineering, Department of Chemistry, and other Department in NITK, Surathkal. Earnest thanks to all Faculty, Staff members and Research Scholars of Mechanical Engineering Department for the healthy support during research work.

I thank my God Almighty for the eternal care and healthy mind, Amma, Dad, Sisters and Brother in law of my family for their constant moral and effective support in achieving this work successfully.

Last, but not the least that deserves the most appreciation to my shrewd and dearest friends Mr. Kamal Babu, Mr. Syed Fahemullah, Mr. Praveen Naik, and other friends who constantly supported me during the experimental work setup and in software development and in conducting the trials.

- **SARAVANA BAVAN D**

ABSTRACT

Natural fibre composites are the recent upcoming materials used in structural and other general engineering applications. They are more popular because of their abundance, native state of recycle, eco friendliness and sustainable material features. Natural fibres as reinforcement in the composite materials have attained new interests to the global sustainable world. As the fossil fuels are depleting, it is the time to focus on alternative material that can compensate with the present synthetic world of composites. Major applications of these natural fibres are in the structural and automotive sectors.

The research work is focused on study of maize fibres and their composites with matrix of thermoset polymer for structural applications processed through Vacuum Assisted Resin Transfer Molding (VARTM) and with hand lay-up methods. Maize composites, Hybrid Composites (maize and jute fabric of single and double layer), and Bio-composites are processed. Maize composites and Bio-composites were processed by VARTM technique. Sandwiched hybrid composites were fabricated by hand layup method. Physical, Chemical, Thermal, Mechanical and Surface characterization tests were carried out for the performance and analysis of maize fibre composites. Natural fibres of pulverized maize stalk and matrix with different weight fractions are considered for the present work. As natural fibres are hydrophilic, chemical treatment of the fibres are carried out in order to make hydrophobic. Work is also concentrated in processing maize stalk particles as fibre and expoxidized soy bean oil as matrix to obtain a bio-composite material.

Finite element analysis is also carried out for thermal studies of composites with maize long fibres with few assumptions. The result shows that the fibre and matrix interactions are better and improved with the chemical treatment. Experimental and Finite element results are compared and the results are presented and discussed.

Key Words: Maize Natural Fibres, Bio-composites, Maize-Jute hybrid composites, Polymers.

TABLE OF CONTENTS

Page No.

Acknowledgement

Abstract

CHAPTER 1

INTRODUCTION

CHAPTER 2

LITERATURE SURVEY ON NATURAL FIBRES AND THEIR COMPOSITES

CHAPTER 3

EXPERIMENTAL WORK IN PROCESSING OF COMPOSITES

CHAPTER 4

PROPERTIES OF MAIZE FIBRE AND THEIR COMPOSITES

CHAPTER 5

ANALYSIS OF MAIZE LONG FIBRE COMPOSITES

CHAPTER 6

CONCLUSIONS

APPENDICES

LIST OF FIGURES

Page No.

LIST OF TABLES

NOMENCLATURE

PMC ----Polymer Matrix Composite

GP----General Purpose

SEM ----Scanning Electron Microscope

TGA---- Thermal Gravimetric Analysis

DSC---- Differential Scanning Calorimeter

XRD ----X-Ray Diffraction

FEA---- Finite Element Analysis

VARTM ----Vacuum Assisted Resin Transfer Molding

FTIR ----Fourier Transform Infrared Spectroscopy

SCRIMP---- Seemann Composites Resin Infusion Molding Process

LCM----Liquid Composite Moulding

NF ----Natural Fibres

NaOH----Sodium hydroxide

DGEBA---- Diglycidyl Ether of Bisphenol-A

TETA---- Triethylene tetramine

ESBO/ESO---- Epoxidized soybean oil

EDAX/EDX---- Energy Dispersive Spectrometry

SS----Hybrid Composites (Single Side)

DS----Sandwiched Hybrid Composite (Double Side)

u---- Darcy's velocity

K---- Permeability tensor

μ---- Viscosity

∇P ----Pressure gradient

P_c---- Total compaction pressure

P_r---- Resin pressure

P_n---- Net pressure

v_v ----Volume fraction

ρ_{tc} ----Theoretical density of composite

ρ_{ac} ----- Actual density of composite

CHAPTER 1

INTRODUCTION

1.1 Over View of Natural Fibre Composites

Natural fibre are becoming popular in recent days especially in the composites sector because they have lot of advantages compared to synthetic fibres in terms of cost, density, biodegradability and other favourable properties including processing. The research activities for the last decade has shown a considerable effort towards the application of natural fibres derived from plant and agro wastes which exist in abundance as a reinforcing material for the production of building, structural components and also for automobile parts. This increased demand for natural fibre composites is because; they are environmental friendly and have potential to replace the traditional fibres (i.e. carbon, glass, and aramid fibre) in many applications. The reasons are renewable, easily available and can be produced at low cost in many parts of the developing world. They are strong, stiff and have the ability to produce composites with similar specific properties to those of E-glass due to their low densities [Wallenberger 2003].

Composite materials formed by natural fibres and polymeric matrices constitute current area of interest in materials research. In natural fibre reinforced composites, fibre acts as reinforcement and exhibits high tensile strength and stiffness. Plant-based or vegetable natural fibres are ligno cellulosic, consisting of cellulose micro fibrils in an amorphous matrix of lignin and hemicelluloses [John and Thomas 2008]. The fibre structure is hollow, laminated with molecular layers and integrated matrix. The mechanical properties of reinforcement have a direct role with tensile strength and stiffness of the composites. The selection of suitable reinforcing fibres follows certain criteria such as thermal stability, fibre-matrix adhesion, long time behaviour, elongation at failure and also processing cost. Owing to their low specific gravity, which are 1.25-1.50 gm/cm^3 as compared to synthetic fibres, especially glass fibres, which are 2.6 gm /cm^3. Ligno cellulose fibres were able to provide a high strength to weight ratio in plastic materials.

Bio-composites or natural fibre composites are composed of biodegradable natural fibres as reinforcement and biodegradable or non biodegradable polymers as matrix [Mohanty et al. 2001]. They have one or more of their phases derived from biological origins, e.g., plant fibres from crops, from recycled wood, waste paper etc. The matrix phase within a bio composite may often take the form of a natural polymer, possibly derived from vegetable oils or starches. Biopolymers are natural polymers obtained from microbial systems, extracted from organisms and also produced from the seasonal crops and plants like corn, wheat, potato and sugarcane. They can be chemically synthesized from the basic biological materials like amino acids, sugars, oils etc.

Natural fibre reinforced polymer composite materials are considered as an important class of engineering materials because they have wide applications in building construction and have shown good mechanical properties and ease of fabrication. Thermosetting polymers are used today in plant fibre composite especially for inner door panels in automotive industry and other applications as a structural building material. Plant fibres in structures are being successfully carried out by many researchers [Herrmann et al. 1998, Dweib et al. 2004, Burgueno et al. 2005, Christian and Billington 2009] and the outcome result has predicted that these plant fibres based composite material can be a good substitute engineering material.

Natural fibre composites find applications in all fields ranging from constructions, to automotive, electronics, packaging etc. Among them, construction and automotive fields grab a large portion of use [Marsh 2003] and similar work is briefed out **[**John et al. 2008] and the view is shown in Figure 1.1. Natural fibre composites in structural and building construction introduces many advantages such as high strength, stiffness to weight, survivability in severe weather conditions, desired ductility, fatigue resistance, and design flexibility [Golbabaie 2006].

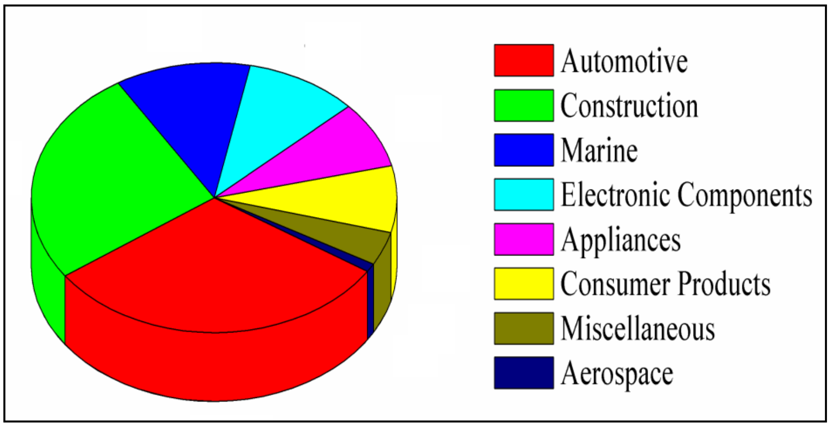


Figure 1.1: Applications of Biocomposite in Various Sectors [John et al. 2008]

A structural composite is an element as one that is required to carry load in use. e.g. load bearing walls, roof systems, subflooring, stairs, framing components, etc. Structural composites can range widely in performance from high performance materials industry to wood-based composites which have lower performance requirements. Dweib et al. 2006 performed work on bio-based composite materials which was tested for suitability in roof structure. Based on the beam results, large scale composite structural panels were developed by the researcher and a soy oil-based resin and cellulose fibre in the form of paper sheets was successfully used for the composite structures. Burgueno et al. 2005 demonstrated that biologically inspired hierarchical designs can help and improve the moderate properties of bio-composites and allow them to compete with conventional materials for load-bearing applications.

1.2 General Composites

A composite material consists of two or more physically and/or chemically distinct, suitably arranged or distributed phases, with an interface separating them and it has the characteristic that are not depicted by any of the components in isolation. Composite materials have bulk phases which are continuous called matrix and one dispersed, non-continuous phase called the reinforcement, which is usually harder and stronger. The essence of the concept of composites is that: the bulk phase accepts the load over a large surface area, and transfers it to the reinforcement, which being stiffer and increases the strength of the composite [Chawla 1987, Hull and Clyne 1996, Callister 1999]. The reinforcing elements are able to sustain high tensile loads whilst the matrix imparts rigidity to the composite. The stresses applied to the composite results in transfer of loads from one fibre to another, via the matrix and the stiffness of the matrix is often accompanied by brittleness.

Fibre reinforced composite materials are special useful composites because they contain a reinforcing phase in which high tensile strengths are realized when they were processed to fine filaments, wires or fibres. In such fibre composites, the matrix glues the fibres together and also transfers stress to them. In many cases, the matrix also protects the fibre from deleterious interactions with the environment, such as oxidation or corrosion. Many fibre reinforced materials consist of a high-strength material imbedded in a much less strong matrix [Chawla 1987]. Fibre reinforced composite materials offers a combination of strength and modulus that was either comparable or better than that of traditional metallic materials. Because of their low specific gravities, strength-to-weight ratios and modulus-to-weight ratios, the composite materials were markedly superior to those metallic materials. In addition, fatigue strength-to-weight ratios, as well as fatigue damage tolerances, of composite laminates are excellent.

1.2.1Classification of Composites

Composite materials are classified as: (a) On the basis of matrix material
(b) On the basis of filler material

a) On the basis of Matrix material it is classified as

- Metal Matrix Composites
- Ceramic Matrix Composites
- Polymer Matrix Composites

b) On the basis of Filler Material

1. Particulate Composites
    - Composites with random orientation of particles.
    - Composites with preferred orientation of particles.

2. Fibrous Composites
    i) Short-fibre reinforced composites.
        - Composites with random orientation of fibres.
        - Composites with preferred orientation of fibres.
    ii) Long-fibre reinforced composites.
        - Unidirectional orientation of fibres.
        - Bidirectional orientation of fibres
        - Laminate Composites

Depending on the type of matrix and reinforcement, manufacturing method processes are used according to the applications. For short or discontinuous fibre reinforcements, compression moulding, extrusion and injection moulding are mainly used as manufacturing processes. In all these processes, either a thermoset or a thermoplastic matrix can be used.

Polymer Matrix Composite (PMC) is one such advanced material consisting of a polymer matrix reinforced by a fibrous material. The polymer matrix binds the reinforcement together and provides surface finish and durability to the composite. The reinforcement is the main load-bearing constituent and provides strength and stiffness to the composite [Gerdeen and Rorrer 2011]. During process, one can easily alter the arrangement of reinforcement to orient the fibres in a required direction and thus tailor the properties of a composite to suit its performance requirements. This is a major advantage from a design perspective and allows one to replace homogeneous metals, alloys and other conventional materials in many applications. The main constituents of PMC materials are the polymeric matrix or resin and fibrous reinforcements.

Polymer matrix composites are widely used in many major engineering structural applications. The matrix serves mainly for two important purposes namely, it bonds the fibrous phase and under an applied force it deforms and distributes the stress to the high-modulus fibrous constituent [Gerdeen and Rorrer 2011]. The ultimate properties of the composites depend on certain distinct properties of the constituents, sizes, shape of the individual reinforcing fibres or particles, structural arrangement and distribution. Composite materials used for structural purposes often have low densities, resulting in high stiffness to weight and high strength to weight ratios when compared to traditional engineering materials [Dweib et al. 2006].

1.2.2 Fibre Materials

The reinforcement materials for composites are fibres, particulates or flakes. Fibres are the load carrying members where as the matrix acts as a load transfer medium and protecting the fibres from the environment.

Generally, fibres are two types namely

a) Synthetic Fibres
b) Natural Fibres

Synthetic fibres are divided into different types based upon their applications and processing techniques namely, carbon fibres, boron fibres, glass fibres, silicon carbide fibres, alumina fibres and aramid (Kevlar) fibres. Commonly used fibres in composites include glass, carbon, and aramid etc.

a) Synthetic Fibres

Glass fibre is the oldest and most familiar fibre. They are also termed as fibre glass and are extremely strong and light weight material. The strength properties are lower than carbon fibre and they are less stiff. The additional property of good strength and weight property make them popular. Glass is the oldest and most familiar performance fibre. The classification of fibres includes A-glass, C-glass, E-glass, and AE-glass. Glass fibres are commonly used for engineering composites and their uses include manufacturing of automotive parts, pipes, structural members etc. Glass fibres are available economically in abundance with good mechanical properties and thus widely used in composite structures. They are available in different forms like continuous, chopped and woven fabrics. Glass fibres behave nearly isotropic resulting in nearly same conductivity properties in any direction of the fibre and the microstructure of fibre plays vital role in carrying heat.

Carbon Fibres are the most used fibres in composites sector for advanced applications and they are the fibres containing 90% carbon prepared with controlled pyrolysis. They are manufactured by thermal treatment of organic precursor’s polyacrylonitrile, pitch or rayon in fibrous form and the most common is polyacrylonitrile fibres (PAN based carbon fibres). Pitch is a complex mixture of aromatic hydro carbons and be made from petroleum, coal, asphalt. Carbon fibres are brittle with higher modulus of fibres, high strength, low density and resistance to chemical environments.

Aramid Fibres have high strength to weight ratio and are produced under the trade name of Kevlar. The chemical composition of Kevlar aramid fibre is poly para-phenyleneterephthalamide also known as PPD-T. The available forms are continous filament, rovings, yarns and woven fabrics and they are heat resistant and strong fibres.

b) Natural Fibres

Natural fibres are class fibres either from plant fibres, animal fibres and mineral fibres. Plant or ligno cellulosic fibres are the most popular fibres in recent times because of ease availability and processing techniques. Plant fibres are mainly composed of different chemical constituents such as cellulose, hemicelluloses, lignin, pectin and wax. Detailed discussions are presented in next chapters on natural fibres and their composites.

1.2.3 Matrix Materials

Matrix materials are available in different types which include metal matrix, ceramic matrix and polymer matrix. Polymer matrices are the most commonly used because of cost efficiency, ease of fabricating complex parts with less tooling cost and have excellent room temperature properties when compared to metal and ceramic matrices. Polymer matrices can be either thermoplastic or thermoset. Matrix protects the reinforcement from environmental factors, serves to transfer load from one fibre to other and they hold the reinforcements and provides a medium for binding.

1.2.3.1 Synthetic Polymers

Polymers are a class of giant molecules consisting of discrete building blocks linked together to form long chains. Simple building blocks are called monomers, while more complicated building blocks are referred to as repeat units [Stevens 1999, Carraher Jr. 2012]. The polymer matrix contains polymeric chains and each chain is made up of 103 to 106 monomer units assembled through chemical reactions. Depending on the type of bond formed between these chains, the polymer matrix can be categorized as either a thermoplastic or a thermoset.

Polymers are widely classified as

- Linear polymers (molecular chain form)
- Thermoplastic polymers (linear or branched polymers and molecules are not interconnected)
- Thermoset polymers (cross-linked polymers and have strong rigid network structure)
- Elastomers (thermoset/thermoplastic having elastic deformation)

a) Thermoplastic Polymers

In thermoplastic polymers, individual molecules are linear in structure with no chemical link between them and they are held by weak secondary bond such as Vander walls bond [Stevens 1999]. They can change the shape and remould because the weak forces reform in new shape when cold. They have better toughness, impact resistance and can be recycled. Few thermoplastics do not crystallize and they are termed as amorphous plastics. Thermoplastic materials are formed by addition polymerization and can be soften or fuse when heated, harden and become rigid after cooling. Unlike thermoset, thermoplastics can be modified or reused upon the need.

Thermoplastics have longer shelf life and higher fracture toughness than thermoset resins. Few thermoplastics are shown in Table 1.1.

Table 1.1: Few Thermoplastics

Sl. No.	Polymer Name
1	Polytetrafluroethylene (PTFE)
2	Polyethylene terephthalate (PET)
2	Polymethyl methacrylate (PMMA)
3	Polyvinyl Chloride (PVC)
4	PolyPropylene (PP)
5	PolyStyrene (PS)
6	Polyethylene(PE)
7	Nylon 66

b) Thermoset Polymers

Thermoset resin is a plastic material which are initially a liquid monomer or oligomers or a pre-polymer, which are cured by either application of heat or catalyst to become an infusible and insoluble material. Thermoset polymers have covalent bonds linking the polymer chains in three dimensions and these links prevent the chains from sliding past one another resulting in a higher modulus and improved creep resistance [Carraher Jr. 2012]. Usually thermosets are more brittle than thermoplastics and the polymer chains in thermosets are below their glass transition temperature and at room temperature making them glassy. Thermoset polyesters are produced by the condensation polymerization of dicarboxylic acids and difunctional alcohols (glycols). Thermoset matrices are formed due to an irreversible chemical transformation of a resin into an amorphous cross-linked polymer matrix. Due to huge molecular structures, thermoset resins provide good electrical and thermal insulation. Thermoset have low viscosity, which allow proper fibre wet out, high rigidity, high dimensional stability, excellent thermal stability and better creep resistance. Mostly thermoset resins can be formulated to give a wide range of properties based upon requirement.

Thermoset polymers also called as network polymers because they are stronger than thermoplastic and with strong network bond, best suited for high temperature applications. They develop three dimensional structures upon curing and usually hardener is added to resin for developing a strong structure. On reheating the cross links prevent true melting and the polymer cannot be hot worked. Different types of thermoset polymers are phenolics, epoxies amines, polyesters, urethanes, silicone. Among the most common thermosetting resins used in composite manufacturing are unsaturated polyesters, epoxies, vinyl esters, amino resin and phenolics.

i) Unsaturated Polyester Resin

Unsaturated polyester resins are cheap and best material used in composite industries and represent approximately 75% of the total resins used. Unsaturated polyesters are divided into classes depending upon the structures of their basic building blocks. Some common examples are orthophthalic (ortho), isophthalic (iso), dicyclopentadiene (DCPD) and bisphenol A fumarate resins. In addition, polyester resins are classified according to end use application as either General Purpose (GP) or Speciality polyesters. Unsaturated polyesters contain an unsaturated material, such as maleic anhydride or fumaric acid, as part of the dicarboxylic acid component. The finished polymer is dissolved in a reactive monomer styrene to give a low viscosity liquid and when this resin is cured, the monomer reacts with the unsaturated sites on the polymer converting it to a solid thermoset structure.

In polyesters, the most important additive is the catalyst or initiator. Typical catalyst are organic peroxide such as methyl ethyl ketone peroxide is used for room temperature. When triggered by heat, or used in conjunction with a promoter (such as cobalt napthenate), peroxides convert to a reactive state (exhibiting free radicals), causing the unsaturated resin to react (cross-link) and become solid. Some additives such as tertiary - butyl catechol are used to slow the rate of reaction and are called inhibitors. Polyester has the advantages of low cost, ease of handling, good chemical resistance with reasonable mechanical properties.

ii) Epoxy Resins

Epoxy resin has excellent adhesion property compared to other resins. In addition to that it has low shrinkage upon curing, good chemical resistance, good adhesion with reinforcement, better moisture resistance and excellent mechanical properties. Epoxies are been used for advanced composites due to their adhesion to wide variety of fibres with superior mechanical, electrical properties and good performance at elevated temperatures. Epoxies are expensive compared to polyester and less resistant to moisture. Polyester and epoxy makes approximately 85% of the fibre reinforced polymer composites.

1.2.3.2 Biopolymers

Biopolymers are also termed as natural polymers because they form under natural conditions during the growth cycles of organisms [Salmoral et al. 2000, Mitrus et al. 2009]. Polymers of natural origin (e.g., starch and cellulose) are modified either by physical or chemical methods in order to make them suitable for processing [Riedel 1999]. Availability of thermoplastic biopolymers includes poly lactic acid, cellulosic plastic, starch plastic, soybean and corn based polymer resins. Poly lactic acid is the most common biopolymer in use and they are derived from corn starch via commercial fermentation and polymerization technologies [Velde and Kiekens 2002, Mohee et al. 2008, Sarasa et al. 2009]. Advantages of biopolymers include biodegradability, compostability, economically sustainable and easily processed.

Bio polymeric materials can be sorted out into four classes as shown below [Velde and Kiekens 2002, Yu et al. 2006, Averous and Pollet 2012] and in Figure 1.2.

- Polymeric materials extracted directly from biomass such as starch, cellulose, proteins, chitin and lipids
- Polymers produced by micro-organisms

- Polymeric material produced by chemical synthesis using renewable bio based monomers such as poly lactic acid
- Polymers from petrochemical products

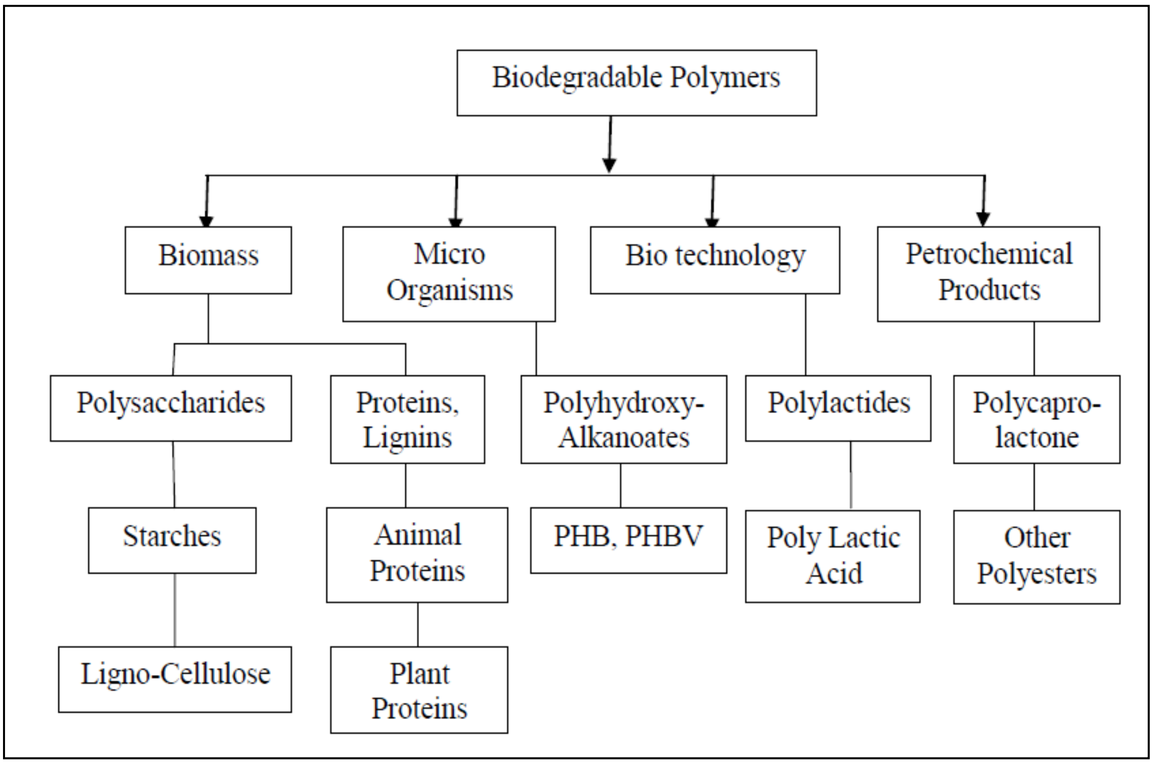


Figure 1.2: Classification of Bio-Polymers

Biopolymers do not produce any harmful gases to the atmosphere and hence environmental friendly [Huang et al. 1990; Wool and Sun 2005]. Bio polymers are obtained from microbial systems, extracted from organisms and also from seasonal crops and plants such as corn, wheat, potato and sugarcane as shown in Table 1.2. They are chemically synthesized from basic biological materials like amino acids, sugars, oils etc. These polymers have wide applications for production of food, clothing fabrics, cosmetics, medicinal materials and industrial plastics etc as shown in Table 1.3 [Yu 2006, 2008, Leja and Lewandowicz 2010, Rapa et al. 2011].

Table 1.2: Natural Sources of Bio-Polymers

Biopolymer	Natural source	Description
Starch	Corn, Potatoes, Wheat	Polymer made up of glucose, carbohydrates are stored in plant tissue. It is not found in animal tissue
Cellulose	Wood, Corn, Cotton, Wheat	Main component of plant cell walls and is made up of glucose
Soy Protein	Soy beans	Naturally occurs in the soy plant
Polyesters	Bacteria	Created through naturally occurring chemical reactions that are carried out by bacteria's

Table 1.3: Few Biopolymers with Source and Applications

Biopolymer	Source	Applications
Starch	Plants - corn, potatoes, rice, barley, wheat	Adhesives, stabilizers, thickeners, soil conditioners, packaging.
Plant Cellulose	Plant - cell wall	Thickening solutions, gels, food, textiles, ceramics, paper coatings, liquid fuels
Lignin	Plants- herbaceous and woody	Non-energy, industrial packaging, tape, binding and thinning agents, chemicals.
Chitin	Plants, insects, shells	Medicine, manufacturing, agriculture, contact-lenses, cosmetics, shopping-bags.
Protein	Plants (soy, zein from corn), animal (collagen)	Films, gels, wetting agent, fibres.

1.3 Processing of Composites

For a thermoplastic matrix and long fibre reinforcements, composite sheet forming and pultrusion are the main processes. Hand lay-up, autoclave moulding, Liquid Composite Moulding (LCM), and filament winding are the main processes for long fibre reinforcements using a thermoset matrix. In addition, based on the moulding arrangement, the last category of processes can also be categorized as either open mould (e.g. Hand lay-up) or closed mould (e.g. LCM) processes. LCM is a general moulding, where a dry reinforcement is placed inside a rigid, semi-rigid or flexible mould. Uncured thermoset resin, in liquid form is injected from a source and infiltrates the reinforcement. After complete infusion of the reinforcement, the resin is allowed to cure. Once the resin is cured, a finished part is extracted [Rudd et al. 1997]. Resin Transfer Moulding, Structural Reaction Injection Moulding, Injection Compression Moulding, Vacuum Infusion etc. are some of the main liquid composite moulding processes [Potter 1997].

Various techniques are available for processing of composites among those techniques; Vacuum bagging techniques is used for fabricating small as well as large components but mainly concerned for complex shapes. The use of Vacuum Assisted Transfer Molding (VARTM) process in manufacturing of high performance composite materials has been greatly recognized as one of the most successful process.

1.3.1 Vacuum Assisted Resin Transfer Moulding (VARTM)

Vacuum Assisted Resin Transfer Moulding (VARTM) is a liquid composite moulding process where both resin injection and fibre compaction are achieved under pressures of 101.3 kPa or less. The use of VARTM process in the manufacturing of high performance composite materials has been greatly recognized as one of the most important processes. The VARTM process is a variant of vacuum-infusion resin transfer moulding. These processes are modified versions and also termed as Vacuum Infusion (VI) or Seemann Composites Resin Infusion Molding Process (SCRIMP)

[Parnas 2000]. The VARTM process though a relatively low cost process can be used to fabricate composite parts of very high quality [Bayldon and Daniel 2009].

The VARTM process involves the following procedures:
(1) Mould preparation
(2) Sealing the mould and creating a vacuum
(3) Mixing of resin, hardener and degassing
(4) Resin impregnation
(5) Curing of fabricated material.

In this process a dry preform is placed on the mould and vacuum is bagged in conjunction with the resin injection and vacuum lines. A low viscosity resin is drawn into the preform with the aid of vacuum. The VARTM process has been used in many applications because of its time-saving and cost-effective characteristics [Yoon et al. 2005, Rachmadini et al. 2010]

1.3.2 Resin Flow in VARTM

The VARTM incorporates resin flow through the preform, compaction and relaxation of the preform, and viscosity with cure kinetics of the resin. Darcy's law combined with the continuity equation for an incompressible Newtonian fluid forms the basis of the flow [Bayldon and Daniel 2009]. Complete filling of the mould with adequate wetting of the fibrous media is the primary objective in resin transfer moulding and VARTM. The potential advantages of VARTM are relatively low cost with sufficient high volume fractions of reinforcement and the process can be readily applied to large-scale structures. VARTM is the most commonly used due to its low tooling cost and ease of use. The flow of resin through porous media such as fibre preforms and resin distribution media is governed by Darcy's Law:

$$\mathrm{u} = \frac{-\mathrm{K}}{\mu} . \nabla \mathrm{P} \tag{1.1}$$

Where,

u is the Darcy’s velocity (defined as the total flow rate per total flow front area),

K is the permeability tensor, m^2 (which characterizes the ease of flow through the fibre perform),

μ is the viscosity of the resin (Pa.sec),

∇P is the pressure gradient or the pressure drop per unit length (Pa/m).

The VARTM process consists of two important mechanisms: flow of the resin through the preform, and compaction and relaxation of the preform under the vacuum pressure. In addition, the resin cure kinetics and viscosity are ensured for complete resin infiltration of the preform before resin gelation [Bayldon and Daniel 2009].

During resin infiltration of the preform, the total compaction pressure is shared by the resin pressure and the pressure supported by the fibre network

$$P_c = P_r + P_n \tag{1.2}$$

Where, P_c is the total compaction pressure, P_r is the resin pressure, and P_n is the net pressure applied on the preform.

1.3.3 Advantages of VARTM

The advantage of VARTM technique is that, it is possible to manufacture very large products with good mechanical properties and creating a void-free laminate with high fibre volume fraction. VARTM has the potential of lower production costs through low-cost tooling approaches, low resin and reinforcement costs and reduced processing time. The process is labour intensive but can save great investment in mould tooling and thus it is suitable for low-mid volume production and for large composite parts [Rachmadini et al. 2010].

The benefits of VARTM when compared to non-vacuum bag curing of composite laminates include [Campbell 2004, Beckwith 2007].

- Better fibre to resin ratio
- Stronger laminate
- Low void content
- Reduces operator exposure to harmful emissions
- Reduced resin usage due to pre-compacted fabric
- Faster ply lay-up

1.4 Research Objectives

The goal of the work is to develop maize fibre reinforced composites for use in structures with distinct technique of VARTM processing and characterization of composite material developed. .

The research objectives of the present work are:

1. Examining the physical, mechanical and thermal properties of the maize fibre and their composites.
2. Explore an advanced method Vacuum Assisted Resin Transfer Molding (VARTM) for processing of maize fibre composites, hybrid composites (maize and jute fabric of single layer) with thermoset polymer and bio-composite material consisting of Maize fibre and epoxidized soy bean polymeric resin. Sandwiching hybrid composite (maize and jute fabric) by traditional hand lay-up method.
3. To study the effect of fibre surface modifications of maize fibres and maize fibre reinforced composites and to investigate, analyze and evaluate the performance of maize fibre reinforced composites through morphological, mechanical and thermal studies.
4. Finite Element analysis for predicting the thermal behaviour of maize fibre (long fibre) reinforced composite material and validating with experimental results.

The Methodology for the present work is shown in Figure 1.3

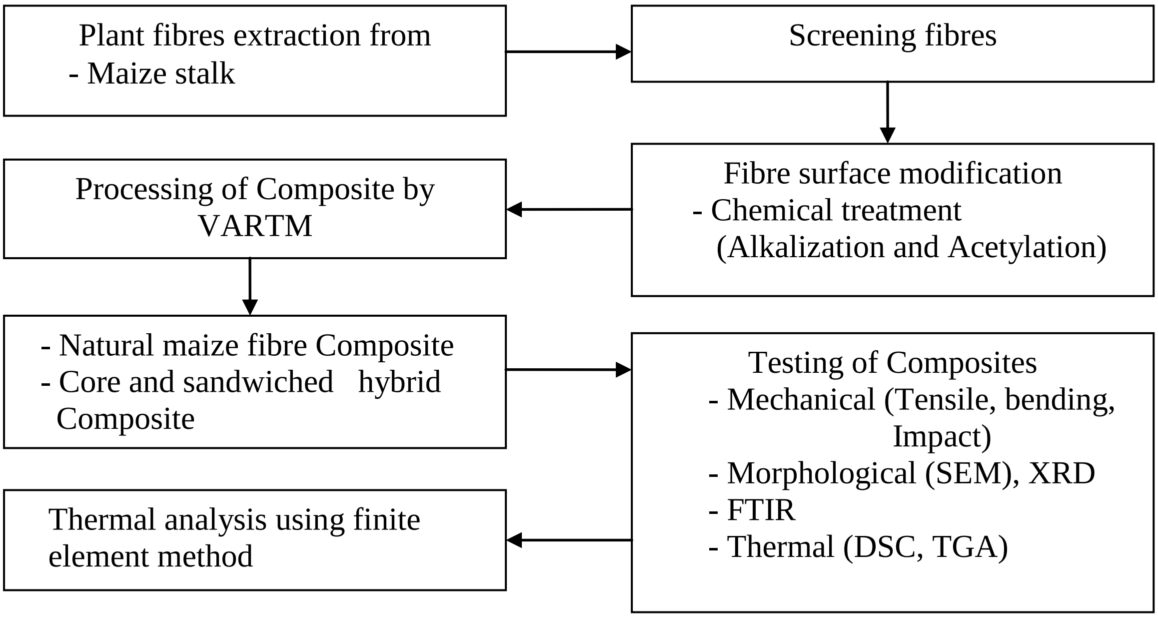


Figure 1.3: Methodology in the Present Work

1.5 Scope and Limitations

Synthetic fibres such as carbon fibre, glass fibre causes lot of harm to the atmospheric world generating lot of oxides and other materials. As petroleum sources are depleting away, there is a need for alternative material in order to substitute the present fibres for composites. Bio polymers can be used as a substitute matrix material for composites in order to develop a fully biodegradable composite material. Processing of composites by VARTM produces material by minimum resin content and superior finishing compared to other processing methods.

Plant Maize fibres are available in plenty worldwide and use of these fibres in composites are very appropriate in the present era of global warming. Maize fibres are local, available in low cost fibres and biodegradable. Therefore, an experimental investigation and analysis of maize natural fibre composites for structural applications are carried out. Fibres extracted from maize plant are processed for composites using VARTM. Effect of chemical treatment of fibres on mechanical properties was studied. Mechnical properties of composites with different weight fraction for epoxy and epoxidized soybean oil matrices are established. The thermal analysis of composites by Finite element method is verified experimentally to validate the results.

1.6 Structure of Thesis

Chapter 1 Introduction focuses on over view on natural fibres and their composites, research objectives, scope and limitation of the carried work. This Chapter outlines composites constituents (matrix and fibres), polymers and processing of composites by Vacuum Assisted Resin Transfer Molding (VARTM).

Chapter 2 Literature survey on natural fibres and their composites, reviews the research articles on natural fibres, extraction of natural fibres, properties of natural fibres and composites. Biodegradation and ecological impact on natural fibre composites are outlined. Application and role of natural fibre composites in present scenario are briefed out.

Chapter 3 Processing of composites, gives detail view of experimental work carried out for processing of maize fibre reinforced polymer composites. VARTM and traditional hand lay-up method are described for processing of composite material. Chemical treatment of natural fibres and fabrication of hybrid composite, sandwiched hybrid composite and bio-composites are discussed.

Chapter 4 Characterization of fibre and composites is focussed on characterization of maize fibre and their composite material i.e., Physical, Mechanical and Thermal tests are carried out for maize fibre and their composite materials. Scanning Electron Microscope (SEM), Thermal Gravimetric Analysis (TGA), Differential Scanning Calorimeter (DSC), X-Ray Diffraction (XRD) and Fourier Transform Infrared Spectroscopy (FTIR) are used for studying the properties of maize fibres and composites. Work on thermal conductivity of natural fibre composite is briefed out along with results and discussions.

Chapter 5 Long maize fibre composite gives experimental and finite element analysis of maize fibre (long fibre) composites. Brief information on the finite element method adapted to model the composite for determining thermal conductivity and is presented with results and discussions.

Chapter 6 Conclusions presents the brief summary of the present work along with scope for the future work.

CHAPTER 2

LITERATURE SURVEY ON NATURAL FIBRES AND THEIR COMPOSITES

2.1 Natural Fibres

Fibres are hair-like materials that are continuous filaments or discrete elongated pieces. Plant fibres themselves can be thought of as composite materials with the stiff and strong cellulose micro fibrils embedded in a hemicelluloses and lignin matrix [Fowler et al. 2006]. The industrial use of plant fibres as reinforcement in composites is supported by a huge number of publications on the many distinctive aspects of plant fibre composites [Eichhorn et al. 2001, Bledzki et al. 2002]. Thus, it is well documented that plant fibres are suitable alternatives for synthetic fibres in composite materials. From an ecological point of view, the use of plant fibres is favourable because of their CO_2 neutrality, biodegradability and cleaner processing conditions. Natural fibres are increasingly used in automotive and packaging materials. The cost factor is more favourable and almost all the leading car manufacturers have switched to the use of plant fibres for several parts in the automotive sector [Marsh 2003].

Asian people had been using natural fibres for many years; for instance, jute available in India has been used as reinforcement in many materials. Increased use of natural fibre composites as raw materials can be found in developing countries like India. Ninety percent of the world’s jute is supplied from India and Bangladesh and 75% of kenaf production from India and China [Ray et al. 2001]. India is the world’s second largest producer and consumer of fibres, textiles, and manufactured products, next to China [India News Letter, 2009]. The country has diverse agro-climatic conditions and consumer preferences, and hence it produces a wide variety of agricultural fibres as shown in Table 2.1.

Table 2.1: Natural Fibre Sources and their Origin [Taj et al. 2007, Ashori 2008]

Fibre Source	Species	Origin	Countries of origin
Abaca	Musa textiles	Leaf	Malaysia, Uganda, Philippines
Bamboo	>5000 Species	Stem	China, South East Asia, India
Coir	Cocos nucifera	Fruit	India, Sri Lanka, Philippines, Malaysia
Cotton	Gossypium sp	Seed	West and central Africa, China, India, Mexico, U.S
Flax	Linum usitatissimum	Stem	Borneo, Egypt
Hemp	Cannabis sativa	Stem	Yugoslavia, China
Jute	Corchorus capsularis	Stem	India, Egypt, Guyana, Jamaica, Ghana, Tanzania
Kenaf	Hibiscus cannabinus	Stem	Iraq, Tanzania, Jamaica, South Africa, Cuba, South Asia
Ramie	Boehmeria nivea	Stem	Honduras, Mauritius
Sisal	Agave sisilana	Leaf	East Africa, Bahamas, Kenya, Tanzania, India

2.1.1 Classification of Fibres

Fibres are two types: natural fibre and man-made or synthetic fibre. Natural fibres are a class of hair-like materials that are obtained from vegetables, animals, or minerals. Some of them can be spun into filaments, thread, or rope and they can be used as reinforcements in composites. Natural Fibres obtained from vegetables constitute cellulose, a polymer of glucose bound to lignin with varying amounts of other natural materials. Synthetic or man-made fibres are generally obtained from synthetic materials such as petrochemicals, but some types are manufactured from natural cellulose, including rayon and lyocell.

2.1.2 Classification of Natural Fibres

The natural fibres are classified based on their origin such as cellulosic (from plants/vegetable), protein (from animals), and mineral fibres as shown in Figure 2.1 [Mohanty et al. 2005, Jacob and Thomas 2008]. The plant fibres are further classified as seed fibres (e.g. cotton), bast fibres (e.g. linen from the flax plant), leaf fibres (e.g. sisal), husk fibres (e.g. coconut). The animal fibres are grouped under the categories of hair (e.g. wool), fur (e.g. angora) and secretions (e.g. silk). Mineral fibre includes asbestos. The bast and leaf fibres lend mechanical support to the plants stem or leaf respectively [Rowell et al. 1997, Nishino 2004, Krishnan et al. 2005]. The surfaces of natural fibres are uneven and rough which provides good adhesion to the matrix in a composite material.

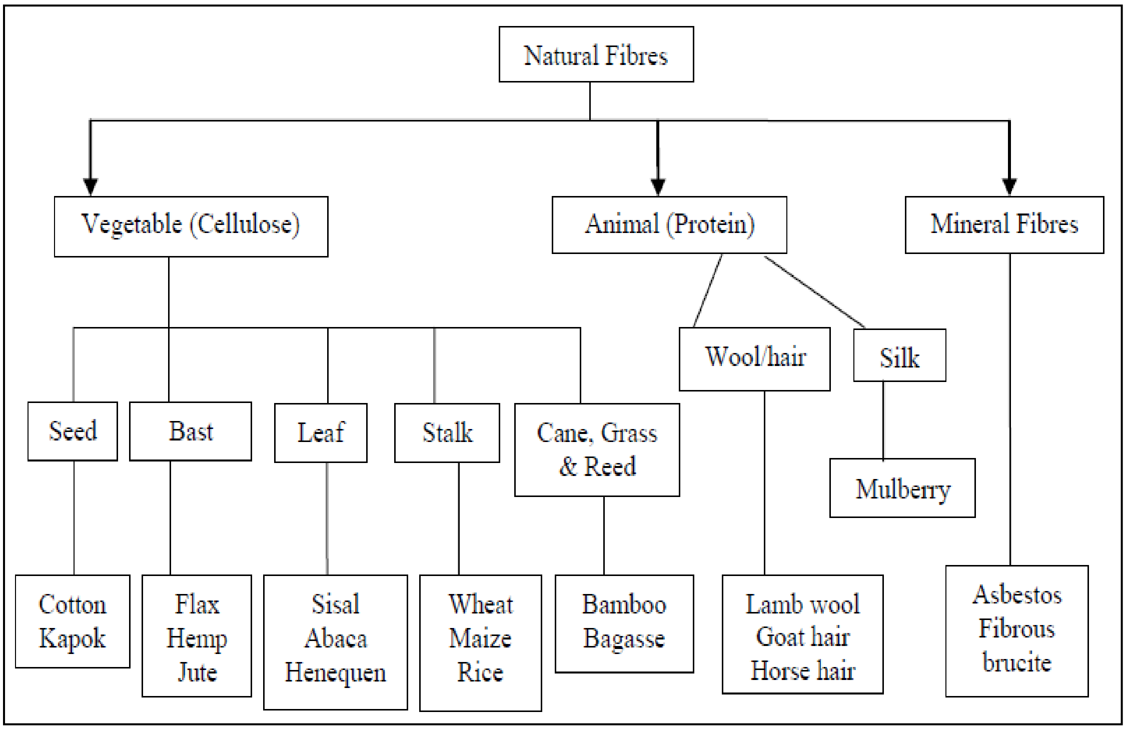


Figure 2.1: Structure of Natural Fibres [Jacob and Thomas 2008]

Vegetable fibres can also be classified as wood or non-wood depending on their origin; while wood fibres can be subdivided into soft wood and hard wood [Bledzki et al. 2002, Mohanty et al. 2005]. These fibres contribute greatly to the structural performance and provide significant reinforcement in plastic composites. Bast fibres are used in textiles, paper, building materials, and other industrial works.

Natural fibre composite can also be classified under three main categories as defined [Marston 2008].

1. Natural Fibres serving as filler in thermoplastics such as polypropylene.
2. Composites where longer fibres are enhanced with compatibilizers and adhesives to attain additional strength and toughness in thermoplastics.
3. Natural Fibres used with thermosetting resins for elements within engineered components.

Fibres (wood fibres) are categorized based on their type and from part of the plant derived [Bledzki et al. 2002].

(1) Soft/Stem or Bast Fibres -these fibres are found in many dicotyledonous plants and are grouped outside the xylem, in the cortex, phloem, and pericycle (bark). They are typically found to be grouped into clusters of several or many cells, and the whole cluster may in some cases serve as the fibre in spinning. Each fibre cell is tenaciously cemented to the adjacent fibre by the pectic middle lamella. The strands are quite strong and durable, and are able to withstand bleaching or other harsh treatments. Example: flax, ramie, hemp, and jute.

(2) Hard/Leaf or Structural Fibres- these are strands of small, short cells found in monocotyledonous plants and constitute supportive and conductive strands primarily in the leaf (few monocots have conspicuous woody stems) and are termed as fibro-vascular bundles. It consists of both xylem and phloem, and various sheathing cells, which are found scattered through a sleek pithy matrix of the leaf or stem. The cells are lignified to a greater or lesser degree and are hard in comparison with the soft fibres found in dicots in which the cellulose is largely associated with pectic materials. The entire fibro vascular bundle serves as a unit fibre and cannot ordinarily be bleached or chemically treated and they are less durable than soft fibres.

(3) Surface Fibres - these fibres are borne on the surface of stems, leaves, fruits, seeds, etc. The most important plant fibre are cotton, others include kapok (used for stuffing). All plant fibres, whether from wood or non-wood origin, are composed of three main cell wall polymers: cellulose, lignin and matrix polysaccharides (such as pectin and hemi cellulose) associated with cellulose and lignin in the cell wall. For example, flax, hemp, and ramie are the cellulose associated with pectin material, while jute fibres are more lignified. High percentage of cellulose in fibres correlates positively with desirable characteristics such as strength and durability. Likewise, low moisture content of the cell wall is usually indicative of physical superiority [Fowler et al. 2006, Alix et al. 2008].

2.2 Extraction of Natural Fibres

Fibre extraction procedures depend on the type, kind of plant and portion from which the fibres are derived such as bast, leaves, stem, fruit, wood, and other factors such as fibre performance and economics. But separation of the fibres from the original plant source is always an important step to obtain the high quality of fibres. Natural plant fibres are extracted and processed by various means that include methods like retting, breaking, scutching, hackling, and combing [Collier et al. 1992, Franck 2005].

Cellulosic fibres are extracted from lignocellulosic by products naturally using bacteria and fungi. Physical, chemical and mechanical methods are also followed to extract the fibre. These methods are carried out in such a manner that the morphological features are improved and processing way is done in a better way and chemical composition is not altered [Bledzki and Gassan 1999]. Physical methods such as stretching, calendaring, thermal treatment can also be carried out. Processing stages for natural fibres extraction is shown in Figure 2.2. Long and short fibres separation depends upon the method adopted by harvesting, extraction conditions, such as water pre-treatment, alkaline treatment, mechanical action, time, and properties of fibre bundle [Collier et al. 1992, Das et al. 2010].

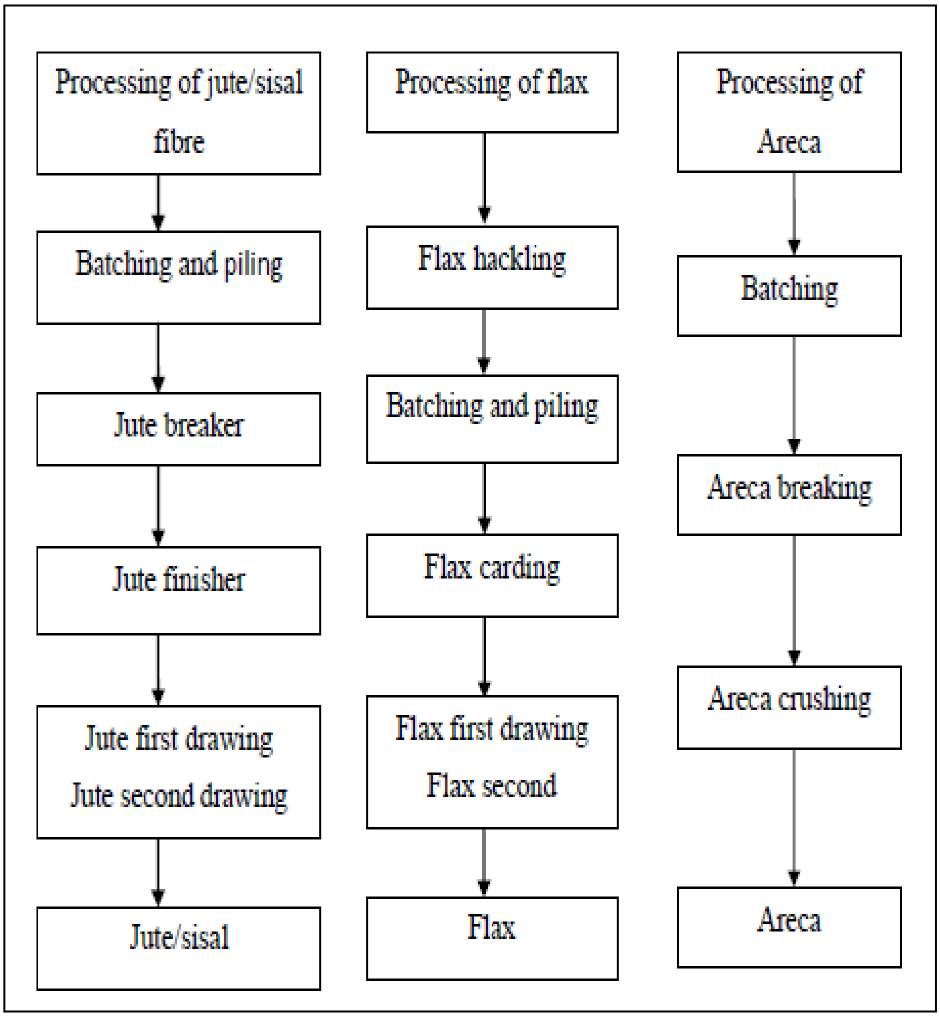


Figure 2.2: Stages Showing Fibres Obtaining from Plant [Das et al. 2010]

2.2.1 Decorticator

A decorticator is a machine that breaks up husk segments through metal bars revolving at high speed. It is the method involved to separate the bark (protecting cover) from the core material and the machine separates non-fibrous materials from the fibre. Decorticator can be operated by means of milling action which involves crushing or grinding operations. Hammer mill, ball mill, and disc mill can be used to operate under milling action of fibres [Fukazawa et al. 1982, Prasad et al. 2005].

2.2.2 Extraction Methods

(a) Traditional and Natural Method of Extraction

Retting, the traditional process to extract fibres uses bacteria and fungi in the environment to remove lignin, pectin and other substances. Most common chemical retting methods use alkalis, mild acids and enzymes for fibre extraction. Bacteria and fungi play an important role in water retting and dew retting. Bacteria retting are based on the natural action of anaerobic bacterior aerobic fungi. Bacillus and Clostridium bacteria are used in water retting, Rhizomucor pusillus and Fusarium lateritium fungi are used in dew retting [Henriksson et al. 1997, Li et al. 2007]. Atmospheric retting also provides better quality fibres, but the time required is more and relatively longer duration which also makes the fibre difficult in processing.

(b) Chemical Method of Extraction

The most common methods include alkalis, mild acids and enzymes for fibre extraction. Sodium hydroxide is the most commonly used chemical for fibre extraction. Sulphuric acid and oxalic acid in combination with a detergent are also been used for fibre extraction [Doraiswamy and Chellamani 1993]. Chemical retting is a low-concentration alkaline solution, and high temperature process. Factors that determine the quality of chemically extracted fibres include chemical concentration, duration of treatment and temperature. Enzymatic fibre extraction includes combination of enzymes such as pectinases, hemicellulases and cellulases which are generally used with a pre or post chemical treatment of fibre [Reddy and Yang 2005].

(c) Mechanical Method of Extraction

Mechanical decorticator machine consists of a pair of feed rollers and a beater as shown in Figure 2.3 and 2.4. Slices will be fed to the beater between the squeezing roller and scrapper roller and the pulp gets separated and fibres are extracted from the machine and air dried [Mukhopadhyay et al. 2008]. Mechanical separation of fibres is carried out by various methods such as decorticating machines, steam explosion (STEX), ammonia fibre extraction, Tilby process and other novel methods [Foscher et al. 1998].

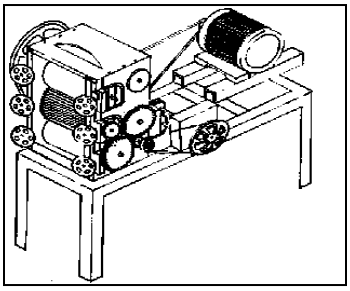


Figure 2.3: Banana Fibre Extractor

[Mukhopadhyay et al. 2008]

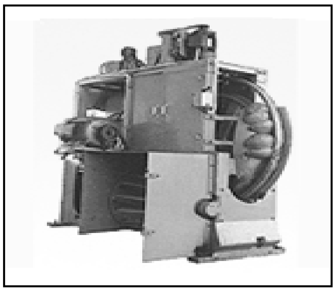


Figure 2.4: Coir Fibre Extractor

[Khan et al. 2007]

2.3 Structure of Natural Fibres

The cell wall of a plant fibre is a heterogeneous membrane and it is built up of two sections: primary cell wall and the secondary cell wall, which is further divided into three layers (S_1, S_2, and S_3). The primary cell wall is the first to be laid down when the cell is formed and are composed of randomly oriented micro fibrils, which allows for expansion of the cell as cell growth take place. The S_2 layer occupies the greatest volume of the cell wall and influences the properties of the cell. This layer comprises at least three hierarchical microstructures: macro fibril, micro fibril and micelle.

The structure of natural fibres is more complex compared to synthetic fibre. The overall properties of fibres depend upon the structure, cell dimensions, micro fibrillar angle and chemical composition of the plant fibres. Natural fibres have a hollow space called lumen, with nodes and inter nodes at irregular distances that divide the fibre into individual cells [Rowell et al. 1996]. The surface is rough, uneven and provides good adhesion to the matrix in a composite structure. Structure properties of natural fibres and plant fibre dimensions are shown in Table 2.2 and 2.3. The cell wall of plant fibres are mainly composed of polymers which includes cellulose, hemicelluloses, lignin and pectin. These polymers differ in molecular composition and structure and therefore display different mechanical properties as well as different water sorption properties. The content of the polymers is highly variable between plant fibres.

The orientation of the cellulose micro fibrils with respect to the fibre axis determines the stiffness of the fibre. They are known to be more ductile, if the micro fibrils have a spiral orientation to the fibre axis. If the micro fibrils are oriented parallel to the fibre axis, then they are inflexible, rigid and have a high strength. Few plant fibres with micro fibril angle are shown in Table 2.4. In general, the tensile strength and Young's Modulus of plant fibre increases with increasing cellulose content of the fibres. The properties of these constituents vary from place to place, according to the climatic conditions, type of harvesting and place of growth.

Table 2.2: Structure Properties of Natural/Bio-Fibres [Mohanty et al. 2005]

Fibre	Fibre structure			Fibre properties	
	Cell dimensions		Crystallinity (%)	Elongation (%)	Moisture Content (%)
	Length (mm)	Width (mm)			
Corn husk	0.5-1.5	10-20	48-50	12-18	9
Coir	0.3-1.0	100-450	27-33	17-47	10-12
Baggase	0.8-2.8	10-34	-	5.5-11.8	8.8
Rice straw	0.4-3.4	4-16	40	-	6.5
Wheat straw	0.4-3.2	8-34	55-65	-	10
Barley straw	0.7-3.1	7-24	-	-	8-12

Table 2.3: Some of the Plant Fibre Dimensions (mm) [Joseph et al.1999]

Type of fibre	Length	Diameter
Cotton	18	0.02
Cereal straw	1.5	0.023
Flax	25-30	0.012-0.027
Jute	2	0.02

Table 2.4: Plant Fibre with Micro Fibril Angle [Mohanty et al. 2005]

Plant fibre	Micro fibril angle (degrees)
Hemp	6
Flax	6-10
Jute	8
Sisal	10-25

2.4 Properties of Natural Fibres and Composites

The various properties of natural fibres such as physical, chemical, mechanical and thermal properties and their composite materials are reviewed in following sections.

2.4.1 Physical Properties of Natural Fibres and Composites

The parameters considered for fibre properties include crystalline cellulose content, micro fibrillar angle, cell number and cell aspect ratio. High cellulose content and low micro fibrillar angle are essential for achieving high fibre strength. Fibres are often characterized by their aspect ratio. The distribution of fibre dimensions in a sample is characterized by assuming a cylindrical geometry and measuring the length and diameter of the individual fibres. Most natural fibres have a maximum density of about 1.5 g/cm^3. Some natural fibres like wood fibres are hollow and have low densities in their original form and can be densified during processing treatment [Saxena et al. 2008]. Other parameters that can be considered are volume fraction of fibres, modulus and strength of the fibres, fibre matrix adhesion, fibre dispersion and toughness of the matrix [Young 1986, Giancaspro et al. 2009].

Bledzki and Gassan 1999 discussed on composites reinforced with cellulose based fibres. Physical properties of natural fibres were determined by factors of physical and chemical composition, cellulose content, cross section, angle fibrils, degree of polymerization and place of growth. The physical structures were modified by treating fibres with alkali and acetylation processes. Mechanical and physical properties of the composite depend on fibre content and coupling agents in the composite material. Influence on treatment with fibre content on creep, quasi-static, cyclic dynamic, impact behaviour of natural fibre reinforced composite are briefed out. Hybrid composite with natural fibres and their properties were also dealt.

2.4.2 Chemical Properties of Natural Fibres and Composites

Natural fibres can be considered as composites of helically wound hollow cellulose micro fibrils held together by a lignin and hemicelluloses matrix with little amount of waxes. The chemical composition of natural fibres varies with type of fibres. Natural fibres are basically rigid, crystalline cellulose, micro fibril-reinforced amorphous lignin and/or with hemi cellulosic matrix. Plant fibres except cotton, are composed of cellulose, hemi cellulose, lignin, waxes, and some water-soluble compounds. Cellulose, hemicelluloses, and lignin are the major constituents [Sarkanen and Hergert 1971]. The Chemical composition of plant fibres are shown in Table 2.5.

Table 2.5: Chemical Composition of Natural Fibres

Fibre	Cellulose (%)	Hemi Cellulose (%)	Lignin (%)	Pectin (%)	Other (%)
Sisal	67-78	10-14.2	8-11	10	3
Jute	61-71	13.6-20.4	12-13	0.2	0.5
Flax	71-78	18.6-20.6	2.2	2.2	5.5
Cotton	82.7	5.7	-	-	0.6
Bamboo	26-43	15-26	21-31	-	-
Abaca	61-64	21	12	0.8	-

Cellulose is the major component of plant fibres consisting of about 40% to 50% of the plant. Cellulose is a linear macromolecule consisting of D-anhydroglucose repeating units joined by β-1, 4-glycosidic linkages as shown in Figure 2.5. Cellulose molecules are extensively hydrogen bond with itself, with another cellulose molecule, and with polar molecules [Young 1986]. The hydrogen bonding plays an important role in chain arrangement and provides cellulose with crystal-like properties.

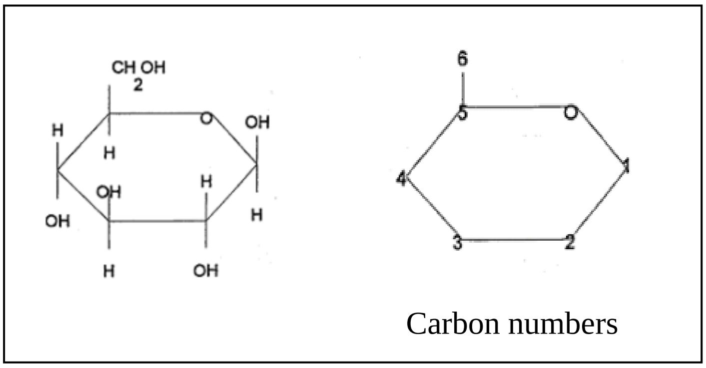


Figure 2.5: Cellulose Chemical Structure

Cellulose crystallinity imparts mechanical strength to the cell wall and makes it relatively unreactive and insoluble in aqueous systems. They occur in nature largely in a crystalline form made up of partially aligned or oriented linear polymer chains. The elementary unit of a cellulose macromolecule is anhydro- D-glucose, which contains three hydroxyls (-OH). These hydroxyls form hydrogen bonds inside the macromolecule itself and also with hydroxyl groups from the moist air. Henceforth, all natural fibres are hydrophilic in nature and their moisture content can reach up to 3-13% [Bledzki et al. 1996]. Cellulose can be altered into cellulose esters, such as cellulose acetate, cellulose acetate propionate, and butyrate, which are used as major components of thermoplastics.

Hemi-Cellulose molecules are hydrogen bonded to cellulose and act as cementing matrix between the cellulose micro fibrils, forming the cellulose hemi cellulose network, which was thought to be the main structural component of the fibre cell [Young 1986]. Hemicelluloses act as interfacial coupling agents between the polar surface of cellulose and relatively non polar lignin matrix. They play an important role in interacting with both cellulose and lignin. Hemicelluloses are composed of b-1, 4 linked D-xylopyranoyl units with side chains of various lengths containing L arabinose, D-glucuronic acid, or its 4-O-methyl ether, D-galactose, and D-glucose [Lawther et al. 1995] and its chemical structure is shown in Figure 2.6. The polymer chains of hemicelluloses have short branches and are amorphous in nature and because of this factor hemicelluloses are partially soluble or swellable in water [Sreekala and Thomas 2003, Sgriccia et al. 2008].

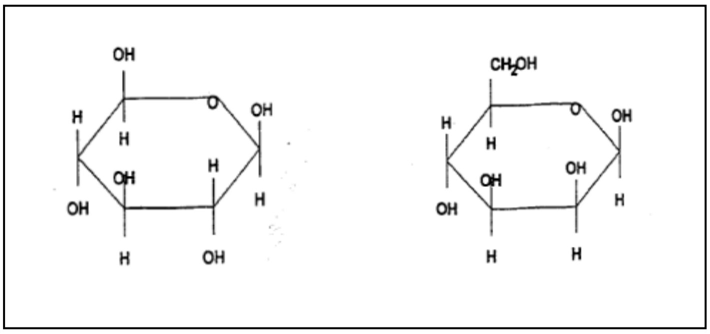


Figure 2.6: Hemi Cellulose Chemical Structure

Lignin contributes too many physiological functions including structural support, cementing polysaccharides, and resistance to microbial degradation. All these functions originate from the hydrophobicity and rigidity of the lignin structure and poly-phenolic nature. Lignins are made up of phenylpropane units [Sarkanen and Hergert 1971] and they are phenolic compound created by the enzymatic polymerization of three phenolic alcohols, known as mono lignols (pcoumaryl, coniferyl and sinapyl alcohols) and have aliphatic and aromatic constituents. The most abundant linkage is the β-O-4 ether bond, which accounts for 40%- 60%. Lignin act as cementing agent in the matrix of cellulose fibre of plant structures and are encrusted in the cell wall and partly covalently bonded with the hemicelluloses polysaccharides. Some of their functions are to provide structural strength, and sealing of water conducting system that links the roots with the leaves [Glasser and Sarkanen 1989; Stark 2001], to protect the plants against degradation and they also have potential ability use as adhesives.

Pectin is a collective name for hetero polysaccharides, which consists essentially of poly galacturon acid. They are complex polysaccharides and the main chain consists of a modified polymer of glucuronic acid and residues of rhamnose. The chains are rich in rhamnose, galactose, and arabinose sugars [Mohanty et al. 2001]. These chains are often cross-linked by calcium ions, improving structural integrity in pectin-rich areas. Pectin is important in non-wood fibres, especially bast fibres. The lignin, hemicelluloses, and pectin collectively function as matrix and adhesive, helping to hold together as cellulosic framework structure of the natural composite fibre. Pectin is soluble in water only after a partial neutralization with alkali.

The **waxes** make the part of the fibres, which can be extracted with organic solutions. Waxy materials consist of different types of alcohols, which are insoluble in water and several acids such as, palmitic acid and oleaginous acid. Fibre Structure and properties of few agricultural stalk residues are shown in Table 2.6 and chemical constituents of stalk fibre are shown in Table 2.7.

Table 2.6: Fibre Structure and Properties of Some Agricultural Stalk Residues

[Reddy and Yang 2005, 2005a]

Fibres	Fibre Structures			Fibre Properties	
	Cell Dimensions		Crystallinity (%)	Elongation (%)	Moisture Content (%)
	Length (mm)	Width (μm)			
Corn Husk	0.5-1.5	10-20	48-50	12-18	9
Sorghum Stalk	0.8-1.2	30-80	-	-	8-12
Rice Straw	0.4-3.4	4-16	40	-	6.5
Wheat Straw	0.4-3.2	8-34	55-65	-	10
Barley Straw	0.7-3.1	7-24	-	-	8-12

Table 2.7: Chemical Constituents of Stalk fibre

Type of Fibre	Source	Cellulose (%)	Lignin (%)	Pentosan (%)	Ash (%)	Silicon (%)
Stalk Fibre	Rice	28-48	12-16	23-28	15-20	9-14
	Wheat	29-51	16-21	26-32	4.5-9	3-7
	Oat	31-48	16-19	27-38	6-8	4-6.5

Jin et al. 2009 characterized, compared degraded and non-degraded hemicelluloses from stalk fibres of barley and maize. Hemi cellulose structure and thermal component of the fibres were quantitatively analyzed and fractionated. The results showed that thermal stability of hemicelluloses decreased with declining molecular weight. Hemi cellulosic polymers were unbounded to the lignin in the cell wall of straw fibres.

Structural, chemical and thermal treatment of maize, rice and rye straw fibre was studied by Xiao et al. 2001. Alkali and lignin content of the fibres were characterized by acid hydrolysis methods and non destructive techniques. Maize and rye straws had dominant of glucuronoarabinoxylans and rice straw had a-glucan and k-arabino, d-xylan. Thermal analysis showed hemicelluloses degrading first whereas cellulose degradation occurring at 250º- 330ºC and proving higher thermal stability than hemi cellulose. Lignin had less degradation and less thermal stability.

Chemical and thermal characterization of cellulose from barley straw fibres was studied by Sun et al. 2005. The samples were characterized by Fourier Transform Infrared Spectroscopy (FTIR) and Nuclear Magnetic Resonance (NMR) spectroscopy method. The cellulose content, crystallinity and thermal stability was found to be higher in purified cellulose than raw cellulose. Isolation of pure cellulose by alkaline peroxide treatments was found to be improved than other methods and the obtained cellulose was free from hemicelluloses and lignin.

Reddy and Yang et al. 2004 and 2005 developed fibre extraction method that results partial delignification of fibres and produced fibres from cornstalks with better mechanical properties. Structural, chemical compositions of the fibres were characterized by X-ray diffraction and scanning electron microscope and found to be different than that of other stalk fibres. Corn stalk fibres had lower percent crystallinity and similar micro fibrillar angle of bast plant fibres. The results showed that cells in corn stalk fibre had smaller dimensions than cotton but larger than flax fibres and crystals providing high fibre strength but low elongation. Agro based bio fibres such as corn, sorghum, wheat, rice, barley, has potential use in industrial and textile applications.

2.4.3 Mechanical Properties of Natural Fibre and Composites

In natural fibre reinforced composites, fibre acts as reinforcement and exhibits high tensile strength and stiffness. The mechanical properties of reinforcement (fibres) have direct relation with the tensile strength and stiffness of the composite. The selection of suitable reinforcing fibres follows certain other criteria such as, thermal stability, fibre-matrix adhesion, long time behaviour, elongation at failure and also others like price and processing costs. Natural fibres have low specific gravity, which is about 1.25-1.50 gm/cm^3 as compared to synthetic fibres, especially glass fibres, which are about 2.5 gm/cm^3 as shown in Table 2.8.

Lignocellulose fibres are able to provide high strength to weight ratio in plastic materials. The influential of surface treatments in natural fibres on the interfacial characteristics were studied [Mohanty et al. 2001a, Joffe et al. 2003]. The micromechanics approach and the thermo-elastic anisotropy [Cichocki and Thomason 2002] of natural fibre composites were also determined by researchers. The mechanical properties of a natural fibre depends on various factors such as climatic and atmospheric conditions, processing features, extracting techniques, type of soil, harvesting methods and other atmospheric conditions.

Table 2.8: Properties of Natural Fibres and Synthetic Fibres

[Mohanty et al. 2001, John and Thomas 2008]

Type of Fibre		Density (g/cm$^{3)}$	Tensile Strength (MPa)	Youngs Modulus (GPa)	Elongation at Break (%)	Moisture Absorption (%)
Natural Fibres	Cotton	1.5-1.6	287-800	5.5-12.6	7.0-8.0	8 -25
	Coir	1.15	131-175	4-6	15-40	10
	Flax	1.50	345-1100	27.6	2.7-3.2	7
	Jute	1.3-1.45	393-773	13-26.5	1.16-1.5	12
Synthetic Fibres	Carbon	1.7	4000	230-240	1.4-1.8	-
	E-glass	2.5	2000-3500	70	2.5	-

The mechanical properties of a natural fibre are shown in below Table 2.9 and the properties depend on other parameters such as fibre length, fibre strength, modulus and orientation of fibres in addition to the fibre matrix interfacial bond strength. The adhesion of natural fibres and matrix are also important in order to obtain good mechanical properties. Basically fibre acts as reinforcement and exhibits high tensile strength and stiffness to the composite material. Natural fibres often show large degree of non-uniformity in most characteristics such as chemical composition, crystallinity, surface properties, diameter, cross-sectional shape, length, strength, and stiffness [John and Thomas 2008]. In addition natural fibre offers good thermal and acoustic insulation properties.

Sreekala and Thomas 2003, investigated on water absorption studies and mechanical performance on treated natural fibre. A palm fibre as (reinforcement) with phenolic matrices was developed and water absorption characteristics were studied for the developed composite material. Hydrophilic nature of the fibre was discussed and various surface modification treatment such as silane treatment, acetylation, mercerization, gamma radiation was used to reduce hydrophilic nature of the fibre. The obtained results showed that mechanical properties of the fibre decreased on sorption of fibres and this decrease is due to physical and chemical changes that occurred on modification of fibres. Performance of mechanical properties, modulus and strength of fibres showed decreased strength on water absorption and regained strength on desorption.

Table 2.9: Physical and Mechanical Properties of Natural Fibres
[Mohanty et al. 2001, Somnuk et al. 2004, John and Thomas 2008]

Properties	Sisal Fibre	Coir Fibre	Jute Fibre	Glass Fibre
Diameter (μm)	100-300	100-400	12-25	13
Density (g/cm^3)	1.45	1.15	1.45	2.5
Moisture (%)	11	10-12	12	-
Elastic Modulus (GPa)	9-20	4-6	10-30	72
Tensile strength (MPa)	511-635	131-175	393-773	2000-3500
Elongation (%)	3-7	15-40	1-2	2.5

Garkhail et al. 2000 developed composite material consisting of plant fibres of flax and polypropylene matrix using film stacking method. Mechanical properties, influence of fibre length and content, volume fraction of fibres, strength and stiffness of the composite material were investigated. Strength and stiffness results were compared with the micro mechanical models and improved results was obtained. Stiffness of the flax fibre reinforced thermoplastics was compared to E-glass fibre and obtained result was better; whereas tensile strength of flax composite was low to E-glass fibre.

Nascimento et al. 2010 obtained composite material of piassava palm fibre with epoxy polymer. Tensile test and characterization studies such as scanning electron microscope were performed. The result shows tensile strength and elastic modulus of the composite decreasing with 30% of the fibres and large amount of fibres accounting for the better performance of composite with 40% volume fraction. The fibres acted as a barrier to transverse crack propagation through the polymer matrix and 40% of the volume fraction of fibre was enough to undergo rupture for tensile strength.

Mercerization on polymer composites and effect of alkali treatment of the composites was studied by Qin et al. 2008. Mechanical properties, structural and morphology studies of the composite material was investigated. The prepared composites consisted of ramie fibres and the tensile strength of the composites improved after mercerization. Alkali treatment improved the tensile strength of the composite from 440 MPa (unmercerized composites) to 540 MPa (mercerized composites) and obtained values were much favourable. Characterization techniques of Raman spectroscopy revealed that orientation of the fibres in mercerized composites were well maintained. Composites with 4% cellulose concentration and fibre volume fraction of 85% had highest tensile strength. Matrix phase, wetting of fibres and interfacial interaction between fibres and matrix also played a major role for resulting efficient composite material.

Bledzki et al. 2001 developed polyurethane composites reinforced with flax and jute and the fabric was processed to produce foam structure. Investigation on influence of fibre, mechanical properties, and micro void content was studied. The micro void content was varied with different resin grade and the size of micro void was analyzed. On having high amount of fibre content, it had an increase in impact strength, Young modulus and shear modulus; whereas increasing the micro void content resulted in low value impact strength, Young modulus and shear modulus. Dynamic mechanical properties are slightly enhanced at higher fibre content. Flax based composites had higher strength and stiffness than jute fibre composites.

Gassan and Bledzki et al. 1997 investigated fibre treatment on composites consisting of jute fibres and their influence on coupling agent treatment. Flexural strength of the composites with treated fibres was high than that of raw fibres and flexural modulus was increased by 90%. Dynamic strength of the modified fibre composite was raised by about 40% strength of the load. Fibre pull out was reduced for chemical treated fibres and there was an improvement in fibre matrix adhesion. Stress transfer from matrix to fibre was improved and the treated fibre gave high damage resistance under dynamic loading and it attained increased dynamic strength.

Ince et al. 2005 dealt with chopping which influenced the properties of the stalk. The bending forces, stress, shearing stress and shearing energy were determined for sunflower stalk. Cantilever test was performed to determine the modulus of the stalk and it was carried for different moisture contents. The bending stress decreased as moisture content was increased and modulus of elasticity decreased. Shearing stress and energy increased as the moisture content was increased. The average value of the bending test at high moisture content was approximately two times greater than that of low moisture content. The effect on cutting had great influence in moisture content because cutting height on shearing energy reduced as moisture content reduced.

Dweib et al. 2004 and 2006 worked on natural fire composites for structural applications such as housing application, structural panels and beams. A composite structural beam panel is processed by Vaccum Assisted Resin Transfer Molding (VARTM) and it consisted of natural fibres of flax and paper with soybean oil based resin. Mechanical testing of beams yielded good results with structural performance. Natural fibres reinforced with 20-55 % weight fraction fibre increased the flexural modulus to 2-6 GPa whereas the same resin reinforced with glass fibre gave flexural modulus of 17 GPa. Bio based composite consisting of reinforced natural fibres and matrix of bio resins have lot of environmental advantage to the traditional ones. The beam had high strength to stiffness to weight, better ductility, design flexibility and better survival in weather conditions. The composite structure was compared to the conventional wood structures and the resulting stress strain diagram and analysis provided that they can compete with traditional method and can be used for manufacturing complete roof for a house.

The water sorption capacity of plant fibres is an essential aspect of plant fibre composites [Sreekala et al. 2002, Espert et al. 2004, Pothan and Thomas 2004, Madsen et al. 2012, Alamri and Low 2013]. Understanding water sorption in the composites requires necessarily an understanding of water sorption in the fibres themselves. The affinity between liquid water and a solid material is characterized by the contact angle at the water-material interface, and the material is denoted hydrophilic when the angle is below 90° and hydrophobic when the angle is above 90º.

2.4.4 Thermal Properties of Natural Fibre Composites

Sombatsompop and Chaochanchaikul 2004 studied on structural, thermal and mechanical properties of wood composites and their moisture effect change in wood composites was discussed. Effect of moisture content with varying weight ratio of the composite was evaluated. Texture characteristics was also examined and the work was carried out with different proportion of wood saw dust contents. The obtained results confirm that at low moisture level, tensile modulus decreased and elongation at break of the composites increased. This is because of reformation of hydrogen bonding between water molecules and saw dust particle. Impact strength of the composite improved with low moisture content and the result was same as that of tensile test. The decomposition temperature of the composite decreased with moisture content and glass transition temperature was independent of moisture content. Greater the saw dust content lower the decomposition temperature.

Lopez et al. 2006 discussed the effect of moisture, temperature and ultra violet exposure on natural fibre plastic composites. Mechanical properties of the composite and performance of the composite for short term and long term test was conducted at natural conditions. Chemical changes were observed in the material and relative humidity affected the modulus of rupture and modulus of elasticity. In short term test, exposure to high relative humidity reduced the modulus of rupture and modulus of elasticity. The temperature effect and ultra violet exposure on the composite had greater effect in the material.

Grozdanov et al. 2006 studied the effects of several chemical treatments (dewaxing, alkali treatment, acetylation) on natural fibre reinforcements and the properties like thermal stability, crystallinity and surface morphology were reviewed.

2.4.5 Comparison of Natural and Synthetic Fibres

Advantages of natural fibres include low specific weight which results in high specific strength and stiffness than conventional glass fibres. Procurement of natural fibres with low cost makes the material an interesting product for low wage countries. Thermal recycling is possible where glass fibre causes problems in combustion furnaces. Natural fibres are renewable resource and the production requires little energy and CO_2 is used while oxygen is given back to the environment. It provides better thermal and acoustic insulation properties. No additional infrastructure requirement is needed for recovery and separation for recycling of fibres. Natural fibres do not cause any allergies or lung diseases if breathed in or came in contact with it. Some of the disadvantages are natural fibres price can fluctuate by harvest or agricultural politics, lower durability, moisture absorption which causes swelling of the fibres, low strength properties particularly its impact strength. Natural fibres are not much processed for commercial use utility as reinforcements in plastics [Joseph et al. 1999, Giancaspro et al. 2009, Gutierrez et al. 2012].

2.4.6 Limitations of Natural Fibre Composites

One of the major problems associated with the use of natural fibres in composites are high moisture sensitivity leading to severe reduction of mechanical properties [Angelini et al. 2000, Lilholt, and Lawther 2000, Rowell 2002, Eichhorn and Young 2003, Wang et al. 2006]. The reduction may be due to poor interfacial bonding between resin matrices and fibres. It is therefore necessary to modify the fibre surface to render it more hydrophobic and also more compatible with resin matrices [Kumar et al. 2005, Harish et al. 2009, Gutierrez et al. 2012, Kestur et al. 2013]. Generally acids and alkali have been used for modifying jute, coir and other natural fibres [Xie et al. 2010, Shekeil et al. 2012]. Strong alkali solutions lead to reduction of strength, an increase in elongation at break and reduction in stiffness of the fibres; whereas lower concentration of alkali (10%) does not cause significant lowering in strength [Sgriccia et al. 2008, Alawar and Hamed 2009, Mohan and Kanny 2012].

The limitations of natural fibres are [Schemanauer 2000, Manfredi et al. 2006, Salemane and Luyt 2006, Monteiro et al. 2012, Bakar et al. 2013].

- Low strength properties
- Poor fire resistance
- Low durability
- Variability in quality, depending on unpredictable influences such as weather
- Moisture absorption, which causes swelling of the fibres
- Restricted maximum processing temperature (up to 220°C)
- Price can fluctuate depending on harvest results
- Processing of fibre is a labor intensive
- Dimensional instability.

2.5 Applications of Natural Fibre Composites

Natural fibre composites has been used widely for making building products such as window, door, siding, fencing, roofing, decking and so on, as shown in Figure 2.7. Composite wood products produced from veneers, wafers, flakes and particles are becoming accepted in structural systems that may have traditionally employed solid wood or steel materials, a reason for this diversity in product application is that these composites can be produced with a wide range of structural properties.

The entire plant (leaves, pith, stock, roots) can be used directly/ indirectly to produce structural, non-structural composites and other materials as shown in Figure 2.8. The office furniture made of wheat straw is shown in Figure 2.9 and automotive component from kenaf fibre is shown in Figure 2.10 [Holbery and Houston 2006]. All the major car manufacturers in Germany and in the world (i.e., Daimler Chrysler, Mercedes, Volkswagen, Audi group, BMW, Ford, and Rover) are using natural fibre composites in their product applications [Tylor 2002] and are listed in Table 2.10.

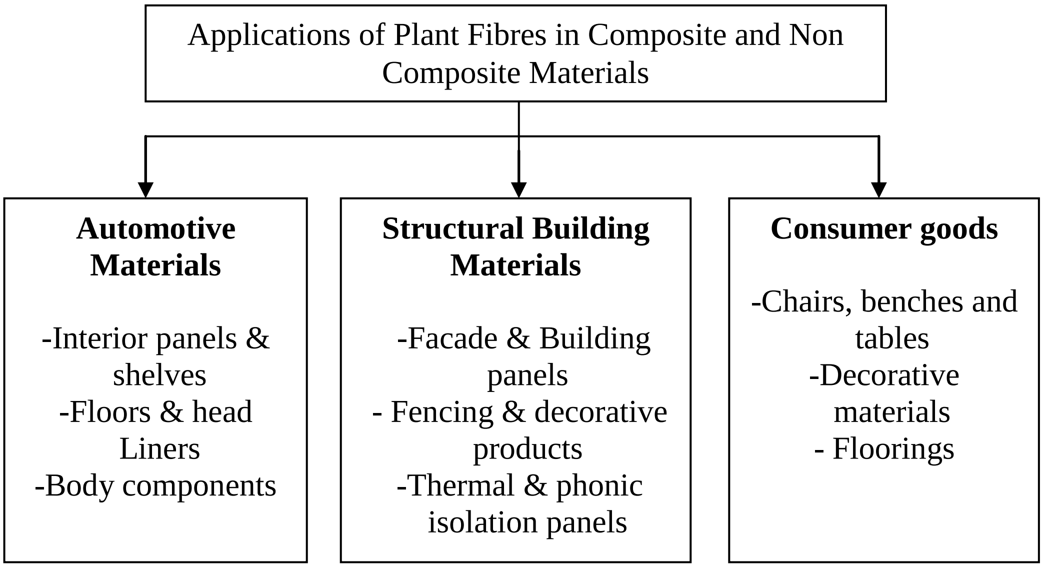


Figure 2.7: Applications of Plant Fibres in Composite Sectors [Puitel et al. 2011]

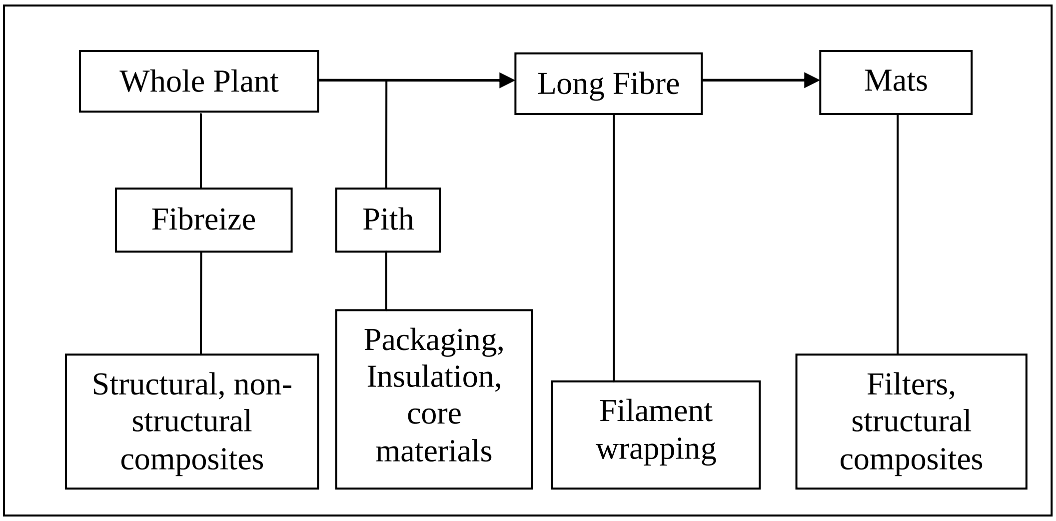


Figure 2.8: Application of Each Part of Plant Fibres [Rowell et al. 1996]

.

Figure 2.9: Office Furniture

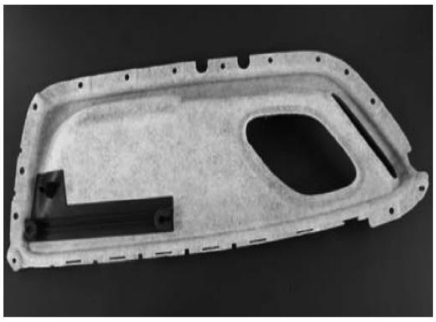


Figure 2.10: Automotive Component

[Greendoor 2000]

Table 2.10: Automotive Manufacturers and Components Utilizing Natural Fibres

[Tylor 2002]

Automotive Manufacturer	Applications
Audi	Seat backs, side and back door panel, spare tyre lining
BMW	Door panels, head liner panel, seat backs
Daimler Chrysler	Door panels, wind shield/dash board, pillar cover pannel
Ford	Door panels, boot liner
Rover	Insulation, rear storage shelf/panel
Volkswagen	Door panel, seat back, boot liner

Daimler Chrysler, already a major user of natural fibre composites, aims to increase the proportion of natural fibre materials in their cars. Interior trim components such as dashboards and door panels using polypropylene and natural fibres are produced by Johnson controls [Green door, 2000]. BMW currently uses 8 kg to over 13 kg of natural fibres per vehicle while Ford uses from 5 to 13 kg (these include wool and cotton). Interior parts of automotive are shown in Figure 2.11 and 2.12.

Figure 2.11: Interior components made of reinforced natural fibre composite for E- Class car [Ellision and Naught 2000; Greencarcongress 2008]

Figure 2.12: Part of hemp and glass plastic bonded with resin solutions for bus seat [Tylor 2002]

2.5.1 Natural Fibres and Composites in Structural Applications

Tita et al 2003, Aly et al. 2010 developed composite beams for structural applications and finite element method approach was used to study the vibration behaviour of composite beams. Glass fibre and polyester resin was used and processed by hand lay-up method. Dynamic test was conducted with different fibre orientations and the experiment results were validated with finite element software package ANSYS. Dynamic characteristics of laminated composite beam was analyzed and changes in fibre angle yielded different dynamic behaviour of the component. Fibre with different orientations and stacking sequences was done and the results were validated with ANSYS. Theoretical and experimental results were compared and had a good agreement with the experimental values.

Natural Fibre composite applications in building industry can be classified into two main groups: structural and non structural natural fibre composites [Rowell 1995]. A structural natural fibre composite can be defined as one that is needed to carry a load in use e.g. Load bearing walls, stairs, roof systems, and subflooring. A non structural natural fibre composite can be defined as one that is not needed to carry a load in use e.g. ceiling tiles, furniture, windows, and doors. The natural fibre composites used for building & construction industries include panels, false ceilings, partition boards, packaging, wall and floor coverings, extruded sections, roof coverings, insulation boards, fittings etc., [Rowell 1997]. However, there are some critical performance requirements that determine the best application for crop-based construction materials and they are: economic performance, social performance and environmental performance. Ligno cellulosic natural fibres such as sisal, coir, jute, ramie, kenaf, pineapple leaf have the potential to be used as a replacement for glass or other traditional reinforcement materials in composites. They have high specific properties such as stiffness [Sherman 1999], impact resistance [Sydenstricker et al. 2003], flexibility [Manikandan et al. 1996], modulus [Eichhorn et al. 2001] and are renewable and biodegradable.

Agro-based fibres suitable for composites are mainly from two sources [Julian and Vincent 2000]. The first is agricultural residues e.g. rice husks or cereal straws, feed crops and the second class is ligno cellulosic grown specifically for their fibre. Crop based fibres have a long application tradition in construction with sisal, cork, coir, bamboo, linoleum and others. Natural fibre composites are used as a substitute for timber in construction purposes and can be moulded into sheets, boards, frames, pallets, structural sections and many other shapes. They can also be used as a substitute for wood, metal or masonry for partitions, false ceilings, fences, railings, flooring, roofing, wall tiles etc. Composition boards including particle board (extruded and plate-pressed), fibre boards and medium density fibre board are quite common in construction, furniture and interior panelling [Chou et al. 1989].

The most common raw material used is wood, but many countries successfully use other agriculturally based residues like flax, hemp shives, bagasse, reed stalks, cotton stalks, jute stalks, grass like miscanthus, rape straw, oil flax straw, small grain straw, peanut husks, rise husks, and palm stalks [Kozlowski et al.1994]. Sisal fibre is one of the best prospective reinforcing materials used for more experiential than technical aspects [Dias et al. 1999]. In developing countries, sisal fibres are used as reinforcement for use in housing and structures. Sisal fibres need a low degree of industrialization for their processing and the role of sisal fibres in cement composites improved the toughness and post cracking of the matrix. Work carried by Savastano et al. 2002, 2005 showed that banana pulp, eucalyptus grandis and sisal wastes had satisfactory adhesion in fibre cement composites in comparison to the synthetic reinforcing fibres [Aziz et al. 1984]. Natural fibre materials for house components are shown in Table 2.11.

Table 2.11: Materials Selected for the Realization of Eco-Houses Components
[Srebrenkoska et al. 2009, ECO-PCCM 2002]

Fibre	Matrix	Application
Kenaf	PLA	Fibre-matrix based panels
Kenaf	PLA	Polymer matrix based panels
Kenaf	PHBV	
Rice straw	PLA	
Rice straw	PHBV	
Recycled cellulose	PLA	
Recycled cellulose	PHBV	

Sisal, hemp, coir, elephant grass fibre are used as reinforcements in soil constructions and other composite elements (e.g. Fibre board's) [Joseph et al. 1999]. Natural fibres are twisted to ropes to tie building elements and structural members in roof construction. Straws are used for thatch roofs and particle boards. In industrial process compressed straw slabs (Stramit) are produced by heat and pressure without any binders. Reeds, bundled are used as boards for various applications such as columns, beams, wall cladding, sun screens and roofing material. Leaves are used for thatch roofs, mats and panels for floors and walls [Swift 1985, Alawar and Hamed 2009]. Natural fibre composites are used for load bearing, sound roof, and good exterior covering and can also offer a durable structure with good thermal performance [Mumma 2002]. Areca fibre composite laminates were prepared with randomly distributed fibres with maize stalk and phenol formaldehyde. The composite laminate was prepared with different proportions of phenol formaldehyde and fibres [Swamy et al. 2004, Kumar 2008]. Baggase cement boards are used for building boards which are suitable for portioning, panelling and door shutter [Green Building Materials Report 2009]. Pulps from sisal and banana pseudostem fibres were produced by chemi thermo mechanical pulping process and used as composites [Savastano et al. 2002].

Table 2.12: Common Wall Construction Materials [Stulz, and Mukerji 1993**]**

Material	Characteristics
Concrete	Expensive, suitable for all climates, load bearing, good durability, resistance to all natural hazards and fire, no surface treatment needed.
Raw clay	Cheap, good material for most climates, durability achieved by good compaction, low earth quake resistance
Burnt clay bricks	Medium costs, suitable for all climates, used for load bearing
Fibre concrete	Low to medium costs, lighter and weaker than ferrocement, mainly sheets and tiles for cladding
Natural fibres, grasses, leaves	Cheap, only used in warm humid climates for light weight, wall panels and claddings, low durability and resistance to natural hazards
Bamboo	Cheap, ideal for infill walls and cladding, similar to fibres, grasses and leaves
Timber	Medium costs, good for most climates, good earth quake and hurricane resistance

A composite board was prepared using mixture of sawdust from pine and coir. They are done with different mixing ratios and using portland cement as binder. Physical and mechanical properties of cemented bonded board were evaluated [Erakhrumen et al. 2008]. Kenaf based composites consisting of thermosetting PLA are mixed to obtain mat and processed by hot press where the fibres are softened promoting their adhesion to kenaf fibres and creating very lightweight panels [Srebrenkoska et al. 2009]. Jute and coir composites also provide an economic alternative to wood for the construction industry and are used for the manufacture of flush door shutters. Some of the construction materials and their characteristics are shown in above Table 2.12.

Silva et al. 2012 developed bamboo composites as a functional graded composite material for construction and bamboo fibres are widely used for low cost building purpose. Bamboo as functionally graded material was studied and finite element method was used for studying mechanical behaviour of the material. Youngs modulus and constitutive properties were obtained from homogenization theory. Tension, bending and torsion load were studied by simulation through homogenization method. Finite element formulations were done to observe the bamboo behaviour and it captured material distribution through the bamboo wall. Numerical analysis technique was carried out for bamboo and it had more influence in mechanical and structural properties.

To improve the performances and to face new challenges on construction materials Central Building Research Institute (CBRI), Roorkee (India) is channelling most of its intellectual and physical resources on (a) development of new, low cost materials from local resources, (b) conversion of industrial/ agricultural wastes into building materials, (c) improvement in behaviour and performance of materials, and (d) savings in energy [Mathur 2002]. Swedish Cement and Concrete Institute are been involved with sisal products for many years and sisal fibre based roof tiles in South Africa are prominent [Rowell 2002]. Construction of inexpensive primary school buildings using jute fibre reinforced polyester in Bangladesh under the auspices of the Co-operative of American Relief Everywhere (CARE) and United Nations Industrial Development Organization (UNIDO). Commonwealth Scientific & Industrial Research Organization (CSIRO), Australia adopted an autoclave process using wood fibres as reinforcement for making asbestos free cement products in the 1980. National Institute of Research on Jute and Applied Fibre Technology (NIRJAFT), Kolkata has also come out with a number of technologies which helped in great extent for the commercialisation of jute/coir based composites. The Central Building Research Institute (CBRI), Roorkee and Regional Research Laboratory (RRL), Bhopal had investigated several techniques for sisal fibre surface modification for the use in the production of roofing sheets. One of the major applications for bio composites that recently gained in the region of North America was in building materials. At the University of Delaware, Affordable Composites from Renewable Sources (ACRES) group are been actively involved in developing bio-based thermosetting resins from plant oils such as soybean for structural applications.

2.6 Bio-Composites

Bio composites consist of bio fibre and bio plastic from renewable resources and thus expected to be bio -degradable. Bio composites derived from plant (natural/ bio fibre) and crop/ bio-derived plastic (biopolymer/ bioplastic) are eco friendly and termed as green composites [John and Thomas 2008] and eco-composites [Kim and Kim 2008].

The processing of bio composites remains same as that of plastics or composite materials which includes thermoset and thermoplastics. Fabrication techniques for manufacturing natural fibre reinforced composites include open mould methods like hand layup techniques or unidirectional fibres/mats and closed mould methods like resin transfer moulding and compression moulding. Pultrusion for continuous fibres, filament winding, and sheet moulding (press moulding) for short and chopped fibres are also used. Most of the bio composites based on thermoplastic polymers such as polyethylene and polypropylene and are processed by compounding and extrusion. Investigators [Fowler et al. 2006] mentioned different procedures such as compounding and extrusion of polymers with natural fibres, comingling of thermoplastic with natural fibres, and processing of thermosetting polymer matrix composites.

Bio based composite materials are new and innovative class of materials are being developed today and consist of environmentally friendly resins and natural fibres [Donnel et al. 2004; Nishino et al. 2004; Phuong and Lazzeri 2012; Alsaeed et al. 2013]. Generally, natural fibre act as reinforcement material and the matrix material is synthetic polymer or bio polymer [Velde and Kiekens 2001, 2002, Oksman et al. 2003; Wool and Sun 2005]. Natural fibres from vegetable are named as ligno cellulosic fibres, where the cellulose provides the strength while the lignin and hemi cellulose provide the toughness and protection of the fibres [Joseph et al. 1999]. Single fibres are themselves made of several micro fibrils and a good orientation angle of these micro fibrils as well as high cellulose content gives better mechanical properties. Life cycle assessment of bio fibres replacing glass fibres were studied and proved that bio fibres are equivalent to glass fibres at some specific conditions and methods [Joshi et al. 2004].

Liu et al. 2005 and 2007 developed green composites of soya based plastic and pineapple leaf fibre using injection molding and twin screw extrusion. Physical, thermal and mechanical properties of the composite was analysed with the help of dynamic mechanical analyzer and united testing system. The bio composite material properties increased with increased fibre content and with the addition of compatibilizer it decreased the water absorption. Tensile, impact and flexural properties of the composite material was studied and observed increase in mechanical properties. 30 wt % of the pine apple leaf fibre composite material had a good improvement. Water absorption of the treated fibre reduced its properties when compared to the untreated fibre composites.

Burgueno et al. 2005 fabricated material of bio composites for housing and panel applications. Hierarchical cellular designs and hybridization techniques were involved to increase the performance of the composites. Bio composite panel consisting of natural fibres and unsaturated polyester resin was used to produce a structural member. Long term performance, indoor and outdoor conditions evaluated for achieving success in practical case. Adhesion between the fibres and resin are to be improved to achieve a successful composite material. Validations of a cellular bio composite material, stiffness, dimensional stability, strength are evaluated through material test and flexural test. Hierarchical plates processed from natural fibres were evaluated analytically and experimentally. The result shows that bio composite material can be an optional material for building structures.

A nano green composite from epoxidixed soyabean oil was investigated by Liu et al. 2008. The composite material was prepared with flax fibre and with organo clay reinforced with epoxidized resin. Flexural modulus increased with fibre content, lower than 10 wt % where as tensile modulus increased with fibre content maximum at 13.5 %. Comparison between conventional traditional plastics and biopolymers are shown in Table 2.13.

Table 2.13: Comparison between Petroleum and Bio- Based Plastics

Sl. No.	Factors	Petroleum based Plastics	Bio - Plastics
1	Sustainable	No	Yes
2	Renewable	No	Yes
3	Degradation	Few degradable by polymer oxidation	Bio- degradable
4	Emissions	High	Low
5	Fossil fuel usage	High	Low

2.7 Biodegradation of Natural Fibre Composites

Degradation action takes place with the help of enzymes (biological), chemical, thermal and photo (light) sources. Bio degradation takes place with the help of living organisms and the process occurs in two steps [Leja and Lewandowicz 2010]. The first step is to break down the polymers into lower molecular mass species and this takes place with the aid of oxidation or hydrolysis. Second step is by, bio assimilation of polymer fragments by micro organisms. The conditions for biodegradation depend on other factors such as processing characteristics, ageing conditions, storage facilities and chemical compositions [Khardenavis et al. 2007, Tokiwa et al. 2009, Vroman and Tighzert 2009].

Generally in degradation process, cellulose fibres are responsible for the formation of tar like products that creates damage effects both on the processing equipment and the composite properties. Hemi cellulose is responsible for the biodegradation, moisture absorption and thermal degradation of the fibre and it shows least resistance. Where as lignin is thermally stable and are responsible for the ultra violet degradation [Albano et al. 1999, Manfredi et al. 2006]. Some of the degradation and their mechanisms are shown in above Table 2.14.

Table 2.14: Degradation Types and their Mechanisms [Rowell 1998]

Type of Degradation	Sources	Reactions	Mechanisms
Biological Degradation	Bacteria, Fungi, Insects, Termites	Enzymatic Reactions	Hydrolysis Oxidation, Reduction
		Chemical Reactions	Hydrolysis, Oxidation, Reduction
		Mechanical	Chewing
Water Degradation	Rain, Acid Rain, Sea, River	Water Interactions	Shrinking, Swelling, Hydrolysis, Dehydration
Weather Degradation	Wind, Heat, Ultraviolet Radiation	Chemical Reactions	Dehydration, Oxidation, Reduction, Hydrolysis
Mechanical Degradation	Wind, Snow, Sand, Dust,	Mechanical Reactions	Cracks, Stress, Fracture, Abrasion

Degradation can be from several types [Andrady and Marine 2011]

1. Biodegradation- action from living organisms
2. Thermal degradation- action from high temperatures
3. Thermo oxidative degradation- slow oxidation with moderate temperatures

4. Photo degradation- action from light sources
5. Hydrolysis- break down reaction with water

2.8 Ecological Impact of Bio-composites

Ecological aspect of the material should be remembered as the first step in fabricating a composite material. For obtaining an environment friendly material minimum roles should be performed, which are shown below.

- Must be free from damage and protect from moisture, dust and other contaminant factors
- Design factors such as shape, size and weight should be considered
- Disposal of the material must be bio degradable
- The material should be feasible, flexible and recycled

Agro waste and industrial waste can be re used after recycling. There is a tremendous potential for these waste and are used for various applications as shown in Table 2.15 for structural and building applications. The applications of these recycled parts are processed in the same manner as that of the original one. Every life cycle of the material consumes certain amount of energy, produce emissions and solid waste and the produced solid waste should be recycled and re used. Some associations in European countries such as European Hemp Association (EIHA) welcome and supports bio based products.

Depending upon the structure of the material, recycling are been carried out. In general, for a polymer composite material recycling involves mechanical recycling, chemical recycling and Incineration (energy recovery). Mechanical recycling involves the process of shredding and granulation of waste composite material. Chemical recycling involves breaking the polymer matrix waste into hydrocarbon that is reused for other polymers and chemicals. Incineration involves in reducing the amount of waste that goes to the landfill [Morris, 1996; Allred, et al. 1997].

Table 2.15: Types and Nature of Solid Wastes and their Recycling and Utilization Potentials [Pappu, et al. 2007]

Type of solid wastes	Source Details	Recycling and utilization in structural building application
Agro wastes	Rice and wheat straw and husk, cotton stalk, ground nut shell, baggage, banana stalk, jute, sisal and other vegetable residues	Particle boards, insulation boards, wall panels, roofing sheets, fibrous building panels, bricks, coir fibre, reinforced composite, cement board
Industrial wastes	Coal combustion residues, steel slag, construction debris	Cement, bricks, blocks, concrete, wood substitute products, ceramic products

The different stages of a life cycle for a natural plant fibre composites are the agricultural stage consisting of cultivating the plants for fibres, followed by pre-processing stage which involves the preparation of fibres, decortication of fibres, cleaning the fibres, extraction and processing the fibres. The next stage is the processing stage, which involves the preparation of natural fibre reinforced polymer composite material and also termed as bio composite material. Processing of natural fibre composites remains the same procedure to that of the conventional composite material. The next stage is the consumption stage, where the users and the usage of the fibres are been done. The final level is the treatment period which involves the re-use, re-cycle, land fill and incineration of the material.

2.9 Discussion on Review and Justification of the Work

From the literature review, it has been observed that the natural fibres are potential to replace traditional fibres in composites in many applications. The physical, chemical, mechanical and thermal properties of fibres and composites revealed more information regarding the behaviour of fibre with the matrix, strength and modulus, crystallinity of the fibre, and thermal degradation of the fibre composite. Mechanical method of extracting the fibres from the plant was easier than the chemical method and the large quantity of fibres are obtained in easier and and faster way. Chemical treatments of fibres have been carried out by many researchers and were shown that it provides good interfacial adhesion between fibres and matrices. Majority of the applications of natural fibres are focussed on automotive and structural sector. Interior applications of natural fibre composites are also focussed and comparatively use of these composites are more than that of exterior applications. Swelling of fibres, moisture absorption and surface degradation are some of the major problems concerned in natural fibre composites. Natural fibre composites are termed to be completely green if the matrix is bio polymer and the derived bio-composite is fully biodegradable. In the present ecological world, natural fibres play an important role in composites and their applications.

From the literature survey it has been confirmed that only a moderate amount of work is focused on natural fibres but no work is reported on maize and hybrid composites (maize and jute) in manufacturing, characterization of natural fibre composites and applications in structural beams. Maize fibres are available at low cost and availability of these fibres is abundant in south India. Most of the top portions of maize stalks are used as a fodder to domesticated animals and remaining portion of the plant are left in farm field with no use. Use of these waste stalks for extraction fibres is appreciated and also helps in disposal of major portion of maize stalk. Much of the literatures were focussed on maize cob and not on the use of stalk part of the plant. Sandwiched hybrid composites with jute fabric and maize fibres are new to the context. Most of the reviewed literature indicated that only a limited work had been carried on processing natural maize fibre composites. In the present global warming era, use of natural fibres for composites is appreciated and to make a clean, green earth.

2.9.1 Why the present work is Unique.

The present work is focused on processing maize fibre composites, biocomposite and hybrid compositse by advanced processing method of Vacuum Assisted Resin

Transfer Molding (VARTM). Different characterization methods were employed for studying the properties of the obtained material. The obtained experimental results of thermal conductivity of maize fibre composites were compared to the finite element method and with suitable analytical model. The various recent works on maize fibre composites performed by various researchers are summarized. As natural plant fibre properties vary the results to region and place of growth and it also has influence on environmental and climatic factors. The present maize fibre is a low cost fibre in this particular region and only little work was focused on studying the properties of this fibre, hence this fibre was choosen.

Maize stalk fibres as fillers in natural rubber composites were developed by Chigondo et al. 2013. The fibres were modified and characterized with FTIR technique. The results showed that treated fibres had high mechanical property and reduced moisture absorption. These natural fibres have the potential to reinforce natural rubber for shoe sole production. Trigui et al. 2012 worked on the thermal properties of maize fibre reinforced with high density polyethylene composites. The composite samples with different fibre contents in the range 10- 40% were choosed. Thermal and mechanical properties were studied and found that the thermal conductivity and diffusivity of the composites decreased with fibre loading. It was also mentioned that good mechanical performance in the composite can be obtained if there is good stress transfer at fibre matrix interface. Kumar et al. 2011 developed composites with zea mays fibre with different fibre weight percentage using hand lay-up method. The fibres were treated with alkali method and mechanical properties for the composite are studied. It showed an increase in property with fibre percentage of 40% and later decreased in properties were observed for high fibre content weight. Characterization of the composites was carried out to evaluate the fibre/ matrix interactions. Dauda et al. 2007 worked on achieving a green composite material consisting of maize fibres and poly e -caprolactone matrix. Sequential molding was employed for achieving the composite material. Tensile strength and modulus of the fibre was evaluated. The tensile strength of the composite increased in increasing the fibre area fraction and it was achieved till 55 % fibre area fraction. The processing factors were optimizied and studied.

CHAPTER 3

EXPERIMENTAL WORK IN PROCESSING OF COMPOSITES

The aim of the experimental work is carried out for processing composites using maize and jute fibres in a matrix of epoxy thermoset polymer through Vacuum Assisted Resin Transfer Molding (VARTM) and hand lay-up technique for sandwiched hybrid composites. Natural fibre composites using maize fibres in matrix are prepared and a layer of jute fabric was stacked to develop hybrid and sandwiched hybrid composites. Surface characterization, thermal and mechanical tests were carried out for the performance of natural fibre composites as shown in Figure 3.1.

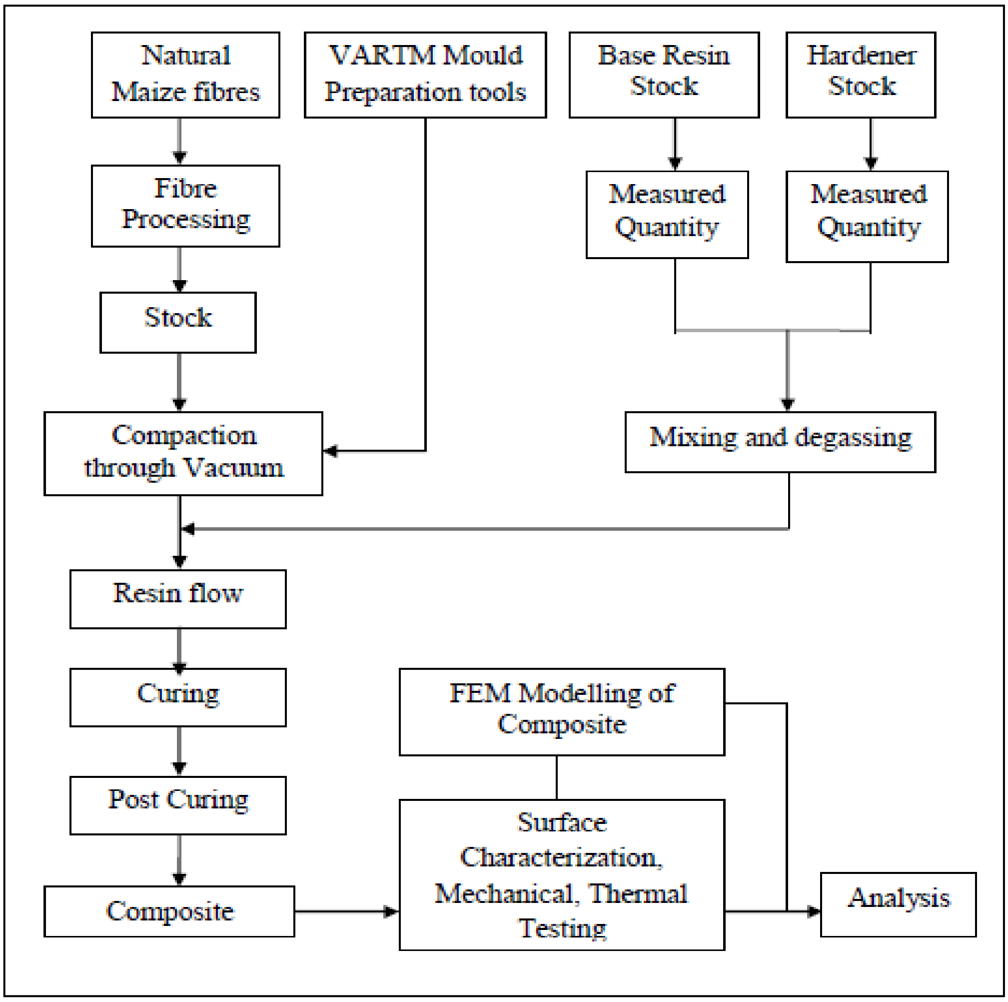


Figure 3.1: Processing of Composites Flow Chart

Natural maize fibres are obtained from dried lower portion of maize stalks. These fibres are chemically treated and used to prepare composites with different weight fractions of fibre and epoxy polymer using VARTM to process maize composites. Hybrid composites were prepared by having maize fibres piled with jute fabric and processed through VARTM. Work is also extended in processing a complete bio composite material by choosing maize fibre in expoxidized soya bean oil processed by VARTM technique. Sandwiched hybrid composites are developed with maize fibres (pulverized) sandwiched between two layers of jute fabric. The following sections explain the method carried out for processing of composites.

3.1 Processing of Maize Fibre Composite

Maize stalks were obtained from a local farm in Davangere, Karnataka (India). The stalks after removing soft pith, were thoroughly cleaned with running water and sun-dried in an open sun for a week. The pulverized raw fibres were chemically treated, initially with alkaline solution and later with the acetylated method. Chemical treatments were carried in order to increase the affinity between fibres and the matrix, and also to reduce the water absorptivity. Further, these fibres were kept in an oven for 60 °C for 7 hours to remove moisture and are used in this work.

3.1.1 Plant Stalk Fibre – Maize

Maize - is a well known crop in most countries in the world. It had its origin from Mesoamerica and southern Mexico and later spread over America and rest of the world. It is a well known food crop cultivated throughout the world and well familiar use in Unites States of America, China, Mexico, India, Brazil, France and South Africa [Erenstein 2010]. In Indian context, food crops such as maize, sorghum, millet, barley are the chief coarse grain and grown in any seasons such as in Kharif season or Rabi season, also well grown in variety of soils. Corn stalks, like other agricultural fibre sources, consist of a pithy core with an outer layer of long fibres. Green maize plant in a farm is shown in Figure 3.2. The dried maize stalks are cleaned, washed and chopped into a convenient size as shown in Figure 3.3 (a to d) to obtain 300 µm particle size (using mesh sieve of 300 µ) for the preparation of maize fibres.

Figure 3.2: Maize Plant in a Farm

(a) (b) (c) (d)

Figure 3.3: Maize Stalks

(a, b) Maize Stalk; (c, d) Choppped Maize Stalk

Jute – Jute fibres are produced from plants genus Corchorus, family Malvaceae and considered golden fibre because of long shiny fibres that can spun into ropes, threads, sacks and mat as shown in Figure 3.4. Jute fibre falls in bast fibre category and second most used vegetable fibre after cotton. They are grown mainly in West Bengal in India and Bangladesh (fertile Ganges delta) and find applications in clothing, carpets, packaging and furnishing applications. Jute fibres are 1 to 4 meter long. In the present work, jute fabric is used and purchased from local market.

Jute Plant

Jute Fibre

Jute Fabric

Figure 3.4: Jute Plant Fibre and Fabric

3.1.2 Chemical Treatment of Natural Fibres

Chemical constituents of maize fibre consist of huge amount percentage of hemicelluloses, lignin and ash compounds. Few chemical compounds have to be minimized for effective bonding between fibres and the matrices. Hence they are chemically treated using alkali treatment and acetylation method. Thereby the -OH compound of the fibre is reduced and making the fibre to be hydrophobic.

Chemical treatments of natural fibre are used to strengthen the interface between fibre and matrix or to achieve a good interfacial bond between natural fibres and the polymeric resin [Bledzki and Gassan 1999, Bismarck et al. 2001, Rout et al. 2001, Li et al. 2007]. Various surface treatments are followed such as alkaline treatment, acetylation, silane treatment, benzoylation treatment, coupling agents, graft polymerization, permanganate treatment and peroxide treatment [Li et al. 2007]. Each method of treatment has its own pros and cons. The most familiar, economical and the older method is the alkaline treatment leading to surface cleaning by partially removing secondary and tertiary constituents such as hemicelluloses and lignin [Esmeraldo et al. 2010].

Alkali Treatment

First treatment: Alkali treatment (Mercerization) improves the fibre-matrix adhesion and also helps to break down the fibre bundles in to smaller fibres. It removes some part of the constituents such as hemicelluloses, lignins and wax. Modification leads to disruption of hydrogen bonding in the polymer structure and increasing the roughness of the surface leading to better mechanical properties [Mohanty et al. 2001, Mwaikambo and Ansell 2002, Li et al. 2007, John and Anandjiwala 2008].

Alkaline treatment is one of the most used chemical treatments for natural fibres when used to reinforce thermosets. In the present work, the raw fibres were treated with 5% of NaOH for 6-8 hours, and later washed with distilled water. It results in the disruption of hydrogen bonding in the network structure, thereby increasing surface roughness. This treatment removes a certain amount of lignin, wax and oils covering the external surface of the fibre cell wall, depolymerizes cellulose and exposes the short length crystallites. Addition of aqueous sodium hydroxide (NaOH) to natural fibre promotes the ionization of the hydroxyl group to the alkoxide.

$$\text{Fibre - OH + NaOH} \longrightarrow \text{Fibre - O - Na} + H_2O \qquad (3.1)$$

Thus, alkaline processing directly influences the cellulosic fibril, degree of polymerization and the extraction of lignin and hemi cellulosic compounds.

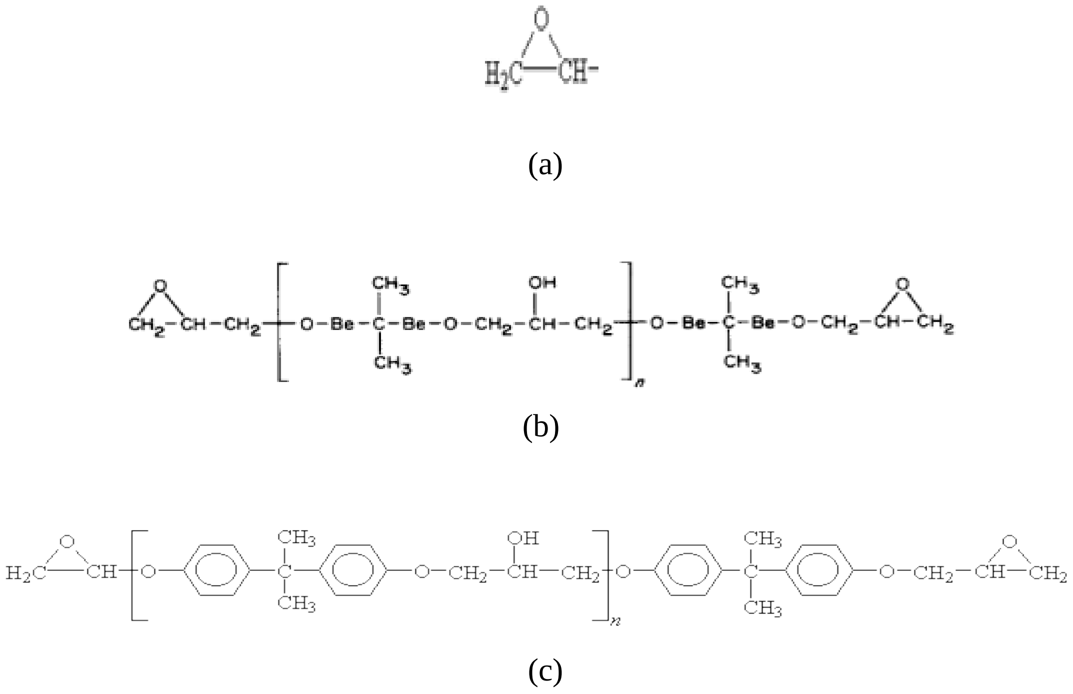
Acetylation Treatment

Second treatment: Acetylation identifies having an acetyl functional group in to an organic compound involving the generation of acetic acid as by product and must be removed from the lignocellulosic material before using the fibre. Alkaline treated fibres were soaked in glacial acetic acid (CH_3COOH) for one hour at room temperature, decanted and again soaked in acetic anhydride containing 1-2 drops of concentrated sulphuric acid (H_2SO_4) for 4-6 minutes, later washed in distilled water and dried in an oven for 40^oC for 24 hours.

$$\text{Fibre- OH} + CH_3CO\text{-}O\text{-}OC\text{-}CH_3 \xrightarrow[\text{Conc.}H_2SO_4]{CH_3COOH} \text{Fibre-O-CO-}CH_3 + CH_3COOH \quad (3.2)$$

3.1.3 Thermoset Epoxy Polymer

Epoxy resins are the most common thermosetting resin polymers and characteristic is their excellent degree of adhesion with good strength, ease of application and good reproducibility. They are characterized by having two or more epoxides group per molecule and the chemical structure of an epoxide group is shown in Figure 3.5 (a) and general chemical structure is shown in Figure 3.5(b). The most common is Diglycidyl Ether of Bisphenol-A (DGEBA) as shown in Figure 3.5(c) which is prepared by the reaction of Epichlorohydrin (ECD) and Bisphenol- A (BPA). ECD is prepared from polypropylene by reacting chlorine with sodium hydroxide. The polymer used for the present work is LAPOX Epoxy Resin and room temperature curing hardener of tetra amine functional group Triethylene tetramine (K6) as shown in Figure 3.5(d). The epoxy resin Lapox resin (L-12) and hardener (K6) is shown in Figure 3.6 are supplied by Atul Polymer Industries, (Valsad -Gujarat) India. These are mixed in the proportion of 10:1 and their densities are shown in Table 3.1.



(a)

(b)

(c)

$NH_2-C_2H_4-NH-C_2H_4-NH-C_2H_4-NH_2$

(d)

Figure 3.5: Chemical Structure of Epoxy Resin

(a) Epoxide group, (b) General chemical structure commercial epoxy, (c) Diglycidyl Ether of Bisphenol-A (DGEBA), (d) Triethylene tetramine (TETA)

Table 3.1: Epoxy Polymer

Trade Name	Chemical Name	Density (kg/m^3)	Parts by weight
Lapox L-12 (Resin)	Diglycidyl Ether of Bisphenol-A (DGEBA)	1120	100
Lapox K6 (Hardener)	Tri ethylene tetro amine (TETA)	954	10

Lapox L-12 (liquid resin) are low temperature curing resin normally between 20-90 ºC with Viscosity (25ºC) of 10000-12000 mPas. Hardener (K6) consisting of Linear Aliphatic Amines are room temperature curing hardeners.

Lapox Resin L-12 Lapox Hardener K-6

Figure 3.6: Epoxy Polymer

3.1.4 Processing of Maize Composites

Maize stalk fibres after chemical treatment were sun dried. These fibres were kept in an oven at around 60ºC for 7 hours and later pulverized in a machine shown in Figure 3.7. Sieve of 300 µm mesh size and maize fibre samples were used in this work shown in Figure 3.8. The maize fibres are now ready to use as a fibre material for processing of composites through Vacuum Assisted Resin Transfer Moulding (VARTM).

Figure 3.7: Maize Pulveriser

(a) (b)

Figure 3.8: Mesh Seive

(a) Seive of 300 µm mesh size, (b) Pulverized Maize samples

3.1.4.1Materials for VARTM

The following items are used for processing of composite by VARTM technique and are shown below and in Figure 3.9. The items are purchased from Vactech Composites Private Limited, Gujarat, India.

a. **Peel Ply** - is a nylon base woven fabric and is placed against the surface. It is porous and weave structure in absorbing excess resin during curing leaving the impression on the surface. The fabric thickness is 0.145 mm with white colour and tracer yarn. The fabric allows for easy removal of the fabricated panel from the mould surface. The porous release material facilitates the resin flow and leaves impression on the part suitable for secondary bonding without surface preparation.

b. **Breather Fabric -** These are synthetic blend (polyester/ nylon) manufactured from combination of fibres which allows good air permeability and absorption property. Its function is to allow air and remove volatiles in the bagging, thus maintaining vacuum throughout the mould in the process. They are also used to absorb excess resin in the system.

c. **Vacuum Bagging Film -** The material is nylon and has excellent elongation and heat ageing characteristics. Bagging film is a cover used to seal the mould surface under vacuum during infusion process. They have excellent physical properties such as toughness, flexibility, high temperature resistance and less permeability. They can perform under high pressure and temperatures up to 190ºC.

d. **Release Film -** The release film is high strength and high elongation with density of 0.963 g/cm^3 and thickness of 27±10% micron. The function is to ensure proper release of the material during vacuum bagging. They also serve to separate the laminate from breather and fabric material and have no release characteristics. Selection of film depends on various factors such as vacuum pressure, cure temperature, resin system and mould component system.

e. **Sealant Tapes -** Sealant tapes are self adhesive and made from butyl compound. They are butyl rubber based with two sided self adhesive sealant tape with good elastic properties.

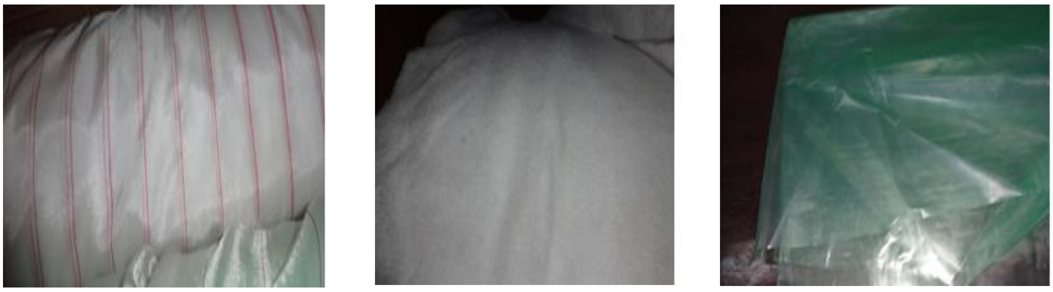
f. **Vacuum Hose -** The material is non toxic and made of high quality PVC. It allows excellent flow characteristic and used for suction and delivery of air from vacuum pump. They are abrasion resistant with much more flexibility and longer life than rubber material.

g. **Spiral Tube -** It is a linear low density polyethylene material used to establish consistent vacuum throughout the mould evenly. They are capable of withstanding negative pressure without collapsing. These are the resin distribution tube and they are laid along the length of stacked fabric lay-up.

Vacuum Trap – Stainless steel vacuum sealed tank trap is placed in between mold and vacuum pump to collect excess resin that enters the vacuum line. Resin trap was purchased from Fibre Glast Developments Corporation, Brookville, U.S.A. The capacity of the trap was approximately 11.5 litres.

Vacuum Pump – Vacuum pump was purchased from HHV Pumps, Bangalore, India. It is a two stage, oil sealed rotary vane vacuum pump, Model ED -15, 0.5 HP, three phase motor. Selection of pump was based on size of the chamber, required vacuum level and time for achieving required vacuum. The pump can be used for medium and high vacuum applications for processing of composites.

Mold Release Agent- It prevents the epoxy resin from sticking it to the mould and facilitates removal of parts from moulds. Frekote 55 NC was used as release agent for vacuum infusion. Frekote -55 NC is a non CFC release agent and are fast drying, semi permanent, non-migratory, multiple release system. They are colourless, solvent based polymer purchased from Henkel-India, Bangalore. Frekote PMC was used to easy removal of parts from the moulds (mould release cleaner) and is highly recommended for preparing mould surfaces before mould sealing and release.



Peel Ply Breather Fabric Vacuum Bagging Film

Sealant tape Vacuum Hose

Figure 3.9: Materials for Processing VARTM of Composites

Spiral Tubing Mold Release Agent

Vacuum trap Vacuum Pump

Figure 3.9: Materials for Processing VARTM of Composites (Continued)

3.1.4.2 VARTM Process

The VARTM (Vacuum Assisted Resin Transfer Moulding) process is a composite fabrication technique by infusing resin into the base materials or fibres using vacuum and shown in Figure 3.10. Compared with the conventional composite fabrication methods, this process is an ideal technique for low cost composite materials without prepregs and autoclaves. In addition, the VARTM is usually an open mold and the fibre preform is placed between the open mold and to the vacuum bag. The process draws resin into a dry reinforcement on vacuum and uses only the partial vacuum to drive the resin and increases the compactness of matrix and fibre content hence reduced void percentage.

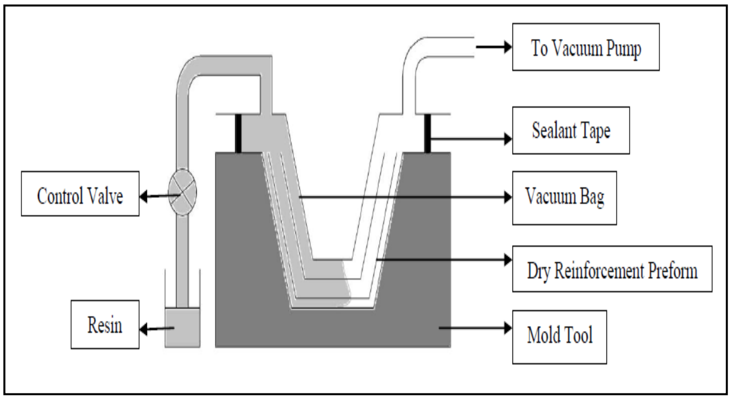


Figure 3.10: Schematic Diagram of VARTM

The main steps of VARTM process are:

1. A dry fabric or preform and accompanying materials such as release films, peel plies are laid on tool surface.
2. The preform is sealed with a vacuum bag and the air is evacuated by a vacuum pump.
3. Liquid resin mixed with hardener from an external reservoir is drawn into the fibre by vacuum.
4. The liquid resin with hardener is infused to the preform until complete impregnation occurs.
5. After Cure and de-molding steps follow to complete the casting process.

The maize fibre pulverized is weighed accurately and the reinforcement is stacked on the mould. The resin inlet is prepared using the distribution spiral at one end of the reinforcement material so that it distributes the amount of resin transferred. The vacuum line is prepared on a layer of peel ply and finally the mould is made air tight using a vacuum bag. Resin mixing and degassing prior to infusion ensures blending of the components and low content of entrapped gases. During the process, the vacuum level is controlled by a regulator. Finally the resin is transfused into the mould completely and Figure 3.11 shows the top view of resin flow. The vacuum source is maintained until the resin system begins to gel and cures. Once the vacuum is turned off, the part is allowed to cure at room temperature. Schematic set-up of VARTM is shown in Figure 3.12. Experimental set-up is shown in Figure 3.13 and 3.14.

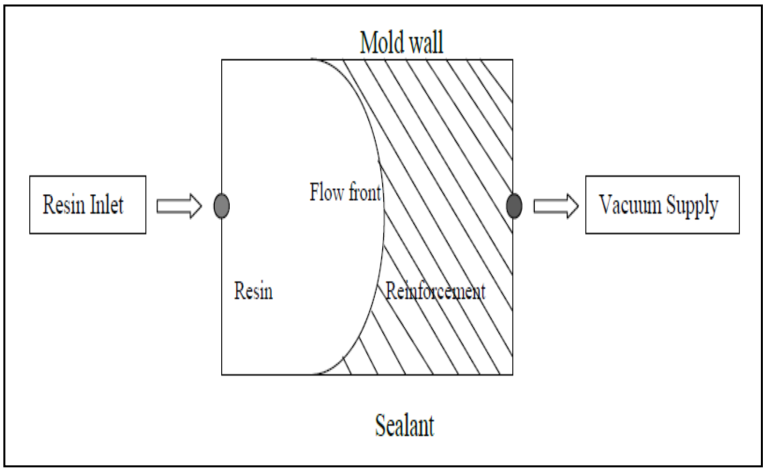


Figure 3.11: Top View Setup of Resin Flow

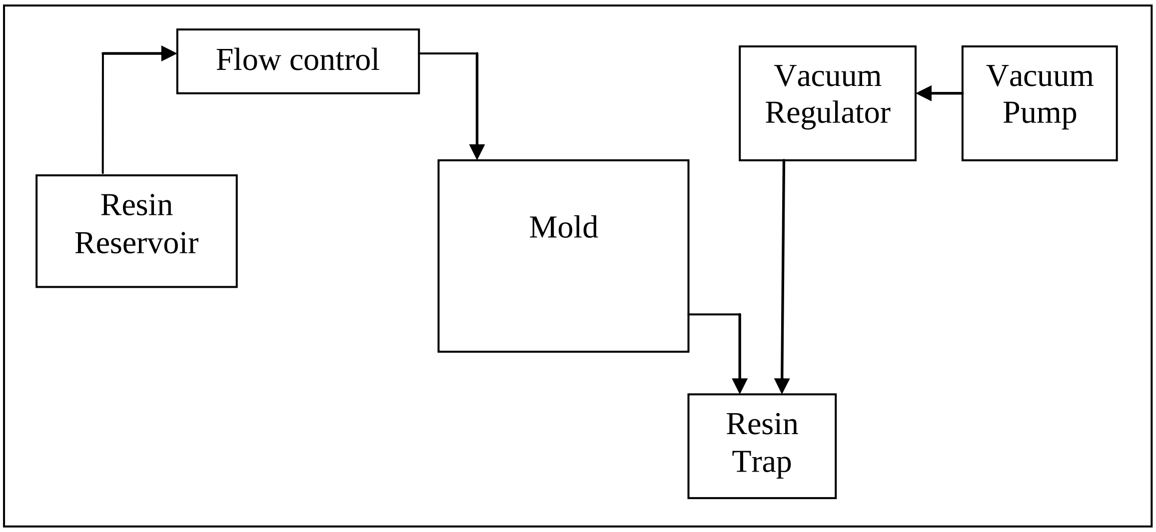


Figure 3.12: Schematic Setup of VARTM Experimental Work

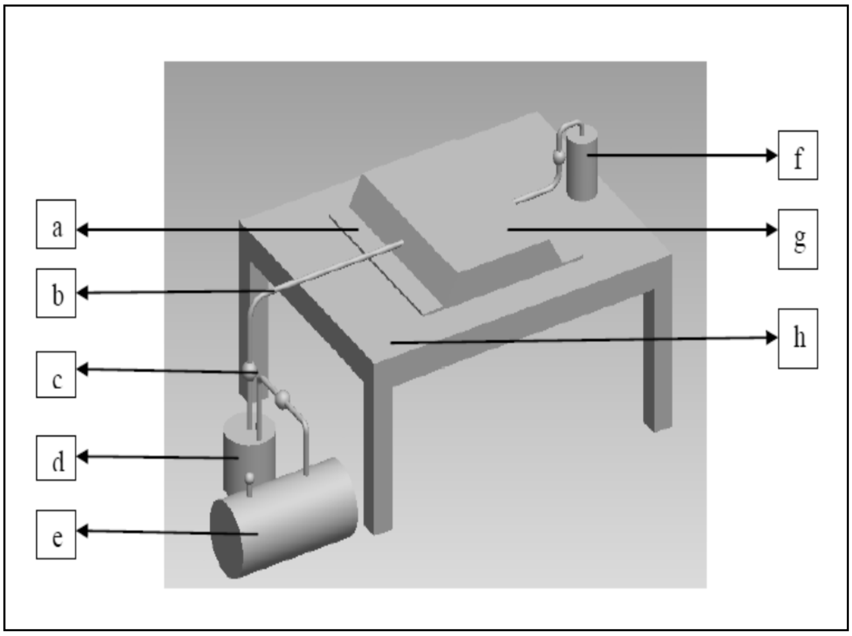


Figure 3.13: Vacuum Assisted Resin Transfer Molding (VARTM)

(a)Vacuum bag, (b) Vacuum side pipe, (c) Resin control valve, (d) Resin trap, (e) Compressor, (f) Polymer matrix Container, (g) Mold, (h) Working table.

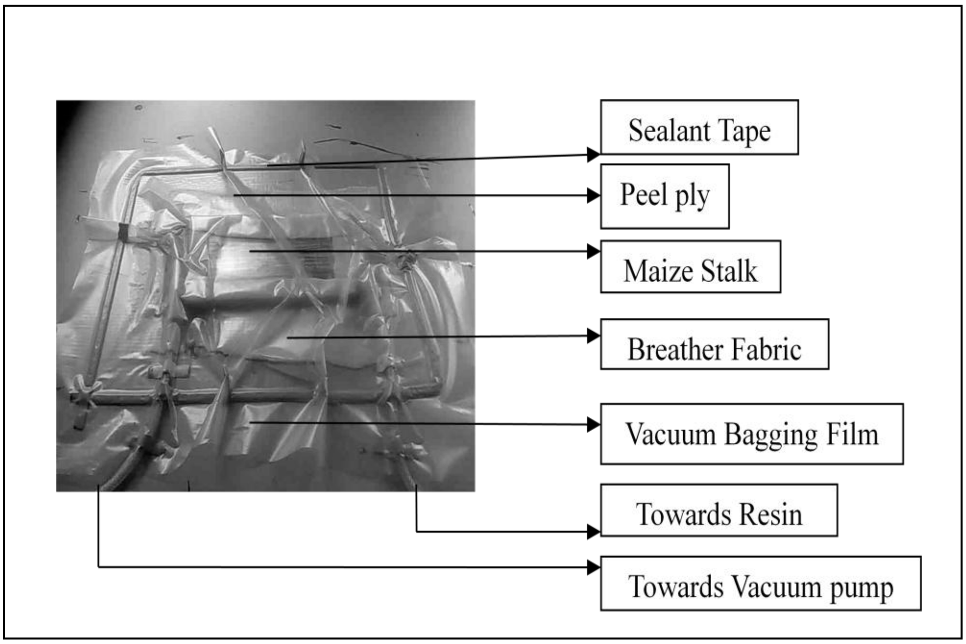


Figure 3.14: VARTM for Processing of Maize Composites

Composite with different weight fractions ranging from 5% to 25% were casted in steps of 5%. Processed maize fibre composite with mould is shown in Figure 3.15. Tensile and flexural specimen as per ASTM standards were cut through water jet machining from the casted composite that was processed by VARTM and are shown in Figure 3.16 and 3.17.

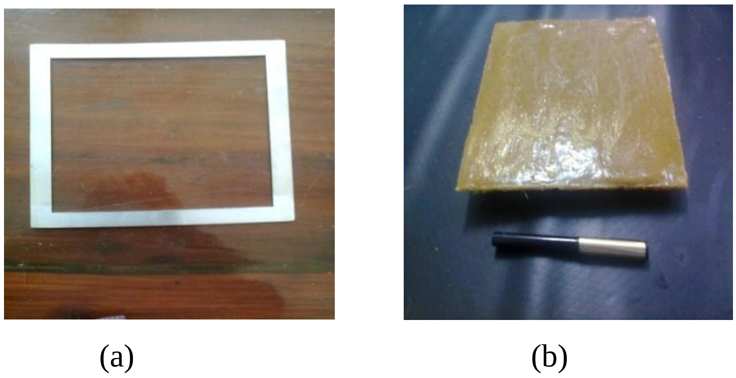

(a) (b)

Figure 3.15: Processed Maize Composites

(a) Mould, (b) Maize fibre composite

Figure 3.16: Tensile Specimens of Maize Fibre Composites

Figure 3.17: Flexural Specimens of Maize Fibre Composites

3.2 Processing of Hybrid Composite Material

Hybrid composite material (Maize and Jute fabric) was processed by means of VARTM and sandwiched hybrid composite by simple hand lay-up method. Natural fibres of maize and jute fabric (single layer) were reinforced with thermoset polymer epoxy resin with appropriate ratio of hardener (as explained in previous section) was used. First layer composed of jute fabric was placed over second layer of maize composite and is shown in Figure 3.18. The processing method was same as that of maize composites processed by VARTM.

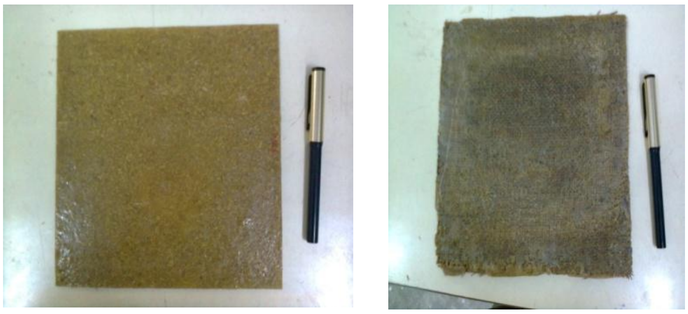


Figure 3.18: Maize-Jute Fibre Hybrid Composite

3.3 Processing of Sandwiched Hybrid Composite Material

3.3.1Materials and Methods

Mould - Moulds were prepared with mild steel with dimensions of 200 x 200 x 3.2 mm with hot chrome plating surfaces.

Mould Release Agent- Poly vinyl alcohol is applied to waxed moulds and it prevent mould from sticking. It is colourless and odourless synthetic polymer and is applied by roller for coating the mould as shown Figure 3.19. Processing of sandwiched hybrid composites was carried out by Simple traditional hand lay-up method. A sandwiched hybrid composite was developed with maize pulverized fibres cores and outer layers with jute fabric.


Roller Release Agent

Figure 3.19: Materials for Hand Lay-Up Process

3.3.2 Hand Layup Method

Hand lay-up is the oldest and simplest method of manufacturing composites. The tools required for the process are mould to accommodate manufacturing according to the desired shape and a roller to facilitate uniform distribution of resin. Virtually any sized composites can be manufactured using this method and this method is the cheapest method of manufacturing, ideal for lower volumes, lower cost to tool, and flexibility. But it has some disadvantages such as long curing time, low production rate, and further the quality of the composite depends on the skill of the worker.

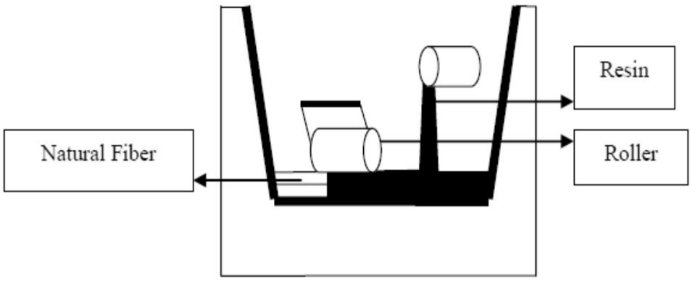


Figure 3.20: Hand Lay-up

Simple hand layup method for processing of sandwiched hybrid composite material (Maize and Jute Fabric) was practiced as shown in Figure 3.20. Poly vinyl alcohol release agent was applied to the open mold. Chemically treated maize stalk fibres and jute fabric were kept in an oven at 60°C for 7 hours and then used for the work. Thermoset polymer of epoxy resin with hardeners was used (as discussed earlier) with a ratio of proportion 10:1. Layers of jute fabric (jute fabric was placed above and below of the maize fibres and are sandwiched them). Totally, the material had three layers, upper and lower layer consist of jute fabric and middle layer consist of maize pulverized fibres as shown in Figure 3.21 The sandwiched hybrid composite are developed with different maize fibre ratios 5% to 25% (interval of 5%) with layers of jute fabric with epoxy resin in mould and were allowed to cure. Tensile and Flexural specimens were cut from the casted component for further testing and characterization work as shown in Figure 3.22 and 3.23.

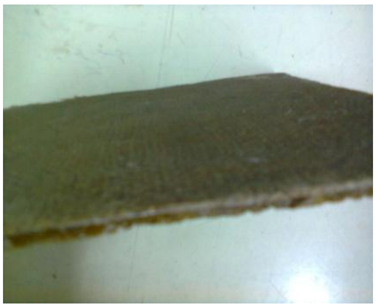


Figure 3.21: Sandwiched Hybrid Composite

Figure 3.22: Tensile Specimen of Sandwiched Hybrid Composite

Figure 3.23: Flexural Specimen of Sandwiched Hybrid Composite

3.4 Processing a Bio-Composite Material

In the present work, maize fibres are the reinforcement material and Epoxidized soybean oil (ESBO/ESO) was the matrix material. Materials used are maize fibres which are brought from a local farm in Davangere, Karnataka, and biopolymer of epoxidized soybean oil from Aldrich Chemicals, Bangalore. Mould was fabricated as explained in early sections. The mild steel mould plate with 200 x 200 x 3.2 mm and hot chromed is shown in Figure 3.24 and is coated with Frekote 55- NC and Frekote PMC (mould release agent).

3.4.1 Biopolymer of Epoxidized Soyabean Oil (ESBO)

Epoxidized soybean oil is a plasticizer and manufactured from soybean oil through the process of epoxidation. Biopolymer of ESBO, styrene, Luperox A-75 Benzoyl Peroxide, N,N Dimethylaniline (curing agent) was purchased from Sigma- Aldrich Chemicals Pvt. Ltd., Bangalore, India.

Mould for Casting Bio-Composite

Soybean Biopolymer Curing Agents

Figure 3.24: Materials for Processing Bio-Composites

3.4.2 Bio-Composites by VARTM

Maize fibres are cleaned thoroughly in running water and chemically treated by alkali method as explained earlier. The fibres were placed in an oven at 60° C for 7 hours before use. The bio polymer soybean matrix is mixed with styrene, Luperox A-75 Benzoyl Peroxide, N, N Dimethylaniline (curing agents). The resins are thoroughly mixed and processed by VARTM technique (as explained previous sections) to produce a composite.

3.5 Summary on Experimental work

Experimental work in processing of composites with fibres and matrix are summarized in Table 3.2. The obtained composites are maize composites, hybrid composites, sandwiched hybrid composites and bio-composites.

Table 3.2: Maize Fibre and Matrix Developed Composite Material

Sl No.	Composite Material	Natural Fibre	Processing Method	Matrix
1	Maize Composite Material	Maize fibre	VARTM	Synthetic Polymer (Thermoset)
2	Hybrid Composite Material	Maize fibre + Jute fabric		
3	Sandwiched Hybrid Composite Material		Hand Lay-up	
4	Bio- Composite Material	Maize fibre	VARTM	Biopolymer (Epoxidized Soy bean oil)

(a) Maize Composite

VARTM processing of composite material leads to casting of composite material those are free of voids and less tooling cost. Manual mixing of maize fibres and resin was a difficult task, because it leads to trapping of air bubbles. These voids leads to low strength and can be easily affected by water absorption, thus change in mechanical properties and uneven swelling characteristics. Hence, degassing of resin was done so that the voids are minimised, this was carried out with the help of vacuum pump and desiccator. The excess resin was collected at the resin trap. The above problems are minimized by appropriate guidance, practice and by experience. If the processing work gets practised by the user, then fabrication of composites by vacuum infusion is easier and it takes less time with minimum wastage of resin. The obtained material was free from entrapped voids and bubbles. Maize fibre composites are developed with different fibre weight ratios 5% - 25% with an interval of 5 %.

(b) Hybrid Composite

VARTM technique was used for fabricating hybrid composite material consisting of maize fibre and jute fabric. The fabrication pattern followed was same as that of the method discussed above (Maize composite). But, the obtained material was hybrid composite material with Maize fibres and jute fabric (skin and core type). The composite materials are developed with different fibre ratios 5% - 25% with an interval of 5 %.

(c) Sandwiched Hybrid Composite

Hand lay-up method was the easiest way of processing a composite material and the materials cost is low compared to other processing techniques. Void content of maize composites decreased with jute fabric and the material had better fibre matrix adhesion because of chemical treatment of fibres. The material had high density property because of additional jute fibre and the jute fibre had good wetting with epoxy resin. The obtained material was sandwiched hybrid composite of maize fibre between jute fabric (sandwiched material) with different fibre weight ratios of 5% - 25% with an interval of 5%. Quality processing on hybrid composites was mainly dependent on type of fibre, fibre content, orientation of fibres, intermingling and size of fibres and adhesion between fibres to matrix. Bonding between fibres and matrix is essential to attain better mechanical properties and these are carried out with the help of chemical treatment of fibres.

(d) **Maize Fibre Bio -Composite**

The material is a fully biodegradable, green composite, maize fibre reinforced biopolymer material. The viscosity of the biopolymers was high as compared to synthetic epoxy resin and there was a bit difficult in vacuum infusion but at different trials it was made easy. The material processed was 15 % weight fraction of maize fibres with ESO biopolymer matrix. The material was free of voids and infusion was carried at atmospheric pressure and allowed to cure in the mould.

The findings in the present work and the novelty of the work carried out are discussed above. However the crticial findings are significant improvement was achieved in processing of composites by vacuum assisted resin transfer molding method (VARTM). This method uses little amount of resin matrix as compared to other processing methods. The finished product had good look and it used little amount of time for processing composites. Different Product Material Obtained from Maize Fibres is shown in Appendix II.

CHAPTER 4

PROPERTIES OF MAIZE FIBRE AND THEIR COMPOSITES

The aim of the chapter is to focus the performance of various tests like physical, chemical, mechanical, morphological and thermal tests for determining the properties of maize fibres, maize composites, hybrid composites (maize and jute fabric with single layer) and sandwiched hybrid composites (maize fibres sandwiched between two jute fabric layers). The following sections explain the detailed information for each test and the results obtained.

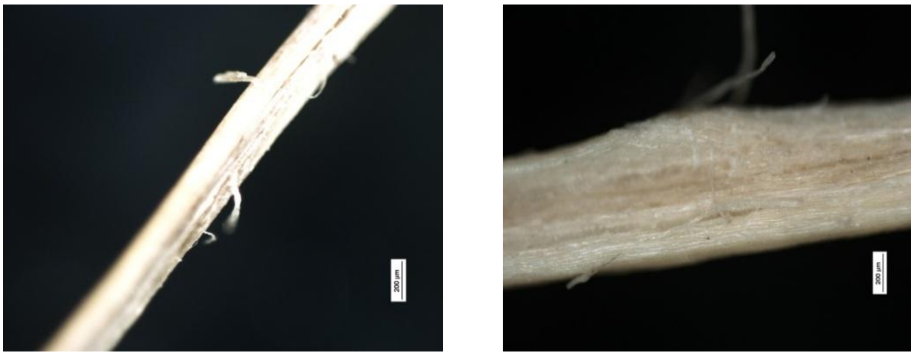
4.1 Properties for Maize Fibres

The properties of maize fibres are studied through various tests such as physical, chemical, mechanical, morphology and thermal tests. The important factors are discussed in the following sections. Physical properties of maize fibre such as length, width and density were determined and reported. These parameters are very important for the study of maize fibre composites.

4.1.1 Physical Properties of Maize Fibres

All maize stalk fibre samples were selected at the bottom portion of the plant. The fibres are extracted by removing soft pith manually from maize stalk they are free from any bacterial/fungal diseases. Healthy stalk fibres were pulverized and samples were measured for length, width and density. The specification of fibres was determined from the optical microscope images as shown in Figure 4.1(a). The variation of thickness of maize fibres is shown in Figure 4.1(b). The thickness of the fibre varies at different location over the fibre length. The average thickness of fibre was 40.7 µm and length of fibre was less than 300 µm. Fibre size may vary with nature of growth and location of the fibres in a plant. Surface image of maize fibre is shown in Figure 4.2 and they clearly shows the unidirectional fibres arranged in a systematic manner along the length.

(a) (Avg 40.7 µm)



(b)Variation of thickness of fibre

Figure 4.1: Photomicrograph of Maize fibres

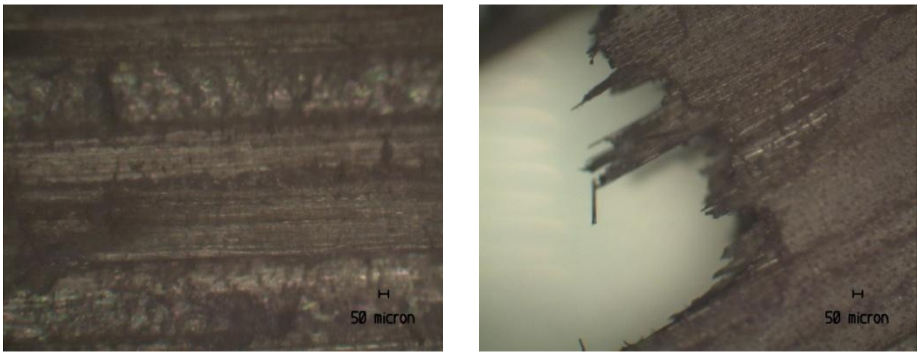


Figure 4.2: Surface Image of Maize Fibre

Density of fibre was determined by Pcynometry. The method consists of gravimetric measurements of fibre placed in a small bottle called pycnometer. Maize fibres were dried in oven and were tested for density measurements. Water was used as a displacement medium.

Density is measured as mass per unit volume

$$\rho = {}^{m}/_{V} \tag{4.1}$$

The density of the maize fibres was 0.44 (g/cm^3)

4.1.2 Chemical Properties of Maize Fibres

Elemental analysis of maize fibre was performed by using Pyrolysis method. This analysis of maize fibre was carried at Central Power Reserach Institute (CPRI), Bangalore, India according to ASTM D 5373-02. The analysis was carried out by using Automatic Carbon-Hydrogen-Nitrogen (CHN) analyzer, (TGA-601 LECO, USA) and is shown in Figure 4.3. The results of various element compositions in terms of weight percentage are determined as shown in the Table 4.1.

Table 4.1: Elemental Analysis of Maize Stalk

Carbon (wt.%)	Hydrogen (wt.%)	Oxygen (wt.%)	Nitrogen (wt.%)	Sulphur (wt.%)
45.8	6.11	42.1	1.99	0.10

Figure 4.3: Automatic Carbon-Hydrogen-Nitrogen (CHN) Analyzer

(Courtesy: CPRI, Bangalore)

Proximate analysis of Maize fibre was carried at Central Power Reserach Institute (CPRI), Bangalore, India according to ASTM D 5142-02A. The analysis was carried out by automatic proximate analyzer, (TGA-601 LECO, USA) as shown in Figure 4.4. The weight fractions of moisture, ash, volatile content and fixed carbon contents are determined as shown in Table 4.2. Chemical compositions of the maize fibres are choosen from the recent literature survey as shown in Table 4.3 and they showed rich content of cellulose when compared to hemicellulose and lignin.

Table 4.2: Proximate Analysis of Maize Stalk

Moisture (wt.%)	Ash Content (wt.%)	Volatile Matter (wt.%)	Fixed Carbon (wt.%)
6.2	5.0	70.7	11.1

Figure 4.4: Automatic Proximate Analyzer

(Courtesy: CPRI, Bangalore)

Table 4.3: Chemical Compositions of Maize Natural Fibres

Fibre	Cellulose (%)	Lignin (%)	Hemicellulose (%)	Pentosans (%)	Ash (%)	References
Maize	38-40	7-21	28	-	3.6-7.0	Reddy and Yang 2004
	40.28	19.35	-	35.06	-	Valchev et al. 2009
	38-42	10-13	21-23	-	-	Kumar 2008

4.1.3 Mechanical Properties for Maize Fibres

Single fibre tensile test and single fibre fragmentation test are widely used for studying fibre-matrix interfacial studies. In this work single fibre tensile test and single fibre fragmentation test were performed using tensile testing machine. The following sections will explain these tests.

4.1.3.1 Tensile Test of Maize Fibre

Tensile test is a most widely used method for determining the mechanical properties of fibres and fibre - matrix interfacial studies. It is an important indicator of the material's behaviour under load in tension. Tensile test provides useful data such as: tensile yield strength, tensile strength at break, ultimate tensile strength, tensile modulus (Young's modulus), and elongation at yield and break. The test was conducted using Instron 3366 (Universal Tensile Machine) with a load cell of 10kN as shown in Figure 4.5.

The tensile strength (σ) is given by,

$$\sigma = F/ bh \tag{4.2}$$

Where,

F= load (N)
b= width of the sample (mm)
h= thickness of the sample (mm)

Strain or Unit elongation is defined as,

$$\varepsilon = \Delta l / l_o \tag{4.3}$$

Where,

Δl= extension (mm)
$l_{o=}$ initial gauge length (mm)

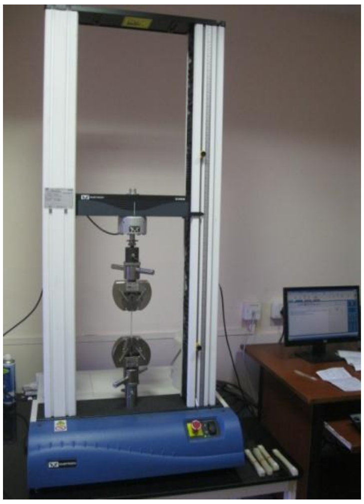
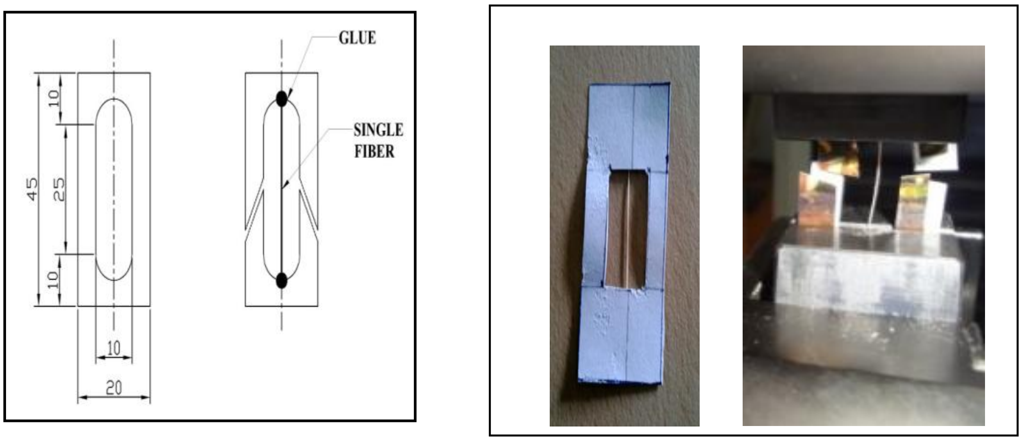


Figure 4.5: Tensile Testing Machine

4.1.3.1.1 Single Fibre Test (Maize Fibre)

Single fibre are taken and glued with both ends to the paper and clamped. There by when the fibre is elongated by tensile test, it shows the strength of the single fibre. A single fibre from maize bundle is selected and carefully placed in a thin paper card with dimensions as shown in Figure 4.6(a). Both ends of the fibre were glued to the card and proper care was taken to make sure that the fibre is placed straight longitudinally in between the card as shown in Figure 4.6(b and c). The paper card with fibre was mounted in Instron tensile machine and subjected to a strain rate of 1 mm/min. The paper card wings were cut on the both ends before the specimen test. Several samples were tested and the average result was plotted.

The load was increased gradually to a maximum value till the fibre fails. The stress and strain data obtained from the experiment was represented in graph as shown in Figure. 4.7. The stress strain relationship is almost linear and failure of the fibre occurs instantly and exhibits brittle failure. The maximum stress was for raw fibre was 296.75MPa and Young's Modulus of 16.16 GPa as shown in Table 4.4. For alkali treated fibre maximum tensile stress was 358.16 MPa and Young's Modulus of 17.04 GPa; for acetylated treated fibre maximum tensile stress was 371.17 MPa and Young's Modulus of 17.21 GPa.



(a) (b) (c)

Figure 4.6: Single Fibre Test Sample

(a) Schematic Representation, (b and c) Experimental Setup of Single Fibre

Table 4.4: Single Fibre Test for Maize Fibres

Fibre	Maximum Tensile Stress (MPa)	Tensile Strain at break (%)	Young’s Modulus (GPa)
Raw	296.75	2.39	16.16
Alkali Treated	358.16	2.27	17.04
Acetylated Treated	371.17	2.05	17.21

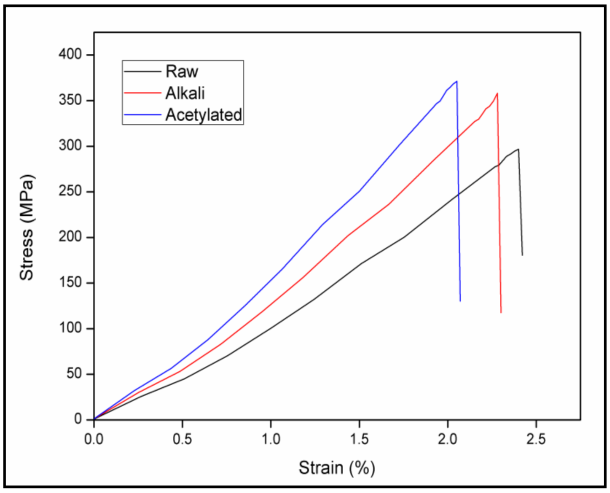
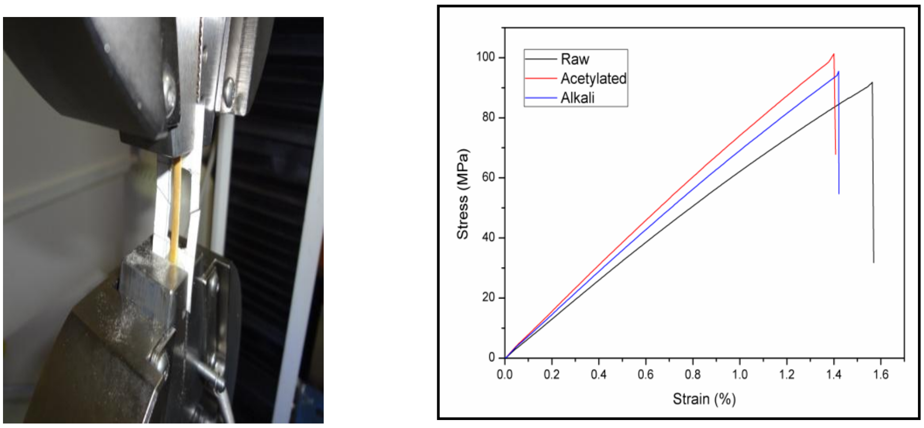


Figure 4.7: Stress Strain Curve for Single Maize Fibres

4.1.3.1.2 Bundle Fibre Test (Maize Fibre)

Bundle fibre test is the collectively fibre test. Determination of bundle fibre has greater importance than single fibre strength test and it gives the strength of fibres in bundle and its behaviour. Bundle fibre has high amount of lignin-pectin matrix with cellulose content and fibre rupture and modulus values can be known for a group of elementary fibres. The similar tensile test was carried for bundle of maize fibres and the test method was same as explained in the previous section. Figure 4.8(a) shows the working of fibre bundle in Universal Testing Machine. The tensile test was carried by increasing load gradually till the fibre bundle fails. The stress strain values are plotted in the graph as shown in Figure 4.8(b).



(a) (b)

Figure 4.8: Stress Strain Curve for Maize Fibre Bundle

(a) Fibre Bundle Test Specimen, (b) Stress Strain Curve for Maize Fibre Bundle

Table 4.5: Maize Fibre Bundle Test

Maize Fibre	Maximum Tensile Stress (MPa)	Tensile Strain at break (%)	Young’s Modulus (GPa)
Raw maize	91.86	1.56	6.38
Alkali Treated	95.44	1.42	6.46
Acetylated Treated	101.27	1.40	6.48

In the graph, the curve tends to vary the path because of some slippage occurring between the outer and inner fibre and with the fixture. The maximum tensile stress for raw fibre was 91.86 MPa and Young’s modulus was 6.38 GPa as shown in Table 4.5. The value of Youngs modulus is very less compared to single maize fibre. For alkali treated fibre the maximum tensile stress was 95.44 MPa and Young’s modulus was 6.46 GPa, where as for acetylated treated fibre the maximum tensile stress was 101.27 MPa and Young’s modulus was 6.48 GPa.

4.1.3.1.3 Single Fibre Fragmentation Test

Single Fibre Fragmentation Test (SFFT) is the most widely used test for studying the interfacial, surface characteristics of a composite material and are used to characterize fibre-matrix interface. In fragmentation test, specimen is dog-bone shape, as the specimen is elongated the fibre inside the resin breaks. It results in matrix cracking or fibre-matrix debonding. Load is transferred from matrix to fibre through interfacial shear. As the fibre breaks, the tensile stress at the fracture location reduces to zero. They are carried out to determine the fibre strength and adhesion.

A schematic model for single fibre fragmentation test is shown in Figure 4.9. The test was carried out by Instron tensile machine 3366 with a load cell of 10kN with cross head speed of 1 mm/min. The single maize fibre is placed in an epoxy matrix material as shown in the Figure 4.10. The sample is subjected to tensile test as a result; the tensile stress is transferred to the matrix and fibre by means of interfacial shear stress. The load was applied gradually at constant strain rate till the specimens fails.

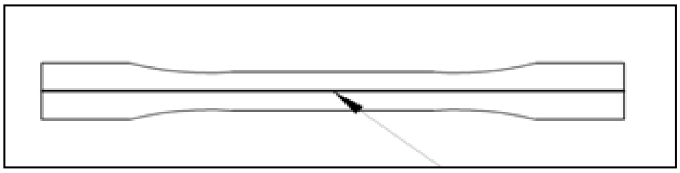


Figure 4.9: Schematic Model for SFFT

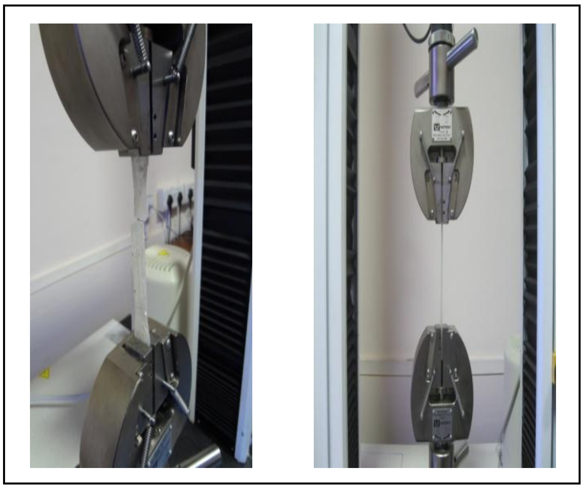


Figure 4.10: SFFT for Maize Fibre Composite

A typical stress versus strain SFFT for maize fibre composites is shown in Figure 4.11. Ten samples were considered for the test and the values of stress and strain were plotted. The strength of the maize fibre epoxy specimen increases with strain till it reaches its ultimate value and then fails. The maximum tensile stress for raw fibre was 14.17 MPa and Young's modulus was 1.27 GPa as shown in Table 4.6. The same test was conducted for alkali treated maize fibre and the maximum tensile stress was 20.14 MPa and Young's modulus of 1.38 GPa. Whereas for acetylated treated maize fibre and the maximum tensile stress was 20.93 MPa and Young's modulus of 1.39 GPa. The fractured surface of the single fibre fragmentation test is discussed in section 4.2.3.2.6.

Table 4.6: SFFT Test for Maize Composites

Maize Fibre	Maximum Tensile Stress (MPa)	Young's Modulus (GPa)
Raw maize	14.17	1.27
Alkali Treated	20.14	1.38
Acetylated Treated	20.93	1.39

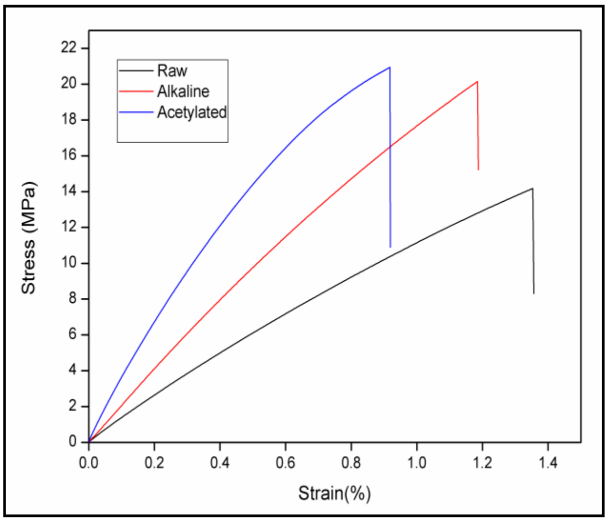


Figure 4.11: Stress Strain Curve for SFFT for Maize Fibres

The mechanical performance of fibres is dependent upon its chemical composition, structure and cellular arrangement. Each individual fibre was composed of fibrils held together by non-cellulosic substances, such as lignin and pectin. Failure of the fibre was gradual upon the application of tensile stress. The result showed the intermediate behavior between brittle and amorphous. As stress gradually increased, some of the fibrils may have slipped out. The total of the stress was then sheared by fewer cells. Further increase of stress led to the rupture of cell walls and decohesion of cells. This resulted in a catastrophic failure of the fibre.

In Single fibre tensile test, the maximum tensile stress for raw fibre was 296.75 MPa and is more than that of the fibre bundle (91.86 MPa). In general, single fibre exhibit always higher value of stress value when compared to the bundle fibre. In fibre fragmentation test, the stress is transferred from fibre to the matrix by means of interfacial shear stress. When the load is increased the fibres tend to fail and cause the fracture of the specimen or when the load is applied continually the fibres in the region can’t withstand the load and fragmentation process occurs, and the sample is no longer sufficient to promote for fractures to occur.

The obtained results were compared to other fibres and are briefed out. The strength of single hemp fibre was in the range of 1695 MPa (15µm diameter) to 2767 MPa (10µm diameter) as reported in Fan 2010. Whereas, in jute fibres, the highest tensile strength value was 306 MPa and Youngs modulus was 38.5 GPa [Fidelis et al. 2013]. In case of sisal fibre, the average tensile strength was 234.30 MPa [Torres and Cubillas, 2005]. For banana fibre tensile strength was 54 MPa and Youngs modulus of 3.5GPa, where as for sisal fibre, the tensile strength is 68MPa and Youngs modulus of 3.8 GPa [Kumar et al. 2014].

Table 4.7 gives the comparison detail of jute fibre to maize fibres and it is shown that maize fibres has less values (mechanical properties) when compared to jute fibres and the density value is also low. Jute values are obtained from Jawaid et al. 2010. The physical and mechanical properties of maize fibres which were performed in this work are tabulated and presented. The comparison shows the physical, chemical composition (cellulose, hemicellulose and lignin content) and mechanical properties. All the values of maize fibres are less than that of jute fibres.

Table 4.7: Physical, Chemical Composition and Mechanical Properties of Jute and Maize Fibre

Fibre	Density (g/cm^3)	Mechanical Property			Chemical Constituents		
		Tensile Strength (MPa)	Youngs Modulus (GPa)	Elongation at break (%)	Cellulose Content (%)	Hemi Cellulose Content (%)	Lignin Content (%)
Jute *	1.3	393-773	26.5	1.5-1.8	58-63	20-24	12-14
Maize Stalk (raw)	0.44	296.75	16.16	2.39	38-42 #	21-23 #	10-13#

* Obtained from Jawaid et al 2010.

Obtained from Reddy and Yang 2004, Kumar 2008

4.1.4 Characterization Analysis for Maize Fibres

The Maize fibre characterization was performed using Morphological analysis by Scanning Electron Microscope (SEM), Fourier Transform Infrared (FTIR) analysis by Fourier Transform Infrared Spectroscopy and X-Ray Diffraction (XRD) analysis by X-Ray Diffractometer.

4.1.4.1 Morphological Analysis

Morphological analysis of raw maize fibre and chemical treated maize fibre was observed by Scanning Electron Microscope (SEM). The morphology changes were analysed and observed using SEM JEOL, JSM-6380LA with an accelerating voltage of 15 kV as shown in Figure 4.12. Maize fibres samples were thoroughly, air-dried and are sputtered with gold coating in JEOL, JFC 1600 (auto fine coater), Japan as shown in Figure 4.13 to avert charge accumulation and later placed in scanning electron microscope for studying the morphological characteristics.

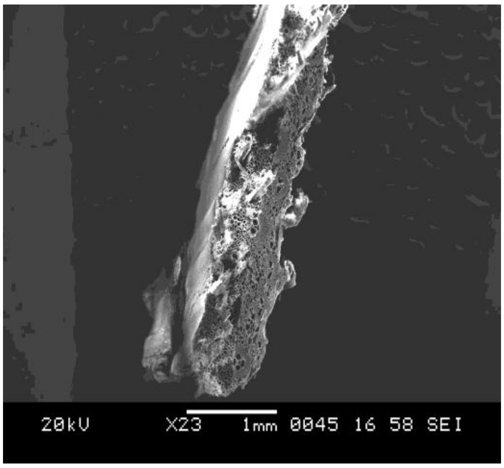
Figure 4.12: Scanning Electron Microscope

Figure 4.13: Sputtering Machine

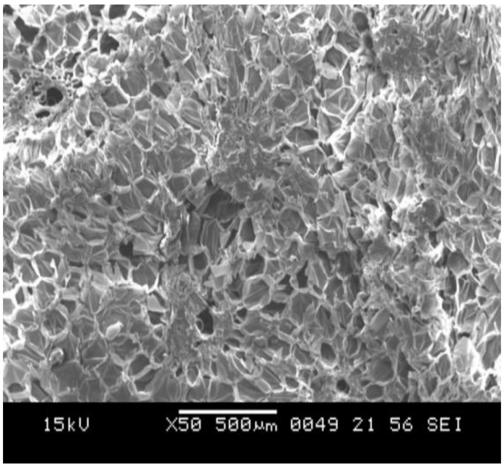
(i) Raw Fibre

The untreated maize fibres were sputtered and observed in Scanning Electron Microscope (SEM). The SEM micrographs samples revealed the information about the morphology or structure of the fibre. Figure 4.14(a, b, c) revealed the cross section of raw soft layer of maize fibre. It had a thick layer of protective cellular deposits and also presence of other constituents such as lumen (inner most part of the fibre) in increasing the absorbency of the fibres. The fibres are arranged in a near circular and honey comb manner at central region of cellular structure interlinked fibre.

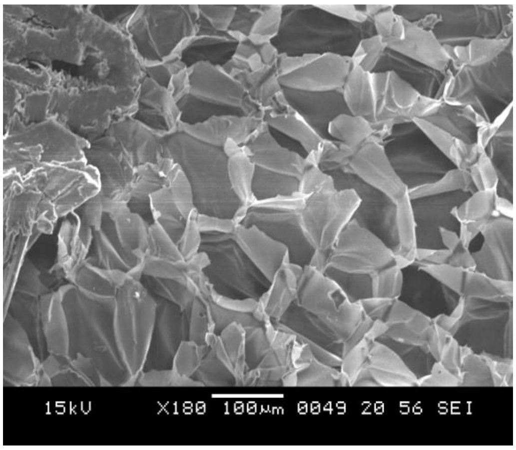
During fibre extraction, most of the vascular fibre bundles are retained. An interesting feature of the fibre cells in maize is the presence of a large lumen as shown in Figure 4.14(d), larger than the width of the cell wall in most cells, reducing the density of the fibres. Figure 4.14(e, f) showed the micrograph of surface of untreated maize fibre. Surface of raw fibre is shown in Figure 4.14(g, h) and the fibre structure had the appearance of epidermis layer, the outer skin of the fibre. Figure 4.14(i) shows the inner layer of maize fibre and had a continuous spiral filament layer which was rarely observed.


20kV X23 1mm 0045 16 58 SEI

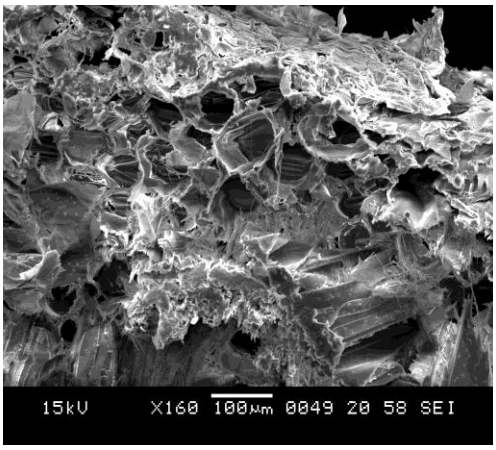
(a)


15kV X50 500µm 0049 21 56 SEI

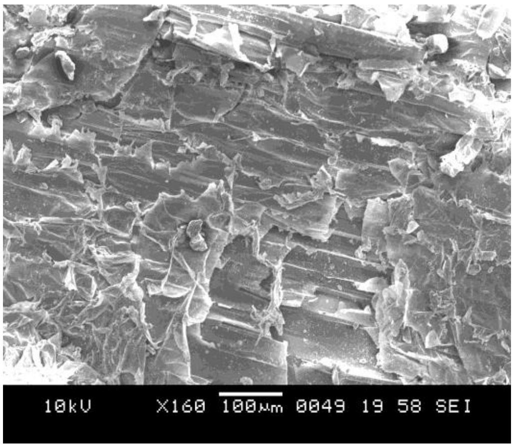
(b)


15kV X180 100µm 0049 20 56 SEI

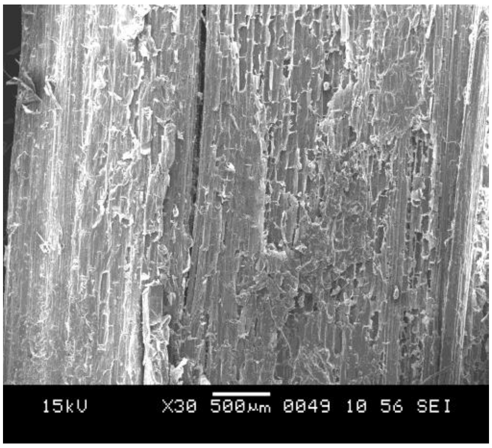
(c)


15kV X160 100µm 0049 20 58 SEI

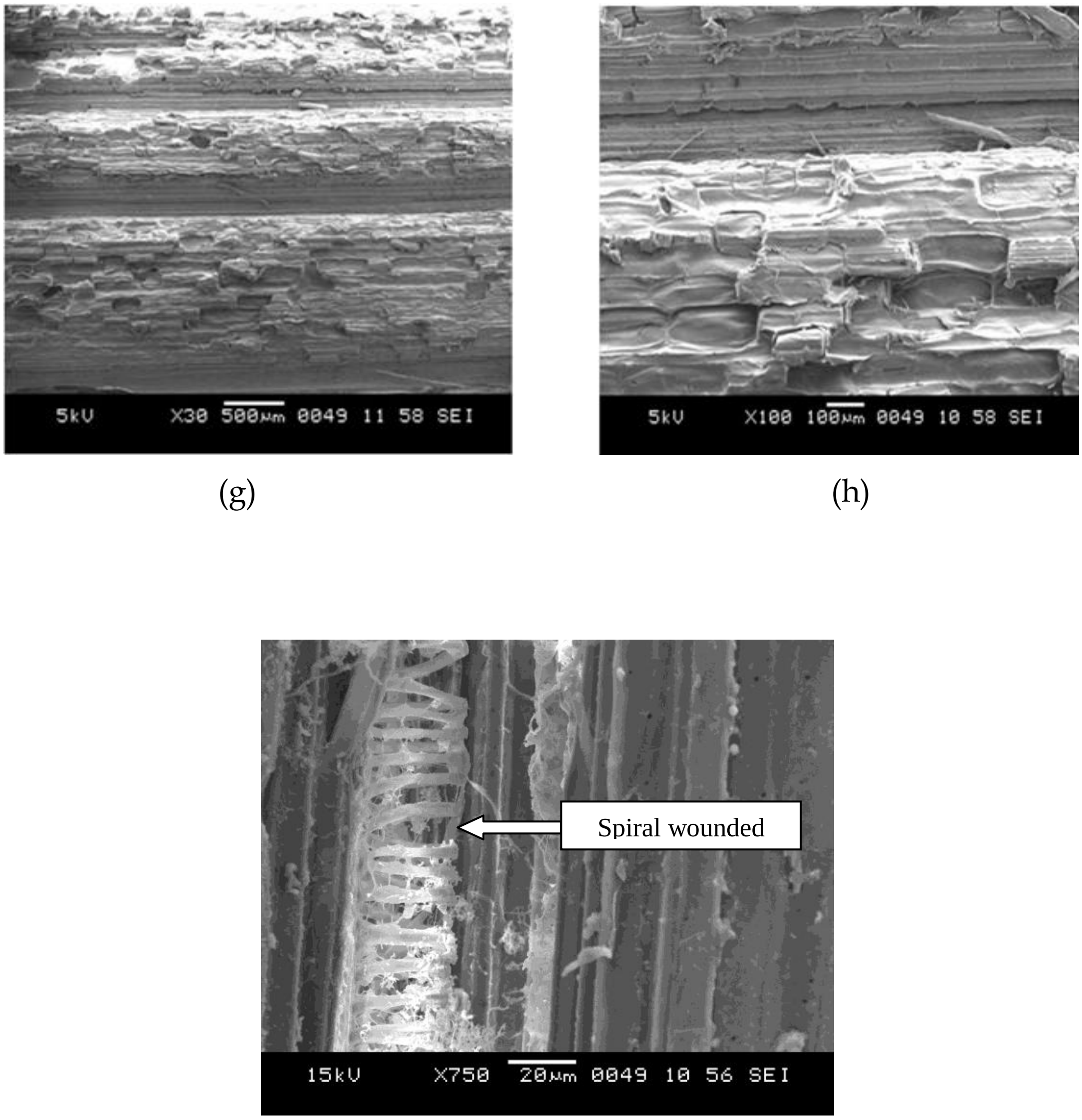
(d)


10kV X160 100µm 0049 19 58 SEI

(e)


15kV X30 500µm 0049 10 56 SEI

(f)



(g) (h)

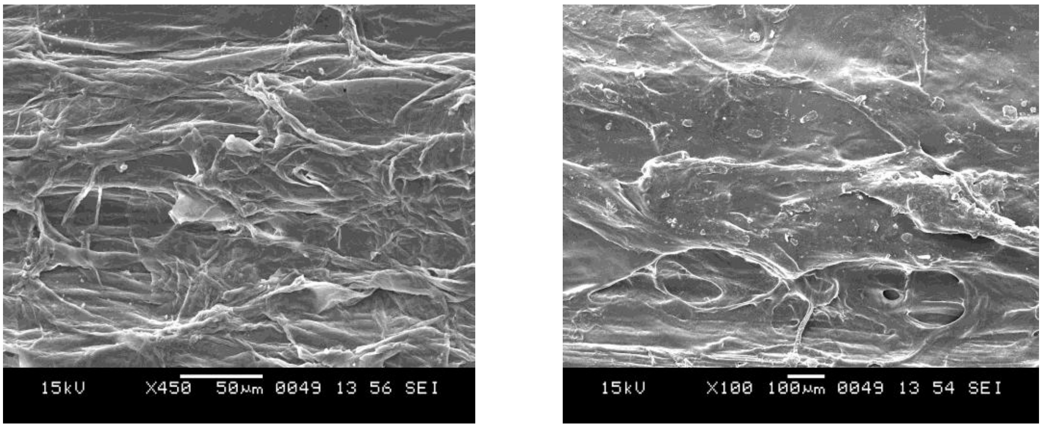
(i)

Figure 4.14: SEM Micrograph of Raw Maize Fibre

(a, b) C/S soft layer of fibre, (c, d) C/S of untreated maize fibre, (e, f) Un-treated straw surface, (g, h) Surface image of maize fibre, (i) Spiral wounded maize fibre

(ii) Chemically Treated Maize Fibres

Natural fibres are hydrophilic, can absorb moisture easily and lead to low mechanical and other properties if untreated; hence these fibres are treated with chemicals to make them hydrophobic and to attain better mechanical properties. The maize fibres were subjected to chemical treatment with alkali and acetylating method. The surface of treated maize fibres is different from natural one and SEM images of surface of these treated fibres are shown in Figure 4.15(a, b). Alkali treated fibres were shown in Figure 4.15(a) and acetylated treated fibre in Figure 4.15(b). They showed the changes in the surface morphology of maize fibre which reflects surface modification because of chemical reaction. Crystalline structure of cellulose was also altered in chemical treatment. The structural changes in the cellulose material were confirmed by X-ray diffraction (XRD) method as shown in Table 4.8 and Figure 4.20. The changes that took place in chemical treatment were the diameter of the maize fibres reducing and interaction between the fibres and the matrix were well established.



(a) (b)

Figure 4.15: SEM Images of Modified Maize Fibres

(a) Alkali treated maize fibre, (b) Acetylated treated maize fibre

4.1.4.2 Energy Dispersive Spectrometry (EDAX) Analysis

The Energy Dispersive Spectrometry (EDAX/EDX) Analysis is carried out by using Energy Dispersive Spectrometry (EDS) detector which is a solid state device designed to detect X-rays and their energy is converted into electrical charge. They are used for identifying the elemental composition of the sample.

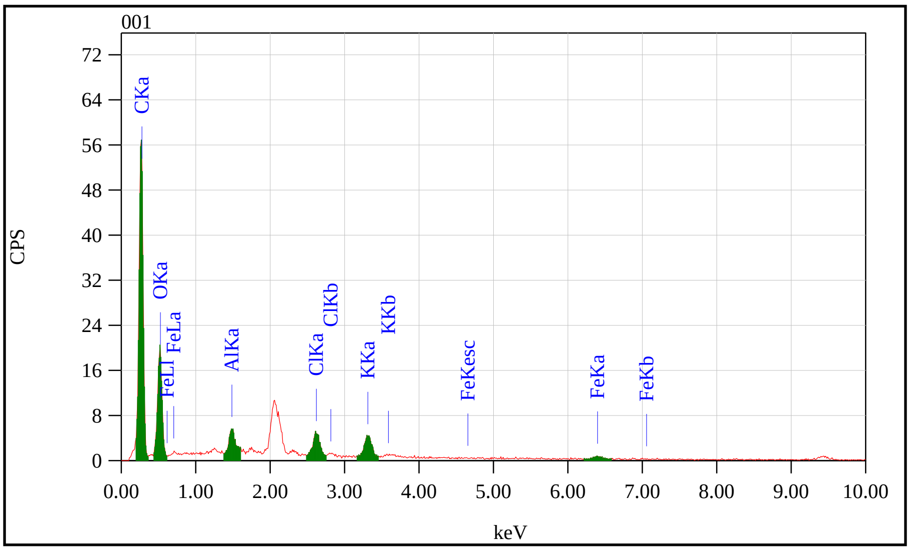


Figure 4.16: EDAX Analysis for Maize Fibre

The analysis is always integrated with Scanning Electron Microscope (SEM). The output of the analysis is an EDX spectrum with peaks corresponding to the energy levels for most X-ray received. The higher the peak in a spectrum, presence and concentrated of element is understood. Figure 4.16 shows the EDAX result obtained for maize fibre. The analysis shows the spectrum indicating the presence of Carbon (C), Oxygen (O), Potassium (K), Iron (Fe) elements with K shell peak identification. These are plant fibre cellular material consisting mainly of cellulose and the presence of Carbon, Oxygen and little traces of Potassium elements.

4.1.4.3 Fourier Transform Infrared (FTIR) Analysis

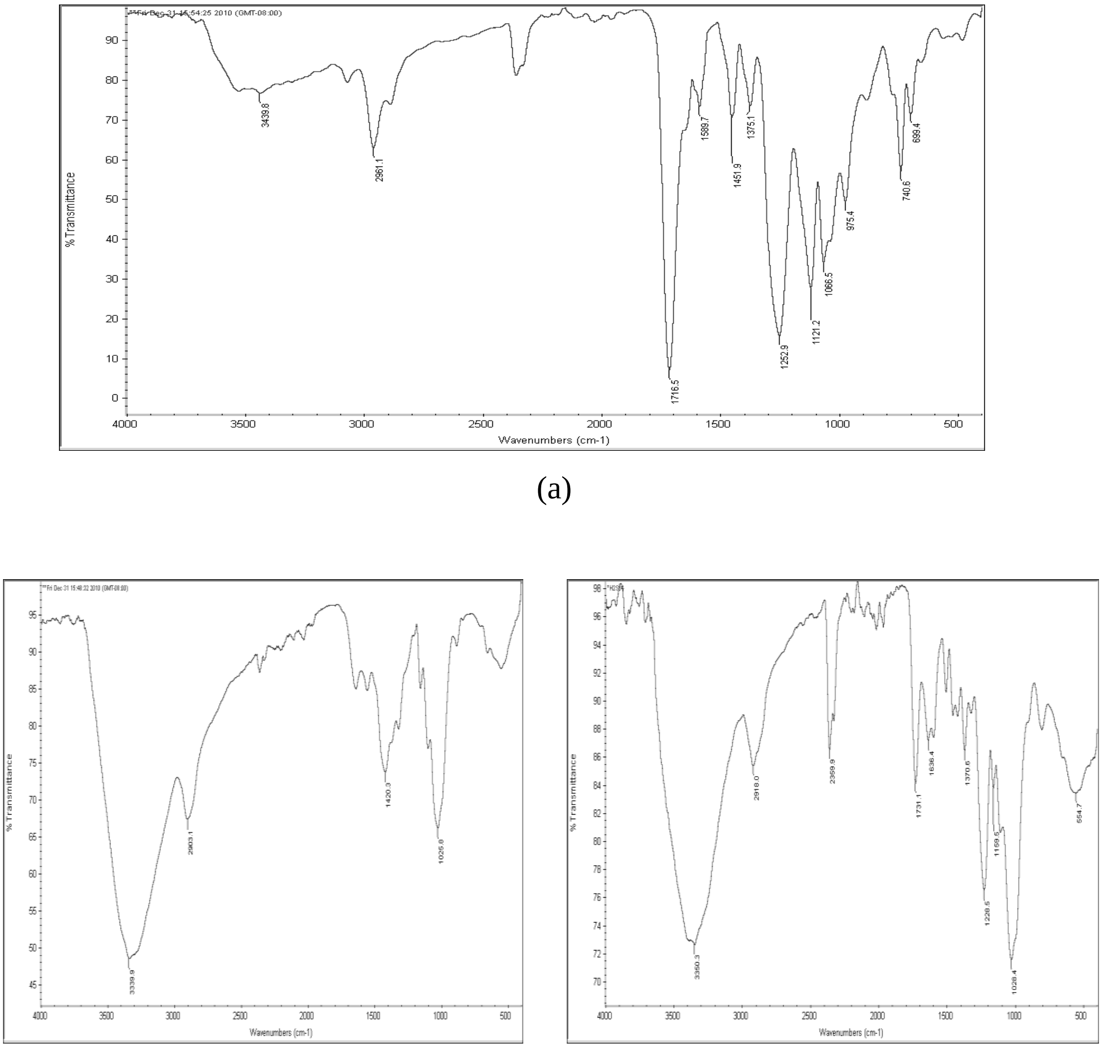
Fourier Transform Infrared Spectroscopy (FTIR) technique was used to characterize the maize fibres and their composites. It serves as a direct means for the identification of the polymer compositions and organic functional groups on the surface of the sorbent using Fourier transfer for infrared spectroscopy and the test was carried out by using Perkin Elmer, maker as shown in Figure 4.17. The analysis allowed the measurement of variations of maize fibre composition before and after the chemical treatment. The main characteristics were assigned to the presence of cellulose, hemicellulose, lignin, and other waxes. The Infrared absorption spectra recorded between 4000 $cm^{-1}$and 400 cm^{-1}.

Figure 4.17: Fourier Transform Infrared Spectroscopy

Fourier Transform Infrared (FTIR) analysis was done using Fourier Transform Infrared Spectroscopy. Raw maize and modified maize fibres was analysed for determining polymer compositions and organic functional groups on the surface of the sorbent material. Infra red spectra of raw (untreated) and alkali treated and acetylated maize fibres are shown in Figure 4.18(a), 4.18(b) and 4. 18(c) respectively.

In Figure 4.18(a) the raw fibre shows that the wide band is between 3,500 and 3,000 cm^{-1}, which are due to OH stretching vibrations of alcohols, attributed to hydroxyl (OH) groups among the fibres. The large band were attributed to the axial deformation of the OH group occurred at the peak of 3440cm^{-1} (for raw fibre) and 3340 cm^{-1}(alkalization fibre as shown in Figure 4.18(b)) and 3350 cm^{-1}(acetylated fibre as shown in Figure.4.18(c)). For raw fibres, 2961 cm^{-1} absorption band was related to the axial deformation of C–H group. The peak at 2500- 2000 cm^{-1} are characteristic of the carbonyl band (C=O) of the hemicelluloses in the maize fibre. The bands in 1716, 1589, 1451 and 1375 cm^{-1} corresponds to the C=C stretching of the lignin. The band at 1252-1020 cm^{-1} was in connection with the deformation of C–O–C of the cellulose and hemicelluloses and it was also be due to C-H and C-O deformations.

In untreated fibre, absorption band existed whereas in treated fibre it no longer exists for NaOH. Fatty substances might also been removed, resulting in disappearance of the peak as shown in Figure 4.18(c). After the reaction of acetylating, new acetyl groups were added to cellulose material. The result of the FTIR analysis confirms that the removal of lignin and hemicelluloses is affected by chemical processes. It also shows the characteristic bands for different polymers in the fibre. Hydroxyl absorption peak shifted to lower wave numbers and this shift confirms that hydrogen bonds were generated.



(a)

(b)

(c)

Figure 4.18: FTIR Analysis for Untreated and Treated Maize Fibres

(a) Raw (untreated) fibre, (b) Alkali treated, (c) Acetylated treated

4.1.4.4 X-Ray Diffraction Analysis

Maize stalk fibres and its crystalline structural characteristics were observed by X-ray diffraction. X-ray diffractogram studies were carried out using JEOL X-Ray Diffractrometer (DX-GE-2P) Japan, using Cu Kα radiation as shown in Figure 4.19. The X-ray unit was operated at 30 kV and 20 Ma. Angular scanning was conducted from 5° to 100°at 1°/min and data were collected using 2 step scan mode with angular intervals of 0.05°. Crystallinity index measures the orientation of the cellulose crystals with respect to the fibre axis. Crystalline and amorphous region was observed at closely at 30° and 23° respectively. Percent Crystallinity (Cr) and Crystallinity index (C.I) can be calculated as follows [Singha and Rana 2010].

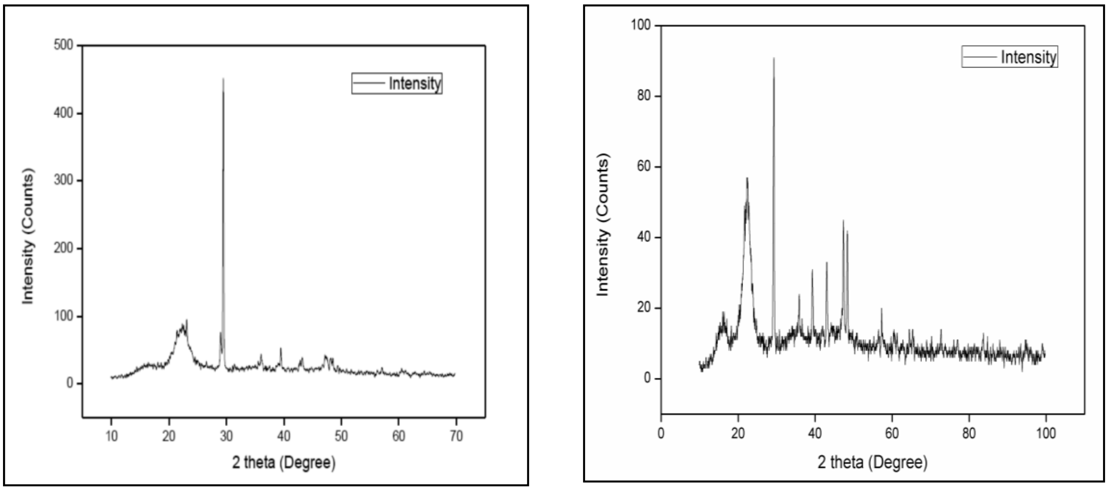
$$\mathrm{Cr}\ (\%) = \frac{\mathrm{I_C}}{\mathrm{I_C} + \mathrm{I_A}} \times 100 \tag{4.4}$$

$$\mathrm{C.I} = \frac{\mathrm{I_C - I_A}}{\mathrm{I_C}} \tag{4.5}$$

Where I_C represents the intensity diffraction of crystalline region and I_A represents the intensity diffraction of amorphous region.

The XRD analysis determined the crystallinity of the maize fibre and was used to indicate the dramatic change in the crystallinity of the maize fibre. All the fibre samples were scanned in 2θ range varying from 5^o to 100° and the tabulate values are shown in Table 4.8. The observed X-ray diffraction peaks for the above materials can be attributed to crystallinity scattering and the diffuse background to disordered regions. The spectrum corresponding to maize raw fibres shows the diffraction peaks of amorphous region and crystalline region at the following 2θ angles at 22.58^o and a high peak nearly at 29.46^o as shown in Figure 4.20(a). For alkali treated fibres, same peaks can be observed at 22.44^o and 29.28^o as shown in Figure 4.20(b). The patterns for the above materials are different however raw fibre (untreated fibre) is more crystalline than the treated fibre. Crystallinity of raw fibre is dropped after chemical treatment and a change in 0.1% crystallinity was observed this factor may be may be due to new hydrogen bonds formed and removal of hemicelluloses from cellulose material and this crystalline change (by alkali method) is significant and is calculated from Ic and Ia as shown in table 4.8. The crystallinity for acetylated treatment was not reported because the initial acetylated sample shows that no observable changes as that of alkali treated samples.

Figure 4.19: X-Ray Diffractrometer



(a) (b)

Figure 4.20: X-Ray Diffractogram for Maize Fibre

(a) Raw fibre, (b) Chemically treated fibre (alkaline method)

Table 4.8: Percent Crystallinity and Crystallinity Index for Maize Stalk Fibres

Maize Stalk Fibre	2 θ(degree)		Crystallinity (%)	Crystallinity Index
	I_C	I_A		
Raw	29.46°	22.58°	56.71	0.044
Alkali treated	29.28°	22.44°	56.61	0.233

4.1.5 Thermal Test

The thermal stability of the fibres was examined using Differential Scanning Calorimeter (DSC) and Thermo gravimetric analysis (TGA). The working method and results of these tests are discussed in the following sections.

4.1.5.1 Differential Scanning Calorimeter

DSC measures the temperatures and heat flow associated with phase transitions in substances as a function of time and temperature. Such measurements provide important information about physical and chemical changes that involve endothermic or exothermic effects, or heat capacity changes. DSC is primarily used to characterize polymers and other organic materials, but it is also applicable for testing some inorganic materials. DSC measurements were carried out using Perkin Elmer DSC 6000 as shown in Figure 4.21. Differential scanning calorimeter thermal analyzer was operated in nitrogen atmosphere. Thermal properties were examined using a Perkin-Elmer DSC. The sample is weighed and about 7 mg of the fibre samples were placed in the DSC pan. Samples of untreated and chemically treated fibre were chosen. The data is obtained on heating the samples from 20ºC to 270ºC at a heating rate of 10 ºC/min under a flow of nitrogen. Glass transition temperature (T_g) can also be determined from the data obtained from the experiments.

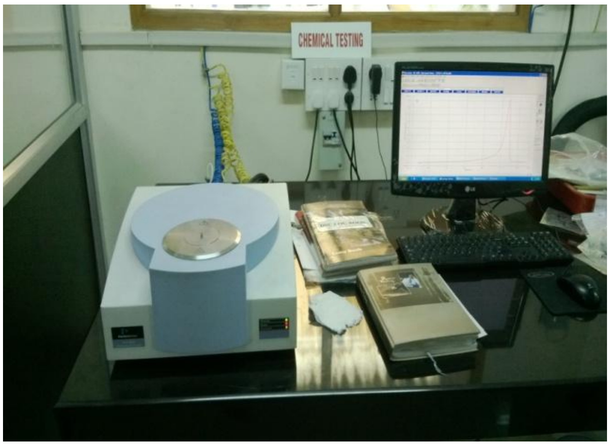
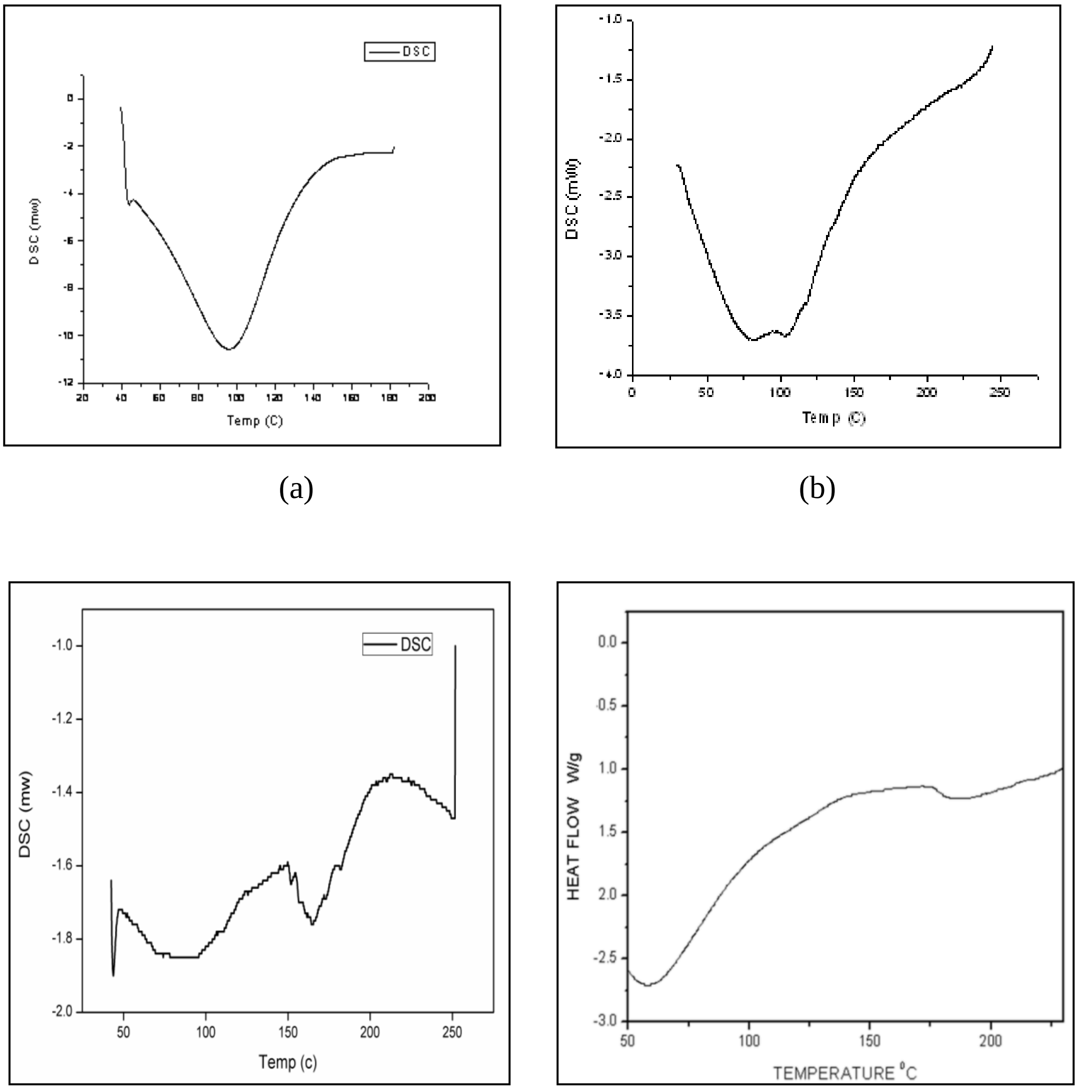


Figure 4.21: Differential Scanning Calorimeter

Figure 4.22(a- d) are the DSC results obtained by heating the specimen of raw maize fibre and fibre treated with chemicals at a constant heating rate of 10^{o} C/min. The plot shows the heat flow as a function of the sample temperature. The raw maize samples were heated at a constant rate of 10°C/min between temperature ranges of 20°C to 270°C and then cooled with nitrogen to 40^{o}C at a cooling rate of 10^{o} C/min with a flow rate of 30 ml/min. For raw fibre, the glass transition event (T_g) was observed at 43^{o}C as an endothermic stepwise decrease in the heat flow or heat capacity and a broad endothermic peak at 97^{o}C as shown in Figure 4.22(a).

For alkali treated fibre, the glass transition temperature was observed at 27^{o}C and a sharp endothermic peak at 104^{o}C and the fibre is stable up to 240^{o}C as shown in Figure 4.22(b).With a further increase in the sample temperature, the fibre undergoes exothermic peak. For acetylated treated fibre, the fibre showed some few peaks and the peak was not consistent it dragged the temperature value from 50ºC to 170ºC of endothermic and raising the value of 250ºC for exothermic values as shown in Figure 4.22(c). For polymer coated fibre, the glass transition temperature was observed at 67^{o}C as shown in Figure 4.22(d). The glass transition took place at 174ºC. The endothermic peak is mostly due to adsorbed moisture and the exothermic peaks are due to the degradation of the maize fibre.



(a) (b)

(c) (d)

Figure 4.22: DSC Thermograph for Raw and Treated Maize Fibres

(a) Raw fibre, (b) Alkaline treated fibre, (c) Acetylated treated fibre, (d) Maize stalk fibre with epoxy polymer

DSC of maize fibre was compared with previous literatures and is discussed below. Ibrahim et al. 2011 reported that the maximum DSc energy peak values for cotton fabric samples was 371.07 mW (undyed) and minimum of 325.48 mw(dyed) and all the peaks were endothermic. For coir fibre, the intial endothermic peak occurred at 105.2°C and final endothermic peak occurred at 336.2°C, but the average peak occurred at 257°C and the first endotherm started from 20°C to 185.1°C and ended with 384°C [Mahato et al. 2013]. For untreated and treated hemp fibres the endothermic peak occurred at 70-90°C and exothermic peak at 260-380°C. The endothermic peak occurred due to evaporation of moisture in the fibres. Lignin, hemicelluloses and α-celluose degraded at exothermic peak [Kabir et al. 2011].

4.1.5.2 Thermal Gravimetric Analyzer

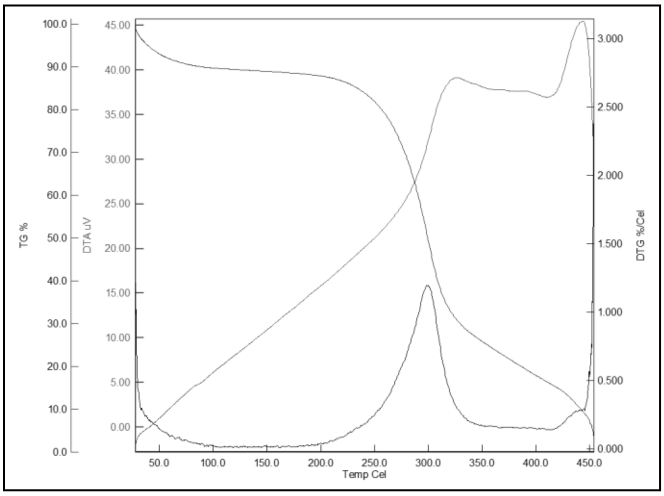
The thermal analysis comprises of different methods such as Thermo Gravimetric Analysis (TGA) / Differential Thermal Analysis (DTA), Derivative Thermo Gravimetry (DTG). Thermo gravimetric analysis (TGA) was conducted to know the degradation characteristics of raw maize fibres. It was studied as a function of % weight loss with the increase in temperature. Is a simple analytical technique that measures the weight loss (or weight gain) of a material as a function of temperature. As materials are heated, they lose their weight from a simple process such as drying, or from chemical reactions that liberate gases. Thermo gravimetric (TG) curves and Derivative thermo gravimetric (DTG) curves of the fibres were obtained by Perkin Elmer TGA (as shown in Figure 4.23) heating the samples under a Nitrogen atmosphere of 50ml/min. The natural fibre sample was heated from room temperature to 470ºC at a heating rate of 10ºC/min.

Figure 4.23: Thermo Gravimetric Analyser

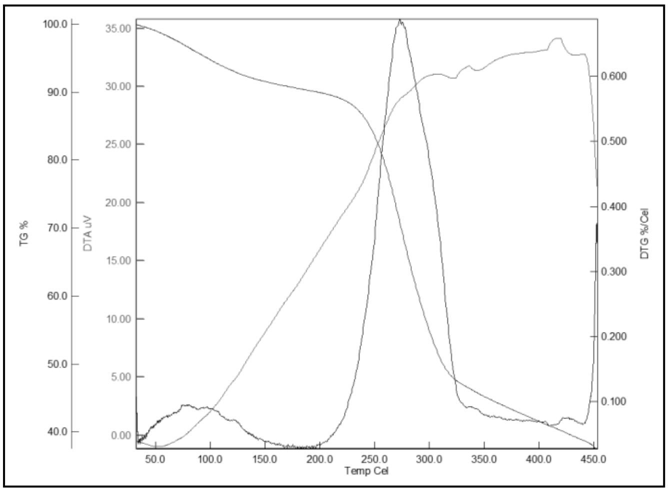
A sample of fibre was transferred into a platinum cup that is supported on an analytical balance located outside the furnace chamber. The balance is adjusted to zero, and the sample cup is heated according to a predetermined thermal cycle. The balance sends the data to the computer for storage, along with the sample temperature and the elapsed time. The TGA curve plots the percent weight change on the Y-axis against the reference material temperature on the X-axis. Differential Thermal Analysis (DTA), in which a small quantity of sample of 10 mg and a thermally inert reference material are separately heated in an electric tube furnace at a controlled rate in a given atmosphere. The temperature difference between the sample and the inert material is continuously measured with a differential thermocouple pair and plotted as a function of furnace temperature. The curve obtained is sometimes called the combustion curve. Differential (Derivative) thermo gravimetric Analysis (DTG), which is similar to TG except a continuous plot of the rate of weight loss with time as a function of furnace temperature is produced.

The thermal degradation of natural fibre studies are further supported by differential thermal analysis (DTA) and the results are shown in Figure 4.24. TGA curve shows two stages of decomposition, initial stage can be due to the decomposition of cellulose and hemi cellulose segments and the later stage due to the degradation of lignin and other segments on the fibre surface. The raw fibre started showing primary decomposition at 150°C as shown in Figure 4.24(a), but the actual first stage decomposition temperatures were from 211°C to 334°and second stage decomposition from 334° to 434°C respectively. Weight loss in the first stage was 47.6 % and in second stage was 13.4 %. The DTGA curves show a single peak at 278ºC may be due to decomposition of some flexible segments. TG curves of maize fibre (alkali treated) confirms the increase of thermal stability of the fibres, first stage decomposition took place at 221°C to 340°C and second stage decomposition occurred at 340° to 444°C as shown in Figure 4.24(b). The maximum temperature was observed ast 284°C. Figure 4.24(c) shows the acetylated treated fibre with fibre losing its decomposition at 150°C but actual decomposition temperature at first stage was from 220°C to 310°C and second stage decomposition was from 310°C to 440° C. Weight loss was 44.6 % and 12.4% in first and second stage and the maximum temperature was at 340°C. Table 4.9 shows TGA Analysis for Maize Fibres.

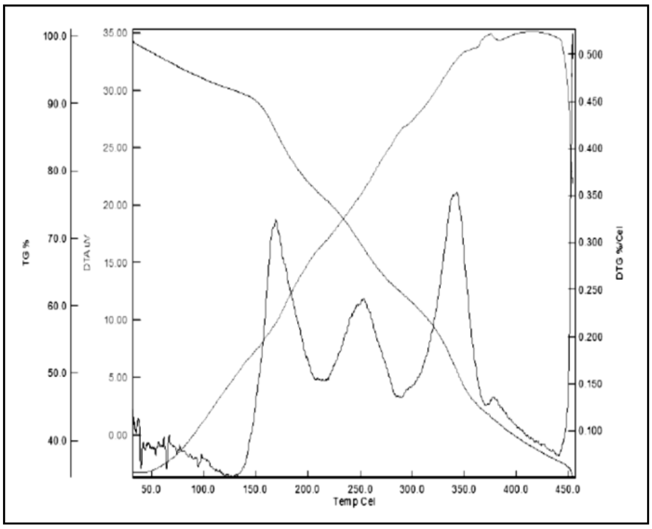
TGA of maize fibre compared with previous literature and are brifed out. Three stages of weight occurred at hemp fibres, the first stage of weight loss from 30 -110°C was due to the release of moisture. The second stage degradation occurred at around 200-300°C, which was due to the degradation of lignin and hemicelluloses of the fibre. The last stage at around 340-380°C, indicates the degradation of α-cellulose and other non cellulosic materials [Kabir et al. 2011]. The DTG peak for cotton stalk occurred at 293.4°C, where as for wood pine it was 311.5°C [Yao et al. 2008].



(a) Raw Maize Fibre



(b) Alkaline Treated Maize Stalk Fibre



(c) Acetylated treated maize stalk fibre

Figure 4.24: Thermo Gravimetric Analysis for Maize Fibre

Table 4.9: TGA Analysis for Maize Fibres

Maize Fibre	Stages of degradation	Decomposition Temperature	Maximum Temp (°)
Raw fibre	I-Stage	211°- 334°	278°
	II-Stage	334°- 434°	
Alkaline treated fibre	I-Stage	221°- 340°	284°
	II-Stage	340°- 444°	
Acetylated Treated fibre	I-Stage	220°-310°	340°
	II-Stage	310°- 440°	

4.2 Properties for Maize Fibre Composites

4.2.1 Physical Properties for Maize Fibre Composites

4.2.1.1 Density and Void fraction

The theoretical density of the composite was determined by

$$\rho_{tc} = \frac{1}{\frac{W_f}{\rho_f} + \frac{W_m}{\rho_m}} \qquad (4.6)$$

Where W and ρ represents the weight and density respectively and subscripts f represents fibre, m represents matrix.

Experimental actual density of the composite was determined by water immersion method (Archimedes principle) and by using equation

$$\rho_{ac} = m/v \qquad (4.7)$$

Then, volume fraction of voids in the composite was calculated by using

$$v_v = \frac{\rho_{tc} - \rho_{ac}}{\rho_{tc}} \qquad (4.8)$$

Where, v_v= volume fraction of voids

ρ_{tc}= theoretical density of composite

ρ_{ac} = actual density of composite

Density of maize fibre composites processed by VARTM is shown in Table 4.10 and Figure 4.25. The void fraction % is calculated from the above formula. The graph shows the variation of void fraction with percentage of weight fraction of composites. The void fraction percentage increases as the fibre content in the composite was increased. The value percentage of voids is very small because of VARTM process. In VARTM, processing of composite is carried out in vacuum hence the composite contains very less voids. Therefore percentage void fraction depend on fibre content in maize composite

Table 4.10: Density of Maize Fibre Composite

Sl. No.	Fibre Content (%)	Theoretical Density (ρ_{tc}) (gm/ cm^3)	Actual Density (ρ_{ac}) (gm/ cm^3)	Volume Fraction of Voids (v_v) (%)
1	5	1.0391	1.0321	0.6736
2	10	0.9691	0.9618	0.7532
3	15	0.9079	0.8998	0.8921
4	20	0.8540	0.8456	0.9836
5	25	0.8061	0.7971	1.116

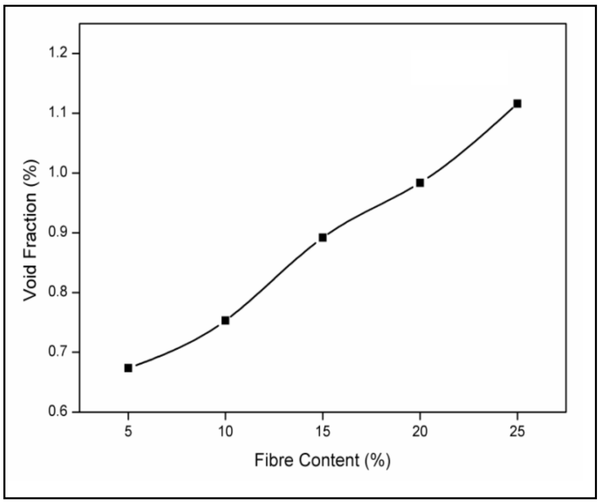


Figure 4.25: Void Fraction of a Composite versus Maize Fibre Content

4.2.2 Mechanical Properties for Maize Fibre Composites

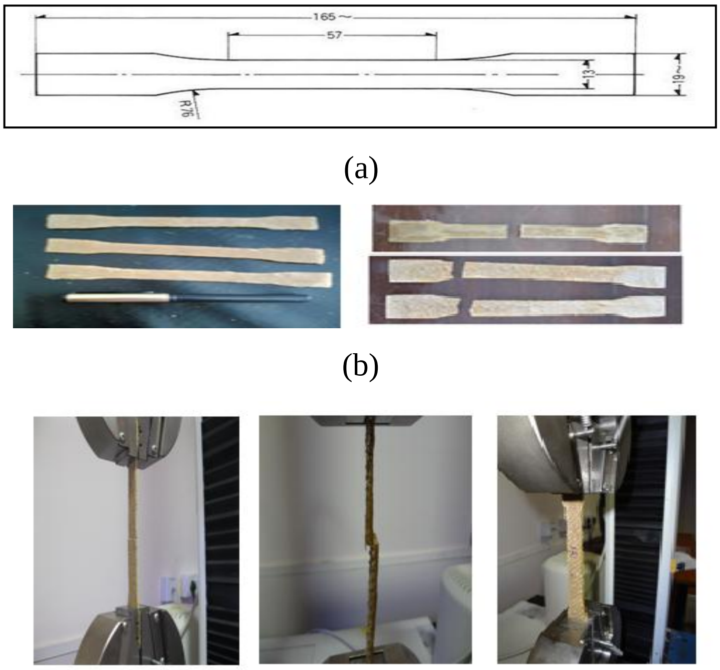
Mechanical test such as tensile test, flexural test (three point bending test), impact test (Charpy test) are performed for Maize fibre composite, Hybrid composite (Maize fibre+ Jute fabric of one layer) and Sandwiched hybrid composite (Maize fibre + Jute fabric of two layer consisting one layer above and below of core maize composite).

4.2.2.1 Tensile Test

4.2.2.1.1Maize Composites

Tensile strength of maize fibre composites and its behaviour were tested according to ASTM standards; ASTM D638-10 (Standard test method for tensile properties of plastics). The samples of size width 13 mm, gauge length of 57 mm, over all length 165 mm, thickness 3.2 mm are prepared as shown in Figure 4.26(a). Prepared samples (maize fibre composites, Hybrid composites, Sandwiched hybrid composites) of different weight fraction of fibres and matrix ranging from 5, 10, 15, 20 to 25 % (in the interval of 5%) was tested. The samples were tested in tensile machine with load cell of 10 kN with cross head speed of 5 mm/min. The specimens were polished and free from dust are clamped in the fixture of tensile machine. Proper care was taken in placing the specimens in the fixture and for each trial initial reading was set to zero. Strain rate plays a major role in determining the modulus value of the composite material. The specimens were tested at cross head speed of 5mm/min as per ASTM standards. Test was conducted at constant strain rate of the order 5 mm/min and load was applied till the failure of the specimen, as shown in Figure 4.26(b & c). Load – extension curve was obtained and the results were plotted for different samples. The test was conducted for 5 samples from 5%, 10%, 15%, 20% and 25% maize fibre composites, maize-jute composites and maize-jute sandwiched composites and the average result were reported. The modulus is determined using the initial tangent modulus in the strain range up to 0.2%

The macroscopic image of failed specimen is shown in Figure 4.26(b) and the failure occurred gage length and others at clamping region, this was because the occurred failure specimen are lateral failure. Fracture initiated at one point and developed across the width of the specimens. In most of the samples, the failure region occurred at the middle part of the specimen, this kind of failure is called lateral gage middle (LGM) failure. Few specimens were failed in the region of lateral grip/tab part (bottom or above). Generally these kinds of failure are accepted to calculate the modulus and strength values. All the specimens failed at sudden and catastrophic failure.



(a)

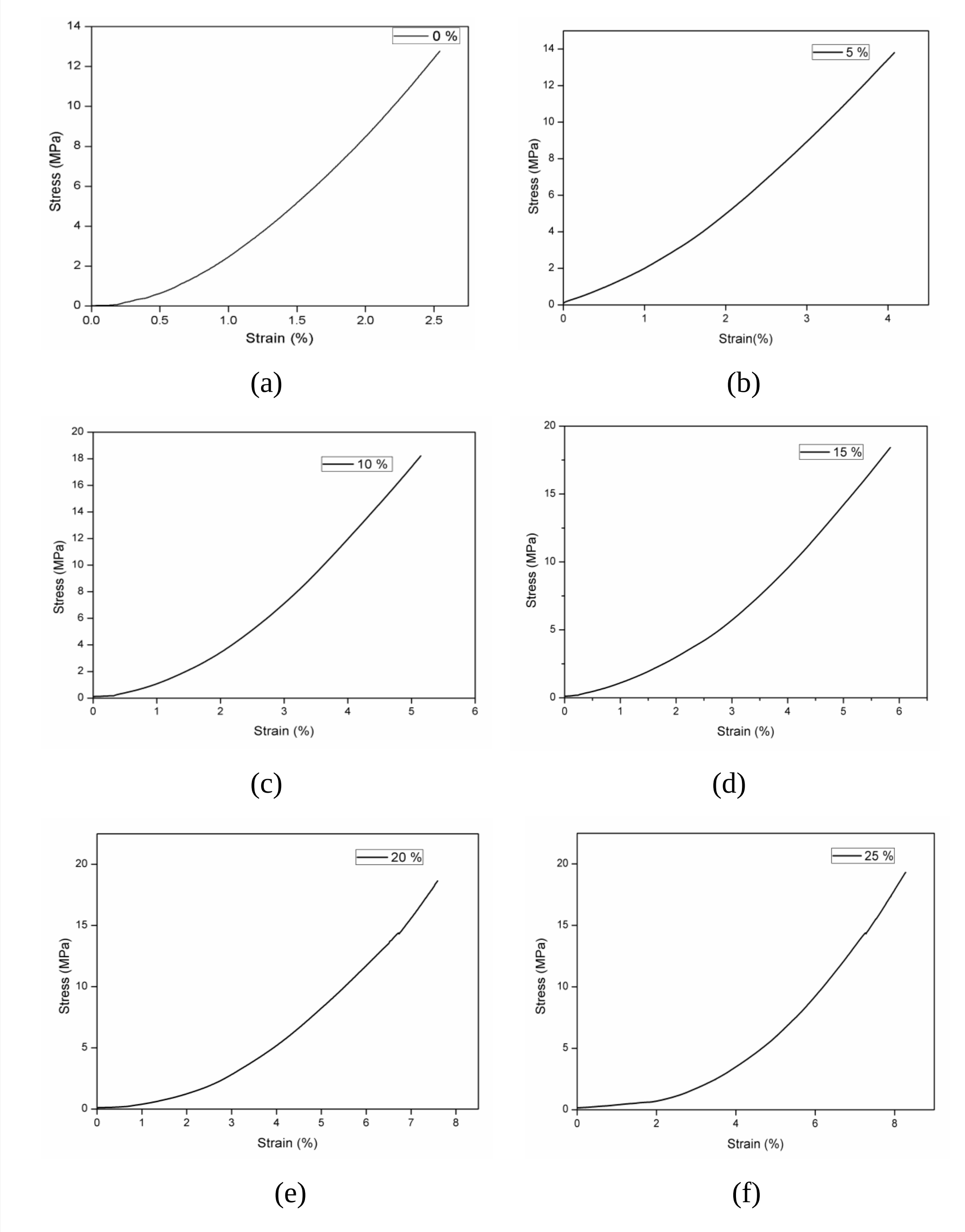
(b)

(c)

Figure 4.26: Tensile Testing of Samples

(a) Specification of specimen as per ASTM D638-I, (b) Tensile test samples, (c) Samples in UTM.

The Maize fibre composites are tested for tensile strength in an Instron Universal Testing Machine (UTM) as explained in the above section. Figure 4.27 shows the stress-strain diagram for natural Maize composite for different weight fractions. Maxmium ultimate strength of 19.29 MPa with strain of 8.27 % was observed for 25% content of maize fibre composites. Maximum Young’s modulus of 1.71GPa was seen in 10% content of maize fibre composites. At higher load values, fibre response becomes linear, while at beginning it is nonlinear. The strength of the maize fibre composite increases till it reaches its ultimate value before break. The apparent variation of tangent modulus with strain confined mostly to the initial, small strain part of the diagram and is attributed to the orientation of the fibrils along the axis of the fibre under load. From the tensile testing results, it shows that the strain rate plays an important role in the behaviour of the curves, nature of failure and the strength of the fibres. The specimens were tested at cross head speed of 5 mm/min.



(a) (b)

(c) (d)

(e) (f)

Figure 4.27: Stress- Strain Curve for Maize Composites

(a) Epoxy polymeric composite (0% wt. Fibre content composite), (b) 5% wt. Fibre content composite, (c) 10% wt. Fibre content composite, (d) 15% wt. Fibre content composite, (e) 20% wt. Fibre content composite, (f) 25% wt. Fibre content composite.

Stress strain curve and corresponding load deflection curve for maize fibre composite is shown in Figure 4.28(a) and 4.29(b). The nature of the curve for both plots is similar except in their unit values. The tensile strength and Young's Modulus for neat epoxy is 12.77MPa and 0.83GPa

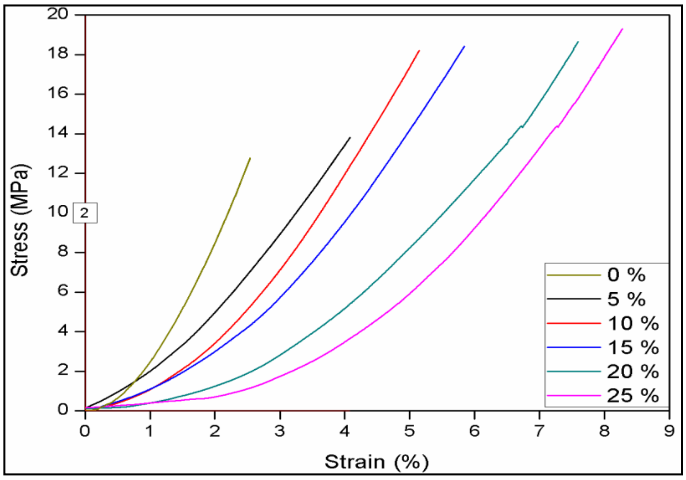


Figure 4.28(a): Stress-Strain Curve for Maize Fibre Composites

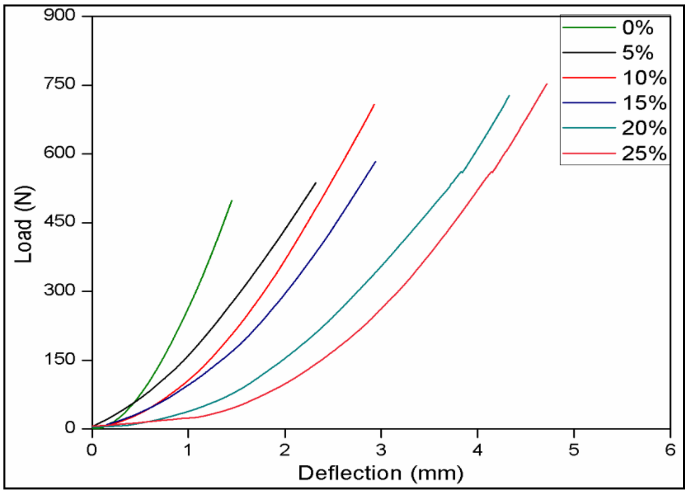


Figure 4.28(b) Load Deflection Plot for Maize Fibre Composites

At 5%, 10% and 15% of maize fibre content the stress strain curve for maize composites are approximately linear without any disturbance at higher loads as shown in Figure 4.27(a, b, c). In 15 % and 20 % the fibre composites had a disturbed variation around 15 MPa this is due to more amounts of fibres in the composite and the interface in the material had some bonding or discontinuity in the fibre thus leading to a sharp increase with the values as shown in Figure 4.27(d, e). Maxmium tensile stress and strain values along with Youngs Modulus are shown in Table 4.11. A composite with 25 % weight content of fibre had high ultimate stress value when compared to other fibre composites as shown in Figure 4.28. Values obtained by tensile strength reflect the bonding quality and the presence of voids at the interface determines the bonding quality. Fractograhic studies for the maize fibre composites was done on the fracture surface of tested tensile specimen and the valid reasons for failure are discussed in section 4.2.3.3.5.

Table 4.11: Stress-Strain values for Maize Fibre Composites

Sl. No.	Fibre Content wt. (%)	Maximum Tensile Stress (MPa)	Tensile Strain at break (%)	Young's Modulus (GPa)
1	0	12.77	2.54	0.83
2	5	13.80	4.07	1.63
3	10	18.21	5.14	1.71
4	15	18.41	5.83	1.52
5	20	18.64	7.59	1.18
6	25	19.29	8.27	1.12

As the load increases, fibre response becomes linear while at beginning it is nonlinear this is because plant fibres display a characteristic of non-linear twist response. The non-linear response could also be the response attributed to the role of micro fibrils. Upon load application the stretch of cellulose micro fibrils is taken into consideration and increased micro fibril angle leads to more non-linear response in tension. At the initial stage of applied load a non-linear behaviour is observed later the response becomes more linear. When the load increases, the fibres become straighter and aligned with the loading direction.

Young's modulus increase then decreases with increase in fibre content this is because Youngs modulus of the composite increase with an increase in fibre content (Happened from 5 to 10% fibre content). The maximum value was at 10% maize fibre. (In this range the fibres were well distributed and there was good interfacial bonding between fibre and matrix). Then the Youngs modulus decreased from 15% fibre content because of irregular distribution of fibre and matrix, improper wettability of fibres and continuity.

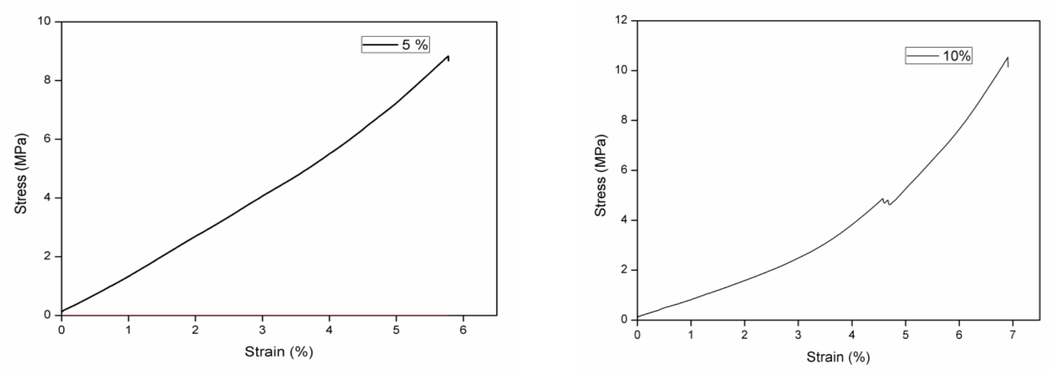
Modulus of strength of fibres was compared with different literature and is briefed out. Kumar et al. 2014 studied banana fibre and sisal fibre composites and their effect of fibre length for natural fibre composite beams. It was noted that tensile strength (30 % fibre wt, for 3mm) for banana composite was 20 MPa and for sisal fibre composite 26.9 MPa. For bamboo based polymer composite the average tensile strength was 35.1 MPa and average Youngs modulus of 4.65Mpa [Okubo et al. 2004]. Kumar et al. 2011 studied the performance of zea mays polymer composites (25% fibre weight) and the observed tensile strength for unidirectional composite was 55.81 MPa and for random composite it was 53.91 MPa and corresponding Youngs modulus was 0.58 GPa and 0.60 GPa for random composites.

4.2.2.1.2 Hybrid Composites

Five samples each in different content of maize fibre composites were tested for hybrid fibre (Maize + Jute fabric of one layer) composites. Jute fabric skin type with single layer and core of pulverized maize fibre was used in the hybrid composite material. Stress strain curves for hybrid maize composite are shown in Figure 4.29(a to e) and the maximum stress and strain values in Table 4.12. For, 5 % and 10 % the composite material behavior almost as a linear curve showing the property of brittleness (as thermoset polymer composites show the property of brittleness). Where as, in 15%, 20 % and 25 % fibre content the composite material show a ductile nature with larger strains before fail. At initial loads, the matrix material was subjected to tensile first, then fibres and matrix are subjected to stresses. When the load was further increased, maize fibres were not able to with stand the increased stresses applied and failure occured at the core (maize fibres) and hence there was a slight change in the smooth curve.

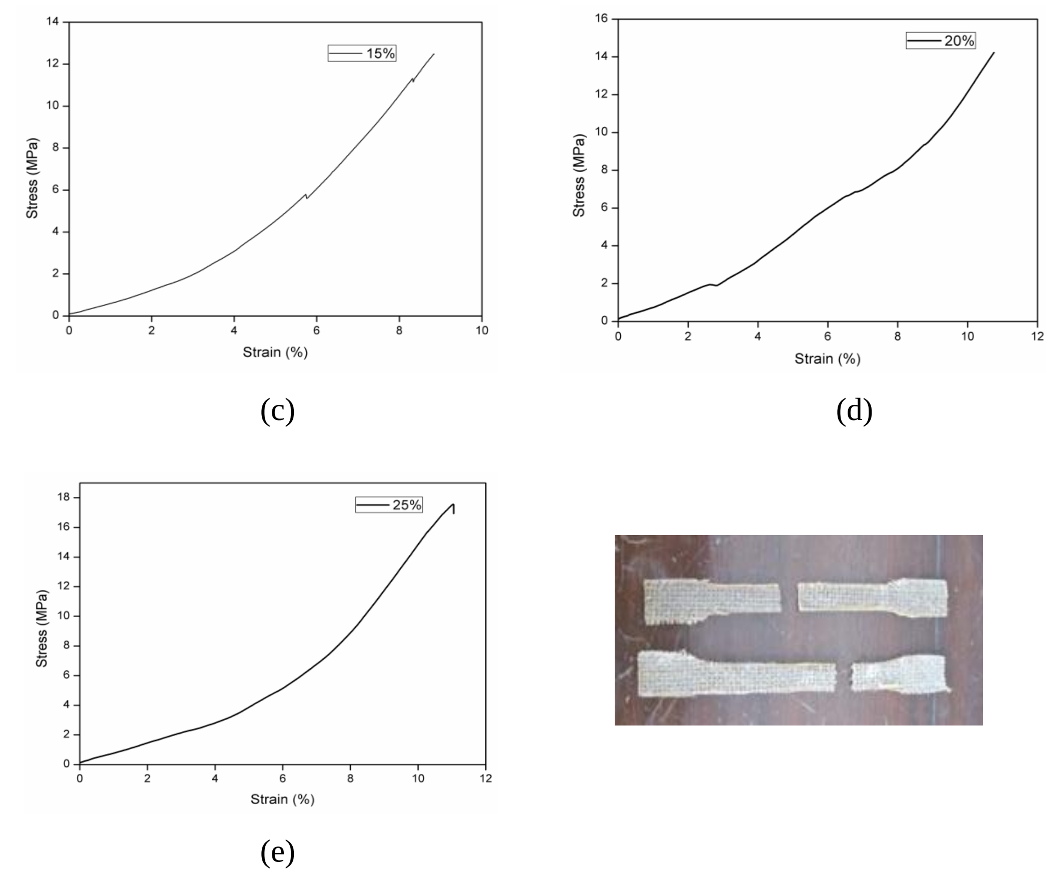
Later, increased load in the skin layer of jute gets additional stresses and hence a raise in curve then after they fail and break. This is clearly visible in the graphs. 25% fibre content hybrid maize fibre composite had more tensile strength when compared to other content fibre composites as shown in Figure 4.30. The specimen failed where the fibres had weak bonding with the matrix, hence initial cracks are developed. This crack tends to propagate linearly with the increased load. The failure can be at neck region or at the middle of the specimen as shown in the figure 4.29. The maximum stress occurred was 17.56 MPa for 25 % fibre content composite and high Youngs modulus of 1.62 was observed in 5% fibre content composite as shown in Table.4.12.

Image of the failure specimen shows different failure location in Figure 4.29, this is because the specimen failed where the fibres had weak bonding with the matrix, and hence initial cracks are developed. This crack tends to propagate linearly with the increased load, and the failure can be at neck region or at the middle of the specimen and the specimen failed where the fibre matrix had weaker section. The obtained image shows that tensile test was carried for hybrid composites.



(a)

(b)



(c)

(d)

(e)

Figure 4.29: Stress-Strain Curves for Hybrid Composites

(a) 5% wt. Fibre content composite, (b) 10% wt. Fibre content composite, (c) 15% wt. Fibre content composite, (d) 20% wt. Fibre content composite, (e) 25% Fibre content composite.

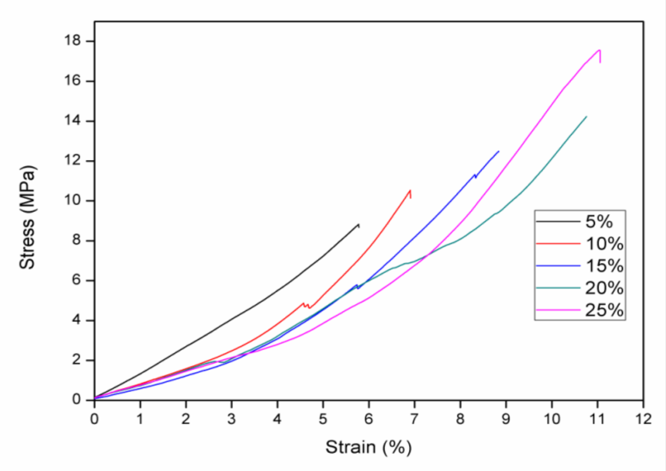


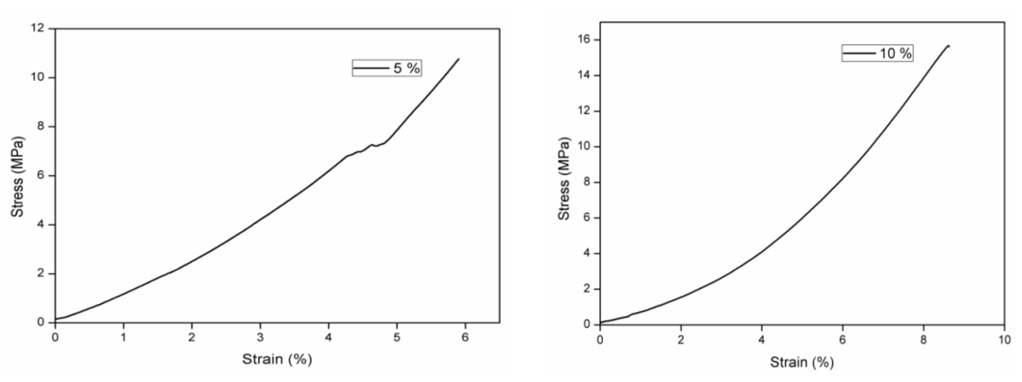
Figure 4.30: Stress- Strain Curve for Hybrid Composites

Table 4.12: Stress- Strain values for Hybrid (Maize + Jute fabric) Composites

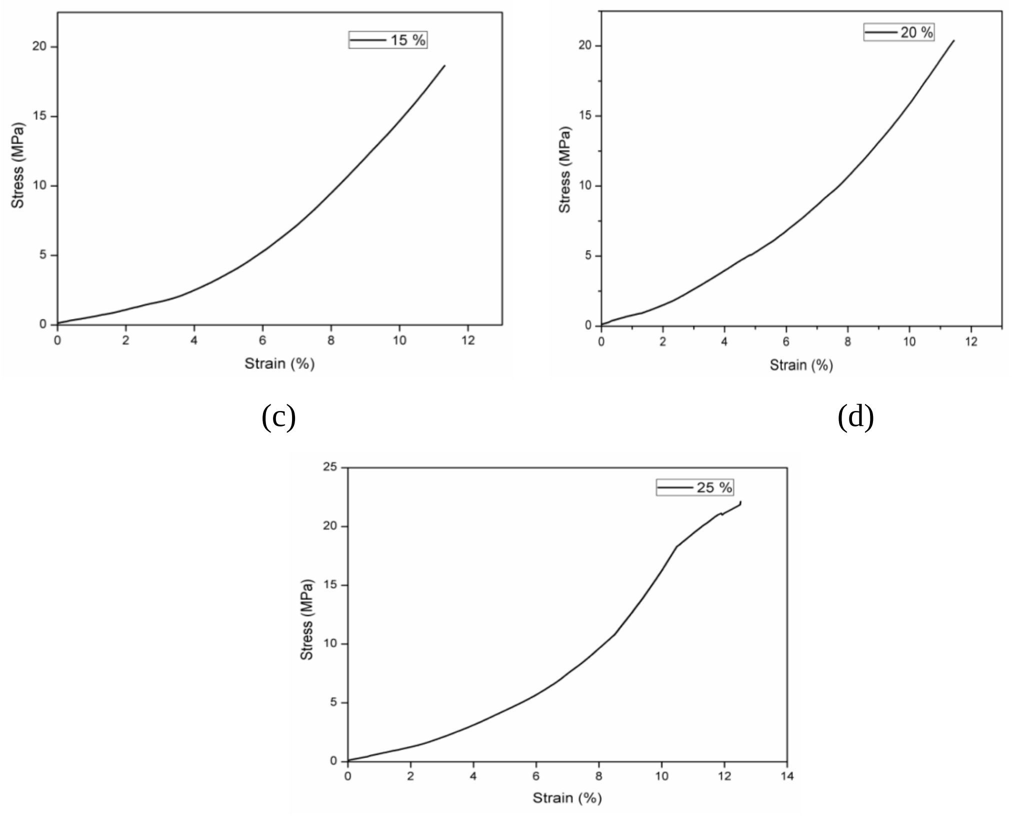
Sl. No.	Fibre Content wt. (%)	Maximum Tensile Stress (MPa)	Tensile Strain at break (%)	Young's Modulus (GPa)
1	5	8.83	5.77	1.62
2	10	10.53	6.90	1.58
3	15	12.48	8.83	1.50
4	20	14.22	10.75	1.42
5	25	17.56	11.05	1.33

4.2.2.1.3 Sandwiched Hybrid Composites

Sandwiched hybrid fibre (Maize + Jute fabric of two layers) composites were processed by hand lay-up method and tested by using Instron universal testing machine as explained in above section. The composite material had maize fibres epoxy core and sandwiched with jute fabric on top and bottom surface layer. The maize fibre content of the composites were same as explained in the previous section and five samples of each wt. % fibre content was tested. Graphs showing stress and strain for sandwiched hybrid composites with 5 % to 25 % of fibre content as shown in the Figure 4.31 (a to e) .



(a) (b)



(c) (d)

(e)

Figure 4.31: Stress- Strain Curve for Sandwiched Hybrid Composites

(a) 5% wt. Fibre content composite, (b) 10% wt. Fibre content composite, (c) 15% wt. Fibre content composite, (d) 20% wt. Fibre content composite, (e) 25% Fibre content composite.

Maxmium tensile stress/ strength of composites increased with increase in % content of maize fibre as shown in Figure 4.31 and the curves were typical with other composites. During test, it has been observed that initial crack had occurred in the core part of the composite (maize fibre-epoxy) and later the crack has slowly extended to the outer part of the composite (Jute fabric mat) when the load had been increased. Thus the material was trying to behave as a non-linear ductile material in high fibre % content. 10%, 15 % and 20 % fibre content of the composite material responded well to the load and had a smooth curve showing the brittle behaviour as shown in Figure 4.31(b, c, d).

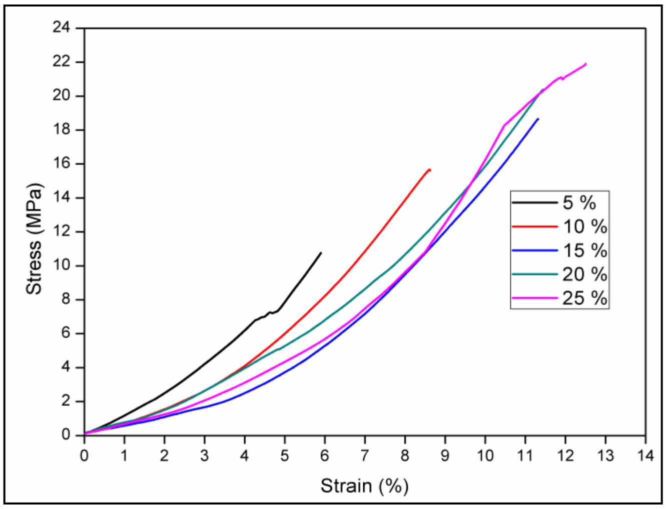


Figure 4.32: Stress Strain Curve for Sandwiched Hybrid Composites

The curve for the stress strain behavior for 5% fibre composite material showed there was a slight increase in strain when it has reached 4.5% strain as shown in Figure 4.31(a). From the Figure 4.32, all stress and strain values for 5% to 25 % fibre composite material is shown. The maximum stress of value 21.89 MPa had occurred in 25 % weight fibre content composite as shown in Table 4.13. Among them, 25% hybrid material had higher stress and strain values having at early stages and some steep values in the curve after 6% strain. 25% of fibre content hybrid material had initial less strain values less than that of 20% fibre material when the load is increased further the curve had a sharp gain stress value. Generally in most of initial experiment, crack was observed in the maize and later with increased to the material failure occurring at the jute fabric of the composite material.

In sandwiched hybrid composite, the initial crack had occurred in the core part of the composite and the crack has extended slowly. This was confirmed by visual while performing the tensile test. Ramesh et al. 2013 studied the mechanical property of sisal - jute fibre reinforced polyester composites. The tensile strength for glass + sisal fibre composite was 176.20 MPa whereas for glass fibre + jute fibre + sisal fibre composite the value was 200 MPa [Ramesh et al. 2013].

Table 4.13: Stress Strain Values for Sandwiched Hybrid Composites

Sl. No.	Fibre Content wt. (%)	Maximum Tensile Stress (MPa)	Tensile Strain at break (%)	Young's Modulus (GPa)
1	5	10.76	5.90	1.83
2	10	15.67	8.62	1.82
3	15	18.65	11.32	1.79
4	20	20.38	11.44	1.76
5	25	21.89	12.50	1.65

4.2.2.1.4 Bio-Composites

The tensile strength for a bio-composite was carried out as the same method explained in the above sections. The bio-composite consisted of 15% fibre content wt. (%) infused with matrix material of epoxidized soy bean oil biopolymer. The tensile strength of the bio-composite material was 10.11 MPa as shown in Figure 4.33 and Young's modulus of the bio-composite material was 1.2GPa with tensile strain at break of 2.71 % strain. Initially the material started with low value then later at 4MPa the values of stresses started increasing rapidy and reaching the value of 10.11 MPa as the ultimate stress. The obtained value of bio composites was lower than that of regular natural fibre composite material consisting of epoxy polymer.

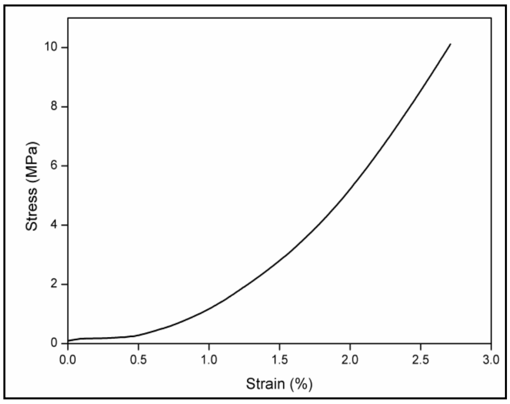


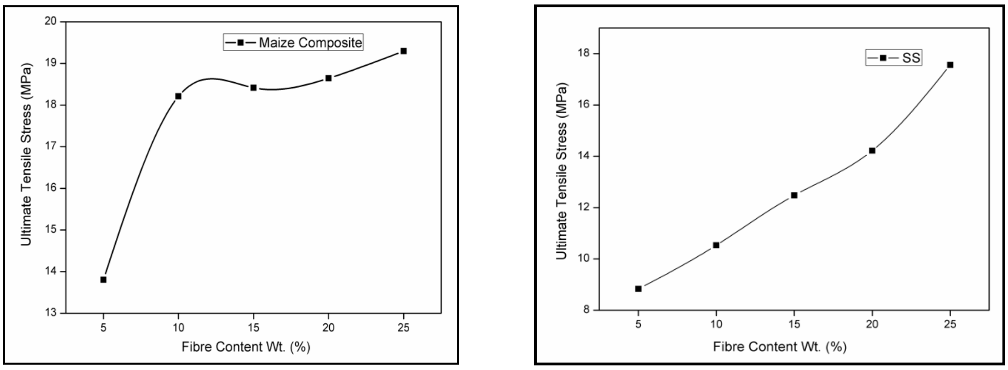
Figure 4.33: Stress Strain for Bio-Composite

4.2.2.1.5 Comparison Study

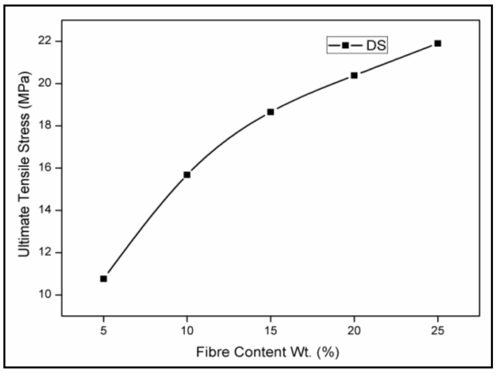
(i) Comparison of Ultimate Tensile Stress for Maize Fibre Polymer Composites

The experimental values of ultimate tensile stress for the composites of 5%, 10 %, 15%, 20% and 25 % wt. of the maize composite, hybrid composite and sandwiched hybrid composite material were plotted as shown in Figure 4.34. The maximum ultimate tensile stress had occurred in 25% fibre content weight composite as shown in Table 4.14. Ultimate tensile stresses was less for 5%, after 15% the values are fairly constant this is because after the fibres matrix interaction relations. Further increase in wt. % fibre content will not improve the strength. The processing technique uses vacuum infusion of resin and that may not results in creating voids.

In hybrid composites the ultimate tensile stresses increased with % fibre content this is because the jute fabric and the maize fibre interaction with matrix resulting in high strength values. In sandwiched hybrid composites the processing technique was hand lay-up method and the fibres had good interaction (the cross section of the composite material are explained in morphology analysis in section 4.2.3.2.3) and the obtained values were also better when compared to other composite material. It observed a maximum value at 25 % fibre content composite with 21.89 MPa.



(a) (b)



(c)

Figure 4.34: Ultimate Tensile Stress Fibre Content Curve For Maize Composites (a) Maize Composite, (b) Hybrid Composite (SS), (c) Sandwiched Hybrid Composite (DS)

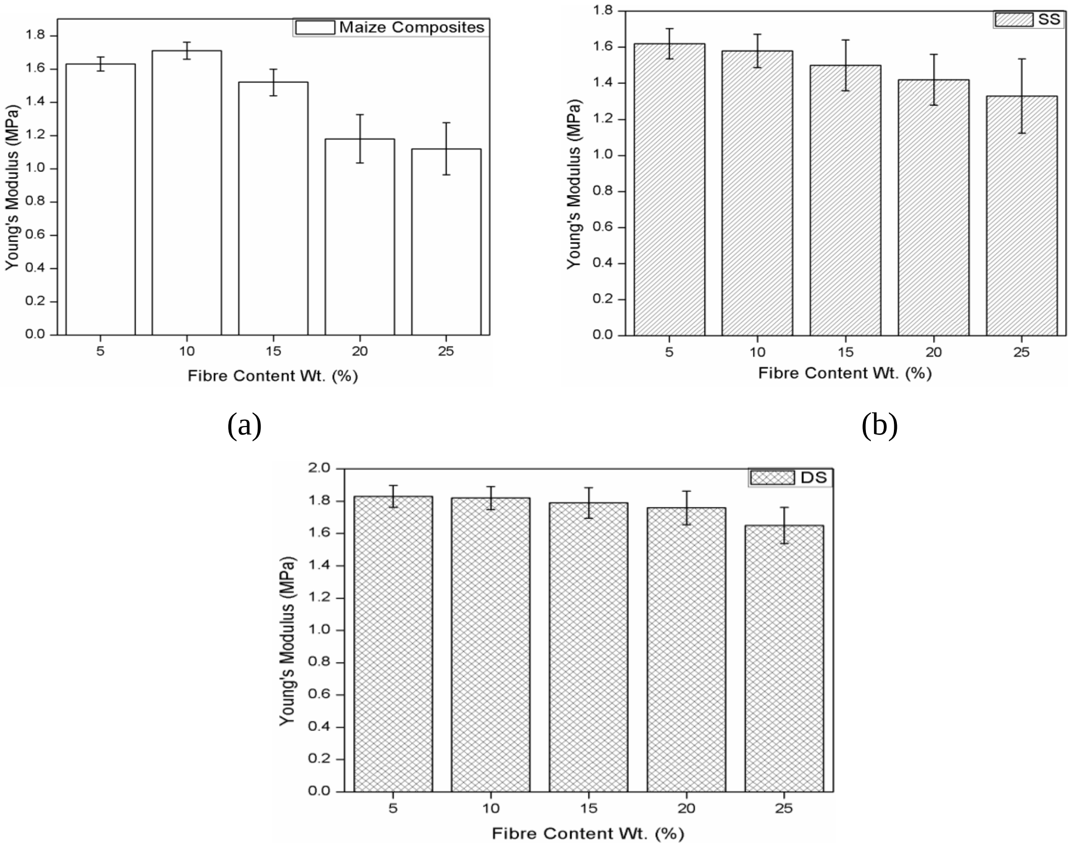
Table 4.14: Ultimate Tensile Stress Values for Maize Composites

Sl. No.	Fibre Content wt. (%)	Ultimate Tensile Stress (MPa)		
		Maize Composites	Hybrid Composites (SS)	Sandwiched Hybrid Composites (DS)
1	5	13.80	8.83	10.76
2	10	18.21	10.53	15.67
3	15	18.41	12.48	18.65
4	20	18.64	14.22	20.38
5	25	19.29	17.56	21.89

(ii) Comparison of Young’s Modulus for Maize Fibre Composites

Young’s Modulus relates tensile stress and strain value with in elastic limit. The Young’s Modulus values were calculated and plotted for maize fibre composites. The maximum Young’s Modulus of 1.71 GPa was observed for maize fibre composites (at 10% fibre content) and 1.62 GPa for hybrid composites (at 5% fibre content) and 1.83 GPa for sandwiched hybrid fibre composites (at 5% fibre content) as shown in Table

4.15 and Figure 4.35 (a to c). Sandwiched hybrid composites were processed by hand lay-up method and had a maximum Youngs modulus of 1.83 (at 5% fibre content) and a minimum of 1.65 (at 25% fibre content). The cross section of the composite is explained in morphology section 4.2.3.2.3. It can be seen in the graph the maize composite material consists of maize had lowest Young’s modulus when compared to hybrid composites and sandwiched hybrid fibre composites. In maize fibre composites, hybrid composites and sandwiched hybrid composites the Young’s modulus were decreasing as fibre content in the composite was increased as shown in Figure 4.36 (Error bar chart). This was because when fibre content was increased the elongation was excessive thus, strain value also increased and this results in turn decreased Young’s modulus values.



(a)

(b)

(c)

Figure 4.35: Young’s Modulus versus Fibre Content curve for Maize Composites (a) Maize Fibre Composite, (b) Hybrid Fibre Composite (SS), (c) Sandwiched Hybrid Composite (DS)

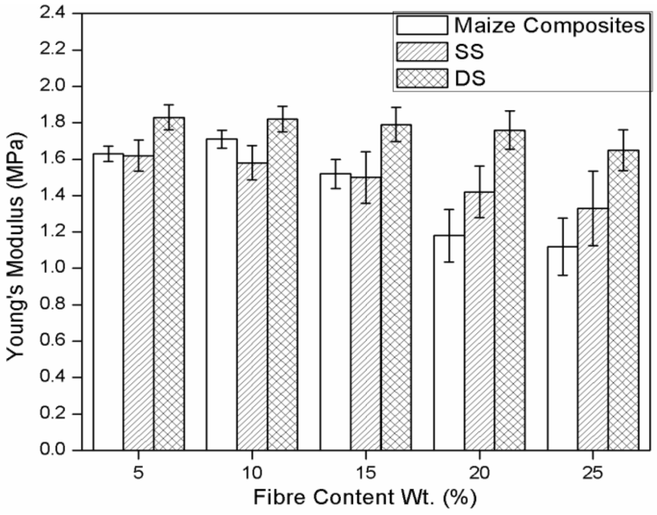


Figure 4.36: Young's Modulus versus fibre content Curve for Maize Composites (Error bar chart)

Table 4.15: Young's Modulus Values for Fibre Composites

Sl. No.	Fibre Content wt. (%)	Young's Modulus (GPa)		
		Maize Fibre Composites	Hybrid Fibre Composites	Sandwiched Hybrid Fibre Composites
1	5	1.63	1.62	1.83
2	10	1.71	1.58	1.82
3	15	1.52	1.50	1.79
4	20	1.18	1.42	1.76
5	25	1.12	1.33	1.65

(iii) Comparison of Tensile Toughness for Maize Composites

Toughness is termed as the ability of a material to absorb energy per unit volume prior to failure in the process. It was determined by measuring the area under the stress strain curve. The tensile toughness is observed by the stress strain curve of a tensile sample. Tensile toughness is plotted for maize fibre composite, hybrid fibre and sandwiched hybrid fibre composites and are tabulated in Table 4.16 and comparison of tensile toughness is shown in Figure 4.37. The tensile toughness is plotted up to break point. The actual stress strain curve for the maize composites is plotted in section 4.2.2.1.1.

Table 4.16: Tensile Toughness Values for Maize Composites

Sl. No.	Fibre Content wt. (%)	Tensile Toughness (MPa)		
		Maize Fibre Composites	Hybrid Fibre Composites (SS)	Sandwiched Hybrid Fibre Composites (DS)
1	5	23.49	23.43	26.33
2	10	34.44	25.99	50.13
3	15	39.79	40.50	72.76
4	20	47.11	59.08	87.34
5	25	48.45	64.87	96.47

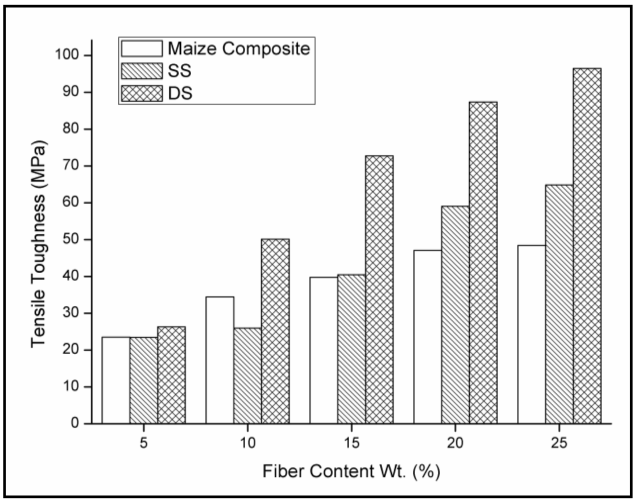


Figure 4.37: Tensile Toughness for Maize Fibre Composites

The Figure 4.37 shows that the sandwiched hybrid fibre composites (at 25% fibre content) had maximum tensile toughness of 96.47 MPa when compared to maize fibre composites having 48.45 MPa. The tensile toughness are been increased when the fibre content is also increased in the composite material. Initially at 5% fibre content hybrid composites had a low tensile toughness of 23.43MPa when compared to maize fibre composites of 23.49 MPa and sandwiched hybrid composite of 26.33 MPa, this was because of low stress values obtained in that particular composite.

Hybrid composite has higher toughness than maize fibre composite because hybrid composite consist of more than one fibre. They have high modulus fibre which provides stiffness and load bearing qualities. High strength and high modulus fibre tends to increase the composite strength with improvements in toughness. In hybrid composites (jute + maize), jute fabric is made as a backing layer, as the critical load increases with the increase of material (composite material having fine maize particles) and with the jute layer, they have high load-deflection response and it which resulted in higher toughness. Jute fabric and geometry had also played role in increasing toughness. However, fibre pull out mechanism may be directly related to composite toughness.

4.2.2.2 Flexural Test

Flexural test also termed as bending test are used to measure the flexural strength and modulus of the materials. It measures the force required to bend the material (beam) under 3 points or 4 point load. The test performed in this work was three point bending test (it consists of two support edges at end of the beam and load at the mid span). The sample was kept on the support and load was applied gradually at the centre of beam through the load nose and thus creating three point bending test as shown in Figure 4.38. The maize composite, hybrid composite and sandwiched hybrid composites were tested according to ASTM standards, ASTM D730-10 (Standard test method for flexural properties of unreinforced and reinforced plastics). The sample specification was 127mm length, 12.7mm wide and 3.2mm thick.

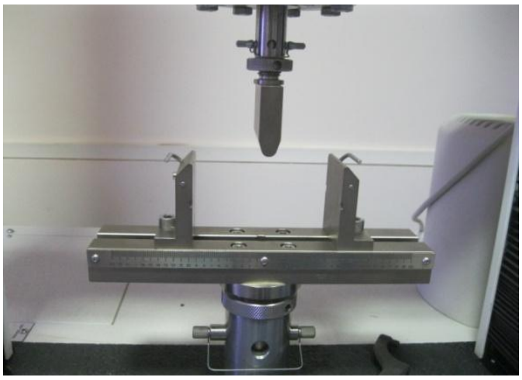


Figure 4.38: Three Point Bending Test

The stress at failure in bending is called the flexural strength and is equivalent to the modulus of rupture in bending.

Flexural strength of the composite can be obtained by using,

$$\sigma_f = {}^{3PL}/_{2bd^2} \tag{4.9}$$

Where, σ_f=Stress in the outer specimen at mid point (MPa)

P= Load at given point on the load deflection curve (N)

L=Support length of span (mm)

b=width of sample (mm)

d= depth of sample (mm)

Flexural modulus is a measure of stiffness during starting of the bending process and it can be calculated by using the equation (Jawaid et al 2010, ASTM D730-10 standards),

$$E_B = {}^{mL^3}/_{4bd^3} \tag{4.10}$$

Where E_B = Modulus of elasticity in bending (MPa)

L=Support length of span (mm)

b=width of test specimen (mm)

d=depth of test specimen (mm)

m= slope of the tangent to the initial straight line portion of the load deflection curve (N/mm)

Modulus of tangent is the ratio within elastic limit of stress to corresponding strain and the tangent line is drawn to the steepest line portion of the load deflection curve.

The rate of cross head can be calculated as $R = {ZL^2}/{6d}$ (4.11)

Where: R = rate of crosshead motion (mm/min)

L = support span (mm), d = depth of beam (mm)

Z = rate of straining of the outer fibre (mm/min), value equal to 0.01

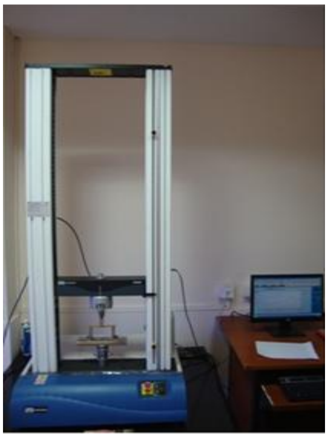


Figure 4.39

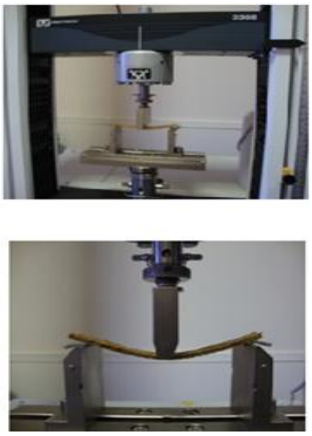


Figure 4.40

Figure 4.39: Universal Testing Machine (Three-point bending test)

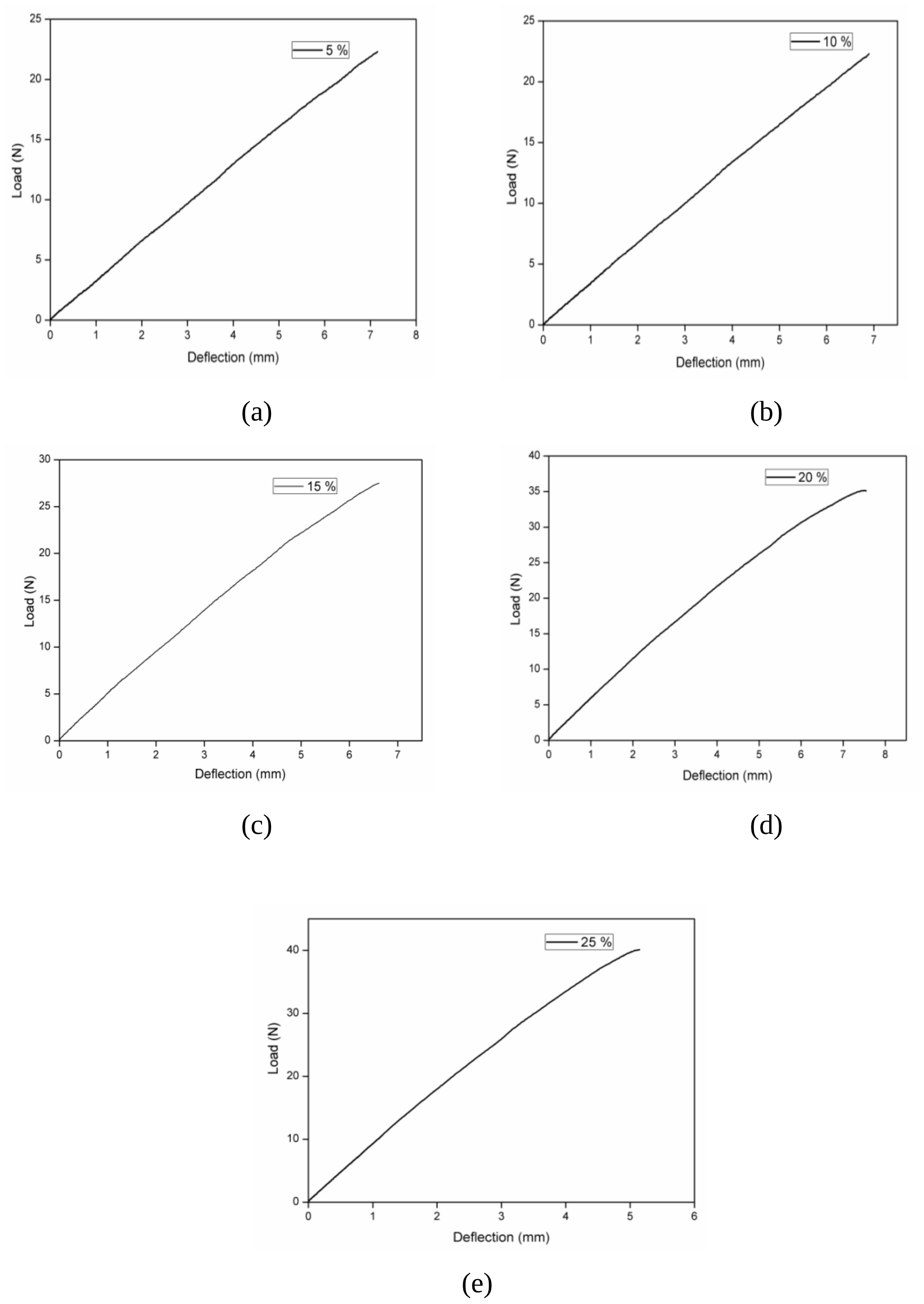
Figure 4.40: Flexural Test for Maize Fibre Composites

The samples prepared (maize fibre composites, hybrid composites, sandwiched hybrid composites) with different weight fraction of fibres and matrix ranging from 5% to 25 % (in the interval of 5%) was tested in Instron 3366 (Computerized Tensile testing machine) as shown in Figure 4.39. The specimens were kept in the fixture in tensile machine. Care was taken while placing the specimens in the fixture and in each trial the initial reading was set to zero. Test was carried out at cross head speed of 5 mm/min and load was applied gradually till the failure of the specimen as shown in Figure 4.40. Load - extension curve was obtained and the average results were plotted for different samples. The maximum deflection for maize fibre composites, hybrid composites and sandwiched hybrid composites were noted.

4.2.2.2.1 Maize Composites

Sample of maize fibre composite material was subjected to three point bending test and typical load versus deflection curves were obtained. Three point bending test requires less material for each test and it eliminates the work of finding out centre point deflection in the equipment machine. Calculations were made for the test as explained in earlier section. The load was applied in the mid of the sample and at the same deflection was noted till the specimen fails. Then the maximum load and deflection of the sample were recorded. Load versus deflection curve was plotted for flexural test and it will clearly indicate the deflection value of specimens. If we know the relation of load-deflection then the material properties such as modulus can be easily known. Materials will have different elastic modulus under bending and tension and it is necessary to know the modulus in bending for interpretation of mechanical behaviour of the material.

The flexural test was carried for neat epoxy, 5%, 10%, 15%, 20% and 25% wt. of fibre content. For neat epoxy, flexural strength and modulus value was obtained as 11.04 MPa and 0.89 GPa respectively. Individual curves for 5%, 10 %, 15%, 20% and 25 % fibre composite material are shown in Figure 4.41(a to e) and it shows the linear relationship and the values are tabulated in Table 4.17 and it was noticed that for 25% fibre content shows the maximum flexural stress with value of 46.24 MPa and load of 40.11 N with deflection of 5.14 mm. The maximum deflection was occurred for 20% wt. fibre sample reaching the value of 7.54 mm deflection. The test was carried out for five samples and the average reading was plotted for maize fibre composites. Due to lesser matrix - fibre compatibility and matix fibre interaction 20% fibre content composite material shows higher deflection and are shown in Figure 4.42.



(a) (b) (c) (d) (e)

Figure 4.41: Load versus Deflection Test for Maize Composites

(a) 5% wt. Fibre content composite, (b) 10% wt. Fibre content composite, (c) 15% wt. Fibre content composite, (d) 20% wt. Fibre content composite, (e) 25% Fibre content composite.

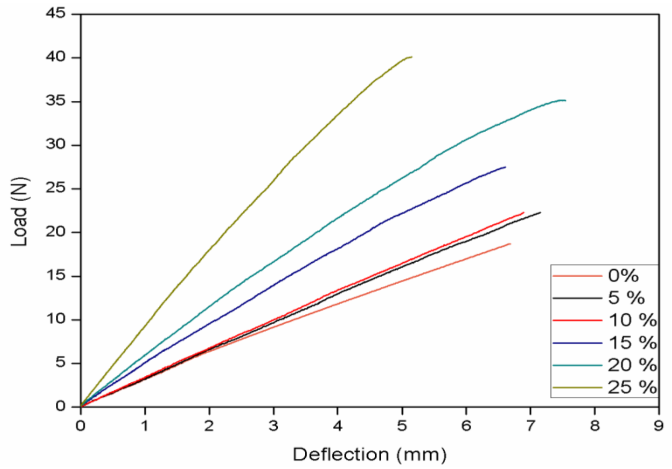


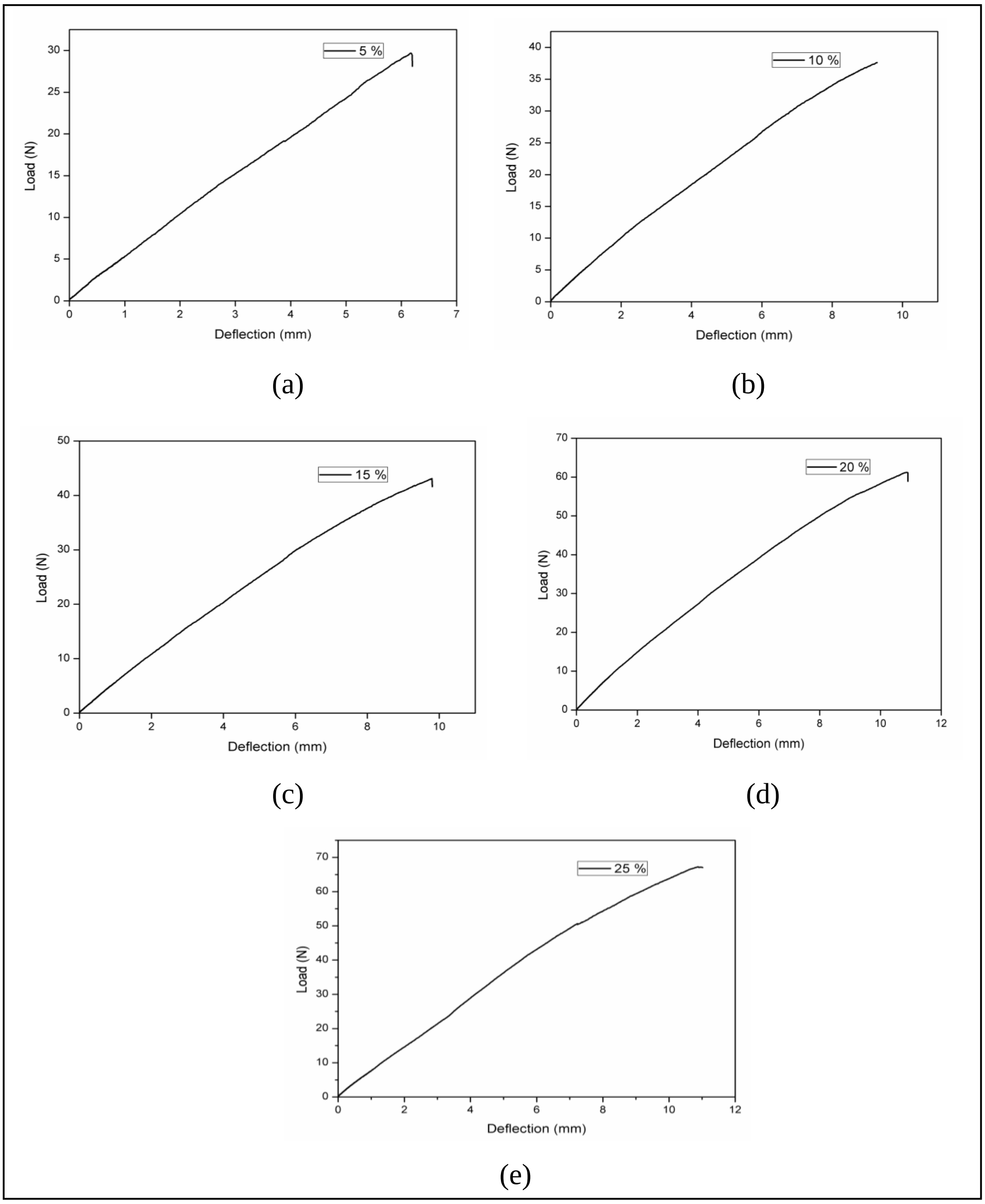
Figure 4.42: Flexural Test for Maize Composites

Table 4.17: Load Deflection Values for Maize Composites

Sl. No.	Fibre Content wt. (%)	Max. Load (N)	Max. Deflection (mm)	Max. Flexural Stress (MPa)	Flexural Modulus (GPa)
1	0	18.71	6.68	11.04	0.89
2	5	22.28	7.15	25.72	1.09
3	10	22.30	6.89	25.69	1.27
4	15	27.50	6.60	31.71	1.37
5	20	35.09	7.54	40.47	1.62
6	25	40.11	5.14	46.24	1.97

4.2.2.2.2 Hybrid Composites

Hybrid composite consist of maize fibres and jute fabric with polymer resin processed by VARTM. The maximum load and deflection data with flexural stress and modulus are tabulated in Table 4.18 and individual load- deflection curves are shown in Figure 4.43(a to e). While performing flexural test for the sample, initial crack was observed on the core part of the sample (maize) and then failure was transferred to the skin part (jute fabric mat) of the sample. This was because of loose and short maize fibre matrix bonding in the core part. The maize fibres in particulate form which made slip and generated crack in the composite. The resin and the fibres had a strong bond for with standing initial high load and failed as the crack propagated.



(a) (b) (c) (d) (e)

Figure 4.43: Load versus Deflection Test of Hybrid Composites

(a) 5% wt. Fibre content composite, (b) 10% wt. Fibre content composite, (c) 15% wt. Fibre content composite, (d) 20% wt. Fibre content composite, (e) 25% wt. Fibre content composite.

Table 4.18: Flexural Test Data for Hybrid Composites

Sl No	Fibre Content wt. (%)	Max. Load (N)	Max. Deflection (mm)	Max. Flexural Stress (MPa)	Flexural Modulus (GPa)
1	5	28.09	6.19	32.40	1.97
2	10	37.61	9.27	43.38	2.33
3	15	41.60	9.80	47.98	2.78
4	20	58.91	10.90	67.94	3.84
5	25	67.04	11.01	72.32	4.02

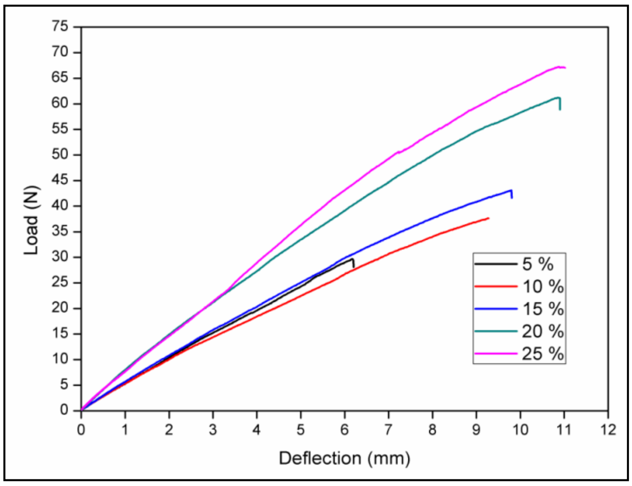


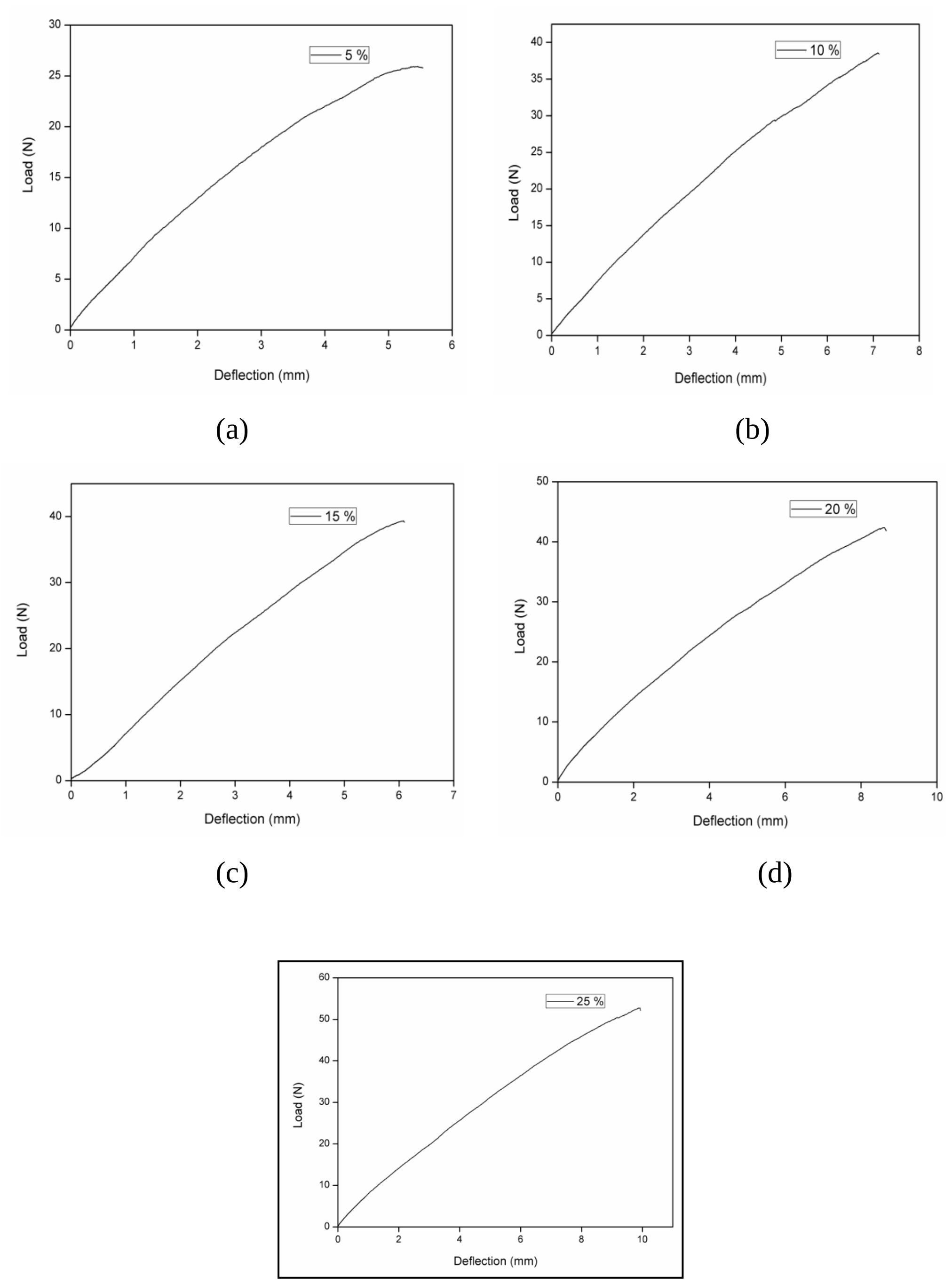
Figure 4.44: Flexural Test for Hybrid Composites

From the Figure 4.44, it can be observed that 5% fibre content sample had less load capabilities (28.09N) and deflection values (6.19 mm) when compared to the other fibre content samples. When the loads are increased at a steady rate, the other fibre content samples of 10%, 15 %, 20% and 25% composite shows the linear relationship with deflection. 25% fibre content composite had shown the maximum deflection value of 11.01 mm. At some point in the mid of the test for 25% fibre content sample composite, the flexural stress dropped (near 50 N force) due to internal phenomenon of fibre debonding in the composite. (The confirmation of this behavior is by mechanism based and the plot reveals the information. As the material is composed of hybrid composite (jute fabric and maize powders), for 25 % fibre content there was not much of bonding between fibres and matrix and hence internal debonding has occurred which resulted in minor crack and flaw, In all the performed flexural tests for hybrid composites, the initial failure occurred in the particulate of maize fibres and then in the outer layer of jute fabric as visualized). Further increase in load, the deflections are also increased to a maximum value of 11.01 mm and then failed. In general, flexural test had shown that the linearity in relation of load and deflection of the beam.

4.2.2.2.3 Sandwiched Hybrid Composites

Load deflection characteristic curves for sandwiched hybrid composites are plotted in Figure 4.45. The stresses induced in the flexural were the combination of compressive and tensile stresses. The load deflection curve for the carried work shows the behaviour of sandwiched hybrid composites. In this sample, maize fibres were in particulate form and the crack developed in the core of composite material and easily propagated to jute long fibres. In this flexural test, the crack was first observed in the maize fibre composite core part and then later in the jute fibres.

Initially for composite 5% fibre content, sandwiched hybrid composites had shown less deflection (5.54 mm) but for composites having fibre content of 25% had more deflection during break. As the fibre content increased in the composite more beam deflection had been observed. This was due to fibre matrix debonding and flexural stress induced that was higher as shown in Figure 4.46. For 25% fibre content composite the maximum load was 52.72N with deflection of 9.93 mm, flexural stress was 57.33 MPa, and flexural modulus was 4.28 GPa. For the various wt. % fibre content composites, the flexural data with flexural stress and modulus are listed in Table 4.19.



(a) (b)

(c) (d)

(e)

Figure 4.45: Flexural Load versus Deflection for Sandwiched Hybrid Composites (a) 5% wt. Fibre content composite, (b) 10% wt. Fibre content composite, (c) 15% wt. Fibre content composite, (d) 20% wt. Fibre content composite, (e) 25% wt. Fibre content composite.

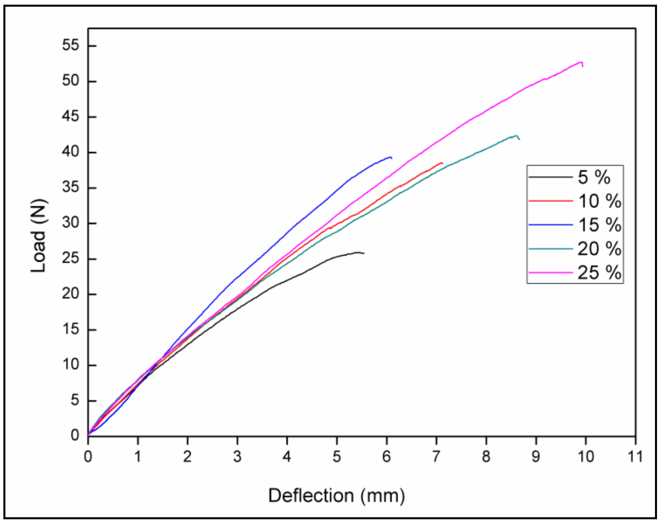


Figure 4.46: Flexural Results for Sandwiched Hybrid Composites

Table 4.19: Flexural Test Data for Sandwiched Hybrid Fibre Composites

Sl. No.	Fibre Content wt. (%)	Max. Load (N)	Max. Deflection (mm)	Max. Flexural Stress (MPa)	Flexural Modulus (GPa)
1	5	25.92	5.54	29.73	2.91
2	10	38.57	7.12	47.21	3.21
3	15	39.16	6.09	45.16	3.08
4	20	42.35	8.66	48.02	3.71
5	25	52.72	9.93	57.33	4.28

The obtained result was compared to the recent work on natural fibre composites and the results are briefed out. Kumar et al. 2014 studied banana fibre and sisal fibre composites and their effect of fibre length for natural fibre composite beams. It was noted that flexural strength (30 % fibre wt, for 3mm) for banana composite was 34.3 MPa and for sisal fibre composite 45.2 MPa.

4.2.2.2.4 Bio -Composites

The flexural test for bio polymers was carried out as explained in the above sections. The biopolymers consisted of 15% fibre content wt. (%) mixed with matrix of biopolymer “Epoxidized soy bean oil”. The flexural strength of the bio composite material was 26.96 MPa and flexural modulus of the bio-composite material was 2.6 GPa with deformation of 6.60 mm as shown in Figure 4.47. The load deflection curve for the material was almost linear till 20N of load then the values started increasing till the sample fail at 27.5N. In this case the flexural strength of bio composite was less than that of maize fibre composite material having matrix of thermoset polymer of epoxy.

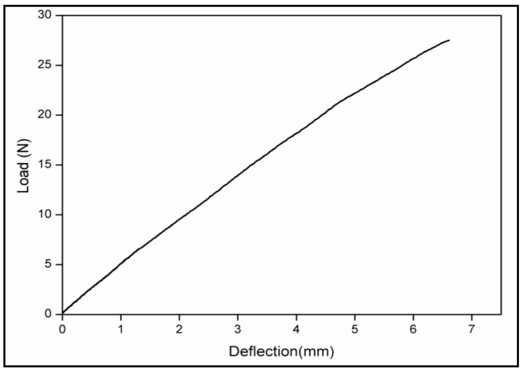


Figure 4.47: Flexural Results for Bio Composites

4.2.2.2.5 Comparison Study

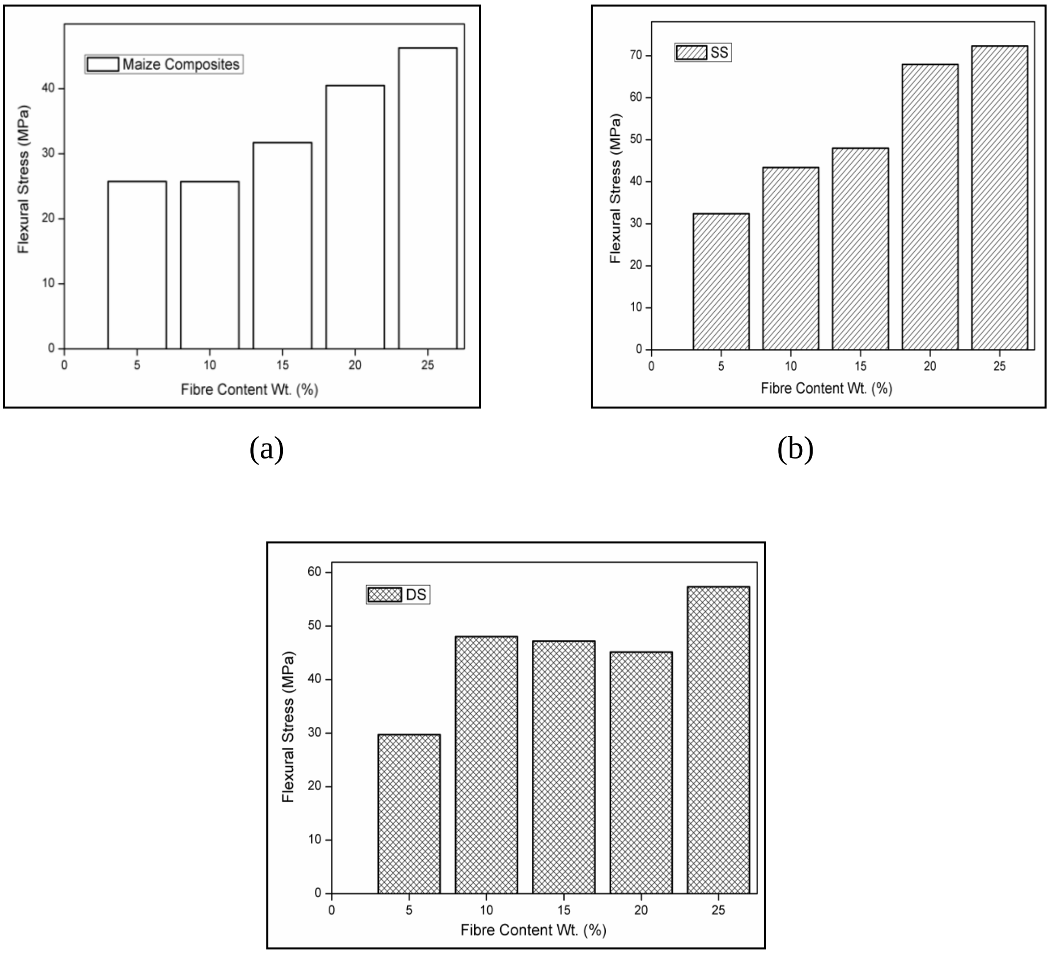
(i) Comparison of Flexural Stresses for Maize Composites

Maximum flexural stress is the highest bending stress developed in the material at the moment of its rupture. The comparison of these stresses was made for maize fibre composites, hybrid composites and sandwiched hybrid composites. When the material was subjected to flexural it experienced maximum compression stress at the concave part and maximum tension stress in the convex part of the material in three point bending. If the material was allowed to bend, then the fibres at the extreme region having larger stresses (part of the region should be free from defect) will have maximum flexural stress. The maximum flexural stress values are shown in Table 4.20 showing hybrid composites having maximum flexural stress of 72.32 MPa (for 25% fibre content) and minimum flexural stress of 25.72 MPa (for 5% fibre content) for maize fibre composites.

Table 4.20: Flexural Stress Values for All Maize Composites

Sl. No.	Fibre Content wt. (%)	Maximum Flexural Stress (MPa)		
		Maize Fibre Composites	Hybrid Fibre Composites	Sandwiched Hybrid Fibre Composites
1	5	25.72	32.40	29.73
2	10	25.69	43.38	48.02
3	15	31.71	47.98	47.21
4	20	40.47	67.94	45.16
5	25	46.24	72.32	57.33

The hybrid composites showed higher flexural stresses and dominant among the composite materials in bending test. Figure 4.48 and 4.49 shows the values for maximum flexural stresses for different fibre composites. Hybrid composites shows better values when compared to maize and sandwiched composites. In sandwiched material, the higher flexural stresses will induce on the outer layer than of the inner layer. But the failure initially occurred in the inner layer (maize fibres) than later in the outer layer (jute fabric). This was because, the maize fibres are in particulate form and the jute fibres are woven fabric. When sandwiched composites were tested for flexural test, as by visual it was observed that the initial crack took place at the maize fibres and when the load is gradually increased, the crack tend to propagate to the jute fibres and catastrophic failure occurred. This behaviour can be supported by fibre-matrix mechanism. The decrease in fibre matrix strength in the sandwiched hybrid material had caused some varied pattern in the graph and later observed some appreciable increase in stresses in the composite (due to high fibre content) sample.



(a) (b)

(c)

Figure 4.48: Maximum Flexural Stress and Fibre Content wt. (%) for Maize Composites

(a) Maize Fibre Composite, (b) Hybrid Fibre Composite, (c) Sandwiched Hybrid Composite

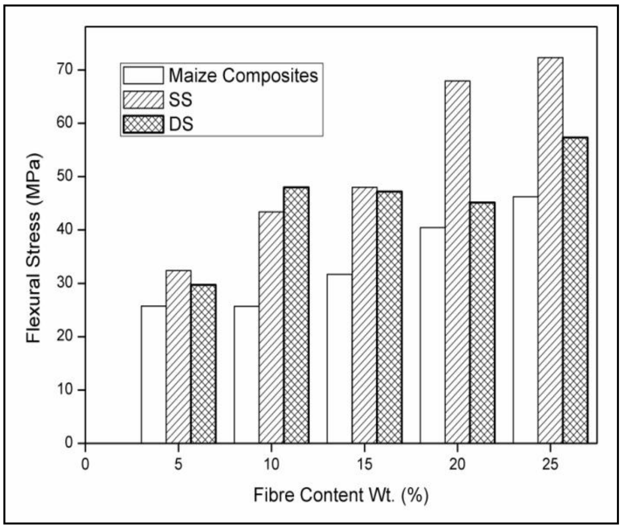
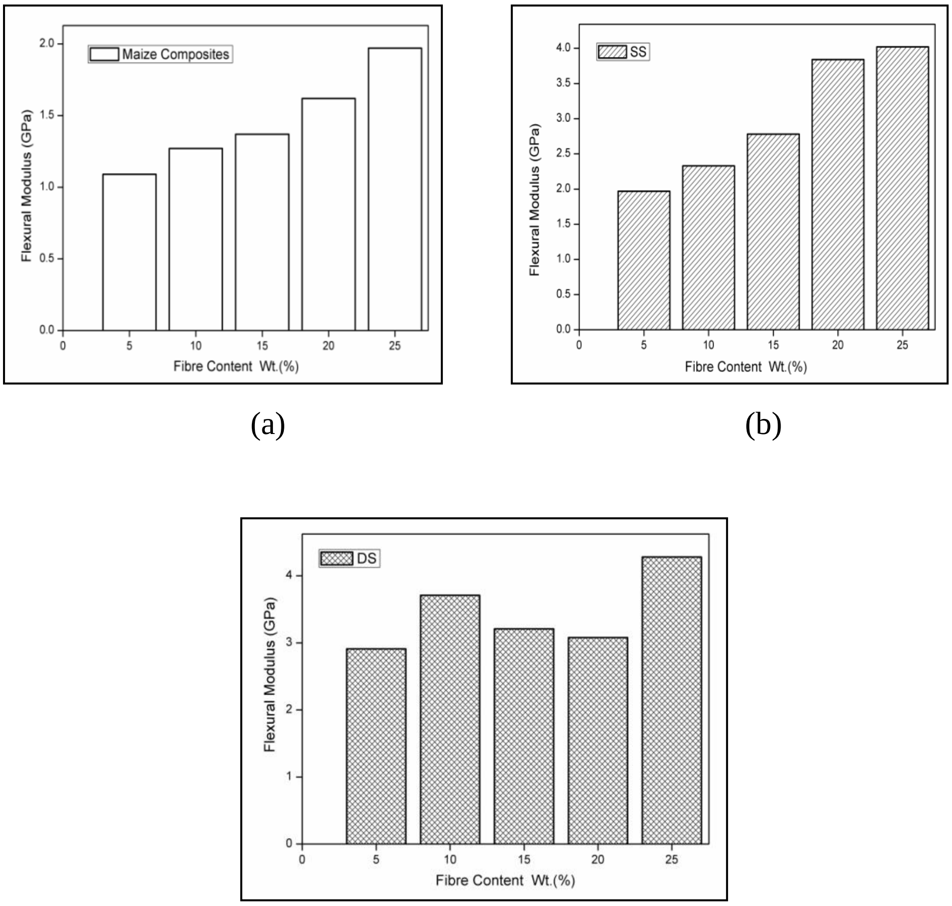


Figure 4.49: Flexural Stress Values for Unified Maize Fibre Composites

(ii) Comparison of Maximum Flexural Modulus for Maize Composites

Flexural modulus is the stiffness parameter and comparison was made for maize fibre composites, hybrid composites and sandwiched hybrid composites. Much of the explanation was briefed out in the above section. The maximum flexural modulus for each samples are tabulated in Table 4.21. Sandwiched hybrid composites observed maximum flexural modulus of 4.28GPa (for 25% fibre content) and minimum flexural modulus of 1.09 GPa (for 5% fibre content) was observed for maize fibre composites. Figure 4.50 and 4.51 shows maximum flexural modulus for different composites. Sandwiched hybrid composites had better flexural modulus value of 3.71 GPa (at 10% fibre content) and then the values started decreasing (at 15% and 20% fibre content) and again increased for 25% fibre content composite, this was probably because of lower deflection in that particular fibre content of the composites. The flexural modulus for sandwiched hybrid composite had higher value of 4.28GPa compared to other composites since the jute fibre content is considerable with maize fibre in these composites.



(a) (b)

(c)

Figure 4.50: Flexural Modulus and Fibre Content wt. (%) for Maize Composites (a) Maize Fibre Composite, (b) Hybrid Fibre Composite (SS), (c) Sandwiched Hybrid Composite (DS)

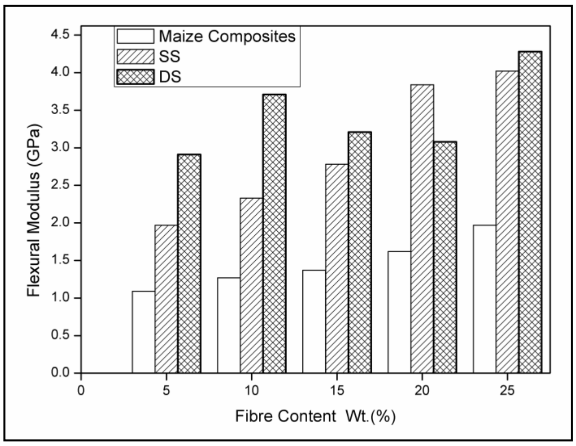


Figure 4.51: Flexural Modulus values for All Maize Fibre Composites

Table 4.21: Flexural Modulus for All Maize Fibre Composites

Sl. No.	Fibre Content wt. (%)	Flexural Modulus (GPa)		
		Maize Fibre Composites	Hybrid Fibre Composites (SS)	Sandwiched Hybrid Fibre Composites (DS)
1	5	1.09	1.97	2.91
2	10	1.27	2.33	3.71
3	15	1.37	2.78	3.21
4	20	1.62	3.84	3.08
5	25	1.97	4.02	4.28

4.2.2.3 Impact Test

Impact resistance of plastics is determined through Izod impact testing machine. Izod impact involves the kinetic energy needed to initiate fracture and continue the fracture until the sample is broken. The samples are notched and test is carried out by Izod impact method with 1 kg pendulum mass. Impact test was carried out by Impact testing machine, Zwick/Roell HIT 50P as shown in Figure 4.52 and Figure 4.53(Schematic set-up). The machine consists of base with fixture to hold specimen, pendulum and a striker rod. The machine has automatic specimen alignment and release of hammer during experiment. The Izod notched was developed as per ASTM D256 (64 x 12.7 x 3.2 mm) standard. The depth under the notch of the specimen was 10.2 mm. Samples are clamped in to the fixture in pendulum impact test machine and the notched side facing to the striking edge of the pendulum. The pendulum strikes the notched sample and required energy lost to break the sample was noted. The result was measured as energy lost per unit to cross sectional area at the notch (kJ/m^2).

$$\text{Impact strength} = \frac{\text{Absorbed impact energy (KJ)}}{\text{Cross sectional area of sample}(\text{m}^2)} \qquad (4.12)$$

Figure 4.52: Izod Impact Testing Machine

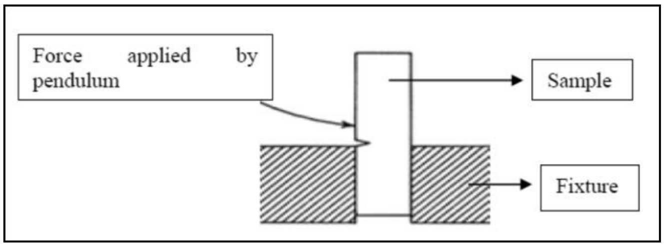
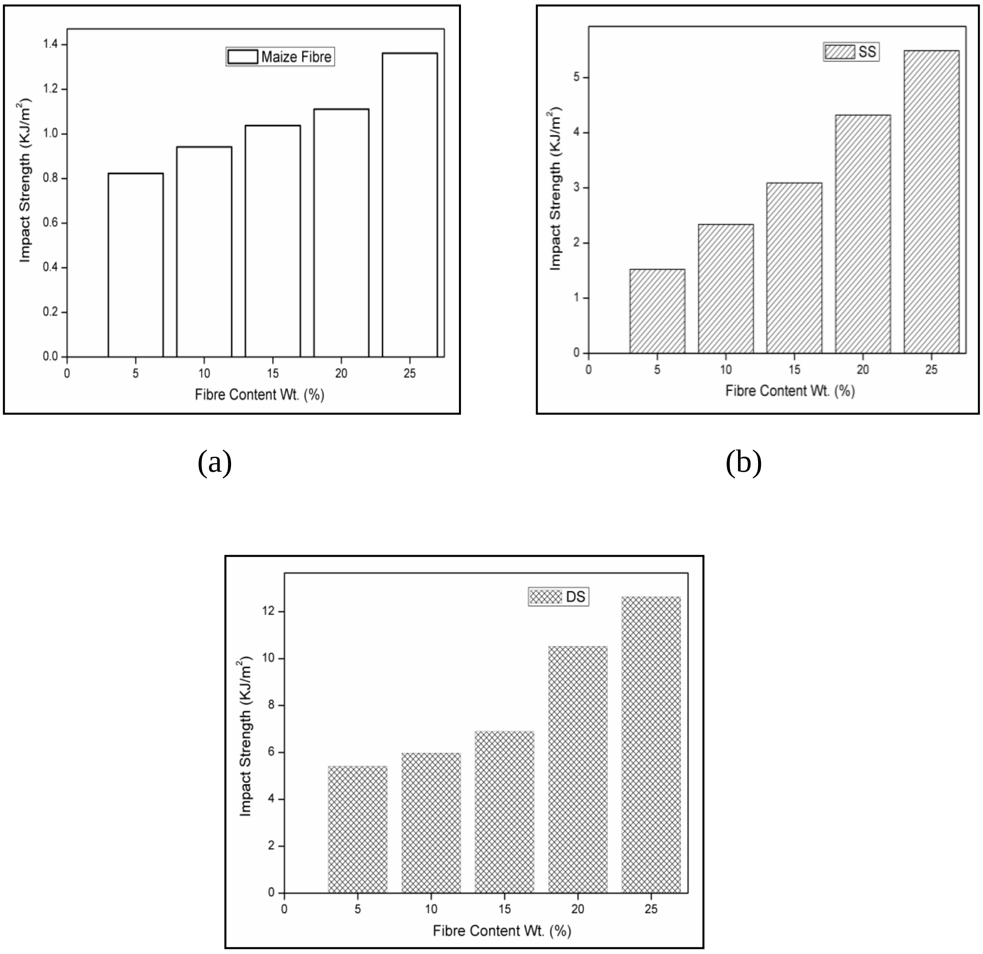


Figure 4.53: Schematic Setup for Impact Test

The impact test was carried out for maize fibre composites, hybrid composites and sandwiched hybrid composites for toughness of the material. Izod impact test having samples with V-notch and is prepared as per ASTM D256 standards. The specimens were held in cantilever manner (vertical position) as shown in Figure 4.53. Five samples of each composite material are tested and the average values are reported.

The impact strength values for maize and hybrid composites and the values are plotted for impact strength versus maize fibre content as shown in Figure 4.54 and Table 4.22. The impact strength values increased with increase in fibre content and the maximum impact strength of 1.36 kJ/m^2 was observed for 25% maize fibre composite material. The impact values indicated that they are are dependent on the amount of reinforcement material and similar rise in impact strength has been observed in other two composites. The sandwiched hybrid material attained maximum impact strength of 12.63 kJ/m^2 and 5.48 kJ/m^2 for hybrid composite material (for 25% fibre content) as shown in Figure 4.55. Comparatively, sandwiched composites had higher impact strength because in general sandwich composite has high specific stiffness and high strength. Impact properties are strongly affected by jute woven fabric structure and the thermoset resin property. The top and bottom part of the composite is normally higher strength than the core part. When a blow from pendulum is given to the composite a crack is developed and this crack travels through the fibre and matrix (resin). When the crack propagates through the composite absorption of energy will be high and hence high impact strength.



(a) (b)

(c)

Figure 4.54: Impact Strength for Maize and Hybrid Composites

(a) Maize Composite, (b) Hybrid Composite, (c) Sandwiched Hybrid Composite

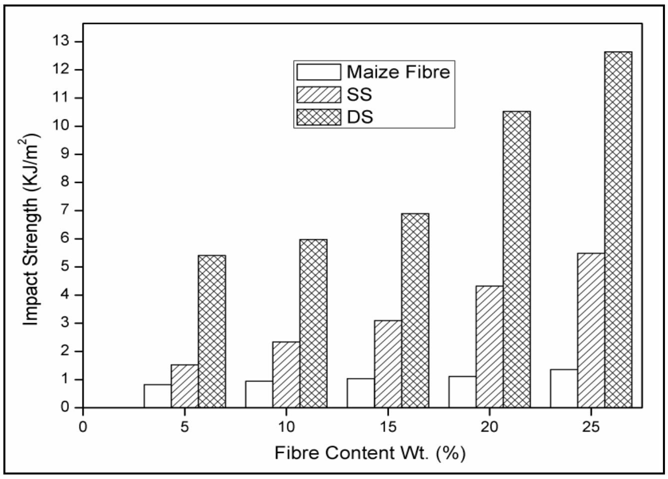


Figure 4.55: Impact Strength of All Maize Fibre Composites

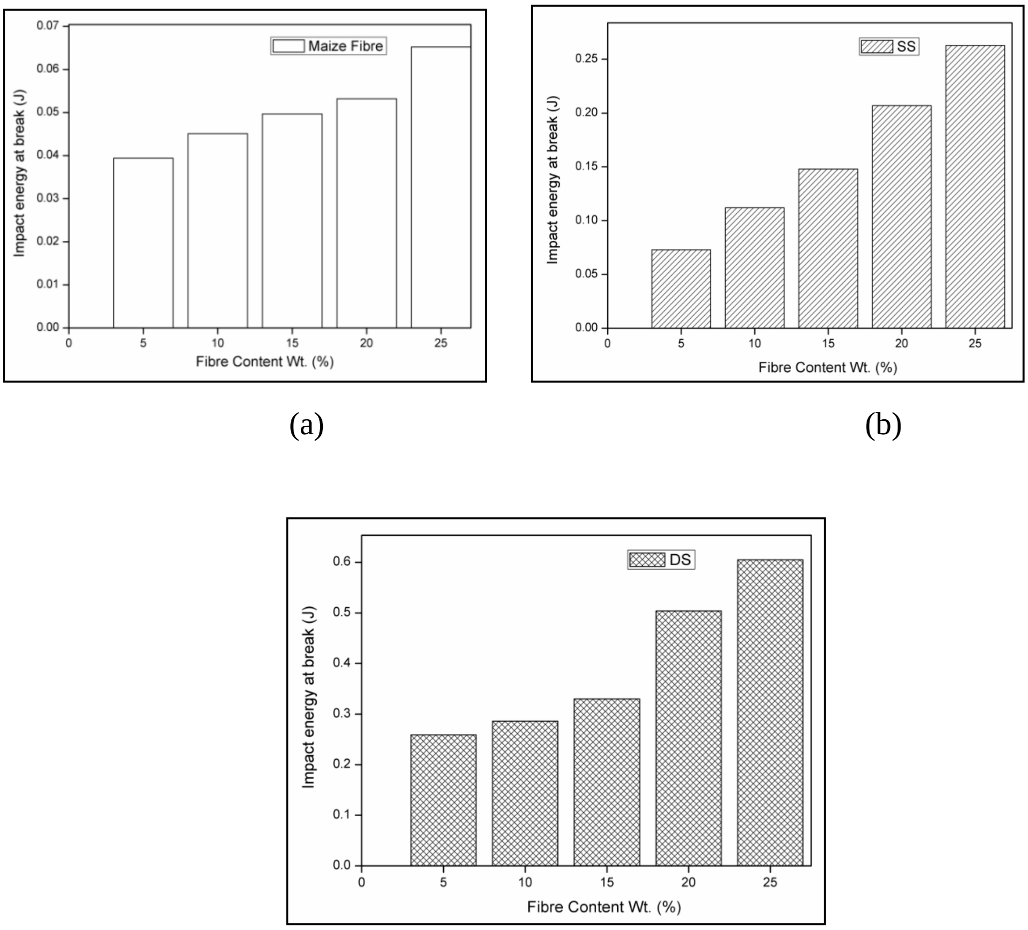
Table 4.22: Impact Strength Values for Maize and Hybrid Composites

Sl. No.	Fibre Content wt. (%)	Impact Strength (kJ/m^2)		
		Maize Fibre	Hybrid Composite	Sandwiched Hybrid Composite
1	5	0.82	1.52	5.40
2	10	0.94	2.33	5.97
3	15	1.03	3.09	6.89
4	20	1.11	4.32	10.52
5	25	1.36	5.48	12.63

The impact energy values for maize and hybrid composites are shown in Table 4.23 and the obtained values are plotted for impact energy at break (J) versus maize fibre content wt. (%) as shown in Figure 4.56. The impact energy at break strength values increased for increased fibre content in a step wise manner and the maximum impact energy at break strength of 0.605 J was observed in sandwiched hybrid composites (for 25% fibre content) and low value of 0.039 J (for 5% fibre content) was observed in maize fibre composites. Figure 4.57 shows the unified values of impact energy at break for all maize fibre composites.

Table 4.23: Impact Energy at Break for Maize and Hybrid Composites

Sl No	Fibre Content wt. (%)	Impact Energy at Break Strength (J)		
		Maize Fibre	Hybrid Composite	Sandwiched Hybrid Composite
1	5	0.039	0.073	0.259
2	10	0.045	0.112	0.286
3	15	0.049	0.148	0.33
4	20	0.053	0.207	0.504
5	25	0.065	0.262	0.605



(a) (b)

(c)

Figure 4.56: Impact Strength for Maize and Hybrid Composites

(a) Maize Composite, (b) Hybrid Composite, (c) Sandwiched Hybrid Composite

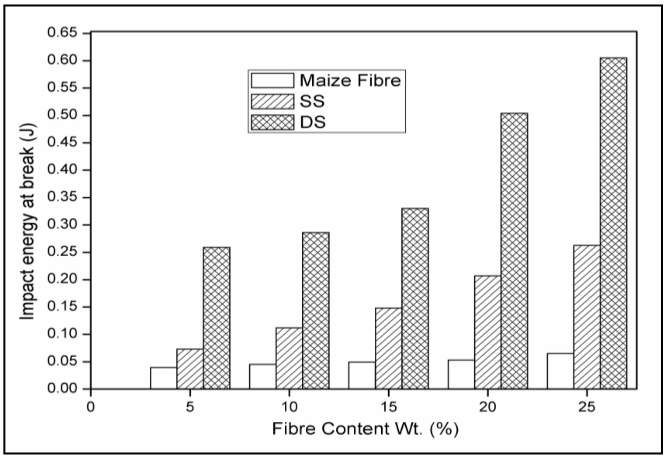


Figure 4.57: Unified Impact Energy at Break of Maize Fibre Composites

4.2.2.4 Hardness Test

Hardness test are useful in material evaluation for manufacturing process. Indentation test are usually carried out to determine the hardness of a material to deform. The term micro hardness refers to the static indentations made with loads not exceeding 1kgf. Vickers and knoop hardness are familiar method for carrying out micro hardness test. The hardness test was carried out with microscopic scale with higher precision instruments. Micro hardness test are small indentations of micron level and are used to determine the hardness of the material to deformation and the force applied is only for 20 seconds. The average length of the diagonals of the indent base is measured and hardness is calculated. It is capable of determining the hardness of the constituents within the structure. The indenter is kept in such a way it would first strike the fibre/matrix part of the composite.

(a) Vickers's Micro Hardness

Vickers's Micro hardness is common method to measure the micro hardness. The maize composite sample was measured out by Clemex micro hardness tester, MMT X7, Matsuzawa Seiki Corp., Japan and it employs a small pyramid shape diamond pyramidal indenter with an apical angle of 136º as shown in Figure 4.58. It is pressed in to the sample at a predetermined load and the indentation is measured in both axis from tip to tip. The axis measurements are converted to Vickers hardness number as given below.

Vickers's hardness number (VHN) was calculated by using the formula.

$$HV = \frac{2F\sin\frac{136^\circ}{2}}{d^2} \tag{4.13}$$

$$HV = \frac{1.854F}{d^2} \tag{4.14}$$

$$\text{And,}\quad d = (d_1+d_2)/2 \tag{4.15}$$

Where, F is the applied load (kgf)

d is the arithmetic mean of two diagonals

d_1 is the horizontal length (mm)

d_2 is the vertical length (mm).

1 HV (Vickers hardness) = 9.8 MPa

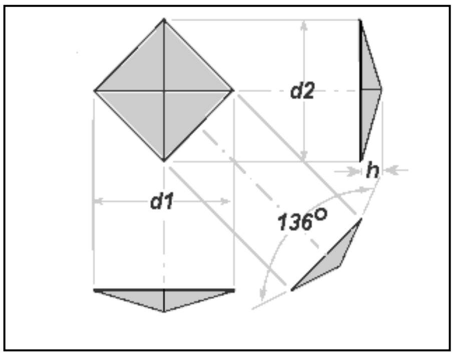
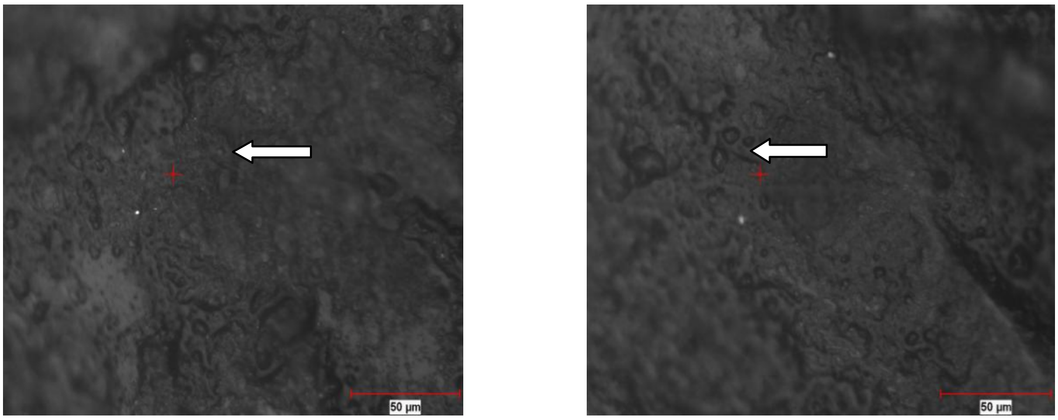


Figure 4.58: Vickers Pyramid Diamond Indentation

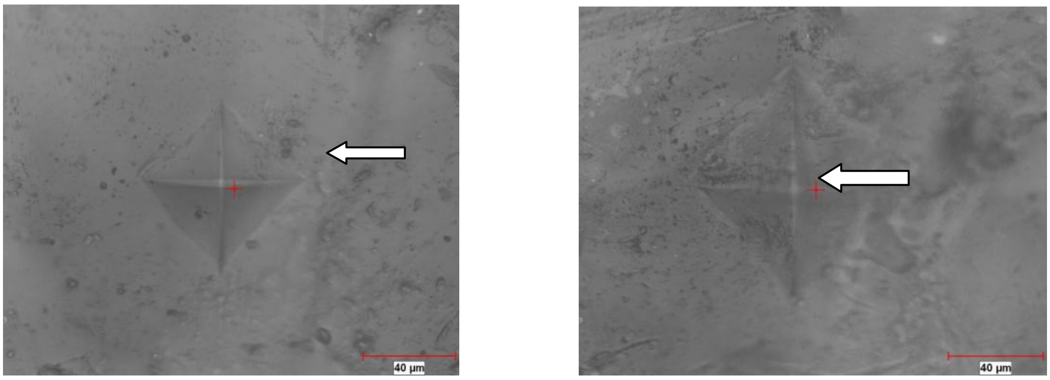
A diamond indenter is placed and a load F is forced into the sample. When the load is removed the two diagonals of the indentation left on the surface of the sample are noted and their arithmetic mean d is calculated. For the present work a load F= 50 gf (0.49N) and dwell time of 10 second was considered. Vickers hardness values are normally expressed as a number only.

High Hardness value can be observed in the area where there are large amount of fibres concentrated on a particular location. The applied stress in the distributed fibres will be more and hence more hardness value. If there are some non distributions of fibres or coarse fibres content then the results may vary. The indentation images for 15 % fibre content composite are shown in Figure 4.59. There is a clear view of pyramidal indenter image seen on the material (indicated by arrow mark). The material was subjected to different load with 10 gf, 25 gf, 50 gf, 100 gf with hardness value of 10 HV, 13HV, 17HV and 18 HV. The higher the load the sample will give good result because it gives larger indentation and good measurement resolution values.



10gf, 10HV

25gf, 13 HV



50gf, 17HV

100gf, 18HV

Figure 4.59: Vickers Hardness Indentation Images of 15% Maize Fibre Composites for Different Forces

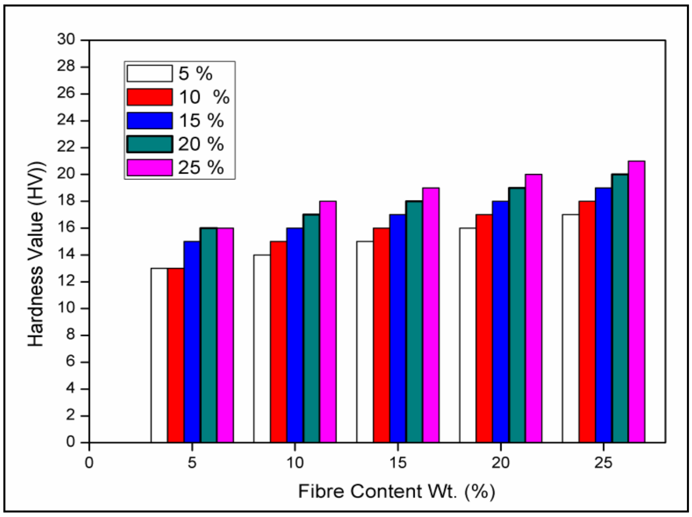


Figure 4.60: Hardness Values for Maize Fibre Composites for Different Fibre Content wt. (%)

If the composite material had good surface finish, smaller load was sufficient for getting better results. If the modulus of the composite material is increased then the hardness of the composite value are also increased, this is due to the reason that hardness is a function of fibre volume and modulus. It can be seen that when the fibre content weight percentage was increased, the hardness value are also increased. From the Figure 4.60, it had been seen that minimum value was observed in maize composite (5% fibre content composite sample) with hardness value of 13HV and Maximum hardness value for 25% fibre content composite sample was 21HV. The increase was almost in step wise increase in values and 15 % fibre content sample composite gave a better average result.

(b) Shore D Hardness test

Durometer is one of the measures for hardness of material. The testing was carried out by Shore D hardness (Digital durometer) tester with pocket sized model (Mextech Instrument Company, Model: HT-6510D) as shown in Figure 4.61. The maize composite samples were placed on a flat surface and the tester was kept in vertical position and it is forcibly pressed for few seconds until it reaches a maximum value. Ten readings at different points are noted and average value is taken.

Figure 4.61: Shore D Hardness Tester

The Shore D hardness values are high for sandwiched hybrid composite and low hardness value for maize composite. When there are more fibres focussed on a particular location it gave maximum value and low fibres in the region with minimum value. The values are dependent on surface matrix fibre bonding of the composite material and the average values are shown in Table 4.24.

Table 4.24: Shore D Hardness Value For Maize and Hybrid Composites (15% fibre content)

Sl. No.	Shore D Hardness Number		
	Maize Composite	Hybrid Composite	Sandwiched Hybrid Composite
1	67	73	82

4.2.2.5 Water absorption (Moisture) test

Water absorption test also termed as moisture test is used to determine the amount of water absorbed under specified conditions. The test was conducted as per ASTM-D570 (2010), standard method for water absorption of plastics. The samples are dried in an oven at 60 ºC for 12 hours and then placed in a desiccator to cool. After cooling the samples are weighed and put in water at 23ºC for 24 hours. Later samples are removed, cleaned and weighed. The samples were approximately cut to 76 x 25 x 3.2 mm using water jet machining. Later for every 24/ hrs (1 day) the test is repeated for 10 days till the equilibrium stage is reached. The percentage of moisture absorbed is expressed as the ratio of increase in mass of the specimen to the initial mass as shown below.

The percentage increase in weight during immersion

$$M_t = \frac{W_t - W_o}{W_o} \times 100 \qquad (4.16)$$

Where, M_t = Percentage of moisture absorbed

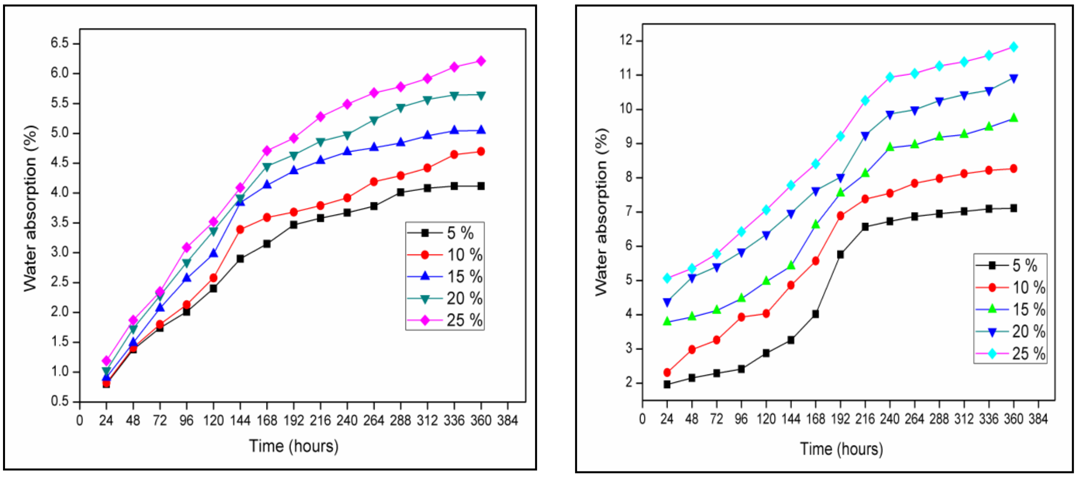
W_t = Weight of the composite at time (t) (wet weight)

W_0= Conditioned weight (dry weight)

Water absorption test was carried for three different composite materials namely maize fibre composites, hybrid fibre composites and sandwiched hybrid composites. The test was conducted as per ASTM 570, initial weight was noted and the material was immersed in water for 24 hours and the sample is weighed again and difference in reading was noted. The procedure was continued for the same sample with wetting the composites in water for ten days or till the saturation point. For every 24 hours, the change in material weight was examined and the values are noted. The moisture absorption values obtained are plotted for different composites and are shown in Figure 4.62. Among them, sandwiched hybrid material with 25% of fibre content had more water absorption percentage of 15.55% and maize fibre composites had least water absorption of 4.11% (for 5% fibre content) and the values are tabulated in Table 4.25.

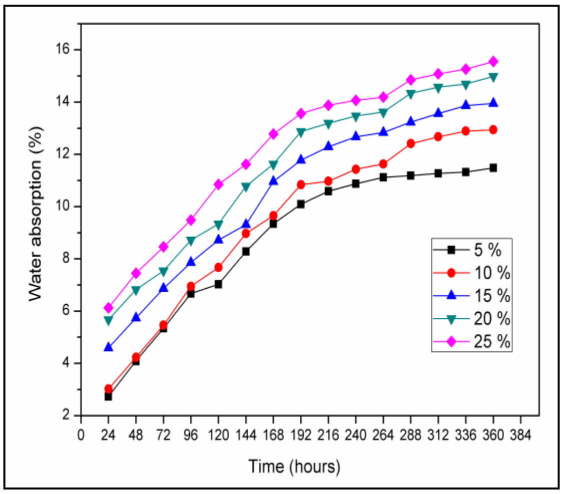
Table 4.25: Water Absorption for Maize and Hybrid Composites

Sl. No.	Fibre Content wt. (%)	Maxmium Water Absorption (%) in10 days (240 hrs)		
		Maize Composite	Hybrid Composite	Sandwiched Hybrid Composite
1	5	4.11	7.11	11.48
2	10	4.29	8.27	13.19
3	15	5.04	9.73	13.95
4	20	5.64	10.93	14.98
5	25	6.21	11.83	15.55



Maize Composites

Hybrid Composites



Sandwiched Hybrid Composites

Figure 4.62: Moisture Test for Maize, Hybrid and Sandwiched Composites

For maize composites with 25% fibre content, the maximum water absorption value was and the percentage values increased almost linearly and for the first 3 days the values raised abruptly and the at most value reached was 6.21% of water absorption. It has observed that the moisture absorption of maize fibre composites are low. For hybrid composites, the reinforced of maize and jute fibres increased the water absorption value to 11.83% (for 25% fibre content) which was maximum and 7.11% (for 5% fibre content) was the minimum value. This is because in hybrid material of jute fabric mat absorbs more moisture and wetting action is more than that of maize fibres. Jute fibres are prone to water absorption and the wetting mechanism is quicker and faster. In 15%, 20% and 25% fibre content the composite material had a drastic change in values in the mid days of water absorption period and for the last three days the absorption percentage values raised.

The similar behaviour was observed in sandwiched hybrid composites, as it consists of jute fabric on both sides, more water absorption was occurred. For the last 4 days, 20% and 25% fibre content shows fluctuated values with value of 14.98% and 15.55%. Whereas for 10% and 15% fibre contents, the absorption value was 13.19% and 13.95% which was nominal value for jute composite material. For 5% fibre content sandwiched composite, values are initially increased linearly and decreased for the third day and further increased for the final days reaching the water absorption value of 11.48%.

Water absorption is higher in the composite having higher % of fibre because natural fibres are hydrophilic in nature and the water absorption is directly proportional to the fibre loading of composites. They are lignocellulosic and have strong presence of polarized hydroxyl bonds. Water absorption is due to presence of lumens, fine pores, hydrogen bonding in the packed material, gaps in the interfaces and micro cracks in the matrix. Cellulose and hemicelluloses are responsible for high water absorption of natural fibres (they contain more hydroxyl groups). In the present work as jute fibres are present more, it may lead to more water absorption factor. As the fibre load increases cellulose content also increases, which results in more absorption of water.

Generally, natural fibres are hydrophilic and they have a tendency to absorb moisture more in a short time. To make hydrophobic chemical treatments are performed such that the wetting action becomes poorer. In raw fibre composite material, the fibres absorb water easily when submerged and hence water adsorption characteristics become dominant. More water absorption characteristics make the composite material to be weak in strength and results in poor mechanical properties.

Material can also gain weight if they are reacted with the moisture in atmosphere or in working environment. As the maize fibres and jute fibres are treated with chemicals they had lesser water absorption characteristics and expected low moisture content. Chemical treatment of fibre and VARTM process results in better interaction of fibre and matrix thereby increasing area of contact without voids or pockets thus enhanced tensile and flexural strength. If there are increased voids and more cellulose content, moisture absorption will be more in composite material and swelling also takes place resulting of low shear stress at the interface that is what happened in hybrid sandwiched composite. This water absorption can be reduced by increasing the interface between fibre and matrix without voids by chemical treatment.

4.2.3 Characterization Tests

Morphological test, thermal test (Differential Scanning Calorimetric, Thermal Gravimetric Analyser), X-Ray Diffraction test, Fourier Transform Infrared Spectroscopy (FTIR) were carried out for these maize fibre composites and the details are explained below.

4.2.3.1 Fibre -Matrix Interface

To achieve good bonding, factor of fibre matrix interface is essential and here the stresses are adequately transferred to the reinforcement providing true reinforcing functions. If the interfacial adhesion breaks down, it allows various toughening mechanisms to become operative. These mechanisms include crack blunting, frictional sliding and debonding fibre fragments within the matrix and fibre fracture. As natural fibres are hydrophilic, they are allowed for chemical treatment analysis such as alkali treatment and acetylation treatment for achieving the composite material as a hydrophobic material. Figure 4.63 shows the microscopic surface image of maize composite material and it shows the presence of globule pattern of thermoset epoxy matrix mixed with the pulverized fibres.

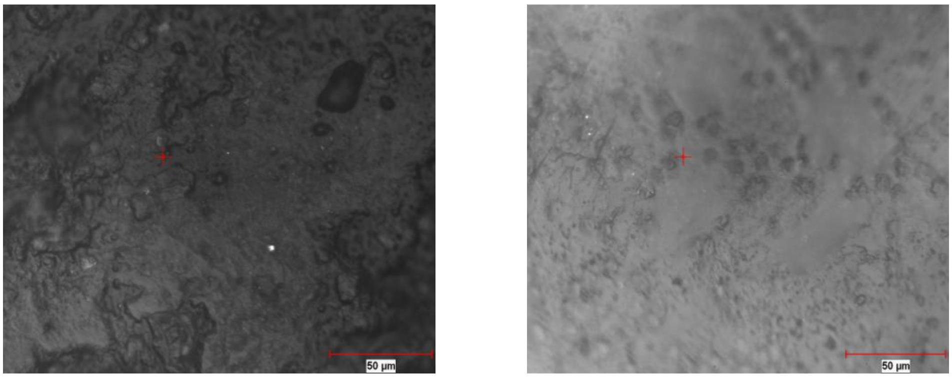


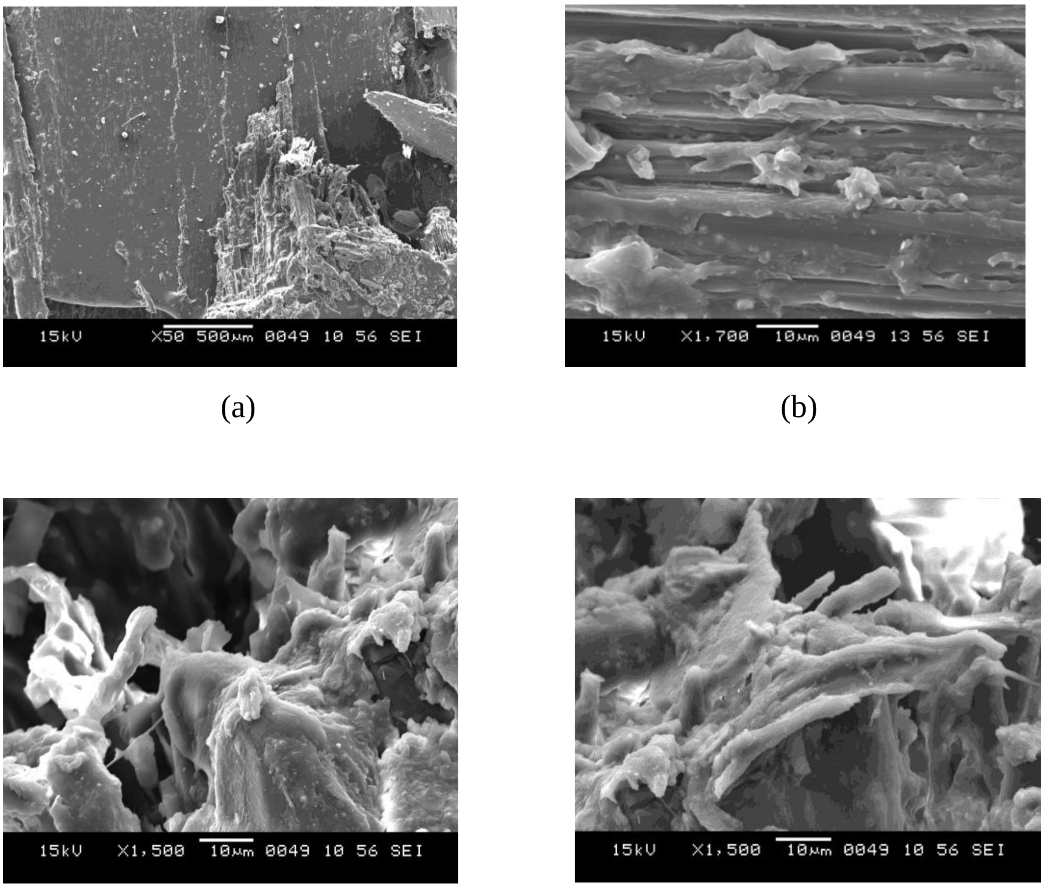
Figure 4.63: Photomicrograph Surface Image of Maize Composites

4.2.3.2 Morphological Analysis Using Scanning Electron Microscope

Morphological analysis and characteristics of treated maize fibre composite, hybrid composite (maize fibre + Jute fabric mat), sandwiched hybrid composite (maize fibre + Jute fabric mat), fractured composites and bio composites were analyzed by Scanning Electron Microscope (SEM). The studies are important to predict fibre interaction with the polymer matrix in composites. These SEM micrographs of the composites show a clear difference in the morphology of the resin and fibre.

4.2.3.2.1 Maize Composites

Figure 4.64 (a to d) shows the maize fibre and matrix are well blended with the resin and it also showed the characteristic intermingling of fibres and matrix thus producing good interfacial adhesion in the composites. The structural changes that expected in chemical treatment were the diameter of the maize fibres reduced and the hemicelluloses, lignin constituents were partly removed. In Figure 4.64(a and b) the surface of the maize composite is shown with globules of polymers settled in the surface.In Figure 4.64(c and d) the micrograph depicts the mixing of resin and maize fibres and the polymeric resin were binded properly. In both chemical treatment methods, the surface morphology, crystalline structure of cellulose of maize fibre, and other constituents of the fibre changed, thus converting the maize fibre composites to hydrophobic material.



(a) (b)

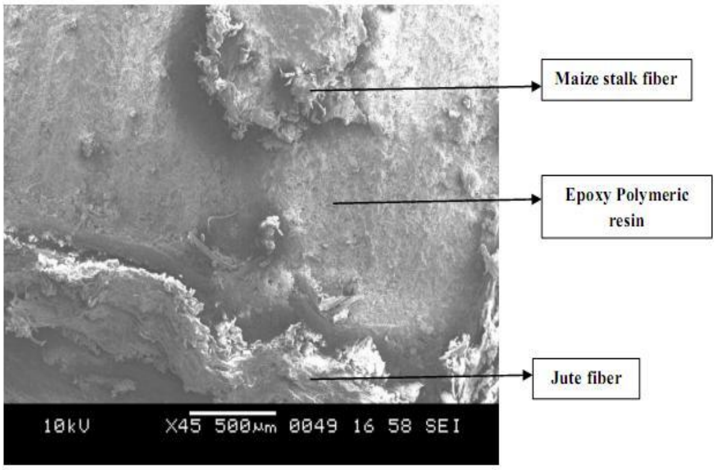
(c) (d)

Figure 4.64: SEM Micrograph for Maize Composites

(a, b, c, d) Maize fibre composite chemically treated with polymeric resin

4.2.3.2.2 Hybrid Composites

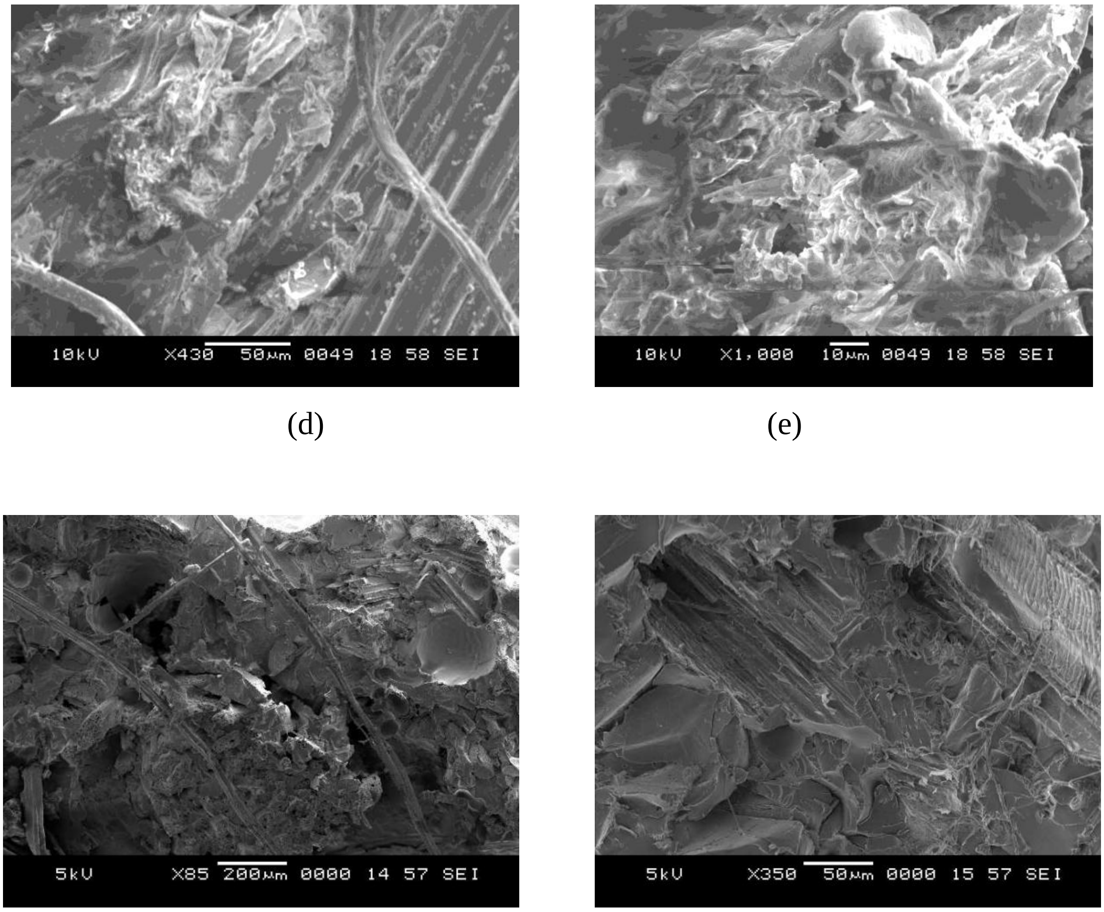
Morphological and characteristic studies of the hybrid composite fibre material consisting of maize and jute mat were analysed by SEM. From the Figure 4.65(a) it can be reported that the rough surface of the hybrid fibre resulted in better wettability and mechanical bonding with the epoxy polymer (Because some impurities located on the surface of maize fibre and the modification of fibre effectively removed the impurities of the fibre surface). It also showed the minglement of jute and maize fibre and with polymeric resin and the wetting of fibres and adhesion of fibres with polymeric matrix was better. In Figure 4.65(b and c) shows the interlacing of fibres in the composite material and fibrillation of fibres was also found out as shown in arrow mark. Features like twinning, interlacing and cross sheathing of the fibres can be seen in Figure 4.65(d and e) and the jute fibres are cross linked to the maize fibres as shown in Figure 4.65(f and g). From the SEM observations it can be concluded that jute fibres are well systematic bonded with the maize fibre and also with epoxy polymeric resin and have achieved better adhesion interfacial characteristics.



(a)

(b)

(c)



(d) (e)

(f) (g)

Figure 4.65: SEM Micrographs of Hybrid Composites

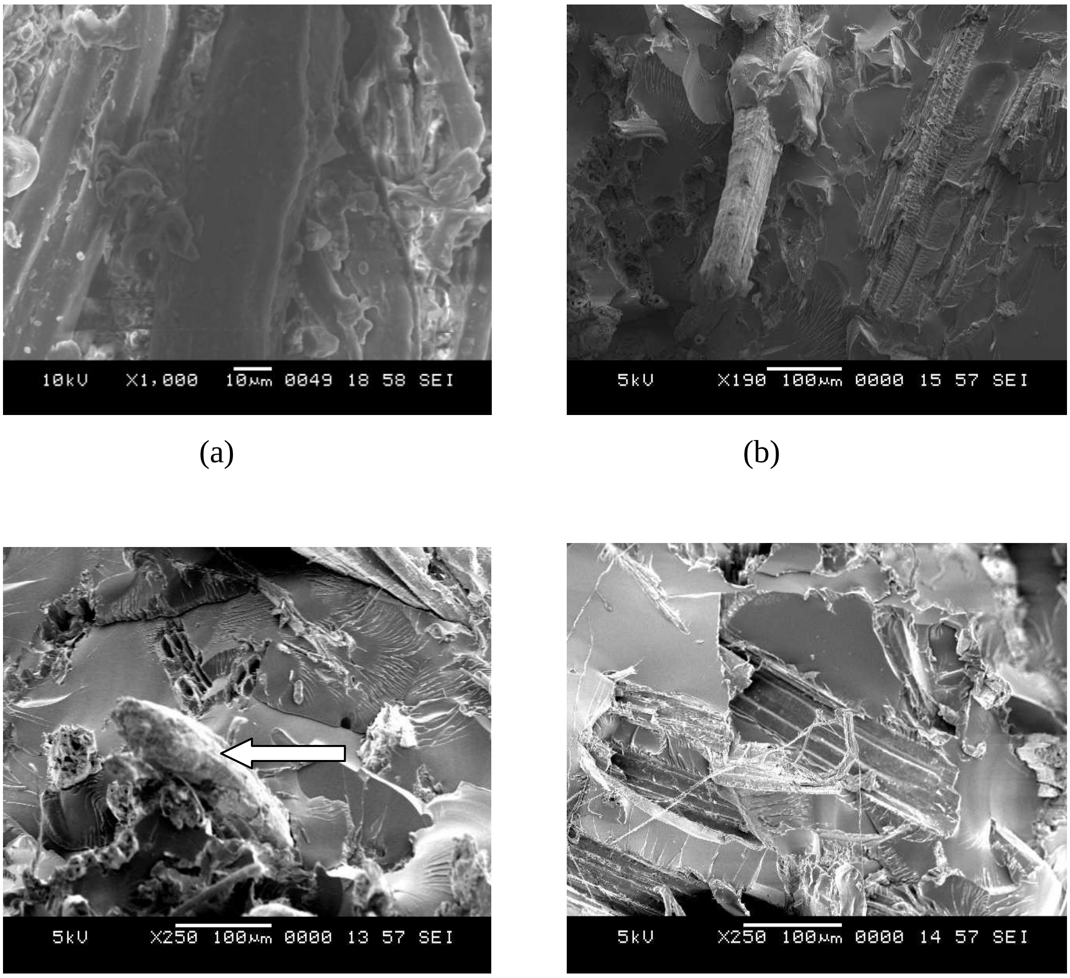
(a) Maize and Jute fibres mixed with polymeric resin, (b, c) Fibrillation of jute fibres, (d, e) Twinning and cross sheathing of fibres, (f, g) Epoxy polymeric resin mingled with jute fibre.

4.2.3.2.3 Sandwiched Hybrid Composite

In Sandwiched hybrid composites the surface material had covered with resin and fibres of maize and jute as shown in Figure 4.66. The wetting phenomenon had taken place good due to the chemical treatment of fibres as shown in Figure 4.66(a) and good compatibility was achieved. In Figure 4.66(b, c) shows the presence of fibres in the matrix and the surface of the fibres showed some micro ridges in the composite material.

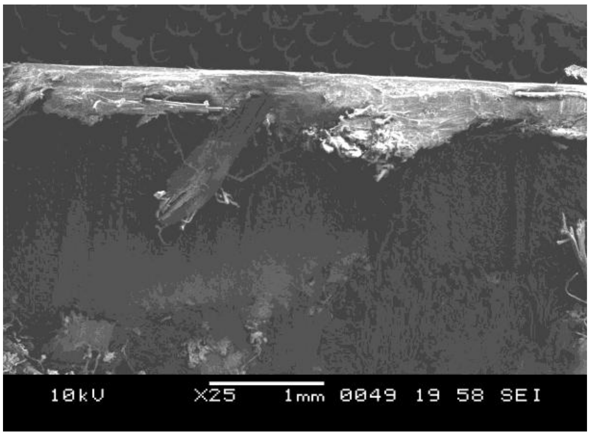
Jute fibres were better dispersed and in chemical treatment the fibres must have split out resulting in thin fibrillation and thin amount of fibres were thrown out as seen in Figure 4.66(d). Splitting of fibres into filaments and breaking the fibres in to smaller fibrils increases the effective surface area, improves contact with the matrix and provides good wetting mechanism. When the waxy layer is removed (during chemical treatment) in fibres, they can expect some increase in roughness and mechanical strength.

Cross section of the sandwiched hybrid composite is shown in figure 4.66(e) with jute fabric and maize composites. The sectional view shows good interaction between fibres and matrix. Smooth fractured surface was encountered and it clearly shows a proper fibre matrix adhesion in the composite.



(a) (b)

(c) (d)



(e)

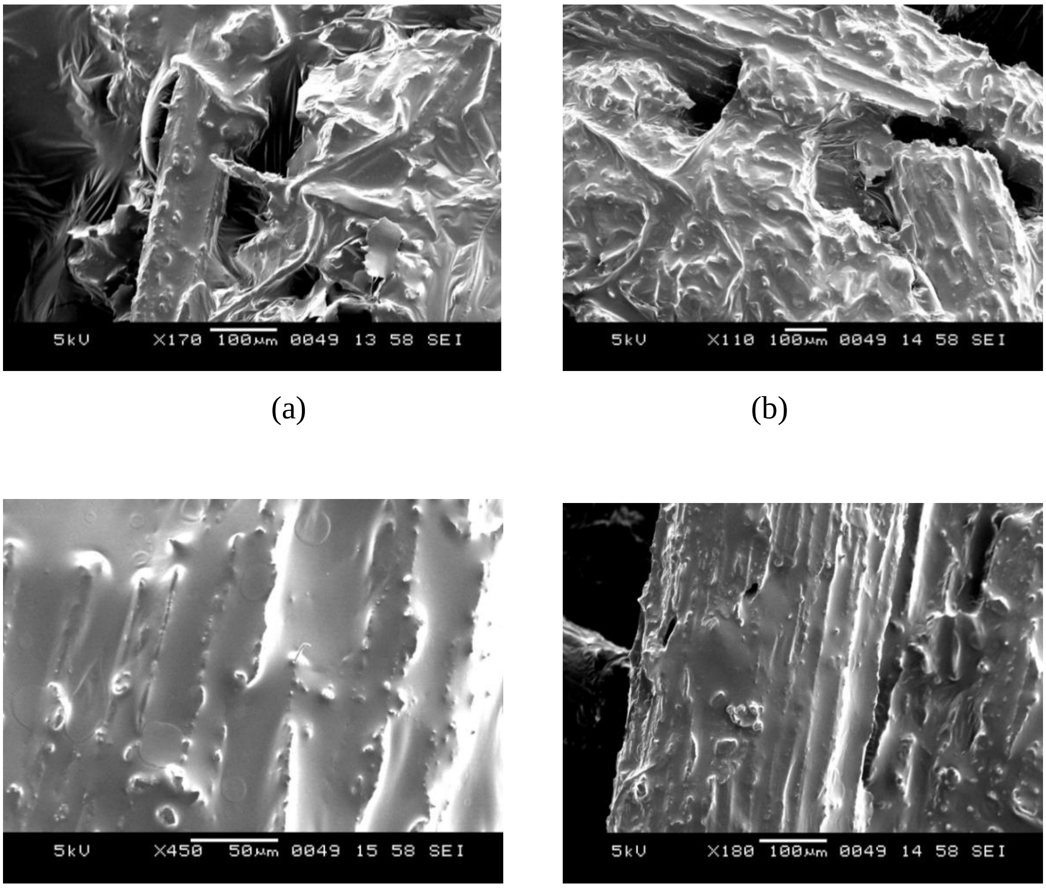
Figure 4.66: SEM Micrographs of Sandwiched Hybrid Composites

(a) Wetting phenomenon achieved in Composite, (b,c) Presence of fibres in the composite, (d) Fibrillation defect in composites, (e) Cross section View.

4.2.3.2.4 Bio-Composite

The Figure 4.67(a and b) shows the fibres had well interacted with bio polymeric resin and the fibres had a neat coating of resin layer. If the fibres are not modified with chemicals then it would have been a poor interaction attachment and thus leading more defects in the composite material. In processing, the fibres are broken out and it is clearly observed some entanglement between fibres and the matrix as shown in Figure 4.67(b) and it is also seen that the fibres are slightly dispersed in the matrix. Figure 4.67(c and d) shows the surface of maize fibres bio composite material and it can be observed some ridges forming on the surfaces and also showing the presence of globules (globules are the presence of hard sphere particles mixed with fibres in polymeric resin. When there is a drop in viscosity in biopolymers or dilute in polymeric solutions, poor globules can be seen) of bio polymeric resin.

Destruction of fibres and splitting of fibres during treatment will be responsible for failure in the composite. The interfacial gap with the fibres and matrix was found to be increased while increasing the fibres in the composite. If there are weak bond, extensive fibre pull out are expected and intial fracture occurs at that particular region and when the load is given the failure of the composite occurs.



(a) (b)

(c) (d)

Figure 4.67: SEM Micrographs Bio-Composites

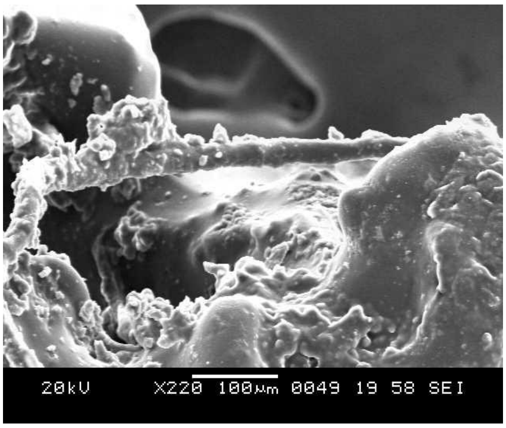
(a ,b) Fibres interacting with matrix, (c, d) surface image of composite

4.2.3.2.5 Fractography Study (Fractured Tensile Sample)

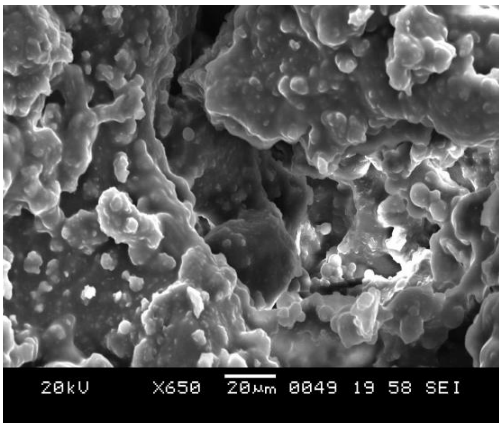
The SEM micrographs were taken for maize and hybrid composites after the fractures of specimen in tensile test. Many factors such as fibre failure, matrix failure, matrix crack and fibre crack were observed in these samples. Energy absorption mechanism such as fibre debonding and fibre pull out were also observed. In Figure 4.68 (a and b) shows the matrix agglomerate forming clusters in the fibre sample. Traces of resin bubbles are noticed at the fracture point and some voids are also seen in the micro graph showing the presence of improper local fibre interactions and the reasons for faster fracture crack. When the tensile load is dropped continually the formation of fibre failure and matrix cracking can be observed.

When the hybrid fibre composites (maize + jute) are subjected to tensile test the fibres break and kinked region and the crack propagating through the fibre matrix interface. The jute fibres are teared from the core part of the maize fibre and separation of fibres in to single fibrils occurs as shown in Figure 4.68 (c and d). The ridges, fibre pull out are also observed in the hybrid composite. Fibrillation is seen at the fracture point and this promotes easy path for the crack to propagate through the jute fibre. Hybrid composites had improved more effectiveness in interfacing and dissipating tensile damage to the composites (In hybrid composites, the effect of fibre matrix interface and the particulate fibre loading in tensile test had made less damage to the composite material. For a good bonded material the applied stress can be effectively transferred to the particles from the matrix. The strength depends on stress transfer between fibre and the matrix.

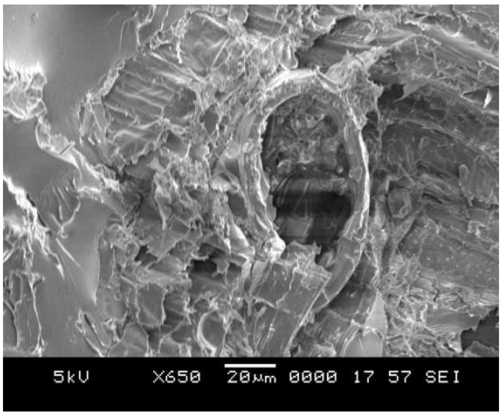
Generally fibres are strong and the matrix plays a minor role in tensile load carrying phenomenon and poor bonding results in easy failure. The maize fibre pull out was projected out as shown in Figure 4.68 (e and f). Fibres are pulled out from the fracture surface, because the presence of more stress concentration in the region (matrix fibre interface). The fibre pull out brings out the effective toughening of composite due to more introduction of fibres. During tensile testing, crack initiated at the weak bond and it propagated through the interface in the stress concentration region and again propagated through the matrix developing brittle fracture. Figure 4.68 (g, h) shows the specimen tested (fractured) for maize polymeric composite indicating maize fibres and epoxy polymers.


20kV X220 100µm 0049 19 58 SEI

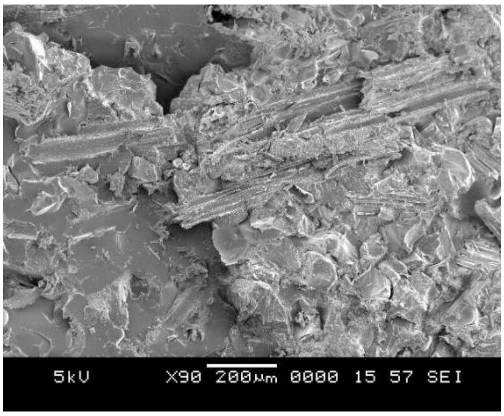
(a)


20kV X650 20µm 0049 19 58 SEI

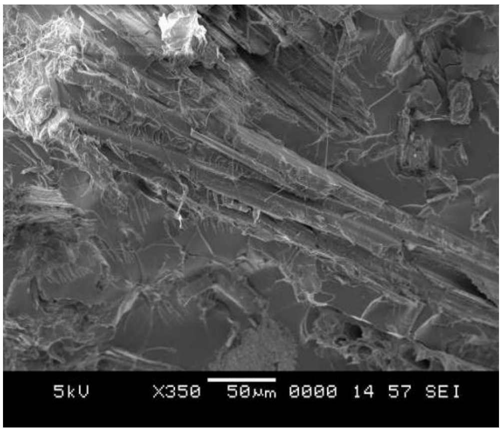
(b)


5kV X650 20µm 0000 17 57 SEI

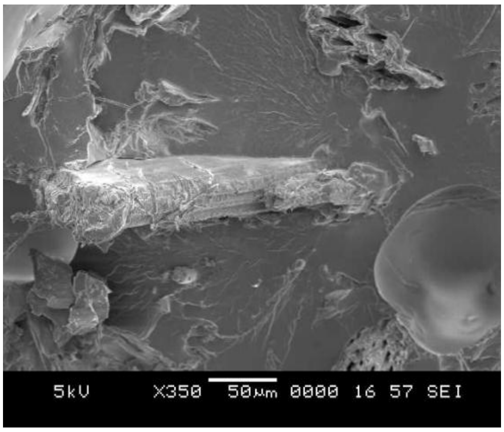
(c)


5kV X90 200µm 0000 15 57 SEI

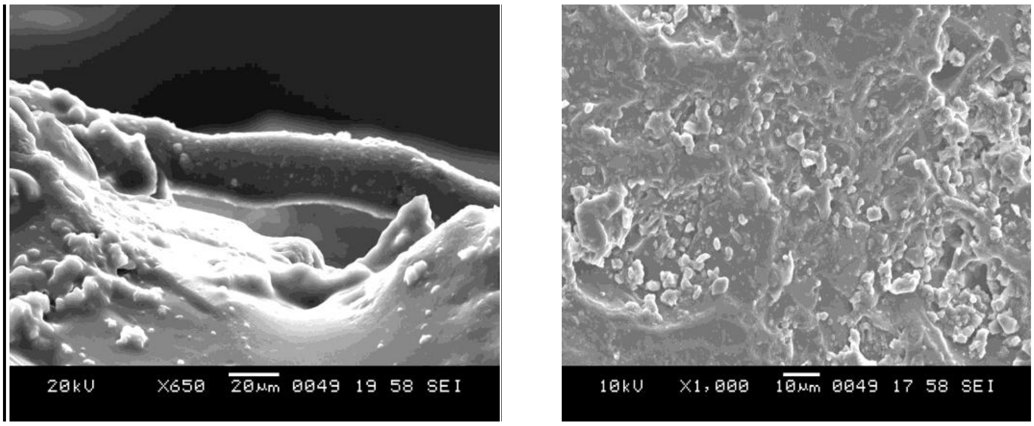
(d)


5kV X350 50µm 0000 14 57 SEI

(e)


5kV X350 50µm 0000 16 57 SEI

(f)



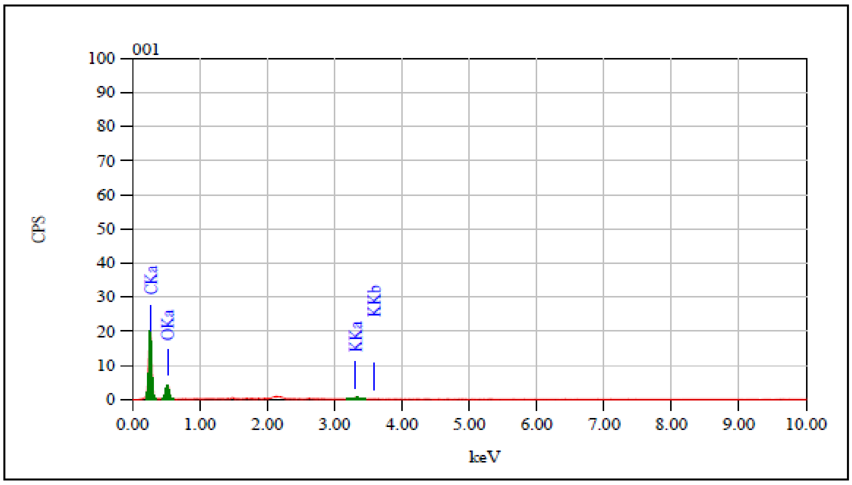
(g) (h)

Figure 4.68: SEM Micrographs for Fractured Maize Composites

(a, b) Maize fibre composites, (c, d) Hybrid composites (Maize + Jute), (e, f) Maize fibre pull out, (g, h) maize fibres with epoxy polymer

4.2.3.3 Energy Dispersive Spectrometry (EDAX) Analysis

The Energy Dispersive Spectrometry (EDAX/EDX) Analysis is carried out for maize hybrid composites and it showed some peaks containing Carbon (C), Oxygen (O), and Potassium (K), with K shell peak identification as shown in Figure 4.69. These are plant fibre cellular material consisting mainly of cellulose and the presence of Carbon, Oxygen and little traces of Potassium elements. K-α peak was observed which means X-rays emitted by an electron in the L-shell going down to the K-shell is identified as K-α (K shell X rays that correspond to atomic numbers 4 through 32). Similarly K-β peak (peak corresponding to X-rays emitted by M-shell electrons going to the K-shell) was observed.



Hybrid (Maize-jute) composite

Figure 4.69: EDAX Analysis for Maize and Hybrid Composites

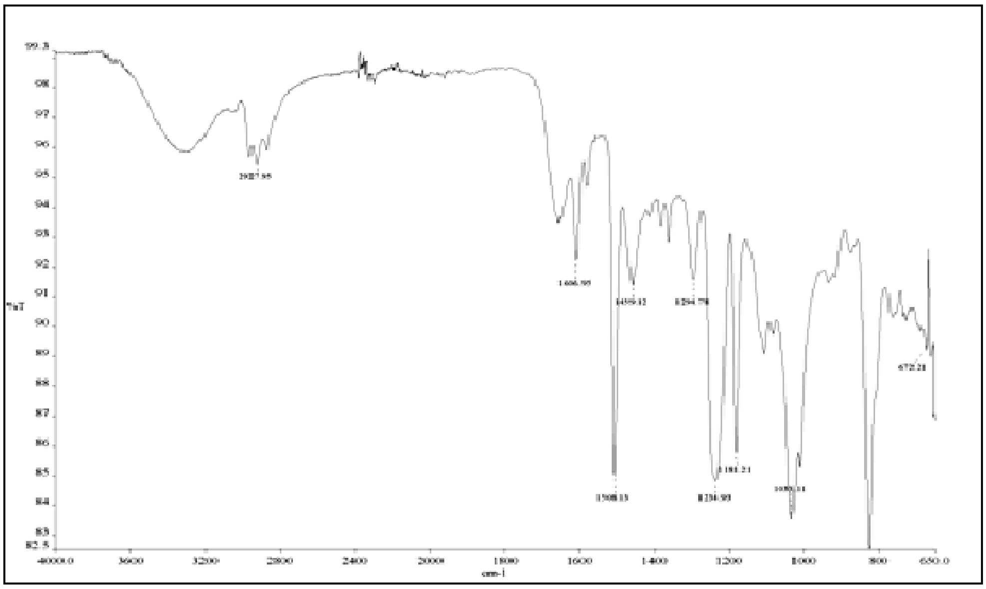
4.2.3.4 Fourier Transform Infrared Analysis

(i) Maize Composites

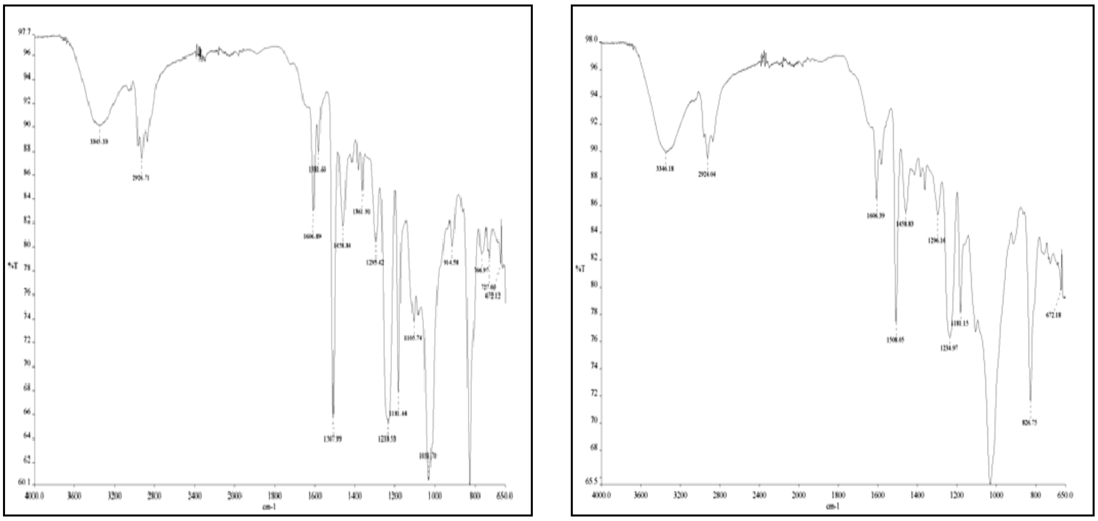
FTIR analysis for maize composites, hybrid composites and sandwiched hybrid composites were carried out for analysing the functional groups in the material. The sample test was conducted for 5%, 15% and 25% fibre content for maize fibre composites and 25 % fibre content for hybrid and sandwiched hybrid composites. Bio composite sample was also analysed for the concerned work and the results are discussed. Band width from 3600-2400 cm^{-1} was attributed to hydroxyl groups and the peak is shown at 2927 cm^{-1}. It also had appearance of C-O bond (crosslink between cellulose and lignin or with hemicelluloses). The peak at 1800- 1400 cm^{-1} were the characteristic of the carbonyl band (C=O) of the hemicelluloses in the maize fibre with a sharp peak at 1508 cm^{-1} as shown in Figure 4.70 (a).

The C=C stretching was observed in 1294, 1234, 1181, 1033 cm^{-1}. C-O-C bond was observed due to the stretching of lignin at 826 cm^{-1} and disappearance of C=O in the maize fibre (fibres treated with NaOH solution) and stretching of C=O in the composites and having a bond of C-H (due to the treatment with alkali of NaOH) in the composite was also observed. When the impurities are removed (such as wax, lignin and others acid) from the fibres C-C bond were observed in the analysis. Similar pattern of stretching of peaks of C=O and disappearance of C=O was observed for 15% Fibre content composite and 25% Fibre content composite as shown in Figure 4.70(b) and 4.70(c). The FTIR analysis for the maize fibre composites was also compared with the peak obtained from the standard epoxy resin as shown in Figure 4.70(d) and the structure resembled the same as previous showing the presence of epoxy polymer.

In hybrid composites, band width from 3600 - 2300 cm^{-1} was attributed to the hydroxyl group with strong peak at 3345 cm^{-1} and 2927 cm^{-1} and stretching of C=C bonds was observed in 1700 to 1200 cm^{-1} with peak occurring at 1606, 1459, 1361 and 1235 cm^{-1} as shown in Figure 4.71. The analysis also had a similar pattern of peaks for sandwiched hybrid composites as shown in Figure 4.72. Band width from 3600-2400 cm^{-1} was attributed to the hydroxyl group with strong peak at 3332 cm^{-1} and 2928 cm^{-1} and stretching of C=C was observed in 1800 to 800 cm^{-1} with peaks at 1508, 1296, 1181, 827 cm^{-1} .

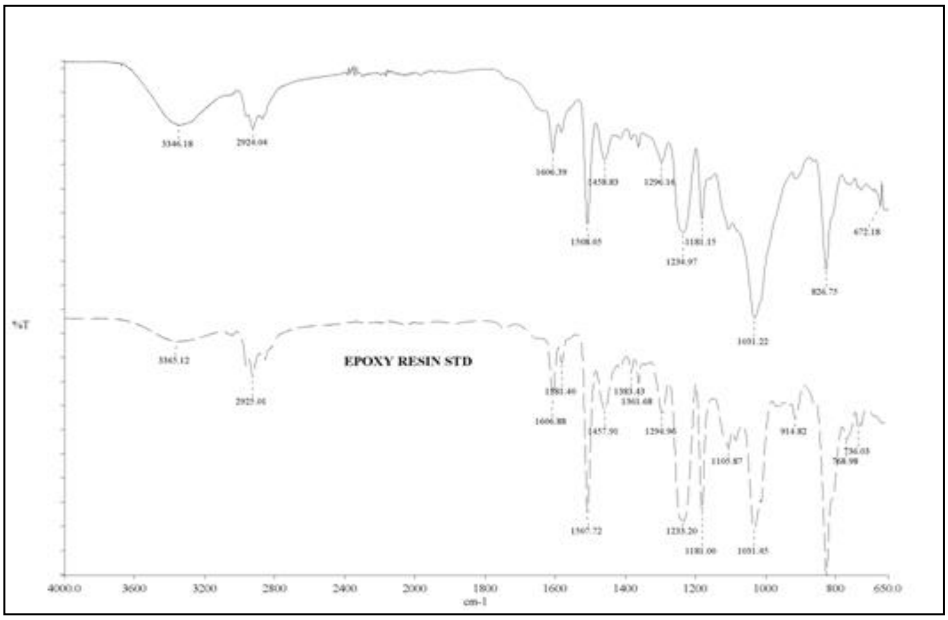


(a)



(b)

(c)



(d)

Figure 4.70: FTIR Analysis for Maize Composites

(a) 5% wt. fibre content composite, (b) 15% wt. fibre content composite, (c) 25% wt. fibre content composite, (d) Maize fibre composite compared with regular Epoxy resin

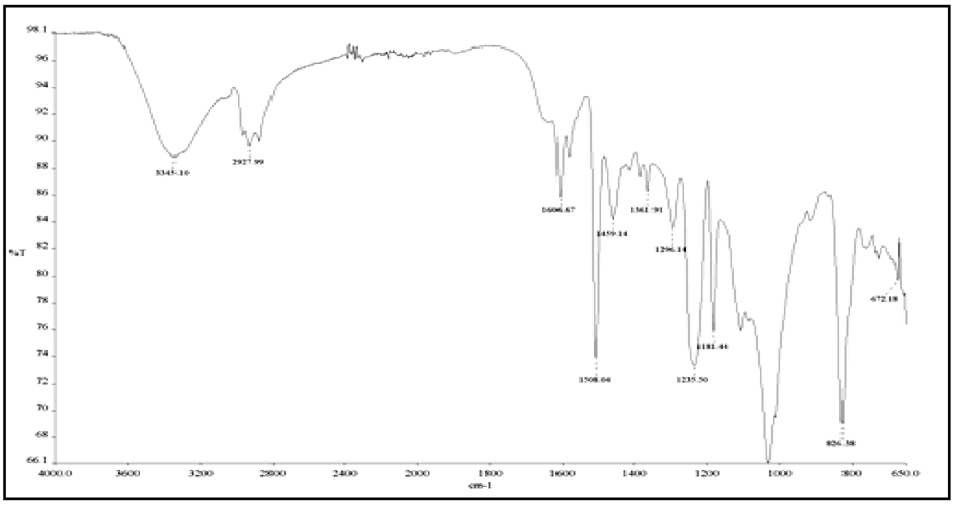


Figure 4.71: FTIR Analysis for Hybrid Maize Composites

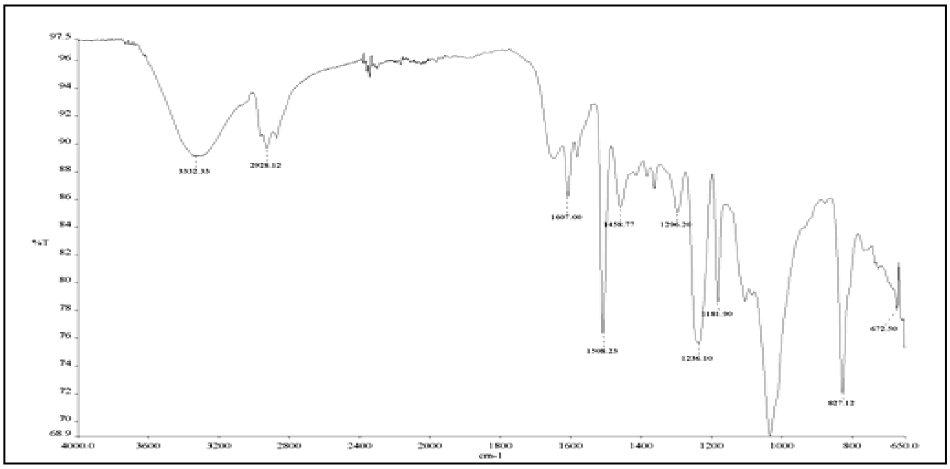
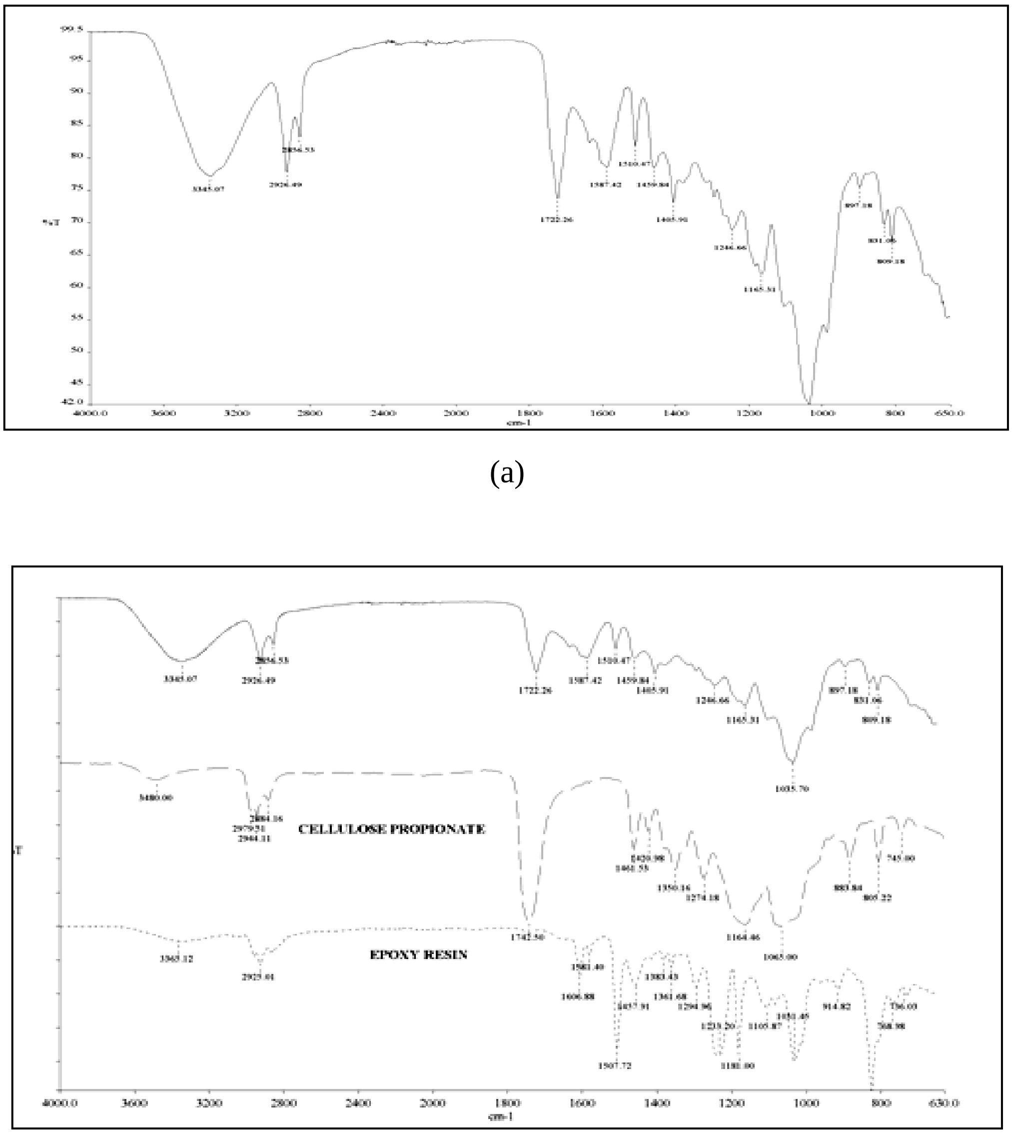


Figure 4.72: FTIR Analysis for Sandwiched Hybrid Maize Composites

(ii) Bio-Composite

Biopolymer composite consisted of epoxidized soy bean oil with maize fibres. Band width from 3400-2800 cm^{-1} was attributed to the hydroxyl group with strong peak at 3345 cm^{-1} and 2856 cm^{-1} and stretching of C=C bonds was observed in 1800 to 1000 cm^{-1} with peaks at 1722, 1405,1244, 1165 cm^{-1} as shown in Figure 4.73(a). The analysis was compared with regular cellulose and epoxy resin as shown in Figure 4.73(b) and the obtained FTIR analysis of bio composite had resembled with that of cellulose analysis conforming the presence of bio resin. Cellulose is the main constitute in natural fibres and it acts as the reinforcing material in the cell wall. Variation of hydrogen bond occurred and it was due to the main interaction with the nitrogen and oxygen group and this bond was due to crystal and amorphous domains. The decrease in wave number shows increase in length of covalent bonds.



(a)

(b)

Figure 4.73: FTIR Analysis for Bio-Composites

(a) Bio-Composite (Maize fibre + Biopolymer), (b) Bio-Composite were compared with Cellulose and Epoxy Polymers.

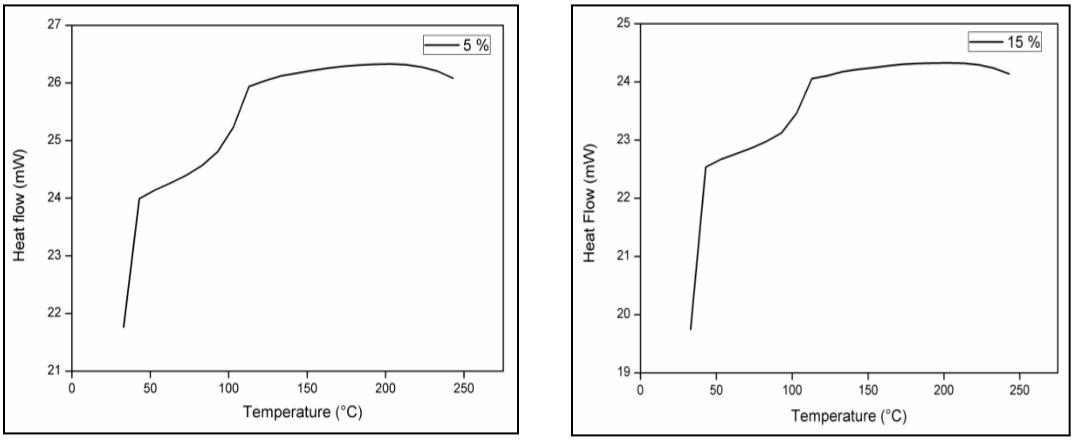
4.2.4 Thermal Analysis for Composites

The thermal stability of the composites was examined using thermal analysis. The thermal analysis comprises of different methods such as thermo gravimetric analysis (TGA) / differential thermal analysis (DTA), derivative thermo gravimetry (DTG) etc. Thermo gravimetric analysis (TGA) studies of samples were carried out in nitrogen atmosphere on a thermal analyser at a heating rate of 10°C/min.

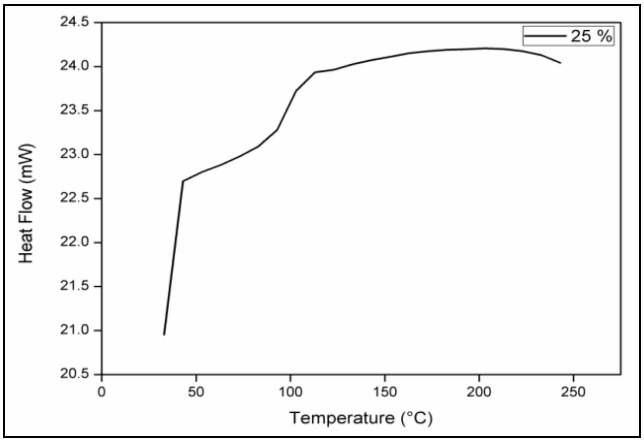
4.2.4.1 Differential Scanning Calorimeter Analysis

4.2.4.1.1 Maize Composite

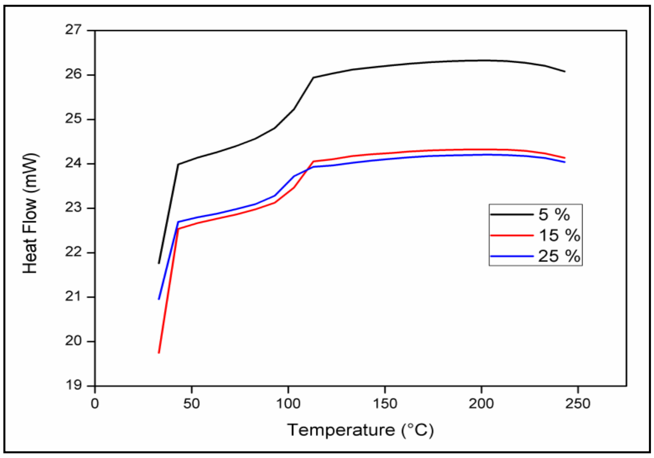
DSC analysis of maize fibre composites are shown in Figure 4.74(a to d). The method of conducting and preliminary work was explained in the previous section (maize fibres). The Samples having size of 6 ± 3 mg were heated at a constant rate of 10°C/min between temperature ranges of 20°C to 270°C and then cooled with nitrogen to 40^oC at a cooling rate of 10^o C/min with a flow rate of 30 ml/min. The trail work was employed for 5%, 15% and 25 % weight content of maize fibre composites.



(a) (b)



(c)



(d)

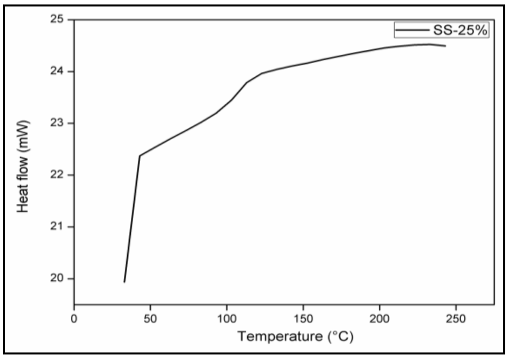
Figure 4.74: DSC Thermograph for Maize Fibre Composites

(a) 5% wt. fibre content composite, (b) 15% wt. fibre content composite, (c) 25% wt. fibre content composite, (d) Unified Maize fibre composite

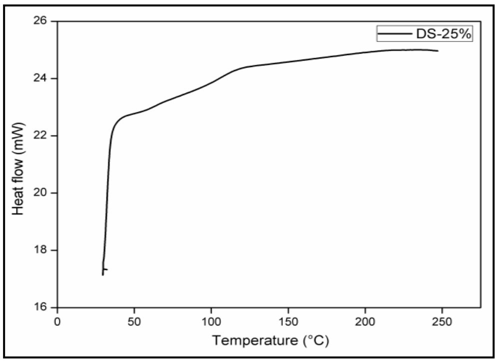
In 5% weight content maize fibre composites, they showed some endothermic peak (initially) with moisture absorption phenomenon later the DSC curve raised from temperature between 120^{o}C to 220^{o}C and then falling to 250^{o}C as shown in Figure 4.74(a). This was similarly observed in 15% and 25% weight content maize fibre composites as shown in Figure 4.74(b) and 4.74(c). The comparison of different content of maize fibre composites is shown in Figure 4.74(d). The exothermic peak observed will be due to the degradation of maize fibre in the composite material.

4.2.4.1.2 Hybrid and Sandwiched Hybrid fibre composites

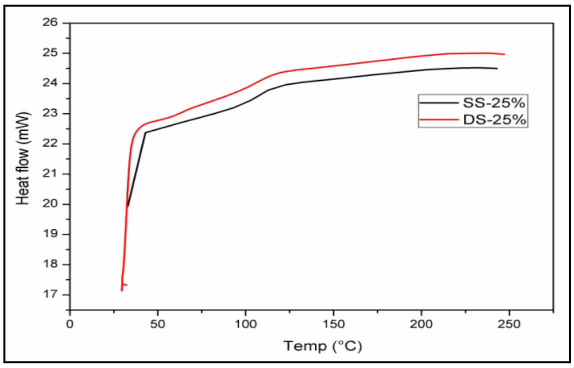
For hybrid and sandwiched hybrid composites, the DSC analysis is shown in Figure 4.75(a to d). 25% weight content maize fibre composites was taken for both hybrid and sandwiched hybrid composites and conducted for the analysis test, the reason was that among all test 25% fibre content gave better results than that of the other composites. These fibre composites, initially showed some endothermic peak (that was due to moisture absorption) and later some glass transition temperature occurred at 121^{o}C (for hybrid composite) and 125^{o}C (for sandwiched hybrid composites) and finally reaching exothermic peak as shown in Figure 4.75(a, b). The comparison of different content of maize fibre composites is shown in Figure 4.75 (d) and it showed sandwiched hybrid composite having better endothermic and exothermic peak. Maize composite with 5% also had the similar appearance having high endothermic and exothermic peak values.



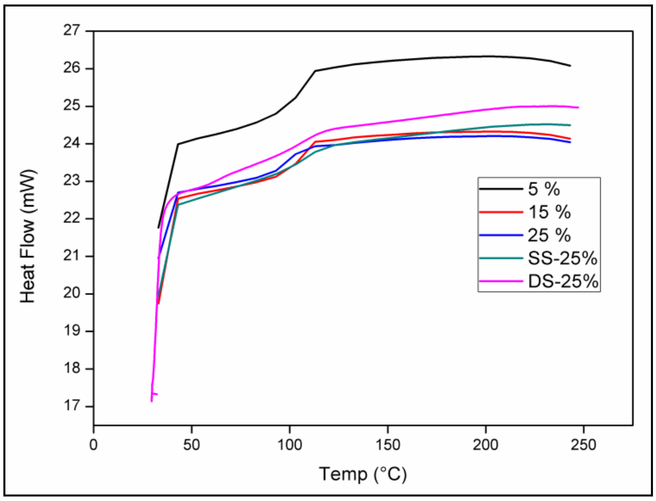
(a)



(b)



(c)



(d)

Figure 4.75: DSC Thermograph for Hybrid and Sandwiched Hybrid Fibre Composites

(a) 25% wt. fibre content hybrid composite (SS), (b) 25% wt. fibre content sandwiched hybrid composite (DS), (c) Unified hybrid fibre composite, (d) Unified Maize fibre composite

The DSC curves in above figures show some positive peaks as the heat flow to the sample was increased; hence the regions show exothermic reactions (release of heat from the sample). The endothermic peak is due to the volatile and water content in the sample. On further heating the sample more than 380°C, it may lead to char. In some cases such as in sandwiched composite a large endothermic was observed, this was due to the energy required to remove the volatile and water adsorbed by adsorbent [Zvinowanda et al. 2009]. Drying characteristics of corn stalk were studied through DSC by Chen et al. 2012. Heat requirements for the corn stalk were calculated and studied with thermal analyser. The hemicelluloses of maize stem mainly consist of xylose, arabinose, glucose and uronic acids. The extraction and characterization of these hemicelluloses from maize stem was studied by Sun et al. 2010. This work is further supported by DSC analysis indicating that because of high thermal stability, DSC curves of the hemicelluloses will very weak exothermic peak.

,

4.2.4.1.3 Bio-Polymer

Bio-composite material consisted of maize fibres and epoxidized soybean oil and the DSC analysis was conducted for 15% maize fibre content composite material. The method and procedure remained same as explained in above section. The bio-composite showed some glass transition at 28°C and followed with sharp endothermic peak temperature at 112°C (water absorption) and then with raise in temperature the peak raised to a temperature of 151°C and settling at exothermic peak (fibre degradation starts) as shown in Figure 4.76.

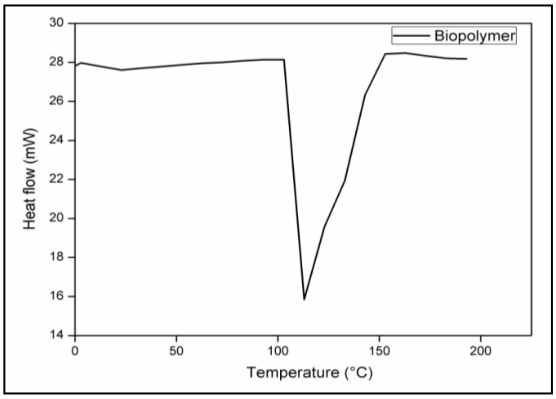
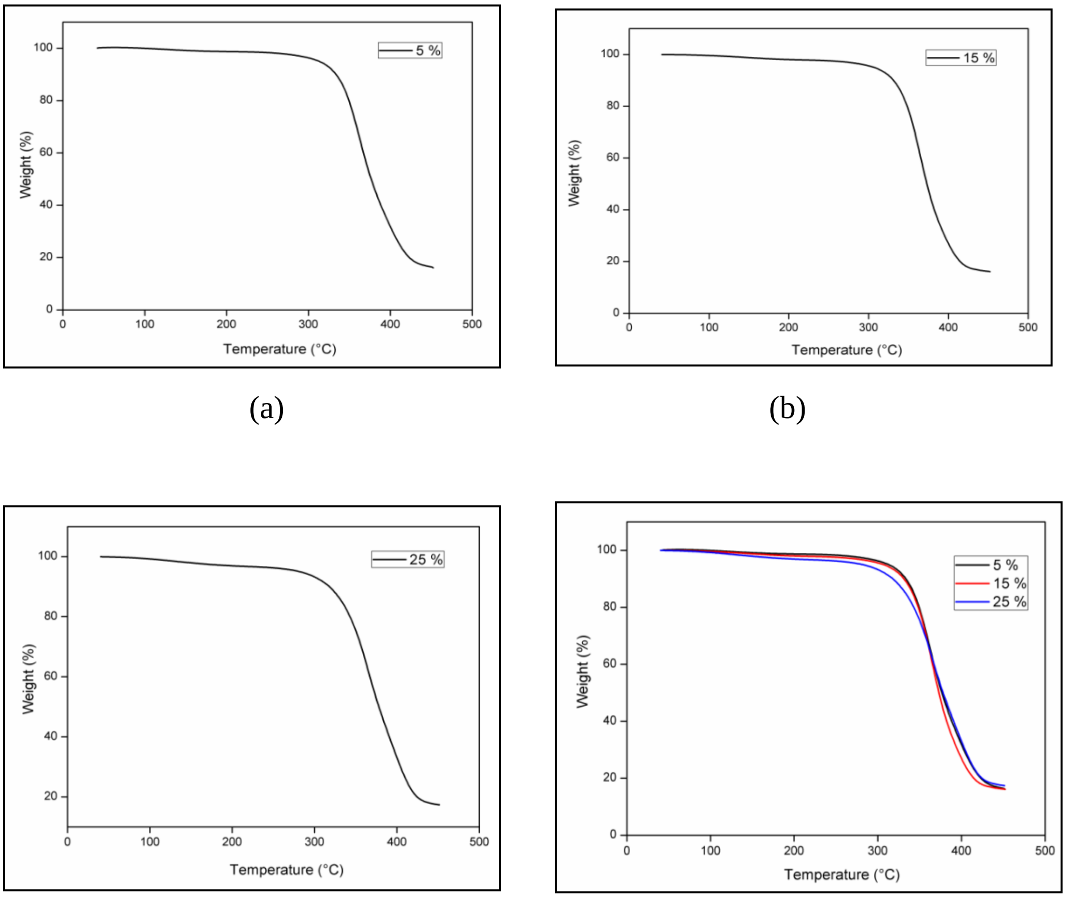


Figure 4.76: DSC Thermograph for Bio-Composites

4.2.4.2 Thermal Gravimetric Analysis

4.2.4.2.1 Maize Fibre Composite

The maize composite sample was heated from room temperature to 470ºC at a heating rate of 10ºC/min and a nitrogen gas flow rate of 50 mL/min. The TG curve for 5%, 15% and 25% maize composite is shown in Figure 4.77 (a, b, c). The TG curve shows two stages of decomposition, first stage can be due to the decomposition of cellulose and hemi cellulose segments and the later stage due to the degradation of lignin and other alkali segments on the fibre surface. For 5% maize fibre composite material, first degradation temperature was occurred nearly at 260°C and final degradation temperature at 381°C as shown in Figure 4.77(a). Similarly it happened for 15% wt fibre composite with first and final degradation temperature values occurring at 261°C and 384°C (as shown in Figure 4.77 (b)) and the resemblance had been the same for 25% wt fibre composite with degradation values of 264°C and 380°C. But for all the cases the maize fibre composite initial degradation temperature started to occur at 180°C. The final comparison of maize fibre composite is shown in Figure 4.77(d) and it can be observed the overlapping of curves in the fibre composite with 15% fibre composite material showing the better values. The DTGA curves are explained in next section.



(a) (b)

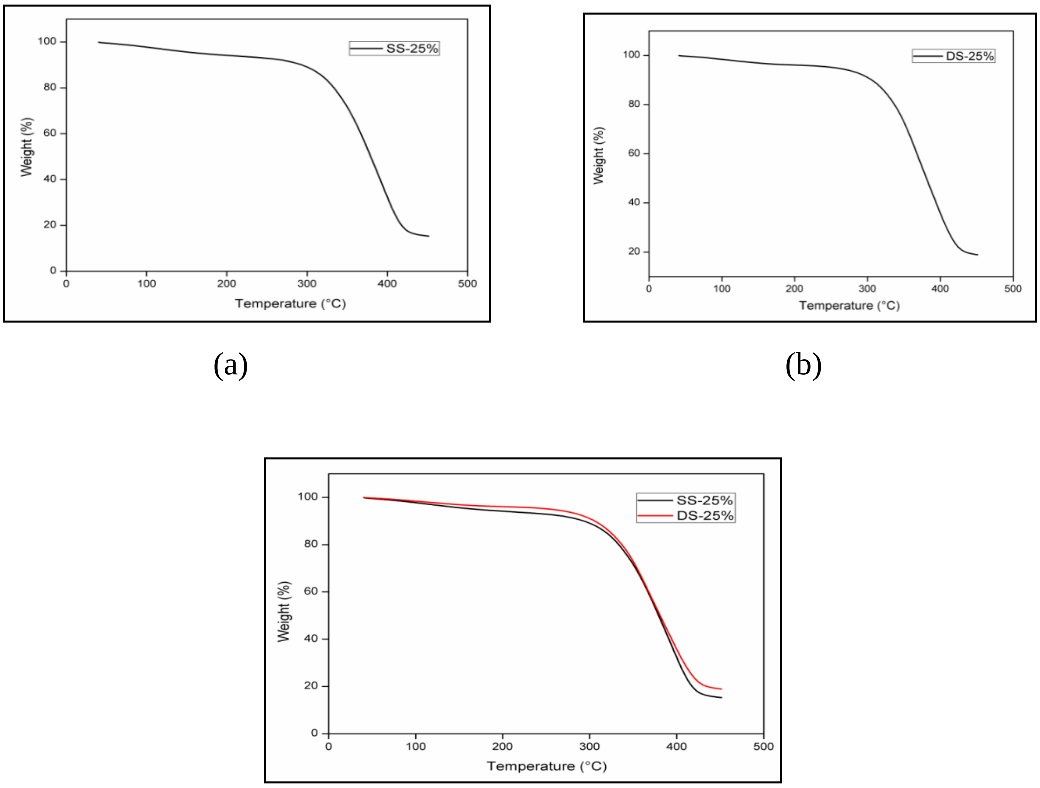
(c) (d)

Figure 4.77: TG Curve for Maize Fibre Composites

(a) 5% wt. fibre content composite, (b) 15% wt. fibre content composite, (c) 25% wt. fibre content composite, (d) Unified Maize fibre composite

4.2.4.2.2 Hybrid and Sandwiched Hybrid composites

The Thermo gravimetric (TG) curves for hybrid and sandwiched hybrid composites are shown in Figure 4.78(a, b, c). For hybrid and sandwiched hybrid composite (25% fibre content composite) the test was performed. In all these curves the moisture escapes on melting and then decomposition occurs with drying, desorption and then sublimation at last step. Initially mass loss will occur with degradation of various components of lignin and hemicelluloses. The initial degradation temperature for all the composites occurred nearly at 187°C with first stage degradation and second stage degradation. In Figure 4.78(c), it is been observed that sandwiched hybrid composite had better values when compared to hybrid composite.



(a) (b)

(c)

Figure 4.78: TG Curve for Hybrid and Sandwiched Fibre Composites

(a) 25% wt. fibre content hybrid composites (SS), (b) 25% wt. fibre content sandwiched hybrid composites (DS), (c) 25% wt. fibre content unified hybrid composites (SS & DS).

The first stage degradation temperature for hybrid composite was 208°C and final degradation temperature was 384°C and ended with 450°C. The first stage degradation for sandwiched hybrid composite was 210°C and final degradation temperature was 393°C as shown in Figure 4.78(a, b). In all these cases the initial degradation of composite started to occur approximately at 167°C (loss of hemicelluloses and lignin content). Primarily water content of the composite will be removed off then followed with removal of waxy substances then with hemicelluloses and lignin and finally with cellulose component.

The obtained results were compared to the previous work on fibre composites. Ioannidou et al. 2009 and Zabaniotou et al. 2008 studied TGA curves for corn residues and stalks. The TGA curve shows three steps of degradation namely first decomposition in the range of 40-110°C showing a moisture absorption, second decomposition (cellulose content) showing the corn stalk in the range of 195-355°C and the third decomposition in the range of 355-900°C and the sample had high amount of volatile upto 91.26 wt %. Region above 350°C shows the lignin decomposition.

The comparison was done for TG of maize composite with hybrid and sandwiched fibre composite. In the present study for all the composites it showed two stages of decomposition, first decomposition with elimination of absorbed water resulting with moisture content of the sample, removal of hemicelluloses segments and the second decomposition occurs at removal of cellulose and lignin constituents. For maize fibre composite initial degradation started to occur at 180°C. Whereas, for hybrid and sandwiched composite the intial degradation occurred at 187°C, this was because of the presence of additional fibre namely jute fibre. The comparison studies shows that sandwiched hybrid fibre composite had better values when compared to hybrid composite. The maize fibre composite had least degradation temperature when compared to the other remaining composite material. The removal of water and few cellulose and hemicelluloses composnents is by endothermic reaction (cause the formation of organic voltiles, whereas the removal of lignin was by exothermic reaction (devolatization, formation of char) [Uzun and Sarioglu 2009].

4.2.4.2.3 Bio-Polymer Composites

Bio polymeric composites did not with stand much temperature than that of the regular maize fibre composites. It showed two stages of degradation as already explained in previous section with first stage degradation temperature occurring at 200°C and final degradation temperature at 349°C. Removal of water content and other impurities occurred at initial stage and is shown in Figure 4.79. Initial degradation temperature of primary substances occurring at 148°C then followed with first stage and second stage of degradation.

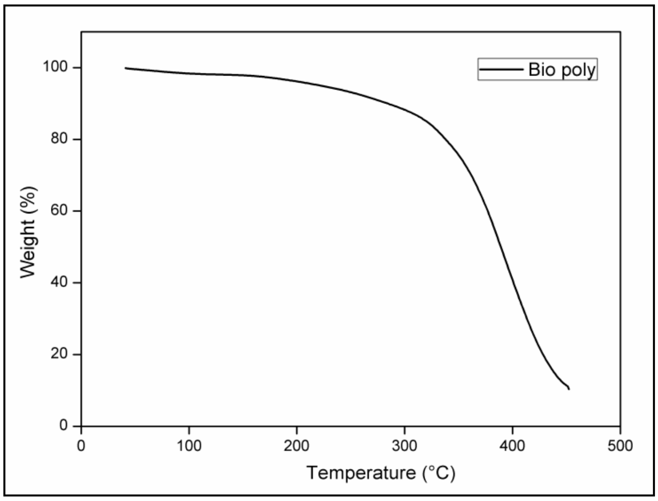
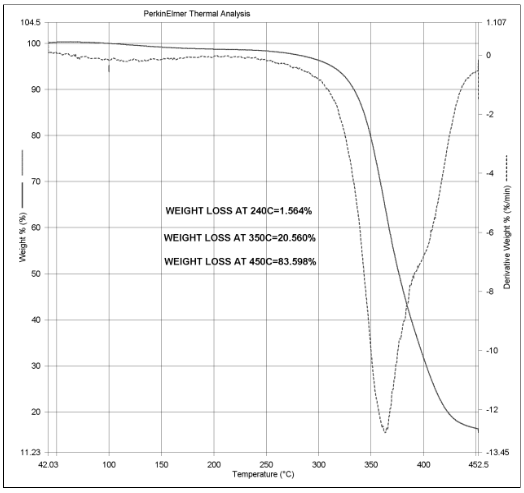


Figure 4.79: TG Graph for Bio-Composites

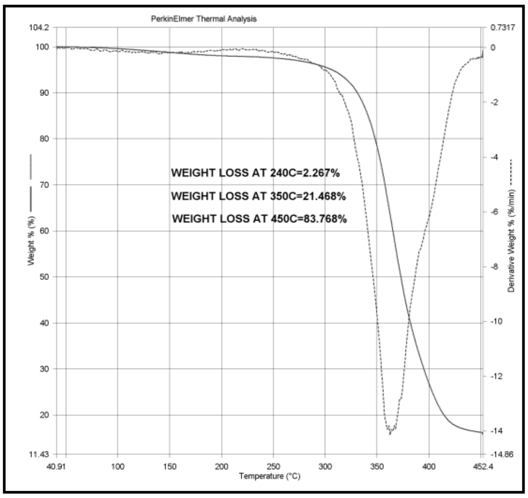
4.2.4.2.4 DTG Curve for Maize Fibre Composites

The weight loss values for maize composites, hybrid composites, sandwiched hybrid composites and biocomposites are shown in Table 4.26. The Derivative Thermo Gram (DTG) curves for the maize composites is shown in Figure 4.80(a,b,c) and the curve confirms the degradation process taking in two step fold and the highest DTG peak for 5% fibre content hybrid composite was at 364°C temperature and maximum weight loss of 83.59% occurred at 450°C. Similarly for 15% and 25% fibre content composite the maximum weight loss was 83.76% (at 450°C) and 82.53% (at 450°C). The weight loss for maize composites (15% wt. fibre content) for 240°C was 2.26%, for 350°C it was 21.46% as shown in Figure 4.80(c). The weight loss for maize composites (25% wt. fibre content) for 240°C was 3.54%, for 350°C it was 24.55%. This clearly shows that 25% fibre content was stable but 15% fibre content composite sample had better weight loss.

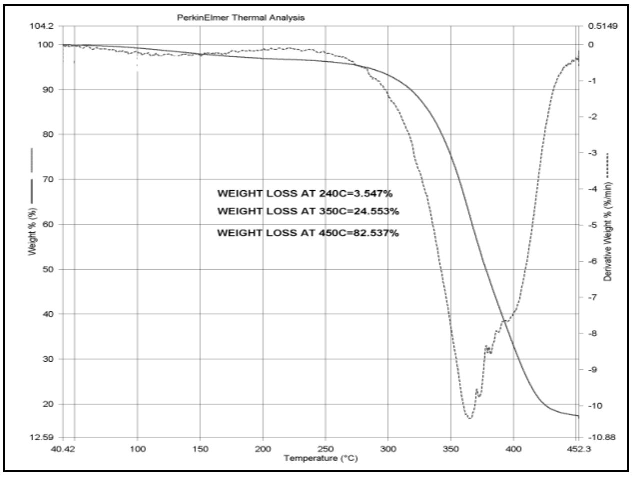
Similarly the weight loss for hybrid and sandwiched hybrid composites was observed. The prepared sample of 25% fibre content composite was tested and the weight loss was found 84.56% at 450°C for hybrid composites. Whereas for sandwiched hybrid composite the weight loss was 80.98% for the same 450°C as shown in Figure 4.81(a) and 4.81(b). Bio composite had the same effect as that of the maize composite material and the details are briefed out in Table 4.26 and shown in Figure 4.81(c). The bio-composite had 10% Fibre content and the weight loss at 450°C was 88.56% and this was the more weight loss when compared to other composite material.



(a)



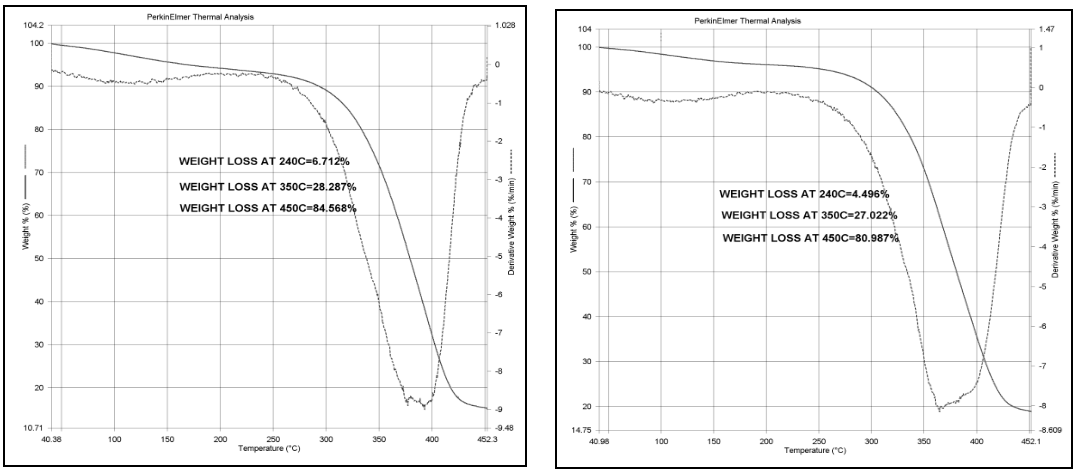
(b)



(c)

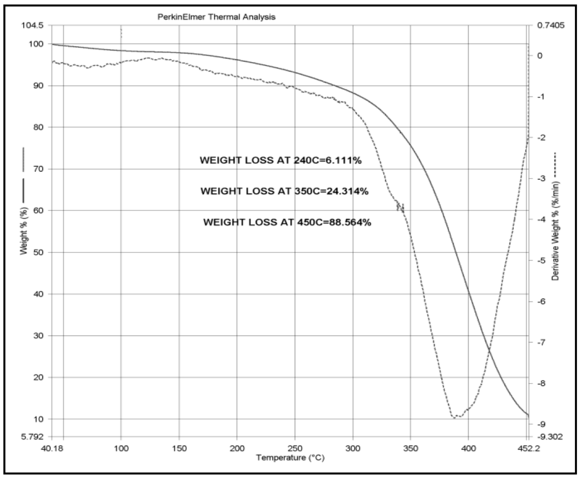
Figure 4.80: DTG Graph for Maize Fibre Composites

(a) 5% wt. fibre content composite, (b) 15% wt. fibre content composite, (c) 25% wt. fibre content composite.



(a)

(b)



(c)

Figure 4.81: DTG Graph for Hybrid and Sandwiched Hybrid Composites

(a) 25% wt. fibre content hybrid composite, (b) 25% wt. fibre content sandwiched hybrid composite, (c) Bio-composite.

Table 4.26: DTG Values for All Maize Composites

Sl. No.	Composites	Fibre Content wt. (%)	Weight loss (%)		
			240ºC	350ºC	450ºC
1	Maize Fibre Composites	5	1.56	20.56	83.59
2		15	2.26	21.46	83.76
3		25	3.54	24.55	82.53
4	Hybrid Fibre Composites (Maize + Jute)	25	6.71	28.28	84.56
5	Sandwiched Fibre Composites (Maize + Jute)		4.49	27.02	80.98
6	Bio-Composites	10	6.11	24.31	88.56

4.2.4.3 Thermal Conductivity for Maize Composites

Thermal conductivity is the property of the material which determines the heat flow in the given temperature field, through the measuring surface under the study resulting in the drop in the temperature in the direction normal to that surface. When a temperature gradient exists in a body, there is an energy transfer from higher temperature region to lower temperature region. The theory of thermal conductivity was proposed by Fourier in 1822. According to Fourier, the fundamental heat conduction equation can be stated as “For a homogeneous solid, the local heat flux is proportional to the negative local temperature gradient”. By definition thermal conductivity means “The material property that describes the rate at which heat flows with in a body for a given temperature change.” For one-dimensional heat conduction the formula can be given as

$$Q = kA\frac{dt}{dx} \tag{4.17}$$

Where,

k = Thermal conductivity of the material, W/m K
A = surface area of the specimen, m^2
dt = Temperature difference, K
dx = Distance between the two Thermocouple, m

The thermal conductivity of a composite material depends on the fibre, resin materials, fibre volume fraction, direction of heat flow and operating temperature.

4.2.4.3.1Materials and Methods

Thermocouple Junction Preparation

K-Type, thermocouple wires of 90 mm length were used in acquiring the surface temperatures, Junction preparation was done for each of the thermocouple wires, initially using nose-plainer the beads where made and later using joining process junction was made as shown in Figure 4.82. This was the common technique for junction preparation and soldering should be avoided for junction preparation as this would add additional metal at the junction. Each of the thermocouple wire junctions were later calibrated using National Instruments Lab View Thermal Module.

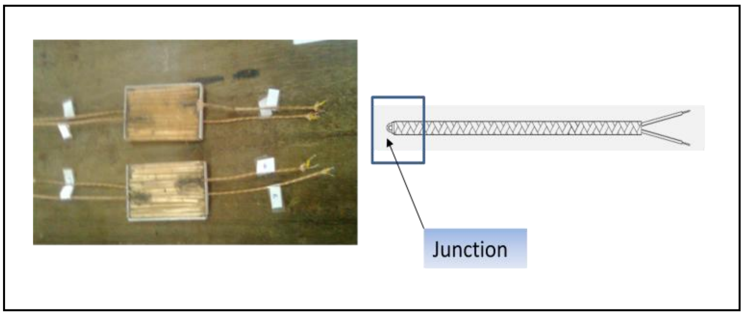


Figure 4.82: Thermocouple Mounting

LAB View - DAQ for Temperature Measurement

The NI USB-9213 is a high-density thermocouple measurement device designed for higher-channel count bus-powered systems for USB as shown in Figure 4.83. It can be easily connected to any desktop, laptop, or notebook and can measure up to 16 thermocouples with the included NI Lab-VIEW Signal Express LE software.

Figure 4.83: NI USB-9213 (National Instruments User Manual)

Initialize the data acquisition system using the DAQ assistant. Select the type of measurement; in this case the parameter to be measured is temperature using ‘K’ type thermocouple; go to acquire signal then analog input. In the analog input select the temperature as a measure and and then select the type of sensor being used. Connect the output from the DAQ asst to the indicators as shown in Figure 4.84.

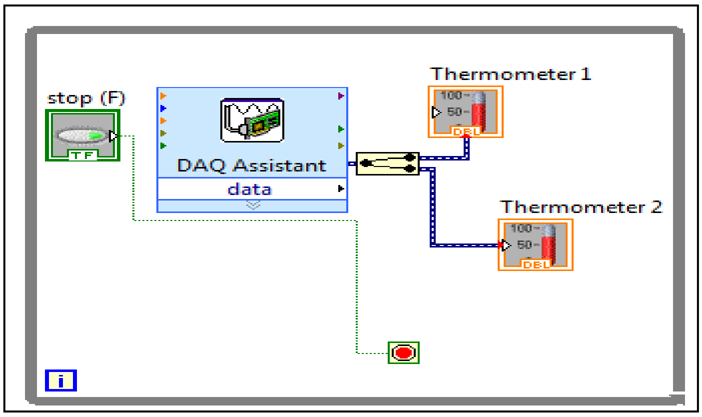


Figure 4.84: Data Acquisition System for Temperature Measurement using NI USB-9213

4.2.4.3.2 Work Set-up

Test was carried out at room temperature. Voltmeter, Ammeter and dimmerstat were used to regulate the heat flow through the heater in transverse direction. Considering the operation of the system, the sample to be tested was prepared as a flat slab and the plates are clamped in order to minimize the air gaps. Representation of experimental set-up is shown in Figure 4.85. It had the metal plate with heater in the middle of the plate and was insulated. The sample (maize composite) was placed in the plate and the temperature control is attached to it.

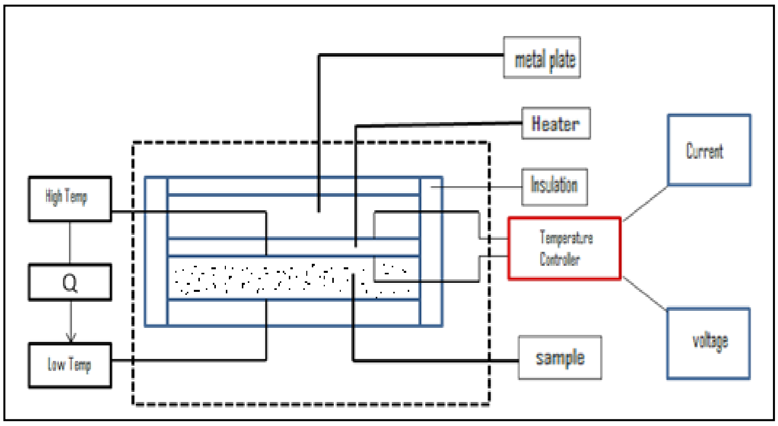


Figure 4.85: Schematic Representation of Experimental Setup

4.2.4.3.3 Results and Discussions

In order to measure thermal conductivity of the composite specimen, percentage of heat from the heat source was estimated by calibrating the setup with specimen of known thermal conductivity, then the composite specimen whose transverse thermal conductivity to be determined was placed. Care was taken in placing the thermo couples, surface temperature was averaged on the top and bottom surface of specimen. The setup designed based on one dimensional heat flow principle, so that thermal conductivity could be easily determined using Fourier’s Law of Conduction.

Basic heat transfer mechanism adopted in composites was followed. It describes the thermal resistances in fibre reinforced composites based on concept of electrical analogy and how a complex problem is represented for analysis. Thermal conductivity reading for the maize fibre composites are tabulated in Table 4.27 and the values are given input for finite element method which was performed by finite element analysis software as explained in next chapter. It can be seen that effective thermal conductivity is mainly dependent on the volume fraction of fibres, and thermal conductivities of fibre and matrix.

Thermal conductivity for Unidirectional composite and derivations for obtaining k_{eff} $_{and}$ k^{+} were included in the Apendix-1.

Table 4.27: Thermal Conductivity of Maize Fibre Reinforced Polymer Composite with Different Volume Fractions

Sl. No.	V_f	k_f / k_m	k_{eff} (Expt)	k^{+} (Expt)
1	0	0.494	0.205	1
2	0.13	0.494	0.192	0.936
3	0.18	0.494	0.189	0.921
4	0.24	0.494	0.169	0.824

From the table 4.27, it can be seen that effective thermal conductivity found to be decrease with the increase in volume fraction. This was because thermal conductivity of the composite depends on fibre, matrix content and value of volume fraction. This behaviour is due to the low thermal conductivity of fibres in the composite. The contributing mechanism would be of influence of particle size, content and increase in volume of natural fibre in the composite. When the specific surface area increases beyond certain volume fraction of fibres, thermal conductivity decreases. The other reasons can be agglomeration of particles at high volume fraction fibres.

4.3 Summary

The findings in this chapter are discussed below with properties of maize fibre and their composites which includes hybrid and sandwiched composites. Mechanical properties such as tensile and flexural test, morphological studies and thermal analysis of the composites are briefed out with outcome results. Characterization techniques results, hardness values and water absorption results show some acceptable values.

The Mechanical and flexural properties of the maize composites depend largely on fibre matrix interaction and the fibre content. In all composites under study; maize composite, hybrid composite and sandwiched hybrid composite with 25% fibre content show better strength and modulus values. Maxmium tensile stress of 19.29 MPa and Youngs Modulus of 1.12 GPa for maize fibre composites, 17.56 MPa and 1.33 GPa for hybrid composites and 27.89 MPa and 1.65 GPa for sandwiched hybrid composites. Maxmium tensile stress of 10.11 MPa and Youngs Modulus of 1.2 GPa was observed for bio-composites. For a given thickness of maize composites, strength remains same for increase in fibre content after 10%. Whereas in other, composites strength was enhanced with increase in fibre content. The flexural properties of the composites depend largely on fibre matrix interaction and the interface quality. Maxmium flexural stress of 46.24 MPa and Flexural Modulus of 1.97 GPa for maize fibre composites, 72.32 MPa and 4.02 GPa for hybrid composites and 57.33 MPa and 4.28 GPa for sandwiched hybrid composites. The flexural stress for biocomposites was 26.96 MPa and Flexural Modulus of 2.6 GPa was observed. Sandwiched hybrid composites had better strength and values compared to the hybrid composites. Tensile strength of sandwiched hybrid composites performed well when compared to hybrid composites as seen from the results.

The SEM micrographs of the sample provided the information about the morphology of the maize fibres and their composites. Fibre debonding, matrix cracking and fibre pull out were observed. In the treated maize composites it showed better bonding with the fibres and the matrix because of good interfaces in the composites. In the bio-composites, it was observed that infusion of resin with the fibre was good and it was maximum for 15% weight fraction of fibres in the composite. Too much resin infusion in the fibre was not favourable for processing of composites through VARTM because of high viscous resin material. Whereas in maize composites, higher weight fraction of fibres was possible and good interfacial bond provided the composite to yield a good result.

The TGA gave the information of maximum degradation temperature that can withstand for maize fibres and composites. Among them hybrid and sandwiched hybrid composite show better favourable values when compared to maize composites and with its respective bio composite material. Results indicated that the presence of cellulose fibres affects the degradation process of the composites. 25% fibre content composite was stable in thermal degradation but 15% fibre content composite material showed better results in TG and DTG curves and they had considerable weight loss. DSC analysis showed the all the composite materials had endothermic region with initial indication of moisture absorption and then entering to a region of exothermic and finally with fibre degradation. 25% weight content maize fibre composites had shown better values than that of the other composite materials. Better thermal results can be obtained if the fibres were subjected to more chemical treatment.

In all XRD and FTIR analysis showed that tthe major changes that occurred for untreated and treated maize fibre composite samples. Crystalline and amorphous content for fibres were revealed and fibres when treated with chemicals showed more crystallinity. More hardness of composites vary with fibre content and increased from

13 to 21 Hv for 5% and 25% fibre content and similarly D shore hardness number was observed from 67 to 82 in maize fibre and sandwiched hybrid composite. Water absorption experimental values shows that the maize composite absorbed 4.11% of moisture, and 7.11% for hybrid composites and for sandwiched hybrid composites with jute fibre increases up to 15.55%

The experimental results of thermal conductivity of maize fibre composite are compared with the analytical method and the analysis was peformed by using finite element method using commercial available software ANSYS which are discussed in chapter 5.

CHAPTER 5

ANALYSIS OF MAIZE LONG FIBRE COMPOSITE

This chapter aims on finite element analysis of maize long fibre composites, carried out by using finite element method with the aid of ANSYS, commercial analysis software. Structural and thermal analysis for the maize fibre composites was analysed using the values of obtained experientally as discussed in the previous chapter.

5.1 Experimental Work for Long Fibre Composite

Maize long fibre polymer composites were prepared using natural maize long fibre runs through out the length of composites. These composites are proposed to avoid pulverizing the maize stalk for obtained fine fibres, where this process is well suit for extruding in the development of composite components. Long fibres are very useful in the preparation of composites plates of larger sizes.

Seasoned and sun dried maize stalks were collected from the field, from this fibres were first extracted and cut into uniform length after removing the soft pith core. Mould release agent is applied all over the mould surface and a brush or roller is used to wrap layering process of the fibres. The stalk long fibres were placed in the mould evenly in a unidirectional manner and thermosetting resin is mixed with promoter and catalyst. Layers of the long maize fibres impregnated with the resin are used to build up the require thickness.

Hand Layup Technique was employed for the preparation of the maize long fibre reinforced composite. A mould of mild steel was used with dimension of 40 x 80 x 10 mm as shown in Figure 5.1 and a base plate with thickness of 4 mm was used. A small beam mould is prepared and made to consist of maize stalks long fibres of approximate length of 80 mm and placed in a unidirectional manner as shown in Figure 5.2.

Unsaturated polyester resin is mixed with Methyl Ethyl Ketone Peroxide (MEKP catalyst) and Cobalt Napthenate (accelerator). The catalyst was mixed in the resin thoroughly before adding the accelerator. The reaction between initiator and accelerator caused the resin to become gel, extra care has taken in the preparation of the resin so that no air bubbles are formed. After curing, the beam specimen was released from the mould to obtain a composite in 20% fibre content and is shown in Figure 5.3.

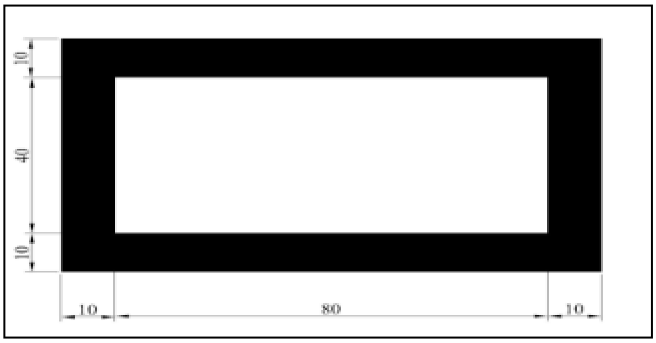


Figure 5.1: Schematic of Hand Lay-Up Mould

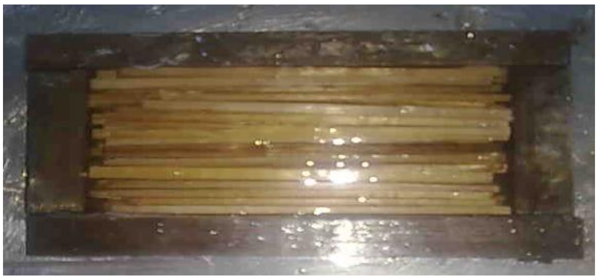


Figure 5.2: Maize Long Fibres with Polymer Matrix in the Mould

Figure 5.3: Specimen of Maize Long Fibre Composite Beam

5.2 ANSYS –Finite Element Analysis Software

ANSYS is a finite element analysis software package which enables an engineer to perform the following the tasks given below.

- To build computer models or transfer CAD models of structures, product, components or systems.
- Applying operating loads or other design performance conditions.
- For studying physical responses such as stress levels, temperature distribution or electromagnetic fields.
- To optimize the design early in the development process and to reduce the production costs.

The ANSYS program has comprehensive graphical user interface that gives users easy, interactive access to program functions, commands, documentation and reference materials. An intuitive menu system helps the user to navigate through the ANSYS program.

ANSYS is a Finite Element Method (FEM) software package that includes pre-processing, solver and post-processing modules. This is very user friendly and can be used to solve many complex geometry problems involving composite materials. Some of the basic steps one has to follow to execute a static analysis are.

- Selection of the appropriate element to discretize the component.
- Supply the required real constants for the geometry, etc.
- Key in the material properties as given by the manufacturer.
- Model the geometry and meshing it.
- Apply the boundary conditions and loads.
- Solve to get the solution.
- Interpret the results.

5.2.1 SOLID 70 Element

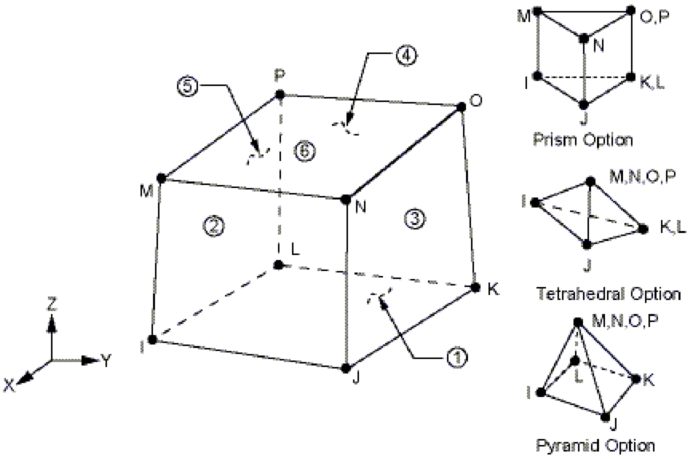


Figure 5.4: SOLID 70 Geometry (ANSYS user guide, Ver.11)

SOLID 70 has 3-D thermal conduction capability and the element has eight nodes with a single degree of freedom, temperature, at each node as shown in Figure 5.4. The element is applicable to a 3-D, steady-state or transient thermal analysis. The element also can compensate for mass transport heat flow from a constant velocity field. If the model containing the conducting solid element is also to be analyzed structurally, the element should be replaced by an equivalent structural element (SOLID 45).

5.2.2 SOLID 70 Assumptions and Restrictions

- The element must not have a zero volume. This occurs most frequently when the element is not numbered properly.
- Elements may be numbered either as shown in Figure 5.2 SOLID70 Geometry or may have the planes IJKL and MNOP interchanged.
- A prism or tetrahedron shaped element may be formed by defining duplicate node numbers as described in Triangle, Prism, and Tetrahedral Elements.
- The specific heat and enthalpy are evaluated at each integration point to allow for abrupt changes (such as for melting) within a coarse grid.

5.3 Finite Element Analysis (FEA) of Maize Long Fibre Composite Material

Fibre composites consist of long fibre and matrix phases and the mechanical behaviour of the composites are much determined by the fibre and matrix properties. Microstructures such as fibre shape, fibre array, and volume fraction of fibres are also more important in determining the mechanical properties [Tsai and Chi 2008]. Micromechanical models are been used to predict the properties starting from the intrinsic properties and their constituents [Rao et al. 2012] and these models show that the fibre strength is not being completely employed as a result of poor fibre matrix interfacial adhesion and fibre length.

5.3.1 Structural Analysis

The Finite Element Method (FEM) was used to model the material behaviour on the basis of micro-mechanical level. The model was assumed to be an isotropic fibre and matrix material with a rectangular section of a beam. This section is then modelled in detailed using volume elements to represent the composite. Each element will have an isotropic property and be positioned corresponding to the fibres and the mesh regions are coarsely meshed (converged solution). The material is modelled using certain assumptions as given below and analyzed for mechanical properties with finite element method software (ANSYS). The composite material is assigned as unidirectional composite by assuming some properties that are given below.

Some of the assumptions used for the analysis work are been gathered by literature [Behzad and Sain 2007, Duigou et al. 2010, Bayat and Aghdam 2012, Balac et al. 2012, Silva et al. 2012, Elbadry et al. 2012]

- Fibres are not porous
- The material property for all the constituents are attributed as isotropic material for both the volumes
- Fibres are uniform in properties with diameter
- Interphase bonding is maintained between fibres and matrix

- Perfect bond between fibre and matrix and no slippage
- Fibres are arranged in unidirectional manner and perfectly aligned
- Composite material are free of voids

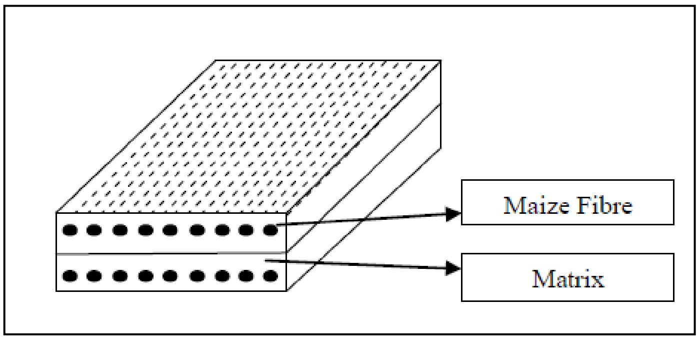


Figure 5.5: Composite Material with Fibre and Matrix

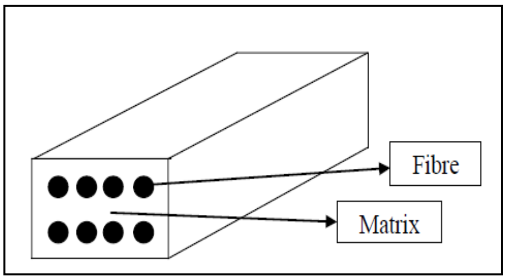


Figure 5.6: Fibre and Matrix Region

The composite material consists of long fibres aligned in unidirectional manner and modelled as a regular uniform arrangement, as shown in Figure 5.5 and 5.6. This model assumed that the fibre is a perfect cylinder of length *l* (80 mm), and diameter, d (1mm) in a matrix.

The model is treated as a linear isotropic problem. The finite element analysis model was constituted of Solid 70 elements, used for fibre-matrix structure as shown in Figure 5.7. The model included the fibre, matrix and fibre-matrix interface. Nine fibres were modelled to the surrounding matrix. The long fibres, with surrounding matrix were selected for stress analysis in this model. These regions were modelled using the coarse mesh (converged solution) as shown in Figure 5.8.

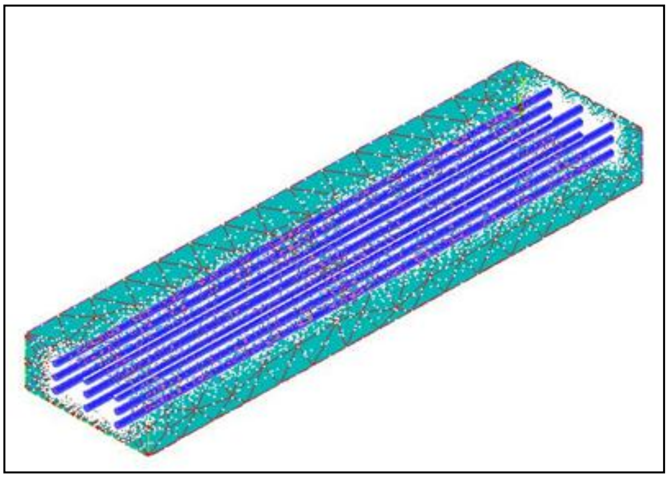


Figure 5.7: 3-D Model of the Composite Material

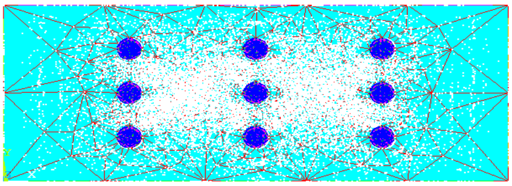


Figure 5.8: Meshing of Fibres and Matrix

Uniform mesh was arrived between fibre and matrix, for the analysis SOLID-70 element was preferred with 8-node brick type. This meshing was sufficient, it is more refined or with excessive number of elements then it may lead to increase in round off error. Hence adequate region of mesh is fine in that suitable region. The geometry model was meshed with SOLID 70 elements with calculated nodal displacement fields in X and Y directions. Boundary conditions were enforced along the side surfaces, and the nodal displacements along the top face with normal directions. The fibre taken as an isotropic material with Young's modulus, E_f = 8.58 GPa for Maize fibre (From experimental work), and E_m= 1.3 GPa for the polymeric resin and Poisson ratio of υ_m = 0.3. Reinforcement content affects the level of stress and stress distribution in fibre, matrix and fibre-matrix interphase. In this model, the volume fibre content was 20 %.

Fibre matrix distribution for the fixed volume of volume fraction was done by rule of mixtures. Rule of Mixtures are mathematical expressions which gives property of composites, and arrangement of its constituents.

5.3.2 Thermal Analysis

Thermal conductivity of a composite depends upon the thermal conductive nature of the fibre and matrix. The thermal conductivity of a polymer composite is based upon the conductivity of fibre and resin, resins are usually insulator and the conductivity is dominated by fibre material. The compactness of fibres per unit area influences the conductivity of the composite. Thermal conductivity of composites is anisotropic in nature and the knowledge of thermal conductivity of composites is required for design. Data about thermal conductivity of resin facilitates to reduce stresses related to shrinkage of composites during cure and mismatch in thermal expansion coefficients. Figure 5.9 shows the flow chart illustrating the finite element method to determine the thermal conductivity of the composite. (Convergence using P-method was obtained. It evaluates the polynomial level of the finite element shape functions. However the best variable to use is strain energy convergence (strain energy to loop pass). The range of study and the optimal value are not counted in this study).

Some of the assumptions are been carried out in achieving the result and they are as follows.

- Locally both matrix and fibre are homogenous and isotropic.
- Thermal contact resistances between fibre and matrix interfaces are negligible.
- Composite laminate are free of voids
- Heat loss due to radiation and convection are neglected.
- Filaments are equal and uniform in shape, size and are symmetrical about x and y axis.

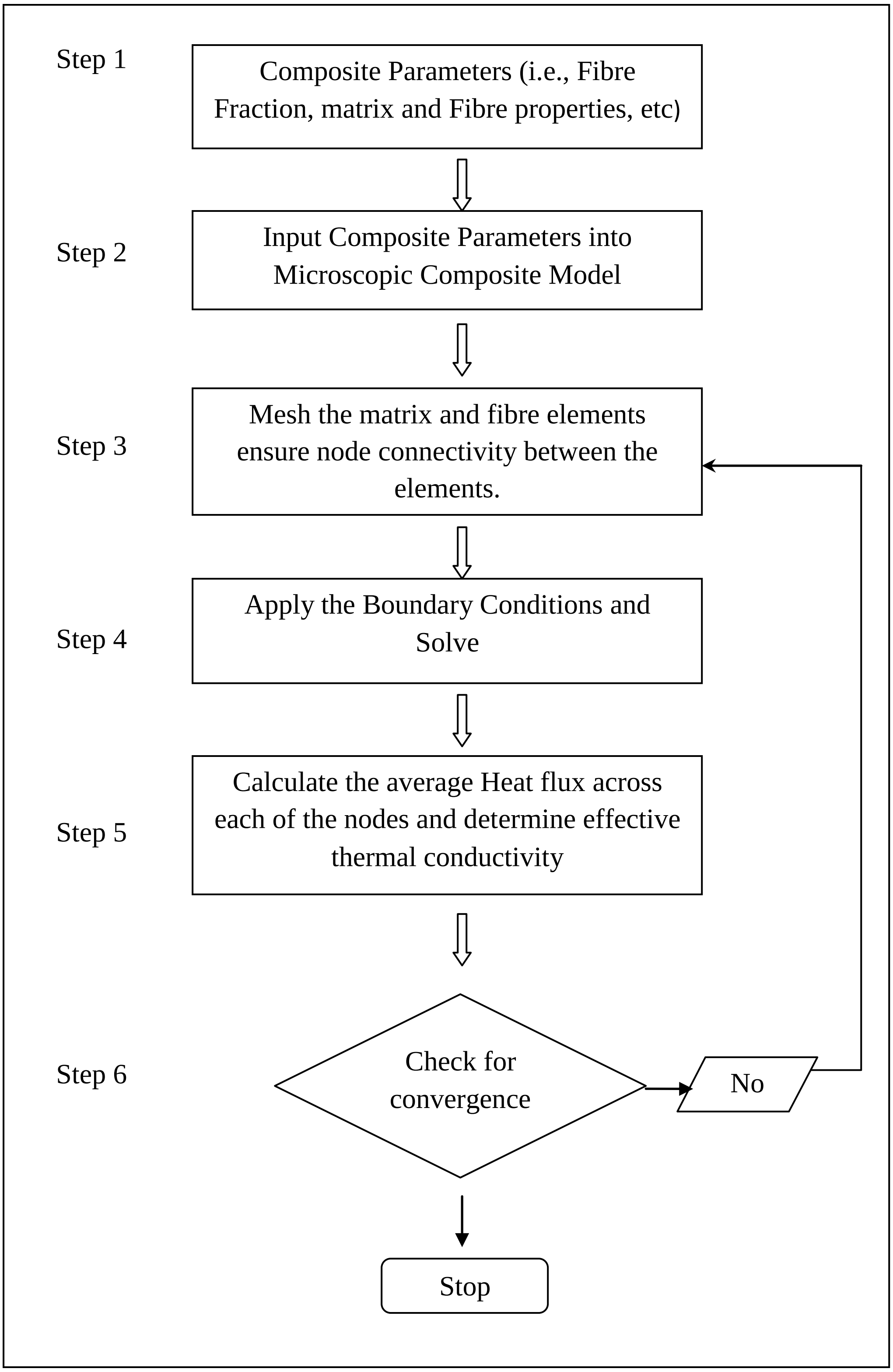


Figure 5.9: Flow Chart illustrating the FEM in Thermal Analysis of a Composite

5.4 Results and Discussions

Finite element analysis for the model developed has been used to analyze structural as well as thermal behavior of maize fibre composite. The following sections explain the comparison of results obtained from FEM and experimental values.

5.4.1 Structural Analysis

The fibre content of the composite was an important factor controlling the mechanical properties of the composites. The level of stresses in fibres was lower than the matrix in the composite with low fibre content. This means the load applied to the composite will be carried by the fibre, when there is a less fibre content in a composite. The deflection in the maize beam composite is shown in Figure 5.10 and stress intensity in Figure 5.11. The von Mises stresses are one of the important factors and they were shown in Figure 5.12. It illustrates the stress distribution for a composite material, fibre and interphase and exhibited complicated stress distribution. It shows the level of stresses in the the fibre matrix region. There exist two important zones of stress concentration they are fibre and fibre-matrix. The most stress concentration was occurred at the interface region of the composite material. The von Mises stress shows high stress concentration in the fibre and interphase region (This was because von crack initiation occurs only at the poor interphase region (fibre/matrix). When load is given to the material, amount of stress concentration will develop at the weak interphase region of fibre/matrix. Hence stress concentration will be more at the interphase region) and von Mises strain is depicted as shown in Figure 5.13 and Nodal displacement along X direction as shown in Figure 5.14. The results obtained from experimental and FEM are in well agreement and is shown in Table 5.1. The obtained deflection in FEM was DMX (Maximum Deflection) of 8.22 where as in experimental work it was 7.54 mm.

Table 5.1: Deflection Results for Maize Fibre Composite

Sl. No.	Composite	Max. FEM Deflection (mm)	Max. Experimental Deflection (mm)
1	Maize Composite	7.54	8.22

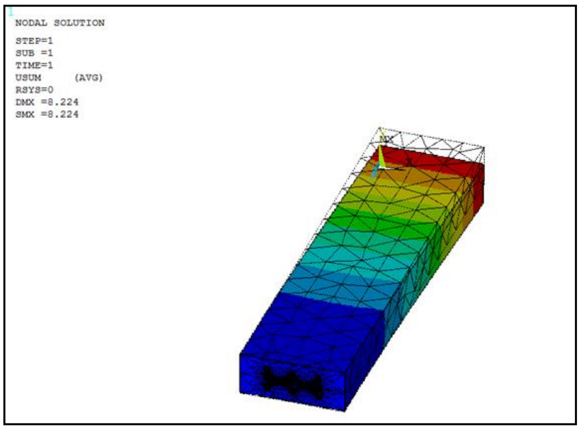


Figure 5.10: Deflection in the Beam Composite

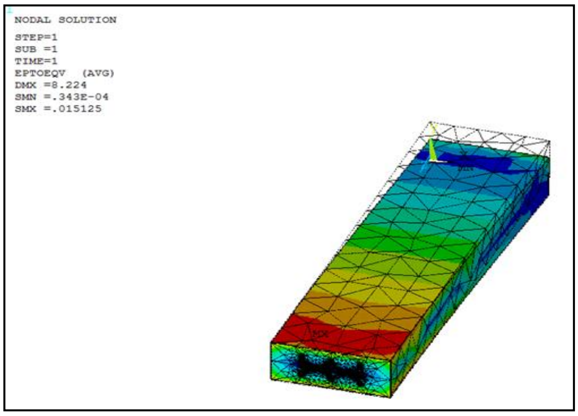


Figure 5.11: Stress Intensity in the Beam Composite

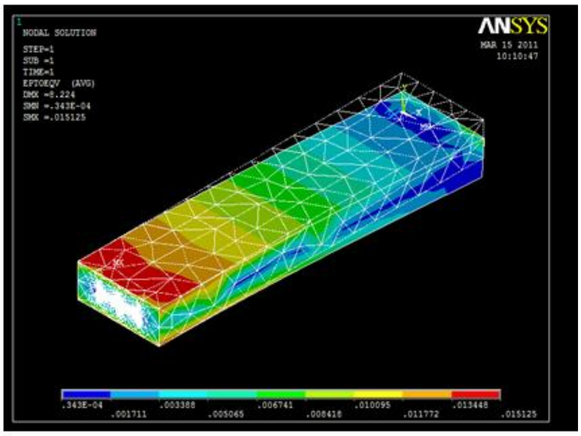


Figure 5.12: Von Mises Stress in the Composite Beam

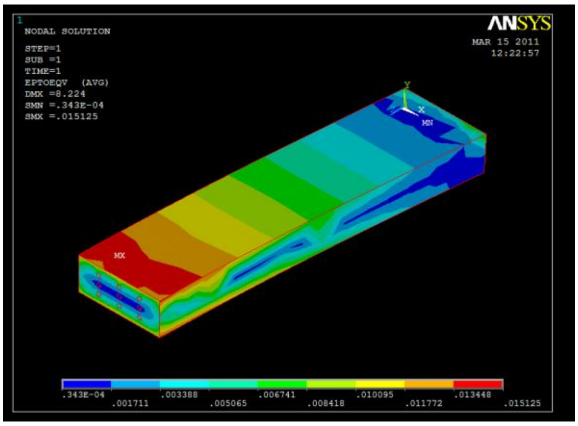


Figure 5.13: Von Mises strain in the Composite Beam

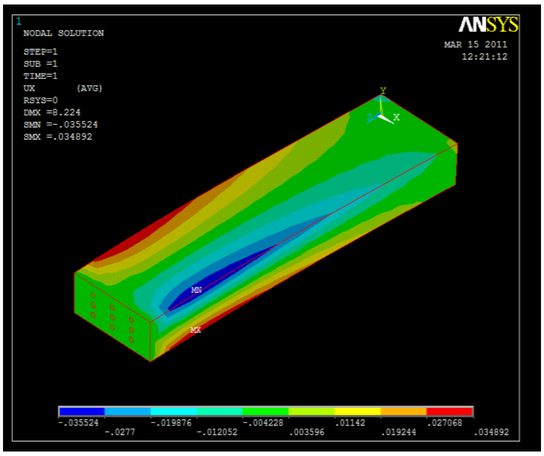


Figure 5.14: Nodal Displacements along X -Direction in the Composite Beam

5.4.2 Thermal Analysis

Figure 5.15 displays the maize long fibre placed with thermoset polymer matrix in 3D mesh. The long fibres are placed in unidirectional manner in a composite model. Figure 5.16 and 5.17 shows the front view mesh and temperature distribution in the composite material. Long fibre and the matrix are glued at the interface for the nodal connectivity. Figure 5.18 shows the contour plot for temperature distribution along the composite. Figure 5.19 and 5.20 shows the heat flux along the composite and vector plot indicating the direction of heat flow.

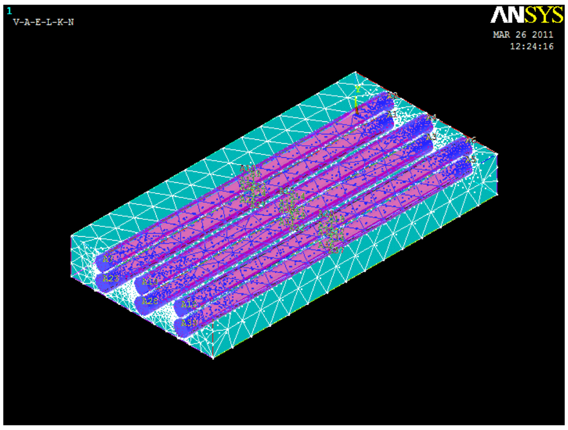


Figure 5.15: Maize Fibre Reinforced With Polymer, 3D Mesh

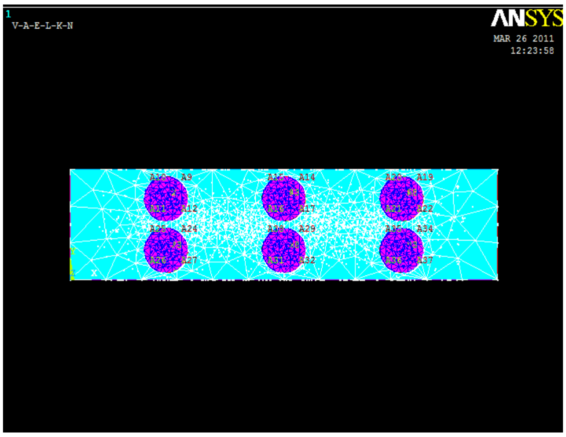


Figure 5.16: Front View – Mesh

Table 5.2 displays the value of k (thermal conductivity) of maize fibre reinforced composite. Figure 5.21 shows the variation in transverse thermal conductivity for different fibre volume fractions for maize fibre reinforced with polymers and thermal conductivity for maize fibre reinforced with polymer composites.

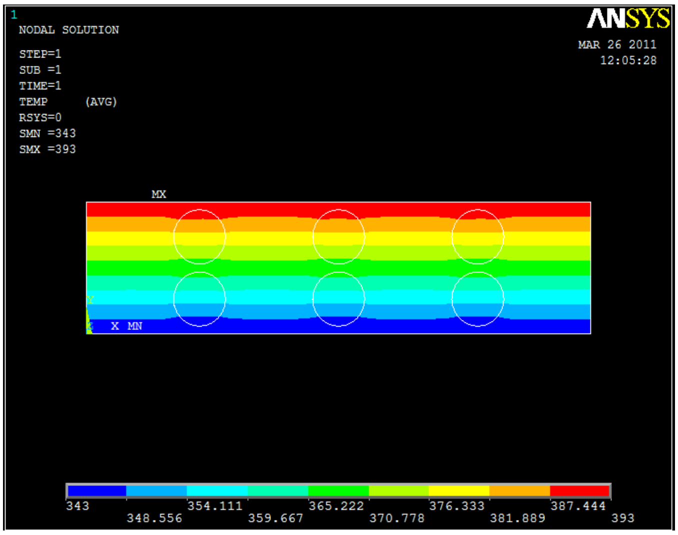


Figure 5.17: Temperature Distributions in the Beam (Front View)

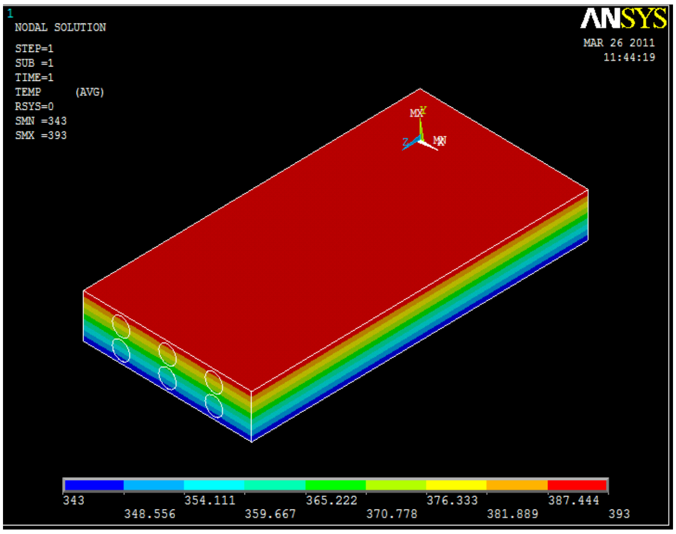


Figure 5.18: Contour Plot for Temperature Distribution along the Composite Beam

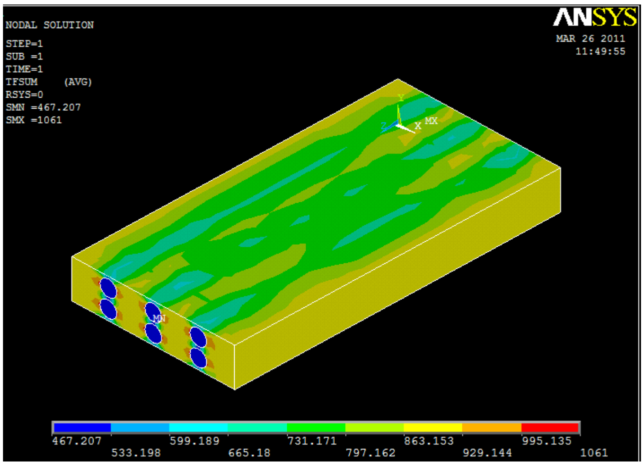


Figure 5.19: Heat Flux along the Composite Beam

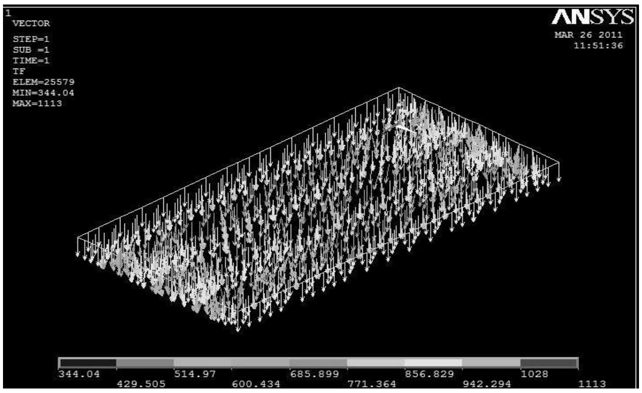


Figure 5.20: Vector Plot Indicating Direction of Heat Flow

Table 5.2: Thermal Conductivity of Maize Fibre Reinforced Polymer Composite with Different Volume Fractions

Sl. No.	V_f	k_f / k_m	k_{eff} (FEM)	k_{eff} (Analy)	k_{eff} (Expt)	k^+ (Expt)	k^+ (Analy)
1	0	0.494	0.182	0.182	0.205	1	1
2	0.13	0.494	0.167	0.164	0.192	0.936	0.901
3	0.18	0.494	0.162	0.158	0.189	0.921	0.868
4	0.24	0.494	0.155	0.151	0.169	0.824	0.829

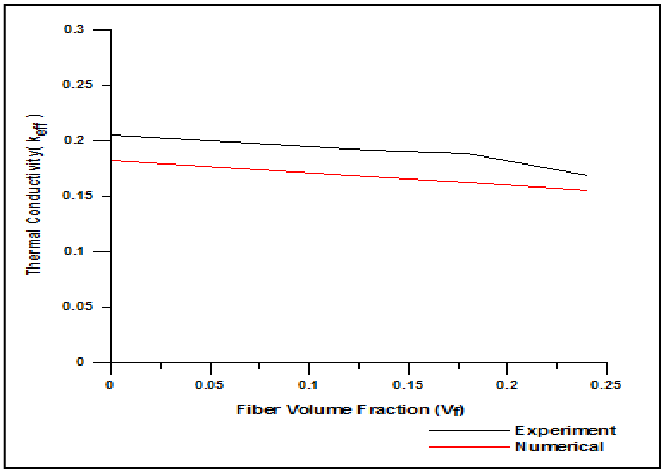


Figure 5.21: Variation of Thermal Conductivity for Maize Fibre Reinforced With Polymeric Resin

Thermal conductivity for Unidirectional composite and derivations for obtaining k_{eff} and k^+ were included in the Apendix-1. The values were substituted in the equations obtained from Appendix-1 for analytical value. The corresponding analytical models (square packing unit cell model) are explained in Appendix-I.

In the table 5.2, it is seen that thermal conductivity decreases with increase in volume fraction, this was because the thermal conductivity has influence on particle size, content and volume of natural fibre in the composite. When the specific surface area increases beyond certain volume fraction of fibres, thermal conductivity decreases. The other reasons can be agglomeration of particles at high volume fraction fibres and poor thermal conductivity of the fibres.

The temperature distributions are very important in composites and their applications. However, many difficulties arising in the design and use of composites are caused by the complexity and poor understanding of the interactions between the composite components. In this study an approximated analytical solution for the temperature distribution along the fibre embedded in a matrix subjected to a differential of temperature was discussed. This analysis is based on the approximate analysis between the maize long fibre and the matrix and helps in understanding the heat dissipation in a fibrous composite.

5.5 Summary

From the FEA analysis, it is confirmed that there is stress concentration in the matrix and fibre interface. Finite element method simulation reveals that the model has more stiffness than the actual. Thermal analysis for thermal conductance from FEM has a close agreement with the results obtained from analytical and experimental. Thermal conductivity varies with % fibre content. Theraml conductivity K_{eff} decrease with increase in fibre content. From the experimental results it is concluded that the maize fibre composite strength can be enhanced with additional layer of jute fibre on outer surfaces. Properties of composites were comparable with wood particle boards.

CHAPTER 6

CONCLUSIONS

Natural fibre composite materials are in much demand in present and future era since they are environmentally friendly green materials. From literature review, it has been concluded that the natural fibres as reinforcement in composite materials have gained new interests in materials sector. Indeed, ecological considerations such as recyclability and environmental friendly products are the new driving forces in our society where pollution and global warming issues have become almost incontrollable. Natural fibres can be potential substitutes for synthetic fibres in many applications where high strength and modulus are not required. The results of many researchers indicated that it can be alternate suitable material for structural applications. In general, hybrid composite materials possess different features that can be used to meet different design requirements with respect to strength and stiffness, thereby improving much more performance in the material.

In the present work processing of composites by Vacuum Assisted Resin Transfer Molding (VARTM) technique for maize natural fibres reinforced with polymers proved out to be a successful method for initiating in small works. The advantage of VARTM technique is that it created a void-free laminate and had lightweight with minimum usage of resin. It is also possible to manufacture large products with good mechanical properties suitable for automotive and aerospace areas. Hand lay-up was the cheapest method of manufacturing but it has some disadvantages such as long curing time, low production rate, and further the quality of the composite depends on the skill of the worker.

The main conclusions are

- An advanced method of processing natural fibre composite (maize fibre composite) by Vacuum Assisted Resin Transfer Molding (VARTM) was carried out at lab level and the method can be improved, modernized and adopted for processing composites at commercial level for the benefit and welfare of the society.

- Green composite familiarly known as Bio-composite was fabricated with the help of biopolymeric resin (epodizied soy bean oil) and was successfully processed by VARTM technique.
- Mechanical test, characterization studies, morphological and thermal analysis of maize fibre and various composites were performed and the out come results were summarized for each test. Among the entire test performed, 25% weight fraction of fibres in the composite material showed a reasonable acceptable values.
- Thermal conductivity for maize fibre was carried out and the results showed satisfactory values. These values were compared with analytical model and the predicted results were appreciated.
- Finite element analysis was carried for long maize stalk fibres and the approximated analytical solution for the temperature distribution along the fibre in a matrix was talked about. This helps in understanding the thermal conductivity in fibrous composite.
- Finally a low cost fibre from nature was developed to build a composite material that can be suitable for structural applications.

6.2 Recommendations for Future Work

The biodegradability of maize fibres can be studied and it may contribute a healthy and good ecosystem. Considering the economic interest of rural areas the maize fibre composite can be processed for interior buildings and constructions because of their low cost and high quality performance. Recycling of maize composites and reuse of these composites can be considered for further work. Sustainability of the composites can also be considered for future work in implementing suitable models.

Some of the futuristic works that can be carried out are

- Optimization techniques such as ANNOVA, Taguchi method can be performed for processing natural fibre composite material for various processing factors.

- Developing a nano-composite material from natural fibres for packaging and water treatment applications.
- Developing more analytical models and analyzing these model results with finite element method.
- Developing natural fibre composite material for exterior applications and large size component structures.

APPENDIX-1

MATHEMATICAL MODEL FOR TRANSVERSE THERMAL CONDUCTIVITY OF UNIDIRECTIONAL FIBRE COMPOSITE

Researchers Islam and Pramila, 1999, Zou et al. 2002, Esparragoza et al 2003, Sulaiman et al. 2006 and Patnaik et al. 2010 had worked on these problems based on finite element method and the obtained method and results shown were part of it. Figure A.1 displays cross sectional view of a fibre-reinforced composite with unidirectional fibres and Figure A.2 is a generalized unit cell (representative cell) of the C–S model [Zou et al. 2002]. One-dimensional heat flow was assumed as shown in Figure A.2 and because of the symmetry of the geometry, top and bottom face of the unit cell they are approximated to be adiabatic. One-dimensional heat flow was applicable and heat conduction in *y*-direction was thus neglected. Further choosen one quadrant of the unit cell for thermal electric analogy analysis of the transverse thermal conductivity is shown in Figure A.3. Assumed functions are ‘a’, and ‘r’ to be the side length of the unit cell and the radius of a cylindrical fibre filament. Let the axial length be unity, R_1 and R_2 are the thermal resistances and further derivations are shown below.

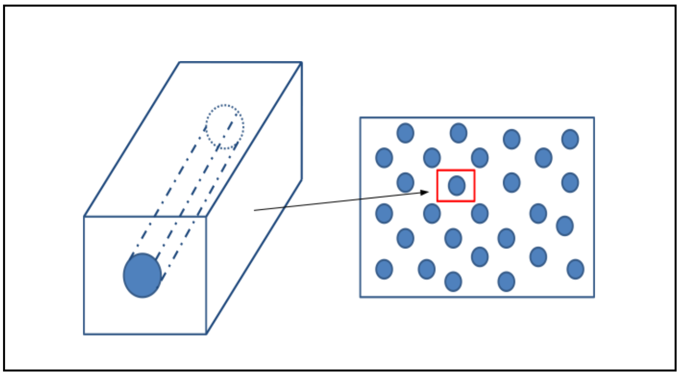


Figure A.1: Cylindrical Filament – Square Packing Unit Cell Model (C-S)

[Zeu et al. 2002]

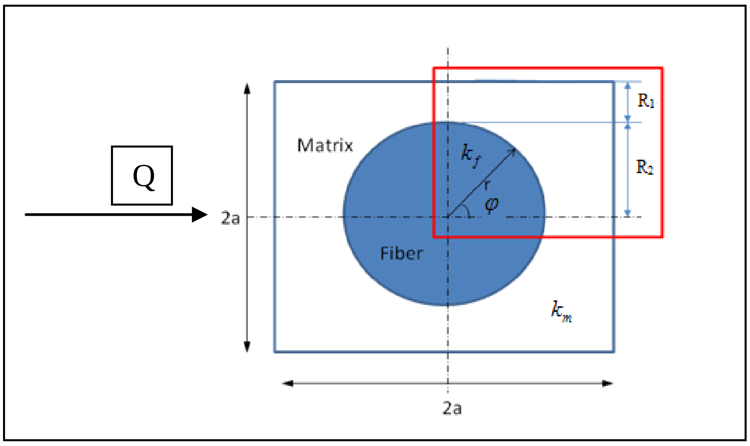


Figure A.2: Schematic of a Single Unit Cell

[Zeu et al. 2002]

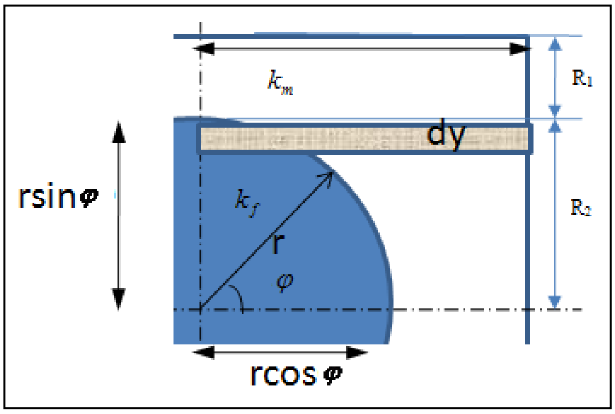


Figure A.3: Enlarged View of a Quarter Unit Cell

[Zeu et al. 2002]

$$R_1 = \frac{a}{k_m(a-r)} \tag{A.1}$$

$$R_2 = R_f + R_m \tag{A.2}$$

$$\int_0^{\pi/2} dr_2 = \int_0^{\pi/2} (dr_f + dr_m) \qquad \text{(A.3a)}$$

$$dr_f = \frac{r\cos\varphi}{k_f dy}$$

$$y = r\sin\varphi$$

$$dy = r\cos\varphi \, d\varphi$$

Here ‘dy’ is the thickness of small element considered and φ varies from 0 to $\frac{\pi}{2}$

$$dr_f = \frac{r\cos\varphi}{k_f r\cos\varphi d\varphi}$$

$$dr_m = \frac{a - r\cos\varphi}{k_m r\cos\varphi d\varphi}$$

$$R_2 = \int_0^{\pi/2} dr_f + \int_0^{\pi/2} dr_m \qquad \text{(A.3b)}$$

$$R_2 = \int_0^{\pi/2} \frac{r\cos\varphi}{k_f r\cos\varphi d\varphi} + \int_0^{\pi/2} \frac{a - r\cos\varphi}{k_m r\cos\varphi d\varphi}$$

$$Q = \frac{k_{eff} A \Delta T}{\Delta x} \qquad \text{(A.4a)}$$

$$Q = \frac{k_{eff} \, 2a \Delta T}{2a}$$

$$Q = \frac{\Delta T}{R_t} \qquad \text{(A.4b)}$$

$$k_{eff} = \frac{1}{R_t} = \frac{1}{R_1} + \frac{1}{R_2}$$

$$k_{eff} = \frac{1}{R_1} + \frac{1}{R_f} + \frac{1}{R_m} \tag{A.4c}$$

$$\frac{1}{R_2} = \frac{1}{\int_0^{\pi/2} \frac{r\cos\varphi}{k_f r\cos\varphi} + \int_0^{\pi/2} \frac{a - r\cos\varphi}{k_m r\cos\varphi}} \tag{A.5}$$

$$k_{eff} = \frac{1}{a/k_m(a-r)} + \frac{1}{\int_0^{\pi/2} \frac{r\cos\varphi}{k_f r\cos\varphi d\varphi} + \int_0^{\pi/2} \frac{a - r\cos\varphi}{k_m r\cos\varphi d\varphi}} \tag{A.6}$$

$$k_{e2} = \frac{1}{\int_0^{\pi/2} \frac{r\cos\varphi}{k_f r\cos\varphi d\varphi} + \int_0^{\pi/2} \frac{a - r\cos\varphi}{k_m r\cos\varphi d\varphi}} \tag{A.7}$$

$$k_{e2} = \int_0^{\pi/2} \frac{\cos\varphi d\phi}{\frac{\cos\varphi}{k_f} + \frac{\frac{a}{r} - \cos\varphi}{k_m}} \tag{A.8}$$

$$k_{e2} = \int_0^{\pi/2} \frac{k_m \cos\varphi d\phi}{\frac{k_m\cos\varphi}{k_f} + \frac{k_m\left(\frac{a}{r} - \cos\varphi\right)}{k_m}} \tag{A.9}$$

$$\frac{k_{e2}}{k_m} = \int_0^{\pi/2} \frac{\cos\varphi d\phi}{\frac{k_m\cos\varphi}{k_f} + \frac{k_m\left(\frac{a}{r} - \cos\varphi\right)}{k_m}} \tag{A.10}$$

Let $\delta = \frac{a}{r}$ (A.11a)

$$v_f = \frac{\pi r^2}{4a^2} \tag{A.11b}$$

$$\delta = \frac{1}{2}\sqrt{\frac{\pi}{v_f}} \tag{A.11c}$$

$$\varepsilon = \frac{k_f}{k_m} \tag{A.11d}$$

$$\eta = \frac{1}{\varepsilon} - 1 \tag{A.11e}$$

$$\frac{k_{e2}}{k_m} = \int_0^{\pi/2} \frac{\cos\varphi d\phi}{\cos\varphi\left(\frac{k_m}{k_f} - 1\right) + \frac{a}{r}} \tag{A.12}$$

Using (A.11c) and (A.11e) in (A.12)

$$\frac{k_{e2}}{k_m} = \int_0^{\pi/2} \frac{\cos\varphi d\phi}{\eta\cos\varphi + \delta} \tag{A.13}$$

$$\frac{k_{e1}}{k_m} = \frac{1}{a/(a-r)} \tag{A.14}$$

$$\frac{k_{e1}}{k_m} = \frac{a-r}{a}$$

$$\frac{k_{e1}}{k_m} = 1 - \frac{1}{\delta} \tag{A.15}$$

$$k_{eff} = k_{e1} + k_{e2} \tag{A.16}$$

$$\frac{k_{eff}}{k_m} = 1 - \frac{1}{\delta} + \int_0^{\pi/2} \frac{\cos\varphi d\varphi}{\eta\cos\varphi + \delta} \tag{A.17}$$

Let $$I = \int_0^{\pi/2} \frac{\cos\varphi d\varphi}{\eta\cos\varphi + \delta} \tag{A.18}$$

$$I = \frac{1}{\eta}\int_0^{\pi/2} \frac{\cos\varphi d\varphi}{\cos\varphi + \delta/\eta} \tag{A.19}$$

$$I = \frac{1}{\eta}\int_0^{\pi/2} \frac{\cos\varphi + \delta/\eta}{\cos\varphi + \delta/\eta} d\phi - \frac{1}{\eta}\int_0^{\pi/2} \frac{\delta/\eta}{\cos\varphi + \delta/\eta} d\phi \tag{A.20}$$

$$I = \frac{1}{\eta}\frac{\pi}{2} - \frac{1}{\eta}\int_0^{\pi/2} \frac{\delta/\eta}{\cos\varphi + \delta/\eta} d\phi \tag{A.21}$$

Let $$\tan\frac{\varphi}{2} = t$$

$$\frac{1}{2}\sec^2\frac{\varphi}{2} d\varphi = dt$$

$$\left(1 + t^2\right) d\varphi = 2dt$$

$$d\varphi = \frac{2dt}{1 + t^2}$$

For $\varphi = 0, t = 0$ and for $\varphi = \frac{\pi}{2}, t = 1$

$$\cos\varphi = \frac{1-t^2}{1+t^2}$$

$$I = \frac{1}{\eta}\frac{\pi}{2} - \frac{1}{\eta}\int_0^1 \left(\frac{\delta/\eta}{1-t^2/1+t^2 + \delta/\eta} \right) \frac{2dt}{1+t^2} \tag{A.22}$$

$$I = \frac{1}{\eta}\frac{\pi}{2} - \frac{2\delta}{\eta^2}\int_0^1 \left(\frac{\frac{dt}{1+t^2}}{\frac{1-t^2}{1+t^2} + \frac{\delta}{\eta}} \right) \tag{A.23}$$

$$I = \frac{\pi}{2\eta} - \frac{2\delta}{\eta}\int_0^1 \frac{\eta dt}{\left(1-t^2\right)\eta + \left(1+t^2\right)\delta} \tag{A.24}$$

$$I = \frac{\pi}{2\eta} - \frac{2\delta}{\eta}\int_0^1 \frac{dt}{\left(\delta-\eta\right)t^2 + \left(\delta+\eta\right)} \tag{A.25}$$

$$I = \frac{\pi}{2\eta} - \frac{2\delta}{\eta(\delta-\eta)}\int_0^1 \frac{\eta dt}{t^2 + \left(\frac{\delta+\eta}{\delta-\eta}\right)}$$

$$I = \frac{\pi}{2\eta} - \frac{2\delta}{\eta(\delta-\eta)}\int_0^1 \frac{\eta dt}{t^2 + \sqrt{\left(\frac{\delta+\eta}{\delta-\eta}\right)}^2} \tag{A.26}$$

$$I=\frac{\pi}{2\eta}-\frac{2\delta}{\eta\sqrt{\left(\delta^2-\eta^2\right)}}\tan^{-1}\left(\frac{\sqrt{(\delta^2-\eta^2)}}{(\delta+\eta)}\right) \tag{A.27}$$

$$\frac{k_{eff}}{k_m}=1-\frac{1}{\delta}+I \tag{A.28}$$

Using (A.27) in (A.28)

$$\frac{k_{eff}}{k_m}=1-\frac{1}{\delta}+\frac{\pi}{2\eta}-\frac{2\delta}{\eta\sqrt{\left(\delta^2-\eta^2\right)}}\tan^{-1}\left(\frac{\sqrt{(\delta^2-\eta^2)}}{(\delta+\eta)}\right) \tag{A.29}$$

Let $k^+=\frac{k_{eff}}{k_m}$, then $\text{for}\left(\delta^2>\eta^2\right)$

$$k^+=1-\frac{1}{\delta}+\frac{\pi}{2\eta}-\frac{2\delta}{\eta\sqrt{\left(\delta^2-\eta^2\right)}}\tan^{-1}\left(\frac{\sqrt{(\delta^2-\eta^2)}}{(\delta+\eta)}\right) \tag{A.30}$$

For $\left(\delta^2<\eta^2\right)$

Using Equation (A.19) $I=\frac{1}{\eta}\int_0^{\pi/2}\frac{\cos\varphi d\varphi}{\cos\varphi+\delta/\eta}$

$$I=\frac{\pi}{2}-\frac{2\delta}{\eta}\int_0^1\frac{dt}{\left(\delta-\eta\right)t^2+(\delta+\eta)} \tag{A.31}$$

Rearranging Equation (A.31)

$$I = \frac{\pi}{2} - \frac{2\delta}{\eta} \int_0^1 \frac{dt}{(\delta + \eta) - (\eta - \delta)t^2}$$

$$I = \frac{\pi}{2} - \frac{2\delta}{\eta(\eta - \delta)} \int_0^1 \frac{\eta dt}{\sqrt{\left(\frac{\eta + \delta}{\eta - \delta}\right)^2 - t^2}} \tag{A.32}$$

Using (A.32) in (A.28)

$$k^+ = 1 - \frac{1}{\delta} + \frac{\pi}{2\eta} - \frac{\delta}{\eta\sqrt{(\eta^2 - \delta^2)}} \log\left(\frac{\eta + \sqrt{(\eta^2 - \delta^2)}}{\delta}\right) \tag{A.33}$$

The Equations (A.30) and (A.33) were used to validate numerical simulations. It can be seen from the above simplified equations, effective thermal conductivity is mainly dependent on the volume fraction of fibres, and thermal conductivities of fibre and matrix.

APPENDIX II

Various shapes of natural plant fibre composite material obtained from maize fibres are shown below.

Figure A.4: Different Product Material Obtained from Maize Fibres

REFERENCES

Alamri, H. and Low, I. M. (2013). “Effect of water absorption on the mechanical properties of nano clay filled recycled cellulose fibre reinforced epoxy hybrid nano composites.” *Compos. A.*, 44(1), 23-31.

Alawar, A. and Hamed, A. M. (2009). “Characterization of Treated Date Palm Tree Fibre as Composite Reinforcement.” *Compos. B.*, 40(7), 601-606.

Albano, C., Gonzalez, J., Ichazo, M. and Kaiser, D. (1999). “Thermal stability of blends of polyolefins and sisal fiber.” *Polym. Degrad. Stabil.*, 66(2), 179-90.

Alix, S., Marais, S., Morvan, C., and Lebrun, L. (2008). “Biocomposite materials from flax plants: Preparation and properties.” *Compos. A.*, 39(12), 1793-1801.

Allred R. E., Doak T. J., Coons A. B., Newmeister G. C. and Cochran R. C. (1997). “Tertiary recycling of cured composite aircraft parts.” *Society of Manuf. Eng*. EM, 97(110), X-17.

Alsaeed, T., Yousif, B.F. and Ku, H. (2013). “The potential of using date palm fibres as reinforcement for polymeric composites.” *Mater. Des.,* 43, 177-184.

Aly, M. F., Goda, I. G. M., and Hassan, G. A. (2010). “Experimental Investigation of the Dynamic Characteristics of Laminated Composite Beams.” *Intl. J. Mech. Mechtncs.*, 10(03), 41-48.

Andrady, A. L. (2011). “Microplastics in the marine environment” *Marine Pollut. Bull.* 62(8), 1596-1605.

Angelini, L. G., Lazzeri, A., Levita, G., Fontanelli, D and Bozzi, C. (2000). “Ramie (Boehmeria nivea L.) Gaud and Spanish Broom (Spartium junceum L.) fibres for composite materials: agronomical aspects, morphology and mechanical properties." *Indl. Crops. Prod.*, 11, 145-161.

Ashori, A. (2008). "Wood–plastic composites as promising green-composites for automotive industries." *Bioresource. Tech.*, 99(11), 4661-4667.

Averous, L. and Pollet, E. (2012). "Biodegradable Polymers", *Environmental silicate nano – biocomposites*, Averous, L. and Pollet, E. eds. Springer -Verlag, 13-39.

Aziz, M. A., Paramasivam, P. and Lee, S. L. (1984). "Concrete reinforced with natural fibres". Natural fibre reinforced cement and concrete, R. N. Swamy, eds. chapter 3, 106-140 (1984). Blackie and Son Ltd, London.

Bakar, B. F. A., Hiziroglu, S. and Tahir, P. M. (2013). "Properties of some thermally modified wood species." *Mater. Des.* 43, 348-355.

Balac, I., Colic, K., Milovancevic, M.., Uskokovic, P. and Zrilic, M. (2012). "Modeling of the Matrix Porosity Influence on the Elastic Properties of Particulate Biocomposites", *FME Transactions*, 40, 81-86.

Bayat, M. and Aghdam, M. M. (2012). "A micromechanics based analysis of hollow fibre composites using DQEM", *Compos: B.*, 43(8), 2921–2929.

Bayldon, J.M. and Daniel, I. M. (2009). "Flow modeling of the VARTM process including progressive saturation effects." *Compos. A.*, 40(8), 1044-1052.

Beckwith, S.W. (2007). "Resin Infusion Technology, Part 3- A Detailed overview of RTM and VIP infusion processing technologies." *Sampe J.*, 43(4).

Behzad, T. and Sain, M. (2007). "Finite element modeling of polymer curing in natural fibre reinforced composites", *Compos. Sci. Technol.* 67(7-8), 1666–1673.

Bismarck, A., Mohanty, A. K., Askargorta, I. A., Czapla, S., Misra, M., Hinrichsen, G. and Springer, J. (2001). “Surface characterization of natural fibres, surface properties and water uptake behavior of modified sisal and coir fibres” *Green Chem,* 3(2), 100-107.

Bledzki, A.K. and Gassan J. (1999). “Composites reinforced with cellulose based fibre.” *Prog. Polym. Sci*., 24(2), 221-274.

Bledzki, A.K., Reihmane, S., and Gassan, J. (1996). “Properties and modification methods for vegetable fibres for natural fibre composites.” *J. Appl. Polym. Sci.,* 59(8), 1329-1336.

Bledzki, A.K., Sperber, V.E. and Faruk, O. (2002). “Natural and wood fibre reinforcement in polymers.” *Rapra Review Reports*, 3(8), 152.

Bledzki, A.K., Zhang, W. and Chate, A. (2001). “Natural-fibre-reinforced polyurethane micro foams.” *Compos. Sci. Technol.*, 61(16), 2405-2411.

Burgueno, R., Quagliata, M. J., Mohanty, A. K., Mehta, G., Drzal, L. T., Misra, M. (2005). “Hierarchical cellular designs for load-bearing bio composite beams and plates.” *Mater. Sci. Eng.*, 390(1-2), 178–187.

Callister, W. D. (2007). Materials Science and Engineering: An Introduction. John Wiley and Sons, New York, 4-70.

Campbell, F.C. (2004). Manufacturing Processes for Advanced Composites, Elsevier Publications, U.K. 3-100.

Carraher, Jr. C.E. (2012). Introduction to Polymer Chemistry, Third Edition, CRC Press. 1-70.

Chawla, K. K. (1987). Composite Materials. Science and Engineering. Springer-Verlag, New York. 3-101.

Chen, D., Li, M., and Zhu, X. (2012). "TG-DSC method applied to drying characteristics and heat requirement of cotton stalk during drying" *Heat Mass Transfer*, 48, 2087-2094.

Chigondo, F., Shoko, P., Nyamunda, C. B., Guyo, U., and Moyo, M. (2013). "Maize Stalk as Reinforcement in Natural Rubber Composites" *Intl. J. Scientific & Tech.* 2(6), 263-271.

Chou, T. W. and Ko, F. K. (1989). *"Textile structural composites*", Composite Materials Series, Elsevier Science Publishers, 1-26. New York/Amsterdam.

Christian, S. J. and Billington, S. L. (2009). Sustainable Biocomposites for Construction, Proceedings for C*omposites & polycon*, 36: 2107-2131. Tampa, FL USA.

Cichocki Jr., F. R. and Thomason J. L. (2002). "Thermo elastic anisotropy of a natural fibre." *Comp. Sci Technol*., 62, 669-678.

Collier, B. J., Collier, J. R., Agarwal, P., and Lu, Y-W. (1992). "Extraction and Evaluation of Fibres from Sugar Cane." *Textile Resrch. J.*, 62(12), 741-748.

Das, P. K., Nag, D., Debanth, S., and Nayak, L.K. (2010). "Machinery for extraction and traditional spinning of plant fibres," *Indian J. Trad. Knwldg*, 9 (2), 386- 393.

Dauda, M., Yoshiba, M., Miura, K., and Takahashi, S. (2007). "Processing and mechanical property evaluation of maize fibre reinforced green composites" *Adv. Composite Mater.*, 16(4), 335–347.

Dias, R., Filho, T., Joseph, K., Ghavami, K. and England, G. L. (1999). "The use of sisal fibre as reinforcement in cement based composites." *Eng. Agrí. Ambiental,* Campina Grande, 3(2), 245-256.

Donnel, L. A. O., Dweib, M. A., and Wool, R. P. (2004). "Natural fibre composites with plant oil-based resin." *Compos. Sci. Technol.*, 64(9), 1135-1145.

Doraiswamy, I. and Chellamani, P. (1993). "Pineapple-leaf fibres." *Textile Progr,* 24(1), 1-25.

Duigou, A. L., Davies, P., and Baley, C. (2010), "Interfacial bonding of Flax fibre/Poly (l-lactide) bio-composites." *Compos. Sci. Technol.,* 70(2), 231-239.

Dweib, M.A., Hu, B., Donnell, A. O., Shenton, H.W. and Wool, R.P. (2004). "All natural composite sandwich beams for structural applications." *Compos. Struct.* 63(2), 147-157.

Dweib, M.A., Hu, B., Shenton, H.W. and Wool R.P. (2006). "Bio-based composite roof structure: Manufacturing and processing issues." *Compos. Struct.* 74(4), 379-388.

ECO-PCCM, "Eco-houses based on Eco-friendly Polymer Composite Construction Materials" FP6- 2002-INCO-WBC.

Eichhorn, S. J., and Young, R. J. (2003). "Deformation micromechanics of natural cellulose fibre networks and composites." *Compos. Sci. Technol.*, 63(9), 1225-1230.

Eichhorn, S. J., Baillie, C. A., Zafeiropoulos, N., Mwaikambo, L. Y., Ansell, M. P. and Dufresne, A. (2001). "Current international research into cellulosic fibres and composites." *J. Mater. Sci.*, 39(9), 2107-2131.

Elbadry, E. A., Hassan, M. S. A. and Hamada, H. (2012). “Mechanical Properties of Natural Jute Fabric/Jute Mat Fibre Reinforced Polymer Matrix Hybrid Composites.” *Advn. Mech. Eng.*, Id 354547.

Ellision, G.C., and Mc Naught, R. (2000). “The use of natural fibres in non woven structures for applications as automotive component substrates.” UK Govt MAFF report reference NF0309, February 2000.

Erakhrumen, A. A., Areghan, S. E., Ogunleye, M. B., Larinde, S. L. and Odeyale, O. O. (2008). “Selected physic mechanical properties of cement bonded particle board made from pine (Pinus caribaea M.) Sawdust-coir (Cocos nucifera L.) mixture.” *Scient. Resrch. Essay.* 3 (5), 197-203.

Erenstein, O. (2010). “The evolving maize sector in Asia: Challenges and opportunities”, *J. New Seeds.* 11(1), 1–15.

Esmeraldo, M. A., Baretto, A. C. H., Freitas, J. E. B., Fechnie, P. B. A., Sombra, A. S. B., Corradini, E., Mele, G., Maffezzoli, A. and Mazzetto, S.E. (2010).”Dwarf green coconut fibres-A versatile natural renewable raw bioresource, treatment, morphology, and physiochemical properties” *Biores.* 5 (4), 2478-2501.

Esparragoza, I. E., Aziz, A. H. and Damle, A.S. (2003), “Temperature distribution along a fibre embedded in a matrix under steady state conditions”, *Compos B*, 34, 429–436.

Espert, A., Vilaplana, F. and Karlsson, S., (2004). “Comparison of water absorption in natural cellulosic fibres from wood and one-year crops in polypropylene composites and its influence on their mechanical properties.” *Compos. A.*, 35(11), 1267-1276.

Fan. M. (2010) “Characterization and performance of Elementary hemp fibres: factors influencing tensile strength”, *Bioresources* 5(4), 2307-2322.

Fidelis, M. E. A., Pereira, T.V.C., Gomes, O.F.M., Silva, F.A., and Filho, R. D. T. (2013). The effect of fibre morphology on the tensile strength of natural fibres, *J. Mater Res. Tech.*, 2(2), 149-157.

Foscher, B., Marzetti, A., Beltrame. P.L. and Avella M. (1998). “Steam exploded biomass for the preparation of conventional and advanced biopolymer-based materials.” *Bioms Bioenrgy.*, 14(3), 187-194.

Fowler, P. A., Hughes, M. J. and Elias, R. M. (2006). “Review Biocomposites: technology, environmental credentials and market forces.” *J. Sci. Food Agric*. 86(12), 1781–1789.

Franck, R. R. (2005). “Bast and other plant fibres.” Wood head Publisher, Cambridge, U.K. 1-158.

Fukazawa, K., Revol, J. F., Jurasek, L., and Goring, D. A. I., (1982). “Relationship between ball milling and the susceptibility of wood to digestion by cellulose.” *Wood sci. Tech*. 16 (1), 279- 285.

Garkhail, S. K., Heijenrath, R. W. H. and Peijs, T. (2000). “Mechanical Properties of Natural Fibre-Mat- Reinforced Thermoplastics based on Flax Fibres and Polypropylene.” *Appl. Compos. Mater.* 7(5-6), 351-372.

Gassan, J. and Bledzki, A.K. (1997). “The influence of fibre-surface treatment on the mechanical properties of jute-polypropylene composites.” *Compos. A.*, 28(12), 1001-1005.

Gerdeen, J.C. and Rorrer, R.A.L. (2011). “Engineering Design with Polymers and Composites”, Second Edition, CRC Press.1-240.

Giancaspro, J., Papakonstantinou, C. and Balaguru, P. (2009). “Mechanical behavior of fire-resistant biocomposite.” *Compos. B*, 40(3), 206-211.

Glasser, W. G. and Sarkanen, S. (Eds.). (1989) "Lignin: Properties and Materials."ACS, Washington, DC. Series 397.

Golbabaie, M. (2006). "Applications of biocomposites in building industry." Department of plant agriculture, University of Guelph.

Green building materials for low cost housing, Science tech Entrepreneur, June 2009.

Green door (2000) - Trim panels use PP and Natural fibres, *Plast. Technol*, 46, 27.

Grozdanov A., Gaceva, B. G., Buzarovska, A., Avella, M., Gentile, G. and Dekanski A. (2006), "Thermal stability of differently treated natural fibre reinforcements for composites." Itnl Conference of the Chemical Societies of the South-East European Countries, Macedonia, Greece.

Gutierrez, M. C., Paoli, M. A. D. and Felisberti, M. I. (2012). "Biocomposites based on cellulose acetate and short curauá fibres: Effect of plasticizers and chemical treatments of the fibres." *Compos. A., 43*, 1338-1346.

Harish, S., Michael, P. D., Bensely, A., Lal, M. D. and Durai, R. A. (2009). "Mechanical property evaluation of natural fibre coir composite." *Mater. Charact.* 60, 44-49.

Henriksson, G., Akin, D.E., Hanlin, R.T., Rodriguez, C., Archibald, D.D., Rigsby, L. L. and Eriksson, K. E. L. (1997). "Identification and Retting Efficiencies of Fungi Isolated from Dew-Retted Flax in the United States and Europe." *Appl. Environ. Microbio.* 63, 3950-3956.

Herrmann, A. S., Nickel, J. and Riedel, U. (1998). "Construction materials based upon biologically renewable resources – from components to finished parts." *Polym. Degrad. Stabil*. 59(1-3), 251-261.

Holbery, J. and Houston, D. (2006). “Natural-fibre-reinforced polymer composites in automotive applications.” *JOM*. 58(11), 80-86.

Huang, J., Shetty, A. S. and Wang, M. (1990). “Biodegradable plastics-A review” Adv. Poly. Tech., 10(1), 23-30.

Hull, D and Clyne, T.W. (1996). An Introduction to Composite Materials. Cambridge University Press, Cambridge, UK.1-.326.

Ibrahim, S.F., El-Amoudy, E. S., and Shady, K. E. (2011). “Thermal Analysis and Characterization of Some Cellulosic Fabrics Dyed by a New Natural Dye and Mordanted with Different Mordants, *Intl. J. Chemistry*, 3(2), 40-54.

IENICA (Ref 1495) Summary Report – Fibre Crops, August 2000.

Ince, A., Ugurluay, S., Guzel, E. and Ozcan, M.T. (2005). “Bending and Shearing Characteristics of Sunflower Stalk Residue.” *Biosystems Engg*, 92 (2), 175-181.

India, the Market for Natural and Man-Made Fibres, Textiles and Textile Manufactures to 2012. International newsletters limited, May, 2009; p. 97. www.researchandmarkets.com/reports/992503.

Ioannidou, O., Zabaniotou, A., Antonakou, E.V., Papazisi, K.M., Lappas, A. A., and Athanassiou, C. (2009). “Investigating the potential for energy, fuel, materials and chemicals production from corn residues (cobs and stalks) by non-catalytic and catalytic pyrolysis in two reactor configurations”, *Renewable and Sustain. Energy Rvws.* 13, 750–762.

Islam, Md. R. and Pramila, A (1999),"Thermal Conductivity of Fibre Reinforced Composites by the FEM", *J. Compos Mater*, 33(18), 1699-1715

Jawaid, M., Khalil, A.H.P.S. and Bakar, A. A. (2010) “Mechanical performance of oil palm empty fruit bunches/jute fibres reinforced epoxy hybrid composites” *Mater. Sci. Engg., A*, 527, 7944-7949.

Jin, A. X., Ren, J. L., Peng, F., Xu, F., Zhou, G.Y., Sun, R.C. and Kennedy, J.F. (2009).“Comparative characterization of degraded and non-degradative hemicelluloses from barley straw and maize stems: Composition, structure, and thermal properties.” *Carbo. Polym.* 78(3), 609-619.

Joffe, R., Andersons, J. and Wallstrom, L. (2003). “Strength and adhesion characteristics of elementary flax fibres with different surface treatments.” *Compos. A.*, 34(7), 603-612.

John, M. J. and Anandjiwala, R. D. (2008). “Recent developments in chemical modification and characterization of natural fibre reinforced composites.” *Polym. Compos.* 29 (2), 187-207.

John, M. J. and Thomas, S. (2008). “Biofibres and Bio composites.” *Carbo. Polym.* 71(3), 343-364.

Joseph, K., Filho, R. D. T., James, B., Thomas, S. and Carvalho, L. H. D. (1999). “A Review on Sisal Fibre Reinforced Polymer Composites.” *Revista Brasileira de Engenharia Agrícola e Ambiental*, 3(3), 367-379.

Joshi, S. V., Drzal, L. T., Mohanty, A. K. and Arora, S. (2004). “Are natural fibre composites environmentally superior to glass fibre reinforced composites.” *Compos. A.*, 35(3), 371-376.

Julian, F.V. and Vincent, V. (2000). “A unified nomenclature for plant fibres for industrial use.” *Appl. Compos. Mater*. 7(5-6), 269-271.

Kabir, M. M., Wang, H., Cardona, F., and Aravinthan, T. (2011). “Effect of chemical treatment on the mechanical and thermal properties of hemp fibre reinforced thermoset sandwich composites” Downloaded from University of Southern Queensland.

Kestur, S. G., Sahagun, T. H. S. F., Santos, L. P. D., Santos, J. D., Mazzaro, I., and Mikowski, A. (2013). “Characterization of blue agave bagasse fibres of Mexico.” *Compos. A*, 45, 153-161.

Khan, B. A. (2007). “Uses of coir fibre, its products & implementation of geo-coir in Bangladesh.” D. I. U., *J. Sci. Technol.*, 2(2).

Khardenavis, A. A., Kumar, M. S., Sandeep, N. M. and Tapan, C. (2007).

“Biotechnological conversion of agro industrial waste waters into biodegradable plastic study,” *Biores Tech.*, 98(18), 3579-3584.

Kim, H.S. and Kim, H. J. (2008). “Enhanced hydrolysis resistance of biodegradable polymers and bio-composites.” *Polym. Degrad. Stabil*, 93(8), 1544-1553.

Kozlowski, R., Mieleniak, B. and Przepiera, A. (1994), “Plant Residues as Raw Materials for Particleboards.” Proc. of the 28th Intern. *Particleboards Composite Materials Symposium*, W. S. U. Pullman, U. S. A.

Krishnan, K. B., Doraiswamy, I. and Chellamani, K. P. (2005). “Jute” *Bast and other Plant Fibres*, R.R. Franck, ed., Wood head Publishing, Cambridge. 24-92.

Kumar, G. C. M, (2008). “A Study of Short Areca Fibre Reinforced PF Composites.”, Proc., *World Congress on Eng*, WCE 2008, London, U.K.

Kumar, K. S., Siva, I., Jeyaraj, P., Jappes, J. T. W., Amico, S.C., and Rajini, N. (2014). “Synergy of fibre length and content on free vibration and damping behavior of natural fibre reinforced polyester composite beams" *Mater. and Design*, 56, 379–386

Kumar, M. A., Reddy, G. R., Mahesh, K. R. V., Chakradhar, K.V.P and Reddy, Y.V.M. (2011). "Performance of Zea mays fibre reinforced epoxy composites" *Intl. J. Fibre and Textile Research,* 1(1), 22-27.

Kumar, M. B., Pavithran, C., and Pillai, R. M. (2005). "Coconut fibre reinforced polyethylene composites: effect of natural waxy surface layer of the fibre on fibre/matrix interfacial bonding and strength of composites." *Compos. Sci Technol.*, 65(3-4), 563-569.

Lawther, J. M., Sun, R. C., and Banks, W. B. (1995). "Extraction, fractionation, and characterization of structural polysaccharides from wheat straw." *J. Agri. Food Chem*., 43(3), 667- 675.

Leja, K. and Lewandowicz, G. (2010)."Polymer biodegradation and biodegradable polymers – A Review" *Polish J. Environt. Stud*., 19(2), 255-266.

Li, X., Tabil, L. G. and Panigrahi, S. (2007). "Chemical treatments of natural fibre for use in natural fibre-reinforced composites: A review." *J. Polym. Envrn.*, 15(1), 25-33.

Lilholt, H. and Lawther, J. M. (2000). "Natural Organic Fibres." *Comprehensive Composite Materials, Fibre Reinforcements and General Theory of Composites* T.W. Chou, Ed. Elsevier, New York, 303-325.

Liu, W., Drzal, L.T., Mohanty, A.K. and Misra, M. (2007). "Influence of processing methods and fibre length on physical properties of kenaf fibre reinforced soy based biocomposites." *Compos. B.,* 38(3), 352-359.

Liu, W., Misra, M., Askeland, P., Drzal, L.T. and Mohanty, A. K. (2005). "Green composites from soy based plastic and pineapple leaf fibre: fabrication and properties evaluation." *Polym.* 46(8), 2710-2721.

Liu, Z. and Erhan S. Z. (2008). "Green composites and nano composites from soybean oil." *Mater. Sci. Eng. A.,* 483/484(1-2c), 708-711.

Lopez, J. L., Sain, M. and Cooper, P. (2006). "Performance of Natural-Fibre–Plastic Composites under Stress for Outdoor Applications: Effect of Moisture, Temperature, and Ultraviolet Light Exposure." *J. Appl. Polym. Sci*., 99(5), 2570-2577.

Madsen, B., Hoffmeyer, P. and Lilholt, H. (2012). "Hemp yarn reinforced composites moisture content and dimensional changes." *Compos. A.*, 43(11), 2151-2160.

Mahato, D. N., Mathur, B. K., and Bhattacherjee, S. (2013). "DSC and IR methods for determination of accessibility of cellulosic coir fibre and thermal degradation under mercerization" *Indian J. Fibre & Textile Research,* 38, 96-100.

Manfredi, L.B., Rodrıguez, E. S., Przybylak, M. W. and Zquez, A. V., (2006). "Thermal degradation and fire resistance of unsaturated polyester modified acrylic resins and their composites with natural fibres." *Polym. Degrad. Stabil.* 91, 255-261.

Manikandan, K. C., Diwan, S. M. and Thomas, S. (1996). "Tensile properties of short sisal fibre reinforced polystyrene composites." *J. Appl. Poly. Sci.*, 60(9), 1483-1497.

Marsh, G. (2003). "Next step for automotive materials." *Materials today*.

Marston, N. J. (2008). "Bio-derived polymer and composites." BRANZ Study report 192, BRANZ Ltd, Judgeford, New Zealand.

Mathur, V. K. (2002). "Base paper presented on new construction materials." CSIR Task Force Programme, Roorkee.

Mitrus, M., Wojtowicz, A. and Moscicki, L. (2009). “Biodegradable Polymers and their Practical Utility” *Thermoplastic starch* L. P. B. M. Janssen, and L. Moscicki, eds, Wiley-Vch Verlag GmbH & Co., Weinheim, 1-33.

Mohan, T. P. and Kanny, K. (2012). “Chemical treatment of sisal fibre using alkali and clay method.” *Compos. A.*, 43(11), 1989-1998.

Mohanty, A. K., Misra, M. and Drzal, L. T. (2001a). “Surface modifications of natural fibres and performance of the resulting bio composites: an overview.” *Compos. Interface,* 8(5), 313-43.

Mohanty, A. K., Misra, M. and Hinrichsen, G. (2001). “Bio fibres, bio degradable polymers and bio composites- An overview.” *Macromol. Mat. Eng.*, 276-277, 1-24.

Mohanty, A. K., Misra, M., Drzal, L.T., Selke, S. E., Harte, B. R. and Hinrichsen, G. (2005). “Natural Fibres, Biopolymers, and Biocomposites: An Introduction” *Natural Fibres, Biopolymers and Biocomposites*, A. K. Mohanty, M. Misra, L.T. Drzal, eds., CRC Press, Taylor & Francis Group; 1-36.

Mohee, R., Unmar, G. D., Mudhoo, A. and Khadoo, P. (2008).”Biodegradability of biodegradable/degradable plastic materials under aerobic and anerobic conditions.” *Waste Mgmt*. 28(9), 1624-1629.

Monteiro, S. N., Calado, V., Rodriguez, R. J. S., and Margem, F. M. (2012).”Thermo gravimetric behaviour of natural fibres reinforced polymer composites-An overview.” *Mater Sci. Eng A.*, 557, 17-28.

Morris J. (1996). “Recycling versus incineration: an energy conservation analysis.” *J. Hazardous Mater.* 47(1-3), 277-293.

Mukhopadhyay, S., Fangueiro, R., Arpac, Y. and Senturk, U. (2008). “Banana Fibres - Variability and Fracture Behaviour.” *J. Eng. Fibres Fabrics,* 3, 39-45.

Mumma, T. (2002). “Efficient Agricultural Buildings: An Overview.” National sustainable agriculture information service.

Mwaikambo, L.Y. and Ansell, M. P. (2002). “Chemical modification of hemp, sisal, jute and kapok fibres by alkalization.” *J. Appl. Polym Sci,* 84 (12), 2222-2234.

Nascimento, D.C.O., Lopes, F. P. D. and Monteiro, S. N. (2010). “Tensile behavior of lignocellulosic fibre reinforced polymer composites: Part I piassava/epoxy.” *Revista Materia*, 15(2), 199-205.

Nishino, T. (2004). “Green Composites: Polymer composites and the Environnent.” *Natural fibre sources,* eds C. Baillie, Wood head Publishing Cambridge, 49-80.

Oksman, K., Skrifvars, M. and Selin J. F. (2003). “Natural fibres as reinforcement in poly lactic acid (PLA) composites.” *Compos. Sci. Technol.*, 63: 1317-1324.

Okubo, K., Fujii,T., and Yamamoto,Y. (2004). “Development of bamboo-based polymer composites and their mechanical properties”, *Compos A*, 35, 377–383.

Pappu, A., Saxena, M. and Asolekar, S. (2007). “Solid wastes generation in India and their recycling potential in building materials.” *Bldg Envrn*, 42(6), 2311-2320.

Parnas, R.S. (2000), Liquid Composite Moulding, Hanser Gardner Publications, Munich. 170.

Patnaik, A., Abdulla, Md., Satapathy, A., Biswas, S., Satyapathy, B. K. (2010) "A study on a possible correlation between thermal conductivity and wear resistance of particulate filled polymer composites", *J. Mater. Design*, 31, 837-849.

Phuong, V. T. and Lazzeri, A. (2012). “Green bio composites based on cellulose diacetate and regenerated cellulose microfibres: Effect of plasticizer content on morphology and mechanical properties.” *Compos. A.*, 43(12), 2256-2268.

Pothan, L. A. and Thomas, S. (2004), “Effect of hybridization and chemical modification on the water-absorption behavior of banana fibre reinforced polyester composites.” *J. Appl. Polym. Sci.*, 91, 3856-3865.

Potter, K. (1997). Resin Transfer Moulding, Chapman and Hall, London. 1-70.

Prasad, B. M., Sain, M. M. and Roy, D. N. (2005). “Properties of ball milled thermally treated hemp fibres in an inert atmosphere for potential composite reinforcement,” *J. Mtrl.Sci,* 40 (16), 4271- 4278.

Puitel, A. C., Tofanica, B.M., Gavrilescu, D. and Petrea, P. V. (2011). “Environmentally Sound Vegetal Fibre Polymer Matrix Composites.” *Cellulose Chem. Technol.*, 45(3-4), 265-274.

Qin, C., Soykeabkaew, N., Xiuyuan, N. and Peijs, T. (2008). “The effect of fibre volume fraction and mercerization on the properties of all-cellulose composites.” *Carbo. Polym.* 71(3), 458-467.

Rachmadini, Y., Tan, V. B. C. and Tay, T.E. (2010). “Enhancement of Mechanical Properties of Composites through Incorporation of CNT in VARTM- A Review.” *J. Reinforced Plast. Compos.* 29(18) 2782-2807.

Ramesh, M., Palanikumar, K., and Reddy, K.H. (2013). “Mechanical property evaluation of sisal–jute–glass fibre reinforced polyester composites”.*Compos B,* 48, 1-9.

Rao, S., Jayaraman, K. and Bhattacharyya, D. (2012). “Micro and macro analysis of sisal composites hollow core sandwich panels,” *Compos B,* 43(7), 2738-2745.

Rapa, M., Popa, M. E., Cinelli, P., Lazzeri, A., Burnichi, R., Mitelut, A. and Grosu, E. (2011). “Biodegradable alternative to plastics for agriculture application” *Romanian Biotech. Letters*. 16(6), 59-64.

Ray, D., Sarkar, B. K, Rana, A. K. and Bose, N. R. (2001). “Effect of alkali treated jute fibres on composite properties.” *Bull. Mater. Sci.*, 24(2), 129-135.

Reddy, N and Yang, Y. (2005). “Biofibres from agricultural byproducts for industrial applications.” *Trends in Biotech*, 23(1), 22-27.

Reddy, N and Yang, Y. (2005a). “Structure and properties of high quality natural cellulose fibres from cornstalks.” *Polym.* 46(15), 5494-5500.

Reddy, N. and Yang, Y. (2004). “Structure of novel cellulosic fibres from cornhusks”. Polymer Preprints, ACS, 45 (2) 411.

Riedel, U. (1999). Natural fibre-reinforced biopolymers as construction materials - new discoveries, *2nd Int. Wood and Natural Fibre Composites Symp*, Germany.

Rout, J., Misra, M., Tripathy, S.S., Nayak, S. K. and Mohanty, A.K. (2001). “The influence of fibre treatment on the performance of coir-polyester composites.”*Compos. Sci. Tech*, 61 (9), 1303-1310.

Rowell, R. M. (1995). “A New Generation of Composite Materials from Agro-based Fibre, Polymers and other advanced materials: emerging technologies and business opportunities.” Proceedings of the 3d international conference on frontiers of polymers and advanced materials; 1995 January 16-20; Kuala Lumpur, Malaysia. New York, NY: Plenum Press: 659-665.

Rowell, R. M. (2002). “Sustainable composites from natural resources from High Performance Structures and Composites.” eds. Brebbia, C.A., Wilde, W.P.D., WIT Press, Southampton, Boston, 183-192.

Rowell, R. M., Sanadi A. R., Caulfield, D. F. and Jacobson, R. E. (1996). “Utilization of Natural Fibres in Plastic Composites: Problems and Opportunities”, *Proc. Int. Symp. on Lignocellulosic Plastic Composites*, Sao Paulo, 23-51.

Rowell, R.M. (1998). “Property enhanced natural fibre composite materials based on chemical modification”, *Science and Technology of Polymers and Advanced Materials Emerging Technologies and Business Opportunities*.” ed. P.N. Prasad., S.H. Kandhil and Z.H. Kafafi. Plenum Press.

Rudd, C. D., Long, A. C., Kendall, K. N. and Mangin, C. (1997). Liquid moulding technologies, Wood head Publishing Limited, U.K.

Salemane, M. G. and Luyt, A. S. (2006). “Thermal and mechanical properties of polypropylene wood powder composites.” *J. Appl. Polym. Sci*., 100(5), 4173-4180.

Salmoral, E. M., Gonzalez M. E. and Mariscal M. P. (2000). “Biodegradable plastic made from bean products” *Indl. Crops and Prd.* 11(2-3), 217–225.

Sarasa, J., Gracia, J. M. and Javierre, C. (2009). “Study of the biodisintegration of a bioplastic material waste” *Bioresour. Technol*. 100, 3764–3768.

Sarkanen, K.V and Hergert, H. L. (1971). “Classification and Distribution, In Lignins: Occurrence, Formation, Structure and Reactions.” K.V Sarkanen and C. H. Ludwig, eds., John Wiley & Sons, New York, 43–94.

Savastano, Jr, H., John, V. M., Agopyan, V. and Gatto, E. G. (2002). “Developing waste cellulose fibre reinforced composites with clinker free cement.” NOCMAT/3, *Int. Conf.on Non-Conventional Materials and Technologies*, Hanoi, Vietnam.

Savastano, Jr. H., Warden, P.G. and Coutts, R. S. P. (2005). “Microstructure and mechanical properties of waste fibre-cement composites.” *Cement Concrete Compos.* 27(5), 583-592.

Saxena, M. R. K., Morchhale, Asokan, P. and Prasad, B. K. (2008). “Plant Fibre Industrial Waste Reinforced Polymer Composites as a Potential Wood Substitute Material.” *J. Compos.Mater*., 42(4), 367-384.

Schemanauer, J. J., Osswald, T. A., Sanadi, A. R. and Caulfield, D. F. (2000). “Melt Rheolocial properties of Natural fibre-reinforced polypropylene.” *Proc. from ANTEC,* 2206-2210.

Sgriccia, N., Hawley, M. C. and Misra, M. (2008). “Characterization of natural fibre surfaces and natural fibre composites.” *Compos. A.*, 39(10), 1632-1637.

Shekeil, Y. A. E., Sapuan, S. M., Khalina, A., Zainudin, E. S. and Al-Shuja, O. M. (2012). “Influence of chemical treatment on the tensile properties of kenaf fibre reinforced thermoplastic polyurethane composite.” *Express Polym. Letters,* 6(12), 1032-1040.

Sherman, L. M. (1999). “Natural fibres: the new fashion in automotive plastics.” *Plast Technol*., 45(10), 62-68.

Silva, E. C. N., Walters, M. C. and Paulino, G. H. (2008). “Modelling Bamboo as a functional graded material” AIP Congress Proceedings, 973, 754-759.

Silva, L. J., Panzera, T. H., Christoforo, A. L., Durao, L. M. P. and Lahr, F. A. R. (2012), “Numerical and Experimental Analyses of Bio composites Reinforced with Natural Fibres”, *Intl. J. Mater. Eng.,* 2(4), 43-49.

Singha, A.S. and Rana, R. K. (2010). “Effect of pressure induced graft copolymerization on the physic chemical properties of biofibres” *BioRes* 5 (2), 1055-1073.

Sombatsompop, N. and Chaochanchaikul, K. (2004). “Effect of moisture content on mechanical properties, thermal and structural stability and extrudate texture of poly (vinyl chloride)/wood sawdust composites.” *Polym. Int*., 53(9), 1210-1218.

Somnuk, U., Ruksakulpiwat, Y., Suppakan, N. and Sutapan, W. (2004). “Characterization of chemical treated vetiver grass.” The 3rd *Thailand Material Science and Technology Conf.* Bangkok Thailand, 420-422.

Srebrenkoska, V., Gaceva, G. B. and Dimeski, D. (2009). “Preparation and recycling of polymer eco-composites- comparison of the conventional molding techniques for preparation of polymer eco-composites.” *Macedonian J. Chem Chemical Eng.*, 28(1), 99-109.

Sreekala, M. S., Kumaran, M. G. and Thomas, S. (2002). “Water sorption in oil palm fibre reinforced phenol formaldehyde composites.” *Compos. A.*, 33(6), 763-777.

Sreekala, M.S. and Thomas, S. (2003). “Effect of fibre surface modification on water-sorption characteristics of oil palm fibres.” *Compos. Sci. Technol.*, 63(6), 861-869.

Stark, N. (2001). “Influence of moisture absorption on mechanical properties of wood flour-polypropylene composites.” *J. Thermoplastic Compos. Mater.* 14(3), 421-32.

Stevens, M. P. (1999). Polymer Chemistry: An Introduction Oxford University Press.1-60.

Stulz, R. and Mukerji, K. (1993). “Appropriate Building Materials: A Catalogue of Potential Solutions.” Third revised edition, SKAT Publications, Switzerland.

Sulaiman, F. A. A., Mokheimer, E. M. A., and Nassar, Y. N. A. (2006). “Prediction of the thermal conductivity of the constituents of fibre reinforced composite laminates”, *Heat Mass Tfr*, 42, 370–377.

Sun, J. X., Xu, F., Sun, X.F., Xiao, B. and Sun, R.C. (2005). “Physico-chemical and thermal characterization of cellulose from barley straw.” *Polym. Degrad. Stabil.* 88(3), 521-531.

Sun, X. F., Fowler, P., Rajaratnam, M., and Zhang, G. (2010). “Extraction and Characterisation of Hemicelluloses from Maize Stem”, *Phytochem. Anal.*, 21, 406-415.

Swamy, R. P., Kumar, G. C. M. and Joseph, V. (2004). “Study of areca reinforced phenol formaldehyde composites.” *J. Reinforced Plastics Compos*, 23 (13), 1373-1382.

Swift, D. G. (1985). “Sisal-cement composites and their potential for rural Africa.” *Compos. Struct.* I. H. Marshall eds., Elsevier science publishers, 1985, 774-787.

Sydenstricker, T. H., Mochnaz, S. and Amico, S. C. (2003) “Pull-out and other evaluations in sisal-reinforced polyester bio composites.” *Polym. Test.*, 22(4), 375-380.

Taj, S., Munawar, M. A. and Khan, S. (2007). “Natural fibre- reinforced polymer composites: Review.” *Proc. Pakistan Acad. Sci*.; 44(2), 129-144.

Tita, V., Carvalho, J. D. and Lirani, J. (2003). “Theoretical and Experimental Dynamic Analysis of Fibre Reinforced Composite Beams.” *J. Braz. Soc. Mech. Sci. & Eng.*, 25(3), 306-310.

Tokiwa, Y., Calabia, B. P., Ugwu, C. U. and Aiba, S. (2009). “Biodegradability of plastics.” *Int. J. Mol. Sci.*, 10(9), 3722-3742.

Torres, F.G., and Cubillas, M. L. (2005) “Study of the interfacial properties of natural fibre reinforced polyethylene” *Poly. Testing* 24, 694–698.

Trigui, A., Karkri, M., Perria, L., Boudaya, C., Candau,Y., Bouffi, S., and Vilaseca, F. (2012). “Thermal and mechanical properties of maize fibres–high density polyethylene biocomposites”. *J. Compos. Mater.* 47(11), 1387-1397.

Tsai, J. L. and Chi, Y. K. (2008), "Effect of fibre array on damping behaviors of fibre composites." *Compos. B.,* 39 (7-8), 1196-1204.

Tylor, A. (2002). "Case study on fibres in composite materials, eg. Hemp in automotive applications." 4th meeting of Government- Industry forum on non-food uses of crops, GIFNFC 4/4 fibres in composite materials, DTI, London.

Uzun, B. B., and Sarioglu, N. (2009). "Rapid and catalytic pyrolysis of corn stalks", *Fuel Processing Tech.* 90, 705-716.

Valchev, I., Nenkova, S., Tsekova, P. and Lasheva, V., (2009). "Uses of Enzymes in hydrolysis of maize stalks", *Biores.* 4(1), 285-291.

Velde, K. V. D. and Kiekens, P. (2001), "Thermoplastic polymers: overview of several properties and their consequences in flax fibre reinforced composites." *Polym. Test.*, 20(7), 885-893.

Velde, K. V. D. and Kiekens, P. (2002). "Biopolymers: overview of several properties and consequences on their applications." *Polym. Test.,* 21(4), 433-442.

Vroman, I. and Tighzert, L. (2009). "Biodegradable Polymers" *Materials.,* 2(2), 307-344.

Wallenberger, F. T. and Weston, N. (2003), Natural fibres, Plastics and Composites, Kluwer Academic Publishers. 3-40.

Wang, W., Sain, M. and Cooper, P. A. (2006). "Study of moisture absorption in natural fibre plastic composites." *Compos. Sci. Technol.*, 66(3-4), 379-386.

Wool, R. and Sun, X. S. (2005). *Bio based polymers and composites,* (Elsevier). 202-255, 292-.396.

www.greencarcongress.com/2008/02/mercedes-to-int.html

Xiao, B., Sun, X.F. and Sun, R. C. (2001). “Chemical, structural, and thermal characterizations of alkali-soluble lignins and hemicelluloses, and cellulose from maize stems, rye straw, and rice straw.” *Polym. Degrad. Stabil.* 74(2), 307-319.

Xie, Y., Hill, C. A. S., Xiao, Z., Militz, H. and Mai, C. (2010), “Silane coupling agents used for natural fibre/polymer composites: A review.” *Compos. A.,* 41(7), 806-819.

Yao, F., Wu, Q., Lei, Y., Guo, W., and Xu, Y. (2008). “Thermal decomposition kinetics of natural fibres: Activation energy with dynamic thermogravimetric analysis” *Polymer Degrad and Stab,* 93, 90-98.

Yoon, M. K., Baidoo, J., Gillespie Jr, J.W. and Heider, D. (2005). “Vacuum Assisted Resin Transfer Molding (VARTM) Process Incorporating Gravitational Effects: A Closed form Solution.” *J. Compos. Mater.* 39(4), 2227-2242.

Young, R. A. (1986) “Cellulose: Structure, modification and hydrolysis.” R. A. Young and R. M. Rowell, eds. John Wiley & Sons, New York.

Yu, L. (2008). “Biodegradable Polymer Blends and Composites from Renewable Resources.” John Wiley & Sons, Inc., USA.1-15.

Yu, L., Dean, K. and Li, L. (2006). “Polymer blends and composites from renewable resources.” *Prog. Polym. Sci.* 31, 576–602.

Zabaniotou, A., and Ioannidou, O. (2008). “Evaluation of utilization of corn stalks for energy and carbon material production by using rapid pyrolysis at high temperature”, *Fuel,* 87, 834–843.

Zeu, M., Yu, B and Zang, D. (2002), “Thermal conductivities of unidirectional fibre composites with thermal barrier”, *J. Physics*, 35, 1867-1874.

Zvinowanda, C. M., Okonkwo, J. O., Agyei, N.M., and Shabalala, P.N. (2008). “Physicochemical Characterization of Maize Tassel as an Adsorbent. I. Surface Texture, Microstructure, and Thermal Stability”. *J.Appl. Polymer Sci.*, 111, 1923-1930.

PUBLICATIONS

INTERNATIONAL JOURNALS

[1] Saravana Bavan D and Mohan Kumar G. C. (2010). “Potential Use of Natural Fibre Composite Materials in India”. *Journal of Reinforced Plastics and Composites*, 29 (24), 3600-3613.

[2] Saravana Bavan D and Mohan Kumar G. C. (2011). “Examination of Tensile Strength and FTIR Features of Maize Fibres Reinforced Polymer Composites”, *International Journal of Recent Trends in Engineering & Technology*, 5(4), 27-30.

[3] Saravana Bavan D and Mohan Kumar G. C. (2012). “Morphological and Thermal Properties of Maize Fibre Composites”, *Fibres and Polymers*, 13(7), 887-893.

[4] Saravana Bavan D and Mohan Kumar G. C. (2013). “Finite Element Analysis of a Natural Fibre (Maize) Composite beam” *Journal of Engineering*, Hindawi Publications, Article ID 450381, 7 pages.

BOOK CHAPTERS

[1] Saravana Bavan D and Mohan Kumar G. C. (2012) “Maize Natural Fibre As Reinforcement With Polymers For Structural Applications”, in *Natural Polymers, Biopolymers, Biomaterials, Blends, and IPNs*, Eds. Sabu Thomas, Neethu Ninan, Sneha Mohan, Elizabeth Francis, Apple Academic Press, Canada, CRC Press, Taylor and Francis Group, (ISBN: 978–1–926895–16–1).

[2] Saravana Bavan D and Mohan Kumar G. C. (2013). “ Re-Use of Natural Plant Fibres for Composite Industrial Applications”, in *Recycling and Reuse of Materials and their Products*, Eds. Yves Grohens, S. Kishor Kumar, Abderrahim

Boudenne, Apple Academic Press, Canada, CRC Press, Taylor and Francis Group, (ISBN: 978-1-926895-27-7).

INTERNATIONAL CONFERENCE PROCEEDINGS

[1] Saravana Bavan D and Mohan Kumar G. C. (2011). "*Conceptual design features and eco-methods for the extraction of natural fibres in the materialistic earth",* in Research in to Design-Supporting Sustainable Product Development, Researching Publishing, Singapore, p 464-471, ISBN: 978-981-08-7721-7.

[2] Saravana Bavan D and Mohan Kumar G. C. (2012). *A detailed study on hybrid plant fibres for packaging applications,* 18th IAPRI World Packaging Conference, DEStech Publications, Inc, Pennsylvania, USA, p 250-257, ISBN: 978-1-60595-084-6.

[3] Saravana Bavan D and Mohan Kumar G. C. (2013). *Significant Aspects on Thermal Degradation of Hybrid Bio composite Material,* RAM 2013, American Institute of Physics Proceedings, p 1318-1319, ISBN: 978-0-7354-1160-9.

[4] Saravana Bavan D and Mohan Kumar G. C. (2013) "*Thermal Properties of Maize Fibre Reinforced Unsaturated Polyester Resin Composites*", Proceedings of the World Congress on Engineering (WCE) 2013, Vol III, WCE 2013, London, U.K. p 2091-296, ISBN: 978-988-19252-9-9.

NATIONAL CONFERENCES

[1] Saravana Bavan D and Mohan Kumar G. C. (2009). *Bio-Composites: An Over View and Recent Trends in Applications,* National Conference on Recent Innovations in Design & Analysis (RIDA-2009), Government College of Engineering, Tirunelveli, Tamil Nadu, India, p 94-99.

[2] Saravana Bavan D and Mohan Kumar G. C. (2010). *Characterization and Effect of polymers in Natural fibre composites,* Third National Symposium for Materials Research Scholars (MR – 10), Indian Institute of Technology (IIT) Bombay, Mumbai, India.

INTERNATIONAL CONFERENCES (IN INDIA)

[1] Saravana Bavan D and Mohan Kumar G. C. (2009). *Modern Aspect of Natural Fibre Composites in the Competitive Field of Structures*, International Conference on Advances in Mechanical and Building Sciences in the 3rd Millennium- (ICAMB-2009), VIT University, Vellore, Tamil Nadu, India, p 1638- 1645.

[2] Saravana Bavan D and Mohan Kumar G. C. (2010). *Prospects of Bio-Polymer in Natural Fibre Composites,* Second International Conference on Polymer Processing and Characterization (ICPPC-2010), Mahatma Gandhi University-Kottayam, Kerala, India.

[3] Saravana Bavan D and Mohan Kumar G. C. (2010). *Characterization of Polymers and their Behavior Study in Achieving Environmentally-Friendly Natural Fibre Composite Material,* Frontiers of Polymers & Advanced Materials (MACRO-2010), Indian Institute of Technology (IIT)- Delhi, India.

[4] Saravana Bavan D, Siad, Kamal Babu and Mohan Kumar G. C. (2011). *Modeling and Finite Element Analysis of Green Composites for Structural Applications,* Second International Conference on Advancements in Polymeric Materials (APM-2011)- Central Institute of Plastics Engineering and Technology (CIPET), Chennai, ISBN: 978-81-309-1790-0.

[5] Saravana Bavan D and Mohan Kumar G. C. (2012). *Morphology study deformation at the interfaces of a maize hybrid fibre biocomposite material*, International Conference on Strength of Materials (ICSMA-2012), Indian Institute of Science (IISc) Bangalore, India.

[6] Saravana Bavan D and Mohan Kumar G. C. (2012). *A Comparison Study of a Maize-Jute Sandwiched Composite Material*, Third International Conference on Natural Polymers and Biomaterials (ICNP-2012), Mahatma Gandhi University, Kottayam, Kerala, India.

[7] Saravana Bavan D and Mohan Kumar G. C. (2013). *An Experimental and Characterization Work on Bio-Composite Material for Structural Applications,* Fourth International Conference on Recent Advances in Composite Materials (ICRACM-2013), Goa, India.

INTERNATIONAL CONFERENCES (ABROAD)

[1] Saravana Bavan D and Mohan Kumar G. C. (2010). *Reinforcement of Maize Fibres in Processing of Bio-Composite Materials by VARTM,* Polymers and the Environment: Emerging Green Technologies and Science, Sheraton Centre Toronto Hotel, Toronto, Canada.

[2] Saravana Bavan D and Mohan Kumar G. C. (2011). *Structural Analysis of a Hybrid Natural Fibre Composite Material Processed Through Vacuum Assisted Resin Transfer Molding,* 19th Bio Environmental Polymer Society (BEPS)-Annual Meeting, Vienna University of Technology, Vienna, Austria.

[3] Saravana Bavan D and Mohan Kumar G. C. (2012). *Processing and data acquisition analysis of a biocomposite material of vetiver and maize fibre* International Symposium on Advanced Polymeric Materials 2012 (ISAPM 2012) under the auspices of IMTCE 2012, Kuala Lumpur, Malaysia.

RESEARCH ACHIEVEMENTS AND AWARDS:

[1] The Paper titled "*A Novel Decorticator Design for Natural Plant Fibres*", achieved **Best Paper and Cash Award** in National Conference on Design and

Manufacturing (NaConDM), at Indian Institute of Information Technology Design and Manufacturing (IIITD&M) Kancheepuram, IIT Madras campus, 27-28 May, 2011.

[2] The Paper titled “A Study on Natural Straw Fibres as a Nano Composite Material and their Applications” was Selected and **Sponsored** by **International Advanced Research Centre for Powder Metallurgy & New Materials (ARCI)** to present the work under the Category of **Young Researchers** at International Conference on Nano Science and Technology (ICONSAT-2012), Hyderabad, January 20-23, 2012.

[3] Participated and Presented Paper “*Characterization Studies on Rheology of Soy based Biopolymer*” at the Sixth National Symposium of Rheology of Complex Fluids and **Sponsored by Indian Institute of Technology** (IIT) Guwahati, January 6-7, 2012.

[4] Paper titled “*Thermal Properties of Maize Fibre Reinforced Unsaturated Polyester Resin Composites*” was **Recommended and Short listed** for Best Paper Award in open competition at World Congress on Engineering (WCE) - 2013, London, U.K.

OTHER PUBLICATIONS

[1] Saravana Bavan D, Kamalbabu P and G. C. Mohan Kumar. *A Novel Decorticator Design for Natural Plant Fibres,* International Journal of Applied Engineering Research, 6(5), 2011, pg.561-567.

[2] Saravana Bavan D and G. C. Mohan Kumar. *Use of Natural Fibres and other Composites in Waste Water Treatment - A Review*, Book Chapter for Nanocomposite for waste water treatment, Ed. Ajay Kumar Mishra, Pan Stanford Publishing, Singapore. (**Accepted and** i**n Process**)

BIO-DATA

SARAVANA BAVAN. D
Department of Mechanical Engineering
National Institute of Technology Karnataka
Srinivasnagar, Surathkal-575025
Mangalore, Karnataka
India
E-mail: saranbav@gmail.com

Research Interests:

Composites, Finite Element Analysis, Polymers, Characterisation Techniques

Education:

ME	**Master of Engineering** Anna University, Chennai- Tamil Nadu, India CAD/CAM – First class with Distinction. Thesis Work: Experimental Investigation and Finite element simulation of ductile fracture Prediction of Powder Metallurgy composites
BE	**Bachelor of Engineering** Visvesvaraya Technological University- Belgaum, Karnataka, India Mechanical Engineering- First Class. Project Work: Fabrication fixture for testing hoists cylinders of BH-85 Dumper
SSLC	**Secondary School Leaving Certificate** Karnataka Secondary Education Examination Board, India First Class with Distinction.

Work Experience:

Industrial Experience-1 year, Teaching Experience-2 year