Experimental Study of Mechanical and Thermal Properties of Bhimal Fiber Reinforced Epoxy Bio-Composite

Thesis

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By

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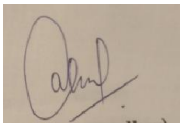
Pantnagar *(Bikraj Upreti)*

July, 2017 *Author*

CERTIFICATE

This is to certify that the thesis entitled "**Experimental Study of Mechanical and Thermal Properties of Bhimal Fiber Reinforced Epoxy Bio-Composite**" submitted in partial fulfilment of the requirements for the degree of**Master of Technology** with major in **Manufacturing Engineering and Management** of the College of Post-Graduate Studies, G. B. Pant University of Agriculture and Technology, Pantnagar, is a record of *bonafide* research carried out by **Mr.Bikraj Upreti,** Id. No.**49606**under my supervision and no part of the thesis has beensubmitted for any other degree or diploma.

The assistance and help received during the course of this investigation and source of literature have been duly acknowledged.



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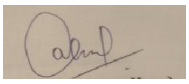
We, the undersigned, members of the Advisory Committee of **Ms. Bikraj Upreti,** Id. No. **49606**, a candidate for the degree of **Master of Technology** with major in **Manufacturing Engineering and Management**, agree that the thesis entitled **"Experimental Study of Mechanical and Thermal Properties of Bhimal Fiber Reinforced Epoxy Bio-Composite"** may be submitted in partial fulfilment of the requirements for the degree.



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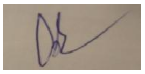

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SYMBOLS

ε	:	Strain
ε_f	:	Flexural Strain
σ	:	Stress
σ_f	:	Flexural stress
σ_u	:	Ultimate tensile strength
ρ	:	Density of material
ρ_f	:	Density of fiber

ABBREVIATIONS

a	:	Depth of notch in Impact test specimen
b	:	Width of test beam
d	:	Depth of test beam
g	:	grams
m	:	Mass of material
s	:	Gradient of initial straight line of load deflection curve
t	:	Thickness of Impact test specimen
w	:	Depth of Impact test specimen
D	:	Maximum deflection of the center of beam
DTA	:	Differential Thermal Analysis
DTG	:	Derivative Thermo Gravimetric Analysis

E	:	Modulus of elasticity
Ef	:	Flexural modulus
HRL	:	Rockwell hardness on L-scale
IS	:	Impact strength
L	:	Support span in bending test
P	:	Load on a given point on the load deflection curve
SEM	:	Scanning Electron Microscopy
TGA	:	Thermo Gravimetric Analysis
V	:	Volume of the material
Wm	:	Weight fraction of matrix
WA	:	Water Absorption

Chapter 1 INTRODUCTION

1.1 Introduction

In this modern world, there is a demand of highly advanced materials with notable blend of properties that are not fulfilled by the inevitable polymers, alloys and ceramics. This is very crucial for the materials that are required in aerospace, underwater and transportation purposes. That is why the Composite is getting used as a broader term in order to overcome these problems.

Composite is any poly-phase material that comprises more than one chemically different elements those are having a distinct boundaries separating them **(Callister, 2015).** Composites are mainly formed of more than one irregular phases which are engrafted in an unremitting phase. The irregular phase is frequently stronger and harder than the unremitting phase and is termed as the reinforcement, whereas the unremitting phase as the matrix. The matrix can be polymeric, metallic or ceramic. The final composite is called as the Polymer Matrix Composite (PMC) if the matrix formed is a polymer. The reinforcement phase can be a particle or fiber and the fiber is known as the natural or biodegradable fibers only if the fibers are a ensuing from living species. Usually, fibers are used to carry more load than matrices; while the main purpose of matrix is to orient the fibers at their desired direction and location and also to protect them from any natural destruction.

In last few years, the natural fibers have been catching the focus of researchers as a reinforcing material for many PMCs from both the ecological and economical point of view. The main source of obtaining the biological fibers is from plants, animals and minerals. The natural fibers have perked up the fuel efficiency and reduced emission in automobile applications due to its greater volume to weight ratio than the synthetic ones. **(Wambua *et al.*, 2003** and **Sahieb and Jog,1999).**

A careful choice of the reinforcing fiber and matrix can help in achieving a preferred composite with a combination of high modulus and strength comparable or even better than the conventional ones **(Jang, 1994)**. The biological fibers like bhimal, animal hair, bird's feather, hemp, coir etc are economic, renewable and can get cremated for energy recovery. They acquire a great calorific value and needs a little attention in terms of

safety and health during handling. This superior environmental friendly attribute makes the resultant materials very admired in the engineering marketplace. This very reason provoked us to explore the thermal and mechanical properties of bhimal fiber reinforced with epoxy resin.

Thermosetting resins like the epoxies, polyurethane, polyester, phenolic, etc are in widespread use together with the biological fiber to make the composites with higher performance applications. They endow with high mechanical properties, in particular strength and stiffness, at suitably low prices. In comparison with the thermoplastic polymers, thermoset polymers are having a better thermal stability and low water absorption quality. But in order to make better recycling thermoplastic polymers are preferred over thermosetting polymers. The term epoxy resin explains a set of thermosetting resins organized by the ring-opening polymerization of compounds comprising more than one epoxy group in one molecule by average **(Irfan, 1998).** Epoxy resins conventionally are prepared by reacting the epichlorohydrin with bisphenol A, which are linear polymers that tends to cross-link, forming a range of thermosetting resins in essence of reaction with the hardeners. For curing amine epoxy resin family plays a major role and thus, no volatile by-products are created during the process. The benefits of epoxy resins have excellent chemical resistance, low polymerization shrinkages, remarkable mechanical strength and good electrical properties. Since they are capable to soak up both the thermal and mechanical stresses better than the linear groups and therefore give the epoxy resin immaculate stiffness, thermal resistance and impact strength. The only few demerits of the epoxy resins are large curing times and their mould release characteristics are also poor.

Biological fibers are classified according to origins, extracted from plants, minerals or animals. All fibers extracted from plants are made of cellulose whereas animal fibers are mostly made of proteins (hair, silk, and bhimal).Fibres those are taken from plants are mostly bast fibres, hard fibres or leaf, fruit, seeds, cereal straw, wood and other grass fibres. In most recent couple of years, such a large number of specialists have been investigating the use of bio-fibers as load carrying component in composite. The utilization of bio-fibers in composites has got more popularity because of their relative low cost, their capability of recycling and due to the fact that they give a tough competition in terms of strength per unit weight of composite. Bio-fibres can be assumed to be naturally occurring

composites consisting mainly of cellulose fibrils embedded in lignin matrix. The cellulose fibrils are aligned along the length of the fibre, which provide good flexural and tensile strengths, in addition to provide stiffness. The reinforcing efficiency of bio-fibre is associated with the character of cellulose and its crystallinity. The main elements of natural fibres are cellulose (a-cellulose), hemi-cellulose, lignin, pectins, and waxes. Natural fibers have been using for a long period of time in engineering field. For example, jute fiber is a mostly used reinforcement in Indian subcontinent. The key market for biological fibers in the year 2000 was in building applications whereas other applications have been growing at an exponential rate, particularly in the automobile industry **(John and Thomas, 2008)**. For illustration, from 1989 till 2004, the use of biological fibers in the composites field in the German automobile commerce increased from 5,000 tons to 19,000 tons per year. The favorable reasons for the relevance of biological fibers in the automobile industry are discussed below **(Joshi *et al*., 2004).**

- Options for latest production technologies and materials.
- Favorable stability, less splintering and accident performance.
- Favorable eco-balance for part fabrication.
- Low density is useful in reducing weight by 10 to 30%.
- Remarkable mechanical properties and good acoustic properties.
- Occupational health profits in comparison of glass fibers during production.
- No off-gassing of contaminated compounds (in contrast to phenol resin bonded wood and recycled cotton fiber parts).
- Favorable ecological balance during vehicle function because of the weight savings.
- Price advantages both for the fibers and the applied technologies.
- Favorable properties in processing, for instance low wear on tools, etc.

Besides the advantages discussed above, the biological fiber composites possess a few disadvantages also. The foremost disadvantage is the deprived compatibility between a hydrophilic fiber and a hydrophobic polymer matrix. This tends to the growth of pathetic interfaces, which results in bad mechanical properties of the formed composites. Other demerits of the natural fiber based composites are their high sensitivity towards water and

also their relatively poor thermal stability. In natural composites water absorption properties is the crucial point to be worked on due to the swelling and dimensional instability due to water absorbed by the natural fibers. Also the mechanical properties are reduced due to the deprivation of fibers and interface between the matrix and reinforcement. **(Bledzki and Gassan, 1999** and **Stamboulis *et al.*, 2001)**. Hence, to recover the adhesion between fiber and matrix, a third factor, known as the compatibiliser, has to be incorporated for the matrix modification on the surfaces of fibers have to be modified to the preparation of the desired composites.

(Mohanty *et al.*, 2001). Several case studies have shown a great impact on a range of chemical alterations on the performance of fiber based composites. A range of surface chemical modifications of biological fibers like as, permanganate treatment, acetylation, isocyanate treatment, alkali treatment, silane treatment, monomer grafting under UV radiation, latex coating etc. have achieved various levels of improvements in fibers mechanical strength and fiber-matrix bond in natural fiber reinforced composites. For the composites that have thermoset matrixes such as epoxy, phenol formaldehyde, polyester, etc. alkali action on biological fibers is mostly used treatment as it improves the surface bonding between matrix and reinforced fiber that is used in improving the properties of composites to a higher extent **(Ray *et al.*, 2013)**. Free energy of fiber surface is increased by doing its alkali treatments. In addition, the alkali treatment can make the surface of fiber clean by eliminating the waxes and dirt. The removal of these substances enhances the surface roughness. Hence, the physical interlocking at the interface could be enhanced.

Bearing in mind the environmental aspects of material range, restoring the artificial fibers by the biological ones is only a first step. Confining the discharge of green house effect causing gases such as CO_2 into the environment and an increasing consciousness of the finiteness of fossil energy resources are leading to development of new materials that are utterly based on renewable resources. The biological fiber composite can be cost effective for the following applications such as window, ceiling, wall, floor and door frames, false ceiling, roof tiles, partition boards, construction industry or pre-fabricated buildings, mobile building which are being used at the time of natural calamities such as cyclones, earthquakes, floods, etc.

1.2 Advantages of Bio-Composite Materials

- Low specific weight, resulting in a higher specific strength and stiffness than glass fiber.
- It is a renewable source, the production requires little energy, and CO_2 is used while oxygen is given back to the environment.
- Producible with low investment at low cost, which makes the material an interesting product for low wage countries.
- Thermal recycling is possible while glass causes problem in combustion furnaces.
- Good thermal and acoustic insulating properties.
- Low density.
- Favorable eco-balance during vehicle operation due to weight savings.
- No off-gassing of toxic compounds (in contrast to phenol resin bonded wood and recycled Cotton fibber parts).
- Price advantages both for the fibres and the applied technologies

1.3 Disadvantages of Bio-Composite Materials

- Lower strength properties (especially impact strength).
- Fluctuation in price depending upon the global demand and production.
- Processing of fibre is labor intensive and time consuming.
- High moisture absorption causing swelling of fibres which brings about dimensional changes in composite materials.

1.4 Statement of the Problem

From the available literature on the subject, it is seen that bhimal fibers are quite a newer kind of bio-degradable fibres. Characterization of this fibre is essential to determine its properties for further use as reinforcing fibre in polymeric, bio-degradable and other kinds of matrix. Literature lacks in the utilization of bhimal fibers with the different types of polymers and due to the abundance of bhimal trees in Himalayan region, it is necessary to use this fiber as commercial material as it would be very beneficial to the firms and the

people who grow these trees in Himalayan region. Hence the further understanding of mechanical and thermal behavior of bhimal fiber based bio-composites is quite essential.

1.5 Objectives of the Present Research Work

The prime idea of this research work is to fabricate a composite that is the combination of a natural fiber (bhimal fiber) and matrix (epoxy resin) having properties good enough to work as an engineering material in comparison with the composites based on synthetic fibers which are expensive, dangerous or depleting materials. Bio composites are more environment friendly, biodegradable, economical, user friendly, lower density and have higher strength to weight ratio as compared to synthetic fiber based composites.

Hence the key objectives are to:

- Fabrication of the composite samples using various ratios of epoxy resin/bhimal fiber i.e. 100/0, 98/2, 97/3, 96/4, 95/5, and 96/10 weight percentage wise.
- To analyze the dispersion, bonding and morphology f matrix and reinforcement by the Scanning Electron Microscopy (SEM) analysis.
- To estimate the mechanical properties of resultant composite material like tensile strength, compressive strength, flexural rigidity, and hardness and impact strength.
- To analyze the thermal behaviour of composite material using the Thermal Gravimetric Analysis (TGA), Differential Thermal Analysis (DTA).
- Reasons for using the bhimal as reinforcing fiber in this investigation are as follows:
- High tensile strength and low density of bhimal fiber is the main reason behind selecting bhimal fibre is it as a reinforcing agent.
- Bhimal fiber is obtained from natural and renewable resources.
- Bhimal fibers are lightweight and environmentally superior alternative to the synthetic fibers.
- Bhimal fibers are biodegradable in nature i.e. when they are dumped; the decomposition is done by the action of micro-organisms.
- Bhimal is easily available in Himalayan region and it is cost effective.

1.6 Approach

The main approach of the research work is to fabricate a bhimal fiber reinforced epoxy composite. Scanning Electron Microscopy (SEM) technique is used to show the microstructure of the resultant composite which shows the distribution of bhimal fiber in epoxy resin matrix and their interaction with each other. In order to check the weight loss of the sample during heating is Thermal Gravimetric Analysis (TGA) and Differential Thermal Analysis DTA is performed to check the physiochemical changes of resultant composite during heating. Along with all these tests some of the mechanical tests such as tensile test, compressive test, hardness test, impact test and flexural test are performed in order to estimate different mechanical strengths

1.7 Organization of Thesis

The present work is divided into five chapters. The topics that are covered in various chapters are as follows:

Chapter 1 has a brief description on introduction to composites, bio-composites material used in fabrication, classification of the materials, selection of materials carefully, problem statement, objective of the research and approach.

Chapter 2 discusses the basic details of the research areas and also provides an insight into the earlier work carried out by various researchers in the field of bio-composites. Most of the literature is based on the study of mechanical and thermal studies of the natural fiber based composites.

Chapter 3 discusses the brief knowledge of processes those are to be carried and the experimental setup for the research work that includes casting moulds, electronic weighing machine, convection oven, grinders, UTM machine, Rockwell hardness testing machine, charpy impact test machine etc. selection of material. In this, selection of material and flow of those materials throughout the experiments is shown with neat diagrams and pictures.

Chapter 4 contains results and discussions. In this all the obtained results are shown in the form of graphs and tables and proper reasons for all results are discussed.

Chapter 5 has the summary of the results along with the scope of thesis for further investigations. References are incorporated at the end of the thesis to support the investigations.

Chapter 2 REVIEW OF LITERATURE

Composites have been a field of immense interest in the last two decades for a number of researchers, who have ingeniously combined various materials to convert them into extraordinary materials. Therefore, it becomes vital to present the major studies related to the polymer composites and the natural fibers. A detailed literature survey has been carried out before the fabrication and characterization of Bhimal-Reinforced-Epoxy bio-composite. Review has been mainly made for Natural fibers based bio-composites and Biodegradable based bio-composites which have been categorized below and presented in subsequent sections.

2.1 Review of Epoxy Resin

Epoxy resin is a thermoset. Thermo-set or network matrices are formed as a result of an irreversible chemical transformation of the resin into an amorphous cross-linked polymer matrix. As a result, they become everlasting hard and do not soften upon heating. Their key properties include good electrical thermal insulation, low viscosity, exceptional thermal stability and enhanced creep resistance. Following is the literature of epoxy resin.

Paramasivam and Abdul Kalam (1974) studied tensile test on dog-bone specimens made of sisal fibers with epoxy resin gave encouraging results, suggesting a greater scope for developing these natural fibers for structural and other commercial purpose. The tensile strength of sisal-epoxy composite varied from 2500kg/cm^2 to 3000kg/cm^2, nearly half the strength of fiber-glass-epoxy composite. Since the density of the sisal-epoxy composite was half that of glass-epoxy composite, the specific strength of sisal composite was nearly the same as that of glass composite.

Leman *et al.* (2008) fabricated the epoxy resin based green composite reinforced with sugar palm fiber and 10% and 20% of fiber were taken. Their objectives behind this research work is check of Fickian diffusivity constant, correction factor and moisture equilibrium content and correction factor of fiber and they found maximum moisture absorption rate at 20% fiber compositions.

Kumar *et al.* (2014) fabricated composites of epoxy reinforced with sundi wood dust and 7 different wt% filler was used and tensile and flexural test were performed at 3 different speeds to check mechanical behaviour. They found improvement in mechanical properties upto certain wt% of filler than decrement observed. Scanning electron microscopy test was performed to examine the change.

Fiore *et al.* (2015) fabricated the epoxy based composite reinforced with kenaf fibers. They made composite first with NaOH treatment and other without NaOH treatment and examined their mechanical properties. Treated composite showed the better mechanical properties than neat. Results of dynamic mechanical analysis showed that above glass transition temperature storage modulus and loss modulus are influenced by alkali treatment.

Prakash and Christu (2016) fabricated the epoxy resin reinforced with human hair. These fiber reinforced composites were successfully prepared by simple and cost effective synthesis methods by hand lay process. To test its suitability, mechanical properties such as tensile strength, compressive strength and hardness were subjected to different composition of the fiber and resin.

Haldar *et al.* (2017) fabricated two separate composites made up of hand layup technique and compression molding process. They fabricated composites reinforced with sisal fiber, one with aluminium powder and other without aluminium powder. They evaluated density test and micro hardness test, impact and tensile test. They found that without addition of aluminium powder, sisal reinforced epoxy composite showed lower density but micro hardness was excellent whereas tensile, impact and elastic modulus of sisal reinforced epoxy composite with aluminium powder was better than without aluminium based sisal-epoxy composites.

2.2 Reviews on Natural Fibers

Biological fibers are classified according to origins, extracted from plants, minerals or animals. All fibers extracted from plants are made of cellulose whereas animal fibers are mostly made of proteins (hair, silk, and bhimal).Fibres those are taken from plants are mostly bast fibres, hard fibres or leaf, fruit, seeds, cereal straw, wood and other grass fibres. In most recent couple of years, such a large number of specialists have been investigating the use of

bio-fibers as load carrying component in composite. Following are some bio-fibers whose literature review has been covered in this chapter.

1. Review on Bhimal (*Grewia Optiva*) fiber based bio-composite
2. Review on Banana Fiber based bio-composites
3. Review on Bamboo Fiber based bio-composites
4. Review on Coir Fiber based bio-composites
5. Review on Flax Fiber based bio-composites
6. Review on Hemp Fiber based bio-composites
7. Review on Jute Fiber based bio-composites
8. Review on Kenaf Fiber based bio-composites
9. Review on Ramie Fiber based bio-composites
10. Review on Sisal Fiber based bio-composites
11. Review on Sugarcane Fiber based bio-composites

2.2.1 Review on bhimal (*Grewia Optiva*) fiber based bio-composite

Bhimal fiber is least known fiber in bio-composite industry due to this very few literatures has been given. This fiber used as cordages in villages as the tensile strength of bhimal fiber is very high. Following is the literature about bhimal fiber based bio-composites:

Singha *et al.* (2008) assessed the properties of compression molded *Grewia Optiva* (Bhimal) fiber reinforced Urea-Formaldehyde (UF) matrix based polymer composites. Reinforcing of the UF resin with bhimal fiber was carried out in the forms such as particle, short and long fiber reinforcement. This work discloses that mechanical properties such as: tensile strength, compressive strength and wear resistance of urea-formaldehyde resin increases to a significant extent when reinforced with bhimal fiber. Analysis of results shows that particle reinforcement is more effective as compared to short and long fiber reinforcement. Thermal and Morphological studies of the matrix and fiber reinforced bio-composites have also been carried out.

Singha *et al.* (2012) have studied effect of surface modification of *Grewia optiva* fibers on their physicochemical and thermal properties. Raw, graft copolymerized and benzoylated bhimal fibers were brought to the assessment of some of their properties like

moisture absorbance, swelling behavior and chemical resistance behavior. It is seen that 5% benzoyl chloride treated and graft copolymerized bhimal fiber gives better resistance towards moisture, water and chemicals when compared with that of raw fiber. Besides this, structural and morphological changes, thermal stability and crystallinity of raw, graft copolymerized, pretreated and benzoylated fibers have also been studied by FTIR, SEM, XRD and TGA techniques. Enhancement in hydrophobic character and chemical resistance behavior of surface modified bhimal fiber has also been observed. All these properties of surface modified fibers could help in their applications especially in the synthesis of bio-fiber reinforced composites for their better end use.

Gupta *et al.* (2014) fabricated bhimal-reinforced-PVA bio-composite and determined its density, and other mechanical properties. The findings thus obtained are compared with other existing natural fiber bio-composites. From the observations, it is concluded that the bhimal fibers are lesser in weight than all existing fibers. Tensile strength of bhimal-reinforced-PVA bio-composite is higher than other natural fiber composites. Hence these could be utilized as reinforcement to produce much lighter weight bio-composites.

2.2.2 Review on banana fiber based bio-composites

Banana fiber is extracted from the pseudo stem Sheath of the plant. This fiber is used in many applications on of the famous utilization of banana fiber is seen in bio-composite fabrications due to its high strength and flexural properties. Following is the literature of it:

Misra *et al.* (2008) examined the Polyethylene/Poly composites reinforced with high-density banana fiber HDPE/PCL and this fiber is made in laboratory to check the mechanical properties. They got maximum tensile and young's modulus at 20 wt% of PLA. This was the reason behind their selection of 80:20 mixture of HDPE/PCL as a matrix material. When they added more than 15 wt% of PCL, ductility drops rapidly. HDPE/PCL plates of composite under self-weight of the bio-composite plate and their calculation of mechanical properties, theoretical dynamic analyses have been examined in this research work. Due to high tensile strength banana fiber reinforced plates stabilized later as compared to HDPE/PCL blend plate (orthotropic). As banana fiber contain increased they examined that damping properties reduced but stiffness improved.

Singh *et al.* (2011) had examined the hybrid bio composites which were reinforced with silica powder and banana fiber. SEM test result showed clearly the well dispersed banana fiber in an epoxy matrix. As they added banana fiber there is an increment in elasticity modulus, but the ultimate tensile strength of the resultant composite decreased as increment in silica. Silica addition gives comparatively better results than banana fiber. Banana fiber enhanced the impact strength of epoxy matrix but water absorption improved with fiber percentage.

Ghosh *et al.* (2011) had fabricated the vinyl ester resin composites which were reinforced with banana fiber. They varied the fiber volume fraction in the matrix and their effects were observed. As they increased the fiber percentage, noticeable improvements in tensile strength were noticed up to a certain percentage. At 35% of banana fiber they observed 38.6 % improvement in tensile strength but specific tensile strength enhanced with 65%. Tensile modulus was improved with 65%.When we compared the lower weight percentage of banana fiber with neat vinyl ester resin there is a reduction in strength. Composite made up of banana fiber reinforcement were lighter in weight so we can use them for interior parts of automobile so that weight of vehicle reduced and fuel consumption also reduced.

Raghavendra *et al.* (2013) had studied rubber composites reinforced with banana fiber and investigated there mechanical properties. They generally used short banana fiber and as matrix they took natural rubber fiber. They tested some of the mechanical tests as tensile test, tear strength test. They also examined the different lengths of banana fiber. SEM test was performed to check interfacial bonding between natural rubber matrix and banana fiber. With the improvement in fiber percentage, tensile strength also enhanced while there in less fiber percentage banana fiber and matrix interfacial bonding were also weak. Addition of banana fiber into natural rubber matrix also enhanced the hardness of resultant composites and strength and toughness also affected by this. 15mm length of banana fiber gave optimum tensile strength.

2.2.3 Review on bamboo fiber based bio-composites

Bamboo fibre has many excellent properties that make it ideal for reinforcing in composite. What people find most surprising is that bamboo fabric is exceptionally soft and

light, almost silky. It is also incredibly hydrophilic, absorbing more water than other conventional fibres such as cotton and polyester:

Shin *et al.* (1989) evaluated the mechanical properties such as compressive, tensile, flexural and shear properties of unidirectional bamboo-epoxy. Observations revealed that specific strength was found to be 3-4 time the specific strength of the mild steel. Mechanical properties were good enough to be compared with the ordinary glass fiber based composites. Further acoustic emission and SEM technique was used to examine the fracture behaviour of bamboo-epoxy composite under various loads. Morphological analysis showed that bamboo itself is a natural fiber composite; its presence in a polymer matrix will help eliminate the risk of cracking due to dehydration and bio-erosion caused by insect pests.

Rajulu *et al.* (1998) fabricated epoxy composite reinforced with short bamboo fiber with changing fiber length. The chemical resistance tests show that the composite materials are impervious to CH_3COOH, HCl, HNO_3, NaOH, Na_2CO_3 and C_6H_5-CH_3. The variety of tensile loading at break with fiber length has been contemplated and the tractable load is observed to be most extreme for the fiber length of 30 mm.

2.2.4 Review on coir fiber based bio-composites

The coir fiber can be used as a potential reinforcing material for making low load bearing thermoplastic composites. This gives an average value of tensile and flexural strength. Following is the brief review of coir fiber based composites:

Ayrilmis *et al.* (2011) examined the physical, mechanical and flammability properties of coir fiber reinforced polypropene (PP) composite panels. In this four different weight percentages of the coir fiber were added such as 40, 50, 60 and 70 wt % with a coupling agent i.e. maleic acid 3 wt%. Observation were made that whenever the fiber content is increased the water resistance and the internal bond strength were reduces whereas the mechanical properties such as flexural, tensile, hardness etc were increased upto the content percentage 60%.

Verma *et al.* (2013) reviewed the utilization of coir fiber and the researches those are done or running on this topic. The main approach of this work was to present the areas in

which coir fibers can be utilized as a part of bio-composites and the properties of coir fibers due to which in different application coir can be used as reinforcing agent, e.g. packaging, furniture etc.

2.2.5 Review on flax fiber based bio-composites

Flax fiber has a great tensile strength, low elongation, good vibration absorbing properties, ultraviolet rays blocking properties, High Water retention. This variety of fibers makes it useful in fabrication of bio-composites. Following is the quick review of flax fiber based composites:

George *et al.* (1999) prepared epoxy composites reinforced with flax fiber by autoclave moulding technique. In this work effect of various fiber parameters on mechanical properties of the composites were investigated. Interaction between the fiber and matrix was improved by using various surface treatment methods such as alkali, isocynate and silane treatments. Scanning electron microscopy (SEM), thermo-gravimetric analysis (TGA) and differential scanning calorimetry (DSC) and atomic force microscopy technique was used to characterize the treated surface. It was observed that surface modification of flax-fiber has noteworthy effect on the mechanical properties of the bio-composites.

Andersons *et al.* (2011) modeled the strength in tension in the fiber direction of an aligned flax fiber-reinforced composite assuming that a cluster of adjacent fiber discontinuities is the origin of fracture. A probabilistic model of tensile strength, developed for UD composites containing a microdefect, is applied. It follows from the theoretical analysis that the experimental tensile strength as a function the fiber volume fraction can be described with acceptable accuracy assuming the presence of a cluster of ca. 4×4 elementary fiber discontinuities.

2.2.6 Review on hemp fiber based bio-composites

Hemp or industrial hemp, typically found in the northern hemisphere, is a variety of the Cannabis sativa plant species that is grown specifically for the industrial uses of its derived products. Following is the brief literature of Hemp fiber based composite:

Baltina *et al.* (2011) very year an ever increasing number of individuals concentrate on sound, natural and ecological cordial living. Ecologically cordial way of life doesn't imply that we are utilizing just normal items, however consideration is indicated the assembling and creation handle moreover. The quick improvement of reused and biodegradable items causes extending utilization of hemp Fibers both in family and specialized materials. The investigation of hemp development and utilization slants on the planet and Europe demonstrates that hemp development and preparing in Latvia has great points of view. Item quality is affected by crude materials. For this situation it is hemp Fiber distinct attributes. Hemp Fibers are regular Fibers and their properties fluctuates as per plant developing territorial climatic conditions, measure of compost, plant thickness, gathering time and pre-treatment mechanical procedures. There are contemplated the impact of previously mentioned factors on synthetic creation, geometrical and physical properties of the Latvian hemp Fiber both nearby and remote cultivars.

Manthey *et al.* (2012) fabricated a woven jute fiber reinforced novel acrylated epoxidized hemp oil (AEHO) based bio-composite. Mechanical properties in the form of flexural and interlaminar shear strength (ILSS) were investigated and compared for the different samples. Results from the mechanical tests showed that AEHO and 50/50 based neat bioresins displayed lower flexural properties compared with the VE samples. However when applied to bio-composites and compared with VE based samples, AEHO bio-composites demonstrated comparable flexural performance and improved ILSS. These results are attributed to improved fiber-matrix interfacial adhesion due to surface-chemical compatibility between the natural fibers and bio-resin.

2.2.7 Review on jute fiber based bio-composites

Jute fiber is the most commonly used fiber as reinforcing material for composite fabrication in India. Following is the brief review of jute fiber based composite:

Abedin (2007) prepared chemically modified Jute fiber (Hessian cloth) and e-glass fiber (mat)-reinforced unsaturated polyester (USP) resin composites using the hand layup technique at room temperature (25^0C). A strong interfacial adhesion between jute and polyester was obtained using chemically modified jute fiber. This modification involves the

introduction of reactive vinylic groups at the surface of the fiber and matrix through esterification of jute hydroxyl groups by methacrylic anhydride. The optimum values of the mechanical properties were observed at the 25 weight % of the jute fiber content. And when different % of glass fiber was added to the composite it showed further increment in the mechanical properties. Among all the resulting hybrid composites, the composite with a jute to glass fiber ratio of (1:3) demonstrate the improved mechanical properties such as tensile strength (TS) 130 %, tensile modulus (TM) 50 %, bending strength (BS) 150 %, and bending modulus (BM) 225 % over untreated jute composite. SEM results of fracture surfaces of unmodified and modified jute-polyester bio-composites clearly exhibited superior fiber-matrix bonding in the case of the latter. No more than two types of formulation are suited for the UL94 standard in the case of vertical burn method, nevertheless most of them have passed Horizontal Burn Tests. Flammability test results have evidenced that a flame retardant content of 20% is enough to manufacture the composites with natural fillers being suited for the flammability risk applications. Thus, further activities should focus on carrying out different tests aimed at both optimizing a formulation of new composites and demonstrating their feasibility as engineering materials.

2.2.8 Review on kenaf fiber based bio-composites

Like other natural fibres, kenaf is arousing a growing interest, and not just on the part of stakeholders in the composite industry. As the environment becomes not only a major concern but also a source for development, public authorities far and wide are also getting involved, and many projects are emerging. Following is the brief review of kenaf fiber based composites:

Udoeyo et *al.* (2012) evaluated the Physical and mechanical characterstics of water-retted Kenaf fiber reinforced mortar composite according to ASTM and other suitable standards. In this work, composites of size 650 × 450 × 8 mm^3 was fabricated. In order to get optimum required properties, three different weight percentages of fiber contents (0.5 %, 1.0 % and 1.5 %) and four different lengths of fiber (20 mm, 30 mm, 40 mm and 50 mm) were taken into consideration in the study. The findings of the research experiment revealed that impact resistance and flexural toughness of the composite were enhanced with the increase in

fiber content when compared to composite without fibers but at the same time the bending capacity of composite decreased with increase in fiber content. The water absorption test and the fire resistance test also gave the satisfactory results specified with concerned standards.

Hao *et al.* (2013) evaluated the mechanical properties of the kenaf and polypropylene non-woven bio-composites (KPNCs). It used needle-punching and carding technique to fabricate composite with equal weight ratio of kenaf fiber and polypropene followed by compression moulding method. The tensile, flexural, shear, and Izod impact tests were conducted in order to estimate the mechanical properties of bio-composite. Thermo-gravimetric analysis (TGA), dynamic mechanical analysis (DMA) and differential scanning calorimetry (DSC) techniques were used in order to check thermal properties of the kenaf/polypropene bio-composite. The sound absorption behavior and sound insulation was also examined. An outstanding sound absorption and insulation properties were shown by adhesive-free sandwich structure. Based on the evaluation of end-use performance, the best processing condition combination of 230°C and 120s was determined, and the correlation between mechanical properties and acoustical behavior was also verified by the panel resonance theory.

2.2.9 Review on sisal fiber based bio-composites

Sisal is a natural fiber derived from the long, green leaves of the *'agave sisalana*' cactus plant. Sisal grows in semi-arid regions in Africa and Brazil. Sisal fibers (which can be up to three feet long) are sustainably harvested by hand from the leaves of the cactus plant. Following is the literature of sisal fiber based bio-composites:

Zhong *et al.* (2007) used the alkali-treated sisal fibers were used as novel reinforcement to get bio-composites with self-synthesized UF (urea-formaldehyde) resin as matrix phase. The composites were fabricated by compression molding technique, and then the impact of weight % of sisal on mechanical properties such as flexural strength, impact strength, and wear resistance were examined. Further water uptake was analyzed and morphological behavior was showed by the scanning electron microscopy (SEM). The bio-composite having 30 wt% showed the outstanding flexural strength, water absorption, and especially the wear resistance revealing that it is having the best bonding and adhesion of all

the composites with various weight %. The composite has significant potential for improving resistance to abrasive wear due to sisal characteristic properties. The fiber itself possesses a better wear resistance than the UF resin and should extend beyond the surface after a passage of time. Therefore, the application of these bio-composites in fiberboard can be expanded.

Naidu *et al.* (2011) combined biological and synthetic fibers in the similar matrix (unsaturated polyester) to make hybrid composites of Sisal and Glass fiber. The compressive and impact properties of these hybrid composites were examined. In order to do this,1%, 2% and 3% by weight percentage of resin of chalk powder was added as additive and effect of various weight % of chalk powder on impact and compressive properties of the bio-composite was examined. In this way, an observation was made that with increasing weight percentage of chalk powder, the compressive and impact strength of the composite was getting reduced. Also the mechanical strength i.e. compressive and impact was checked with the weight percentages of the fibers (sisal and glass). Therefore it was concluded that Sisal and Glass fiber hyrid component was having the better mechanical properties such as than the resin reinforced with sisal fiber but lower than the resin reinforced with the glass fiber. In case of loading the load is transferred to glass fiber after the failure of the sisal fiber. So the existence of glass fiber in the hybrid bio- composite of sisal and glass fibers causes to improve the impact and compressive strength.

2.2.10 Review on sugarcane fiber based bio-composites

Bagasse is the fibrous residue which remains after sugarcane stalks are crushed to extract their juice. It is mainly used as a burning raw material in the sugar mill furnaces. When appropriate modifications and manufacturing procedures are applied, bagasse displays improved mechanical properties such as tensile strength, flexural strength, flexural modulus, hardness, and impact strength. The literature review of the sugarcane fiber based bio-composites is as follows:

Xu *et al*. (2010) have studied the creep behavior of composites based on bagasse fiber with virgin and recycled polyvinyl chloride (B/PVC) and polyethylene (B/HDPE) of high density. The immediate deformation and rate of creep of all bio-composites at the equal level of loading increased at high temperatures. At an unvarying level of loading, B/PVC bio-

composites had superior creep resistance than B/HDPE systems at lower temperatures. Nevertheless, B/PVC composites illustrated more temperature-dependency. Numerous creep models (i.e. Findley's power law model, Burgers model and a simpler two-parameter power law model) were utilised to fit the calculated creep data. Time–temperature superposition (TTS) was done for long-run creep prediction. Temperature showed a large impact on creep behaviour of all of the composites. B/PVC had superior creep resistance than B/HDPE at low temperatures, but they illustrated higher temperature-dependence. The TTS principle was applied to these composites and smooth master curves were obtained. The TTS better predicted long-run creep behaviour from short-term creep tests for the PVC composites than the HDPE composites.

Cerqueira *et al.* (2011) have evaluated the impact of chemical change on mechanical properties of bagasse Fiber/PP composites. Fibers were pretreated with 10% H_2SO_4 solution, in addition to delignification with 1% NaOH solution. These Fibers were blended with the polypropene in a thermo-kinetic mixer, and compositions with 5-20 weight % of Fibers were obtained. Tension test, flexural and impact tests were used to evaluate the mechanical properties.

Further, SEM (scanning electron microscope) was performed in order to do fracture analysis. Finally sugarcane bagasse fibers were modified successfully and it was confirmed that the whole process effectively improves mechanical and morphologiocal properties when compared to the pure polymer and untreated composite.

The summery of the above literature review has been summarized in the form of table 2.1 for the epoxy resin and bhimal fiber based bio-composite.

Table 2.1: Summary of literature survey

AUTHORS	YEAR	RESEARCH WORK
Singha *et al.*	2008	Found the properties of compression molded *Grewia Optiva* (Bhimal) fiber reinforced Urea-Formaldehyde (UF) matrix based polymer composites and showed that mechanical properties such as tensile strength, compressive strength and wear resistance of urea-formaldehyde resin increases to a significant extent when reinforced with bhimal fiber.
Singha *et al.*	2012	Studied effect of surface modification of *Grewia Optiva* fibers on their physicochemical and thermal properties. It was found that 5% benzoyl chloride treated and graft copolymerized bhimal fiber gives better resistance towards moisture, water and chemicals.
Gupta *et al.*	2014	Investigated the effects of bhimal fibers in mechanical strength of the PVA resin and concluded that addition of fiber to the PVA resin greatly improves its mechanical properties in comparison of other bio-composite.

Prakash and Christu	2016	Fabricated the epoxy resin reinforced with human hair To test its suitability, mechanical properties such as tensile strength, compressive strength and hardness. The results showed that there was significant improvement in all the mechanical properties.
Haldar *et al.*	2017	Fabricated two separate composites made and found that without addition of aluminium powder, sisal reinforced epoxy composite showed lower density but micro hardness was excellent whereas tensile, impact and elastic modulus of sisal reinforced epoxy composite with aluminium powder was better than without aluminium based sisal-epoxy composites.

2.3 Conclusion

The literature reviewed in the above sections found that researches are still in vogue to improve mechanical and micro-structural properties of bhimal based bio-composite. In this chapter summary of various research papers and journals, related to the study of several properties of different bio-composite is mentioned. These papers show the consequence of addition of several natural fibers, coatings and surface treating chemicals. In the present research work, the mechanical and thermal properties of bhimal fiber based composite is examined as it was observed from literature that very rarely research has been done on bhimal fiber.

Chapter 3 MATERIALS AND METHODS

The subsequent chapter depicts the materials and methods that are used for the fabrication and processing of the composites under this investigation. It also showcases the detailed version of characterization and experimentation which the composite samples are subjected to.

3.1 Materials

The various materials which have been used in the production of composite like the epoxy resin, hardener/curing agent, reinforcement fiber, chemicals and other resources are discussed below.

3.1.1 Matrix material

Matrix material in this present context binds the fibers together and serves as an intermediate by which an externally applied stress is transmitted and distributed to the fibers. It also protects the individual fibers from the surface damage and behaves as a barrier to crack propagation.

3.1.1.1 Epoxy resin (CY-230)

The matrix phase of fibrous composites may be of different categories like ceramics, metals and polymers. In broad-spectrum, the polymer-matrix composites are being used due to its cost effectiveness, easiness of fabricating the complex parts and their stable room temperature properties. Polymer matrices can be classified into thermo-set or thermoplastic. Thermo-set or network matrices are formed as a result of an irreversible chemical transformation of the resin into an amorphous cross-linked polymer matrix. As a result, they become everlasting hard and do not soften upon heating. Their key properties include good electrical thermal insulation, low viscosity, exceptional thermal stability and enhanced creep resistance. Normally, these resins include epoxies, phenolic, vulcanized

rubbers and some polyesters. The physical and chemical properties of epoxy resin are shown in table 3.1.

Table 3.1: Physical and chemical properties of epoxy resin (Araldite CY-230)

S. No	Epoxy Resin (Araldite CY-230)	
1.	**Physical Property**	Yellow-brown, tasteless, odorless and completely non-toxic in nature
2.	**Chemical property**	Product of reaction between bisphenol-A and epichlorohydrin

In general, the thermo-set matrices are stronger and harder than the thermo-plastic ones. The word resin is repeatedly been used to denote a high molecular weight reinforcing plastic. The major circumstances for using the resin for a lengthy period of time are cool storage and dry conditions in the original containers. In the present investigation, Araldite CY-230 (thermo-set epoxy resin) which has been purchased from M/s CIBATUL Limited, India is being used as the matrix material. The chief motive for using the epoxy resins are their outstanding adhesion to variety of fibers, enhanced resistance to atmospheric attack and excellent performance at elevated temperatures. On the contrary, epoxies are more costly and non-biodegradable.

3.1.1.2 Hardener (HY-951)

Hardener (HY-951) purchased from M/s CIBATUL Limited, India has been used as the curing agent. It is a yellowish-green colored liquid and during this exploration an amount of 9 wt. % of hardener has been used in the development of the composite. In the

present investigation, the weight percentage of hardener used is as per the recommendation of **Singh (2002)** (Fig. 3.1(a)).

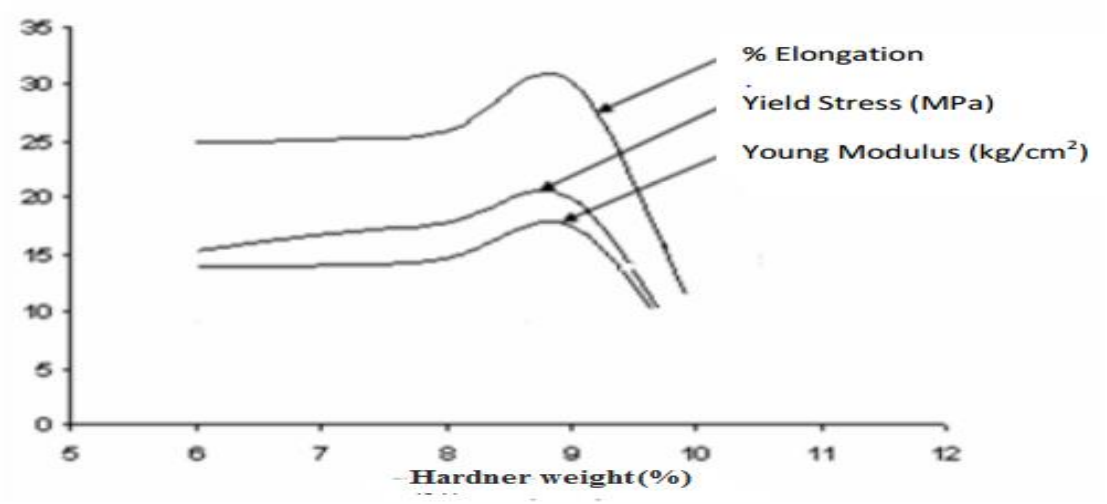

Fig. 3.1(a): Effect of wt. % of hardener (HY-951) on mechanical properties of epoxy resin (Singh, 2002)

3.1.2 Reinforcement material

Normally, by accumulating the reinforcement to the matrix phase provides a high degree of crystalline perfection to the composite which results in high strengths. The reinforcement used in this current research is the bhimal fiber mainly to improve the properties of the resultant composite.

3.1.2.1 Bhimal fiber

Bhimal fires are quite a newer kind of bio-degradable fibers. They have very rarely been heard before in literatures from the point of view of their utilization as engineering material. These fibers have been utilized for investigation of their properties. It is also known as,

- Biological name: *Grewia Optiva*.
- Local names: Hindi (Dhaman, Biung, Biul, Bihul, Bhimal, Bhengal, Bewal).

- Nepali: (Shyalphusro, Phusre, Ghotli, Bhimal).
- Trade name: (Dhaman, Biul).

Bhimal (Fig. 3.1 b) is a small to medium-sized deciduous tree, 9-12 m in height; crown spreading, and about 1m in diameter. Its branches are smooth, pale silvery-brown in colour, bark is dark brown, thick and roughish.

Fig. 3.1 (b) Bhimal trees, (K. Chandrasekhar *et al*., 2011)

The timber is used for oar shafts, poles, frames, tool handles and other purposes where strength and elasticity are required. The bark yields a fiber that is used for cordage as shown in Fig. 3.1 (c).

Fig 3.1 (c) Cordage made from bhimal fibers, (K. Chandrasekhar *et al*., 2011)

3.1.3 Potassium hydroxide (KOH)

Potassium Hydroxide (KOH) was supplied by the M/s Allied Business Limited, India. It was used for the alkali treatment of bhimal fibers. Bhimal fibers were soaked in the 5% concentration of KOH for almost 3-4 hours at 28°C. After that they were thoroughly washed in the distilled water and finally put to dry at 60°C in the oven.

3.2 Methods and Experimental Setup

In this, the whole procedure of fabrication such as mould preparation, alkali treatment of fiber, Casting and preparation of the composite is discussed. All experimental setups are shown with help of pictures. Following are the steps involved in preparation of bio-composite;

1. Mould preparation
2. Casting
3. Alkali treatment
4. Preparation of bhimal fiber reinforced epoxy resin

3.2.1 Mould preparation

In this present experimentation, simple hand lay-up technique was used. The mould was prepared using a galvanized iron sheet having the dimensions of 200 mm × 100 mm × 15 mm which was open from the top as shown in Fig. 3.2(a). The mould was uncontaminated with the help of acetone and then the releasing agent i.e. silica gel was applied on its surface for easy removal of the casting and finally the mould was ready.

Fig. 3.2(a): Model of the mould box

3.2.2 Casting

In this scheme, the bhimal fibers at varying weight percentages are thoroughly mixed in the epoxy resin in such a way that no bubble formation takes place. The solution thus obtained is kept in the oven (shown in Fig. 3.2(b)) at a temperature of 100 ^{0}C for two hours as per the recommendation of **Singh and Gope (2010).** Then the blend is cooled to a temperature of 40 ^{0}C followed by the addition of hardener to the mixture. Finally, the mixture is all set to be transferred inside the mould. The convection oven used for heating is having the temperature range of 0-240 ^{0}C.

3.2.3 Alkaline treatment

Biological fibers have a characteristic property of lacking strong interfacial adhesion with the polymers. That's why an alkaline treatment is used to boost the interfacial adhesion between the synthetic matrices and the biological fibers. Alkaline treatment removes a definite quantity of oils, wax and impurities that coats the exterior surface of the fiber. The significant amendment attained with this treatment is the interruption of hydrogen bonding in the complex structure, resulting in increase of the surface roughness. In this episode, the bhimal fibers (Fig. 3.2(c)) were drenched in 5%

KOH solution for 1-2 hrs at 30 ^{0}C followed by washing with the distilled water and then drying at 70 ^{0}C.

3.2.4 Preparation of bhimal fiber - reinforced composite Epoxy resin

Chopped bhimal fiber of 0.5 to 3 mm length at different percentages were totally dissolved in the epoxy resin and then put to oven. The resultant epoxy resin was kept in the motionless air to cool down a temperature of 40^{0}C, as the temperature accomplishes 40^{0}C

Fig. 3.2(b): Prepared composite of various fiber wt% of bhimal fiber

to the hardener was poured into the mixture and finally kept to solidify. The combination thus acquired was emptied in the moulds which were already cleaned with acetone and layered with the silica gel. Finally, the mould was opened after 2-3 days of time period and the required specimens were cut from it.

In order to prepare composite of different bhimal fiber content following steps were taken;

1. For neat epoxy resin casting, take 91 parts of epoxy and 9 parts of hardner with 0 parts of bhimal fiber.

2. For 2 wt. % bhimal fiber reinforced epoxy resin, take 91 parts of epoxy and 9 parts of hardner with 2 parts of bhimal fiber.

3. For 3 wt. % bhimal fiber reinforced epoxy resin, take 91 parts of epoxy and 9 parts of hardner with 3 parts of bhimal fiber.

4. For 4 wt. % bhimal fiber reinforced epoxy resin, take 91 parts of epoxy and 9 parts of hardner with 4 parts of bhimal fiber.

5. For 5 wt. % bhimal fiber reinforced epoxy resin, take 91 parts of epoxy and 9 parts of hardner with 5 parts of bhimal fiber.

6. For 6 wt. % bhimal fiber reinforced epoxy resin, take 91 parts of epoxy and 9 parts of hardner with 6 parts of bhimal fiber.

Fig. 3.2(c): Convection Oven

3.3 Physical Tests

In this, the physical properties suc as density and % water absorption were estimated by using conventional methods. Tests which were carried out are as follows.

1. Density test
2. Water absorption test

3.3.1 Density test

Density is a physical property of the material which determines the mass per unit volume of any material. It is mostly determined in the units of kg/m^3 or gm/cm^3 For density measurement, the sample of a bundle of bhimal fiber was taken whose weight was computed on the electronic weighing machine which has a least count of 10^{-2} g and finally all the material were dipped in water in a measuring flask of 50 ml having a least count of 0.5 ml and the change in volume was recorded

The actual density of the composite sample was calculated by the formula:

$$\rho = \frac{m}{V} \qquad \text{...(3.1)}$$

Where *m*, *V* and ρ symbolize the mass, volume and density respectively.

Weights of the Bhimal Fibres have been measured from digital electronic balance, which is shown Fig. 3.3 (a) and its specifications are as follows.

- Maximum weight capacity = 120 gm.
- Readability = 0.1 mg or 0.0001 gm.

Thus, the density of bhimal fibres is calculated as below.

- Density of water = $1gm/cm^3$

Fig. 3.3 (a) Digital electronic balance

Density of water = 1gm/cm^3

$$\frac{Density of\ Bhimal\ Fibres}{Density of\ water} = \frac{Weight of\ Bhimal\ Fibres}{Weight of\ water\ displaced} \quad ...(3.2)$$

3.3.2 Water absorption test

Water absorption test is done by taking a small bundle of fibers by dipping them into water for couple of hours. Then wt. % of absorbed water at different time interval for different number of fibres were recorded. The weight of a small bundle of fibres is measured by using electronic balance. Finally a graph was plotted in order to check the pattern of variation in % water absorption of the bhimal fiber with respect to time. Percentage water absorption was calculated by given formula.

$$\%\, water\, absorption = \frac{weight\, of\, water\, after\, dipping - initial\, weight\, of\, water}{initial\, weight} \times 100 \quad ...(3.3)$$

Water absorption test is done by taking a small bundle of fibres shown in Fig. 3.3 (b).

Fig. 3.3 (b) Bhimal Fibers (dipped in water)

The weight of a small bundle of fibers is measured by using electronic balance. Water absorption test is done by taking a bundle of 10, 20, 50 and 100 fibers. First the bundle of fibers are weighed in dry state and then dipped in water. The weight is taken at an interval of 2 hours up to 14 hours and then experiment has been carried-out for a total of 48 hours. During weighing each time at 2 hours interval, the bundle was rinsed of the water, but not squeezed.

3.4.1 Micro - Structural Tests

Micro-structural test is done with the help of Scanning Electron Microscopy (SEM) test In this technique, the morphology of a specimen to be scrutinized is scanned with beam of electron and the back scattered beam of electron is accumulated, and then the displaying of image at the same scanning rate on a CRT television screen is done with a high resolution. In the present exploration, SEM study has been done to see the distribution of bhimal fiber in epoxy resin. The images are achieved through microscopic

inquiry with the model LEO435V6. To acquire the scanning electron micrographs of each percentage sample, the fractured surface (Fig. 3.5(a)) is taken into

Fig. 3.4(a): Specimen for the SEM Test

consideration and is gold coated to keep away from any artifacts that are associated with sample charging and finally are placed inside a compartment in which an electron beam falls on the sample. The accelerated voltage in the machine used was 10 kV and the results being in the form of images taken at different magnification ranges. The micrographs obtained by the SEM technique are shown in the Fig. 4.2 (a-n).

3.5 Mechanical tests

Numerous mechanical tests were conducted to determine the distinctiveness of the composite material, which are determined below:

1. Tensile test
2. Compression test
3. Flexural test
4. Hardness test
5. Impact test

3.5.1 Tensile test

Tensile test is one of the most universal mechanical stress-strain tests. It is utilized to determine the young's modulus, elastic limit, percentage elongation, proportional limit, yield strength and yield point. The sample construction is shown in the Fig. 3.5 (a) and 3.5 (j). The tensile tests were carried out on the 25 kN Tinius Olson Tensometer, Fig. 3.5(e). The testing speed i.e. the rate of separation of the gripping jaws was taken to be 1 mm/min. The relative humidity and the temperature at the testing time were 50% and 29°C respectively. The consequence in the form of stress-strain diagram is showcased in Fig. 4.3(a). All the samples for tensile testing were organized according to the ISO 527:2012 standard.

3.5.2 Compression test

The compression stress-strain test is merely the overturn of tension test in regard to the direction of loading. In brittle and fibrous materials, the compressive strength is noticeably diverse from the tensile strength. Therefore, it becomes very much essential to conduct the tension and compression tests individually. It results in the determination of mechanical properties that mainly includes the compressive ultimate strength, compressive yield strength and percentage reduction in length etc. All the compression tests were conducted on 25 kN servo Tinius Olson Tensometer, Fig. 3.5(e). Sample configuration used for the compression test is revealed in Fig. 3.5(d). Sample for the compression test are arranged according to the ISO 604:2002 standard. All the tests are conducted under the displacement mode of control at a test speed of 1 mm/min. Here the test speed refers to rate of approach of the plates of the machine during the testing time. The ambient

conditions remain the same as already defined for the tensile test. The resultant stress-strain diagram for composites tested under the compressive test is shown in Fig. 4.3(c).

3.5.3 Flexural test

The main parameters that a 3-point flexural/bending test provides are the values of flexural stress-strain and flexural modulus of the composite material. Its main benefit is the ease of preparing the sample and the test set up. The sample geometry and the test set up are showcased in the Fig. 3.5(b) and Fig. 3.6(g). The main parameters that govern the flexural test are the specimen properties, loading condition and strain rate. The temperature recorded was 27°C and the relative humidity (RH) was 51 %. The corresponding test standard followed was ISO 178:2010. In this test, a test speed i.e. the rate of relative movement between the specimen supports and the loading edge of 1 mm/min was employed. The flexural stress (σ_f), flexural modulus (E_f) and flexural strain (ε_f) for a rectangular cross section are determined by the formulas given underneath:

$$\sigma_f = \frac{3PL}{2bd^2} \quad \text{...(3.4)}$$

$$E_f = \frac{L^3 s}{4bd^3} \quad \text{...(3.5)}$$

$$\varepsilon_f = \frac{6Dd}{L} \quad \text{...(3.6)}$$

Where,

P = load

b = width

d = thickness

L = supported span length

D = maximum deflection

3.5.4 Hardness test

The digital hardness testing machine on L scale was used to carry out the Rockwell hardness test. The main advantage of the Rockwell hardness test is its quick and direct reading; no calculations are to be performed. For polymer matrix composite, the ball indenter having the diameter $^{1}/_{4}$ inches (6.35 mm) is selected as specified by the standard ISO 2039:2001. The ball indenters are normally prepared of tungsten carbide or hardened tool steel. In this test an indenter is firstly seated in the material to be tested by the application of load to a predefined set point. Then a load of 60 kgf is applied for a dwell time period of 3 seconds and then the unloading is done and finally the hardness is interpreted from the digital indicator on the machine. There is a red colored light which indicates whether the hardness is small, high or within a working range. The hardness was taken at various points on the sample and the average value was calculated. The environmental temperature at the time of test was recorded to be 27°C and the relative humidity (RH) was 52 %. The Digital Rockwell hardness machine used for this present investigation is shown in the Fig. 3.5 (j).

3.5.5 Impact test

The impact strength of any substance is firmed with an Izod or Charpy impact test named after their inventors and were initially developed in the early 1900's. The primary difference these tests is that the Charpy test uses a simply supported beam while the Izod uses a cantilever beam. The Izod impact test deals with the energy required to fracture a notched sample. The sample smacks with a weighted pendulum hammer of known kinetic

energy and by determining the energy vanished by this hammer, a relative capacity of material's toughness can be firmed. The ambient temperature at the testing time was 28°C accompanied by a relative humidity (RH) of 49 %. Standard specimen geometry as specified by the ISO 180:2000 standard and the actual specimen geometry for the impact test are shown in Fig. 3.5 (c) and 3.5 (h) respectively. In the current examination, Izod test was carried out on the impact testing machine as shown in the Fig. 3.5 (i). After that all the dimensions of sample are entered in the equipment and the weighted pendulum hammer is made to strike at the composite sample. Finally, the outcomes are copied from the display given in the device.

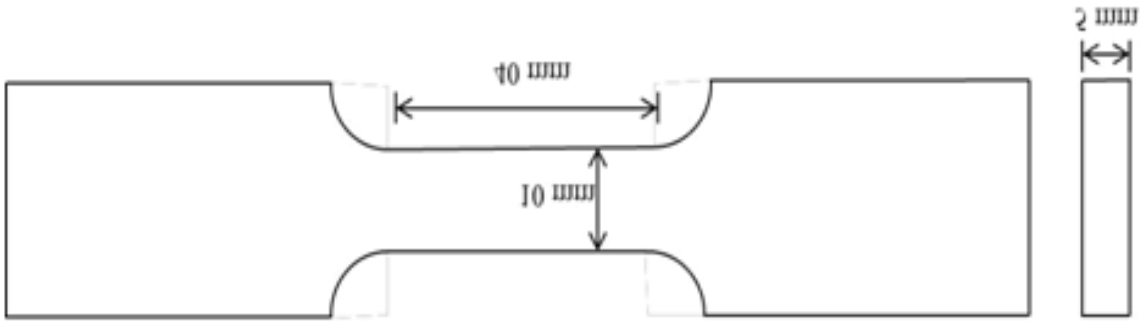

Fig 3.5 (a): Dimensions for tensile test specimen

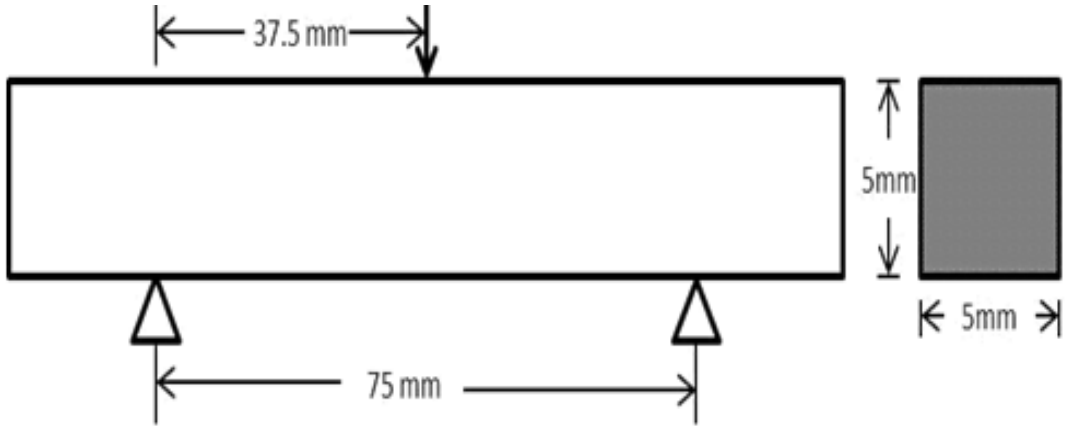

Fig 3.5(b): Specimen dimensions for a 3-point flexural test

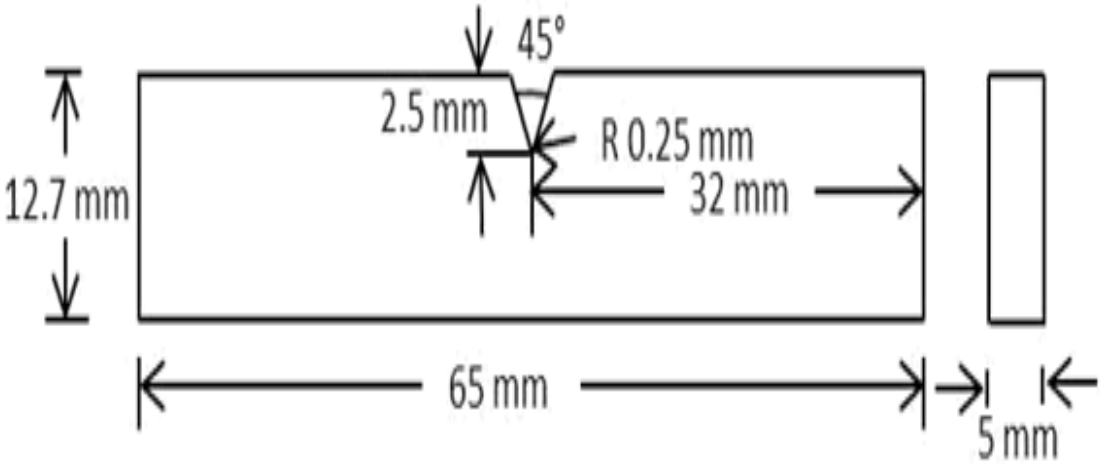

Fig 3.5 (c): Specimen dimensions for the impact test

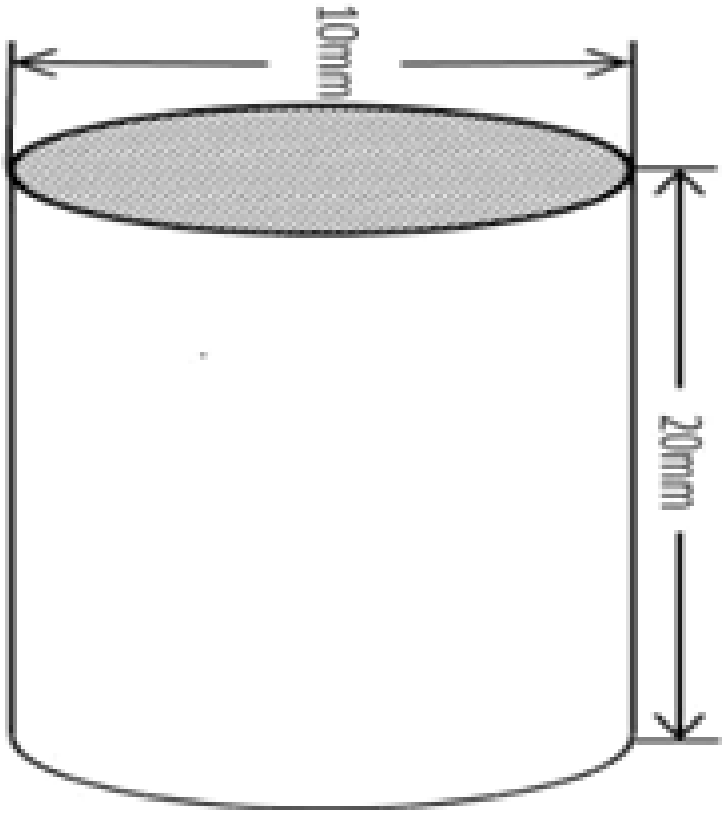

Fig 3.5 (d): Specimen dimensions for the compression test

Fig. 3.5 (e): Tinius olson tensometer

Fig 3.5 (f): Fixture used for the compression test

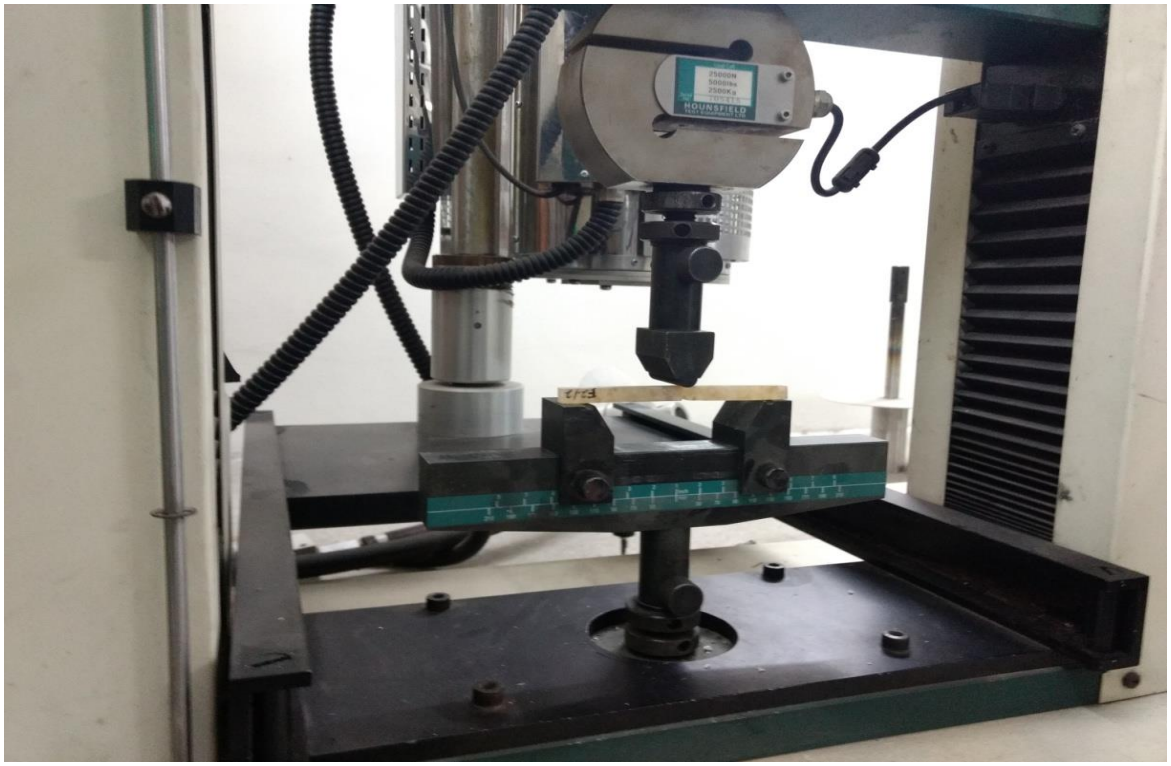

Fig 3.5 (g): Setup for a 3-point flexural test

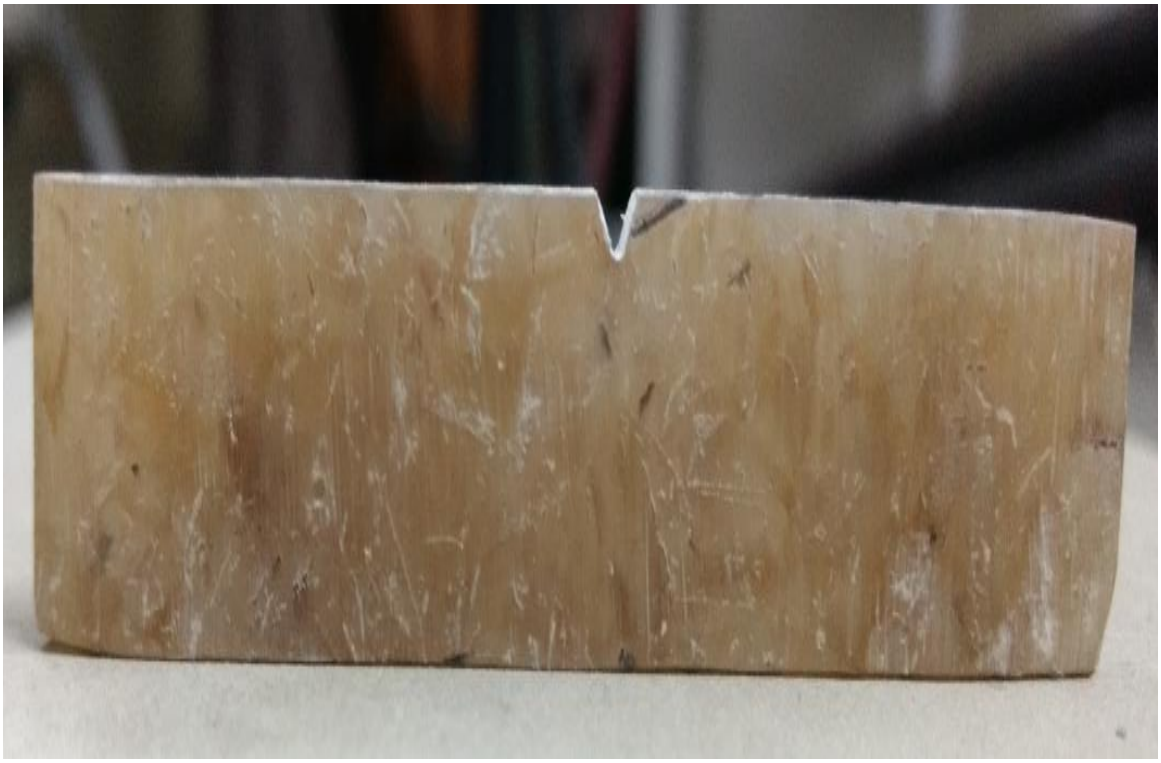

Fig 3.5(h): Actual specimen for impact test

Fig 3.5 (i): Impact testing machine

Fig. 3.5 (j): Digital Rockwell hardness testing machine

Fig. 3.5 (k): Actual specimen for tensile test

3.6 Thermal Tests

Thermal analysis is a division of science and technology where the properties of resources are studied as they transform with a change in temperature value. In thermal analysis for the present investigation, Differential Thermal Analysis (DTA) and Thermo Gravimetric Analysis (TGA) are done. This scrutiny was done using the TGA analyzer (SII 6300 EXSTAR) at the Institute Instrumentation Centre, IIT Rookie by heating the samples in the still air (200 ml/min) at the rate of 10°C/min from 0^0C to 800^0C. The plots are exposed in the Fig. 4.4(a-f) of this effort.

3.6.1 Thermo gravimetric analysis (TGA) test

Thermo-gravimetric analysis or thermal gravimetric analysis (TGA) is a branch of thermal science which examines the property change of a given sample as a function of change in temperature when heating rate is constant or as a function of time when

temperature is constant. TGA is mainly used to determine the organic as well as the inorganic composition of the sample and also to inspect the reaction kinetics and degradation mechanisms that occur in the model. Fig. 3.7(a) shows a classical thermo-gravimetric (TG) graph which characterize for a given substance because of the exclusive series of the physiochemical reaction that happens over a precise temperature range and predefined heating rates. In this context, the change in mass of a sample on heating under a restricted environment is known as Thermo-gravimetric analysis (TGA). Its key applications include measurement of a resource thermal composition and stability.

3.6.2 Differential thermal analysis (DTA) test

It is one of the most extensively used schemes of thermal analysis. In DTA technique, temperature of the specimen is put side by side with that of an inert reference substance for comparison during a predefined change in temperature. The temperature during the entire process should be constant i.e. it should be an isothermal process until the thermal event arises for example in melting, decomposition or change in the crystal structure. Suppose an endothermic event occurs in the sample, temperature of the specimen will be less than that of the reference one and as a result a minimum will be seen on the curve. On its contrary side, if an exothermal event occurs, then temperature of the specimen will be more than that of the reference material and a

maximum will be seen on the curve as shown in the Fig. 3.7(b). The graph peaks give an idea about whether there is an energy dissipation or energy absorption i.e. the reaction inside the matter is exothermic (EX) or endothermic (EN). As a result, we get the area under the endotherm or exotherm which is linked to the enthalpy of the corresponding thermal event, ΔH.

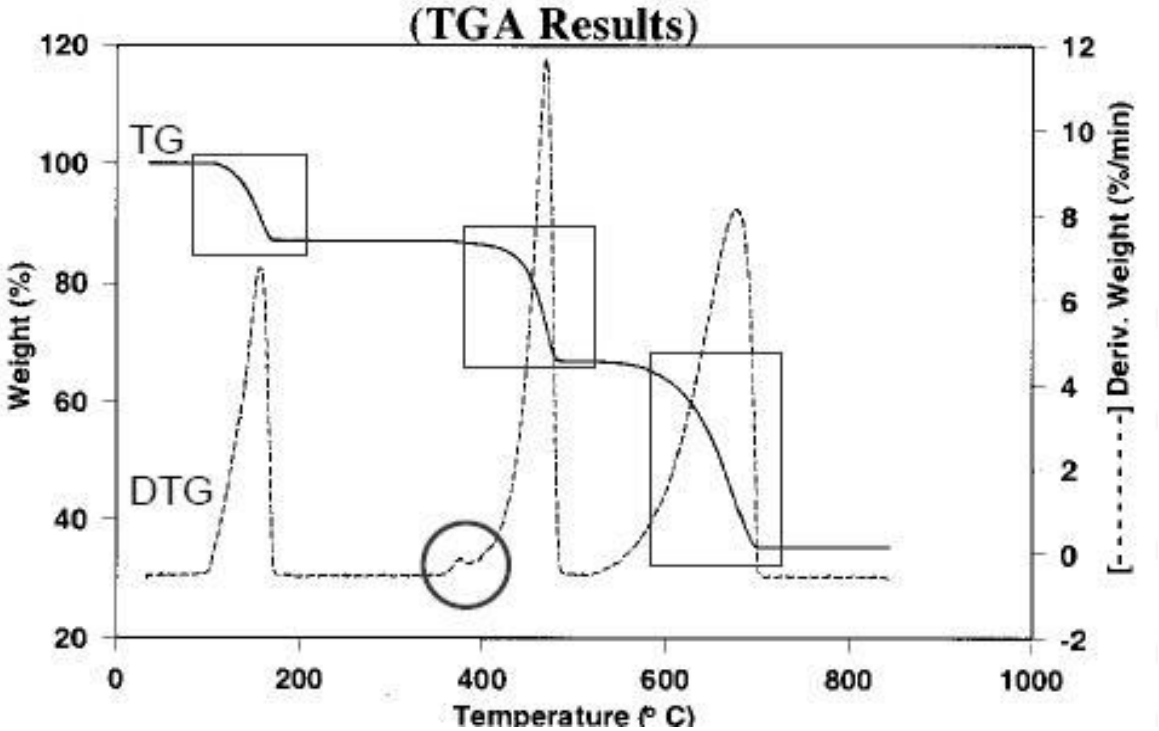

Fig. 3.6(a): Schematic representation of a TGA curve

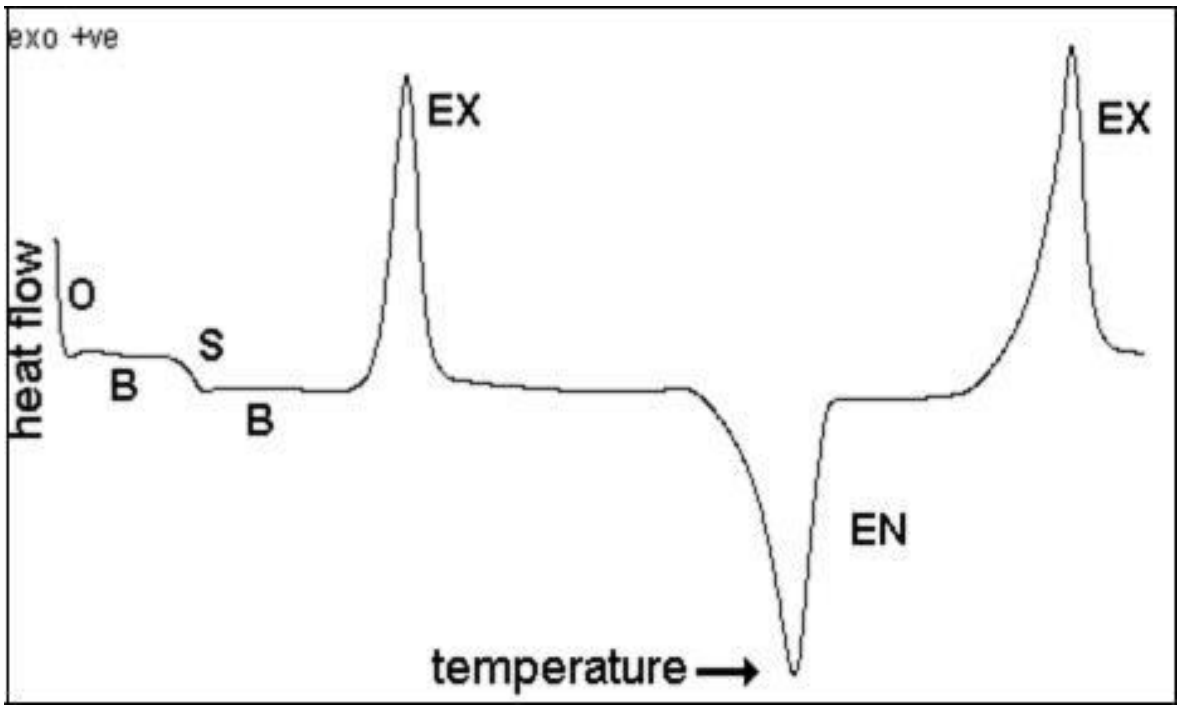

Fig. 3.6(b): Example of a DTA

Chapter 4 RESULTS AND DISCUSSION

In this chapter, the outcomes obtained from physical tests such as density test and water absorption test of bhimal fiber, scanning electron microscopy, mechanical and thermal tests of bhimal fiber reinforced epoxy resin are presented and discussed.

4.1 Results of Physical Tests of Bhimal Fiber

In this result of the physical tests such as density test and water absorption test is discussed. Results are as follows

4.1.1 Density test

By putting all values in equation (3.2) we get that,

Density of bhimal fiber $= 0.453\ gm/cm^3$

This value of density of bhimal fiber is comparatively lower than that of the other conventional natural fibers such as banana, sisal, hemp, sugarcane etc. This can be seen from given table;

Table 4.1 Density comparison with other Fibres (Xia et al. 2009)

S. No.	Fibres	Density (gm/cm^3)
1.	Palmyra Fibre	0.69
2.	Ramie Fibre	1.55
3.	Sisal Fibre	1.45
4.	Coir Fibre	1.15-1.46
5.	Flax Fibre	1.50
6.	Jute Fibre	1.30-1.49
7.	Hemp Fibre	1.47
8.	Cotton Fibre	1.50-1.60
9.	**Present work**	**0.453**

4.1.2 Water absorption test

The water absorption characteristics curve for bhimal fibre is steep at start and after some time it becomes approximately horizontal. It is because the fibres initially absorb more water and after some time it absorb water at very slow rate. The percentage weight gain varies from 3.97 % to 96.06 % for different bundle of fibres.

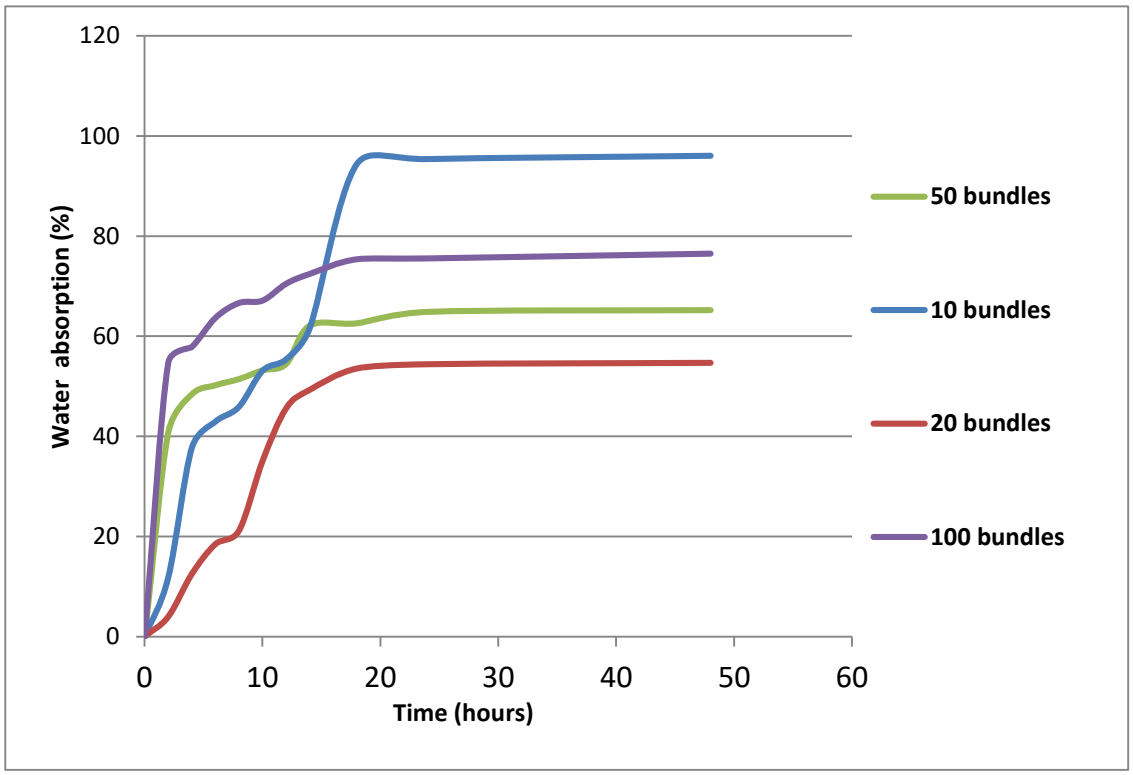

Fig.4.1 (a) Water absorption characteristics of bhimal fibres

4.2 Results of Micro-Structural Tests of Bio-Composite

The morphology or surface characteristics of the resultant fabricated composite materials are considered through the Scanning electron microscopy (SEM) technique. The cross sectional scrutiny of the fabricated composite materials consisting of epoxy resin, various wt% of bhimal fiber and hardener are offered in the Fig. 4.2(a-n). The Scanning Electron Microscopy (SEM) images are taken to monitor the internal cracks, interfacial properties and internal structure of the fractured surfaces of fabricated composite materials. Fig. 4.2(a-n) give you an idea about the SEM pictures of fractured surfaces of various composite materials fractured under the tensile loading in the present work. Fig. 4.2(a) shows morphology of neat epoxy resin, Fig. 4.2(b-c) of epoxy resin with 2 wt% of bhimal fiber, Fig. 4.2(d-f) of epoxy with 3 wt% bhimal fiber, Fig. 4.2(g-j) of

epoxy resin with 4 wt% of bhimal fiber, Fig. 4.2(k-l) of epoxy resin with 5 wt% bhimal fiber and Fig. 4.2(m-n) of epoxy resin with 6 wt% bhimal fiber.

Fig. 4.2(a) displays the fractured surface of neat epoxy resin matrix under tensile loading. Fig. 4.2(b) showcases the fractured surface of epoxy resin amalgamated with 2 wt% of bhimal fiber, as we can see from this Fig. a very few bhimal fibers can be viewed on the fractured surface due to a less wt% of fiber present. In addition to this the failure reason is the fiber pull off from the fractured surface, a void formed resulting from the fiber pull off can be easily seen in the Fig. 4.2(c). Fig 4.2 (d-e) shoes the interfacial bonding between the fiber and matrix. Fig. 4.2(f) shows the failure of the material. The failure mechanism is due to the crack growth which leads to splitting of bhimal fiber from the surface. This type of failure is generally seen at lower concentration of fiber having a strong bond with the matrix which establishes with matrix cracking then the fiber and followed up by de-bonding of matrix inter-phases, followed by another fiber failure and so on leading to whole composite failure. Fig. 4.2(g-h) shows the morphology of cracked surface of epoxy resin reinforced with 4 wt% of bhimal fiber and a single strand of bhimal fiber. Further, as the fiber wt% is increased we can see the development of mat (non-uniform) between bhimal fibers (4 wt%) and epoxy resin matrix which in turn resulted in a relatively high strength. In Fig. 4.2 (i-j) an example of unwrapping of bhimal fiber from epoxy resin matrix and finally getting broken can be seen. Similarly in Fig. 4.2 (k-l), as the fiber wt% is further increased to 5 wt%, this resulted in the highest tensile and compressive strength amongst the various compositions tried due to the uniformity of the fiber throughout the matrix and proper load transmission through the fibers. From the Fig.s it can be easily seen that almost all fibers are getting properly broken that means the load is getting transferred successfully .Finally in Fig. 4. 2 (m-n) the distribution of bhimal fiber in cluster form and crack formed is shown. The composite containing 6 wt% of bhimal fiber showed the lower tensile strength because of low bonding between fiber and matrix. Density of bhimal fiber is approximately 0.453 gm/cm^3 due to this even in 6 wt% of fiber, it takes a large volume of matrix so the improper bonding between fiber and matrix occurs due to which material gets failed even at low loading condition. Also such a high fiber percentage leads to formation of clusters of fiber that deteriorates the strength. Therefore, the SEM photographs clearly correlate with the mechanical properties in this investigation.

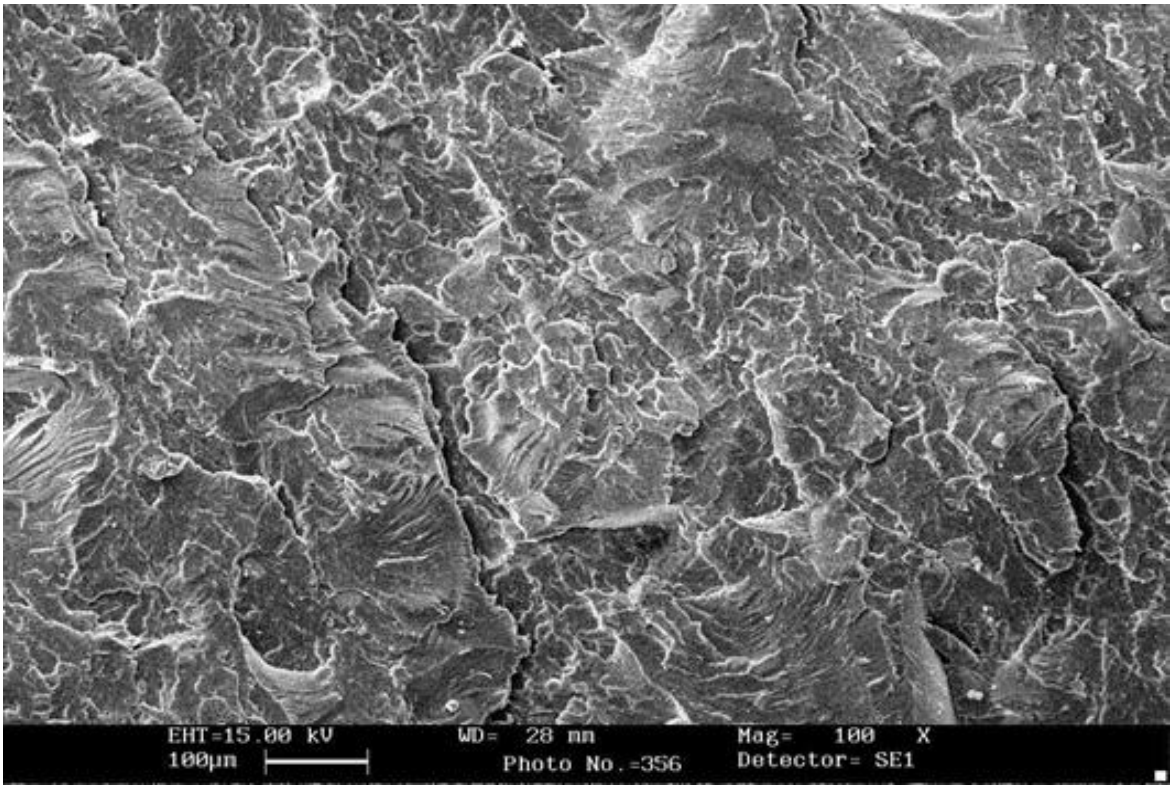

Fig. 4.2 (a): Scanning electron micrograph for fractured surface of Epoxy resin (CY-230 + 9 wt% HY-951)

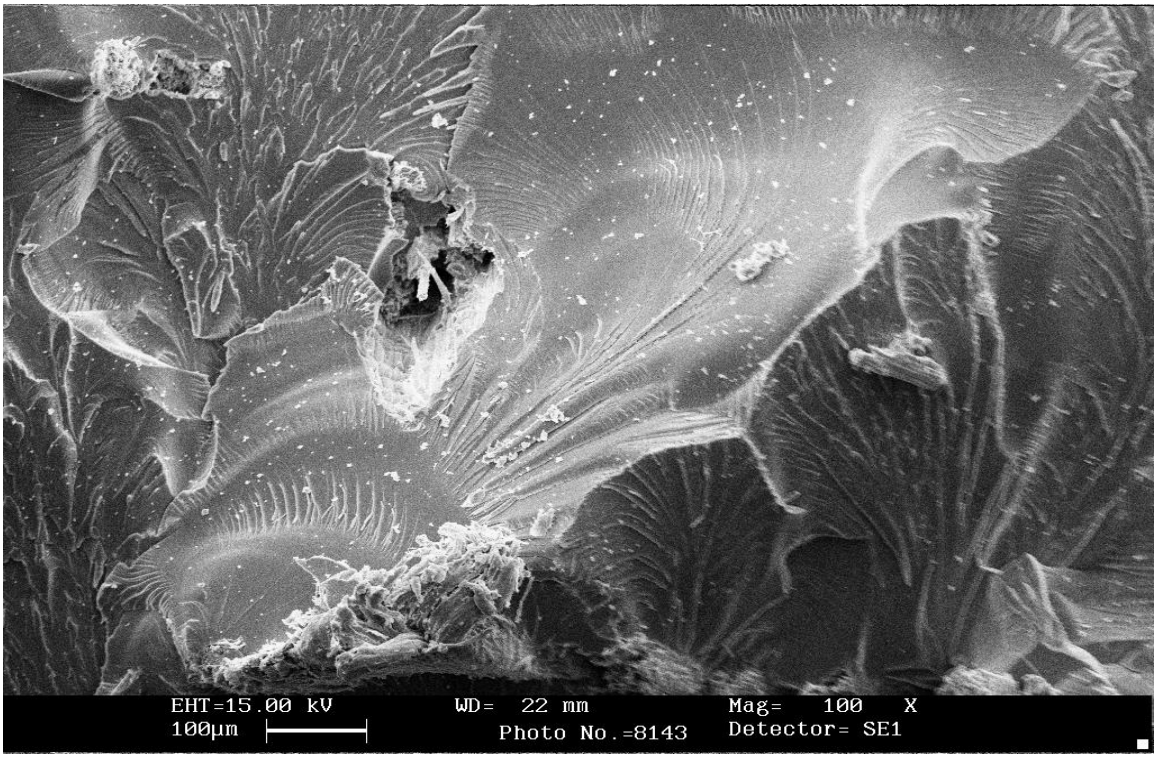

Fig. 4.2 (b): Scanning electron micrograph showing the fractured surface for Epoxy resin and 2 wt% of bhimal fiber

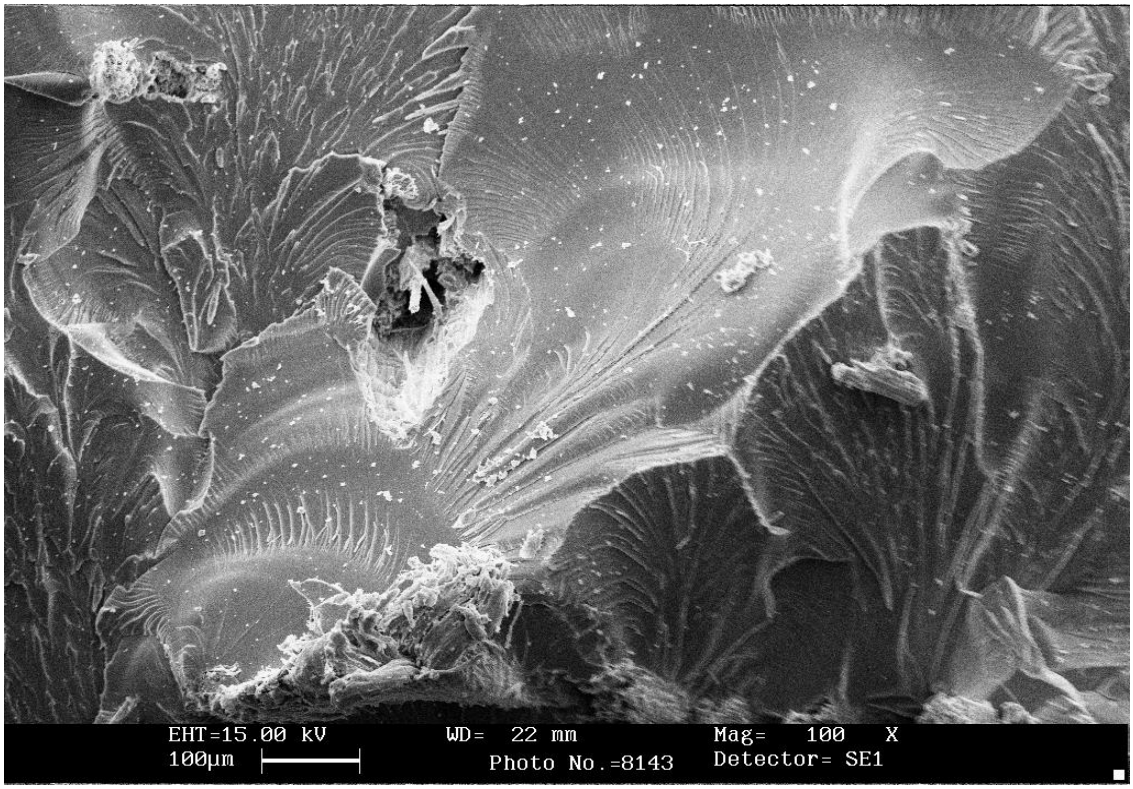

Fig. 4.2 (c): Scanning electron micrograph for 2 wt% bhimal fiber in the Epoxy resin showing fracture due to fiber pull off

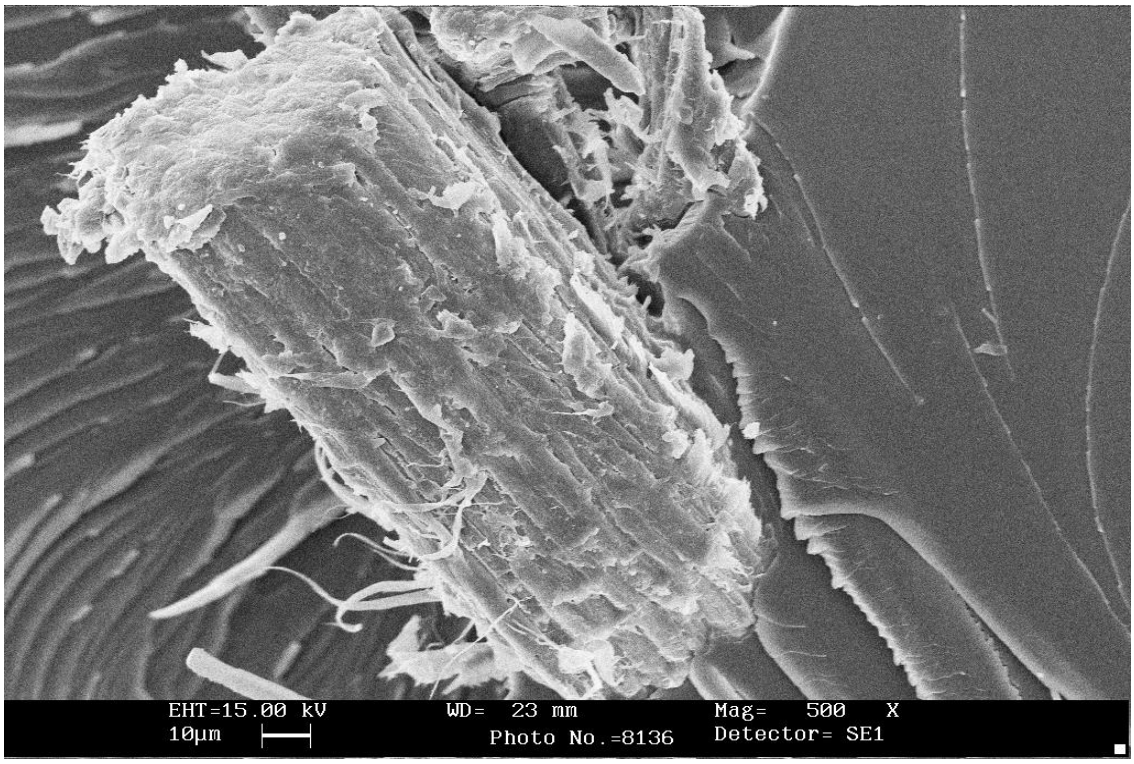

Fig. 4.2 (d): Scanning electron micrograph showing bonding between bhimal fiber (3%) and epoxy resin at 500X

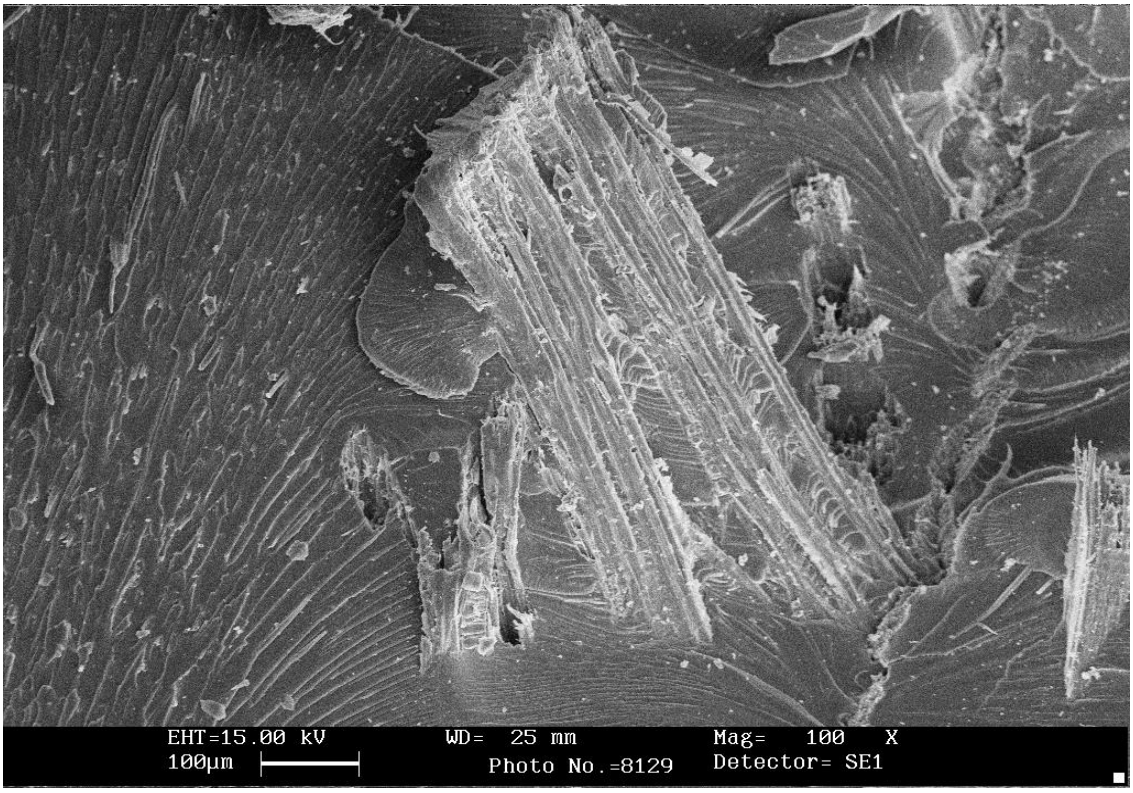

Fig. 4.2 (e): Scanning electron micrograph for showing the interfacial bonding between bhimal fiber and Epoxy resin

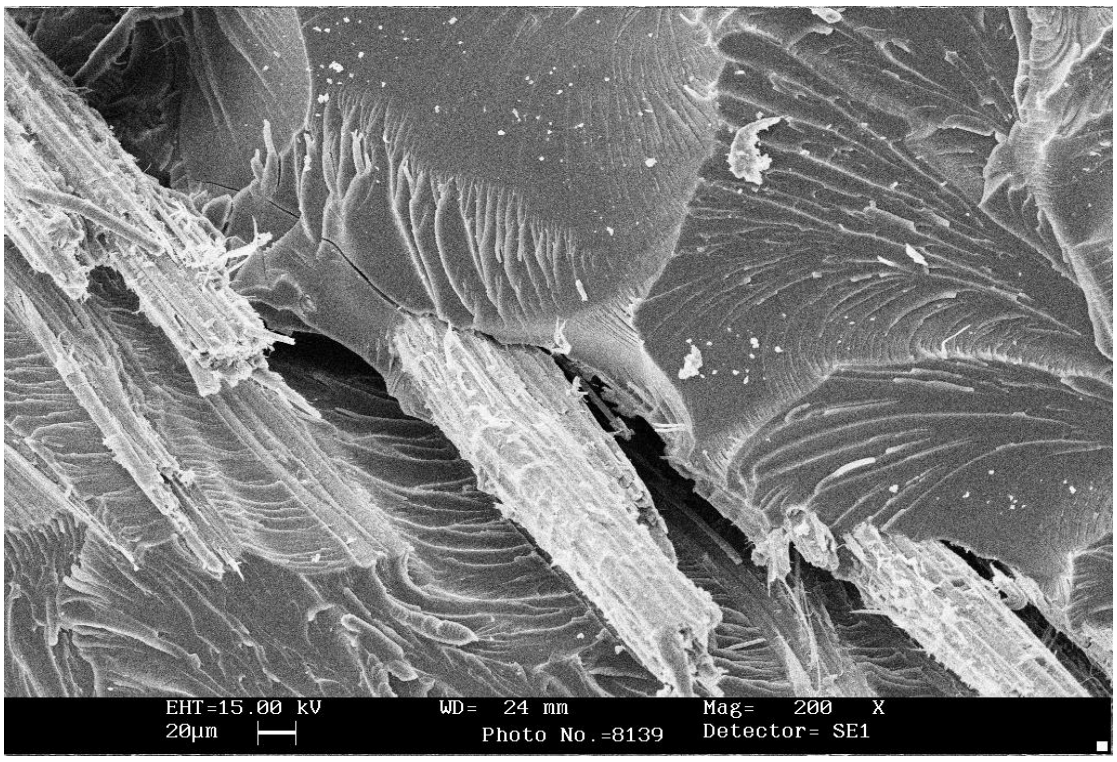

Fig. 4.2 (f): Scanning electron micrograph displaying crack formation through bhimal fiber (3 %) in the Epoxy resin

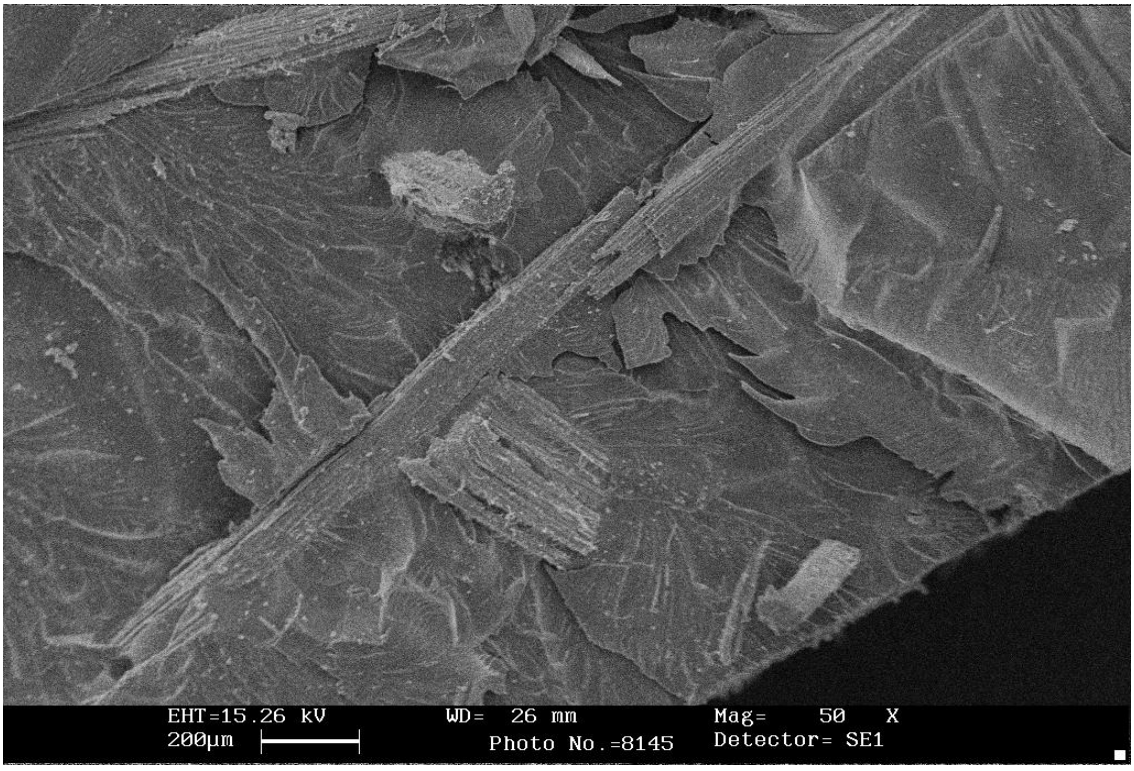

Fig. 4.2 (g): Scanning electron micrograph showing the fractured surface for Epoxy resin reinforced with 4 wt% of bhimal fiber

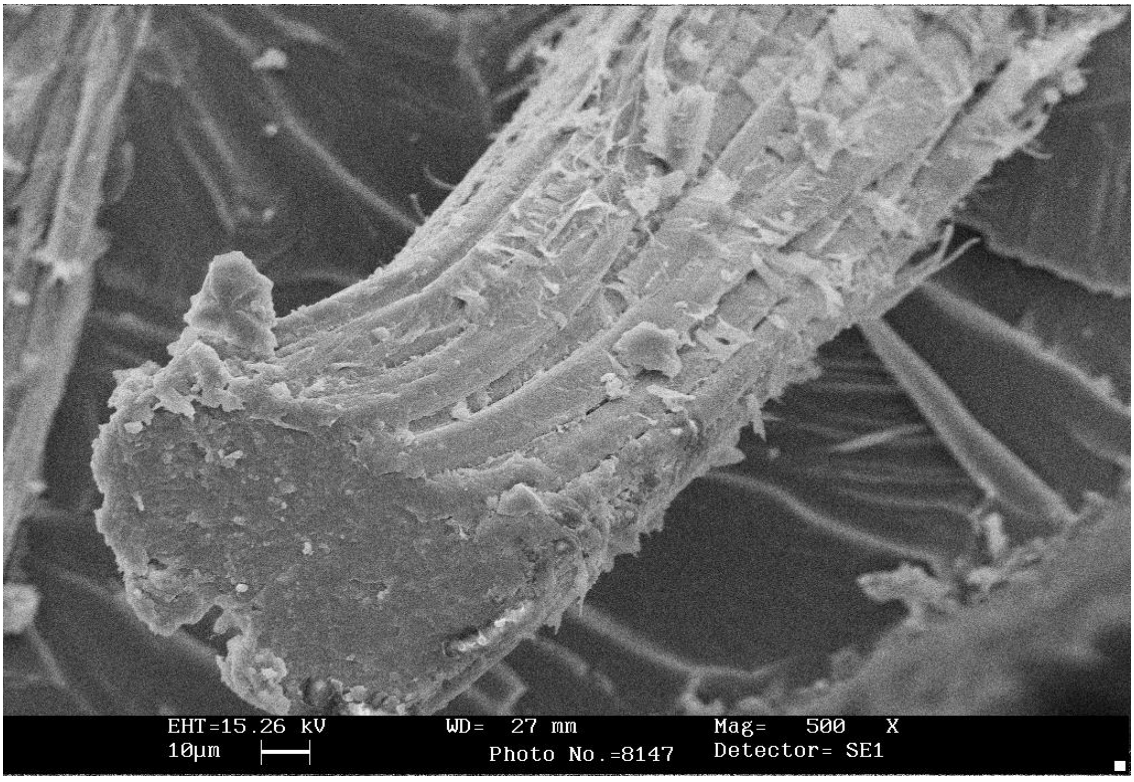

Fig. 4.2 (h): Scanning electron micrograph showing the single strand of bhimal fiber reinforced in epoxy resin

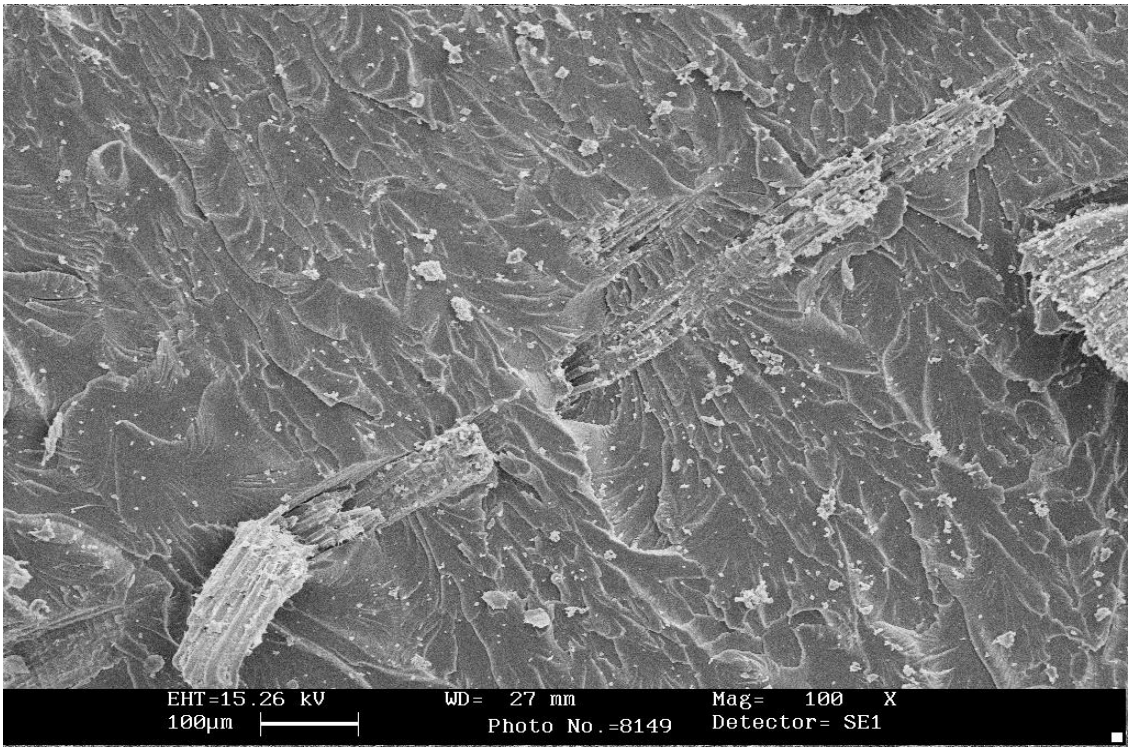

Fig. 4.2 (i): Scanning electron micrograph showing the impression formed due to unwrapping of fiber and breakage of bhimal fiber (4%)

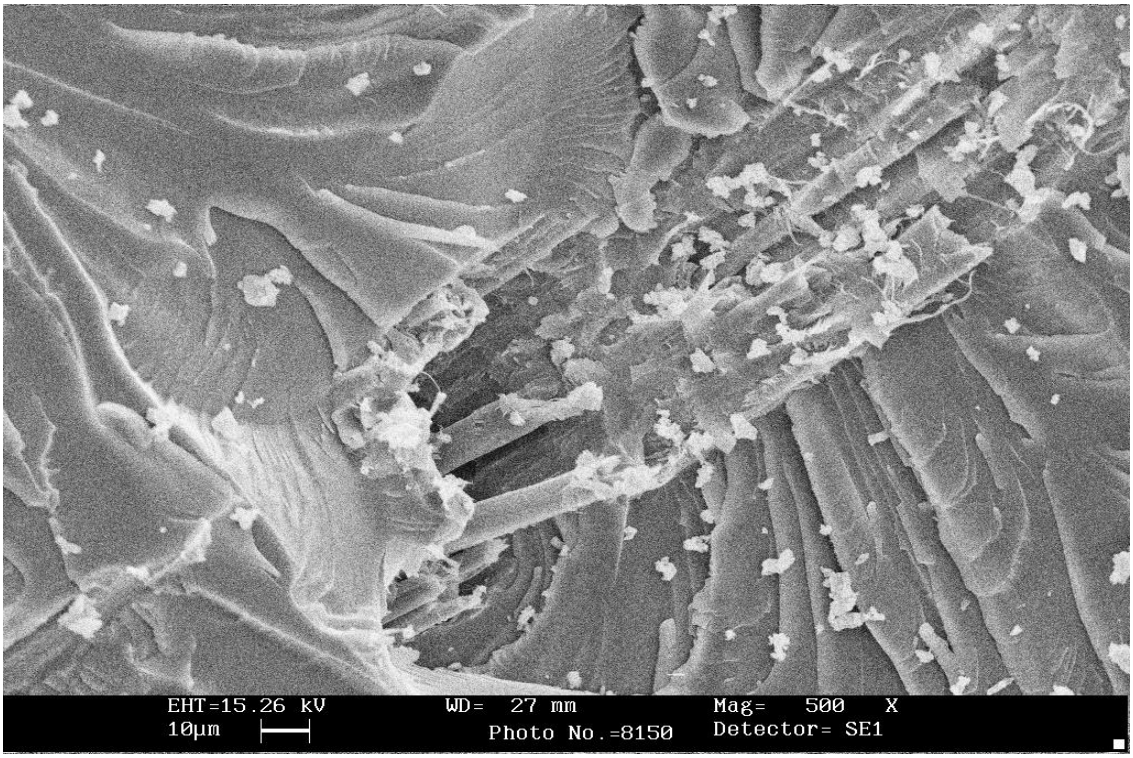

Fig. 4.2 (j): Scanning electron micrograph showing the magnified view of fiber breakage in bhimal fiber (4%) reinforced epoxy resin

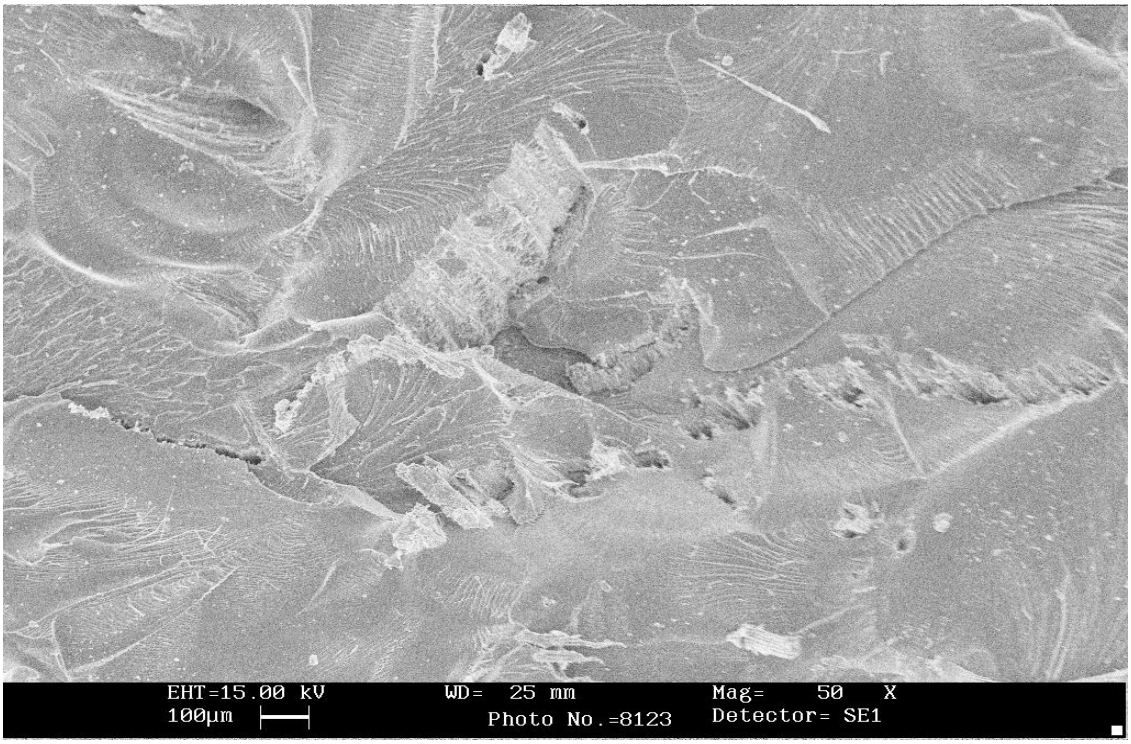

Fig. 4.2 (k): Scanning electron micrograph showing the breakage of fibers in Epoxy resin reinforced with 5 wt% of bhimal fiber

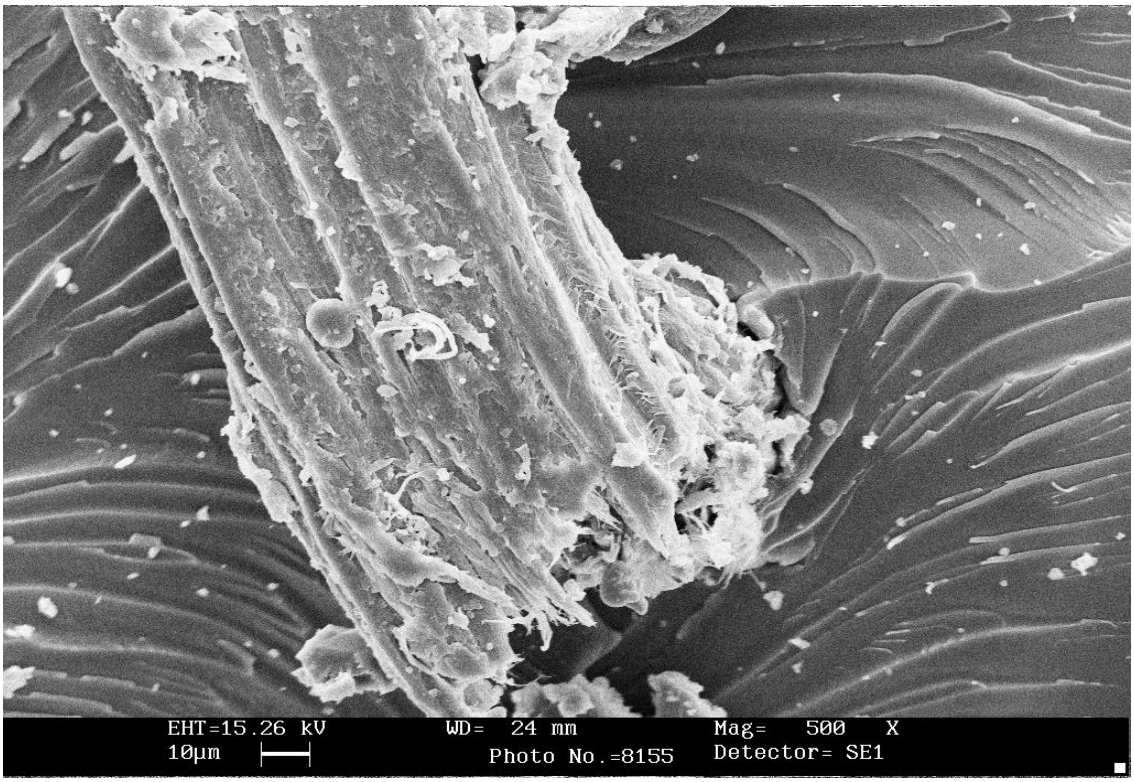

Fig. 4.2 (l): Scanning electron micrograph showing the magnified view of fiber breakage in bhimal fiber (5%) reinforced epoxy resin

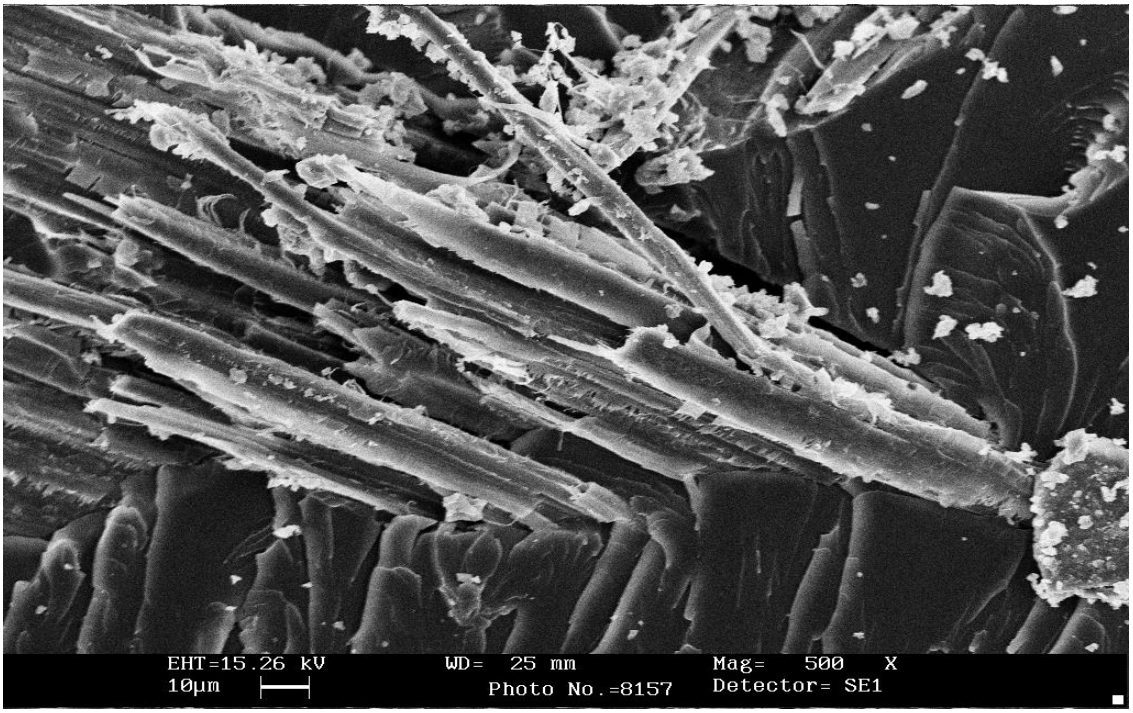

Fig. 4.2 (m): Scanning electron micrograph showing the distribution of fibers in form of clusters in bhimal fiber(6%) reinforced epoxy resin

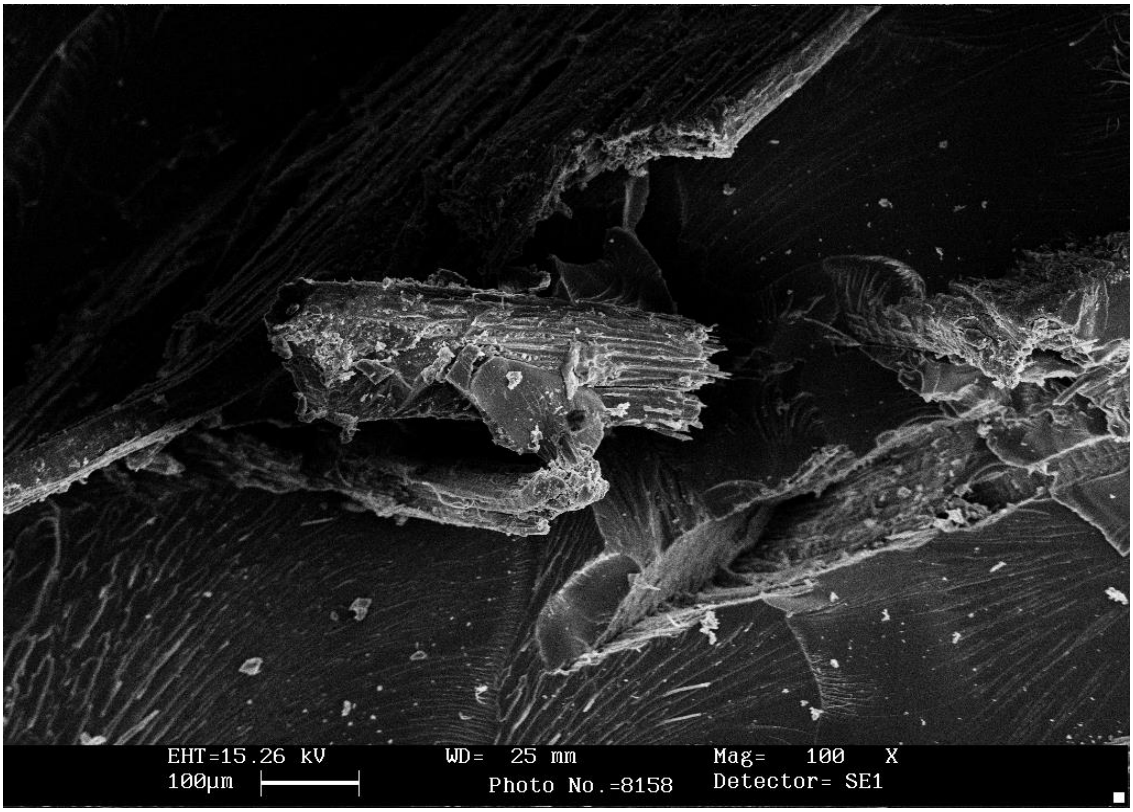

Fig. 4.2 (n): Scanning electron micrograph showing the crack formed in bhimal fiber (6%) reinforced epoxy resin

4.3 Results of Mechanical Tests

The diverse compositions of the composite material were mechanically tested and the values of mechanical properties were determined accordingly.

4.3.1 Tensile test

Tensile testing of composites has been performed on 25 KN servo hydraulic universal testing machine (Tinius Olson Tensometer) at a fixed strain rate 1 mm/min under the displacement control mode. The tensile tests were conducted for different compositions and the resultant graphs obtained are shown in the Fig. 4.3 (a & b) and values are provided in the table 4.3.

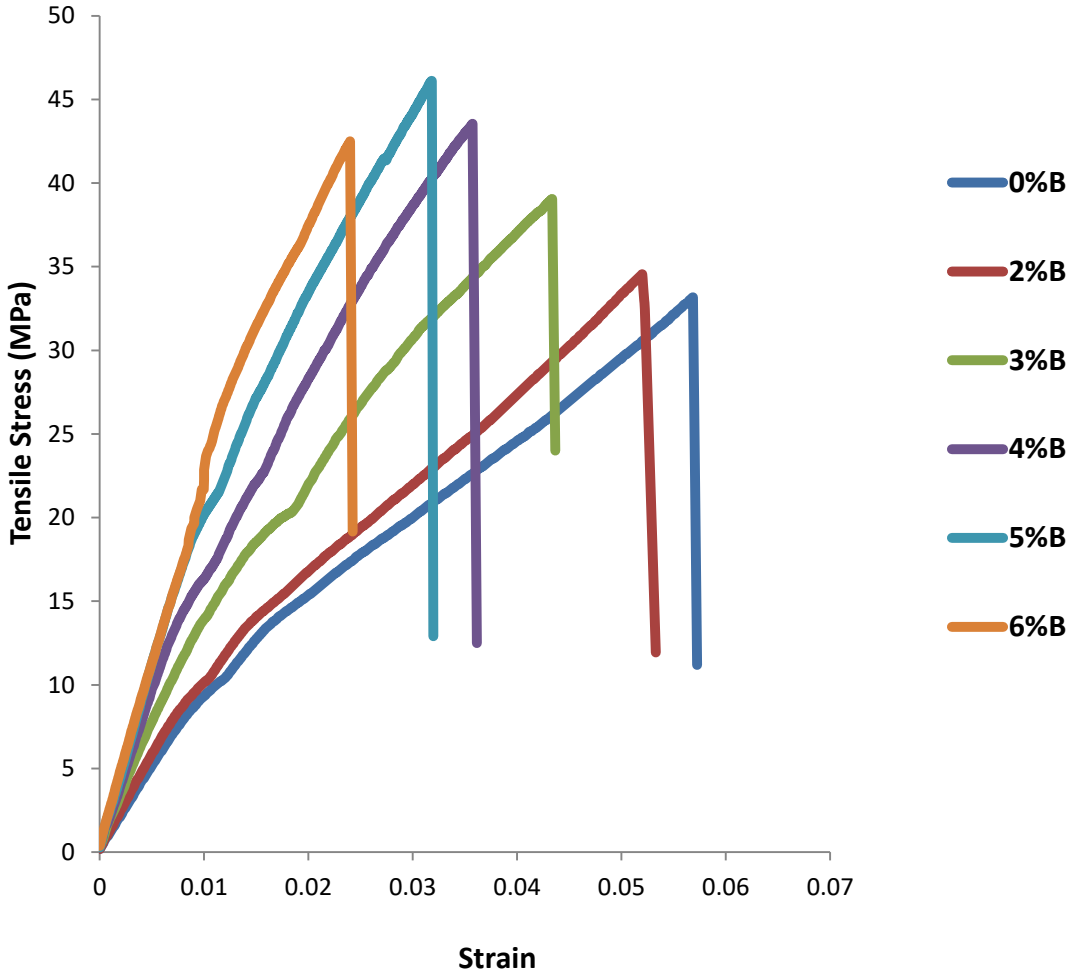

Fig. 4.3 (a): Tensile Stress-Strain Diagram for various compositions

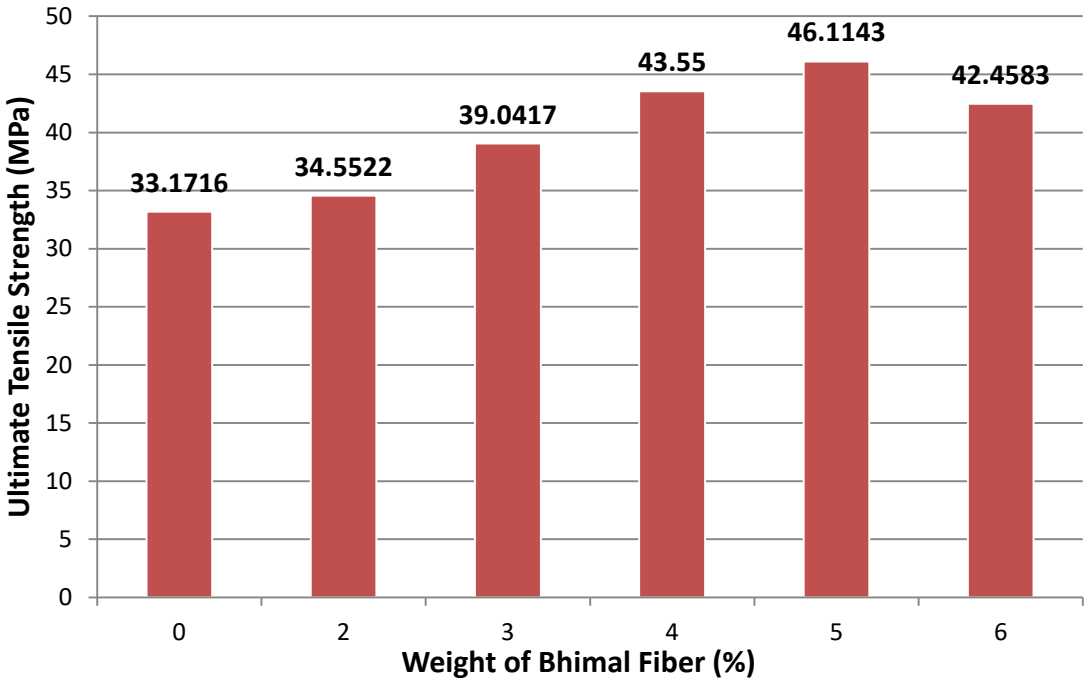

Fig. 4.3 (b): Effect of fiber weight % on Ultimate Tensile Strength

Table 4.2: Tensile properties for various compositions of epoxy resin - bhimal fiber based composite

Weight % of bhimal fiber	Ultimate tensile strength (MPa)	Strain
0	33.17	0.057
2	34.55	0.053
3	39.04	0.043
4	43.55	0.036
5	46.11	0.032
6	42.45	0.024

From the graph, remarkable differences can be seen in the magnitude of ultimate tensile strength. It can be observed from the results that for all specimens under investigation, the ultimate tensile strength is highest for the composition (Epoxy resin + Hardener + 5 wt%

bhimal fiber) which is approximately 46.11MPa. If we see Fig. 4.3(b), it can be clearly seen that with the increase in fiber wt% in the matrix there is an increase and then decrease in the tensile strength trend.

The foremost reasons behind this trend are listed below:

- At 2-4 wt% of bhimal fiber, the load distribution was almost equal between the matrix and fiber, thus leading to a moderate increase in the tensile strength as compared to the neat epoxy resin matrix tensile strength.
- At 5 wt% of bhimal fiber, a higher magnitude of load was transferred to the fiber i.e. the contribution of fiber increased that lead to increase in the tensile strength. This was supported by the occurrence of fiber breakage (Fig. 4.2(j)). As already mentioned, the tensile strength value of bhimal fiber is very high.
- At 6 weight% of bhimal fiber, as the fiber wt% increases the tensile strength started deteriorating because of the lack of bonding between the fiber and matrix because fibers get accumulated in some areas and epoxy resin in the remaining areas (Fig. 4.2(m)).

All the above mentioned reasons are fully validated by the SEM photographs as mentioned. When fibers are treated with KOH (alkali), it reacts with the surface of fiber, thus breaking the hydrogen bond at the surface and removing all the impurities leaving the surface rough. As a result, strong bonding occurs between the matrix and fiber therefore to rupture the bonding higher stresses are to be applied.

4.3.2 Compression test

The compressive properties of the fabricated composite material were determined by the 25 kN servo controlled universal testing machine (Tinius Olson Tens meter) at a fixed test speed 1 mm/min under displacement control mode. The compressive stress-strain curve is shown in the Fig. 4.3(c). It can be examined from the outcome that from all the samples, the ultimate compressive strength is highest for the composition i.e. Epoxy resin + Hardener + 5 wt% bhimal fiber which is approximated to be 127.69 MPa. This compressive strength is about 1.57 times the ultimate compressive strength of the neat epoxy resin. If we see Fig. 4.3(d), it can be clearly viewed that with the increase in fiber wt% there is an increase and then decrease in the compressive strength trend. The

magnitudes to support the above statement are provided in the table 4.2. The basis for the above described trend is similar to that of the tensile test.

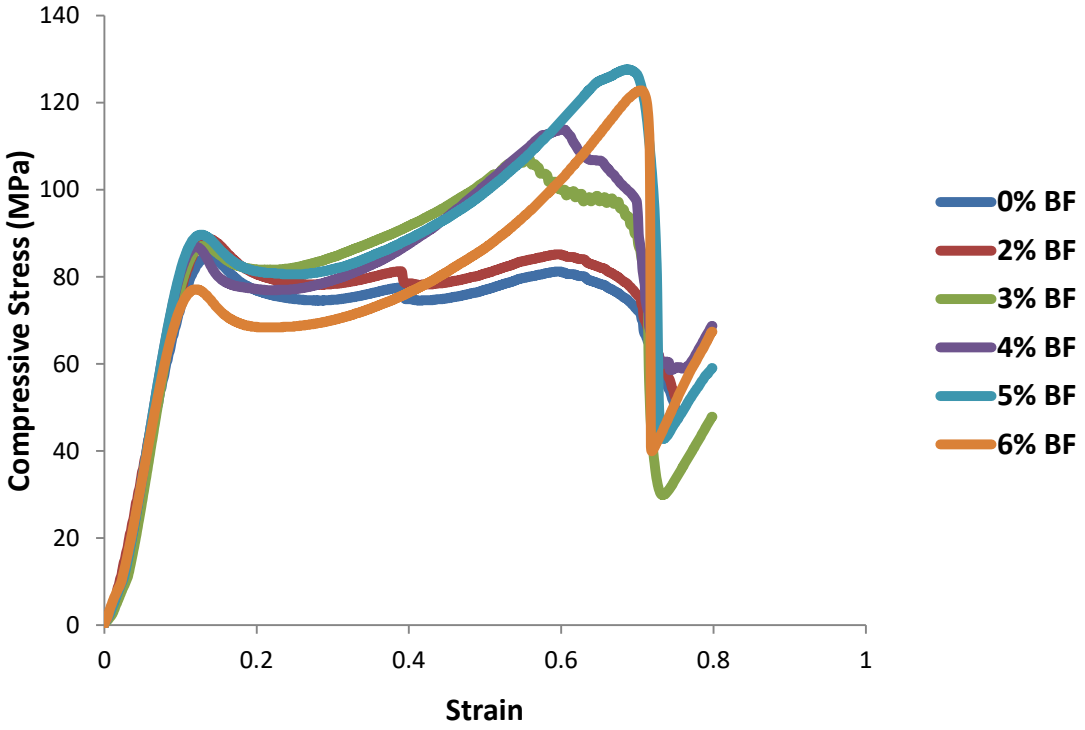

Fig. 4.3 (c): Compression Stress-Strain Diagram for various compositions

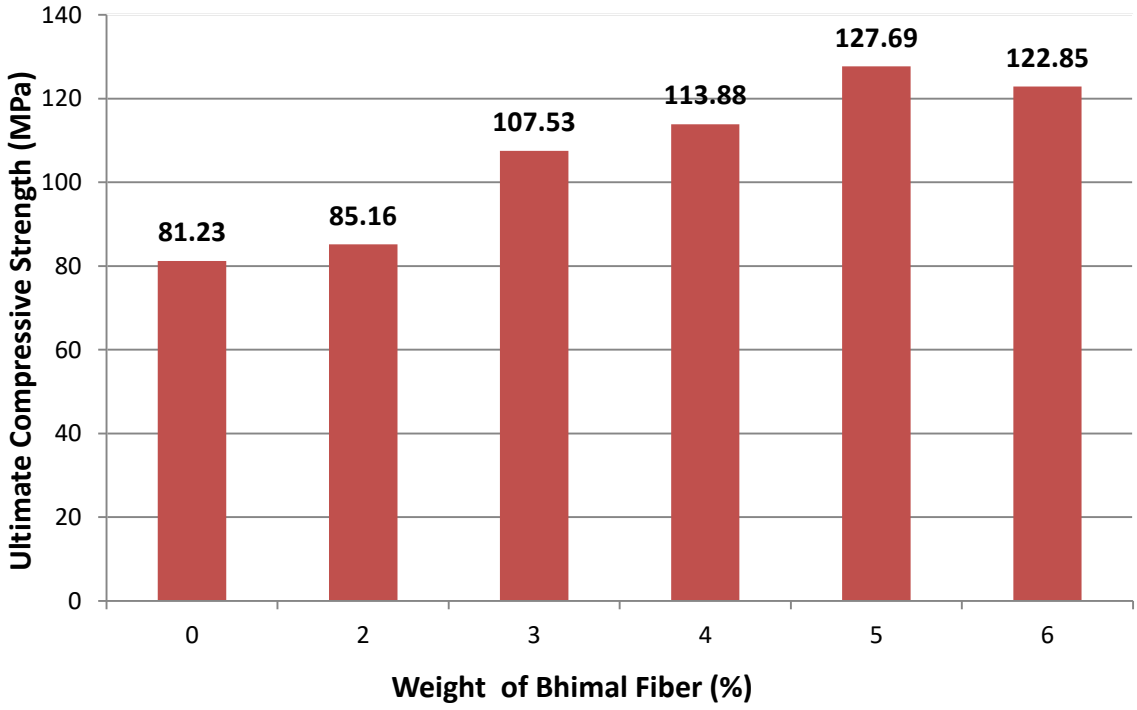

Fig. 4.3 (d): Effect of fiber wt% on Ultimate Compressive Strength

Table 4.3: Tensile properties for various compositions of epoxy resin - bhimal fiber composite

Weight % of bhimal fiber	Ultimate compressive strength (MPa)	Strain
0	81.23	0.798
2	85.16	0.798
3	107.53	0.798
4	113.88	0.798
5	127.69	0.798
6	122.85	0.798

The key reason for having more compressive strength than the tensile strength is contributed to the internal flaws that are present inside the composite. The crack propagation is much rapid from these internal flaws under the tensile load but the behavior is just opposite in the case of compressive load. In fact, the compressive load leads to the shutting of crack thus concluding a higher compressive strength.

4.3.3 Flexural test

Flexural strength/modulus of rupture/bend strength is a mechanical factor which is defined as the stress experienced in a material before it tries to yield in a flexure test. The most frequently used transverse bending test employs a specimen having either a rectangular or circular cross-section which is bent further until the fracture or yielding occurs generally using a three point flexural test method. The flexural properties play a very vital role in the structural applications. The flexural stress, flexural modulus and flexural strain are computed by the equations (3.5), (3.6) and (3.7) respectively. The flexural tests were done for diverse compositions and the experimental graphs obtained are shown in the Fig. 4.3(e-f), while the corresponding values are provided in the table 4.3. From the Fig. 4.3(e), it can be observed that for all the samples under investigation,

the flexural strength is highest for the composition having 5wt% of fiber reinforcement having an approximate value of 93.022 MPa which is approximately 1.518 times that of the neat epoxy resin matrix.

Table 4.4: Tensile properties for various compositions of Epoxy resin – bhimal fiber based composite

Weight % of bhimal fiber	Flexural strength (MPa)	Flexural strain	Flexural modulus (GPa)
0	61.25	0.0321	1.902
2	66.294	0.0309	2.139
3	75.254	0.0298	2.563
4	85.725	0.0288	2.982
5	93.022	0.0283	3.428
6	86.955	0.0293	2.986

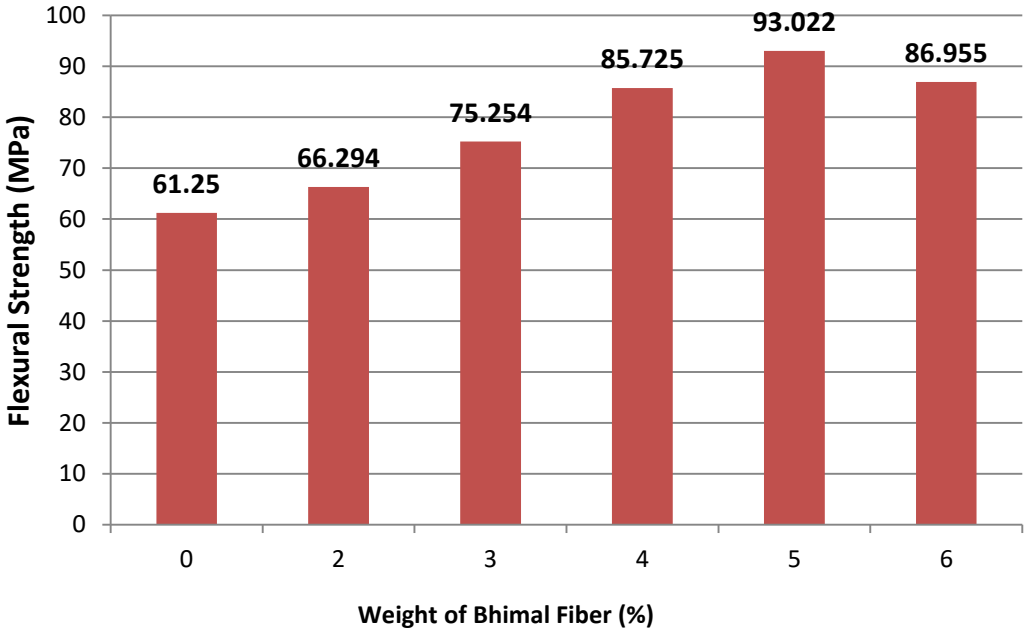

Fig. 4.3 (e): Effect of fiber weight% on flexural strength

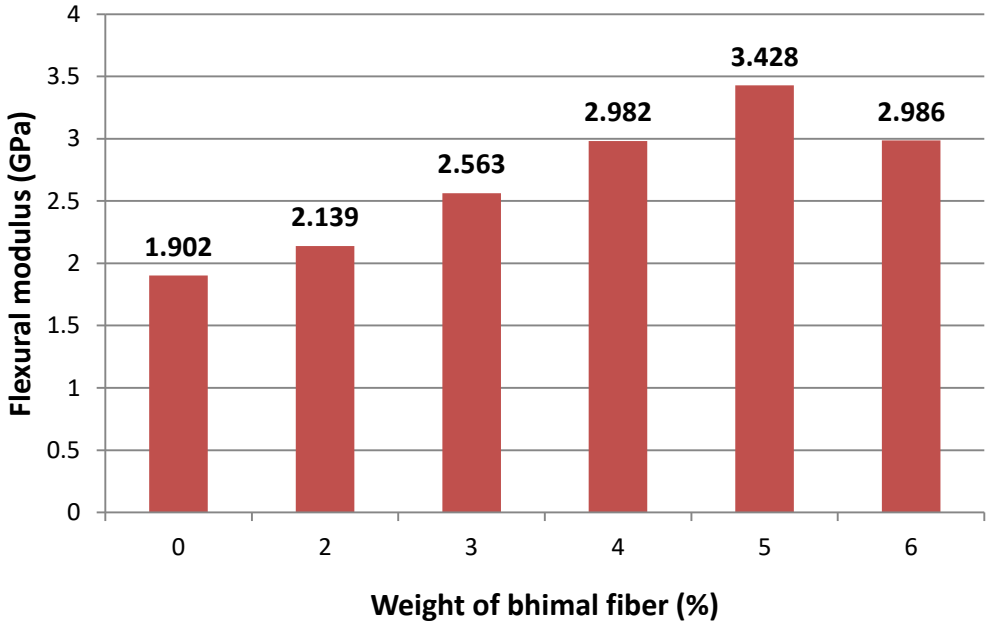

Fig. 4.3 (f): Effect of fiber weight% on flexural modulus

Similarly, from Fig. 4.3(f) the variation between flexural modulus and wt% of bhimal fiber can be viewed. The flexural modulus is found to be maximum for the composition having bhimal fiber 5% by weight with a magnitude of 3.428 GPa. From both the graphs, the trend seen is that firstly the value increases and after attaining a maximum at 5 wt% it starts decreasing. The explanation for the above depicted trend is quite similar to that of the tension and compression test.

4.3.4 Hardness test

The resistance to plastic deformation is called Hardness. It is also a surface property, if the surfaces have voids, cuts or any other deformity the hardness will be low for such material. In composite material science, the reinforcing element weight fraction significantly affects the hardness magnitude. In this scrutiny, the hardness test has been carried out on the L scale with the help of a Digital Rockwell hardness testing machine. The hardness tests were done for various compositions and the experimental graphs thus obtained are shown in the Fig. 4.3(g) and corresponding values are provided in the table 4.5.

Table 4.5: Rockwell hardness values on L scale for a range of compositions

Weight % of bhimal fiber	Hardness values (HRL)			Average hardness value
	Trial 1	Trial 2	Trial 3	
0	125	127	129	127
2	132	135	136	134.33
3	136	139	141	138.66
4	137	140	145	140.66
5	135	136	138	136.33
6	135	134	135	134.66

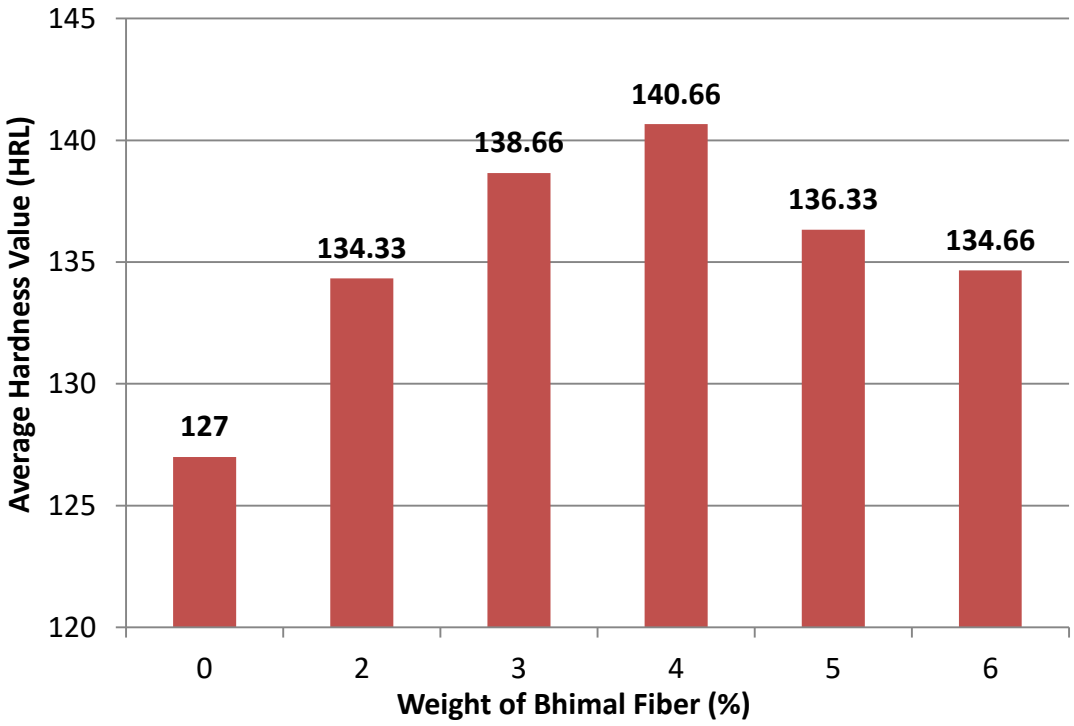

Fig. 4.3(g): Effect of fiber wt% on rockwell hardness values

As seen from the Fig. 4.3(g) and table 4.5, the hardness value increases upto 4 wt% and after that it starts decreasing. The maximum value attained is 140.66 which is approximately 1.1 times greater than that of the neat epoxy resin. The chief reason behind this behaviour is that till 4 wt% the fiber was able to bear the indentation load but after this composition due to the presence of epoxy as matrix was not good enough to encapsulate the bhimal fibres. Some fibres were left uncovered and physical bonding was only between fibre strands. The stress transferred due to load bearing was hindered and weaken the interfacial adhesion between the matrix and the fibres in the bio-composites. As a result, agglomeration occurred due to these fibre to fibre bondings. This resulted in discontinuities in the matrix, producing stress concentration points in the composite. Hence it started giving lower values of hardness at higher wt% of bhimal fiber.

4.3.5 Impact test

Impact strength is the material ability to resist fracture under the stress applied at a high speed or generally shock loads. The outcomes obtained by the charpy impact tests are tabulated in the table 4.6. The impact strength is calculated by the formula given in the equation (3.8). Fig. 4.3(h) and 4.3(i) give you an idea about the impact energy and impact strength variation with various compositions of the composite.

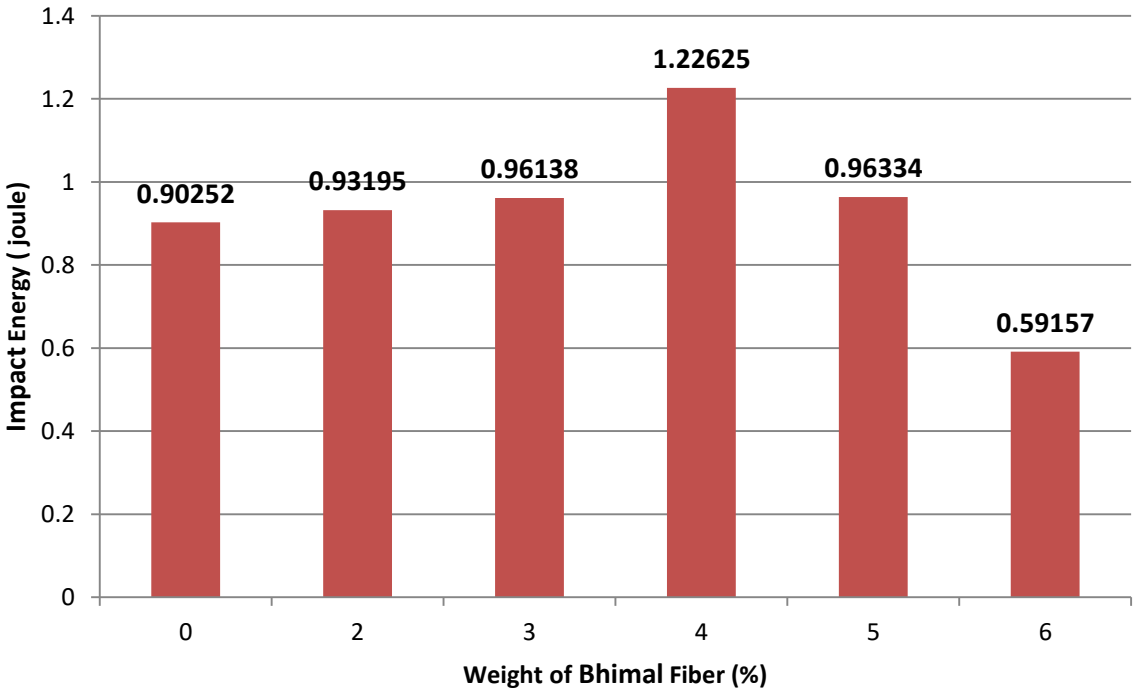

Fig. 4.3 (h): Effect of fiber wt% on Impact energy

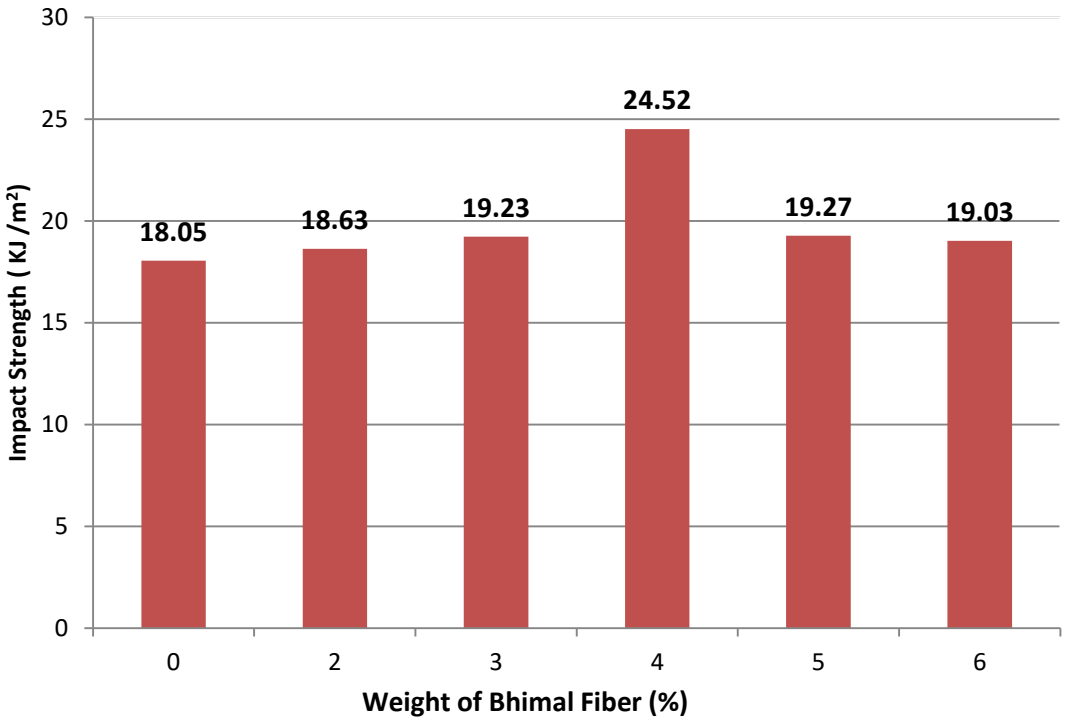

Fig. 4.3 (i): Effect of fiber wt% on Impact strength

Table 4.6: Impact test parameters for various fabricated compositions

Weight % of bhimal fiber	Impact energy (Kgm)	Impact energy (Joule)	Impact strength (KJ/m^2)
0	0.092	0.9025	18.05
2	0.095	0.9319	18.62
3	0.098	0.9614	19.22
4	0.125	1.2262	24.52
5	0.098	0.9633	19.26
6	0.097	0.9515	19.03

As seen from the graphs, the impact energy and impact strength is increasing as the wt% of bhimal fiber increases attaining a maximum magnitude of 1.2262 J and 24.52 KJ/m^2 respectively. The ultimate impact strength is about 1.35 times the value for neat

epoxy resin matrix. The reason behind this increase is that at higher fiber concentration the formation of mat (cross linking phenomenon) occurs which takes the load first and after that transfers it to the matrix. In addition, the bhimal fiber is quite flexible in nature. Hence, when we keep on increasing the fiber weight percentage the fiber keeps on absorbing the energy within it which results in monotonically increase of impact energy and strength.

As already seen, the fabricated composite is having higher tensile, compressive, impact and also the impact strength than the neat epoxy resin matrix so it would be more interesting to note the thermal analysis of the various compositions of composite.

4.4 Results of Thermal Tests

The samples thermal analyses were performed at the Institute Instrumentation Centre, Indian Institute of Technology, Roorkee. For the thermal analysis to be carried out satisfactorily, the specimens were initially converted into the powder form by grinding process. The reference base substance taken was alumina powder with a weight of 10.250 mg, which was thoroughly mixed with the given composite samples in equal proportions. The instrument used in this process is EXSTAR TG/DTA 6300 having the medium in which the tests were conducted as air flowing at a rate of 200 ml/min. The experimental rate of change of temperature was taken to be 10°C/min and the range of temperature was from 35 - 800 ^{0}C. The upcoming graphs demonstrate the thermal variation through DTA, DTG and TG curves separately. From the following results we can see that as the fiber weight percentage is increased, the more residue percentage of initial weight is obtained so we can conclude that upto fiber percent of 5% thermal stability increases and then starts decreasing.

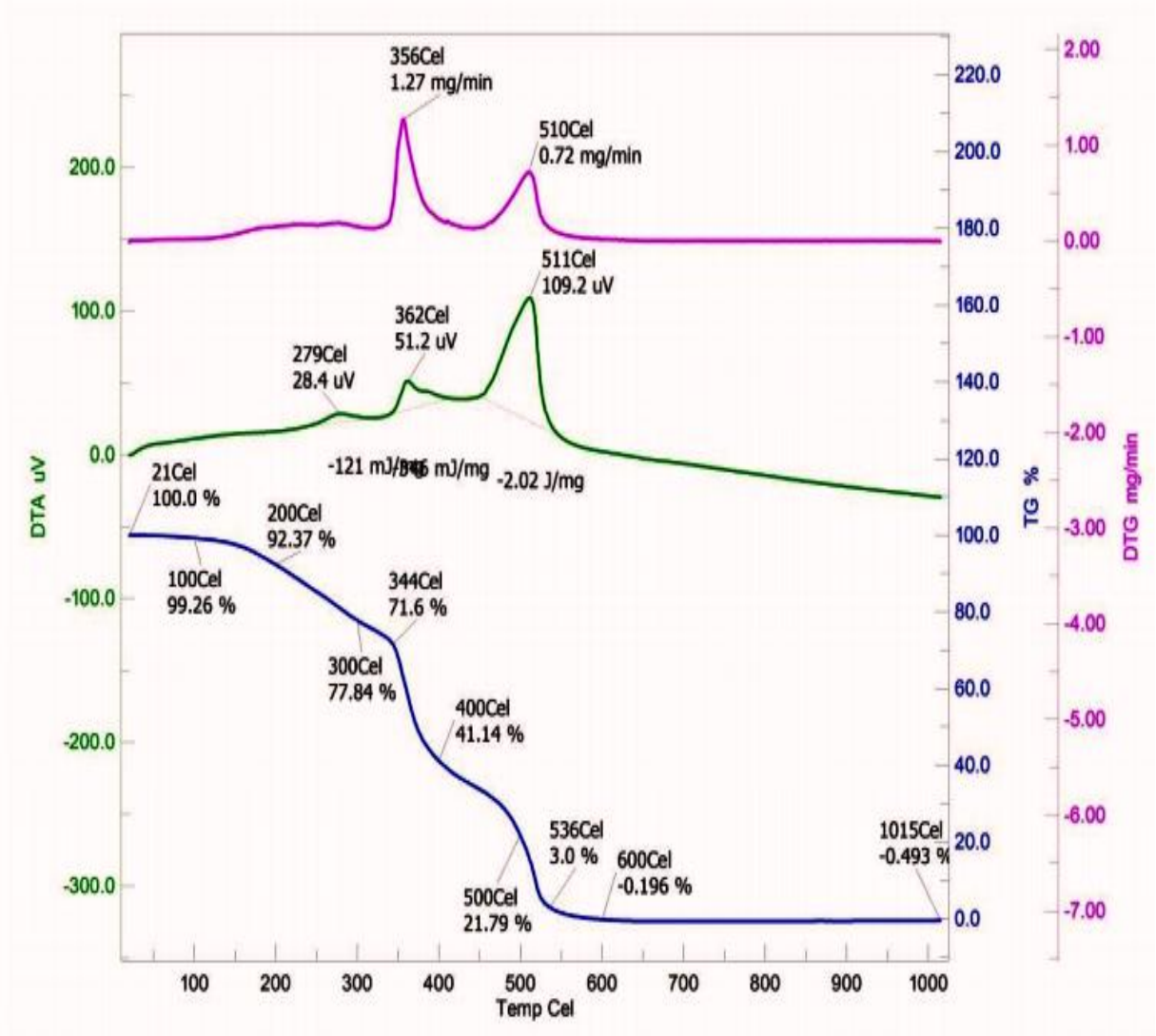

Fig. 4.4 (*a*): Thermogram of neat epoxy resin

Fig. 4.4(*a*) showcases the thermo gram of neat epoxy resin having 9 wt% hardener blended in it. According to the DTG curve, the decay of this composite has been acquired under two phases that are ranging between 356^0C to 510^0C having the rate of decomposition 1.27 mg/min to 0.72 mg/min respectively. Before 200^0C, the weight loss of 7.63% may be accredited to the ejection of moisture, low molecular mass molecules and volatile stuff linked with the composite as illustrated in the TG curve. The utmost rate of decomposition of 1.27 mg/min has been observed at 356^0C. Such decomposition has been sustained with a fusion heat of -2.487 J/mg centered in the temperature range of 279^0C to 511^0C with a corresponding DTA signal of 28.4 µV to 109.2 µV. The decomposition of the composite had been accomplished at 536^0C leaving a char residue 3% of the initial weight.

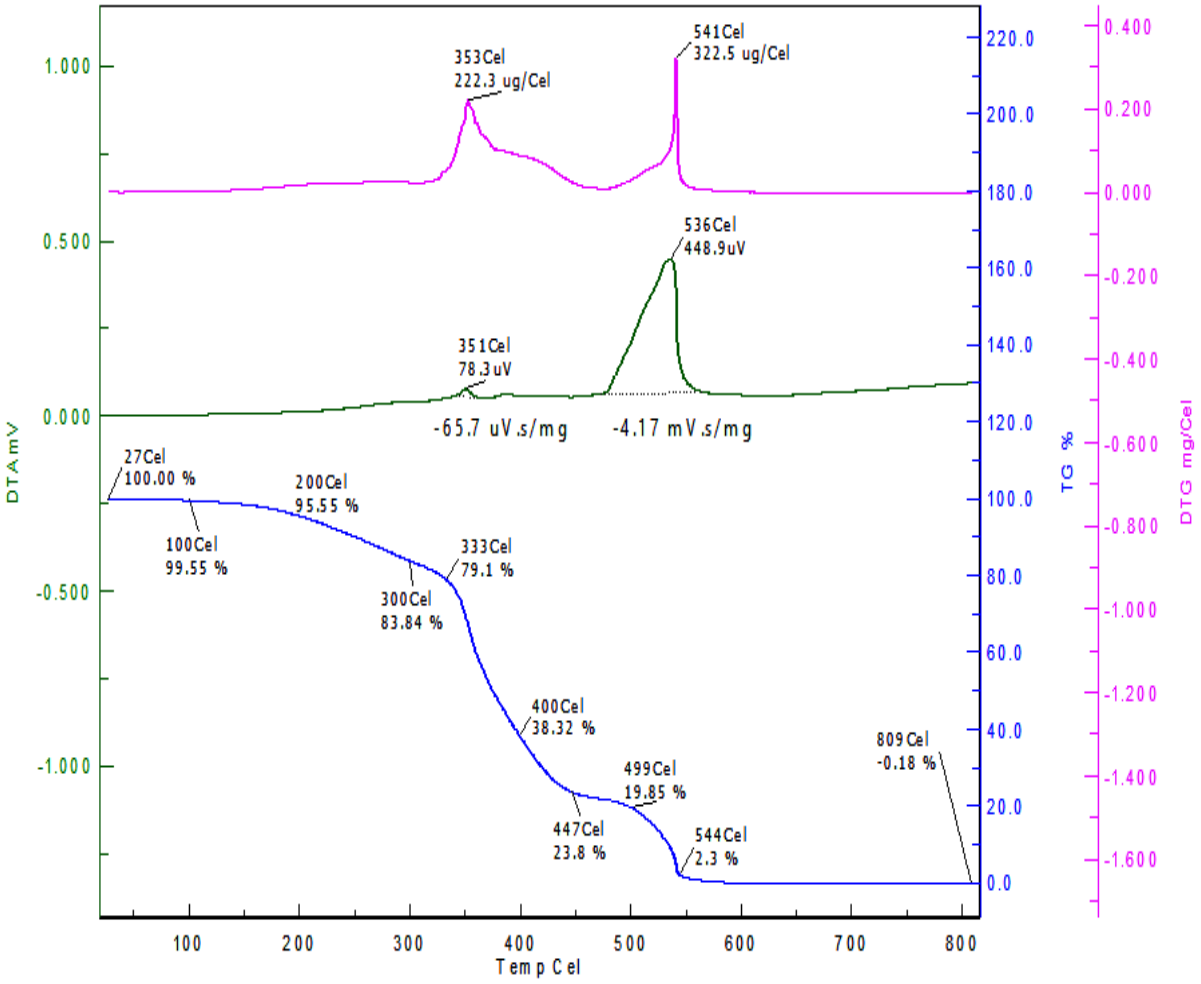

Fig. 4.4 (b): Thermal analysis of epoxy resin with 2% bhimal fiber

Fig. 4.4(b) displays the thermo gram of epoxy resin having 2 wt% of bhimal fiber and 9% of hardener merged in it. Here the initial sample weight is taken to be 20.200 mg. Through the DTG curve analysis, we can conclude that the decay of this composition has been accomplished under two stages that range between 353^0C to 541^0C with the rate of decomposition 222.3ug/cel to 322.33ug/cel respectively. Prior to 200^0C, the weight loss of 4.45% may be credited to the expulsion of moisture, low molecular mass molecules and volatile objects associated with the composite as illustrated by the TG curve. The extreme rate of decomposition of 322.5 ug/cel has been observed at 541^0C. Such disintegration has been sustained with the fusion heat of -108 uV.s/mg at 354^0C and -4.11 mV.s/mg at 513^0C accompanied by a DTA signal of 0.089 mV and 0.51 mV respectively. The decomposition of the composite material had been concluded at 809^0C leaving a char residue 0.10% of the initial weight.

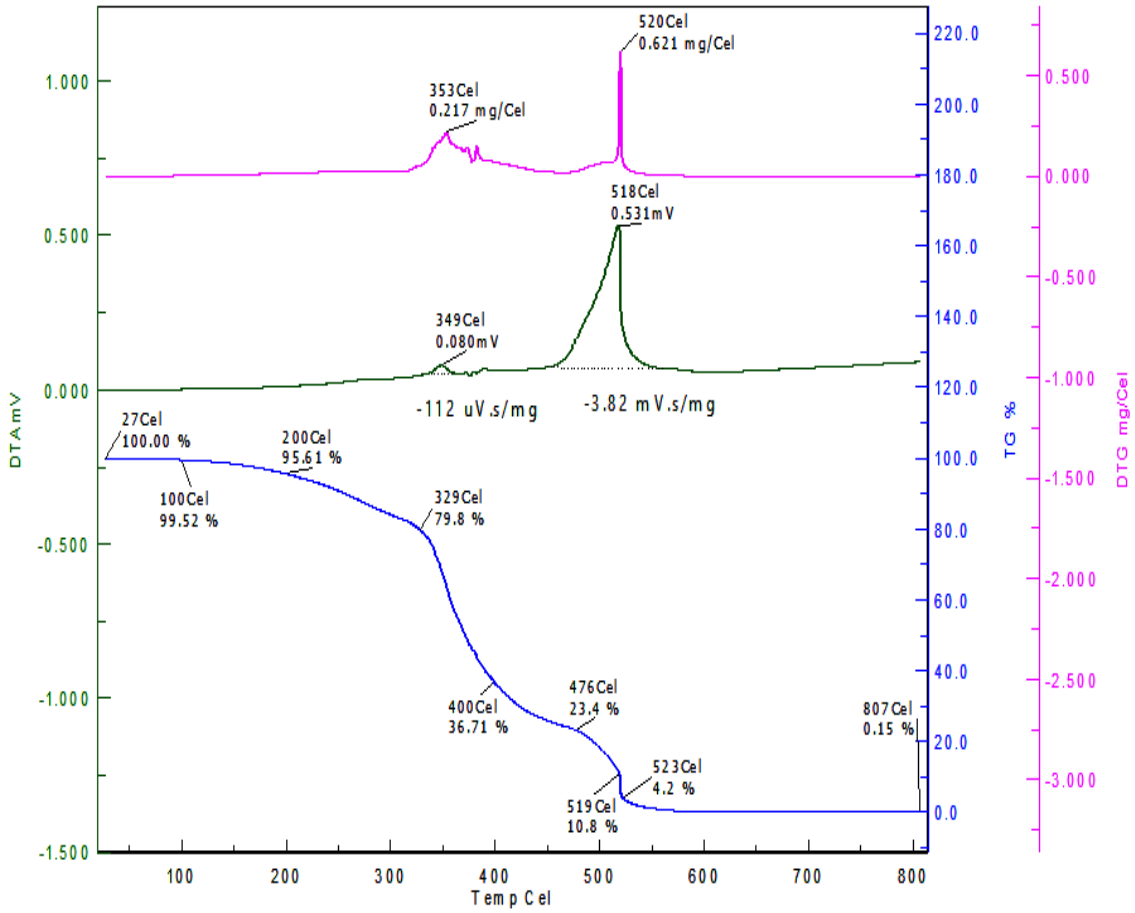

Fig. 4.4 (c): Thermal analysis of Epoxy resin with 3% bhimal fiber

Fig. 4.4(e) displays the thermo gram of epoxy resin having 3 wt% of bhimal fiber and 9% of hardener merged in it. Here the initial sample weight is taken to be 20.570 mg. Through the DTG curve analysis, we can conclude that the decay of this composition has been accomplished under two stages that range between 353^{0}C to 520^{0}C with the rate of decomposition 0.217 ug/cel to 0.621 ug/cel respectively. Prior to 200^{0}C, the weight loss of 4.39% may be credited to the expulsion of moisture, low molecular mass molecules and volatile objects associated with the composite as illustrated by the TG curve. The extreme rate of decomposition of 0.621 mg/cel has been observed at 520^{0}C. Such disintegration has been sustained with the fusion heat of -112 uV.s/mg at 349^{0}C and -3.82 mV.s/mg at 518^{0}C accompanied by a DTA signal of 0.08 mV and 0.531 mV respectively. The decomposition of the composite material had been concluded at 807°C leaving a char residue 0.15% of the initial weight.

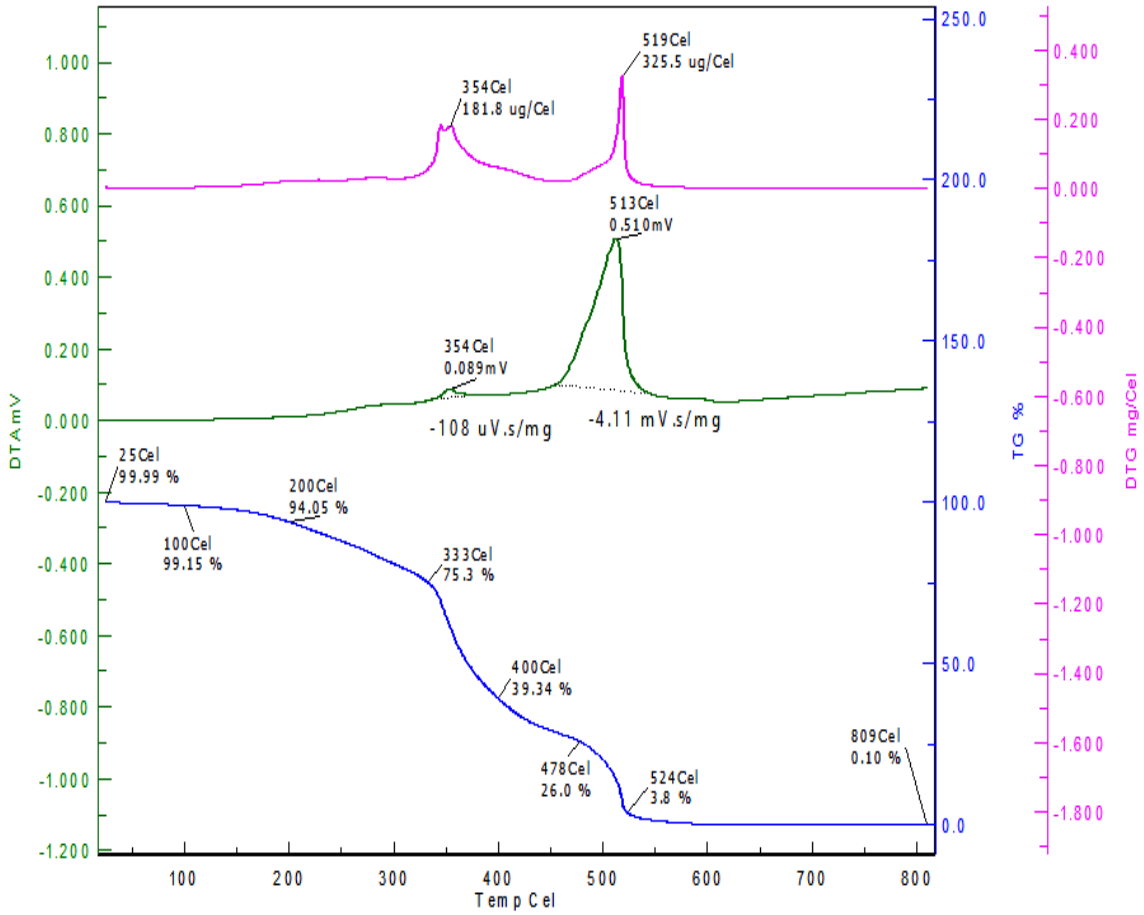

Fig. 4.4 (d): Thermal analysis of epoxy resin with 4% bhimal fiber

Fig. 4.4(e) displays the thermo gram of epoxy resin having 4 wt% of bhimal fiber and 9% of hardener merged in it. Here the initial sample weight is taken to be 20.46 mg. Through the DTG curve analysis, we can conclude that the decay of this composition has been accomplished under two stages that range between 354^0C to 519^0C with the rate of decomposition 4.2 ug/Cel to 2.3 ug/Cel respectively. Prior to 200^0C, the weight loss of 3.95% may be credited to the expulsion of moisture, low molecular mass molecules and volatile objects associated with the composite as illustrated by the TG curve. The extreme rate of decomposition of 181.8 mg/cel has been observed at 354^0C. Such disintegration has been sustained with the fusion heat of -108 uV.s/mg at 354^0C and -4.11 mV.s/mg at 513^0C accompanied by a DTA signal of 0.089 mV and 0.510 mV respectively. The decomposition of the composite material had been concluded at 809^0C leaving a char residue 0.10% of the initial weight.

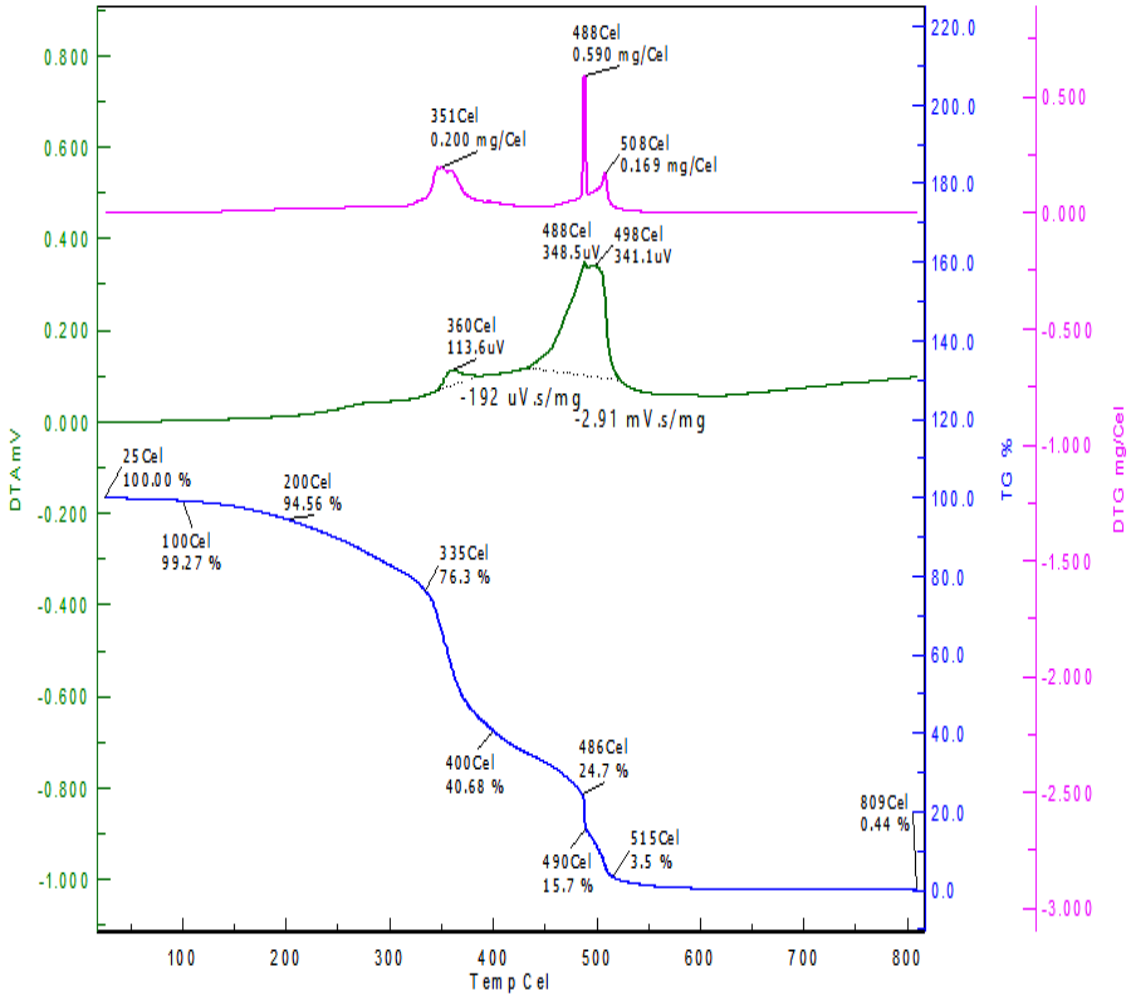

Fig. 4.4 (e): Thermal analysis of epoxy resin with 5% bhimal fiber

Fig. 4.4(e) displays the thermo gram of epoxy resin having 5 wt% of bhimal fiber and 9% of hardener merged in it. Here the initial sample weight is taken to be 20.53 mg. Through the DTG curve analysis, we can conclude that the decay of this composition has been accomplished under three stages that range between 351^0C to 508^0C with the rate of decomposition ranging from 0.20 mg/Cel to 0.59 mg/Cel. Prior to 200^0C, the weight loss of 3.44% may be credited to the expulsion of moisture, low molecular mass molecules and volatile objects associated with the composite as illustrated by the TG curve. The extreme rate of decomposition of 0.590 mg/Cel has been observed at 488^0C. Such disintegration has been sustained with the fusion heat of -192 uV.s/mg at 360^0C and -2.91 mV.s/mg at 498^0C accompanied by a DTA signal of 113.6 uV and 341.1 uV respectively. The decomposition of the composite material had been concluded at 809^0C leaving a char residue 0.44% of the initial weight.

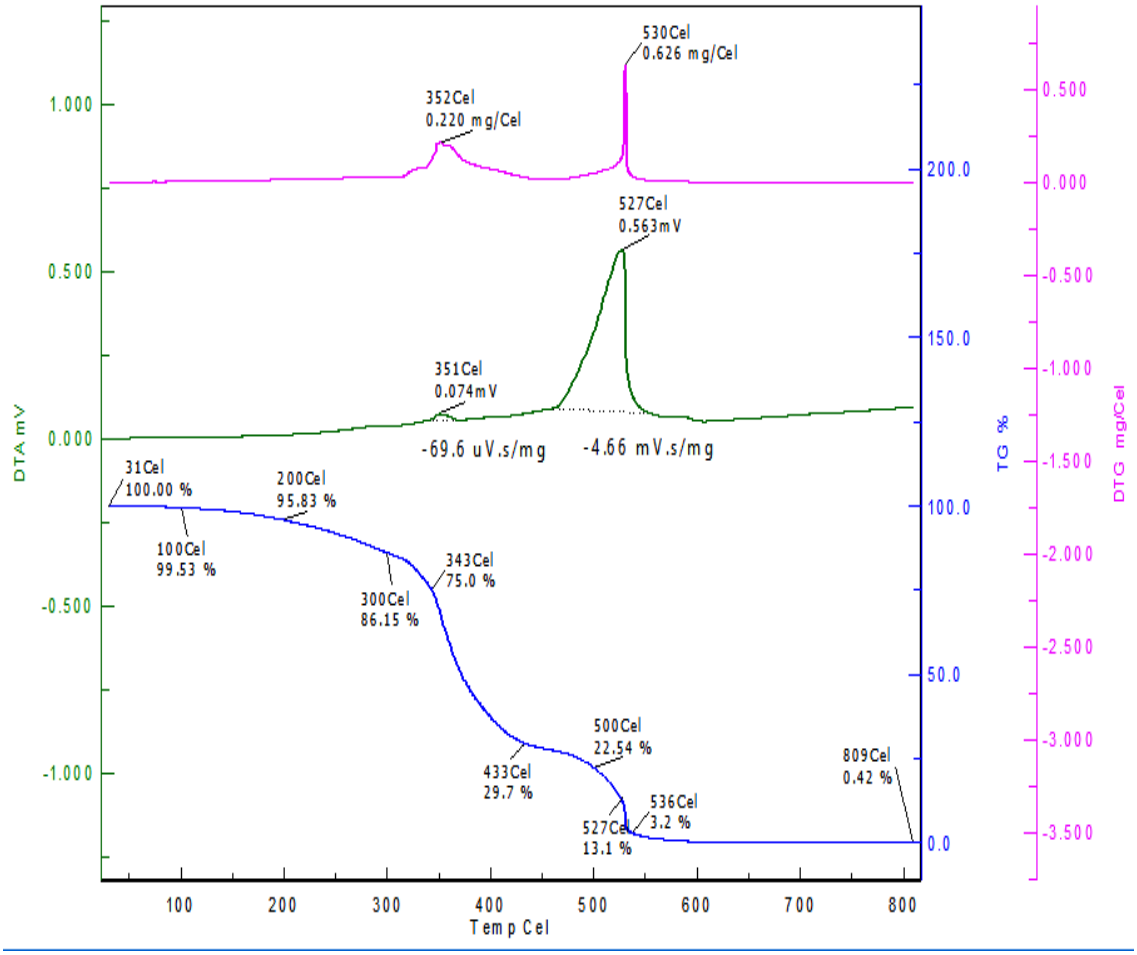

Fig. 4.4 (f): Thermal analysis of epoxy resin with 6% bhimal fiber

Fig. 4.4(e) displays the thermo gram of epoxy resin having 6 wt% of bhimal fiber and 9% of hardener merged in it. Here the initial sample weight is taken to be 20.59 mg. Through the DTG curve analysis, we can conclude that the decay of this composition has been accomplished under two stages that range between 352^0C to 530^0C with the rate of decomposition 0.22 mg/Cel to 0.626 mg/Cel respectively. Prior to 200^0C, the weight loss of 4.17% may be credited to the expulsion of moisture, low molecular mass molecules and volatile objects associated with the composite as illustrated by the TG curve. The extreme rate of decomposition of 0.626 mg/cel has been observed at 530^0C. Such disintegration has been sustained with the fusion heat of -69.6 uV.s/mg at 351^0C and -4.66 mV.s/mg at 527^0C accompanied by a DTA signal of 0.074 mV and 0.563 mV respectively. The decomposition of the composite material had been concluded at 809^0C leaving a char residue 0.42% of the initial weight.

Chapter 5 SUMMARY AND CONCLUSIONS

5.1 Summary

The research work presented in this thesis has provided the portrayal about fabrication and study of mechanical and thermal properties of bhimal fiber filled epoxy resin composites. By incorporating the bhimal fiber with epoxy resin, outstanding outcomes, as expected were achieved in the form of perked up mechanical and thermal properties. Inclusion of the bhimal fiber improved the load bearing capacity i.e. tensile strength and the ability to withstand bending i.e. flexural strength of the composites up to a certain limit. This behavior was well supported by the SEM images and thermo-grams. Also the physical properties of the bhimal fiber such as density and water absorption behavior were analyzed.

5.2 Conclusions

This experimental research on the bhimal fiber fibers filled epoxy resin composites have led to the subsequent precise conclusion as follows;

1. Successful fabrication of epoxy based bhimal fiber fiber filled composites by the hand lay-up technique.
2. Incorporation of the bhimal fiber in epoxy resin matrix resulted in improvement of tensile, compression, flexural and impact strength to a higher magnitude. Hardness and density of the resultant composites are also significantly influenced by the existence of bhimal fibers. In our investigation, the optimum weight % of bhimal fiber 5.
3. The biological fibers used have certain kind of waxes, impurities, hemi-cellulose, pectin and a part of lignin stuck to their surface which tries to reduce the bonding of fiber to the matrix. When the fibers were surface treated with KOH, the surface become free from the constituents listed above and become rough in nature. As a result, the bonding improves which leads to better mechanical and thermal properties.
4. Through SEM we could actually spot how the fibers were dispersed in the matrix, how they are bonded to matrix and each other, how at higher wt% form

clusters and composite failure due to fiber pull out or splitting of fiber and validate our results.

5. The thermal analysis of various compositions was performed using the TGA/DTA curve. The separate DTA, DTG and TG curve for every sample gave the total information about the decomposition rate, thermal characteristics and thermal stability.

Thus, it can be concluded that 5wt% bhimal fiber filled in epoxy resin gave the satisfactorily results from various other combinations. Hence, the properties have significantly improved for the composite as compared to pure epoxy based composite.

5.3 Recommendations for Future Work

The presented effort leaves a huge extent of research work for the future generation of researchers to come. Some suggestions for the future research that can be incorporated are as follows:

- Try out the different orientations (angles) and sizes of bhimal fiber in the matrix.
- Possible use of new matrixes other than epoxy resin.
- Obtain the reaction of these composites to many wear modes such as sliding, erosion and abrasion.
- Attempt to find the thermal and electrical conductivities of these composites.
- Study the tribology and friction behavior of the composites.

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Table A: Experimental Observation to Determine Water Absorption of 10 Fibres Bundle

S. No.	Time (hours)	Weight of fibres at different time interval (gm)	*Water soaked weight of fibres (gm)	**Change in weight of the fibres at different time interval (% wt. gain)
1.	0	0.0305	0	0
2.	2	0.0341	0.0036	11.80
3.	4	0.0420	0.0115	37.70
4.	6	0.0436	0.0131	42.95
5.	8	0.0445	0.014	45.90
6.	10	0.0467	0.0162	53.11
7.	12	0.0474	0.0169	55.40
8.	14	0.0493	0.0188	61.63
9.	18	0.0593	0.0288	94.42
10.	24	0.0596	0.0291	95.40
11.	48	0.0598	0.0293	96.06

Table B: Experimental Observation to Determine Water Absorption of 20 Fibres Bundle

S. No.	Time (hours)	Weight of fibres at different time interval (gm)	*Water soaked weight of fibres (gm)	**Change in weight of the fibres at different time interval (% wt. gain)
1.	0	0.0704	0	0
2.	2	0.0732	0.0028	3.97
3.	4	0.0792	0.0088	12.50
4.	6	0.0834	0.0130	18.46
6.	8	0.0853	0.0149	21.16
7.	10	0.0951	0.0247	35.08
8.	12	0.1025	0.0321	45.59
9.	14	0.1051	0.0347	49.28
10.	18	0.1081	0.0377	53.55
11.	24	0.1087	0.0383	54.40
12.	48	0.1089	0.0385	54.68

Table C: Experimental Observation to Determine Water Absorption of 50 Fibres Bundle

S. No.	Time (hours)	Weight of fibres at different time interval (gm)	*Water soaked weight of fibres (gm)	**Change in weight of the fibres at different time interval (% wt. gain)
1.	0	0.1712	0	0
2.	2	0.2410	0.0698	40.77
3.	4	0.2541	0.0829	48.42
4.	6	0.2572	0.086	50.23
6.	8	0.2593	0.0881	51.46
7.	10	0.2623	0.0911	53.21
8.	12	0.2645	0.0933	54.49
9.	14	0.2776	0.1064	62.14
10.	18	0.2784	0.1072	62.61
11.	24	0.2823	0.1111	64.89
12.	48	0.2829	0.1117	65.24

Table D: Experimental Observation to Determine Water Absorption of 100 Fibres Bundle

S. No.	Time (hours)	Weight of fibres at different time interval (gm)	*Water soaked weight of fibres (gm)	**Change in weight of the fibres at different time interval (% wt. gain)
1.	0	0.4132	0	0
2.	2	0.6391	0.2259	54.67
3.	4	0.6523	0.2391	57.86
4.	6	0.6763	0.2631	63.67
6.	8	0.6886	0.2754	66.65
7.	10	0.6906	0.2774	67.13
8.	12	0.7045	0.2913	70.49
9.	14	0.7126	0.2994	72.45
10.	18	0.7247	0.3115	75.38
11.	24	0.7254	0.3122	75.55
12.	48	0.7293	0.3161	76.50

VITA

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Department	:	Industrial and Production Engineering			
Thesis Title	:	**Experimental Study of Mechanical and Thermal Properties of Bhimal Fiber Reinforced Epoxy Bio-Composite**			
Advisor	:	Dr. Arun Kumar Chaudhary			

ABSTRACT

Bio-composites are putting forth a concentrated effort in almost every engineering field due to their cost viability and biodegradable nature. In a huge variety of natural fibers, bhimal fiber is a very promising fiber that still needs to be explored as an engineering material, because it has very rarely been used by researchers in their research work. Bhimal fiber is a cellulosic fiber extracted from the stems of bhimal tree. It has a great tensile strength and flexibility. In present work bhimal fiber reinforced epoxy bio-composite was successfully prepared by hand lay-up casting technique in order to examine its mechanical and thermal properties for varying content of bhimal fiber (i.e. 2, 3, 4, 5 & 6 weight %). Before the preparation of composite, the surface of bhimal fiber was treated with potassium hydroxide to improve its adhesion properties. From the experiments it was observed that while increasing the fiber content up to 5 wt%, the tensile, flexural and compressive strength continuously increased then started decreasing. Similarly hardness and impact strength were also observed to be increasing up to a peak value at 4 wt% of bhimal fiber. All these results were supported by the SEM, TGA and DTA results. From the observations, it has been concluded that the bhimal fibers are lighter in weight than all existing fibers and strength to weight ratio of the bhimal fiber reinforced epoxy bio-composite is quite higher than the other conventionally used natural fiber based composites. Hence these can be used as reinforcement to produce much lighter weight bio-composites.

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मुख्य विषय : विनिर्माण अभियांत्रिकी एवम् प्रबंधन

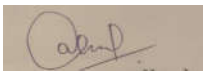
विभाग : औद्योगिक और उत्पादन अभियांत्रिकी

शोध का शीर्षक : भिमल रेशा प्रबलित एपॉक्सी जैव-समग्र के यांत्रिक और तापीय गुणों का प्रायोगिक अध्ययनाः

सलाहकार : डॉ0 अरुण कुमार चौधरी

सारांश

जैव-समग्र अपनी लागत व्यवहार्यता और जैवसंयोजी प्रकतों के कारण लगभग हर इंजीनियरिंग क्षेत्र में एक केंद्रित प्रयास कर रहे हैं। प्राक्रतिक रेशे की एक विशाल विविधता में, भीमल रेशा एक बहुत ही आशाजनक रेशा है, जिसकी अभी भी इंजीनियरिंग सामग्री के रूप में खोज की जानी चाहिए, क्योंकि इसके शोध कार्य में शोधकर्ताओं द्वारा बहुत कम उपयोग किया गया है। भीमल रेशा एक सेलयूलॉजी रेशा है, जिसे भीमल पेड़ की टहनियों से निकाला जाता है। इसमे तन्य शक्ति और लचीलापन बहोत अधिक है। वर्तमान कार्य में भीमल रेशा प्रबलित इपॉक्सी जैव-सामग्र की भीमल रेशे की अलग-अलग मात्रा (यानी 2, 3, 4, 5 और 6 वजन %) पर अपनी यांत्रिक और तापीय गुणों क़ी जाँच के लए हाथ ले-आप कास्टिंग तकनीक द्वारा सफलतापूर्वक तैयार किया गया था। समग्र तैयार करने से पहले, भीमल रेशे की सतह का इसकी आसंजन गुणों में सुधार के लए पोटेसीयम हाइड्रोक्साइड के साथ उपचार किया गया था। प्रयोगों से यह पाया गया की रेशे की मात्रा की 5% तक बढ़ाने पर, तन्यता, सूनमयता और संपीडय शक्ति मे लगातार व्रद्धी हुई फिर घटनी शुरू हो गई। इसी तरह कठोरता और टक्कर शक्ति को भीमल रेशे के 4 % पर अपने अधिकतम मान तक बढ़ता हुआ प्रेक्षित किया गया। इन सभी परिणामों की एसईएम, टीजीए और डीटीए परिणाम के द्वारा समर्थित किया गया था। टिप्पणियों से यह निष्कर्ष निकाला गया है की, भीमल रेशा सभी मौजूदा रेशों की तुलना में वजन में हल्का हैं और भीमल रेशा प्राबलित एपोक्सी जैव-सामग्र का शक्ति से भार अनुपात अन्य परंपरागत रूप से प्रयुक्त प्राक्रतिक रेशा आधारित सामाग्रो की तुलना में काफ़ी अधिक है। इसलिए इसे कम वजन के जैव-सामग्रा उत्पादन मे सुदृढ़ीकरण के रूप में इस्तेमाल किया जा सकता है।



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सलाहकार **लेखक**