# PREPARATION AND CHARACTERIZATION OF BAGASSE-WHEAT STRAW FIBER/ EPOXY BLENDED COMPOSITES

**Ph.D THESIS**

*by*

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**JUNE, 2016**

# PREPARATION AND CHARACTERIZATION OF BAGASSE-WHEAT STRAW FIBER/ EPOXY BLENDED COMPOSITES

**A THESIS**

***Submitted in partial fulfilment of the***
***requirements for the award of the degree***

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***by***

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# INDIAN INSTITUTE OF TECHNOLOGY ROORKEE
# ROORKEE

## CANDIDATE’S DECLARATION

I hereby certify that the work which is being presented in the thesis entitled “**PREPARATION AND CHARACTERIZATION OF BAGASSE-WHEAT STRAW FIBER/ EPOXY BLENDED COMPOSITES**” in partial fulfillment of the requirements for the award of the Degree of Doctor of Philosophy and submitted in the Department of Chemical Engineering of the Indian Institute of Technology Roorkee, Roorkee is an authentic record of my own work carried out during a period from January, 2013 to June, 2016 under the supervision of Dr. Shishir Sinha, Professor, Department of Chemical Engineering, Indian Institute of Technology Roorkee, Roorkee.

The matter presented in the thesis has not been submitted by me for the award of any other degree of this or any other Institute.

**(Varun Mittal)**

This is to certify that the above statement made by the candidate is correct to the best of my knowledge.

**(Shishir Sinha)**
**Supervisor**

The Ph.D. Viva-Voce Examination of................................................., Research Scholar, has been held on ..............................

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This is to certify that the student has made all the corrections in the thesis.

**Signature of Supervisor (s)** **Head of the Department**

**Dated:..........................**

# ABSTRACT

Research in lignocelluloses materials is important because of their advantages over conventional reinforcing materials, namely, low density, high specific properties, non-abrasive nature, high level of filler loadings, high amount availability, renewability, safe working environment, etc. Because of low cost and good mechanical properties bagasse and wheat straw fiber is treated as most abundantly available fiber among all similar types of resources. It can be used as an effective reinforcing material with thermoplastic and thermosetting polymer.

The present work, reports the experimental investigation on the development and characterization of blended natural fiber based composite along with bagasse and wheat straw fiber reinforced with epoxy. Initially composite made with the bagasse and wheat straw fibers reinforced with the epoxy resin separately and identify the optimum condition which gives a superior mechanical strength.

Firstly, mechanical, thermal and water absorption properties of bagasse fiber reinforced epoxy composites has been studied. For this purpose, the epoxy resin is reinforced with 5, 10, 15, 20, and 25 weight % of the bagasse fiber with the help of a hand lay-up technique. Bagasse fibers are subjected to chemical treatment with 1 % sodium hydroxide followed by 1 % acrylic acid at ambient temperature to enhance the bonding strength between the fiber-polymer so as the results give high value of mechanical properties and reduces the water absorption properties of the composites. The optimum value of the bagasse fiber content are examined been found at 15 % treated fiber loading which gives better in mechanical properties simultaneously the lower in water absorption properties. Thermal analysis results prove that the treated bagasse fiber reinforced epoxy composites are 3 % more thermal stable than the untreated bagasse fiber/epoxy composites.

Further, the feasibility of using wheat straw fiber with epoxy resin for developing the natural fiber-polymer composite has been studied. In order to improve the composite characteristic, wheat straw fibers are treated with three different concentrations of alkali (1, 3 and 5 %). The mechanical, thermal and water absorption properties of treated fiber composites are characterized and compared with untreated fiber filled epoxy composites. It is observed that the mechanical properties and water resistance are reduces with an increment in the wheat straw fiber loading from 5 to 25 wt %. Among the three levels of alkali treatment, composite made with 3 % alkali treated fiber exhibits superior mechanical and water absorption properties than the other treated fiber composite, which pointed to an effective fiber-matrix adhesion. The thermal properties of all composition of untreated and alkali treated wheat straw fiber

reinforced epoxy composites are investigated. Upon analysis through Thermo Gravimetric Analysis (TGA) and Differential Thermal Gravimetric (DTG), it has been observed that the thermal stability of the high concentration alkali treated WSF/epoxy composite is 4 % superior to the untreated fiber composite.

Finally, wheat straw fiber is blended with bagasse fiber. The wheat straw and bagasse fibers are prepared with various weight ratios (80/20, 60/40, 40/60 and 20/80) whereas total fiber content in epoxy matrix is 15 % and then incorporated into the epoxy matrix by molding technique to form composites. The mechanical, thermal and water absorption characteristics are examined using blended composite samples. In this part of work, it is found that an increases the bagasse fiber content in wheat straw/epoxy composites results that the 18 % increase in tensile strength, a 22 % increase in flexural and 46 % increase in impact strength.

Epoxy resin reinforced with bagasse/wheat straw fiber composites proved to be the ideal low cost material with superior qualities. Raw material which were used in the present work is waste material and it is utilized in optimum manner which is not only cost effective but also helping the environment.

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At the last but not the least, I pay my prayer to thank the God for strengthening me to complete my thesis work.

**June 2016**

**(Varun Mittal)**

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भारतीय प्रौद्योगिकी संस्थान रुड़की
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# NOMENCLATURE

| | |
|---|---|
| BF | Bagasse Fiber |
| DSC | Differential Scanning Calorimetry |
| DTG | Differential Thermal Gravimetric |
| FT-IR | Fourier Transform Infrared Spectroscopy |
| h | Hour |
| min | Minute |
| MPa | Mega Pascal |
| rpm | Revolution per minute |
| SEM | Scanning Electron Microscopy |
| s | Second |
| TGA | Thermo Gravimetric Analysis |
| WSF | Wheat Straw Fiber |
| wt | Weight |

# 1

# INTRODUCTION

The significance of composite material cannot be overlooked. It is evident in various formations in nature. Mankind has been using natural fiber composites since prehistoric era. One such example is wood, a stringy composite containing cellulose fibers joined with lignin matrix. Plants and trees are also a representative of a natural composite that defends the soil on the river banks to oppose the water flow in the invert direction. Wall is also a composite that has brick as reinforced phase and cement as matrix phase. Various composites given by nature have been used for a very long time in multiple applications.

During the last couple of decade, there has been an enormous development of natural plant-fiber based polymer composite which are predominantly used in several industries like automotive, sports goods, electrical, building and construction, optical application, marine, etc. This equated to the other synthetic fiber (glass and carbon) composites. Natural plant fibers are more attractive because of low cost and environment friendly nature. They possess good specific properties- like low density, renewable, recyclable making them a good substitute for the conventional stuff like metals and wood products (Teng et al., 2015). In India, there is an abundance of bio-fibers such as bagasse, jute, wheat straw, coir, sisal, pineapple, ramie, bamboo, banana etc. They are being used as fillers in various thermoplastic and thermoset polymer.

Fibers produced from the agricultural by-products or waste, such as bagasse, wheat straw, stalk and rice husk have received considerable attention due to their nature as a by-product obtained during the procurement of various edible items. In many cases, these agriculture by-products are burnt out in the open field that release a huge quantum of $CO_2$, resulting in further losses of energy and pollution (Pant and Kunzru, 1997; Pant and Kunzru, 1998; Lee et al., 2015; Mogalicherla and Elbashir, 2011). Emission of $CO_2$ also involves in global warming, which results in rise of sea level (Yongpinga et al., 2010; Chen et al., 2012; Patchigolla and Oakey, 2013).

## 1.1 COMPOSITE

A composite is a combination of materials (framing of two or more materials) mixed or bonded on a macroscopic scale. Generally, composites are made up of reinforcing phase (in the form of fibers, sheets, or particles) embedded in other material called the matrix phase.

Jartiz (1965) defines composites are multifunctional material systems that provide characteristics not obtainable from any individual material. They are a combination of two or more materials which are unlike in composition and characteristics. The other definition given by Berghezan, (1966) composites are the compounds which are distinct from the alloys by the fact that the single components retain their characteristics, but are so incorporated into the composite as to take advantage only of their attributes and not of their shortcomings, in order to obtain an improved material.

Kelly, (1967) observed that the composites should not be regarded simply as a combination of two materials. It is the joining of its own distinctive properties. In terms of strength or resistance to heat or some other desirable quality, it is better than either of the components alone or radically different from either of them.

Suchetclan, (1972) defined as composite materials as heterogeneous materials containing two or more solid phases, which are in confident contact with one another on a microscopic scale. They can be also considered as homogeneous materials on a microscopic scale in the sense that any portion of it will have the same physical property.

## 1.2 PROPERTIES OF COMPOSITE

Composites properties mainly depend on the properties of the materials used, their dispersion and the reinforcement type. It may be affected by a volume fraction of the properties of the components. Also, nature of material and material dimensions like shape, size, distribution factor acts upon the properties of the composite to a great magnitude. The properties of composites also bear upon the reinforcement immersion and orientation (Hui et al., 2010).

Deciding the extent of the interaction between the reinforcement and the component matrix can be done on the basis of the pattern of the discontinuous phase like (spherical, cylindrical, or rectangular cross-sanctioned prisms or platelets) and volume fraction (Kumar et al., 2010). Composites properties cannot be regulated by a single parameter, and also, exhibit an easy change in the  properties of composites by variation of any one parameter.

In engineering aspect, We can divide the properties in three spectra. Mechanical properties: Tensile, Flexural and Impact; Thermal properties: Thermo Gravimetric Analysis (TGA), Differential Thermal Gravimetric (DTA), Differential Scanning Calorimetry (DSC); Other consideration: Water absorption analysis, Fourier Transform Infrared Spectroscopy (FT-IR), and Scanning Electron Microscopy (SEM) analysis.

Study of the mechanical properties of the composite material is very significant because various applications require the mechanical loading. Selection of composite material under loading is based on the tensile strength, tensile modulus, flexural strength, flexural modulus and impact strength (Basuli et al., 2010).

Thermal analysis of the composite materials is as important as the mechanical and chemical analysis. Fiber content, surface treatment, crystallinity, molecular orientation, surface and bonding between the fiber & polymer matrix phase have a considerable effect on the thermal properties of the composite material (Chattopadhyay et al., 2010).

Water absorption of natural fiber composites is dependent on factors such as a property of the portions and their interface compatibility. One of the main disadvantages of natural fibers using the composite is that it absorbs more moisture as compared to the neat polymer resin. Many researchers observed that cellulose and hemicellulose are mainly responsible for the more water uptake of biofibers.

## 1.3 CLASSIFICATION OF COMPOSITE

Composite materials can be classified into many categories. Based on the matrix material it can be classified as metal, ceramic, and polymer composite.

### 1.3.1 Metal matrix composite

Metal matrix composites are composed of a metallic matrix (magnesium, copper, iron, aluminum, cobalt) and a dispersed metal phase (lead, tungsten, molybdenum). In comparison to organic matrix, metal matrix composites have some better characteristics like strength retention at higher temperatures, good transverse strength, high electrical and thermal conductivity (Tian et al., 2014). Despite this, the main limitation of metal matrix composite is that it possesses the high density and low specific mechanical properties as compared to a polymer matrix (Mogalicherla et al., 2014). The fabrication process of metal matrix composites needs more energy.

### 1.3.2 Ceramic matrix composite

Ceramic matrix composites are composed of a ceramic (matrix) and embedded fibers of other ceramic material (dispersed phase). Composite made with ceramic matrix exhibits very high-temperature applications. However, it has very bad tensile and flexural properties. These composites are used in places having high-temperature environmental conditions and unwanted environment attack.

### 1.3.3 Polymer matrix composite

Polymer matrix composites are composed of a matrix thermoset (unsaturated polyester, epoxy) or thermoplastic (polyvinyl chloride, polystyrene, nylon) and dispersed phase as fibers (synthetic and natural fibers) etc. The selection of the polymeric matrix on the basis of their adhesion power, heat resistance, fatigue resistance, chemical and moisture resistance etc. It must be easy to use in the fabrication process. Table 1.1 shows the major advantages and limitations of polymer matrix over the metal matrix. Besides these characteristics, the polymer matrix must be adequate to wet and penetrate into the packets of fibers which provide the better reinforcement with them, removing the void there in and providing those physical characteristics capable of improving the fiber performance (Tian et al., 2015).

**Table 1.1: Advantages and limitations of polymer matrix materials over metal matrix material (Deo, 2010; Mitra et al., 2004)**

| Advantages | Disadvantages |
|---|---|
| Low-density | Low transverse strength |
| Low electrical conductivities | Low operational temperature limits |
| Low thermal conductivities | |
| Good corrosion resistance | |
| Aesthetic color effects | |
| Translucence | |

This can further be classified into thermoset composites and thermoplastics composites.

#### 1.3.3.1 Thermoplastics composite

Thermoplastic composites use thermoplastic polymer as matrix (polyvinyl chloride, polyethylene, polystyrene, Polymethyl methacrylate, etc) (Garg and Gupta, 1999; Garg et al., 1999; Upreti et al., 2005) and reinforcement as fibers (synthetic and natural). Depending upon the property required, a various variety of fillers can be used in composites. Various advantages are associated with the addition of filler like: reduction in cost, density control, higher thermal conductivity, reduced thermal expansion, and improvement in the mechanical properties.

### 1.3.3.2 Thermosets composite

Thermosets composites use thermosets polymer (unsaturated polyester, epoxy resin, etc.) as matrix and reinforcement as fibers (synthetic and natural). The reaction takes place in a monomeric or oligomeric state when the matrix has low viscosity. Thermoset composites utilize natural fibers which improve modulus, creep, impact, and heat resistance properties. Though the addition of fibers gives the higher viscosity, reduce the cost and bring more wear, tear on mold and machinery.

## 1.4 COMPONENTS OF A COMPOSITE MATERIAL

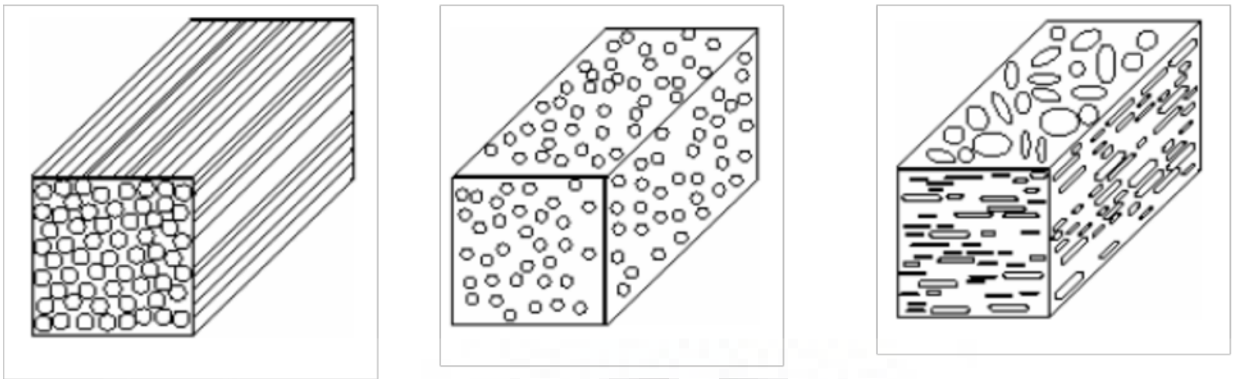
There are three integral part of the component of composite material includes matrix, reinforcement, interface.

### 1.4.1 Matrix

Variety of natural fibers have very good strength, but they should be attached by a compatible matrix. The primary function of the matrix is to combine the fibers to one another in order to keep abrasion and form new surface flaws and acts as a bridge to hold the fibers in place. It may be polymeric, metallic, or ceramic in origin. Shape, surface appearance, environmental tolerance and overall durability of composites are given by the matrix while the fibrous reinforcement carries most of the structural load, thus giving macroscopic stiffness and strength. A beneficial matrix should have the ability to deform easily under applied load, distribute the load onto the several fibers and stress concentration uniformly.

### 1.4.2 Reinforcement

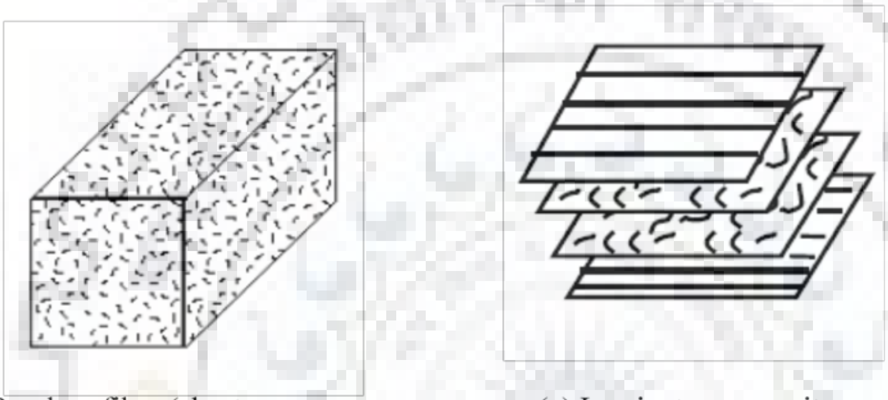
The mechanical strength of the resin system can be increased by the reinforcement. The fibers used in the composite have distinct properties that affect the composite properties in various ways. Sometimes, fibers are arranged in the form of sheet, called as fabric, to ease handling and processing. A variety of characteristics is achieved by the distinct arrangement of the fibers in a single matrix system. Figure 1.1 shows the various arrangement of fibers in the composite materials.



(a) Continuous fiber composite

(b) Particulate composite

(c) Flake composite



(d) Random fiber (short fiber) composite

(e) Laminate composite

**Figure 1.1: Diagram of various arrangement of fibers in the composites (Deo, 2010)**

### 1.4.3 Interface

The interface is also called as bounding surface or zone at which a discontinuity is observed: physical and chemical. Sometimes, coupling agents are frequently used to increase the bonding power between the matrix and reinforcement. The application of the load at the given requirement should be transferred from the matrix to the fiber through the interfaces. Thus, the interface must be a long surface and should exhibit good attachment between the fibers and matrix. Failure of interface destroys the composites.

## 1.5 APPLICATION OF COMPOSITES

The composite material has the variety of applications like automotive industries, construction application, electrical application, sports application, etc. Composite usage in these fields is increasing rapidly due to properties like light weight, good strength, design flexibility, safety, corrosion and electrical resistance.

### 1.5.1 Automotive

Composites play an important role in the field of the automotive industry. The first car-maker to use fiber-polymers based composites was Mercedes-Benz® in the nineties. They fabricated the door panels by composites made by the jute fiber. In Germany, many car manufacturers (like Mercedes, Volkswagen, Audi Group, BMW, Ford and Opel) use natural fiber composites in their parts. There are some important advantages of polymer composite used in the automobile: like safety, light weight, mileage enhancement, reduced transport cost (Suddell and Evans, 2005). Flax and sisal fibers are reinforced with the epoxy resin, to make the various car component as shown in the figure 1.2. By this way 20 % reduction in weight is achieved.

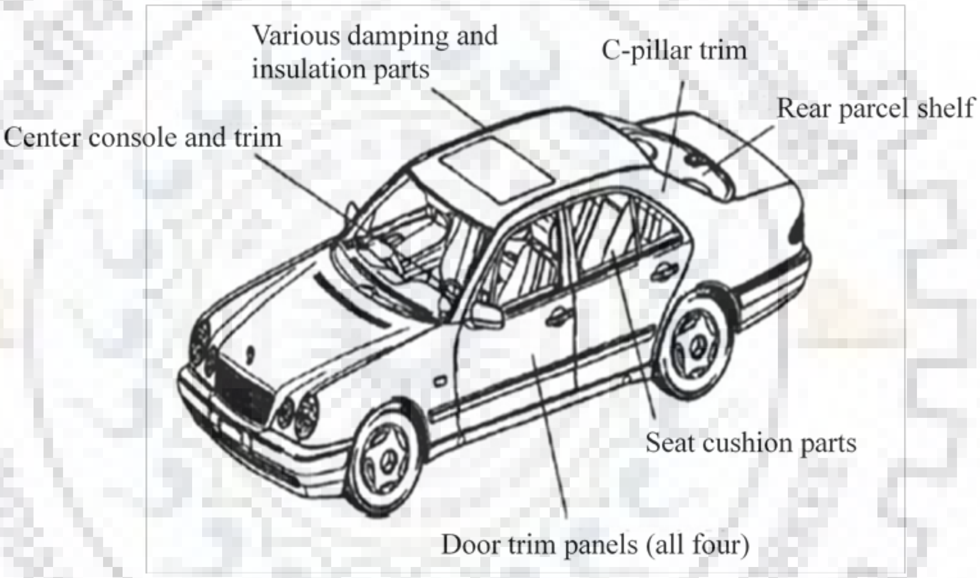


**Figure 1.2: Diagram of car (E-Class Mercedes-Benz®) components of composites (Schuh, 2004).**

### 1.5.2 Construction

Polymer composites have become important engineering material in the past decade and their applications in the construction industries are expanding rapidly. It is one of the important divisions after automotive industry where composite are in huge demand (Kumar et al., 2011). Materials used in the building and construction application must possess properties such as good mechanical strength, resistance to corrosion, weathering, ease of installation, less maintenance, etc. It is used to provide insulation in buildings, which saves a lot of energy. Interior roofing, walls of a building require thermal insulation. Figure 1.3 and figure 1.4 shows the various building and household components of composite materials respectively.

**Figure 1.3: Diagram of building components of composites**

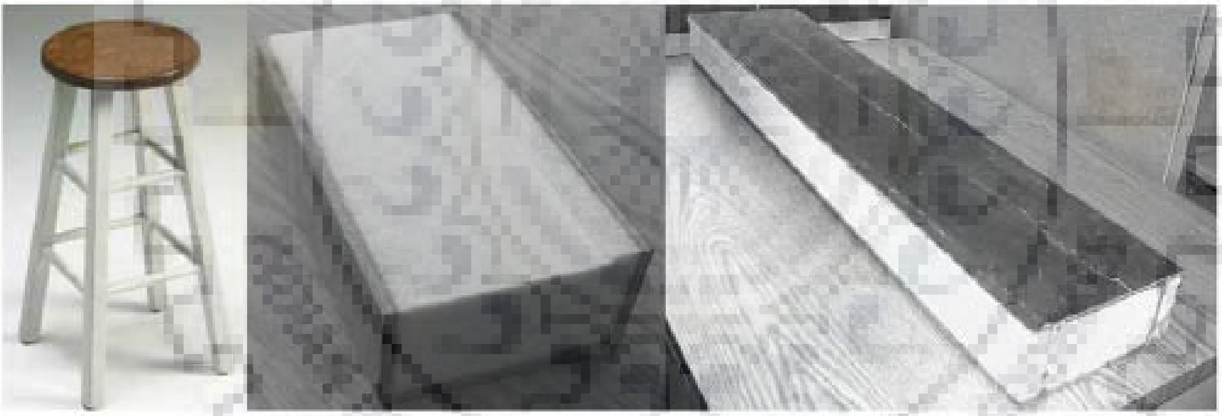


**Figure 1.4: Diagram of household furniture of composites**

**[Teel GRT. Natural fiber products and composites: http://www.teelgrt.com; accessed in September, 2015]**

### 1.5.3 Electrical

Polymer composites possess the property of insulation i.e. they don’t conduct electricity or heat. Now, a day's scientists have proved that current can flow in polymer composite to behave as conducting materials. The important advantages of polymer composite are low density, easy process, a  complex shape that make useful in electrical application.

## 1.6 NATURAL FIBER COMPOSITES

Natural fiber composites have natural fiber which acts as reinforcement phase and the polymer resin which acts as the matrix. However in the fiber section, it is usually referred to as fiber of wood and agro-based bast, seed, leaf, and stem fibers. vegetable, animal, and mineral fibers are part of the natural fibers (Faruk et al., 2012). These fibers give a superior strength to a composite on the reinforcement with the polymer matrix. Huge availability of bio fibers and comfort fabricating have influenced the researchers to attempt the locally available inexpensive fibers. The research done on lignocelluloses materials is important to the benefits given by these fibers over conventional reinforcing materials like low density, high specific properties, non-abrasive nature, high level of filler loadings, high amount availability, renewability, safe working environment, etc (Bledzki et al., 2008; Li et al., 2004b; Shibata et al., 2002; Shibata et al., 2003; Sinha et al., 2008). It is a better substitution for highly useable synthetic fibers.

Due to environmental issues and to fulfill the market demands, manufacturing of composite is on a rising trend, especially the use of natural fibers in composites with the expectation of reaching more and more. Natural fiber composite materials are attractive because they have merged properties which are not easily found in nature.

## 1.7 NATURAL FIBER

Natural fibers may be obtained from plants, animal, and mineral sources. Plants are producing two types of natural fibers: one is primary fiber and other is secondary fiber. Fiber which is directly obtained from plant root is called the primary fibers while secondary fibers are the byproducts from the utilization of the primary fiber. Primary fibers include jute, kenaf, cotton, sisal, and hemp while secondary plants include wheat, bagasse, pineapple, cereal stalks, agave, oil palm and coir (Pant and Mohanty, 2014). Though fiber mainly originates from the wood, plant straws and stalks mixed are potentially a larger source of fiber than wood.

Natural fibers including hemp, banana, flax, sisal, oil palm and jute have the various amount of techno-economical and ecological advantages against the synthetic fibers. There are a number of industrial applications, which prefer bio-fiber reinforced composite because it has a superior combination of mechanical and physical properties with their eco-friendly character. Table 1.2 shows the advantages and limitations of natural fibers used in composites.

**Table 1.2: Advantages and disadvantages of using natural fibers in composites (Sreekumar, 2008)**

| Advantages | Disadvantages |
|---|---|
| Renewable resources, production require less energy and low $CO_2$ emission | Lower strength, especially impact strength |
| Low specific weight results in a more specific strength and stiffness than glass | Lower durability |
| The processing atmosphere is worker-friendly with better working conditions | Lower fire resistance |
| Biodegradability | Poor moisture resistance |
| Good thermal and acoustic insulating properties | Lack of fiber-matrix adhesion |
| High electrical resistance | |

### 1.7.1 Chemical composition of natural fibers

Characteristics of lignocellulosic fibers depend primarily on the nature and type of the plant; locality in which it grows, plant life, and the extraction method used. The living tree body has water as a major chemical component. However, walls of most of the plant cell contained principally of sugar-based polymers which are (cellulose, hemicellulose) which connected with lignin, with small add up of inorganic, extracts, protein and starch (Nanda et al., 2013a; Nanda et al., 2013b). The cell wall structure of a plant is distributed in two parts, one is primary and other secondary, they are merged with the chemical components. Every plant has a different chemical composition, and it also differs from various regions of the particular plant. Table 1.3 shows the range of the average chemical constituents of various natural fibers.

**Table 1.3: Chemical composition of some common natural fibers (Hattallia et al. 2002; Hoareau et al. 2004; Marti-Ferrer et al. 2006)**

| Fiber | Cellulose (wt%) | Hemicellulose (wt%) | Lignin (wt%) | Waxes (wt%) |
|---|---|---|---|---|
| **Bagasse** | 55.2 | 16.8 | 25.3 | – |
| **Sisal** | 65 | 12 | 9.9 | 2 |
| **Flax** | 71 | 18.6–20.6 | 2.2 | 1.5 |
| **Rice straw** | 41–57 | 33 | 8–19 | 8–38 |
| **Kenaf** | 72 | 20.3 | 9 | – |
| **Jute** | 61-71 | 14–20 | 12–13 | 0.5 |
| **Hemp** | 68 | 15 | 10 | 0.8 |
| **Bamboo** | 26-43 | 30 | 21-31 | – |
| **Ramie** | 68.6–76.2 | 13–16 | 0.6–0.7 | 0.3 |
| **Curaua** | 73.6 | 9.9 | 7.5 | – |
| **Coir** | 32–43 | 0.15–0.25 | 40–45 | – |
| **Abaca** | 56–63 | 20–25 | 7–9 | 3 |
| **Oil palm** | 65 | – | 29 | – |
| **Wheat straw** | 38–45 | 15–31 | 12–20 | – |
| **Pineapple** | 81 | – | 12.7 | – |
| **Rice husk** | 35–45 | 19–25 | 20 | 14–17 |

### 1.7.2 Properties of natural fibers

Natural fiber properties are distinct among referred works due to dissimilar fibers being used in different moisture conditions in combination with distinct testing procedure. Performance of natural fiber based reinforced polymer composite depends on various factors like: fiber chemical composition, the dimension of the cell, fiber microfibrillar angle, fiber surface defects, fiber structure, physical and mechanical properties, and also the reinforcement of a fiber with the polymer matrix. Nowadays, the use of natural fibers is increasing for making the composite material to enhance their performance. It is essential to know the properties of the fiber. The mechanical properties of the natural fiber are less than the synthetic fiber i.e. glass fiber because it has distinct fiber structure due to the overall environment conditions during the growth period. Table 1.4 shows the some important physiomechanical properties of usually used natural fibers.

**Table 1.4: Mechanical properties of some common natural fibers (Hattallia et al., 2002; Hoareau et al., 2004)**

| Fiber | Tensile strength (MPa) | Young's modulus (GPa) | Elongation at break (%) | Density [g/cm$^3$] |
|---|---|---|---|---|
| **Bamboo** | 140-230 | 11–17 | – | 0.6–1.1 |
| **Abaca** | 400 | 12 | 3–10 | 1.5 |
| **Hemp** | 690 | 70 | 1.6 | 1.48 |
| **Bagasse** | 20-290 | 17 | – | 1.25 |
| **Kenaf** | 930 | 53 | 1.6 | – |
| **Jute** | 393–773 | 26.5 | 1.5–1.8 | 1.3 |
| **Sisal** | 511–635 | 9.4–22 | 2.0–2.5 | 1.5 |
| **Flax** | 345–1035 | 27.6 | 2.7–3.2 | 1.5 |
| **Oil palm** | 248 | 3.2 | 25 | 0.7–1.55 |
| **Pineapple** | 400–627 | 1.44 | 14.5 | 0.8–1.6 |
| **Ramie** | 560 | 24.5 | 2.5 | 1.5 |
| **Coir** | 175 | 4–6 | 30 | 1.2 |
| **Wheat straw** | 32 | 6.6 | - | 1.15 |

### 1.7.3 Surface modification of natural fibers

The major drawback of all natural fibers is its hydrophilic nature. They show the poor compatibility with polymer matrix, if used directly reinforcement without any modification. The non-crystalline parts and voids of the fiber are responsible for the moisture content of the fibers. The mechanical properties are also affected by the hydrophilic character of natural fiber. In order to enhance their bonding with various matrices and reduce the hydrophilic character, natural fibers needs to be modified. Strong interface leads to more strength and stiffness that is very brittle in nature resulting in crack propagation through the matrix and fiber whereas weak interface results in less efficiencies stress transfer from matrix to the fiber. Prima facie these modification need to be carried out at the surface of fiber. There are two surface modification methods available in the literature, physical, and chemical.

#### 1.7.3.1 Physical method

Structure and surface characteristics of the fiber can be changed by physical methods thereby affect the mechanical connectivity between the fiber and polymer matrix. It cannot change the chemical composition of the natural fibers (Faruk et al., 2012). Therefore, the interface is normally improved via an enhancement of the mechanical bonding between the fiber and the matrix. Stretching, calendaring and thermo-treatment are parts of this method.

Physical method is two types.

##### 1.7.3.1.1 Corona treatment

Corona treatment is one of the most interesting techniques for surface oxidation activation. Corona treatment modifies the surface energy of the natural fibers by the surface oxidation process. It is an effective work for the betterment of the compatibility between hydrophilic fibers and a hydrophobic matrix. This treatment does not harm the material and offer manufacturer an eco-friendly solution.

##### 1.7.3.1.2 Plasma treatment

It has the same function as corona treatment. Plasma makes changes in the material surface by creating a roughness on the surface of the fibers. The surface energy of the fiber can be changed. Free radicals and cross-linking of surfaces can be obtained by this technique. The use of this treatment in the automotive industry is growing due to increased use of plastic materials in the manufacturing of interiors and exteriors component for light motor vehicle.

#### 1.7.3.2 Chemical method

Natural fibers have poor adhesion nature with polymer due to their hydrophilic character. Materials having several coupling mechanisms such as weak boundary layers, restrained and deformable layers, wettability, chemical bonding, and acid–base effect. It includes the fiber surface morphology, the acid-base reactions in the interface, surface energy, and the wetting phenomena. The aim of the chemical method is to improve the reinforcement efficiency between the fiber and the polymer matrices. This method can be divided in several ways.

#### 1.7.3.2.1 Silane treatment

Silane coupling agents may lower the hydrophilic nature of the fiber by removal of the hydroxyl groups present in the natural fiber. Silane is a chemical compound having chemical formula $SiH_4$. In the presence of moisture, the hydrolyzable alkoxy group extends to the formation of silanol. The silanol further reacts with the hydroxyl group present in the fiber forming stable covalent bonds to the cell wall that are chemically joined with the fiber surface (Agrawal et al., 2000).

$$CH_2CHSi(OC_2H_5)_3 + H_2O \rightarrow CH_2CHSi(OH)_3 + 3C_2H_5OH$$

$$CH_2CHSi(OH)_3 + \text{Fiber–OH} \rightarrow CH_2CHSi(OH)_2O\text{–Fiber} + H_2O$$

#### 1.7.3.2.2 Alkaline treatment

Alkaline treatment is one of the major applied chemical treatment for application of the reinforcement of the natural fiber with the polymer matrices. This treatment disturbs the hydrogen bonding present in the cellulose and hemicellulose, thereby increasing the roughness of the fiber surface. It also takes out a some amount of lignin, oils, wax and other impurity presents at the external surface of the fiber, depolymerizes cellulose and discloses the short length crystallites (Mohanty et al., 2001a).

$$\text{Fiber–OH} + NaOH \rightarrow \text{Fiber–O–Na} + H_2O$$

#### 1.7.3.2.3 Acetylation

Acetylation treatment of the natural fibers is a well-known esterification method causing plasticization of cellulosic fibers. The main purpose of acetylation treatment is to pick the hydroxy groups of fibers which are responsible for their hydrophilic character and make them hydrophobic in nature. It raises the dimensional stability of composites.

$$\text{Fiber–OH} + CH_3 – C(=O) –O–C(=O)–CH_3 \rightarrow \text{Fiber–OCOCH}_3 + CH_3\,COOH$$

#### 1.7.3.2.4 Maleated coupling

Maleated coupling is also used for the fiber surface modification method to enhancement the adhesion between the polymer and fiber. The main aim of this treatment is to modify the fiber surfaces as well as to provide the superior adhesion between the fiber and polymer matrix thus improving the mechanical strength of the composite (Gassan and Bledzki, 1997; Van and Peijs, 1998).

#### 1.7.3.2.5 Acrylation treatment

Acrylation treatment is widely used to give the high strength of the natural fiber based composites. The free radicals of cellulose molecule are responsible for this reaction to occur. Cellulose can be treated with high energy radiation to generate radicals together with chain scission. Tensile strength and tensile modulus of fibers can be enhanced because it develops the strong covalent bond (Paul et al., 1997).

$$\text{Fiber}{-}\text{OH} + \text{CH}_2{=}\text{CHCOOH} \quad \rightarrow \quad \text{CH}_2{=}\text{CHCOO}{-}\text{Fiber} + \text{H}_2\text{O}$$

#### 1.7.3.2.6 Enzyme treatment

The application of enzyme treatment is increasing significantly for the processing of natural fibers due to its eco-friendly in nature. Specific catalytic reactions are involved in this treatment (Bledzki et al., 2010a; Wu et al., 2013a; Wu et al., 2013b).

## 1.8 OBJECTIVES OF THESIS

In view of an extensive literature review, it has been observed that less quantum of work has been carried out on wheat straw and bagasse fiber reinforced polymer composite. As far as blends are concerned, no work has been reported so far on a blend of wheat straw and bagasse fiber. Based on their mechanical properties we have selected these fibers which are inexpensive and easily available in the environment. Matrix phase selected is epoxy resin due to its better properties and easy workability.

To prepare a blended fiber composite by using two natural fibers (bagasse and wheat straw) which are abundant, inexpensive and eco-friendly; reinforced in a polymer matrix (epoxy resin) and promote the new era of green composites.

- To study the effect of fiber loading and chemical treatment on the mechanical, and water absorption properties of bagasse fiber reinforced epoxy composites and to characterize the effect of surface modification of bagasse fiber for the enhancement of mechanical properties of composites by the Scanning Electron Microscopy (SEM) analysis.
- To observed the effect of fiber loading and chemical treatment on the thermal stability of bagasse fiber reinforced epoxy composites and to analyze changes in the functional group of the fibers after chemical treatment by using Fourier Transform Infrared Spectroscopy (FT-IR).
- To study the effect of fiber loading and chemical treatment on the mechanical, and water absorption properties of wheat straw fiber reinforced epoxy composites and to characterize the effect of surface modification of bagasse fiber for the enhancement of mechanical properties of composites by the Scanning Electron Microscopy (SEM) analysis.
- To observed the effect of fiber loading and chemical treatment on the thermal stability of wheat straw fiber reinforced epoxy composites and to analyze changes in the functional group of the fibers after chemical treatment by using Fourier Transform Infrared Spectroscopy (FT-IR).
- To prepare a composite of wheat straw and bagasse blended natural fiber reinforced in epoxy and to study the effect of blend ratio of bagasse and wheat straw fiber on the mechanical and thermal properties of the respective blended composite

## 1.9 OVERVIEW OF THESIS

Chapter 1 is having an introduction to composites, properties, application of composites, natural fibers, various modification techniques of natural fibers. Chapter 2 contains the literature review on the basis of reinforced phase (bagasse and wheat straw fiber) and reinforced matrix (epoxy) used in this work. The materials, methods and instrumentation used in this work are described in Chapter 3. The results and discussion on the natural fiber (bagasse and wheat straw) reinforced matrix (epoxy) composite characterization via tests (mechanical, thermal properties, water absorption, FT-IR, and SEM analysis) are discussed in Chapter 4. Finally, the conclusions and recommendations are presented in Chapter 5.

# 2

# LITERATURE REVIEW

Natural fiber reinforced composite materials are attractive because they allow combining properties in ways that are not found in nature. These materials offer good mechanical properties with light weight structure at low cost. A composite consists of a discontinuous reinforcement phase implanted in a continuous matrix phase. A concise review of the existing literature on the natural fibers (bagasse and wheat straw) and matrix phase (epoxy resin) is presented in this chapter.

## 2.1 BAGASSE FIBER REINFORCED POLYMER COMPOSITES

Present section deals with the study of bagasse fiber reinforced polymer composite.

### 2.1.1 Effect of surface treatment on mechanical and thermal properties

**Sousa et al. (2004)** examined the consequence of pre-treatment of fiber, the size of fiber and molding pressure on the mechanical behavior of chopped bagasse-polyester composites. They found that composite made of bagasse fiber (size below mesh #20 sieve) gives better mechanical strength, this is due to the availability of more surface area for the adhesion. It was reported that the molding pressure also affects the mechanical behavior due to reduction in voids of composites.

**Cao et al. (2006)** studied the effect of the alkali treatment of bagasse fiber on the mechanical properties of bagasse-polyester reinforced composites. They reported that composite which were prepared with the 1% alkali treated fiber has superior mechanical strength than the composites manufactured with untreated fiber. It was observed that surface modification improves the fiber-matrix adhesion.

**Zheng et al. (2007)** worked on the surface treatments of bagasse fiber with benzoic acid and examined the mechanical properties of the Polyvinyl chloride reinforced composite by the orthogonal optimal method. They found that interface modifier improves the tensile strength and small changes occurring in the impact strength of the composite.

**Luz et al. (2008)** studied the effect of acetylation on the mechanical and thermal properties of sugarcane bagasse reinforced polypropylene composites. They confirmed that chemical modification efficiency of fiber by the Fourier Transform Infrared Spectroscopy (FT-IR) and Scanning Electron Microscopy (SEM) analysis. It was observed that acetylation reduces the OH bonds of the fiber subsequently modify the fiber surfaces which results from the losses of mechanical property. The composites fabricated by the untreated fibers were found more crystalline than pure polypropylene.

**Vilay et al. (2008)** analyzed the influence of surface treatment and content of fiber on the properties of bagasse fiber/ unsaturated polyester composites. High tensile strength, high flexural strength and less water absorption properties were achieved up to 20 wt % bagasse fiber (acrylic acid treated) polyester composites compared to those of untreated fiber based composites. The chemical treatment modified the surface of the fiber that further improved the fiber–matrix interaction. The treated fiber composites exhibit less water absorption than the untreated fiber composites.

**Mulinari et al. (2010)** analyzed the bagasse/high-density polyethylene composites. Bagasse fibers modified by zirconium oxychloride for the purpose of improvement in the mechanical properties. They found that mechanical properties of chemically modified fiber reinfored high density polyethylene composite are superior to bleached fiber composites. It was reported that the fracture surface of the modified fiber composite has superior bonding strength than the bleached fiber composites.

**Acharya et al. (2011)** studied the effect of alkali treatment on the flexural properties of the bagasse fiber/epoxy composite. They reported that alkali treatment gives a positive effect on the flexural strength of the bagasse fiber reinforced epoxy composites. Upon analysis through SEM, they found that surface one treated with alkali have effective adhesion capability than the untreated one.

**Cerqueira et al. (2011)** determined the effect of chemical modification on the mechanical behavior of bagasse fiber reinforced polypropylene composites. They found that composites fabricated with the bagasse fiber (treated with 10 % sulfuric acid solution, followed by the 1 % sodium hydroxide solution) exhibit superior strength compared with the pure polymer.

**Rodrigues et al. (2011)** examined the influence of estherification on the tensile properties of sugarcane bagasse fibers reinforced polyester composites. They reported that estherification through anhydride of sugarcane bagasse fibers has high tensile modulus as compared to neat polymer.

**Brugnago et al. (2011)** analyzed the influence of steam explosion on the development of bagasse reinforced polyester composites. They observed that composites manufacture with steam explosion treated fibers showed effective improvement in water absorption and thermal stability properties over composites manufacture with untreated fibers. This is due to the fact that steam explosion decreases the acid-soluble lignin content present in the bagasse fiber.

**Huang et al. (2012)** studied the effect of mechanical activation and modification of aluminate coupling agent on the mechanical characteristics of bagasse reinforced Polyvinyl chloride composites. FT-IR analysis, revealed that mechanical activation increases the condensation reaction between aluminate coupling agent and the hydroxyl group present in the bagasse fibers. By this way, hydrophobicity of fiber is increases that affect the mechanical properties of the composites.

In order to examine the un-carbonized and carbonized bagasse reinforced recycled polyethylene composites properties **Agunsoye et al. (2013)** developed composite sheets by the compression molding method. Morphological studies revealed that fiber is uniformly distributed in the polymer matrix so better adhesion is achieved. Therefore, mechanical characteristics of respective composites effectively improved. The tensile strength and flexural strength of the composite rises with increasing the amount of the bagasse fiber (up to 20 wt %) for un-carbonized type and (up to 30 wt %) for carbonized type reinforced recycled polyethylene composites. On the other side, impact strength and fracture toughness reduces while increasing the bagasse fiber content.

To study the effect of extractive and fiber content on the mechanical characteristic of the bagasse flour/high-density polyethylene composites **Sheshmani (2013)** fabricated the composites by the injection molding. It used three different solvents ethanol–benzene, 1 % NaOH and hot-water for the removal of extracts from the fibers. It was found that composites fabricated with extracted bagasse possess good water absorption and tensile strength than the unextracted composites under same filler content; this is due to extractives those are blocked the OH groups. Significant increment in water absorption and tensile strength is reported upon increasing in fiber content.

**Arrakhiz et al. (2013)** studied the mechanical behavior of alfa, coir and bagasse fibers reinforced polypropylene composites. They applied the alkali treatment on all of three fibers and observed that composite developed with treated fibers has superior mechanical strength than the composites prepared with untreated fiber. Alkali treatment withdraw the waxes and other impurities which present on the surface of the fiber. They also found that composites developed with a 30 wt % fiber loading exhibit the higher mechanical characteristics.

### 2.1.2 Effect of compatibilizer/coupling agent on mechanical and thermal properties

To study the interfacial behavior of sugarcane bagasse fiber/ recycled poly (ethylene terephthalate) composites. **Corradini et al. (2009)** used two different compatibilized agents naming ethylene/n-butyl acrylate/glycidyl methacrylate (EBGMA) and ethylene–methyl acrylate (EMA) further compared the torque values. They found that EBGMA enhances the interaction between the constituents more effectively than the EMA. They also reported that bagasse fiber content does not affect the Young modulus, but reduce the tensile strength and elongation of poly (ethylene terephthalate) composites.

**Mulinari et al. (2009)** examined the effect of cellulose (10 %) and Cell/$ZrO_2$ $nH_2O$ (10 %) on the mechanical behavior of bagasse reinforced high-density polyethylene composites. They observed that the modification of bagasse cellulose with 10 wt % zirconium oxychloride gives positive results and it effectively improves the tensile modulus of the respective composite. Morphological studies revealed that modified fiber composites possess less void than the untreated fiber composites.

**Filho et al. (2011)** worked to develop, characterize and compare the chipboard panels prepared by bagasse fiber with urea formaldehyde and melamine formaldehyde resins individually. They shaped the panels by addition of pine, eucalyptus, and paraffin separately with the bagasse fibers. It was reported that panels contained pine particle exhibit the better tensile properties than the panels prepared with eucalyptus.

**Samariha et al. (2013)** studied the effect of filler loading and coupling agent (maleic anhydride Polypropylene) on the mechanical properties of bagasse flour/ polypropylene composites. They observed that reducing the impact strength and enhancement in tensile properties of the composites with increases the bagasse fiber loading from 25 to 45 %. Coupling agent also improved the composites properties because it improvises the fiber/polymer adhesion.

**Ashoria et al. (2013)** examined the influences of multi-walled carbon nanotubes (MWCNTs), and maleic anhydride grafted polyethylene (MAPE as a coupling agent) on the mechanical characteristics of bagasse/ high-density polyethylene composites. They found that tensile and flexural properties reach their highest values when 1.5 % MWCNTs and 4 weight % MAPE added into the composite. This is due to the fact that at higher values of MWCNTs attribute the agglomeration and stress transfer gets blocked. MAPE provide an effective bonding between the fiber and polymer.

### 2.1.3 Effect of fiber orientation on mechanical and thermal properties

**Lee and Mariatti (2008)** comprise the two components of bagasse: rind (outer part) and pith (inner part) which was used as reinforcement in unsaturated polyester composites. It was reported that the composites prepared with rind fiber gives better flexural, impact properties and poor water absorption rate compared to the pitch fiber composite.

**Mishra and Acharya (2010a)** worked on the abrasive properties of bagasse fiber reinforced epoxy composite which contain fiber in dissimilar directions (normal, parallel and anti-parallel). They observed that composite made with fiber in the parallel orientation have highly wear resistance than the composite made with fiber in the anti-parallel or normal orientation. Further, noticed that parallel orientation composite has an abrasion due to a micro ploughing, whereas in anti-parallel or normal orientation composite have micro cutting. In another study **Mishra and Acharya (2010b)** examined the influence of impingement angle and particle velocity on the solid particle erosion behavior of composites prepared with bagasse fiber. They reported that impingement angle (at 90°) gives brittle behavior with best erosion rate. Upon analysis through SEM, it was found that overall erosion damages the composite matrix material, removed the resin and break the fiber which is present in the composite.

### 2.1.4 Effect of process parameters on mechanical and thermal properties

**Raj and Kokta (1991)** studied the effect of process conditions and binding material on the mechanical properties of bagasse fiber-polyethylene composites. They found that composite which are fabricated at a high molding temperature have better mechanical strength than the composite fabricated at lower temperature and mechanical properties also increase by the addition of polyethylene up to 20 wt %.

**Bilba et al. (2003)** analyzed the influence of various parameters on the setting properties of the bagasse/cement composite like botanical components of fiber, thermal treatment of fiber and loading of fiber. They found that setting properties of the respective composite are affected by the concentration of lignin and thermal treatment (200°C) of bagasse.

**Talavera et al. (2007)** worked on the influence of the process temperature, pressing time, bagasse content and pressure on the bending properties, thickness swelling and water absorption characterstic of bagasse-plastic composite boards. They found that bending strength and bending modulus of elasticity rises proportionally with press temperature, pressing time and bagasse content. It was found that increment of pressure reduces the bending strength and bending modulus of elasticity. Operation temperature and pressing time give positive effect on the water absorption and thickness swelling property of the respective boards.

**Luz et al. (2007)** performed injection and compression molding processes to determine the best mixer method for bagasse-polypropylene composites. They found that injection molding under vacuum process is the best method for making for bagasse-polypropylene composites. Upon analysis through SEM, they found that composite made by this method having homogeneous distribution of fibers.

**Pinto et al. (2009)** analyzed the influence of molding pressure on the structure and thermal properties of bagasse fibers/poly (hydroxybutyrate) composites. It was found that spherulite size increases and thermal transitions take place with raising the pressure. Differential Scanning Calorimetry (DSC) curves state that; absence of one peak of poly (hydroxybutyrate) composites.

**Huang et al. (2009)** examined the effect of mechanical activation on the graft copolymerization of bagasse fiber and acrylic acid. They observed that the mechanical activation could break fiber bundles and decrease the crystallinity. It was found that graft copolymerization is raises with the mechanical activation.

**Xu et al. (2010)** studied the creep behaviors of bagasse/polyvinyl chloride (recycled and virgin) composites, bagasse/high density polyethylene (recycled and virgin) composites and commercial wood/high density polyethylene composites. It was reported that temperature impacts heavy effect on the creep behavior of all of the composites. They found that bagasse/polyvinyl chloride composite is having good creep resistance than bagasse reinforced high density polyethylene composites at low temperature. They used various models to fit the measured creep data like (i.e., Burgers model, Findley's power law model, and a simpler two-parameter power law model).

**Beninia et al. (2011)** studied the influence of weather condition on the visual appearance and mechanical characteristic of high impact polystyrene reinforced bagasse fiber composites. They observed that bleached fibers reinforced composites are less susceptible to weathering exposure than the mercerized fibers reinforced composites due to higher lignin content in mercerized fibers.

**Monteiro et al. (2016)** characterized the raw bagasse or extracted bagasse fibers reinforced epoxy composites. They compared the ballistic performance of bagasse fiber composites plate with the kevlar composites which are used in commercial multilayered armor systems. From this study, it has been found that both composites satisfied the National Institute of Justice (NIJ) norm, and displayed similar depths of indentation in a clay witness. Economical analysis revealed that armor with bagasse fiber composite is nearly 180% less expensive than a corresponding armor with Kevlar.

## 2.2 WHEAT STRAW FIBER REINFORCED POLYMER COMPOSITES

Present section deals with the study of wheat straw fiber reinforced polymer composite.

### 2.2.1 Effect of surface treatment on mechanical and thermal properties

**Avella et al. (2000)** examined the thermal and mechanical properties of the wheat straw reinforced polyester composites. Initially, wheat straw fiber is treated with the high temperature steam which providing the fiber more reactive and more cellulose content. They reported that treated fiber composite has a higher value of the young modulus than the neat polyester. From thermal study, it was found that the presence of wheat straw fibers in composite increases the rate of polyester crystallization; this is due to the of nucleating effect, while the crystalline of samples remains unchanged. The content of wheat straw does not reduce the biodegradation rate of wheat straw/polyester composites in liquid environment. But 20/80 and 30/70 wheat straw/polyester composites shows a low rate of biodegradation as compared with both neat Polyester and 10/90 wheat straw/polyester composite.

**Xiaoqun et al. (2003)** analyzed the various properties of medium-density wheat straw particle board using different adhesives like methylene diphenyl diisocyanate, urea formaldehyde, soybean protein isolate and soybean flour as well as chemical treatment of the wheat straw. They reported that within all adhesives, particleboard made from methylene diphenyl diisocyanate showed better mechanical performance and water resistance than the other adhesives. Treated wheat straw particleboard has improved mechanical performance over that untreated wheat straw particleboard.

In order to analyze the morphology, physical, mechanical and thermal properties of fiber reinforced polypropylene composites **Panthapulakkal et al. (2006)** fabricated the composites with mechanical and chemical processes. After applying the chemical treatment on the fiber; they show the better mechanical properties than the untreated fibers. It was reported that chemically processed fiber composites showed lower strength properties than mechanically processed fiber composites; this is due to the fact of lack of dispersion.

**Alemdar and Sain (2008)** analyzed the influence of (chemical treatment followed by a mechanical treatment) of the wheat straw nanofibers (diameter: 10–80 nm and length: 1000-4000nm) on the thermal and mechanical properties of wheat straw nanofiber starch-based thermoplastic polymer nanocomposites. It was found that treated fibers have 35 % more crystalline than the untreated fibers. Treated nanocomposites exhibit better thermal and tensile property as compared to both neat thermoplastic starch and untreated fiber composites.

**Kaushik et al. (2010a)** studied the effect of (steam explosion, acidic and mechanical treatment mechanical activation) on the mechanical and barrier properties of the wheat straw nanofibrils/thermoplastic starch composites. From TGA, they observed that increment in the amount of residue left, with the addition of fibril in the polymer matrix. Mechanical and barriers properties also improved with the addition of nanofiller up to 10 wt%.

**Babaei et al. (2014)** analyzed the effect of (3 levels of both Azodicarbonamide and nano clay) loading on the mechanical and foaming properties of wheat straw flour reinforced high-density polyethylene composites. They observed that the average cell size and cell density improves

with the addition of azodicarbonamide but simultaneously addition of both (azodicarbonamide and nanoclay) reduce the cell size and improve the cell density. Mechanical resistance also goes down with hike the content of the azodicarbonamide while raising the content of nano clay up to 2 % enhance the mechanical resistance. Water absorption and thickness swelling property also increase with the addition of azodicarbonamide.

**Trana et al. (2014)** examined the influence of alkaline and silane treatment on the mechanical and thermal properties of rice & wheat husks reinforced poly lactic acid composites. They found that the alkaline treatment removes the surface impurity (lignin, waxes, and hemicelluloses) and rise the polarity of fiber surface; therefore they have high moisture sensitivity. Silane treatment enhances the surface energy and reduces the moisture sensitivity of the husks. A composite reinforced by the husk (alkaline treatment followed by silane treatment) have more bending modulus and stresses than the composite reinforced by husks (untreated and only silane treated). From this study, they observed that this treatment improves surface adhesion in between the fiber and polymer matrix.

### 2.2.2 Effect of fiber: content, size, orientation; on mechanical and thermal properties

**Pfister and Larock (2010)** analyzed the effect of wheat straw fiber loading, size; density of the matrix, compatibilizer and molding pressure on the structure, mechanical, thermal and water absorption properties of the wheat straw reinforced conjugated linseed oil-based resin composites. Extensive improvement is observed in the mechanical and thermal properties of the composite with raising of wheat straw fiber content, a density of the matrix and the molding pressure. Mechanical characterstics can be further improved by using a compatibilizer (Maleic anhydride). They observed the significant enhancement in water absorption by increasing the fiber loading and size.

**Zou et al. (2010)** studied the consequences of wheat straw fiber length, fiber concentration and split configuration (half, quarter, and mechanically split) of fiber on the flexural and tensile behavior of the wheat straw/polypropylene composites. They reported that mechanically split fiber reinforced composites possess superior flexural and tensile properties than the entire fiber reinforced composites; this is due to the fact that split fiber has a more surface available for adhesion with the polymer matrix. They also compared these composites with the jute/polypropylene composite: it was observed that wheat straw split fiber reinforced composites have better properties than jute reinforced polypropylene composite.

**Al-Sultani (2010)** analyzed the effect of fiber content, fiber size (fine & coarse) and coupling agent (maleic anhydride) on the mechanical behavior of the wheat straw reinforced high-density polyethylene composites. They reported that highly increment in mechanical properties of composites with raising the fiber loading up to 30 %. A composite manufactured with fine fiber size shows superior properties than the composite prepared with coarse fiber size. It was observed that composite attains a maximum mechanical strength by the combination of wheat straw (30 wt %) and maleic anhydride (1.5 wt %).

**Bledzki et al. (2010b)** studied those physical, chemical and surface properties of wheat husk, rye husk, and soft wood & their polypropylene composites. They found that wheat husk and rye husk are thermally stable as low as 235°C and 210°C, respectively by the help of thermal studies. The surface of wheat and rice husk are more carbon and silicon-richer than the softwood fiber. A composite prepared by wheat husk exhibit 15 % more impact strength than the composites developed by soft wood while composite prepared by rice husk shows 110 % more elongation at break than the composites developed by soft wood.

**Zhang et al. (2011)** analyzed the mechanical, thermal and water absorption behavior of the wheat straw reinforced with urea-formaldehyde adhesives composite. They observed that particle board formed with wheat straw fiber shows an efficient mechanical strength; hence it is desirable material to make these types of board. Upon analysis through X-ray photoelectron spectroscopy, they found that urethane groups (-NH-C=O) are join with the wheat straw surface which gives a positive effect on the strength and thermal stability of composites.

**Dong and Davies (2011)** examined the effect of fiber content on the flexural properties of wheat straw/polyester composites. They compared the both experimental and calculation results and found that flexural modulus reduces by increasing of wheat straw; this is due to the fact of process-induced voids. Voids can be decreases with the help of effective treatment of the fiber and using of better processing technique for making the composites.

**Montano-Leyva et al. (2013**) studied the influence of fiber size (fiber median diameter $d_{50}$ = 1.1 mm, 62 µm and 8 µm) on the mechanical properties of the composites which are fabricated by successive grinding processes (cut milling, impact milling, and ball milling) respectively. They reported that tensile modulus and stress at break are improves while strain at break is reduces by adding the fiber content up to 11.1 Volume %. It was found that hydrophobicity of fibers can be increases by successive grinding and small fibers have a large specific area for bonding. Interfacial adhesion is better in the $d_{50}$ = (8µm > 62µm > 1.1mm) order, whereas the fiber elongation (which reflects the fiber aspect ratio) decreases in the order $d_{50}$ = (1.1mm > 62µm > 8µm). It was also proved that the net cost of composites is decreased with the addition of fibers without varying the mechanical characteristics.

### 2.2.3 Effect of compatibilizer/coupling agent on mechanical and thermal properties

**Halvarssona et al. (2008)** studied the mechanical and water absorption properties of medium density fiberboard composites which are produced by various verity of wheat straw and different resin contents (14–17 %) naming urea-melamine formaldehyde, a mixture of urea-melamine formaldehyde resin and urea melamine phenol formaldehyde. They observed that the average density is the factor which affects the properties like the internal bond, modulus of rupture, modulus of elasticity. The water absorption and material swelling properties of composites are decreases on increasing the density of straw. It was reported that by increasing the melamine content in composites implies the improvement in the water resistance and tensile properties.

**Pan et al. (2009)** examined the influence of wheat straw fiber content, size and coupling agent on the mechanical characteristics of wheat straw fiber reinforced polypropylene composites. They observed that by rising the wheat straw fiber content up to 40 % gives a higher value of tensile strength and modulus but lower value of elongation. It was found that composites made by fine particle fiber (>35 mesh) possess a little bit high value of tensile strength. Composite attained a maximum value of mechanical strength at 10 wt % maleic anhydride grafted polypropylene concentration, but magnitude of flexural modulus is remain the same.

**Kuang et al. (2010)** examined the influence of coupling agent naming (polyacrylate latex, hybrid prepared from polymethylene polyphenylene isocyanate and polyacrylate latex) and fiber loading on the mechanical and thickness swelling stability of wheat straw reinforced low density polyethylene composites. They observed that polyacrylate latex enhances the mechanical strength and reduces the thickness swelling ability of the composites. However, using the various ratios of hybrid (prepared from polyethylene polyphenylene isocyanate and polyacrylate latex) further enhances the mechanical property of the composites this may be due to the fact that coupling agent develops stiff hydrogen bonding in-between the fiber and polymer. It was found that composite exhibit maximum strength at coupling agent (polymethylene polyphenylene isocyanate/polyacrylate) ratio 30/70 while the modulus of rupture, modulus of elasticity is similar at coupling agent (polymethylene polyphenylene isocyanate/ polyacrylate) ratio 70/30.

**Reddy et al. (2010)** examined the influence of fiber content and coupling agent (maleic anhydride grafted polypropylene) on the water absorption and flexural properties of wheat straw fiber reinforced clay composites. They reported that flexural modulus and water absorption resistance improves by raising the wheat straw fiber and the coupling agent concentration. Morphological studies revealed that addition of coupling agent increases the interfacial adhesion between the fibers and polymer matrix which affected the flexural properties of composites.

**Farsi (2012)** examined the influence of fiber content and coupling agent (polypropylene grafted maleic anhydride) concentration on the mechanical, thermal, and viscoelastic behavior of the wheat straw reinforced polypropylene composites. It was reported that the tensile properties increases while impact properties reduces by the addition of fiber up to 30 %. Composite treated with coupling agent possess a better property than untreated this is due to fact coupling agent establish a strong bonding between the fiber and polymer matrix. Thermal analysis revealed that huge mass loss of composites occurs at a temperature above 400°C. Wheat straw/polypropylene composite is thermally less stable than virgin polypropylene. Dynamic mechanical, thermal analysis proved that composites having wheat straw (30 %) and coupling agent (3 %) possess high value of storage modulus.

**Obele et al. (2012)** analyzed the effect of filler content and coupling agent (maleic anhydride) on the mechanical behavior of wheat straw /polypropylene composites. They found that tensile strength, elongation at break, and impact strength of the composites are decreases while the tensile modulus rises by the addition of filler. Further improvement in these characteristics of the composite is observe by the addition of maleic anhydride.

**Liany et al. (2013)** examined the influence of (four grades of magnesium hydroxide and two grades of nano clay) content on the mechanical properties of the straw fiber reinforced high- density polyethylene composites. They found that composites burning rate is reduces with increment of nanoclay and Mg(OH). Small reduction observed in tensile and impact properties of the composite with the addition of $Mg(OH)_2$ up to 30 %. Young modulus and impact strength is increases with the addition of nano clay up to 3 %.

### 2.2.4 Effect of process parameter on mechanical and thermal properties

**Panthapulakkal and Sain (2006)** studied the effect of molding techniques, compatibilizer, fungal treatment of wheat straw and corn stem on the mechanical properties of the wheat straw/corn stem composites. They reported that higher shear compounding of wheat straw fiber composites shows similar properties to that composite produced by the milled wheat straw this is due to fiber breakage is occurring during the high shear compounding that affect the similar aspect ratio to that of milled straw. Compatibilizer and fungal treatment also enhanced the tensile and flexural properties of the wheat straw polypropylene composites.

## 2.3 NATURAL FIBER REINFORCED EPOXY RESIN COMPOSITES

Present section deals with the study of natural fiber reinforced epoxy resin composite.

### 2.3.1 Effect of surface treatment on mechanical and thermal properties

**Newman et al. (2007)** analyzed the influence of NaOH treatment on the water absorption and flexural characteristics of the tenax leaf fiber reinforced composites. This treatment is not suitable for modification of leaf fibers for enhancing the flexural properties of composites. It was reported that water absorption properties is not affected by increasing the treatment time while increases the concentration of treatment gives a positive effect on water absorption properties of composites.

**Bachtiar et al. (2008)** studied the influence of the sodium hydroxide treatment on the tensile behavior of the sugar palm/epoxy composites. They found that the hydrophilic character of the palm fiber may creates a problem in adjoining with the resin, yet they need a suitable treatment that makes the fiber hydrophobic. The tensile strength of the respective composites did not get improved significantly, yet modulus had much better value than the untreated fiber composites.

**Mylsamy and Rajendran (2011a)** investigated the influence of sodium hydroxide treatment on the mechanical and water absorption characteristics of the agave/epoxy composites. They found that sodium hydroxide treated fiber composite possess superior mechanical characteristics than untreated fiber composites. Morphological studies with SEM reported that the alkali treated fiber is more deeply linked to the polymer than the untreated fiber. They observed that sodium hydroxide treated fiber composite exhibits the low value of water absorption as compared with untreated fiber composites.

**Denise et al. (2012)** analyzed the influence of fiber loading and alkali treatment on the mechanical characteristics of the piassava fiber/epoxy composites. It was observed that by increment of fiber content in the epoxy resin gives positive effect on all mechanical characteristics of composites; subsequently properties are further improved by the treatment of fiber.

**Yousif et al. (2012)** analyzed the influence of alkali treatment on the flexural characteristics of the kenaf fiber/epoxy composites. They found that the flexural strength of the piassava fiber composites improves by increasing the untreated fiber loading. It was observed that composite made from the treated fiber possesses a higher flexural strength than the composite prepared from the untreated fiber. This is due to the fact that alkali treatment enhances the surface adhesion of the fibers and the porosity of the composites which prevents the detachments of fibers.

**Venkateshwaran et al. (2013)** determined the effect of the several concentrations of alkali treatment on the mechanical and viscoelastic properties of the banana/epoxy composites. They observed that 1% alkali treated fiber composites possess good mechanical properties. Dynamic mechanical study says that storage modulus and damping parameter are affected by the frequency.

**Lu et al. (2013)** examined the influence of sodium hydroxide and silane treatment on the mechanical behavior of the bamboo fiber/epoxy composites. It was evident from their work that alkali treated fiber composites give rise to a lesser extent to tensile and elongation at break. However, silane coupling agent treated composite gives much rise to those properties. FT-IR analysis revealed that NaOH removes the lignin present in the fiber. Fibers are split into a shorter one subsequently reduces the voids in the composites that provide the better bonding between the fiber and polymer matrix. However, silane coupling agent helps to make a new chemical bond among the fiber and polymer.

**Azwa and Yousif (2013)** investigated the effect of alkalization on the thermal behavior of the kenaf fiber reinforced epoxy composites. Thermal studies revealed that charring and thermal stability of neat epoxy composite improves by the addition of kenaf fiber. Decomposition temperature of the respective composite decreases by alkalization and produces less amount of char. Weight loss is observed due to evaporation of water and principal component at low and high temperature respectively.

**Srisuwana et al. (2014)** analyzed the influence of alkali and silanized woven sisal fiber on the mechanical characteristics of natural rubber/epoxy composites. They found that 1 wt % methyl methacrylate/glycidyl methacrylate grafted depolymerized natural rubber possesses higher impact value than the epoxy resin. Composites made by silanized sisal fiber exhibits good flexural modulus than other. 7 wt % silanized sisal fibers composite improves the impact strength upto 230 % in comparison with grafted depolymerized natural rubber/epoxy resin blend composite.

**Kumar et al. (2014c)** examined the mechanical and thermal properties of the untreated and alkali treated coconut sheath fiber composites. They reported that composites made by alkali treated coconut sheath fiber possess good mechanical and thermal properties compared to a composite prepared by untreated (raw) coconut sheath fiber. Morphological studies with SEM proved that treated fiber composite shows good fiber–matrix adhesion and fewer voids compared to the untreated fiber composite.

**Liu et al. (2014)** investigated the influence of silane treatment and mercerization on the thermal properties of abaca fiber/epoxy composites. They found that both treatments positively affect the bonding in between the fiber and polymer. The treated fiber composite has better thermal conductivity and less void content. The transverse thermal conductivity of composites are enhances by the suitable chemical treatments of the fiber this is due to fact treatment changes the cell wall structure of the same.

**Fiore et al. (2015)** investigated the influence of alkaline treatment on the mechanical behavior of kenaf fibers reinforced with the epoxy composites. They examined that composite fabricated by the alkali treated fiber have more modulus than neat polymer. It was reported that the alkali treated fiber composites contributes the reduction of damping factor peaks shifting them to higher temperatures.

**Anbukarasi and Kalaiselvam (2015)** examined the impact of fiber shapes, mass of fiber and alkali treatment on the mechanical, thermal and water absorption behavior of luffa/epoxy composites. They found that 40 % treated fiber composite have superior mechanical strength than untreated fiber composites. Morphological studies revealed that treated composite have a better interface bonding compared to untreated fiber reinforced composite.

### 2.3.2 Effect of fiber: content, size, orientation; on mechanical and thermal properties

**Sapuan et al. (2006)** analyzed the tensile and flexural characteristics of the banana fiber woven composites by varying the direction of fiber woven. They reported that tensile strength and modulus of the composite have slightly more value in the x-direction(fiber orientation) than the y-direction.

**Low et al. (2007)** determined the influence of the fiber loading on the mechanical behavior of the cellulose fiber/epoxy composites. It was observed that raising the content of cellulose fiber in the polymer matrix enhances the mechanical properties. The breakup of the microstructure of the composite specimen occurs on the application of the mechanical tests (tensile, flexural) due to the fiber pull out from the polymer.

**Liu and Hughes (2008)** analyzed the influence of fiber density, weave configuration, and stacking sequence on the fracture and toughness properties of woven flax/epoxy composites. They observed that fracture strength and toughness of the composite proliferate by increases the fiber density. Composite toughness is mainly dependent on the fiber content.

**Leman et al. (2008)** studied the moisture content and Fickian diffusivity constant of the sugar palm fiber/epoxy composites. They observed that the composite made with the 20 wt% fiber content has highest moisture absorption having a value of Fickian diffusivity constant ($3.76*10^{-7}$ $mm^2/s$). It was concluded that moisture content is highly attributed to the presence of fiber loading.

**Newman (2009)** examined the water diffusivity of the weave flax fiber reinforced composites. It was reported that water uptake of flax epoxy composites is higher compared to e-glass fabric reinforced polyester composites. However, flax composites become swollen after the water absorption and on drying; the fiber shrinks faster than the matrix.

**Yousif et al. (2010)** studied the effect of particle size on the wear and frictional behavior of a betelnut fiber (treated) composite. They reported that the frictional coefficient of coarse particle-fiber composite is having large value while the abrasive wear can be mainly attributed to particle size and sliding velocity. The specific wear rate for the composite possesses the following order in terms of size: coarse > grain > fine.

**Rosa et al. (2010)** examined the effect of fiber orientation on the mechanical and thermal characteristics of the phormium tenax fiber/epoxy composites. They observed that mechanical properties of the quasi-unidirectional fiber composite were better compared to the epoxy resin. Short fiber reinforced composite is also better mechanical strength than the same. Phormium tenax fiber/ epoxy composite exhibits more thermal stability than fiber and epoxy resin separately.

In another study **Mylsamy and Rajendran (2011b)** analyzed the influence of the fiber length on the mechanical characteristics of the agave fiber composites. They found that composite made with the alkali treated short fiber (3mm) possess superior mechanical properties. Dynamic mechanical analysis proves that the alkali treated fiber has a high damping factor in the low-temperature region and low damping factor in the high-temperature region.

**Alamri and Low (2012a)** studied the effect of fiber loading on the mechanical and water absorption characteristics of recycled cellulose fiber/epoxy composites. They reported that significant enhancement in the mechanical properties(flexural strength, impact strength and fracture toughness) of composites by increment the fiber content. However, the maximum value is achieve at 46 wt % fiber loading. It was also reported that composite possess higher water absorption on increasing the fiber loading. After water uptake of the composite, all mechanical properties got reduces except impact strength.

**Romli et al. (2012)** examined the effect of fiber content, period of curing and compression load on the tensile behavior of the coir/epoxy composites. They reported that the tensile characteristics of the composites are mainly affected by the fiber content and curing time while a load of compression has no effect on it.

**Nirmal et al. (2012)** investigated the influence of fiber orientation on the wear and fractional properties of the bamboo fiber/epoxy composites. They noticed that anti-parallel orientation of the bamboo fiber composite has a good adhesive wear performance than any other orientation. Frictional performance is also better for an anti-parallel orientation of the bamboo fiber composite at low sliding velocity.

**Muralidhar (2013)** analyzed the mechanical characteristics of the flax-rib knitted/ epoxy composites. Their research made them realize that knitted composite has more tensile strength in the wale direction compared to course direction. The elastic modulus of flax-rib knitted fiber composite depends on the thickness and number of perform layers.

**Mishra and Biswas (2013)** examined the response of fiber volume fraction on the mechanical strength of the jute fiber/epoxy composites. They reported that composite void content is reduce by the addition of fiber up to a certain limit. The composite exhibits superior flexural and inter-laminar shear strength at 48 wt % fiber content this is due to less void content at this point.

**Coroller et al. (2013)** studied the effect of fiber content on the tensile properties of the flax fiber reinforced epoxy composites. Morphological studies with SEM found that the developed composite has a proper dispersion of fiber and less number of fiber bundles. The value of tensile modulus of respective composites is proving that the rule of mixture prediction. It was used the Rosen model to relate the experimental data with the evaluated data.

**Gupta and Srivastava (2014)** analyzed the tensile and flexural behavior of unidirectional and mat from sisal fiber/epoxy composite. They observed that 30 wt% unidirectional sisal fiber composites possess high tensile and flexural properties.

**Kumar et al. (2014a)** analyzed the influence of fiber loading on the mechanical characteristics of wood dust/epoxy composites using the Taguchi method. They reported that load and tensile strength are affected by the speed of conducting the test while flexural properties mainly correspond to the fiber loading. In another study **Kumar et al. (2014b)** determined the effect of fiber content on the mechanical characteristics of wood dust reinforced epoxy composite. They reported that 10 % fiber content composites exhibit good mechanical properties than the other ratio of fiber content.

**Mahjoub et al. (2014)** examined that the effect of fiber loading on the mechanical behavior of kenaf fiber/epoxy composites. They have found that the Rule of Mixture (ROM) analytical model validates the tensile property data of the respective composites which was obtained from the experimental results. Composite made with 40 % fiber content exhibits better value of tensile, flexural and impact strength.

### 2.3.3 Effect of compatibilizer/coupling agent on mechanical and thermal properties

**Shih (2007)** analyzed the influence of the coupling agent on the mechanical and thermal behavior of the bamboo fiber/epoxy composites. They observed that fibers treated with coupling agent exhibits superior adhesion with the polymer than the raw fibers. The thermal stability also enhances by the fiber increment in a polymer matrix. Composite developed with treated fiber exhibits a better mechanical characteristic than the untreated fiber composites.

### 2.3.4 Effect of various additives on mechanical and chemical properties

**Biswas and Satapathy (2010)** examined the loading of red mud on the mechanical characteristics of the bamboo fiber/epoxy composite and equated with the same set of glass/epoxy composites. They reported that the mechanical strength of bamboo fiber composite is smaller than the glass reinforced epoxy composites while wear properties are superior to the glass/epoxy composites. Both properties are further enhanced by the addition of red mud content in both composite.

**Mohan and Kanny (2011)** analyzed the influence of nano-clay on the mechanical and water absorption behavior of the sisal fiber reinforced epoxy composites. The tensile and wear characteristic of composite slightly reduces after the water absorption of the composites. It was reported that composite made with the 5 wt % nano-clay possess low water absorption characteristics. However, these mechanical properties slightly enhances by increasing the nano-clay content.

**Benjamin et al. (2011)** examined the influence of lignin on the mechanical properties of the hemp/epoxy composites. They observed that composite shows good impact strength on the addition of 5% w/w lignin. Modulus of elasticity and the flexural modulus of the composite found to be increases on raising the concentration of lignin up to 2.5 wt %.

**Alamri et al. (2012)** investigated the influence of clay concentration on the mechanical and thermal behavior of the recycle cellulose fiber/epoxy composites. They found that the mechanical strength (toughness and impact) of composite increment on the addition of clay up to 1 wt % in cellulose fiber/epoxy matrix. The addition of nano-clay causes increases the thermal stability of the respective composite and also improves the char residue over neat epoxy.

**Hoyos and Vazquez (2012)** examined the effect of environmental condition, addition of montmorillonite and alkali treatment on the flexural characteristics of fique fiber/epoxy composites. They found that composite achieves good flexural properties with the treated fiber and montmorillonite. The flexural value of fique fiber composite is also better than the wood.

**Alamri and Low (2012b)** determined the effect of nano-silicon carbide particles on the mechanical and water absorption characteristics of cellulose fiber/epoxy composites. They reported that use of 5 wt % nano-silicon carbide causes a positive effect on the flexural properties and fracture toughness of the composites. Nano-silicon carbide increases the adhesion at the interfaces of fiber and polymer. It was found that water absorption of composites decreases by the addition of nano-silicon carbide particles. Composite possess low flexural properties and fracture toughness after the water absorption.

**Ahmed et al. (2012)** determined the effect of the silicon carbide and $Al_2O_3$ content on the wear properties of the jute/epoxy composites. They observed that composite exhibits a superior wear resistance after the addition of silicon carbide and $Al_2O_3$ individually. However, a composite which having a $Al_2O_3$ possess good wear resistance than composite containing silicon carbide.

**Gabr et al. (2014)** studied the effect of the loading of cellulose acetate on the thermal and surface morphology behavior of the electrospun nano-cellulose/epoxy composites. They observed that thermal and fracture resistance of nanocomposites made with cellulose acetate is better than nanocomposites prepared without cellulose acetate.

**Subagia et al. (2014)** analyzed the influence of tourmaline micro/nanoparticle content on the mechanical characteristics of basalt fiber/epoxy composites. They found that 1 wt% tourmaline loading with surfactant gives a superior value of tensile and flexural properties compared to neat basalt/epoxy composite. This is due to the presence of tourmaline which provides more surface interaction between the fiber and polymer.

**Vijaykumar et al. (2014)** analyzed the influence of the filler loading on the mechanical behavior of the kenaf and coconut spathe fiber/epoxy composites. They found that composite attributes good mechanical properties up to a certain limit of filler content, thereafter the same properties are reduces due to lack of bonding between the fiber and polymer.

### 2.3.5 Effect of process parameters on mechanical and thermal properties

**Ali et al. (2010)** analyzed the influence of the aging on the mechanical properties of the arenga pinnata fiber composites. They reported that the tensile properties of the aged composite possess more value than those of the original composite specimen while impact value of the aged composite is significantly similar to that of the original.

**Scida et al. (2013)** examined the effect of hygrothermal aging on the mechanical and damage behavior of flax-fiber/epoxy composite. They observed that tensile strength of the composite is mainly impacts on the hygrothermal aging; this is due to the reorientation of micro-fibrils of the used fibers. A water-saturated composite faces a reduction in tensile properties. It was found that breakage of fiber in the polymer matrix reduced due to hygrothermal aging.

**Gu et al. (2014)** investigated the effect of fiber content and pre-compaction operation (vacuum pressure and temperature) on the mechanical characteristics of ramie fabric/epoxy composites. Extensive improvement is observe in mechanical strength of the composite (fabricated at hot compaction) with raising of ramie fiber content. But on exceeding the temperature and pressure of hot compaction beyond certain values, the mechanical properties of ramie fiber reinforced composite were found to be dwindling.

### 2.3.6 Other considerations

**Amor et al. (2010)** studied the dielectric characteristics of the palm tree composites. They reported that ionic relaxation occurred due to the high mobility of electric charges and this can be attributed to the presence of cellulose fibers in the composite.

**Bonnafous et al. (2012)** analyzed the experimental and mathematical analysis of the hemp fiber reinforced epoxy composites. They developed a finite element model to simulate the fragmentation process of the respective composite. The model has a strain criterion based on the experimental characterization of the single hemp yarn behavior and uses an optimized tangential frictional contact at the interface. It was reported that numerical simulations were closely related to the experimental fragmentation patterns.

**Ataollahi et al. (2012)** examined the effect of composite length on the absorption energy and the compression failure of the silk reinforced epoxy composites. They found that peak load and average load values are much different in all specimens which lead to low crash force efficiencies. A lengthier composite specimen exhibits a higher value of energy absorbed and takes more time to reach the compaction zone. However, short length tubes have the highest value of absorbed specific energy.

**Oshkovr et al. (2012)** determined the influence of length and thickness of the composite tubes on the absorbed energy of natural silk/epoxy composite. They reported that composite fabricated with a 30 layer of silk and epoxy shows the highest value of total absorption energy. However, crash force efficiency is the highest for a 12 layer of silk and epoxy composite at the same length.

**Yan and Chouw (2013)** investigated the influence of crashworthiness behavior on the energy absorption of flax fiber/epoxy composite tubes. They reported that crushing energy absorption rises with increasing the number of plies at the same inner diameter and length of the specimens. It was found that, it is mainly dependent on the dimension of the tube.

**Eshkoor et al. (2013)** determined the effect of triggered and non-triggered on the energy absorption of silk reinforced epoxy composites. They observed that specific energy absorption of composite reduces on raising the composite specimen length in both triggered and non-triggered tubes. However, total energy was found to be improved on increasing the composite specimen length in both triggered and non-triggered tubes. Non-triggered specimens have twice peak load compared to triggered specimens. The non-triggered composite specimen shows catastrophic failure while progressive failure possess by the triggered specimen.

**Vasconcellos et al. (2014)** studied the effect of low-velocity impact and impact damage on the quasi-static tensile and fatigue properties of woven hemp fiber/epoxy composites. They found that impacts leads to damage at the fiber interface and micro-cracks in the matrix. It was used analytical model to calculate the residual fatigue life of the composite for any low-energy impact level.

**Guen et al. (2014)** examined the influence of polyol on the vibration damping behavior of flax fiber/ epoxy composite. They observed that composites made by polyol-treated flax have larger damping coefficients than composites made by the non-treated flax and carbon-fiber composites at standardized conditions.

**Liang et al. (2014)** examined the fatigue property of flax fiber/epoxy composites. As per their results, composites possess more fatigue resistance having more static strength. The gain in the longitudinal Young's modulus of composites depends on the ratio of parallel to load direction of the fiber.

**Yan et al. (2014a)** investigated the effect of tube thickness, tube diameter, and content of polyurethane-foam on the crushing and energy absorption capacity of flax fabric/epoxy composite tubes. They reported that polyurethane-foam filled composite tubes spearheaded tendency for deformation. By using polyurethane-foam filled composite tubes, the energy absorption of the tubes got enhances during the flattening process. The polyurethane-foam filled composite tubes shows superior crashworthiness compared to the empty tubes. It was reported that the specific energy of the empty and foam filled flax/epoxy tubes in lateral crushing is significantly lower than that in axial crushing. In another study **Yan et al. (2014b)** examined the response of foam content, thickness of the tube, triggering on the crushing property and energy absorption capacity of flax-reinforced epoxy composite tubes. The composite containing foam exhibits good value of all: crush force efficiency; more total; and specific absorbed energy. However, triggering has no significant effects on the composites properties in the absence of foam. A composite tube developed with (triggered and foam-filled) has better characteristics compared to the composite tube made either of foam or triggered.

**Duigou et al. (2014)** investigated the real fiber surface that participates in the practical adhesion with the two types of flax fiber (Hermes and Electra) reinforced epoxy composites. They stated that adhesion determined by micro bond tests between Hermes (Shear strength = 22.5 ± 1.5 MPa) and Electra (Shear strength = 13.2 ± 3.2 MPa) fiber composites. Flax fiber varieties/epoxy systems cannot be explained by their superficial surface chemistry using X-ray Photoelectron Spectroscopy. It was found that the total participating surface will be a repercussion of the overall area where the resin and fiber are in contact, i.e. the total area penetrated by resin.

**Kandare et al. (2014)** investigated the fire reaction properties of flax-reinforced epoxy sandwich composites. They observed that no significant variation in the time-to-ignition by the addition of the glass fiber veil with ammonium polyphosphate onto the composite surfaces took place. It was reported that the glass fiber veil reduces the thermal damage of underlying substrate composites with the help of through-thickness temperature profiles across the sandwich composite.

In order to determined the influence of energy absorption on the woven natural silk reinforced epoxy composite **Eshkoor et al. (2014)** used a trigger mechanism. They reported that specific energy absorption value of composite reduces by increasing the composites length. However, total energy absorption rises by improving the length of the composites.

**Landro and Janszen (2014)** analyzed the permeability, rheological and thermal properties of the hemp/epoxy composites. Their work concluded that laminated composites are produce using vacuum assisted resin transfer molding without any difficulty. Upon analysis through vibration tests, mechanical characteristics of the composite were found to be directly linked to the density and the damping properties.

From the above discussion, there are so many studied done by the various researchers in several research articles. It has been found that the many of them done there research in the field of mechanical behavior of the natural fiber reinforced composites. Some people worked on the thermal stability of the natural fiber based composites. So, we have found that there is no work available of acrylic treated bagasse fiber reinforced with the epoxy resin and alkali treated wheat straw fiber reinforced with epoxy resin. Therefore, we trying to fill this research gap. As far as blend, no study has been reported so far on a blend of wheat straw and bagasse reinforced epoxy composites.

# 3

# MATERIALS AND METHODS

Presents chapter deals with the materials, methods, and analytical procedures adopted during the experimental studies for the development and characterization of blended natural fiber based composite along with bagasse and wheat straw fiber reinforced with epoxy.

## 3.1 MATERIALS

This section contains the details of the natural fiber: bagasse and wheat straw; polymer: epoxy resin; surface modifier: sodium hydroxide and acrylic acid.

### 3.1.1 Bagasse Fiber (BF)

There are various varieties of sugar cane grow abundantly in northern and southern parts of India. To produce sugar from sugarcane it is crushed in mill having big rollers arranged in consecutive order. Crushing attributes to breakage of cane stalk into minor pieces and juice used to spread out by the succeeding milling. The juice is gathered and processed for sugar production. The leading crushed and squeezed cane stalk, called bagasse, is considered to be a by-product of the milling process (Elsunni and Collier, 1996). Bagasse is basically a waste product that causes mills to incur extra disposal costs. Presently, it is used as a renewable natural fiber for the development of composite materials. Wet milled bagasse contained about 50% moisture, 47% fibrous material, 1.5% sugar, and little amount of ash (0.5%).

The sugar cane residue was collected from a local sugar mill, Saharanpur, Uttar Pradesh (India). BF is sieved from the sugarcane residue for the separation purpose. After separation, the moisture content of fiber is found to be 6–7%. The length of BF varied from 5 to 10 mm with an average of 8 mm. The fibers are washed with distilled water to remove any unwanted adhering impurities, followed by drying in a hot air oven at 70 ° C for 48 h to remove moisture. Properties of bagasse fiber are listed in table 3.1.

**Table 3.1: Properties of bagasse fiber**

**(Hattallia et al., 2002; Hoareau et al., 2004; Marti-Ferrer et al., 2006)**

| **Properties** | **Bagasse fiber** |
|---|---|
| Cellulose (wt %) | 55.2 |
| Hemi-cellulose (wt %) | 16.8 |
| lignin (wt %) | 25.3 |
| Diameter (μm) | 10-34 |
| Density (g/cm$^3$) | 1.5 |
| Elongation at break (%) | 3-10 |
| Tensile strength (MPa) | 20-290 |
| Young's modulus (GPa) | 17 |

**Figure 3.1: Bagasse fiber**

### 3.1.2 Wheat Straw Fiber (WSF)

Wheat straw is a farming by-product of the dry stalks of wheat plants, after the removal of grain and chaff. Straw makes up about half of the yield of cereal crops such as wheat. This is one of the most significant agricultural residues. It is an annually renewable fiber resource which is available in huge quantity in various area of the world. Wheat straw is similar to wood and could also be viewed as a natural composite material. It consists primarily of cellulose, hemicellulose, and lignin (White and Ansell, 1993).

Wheat straw fiber was picked up from a local agricultural farm, Meerut, Uttar Pradesh (India). The moisture content of fiber is found to be 5-6 %. The length of WSF varied from 8 mm to 15 mm with 12 mm of an average length. The fibers are washed with distilled water to remove undesirably adhered impurities, followed by drying in an oven at 70°C for 48 h for the moisture removed. Properties of WSF are listed in table 3.2.

**Table 3.2: Properties of wheat straw fiber**

**(Hattallia et al., 2002; Hoareau et al., 2004; Marti-Ferrer et al., 2006)**

| **Properties** | **Wheat straw fiber** |
|---|---|
| Cellulose (wt %) | 38-45 |
| Hemi-cellulose (wt %) | 15-31 |
| lignin (wt %) | 12-20 |
| Density (g/cm$^3$) | 1.2 |
| Diameter (µm) | 5-7 |
| Tensile strength (MPa) | 32 |
| Young’s modulus (GPa) | 6.6 |
| Elongation at break (%) | -- |

**Figure 3.2: Wheat straw fiber**

### 3.1.3 Epoxy resin and curing agent

Owing to high strength, toughness, and resilience; the epoxy resin (AW106) and the curing agent (HV953IN) are taken. When both are mixed, they give excellent resistance to chemical and moisture attack, with outstanding electrical insulating properties (Tripathi and Srivastava, 2009; Mitra, 2010; Yadav and Srivastava, 2014; Majumdar et al., 2005). They can be cured at ambient temperature without any pressure by using a curing agent (Srivastava et al., 2008). It can be easily bonded with all material like wood, glass, and natural fibers, metal. They exhibit little or no shrinkage after curing. It has a density 1.1 g/cm$^3$. This is procured from M/S Petro Araldite Pvt. Limited, Manali, Chennai, India. Properties (MSDS) of epoxy resin are listed in table 3.3.

**Table 3.3: Properties of epoxy resin**

| **Appearance** | **A clear pale yellow liquid** |
|---|---|
| Specific gravity at 25$^{o}$C (g/cm$^{3}$) | 1.12 |
| Solid Content (%) | 84 |
| Tensile Strength(MPa) | 31 |
| Flexural Strength (MPa) | 67 |
| Impact strength (Kg/m$^{2}$) | 9 |

### 3.1.4 Surface modifier

The use of the surface modifier is to enhance the surface properties of the fibers that are used in making of the composites. It cleans the fiber surface and improves the fiber adhesion.

#### 3.1.4.1 Sodium hydroxide

Another name of sodium hydroxide is caustic soda having chemical formula NaOH. This is a white solid organic which is having base and alkali, salt. It is available in flakes, pellets, granules, and as prepared solutions at a number of dissimilar concentrations. Drops of sodium hydroxide solutions can decompose proteins and lipids in the skin, eyes or other living tissues. It may cause permanent blindness while in contact with eyes.

In the present study, it is used for the purpose of the fiber surface treatment. This treatment is disturbed the hydrogen bonding in the fiber structure, thereby increasing surface roughness. It also removes a certain amount of lignin, wax, and oils covering the external layer of the fiber surface. It (Min-assay 98%) is procured from M/S Himedia Laboratories Pvt. Limited, Mumbai, India. Properties (MSDS) of sodium hydroxide are listed in table 3.4.

**Table 3.4: Properties of sodium hydroxide**

| **Molecular formula** | **NaOH** |
|---|---|
| Molar mass | 39.9971 g mol$^{-1}$ |
| Appearance | White, waxy, opaque crystals |
| Solubility in water | Miscible |
| Density | 2.13 g/cm$^{3}$ |
| Boiling point | 1388 °C |
| Melting point | 318 °C |

### 3.1.4.2 Acrylic acid

Acrylic acid is the unsaturated carboxylic acid. It has a chemical formula $CH_2$=CHCOOH. It consists of a vinyl group which is joined with a carboxylic acid. It is a colorless liquid and has an acrid or tart smell. It is miscible with ethers, water, chloroform, and alcohols. If contact with the skin, it behaves as irritating and corrosive. Eye contact may result in terrible and irreversible injury.

In this study, it is used for the purpose of removal of sodium hydroxide, which is stuck due to the surface treatment of the fiber. Acrylic acid (Min- assay- 99%) is procured from M/S Loba Chemie Pvt. Limited, Mumbai, India. Properties (MSDS) of acrylic acid are listed in table 3.5.

**Table 3.5: Properties of acrylic acid**

| **Molecular formula** | $\mathbf{C_3H_4O_2}$ |
|---|---|
| Molar mass | 72.06 g $mol^{-1}$ |
| Appearance | Clear, colorless liquid |
| Solubility in water | Miscible |
| Boiling point | 141 °C |
| Flash point | 68 °C |
| Density | 1.051 g/ml |
| Melting point | 14 °C |

## 3.2 METHOD USED

This section contains the detail of the procedures adopted during the experimental studies on the development and characterization of bagasse/wheat straw reinforced epoxy composites.

### 3.2.1 Untreated bagasse fiber

Bagasse fiber was washed with distilled water to remove any unwanted adhered impurities, followed by drying in hot air oven at 70°C for 48 h for the removal of excess water. The dried fibers are assigned as untreated fibers.

### 3.2.2 Untreated wheat straw fiber

Wheat straw fiber was rinsed with distilled water to remove any undesirably adhered impurities, followed by drying in an oven at 70°C for 48 h for the removal of excess water. The dried fibers are assigned as untreated fibers.

### 3.2.3 Treatment of the bagasse fiber

Bagasse fiber was soaked with 1% sodium hydroxide (NaOH) for a time of 30 min, followed by a 1% acrylic acid for a time of 1 h at ambient temperature (Li et al., 2004a). The fibers are washed with distilled water to remove any unwanted adhered impurities, followed by drying in hot air oven at 70 $^{O}$C for 48 h. The liquor ratio was maintained at 20:1 during the process.

### 3.2.4 Treatment of the wheat straw fiber

Wheat straw fiber was soaked in each 1, 3, and 5% NaOH solution at ambient temperature for 2 h, (Cao et al., 2006) followed by washing with distilled water to remove withdraw any NaOH solution adhere to the wheat straw fiber further dried in an air oven for 48 h at the temperature of 70 $^{O}$C.

### 3.2.5 Preparation of composites

Epoxy resin (AW106) and curing agent (HV953IN) are mixed as per the recommendation by manufacturer followed by addition of various weight fractions of the untreated fiber and treated fiber. The contents are stirred in a mixer for 10 to 15 min at a rotational speed of 2000 rpm to get a homogenous mixture. The mixture is poured into the mold having a size of 300 X300 X10 mm$^3$ and cured for 24 h at ambient temperature. All composite materials made for this research is manufactured using the Hand lay-up technique (Mishra and Acharya, 2010a). An overview of the experimental setup is shown in figure 3.3. In order to avoid the problem of sticking of composite material, the mold has been coated with Teflon sheet, which may lead to a problem of formation of the bubble. To overcome such problem, roll over the sheet with a heavy roller is incorporated. Every composite cast is cured under a load of 25 kg for 24 h before it removed from the mold. After 24 h the samples are taken out from the mold, cut into different sizes (as per the ASTM standard) and kept in an airtight container for further experimentation.

Figure 3.3: Experimental set up(Mixing chamber and Mold)

## 3.3 CHARACTERIZATION OF COMPOSITES

Prepared composites sheet needs to be characterized. Characterization process can be divided into following:

### 3.3.1 Mechanical properties

Study of the mechanical properties of the composite material is very significant because most of the applications require the mechanical loading. Selection of composite material under loading is based on the tensile strength, tensile modulus, flexural strength, flexural modulus and impact strength. Three specimens are tested in each type of composite and values are taken as the average of three specimens. In all characteristics, due care is taken to check the reproducibility of data.

#### 3.3.1.1 Tensile test

The tensile test determines the ability of the material to withstand the force that tends to pull apart the specimen and the extent to which specimen stretch before breaking. Tensile modulus is a denotation of the relative stiffness of the material and can be obtained from the slope of the stress-strain plot.

Tensile strength and tensile modulus are determined by numerically as follows.

Tensile strength = Force/ Cross section area = F/A

Tensile modulus = Tensile stress/ Tensile strain= $\frac{F/A}{\Delta L/L}$

Where,

F = Force exerted on a specimen under tension

A= cross-sectional area of specimen through which force is applied

$\Delta$L = the amount by which the length of the specimen changes

L = original length of the specimen

The tensile strength and tensile modulus of the composites are evaluated with a 2716-002 Instron Model 5982. The specimen is shown in figure 3.4 and overview of a machine is presented in figure 3.5. This property is determined in accordance with the ASTM D3039 procedure at a cross head rate of 2 mm/min. The dimension of a tensile specimen is 250 mm long and 25 mm wide of the material which is cut from a composite sheet of the respective material.

Universal testing machine requires a constant rate of movement. The machine consists of one grip which is in stationary mode and a second grip having movable mode. A load indicating mechanism capable of indicating the tensile load is used with good accuracy. An extensometer is also used to find out the distance between the two points placed within the gauge length of the test sample when the sample got stretched.

Procedure for conducting a tensile test

- The specimen is tightened vertically in the grips of the machine (figure 3.6), it should not slip.
- The dimension of the specimen given to the machine, testing speed is set and the machine is started.
- Specimen start to elongate, resistant of the specimen is recovered by the load cell.
- Elongation of the specimen continued until the breakup occurs.
- Finally display of machine shown a value of tensile strength, tensile modulus, stress vs strain curve, elongation etc.

Figure 3.4: Specimen for a tensile test

Figure 3.5: Universal Testing Machine (2716-002 Instron Model 5982)

**Figure 3.6: Specimen is tightened vertically in the grips of the machine**

#### 3.3.1.2 Flexural test

Flexural strength is the ability of the material to withstand the deflecting force applied perpendicular to its longitudinal axis, a stiffness of the material can also obtain from this test. Flexural modulus is the ratio of stress to strain in flexural deformation, or it is the tendency of a material to bend. It is determined from the slope of a stress-strain curve produced by a flexural test.

Flexural strength and flexural modulus (3-point loading system, Figure 3.7) are determined by numerically as follows.

Flexural strength =$3FL/2wh^2$ , Flexural modulus = $L^3F/4wh^3d$

Where,

F = Force at the fracture point , L= is the length of the support span, w = width of the specimen, h = thickness of the specimen, d = deflection due to the load applied at the middle of the specimen

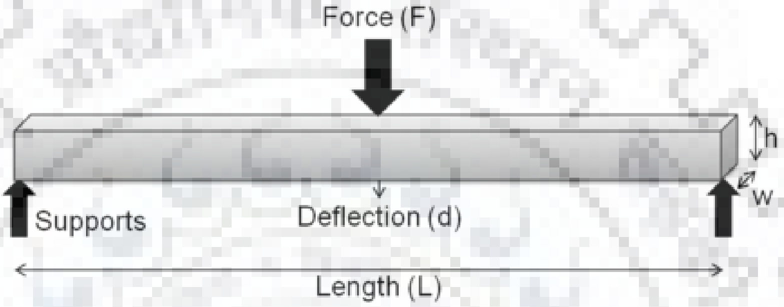


**Figure 3.7: Pictorial depiction of flexural measurement**

Flexural property of the respective composites is evaluated with a 2716-002 Instron Model 5982(Figure 3.5). The flexural properties are determined in accordance with the ASTM D790 (Kaushik et al., 2006; Kaushik et al., 2010b) procedure respectively at a cross head rate of 2 mm/min. The dimension of flexural specimen is 100 mm long and 25 mm wide of the material which is cut from a composite sheet of the respective material and specimen shown in Figure 3.8.

**Procedure for conducting a flexural test (3-point loading system)**

In a 3-point flexural test, the area of uniform stress is quite small and concentrated under the center loading point.

- The specimen bar rests on two supports (figure 3.9) and loaded by means of loading nose midway between the supports.
- Loading nose and the support must have the cylindrical surfaces to avoid the stress concentration.
- The dimension of the specimen given to the respective machine and load is applied to the specimen at the specified speed.

- Specimen start to deform, resistant of the specimen is recovered by the load cell.
- Bending of the specimen continued until the breakup occurs.
- Finally display of machine shown a value of flexural strength, flexural modulus, Stress vs strain curve etc.

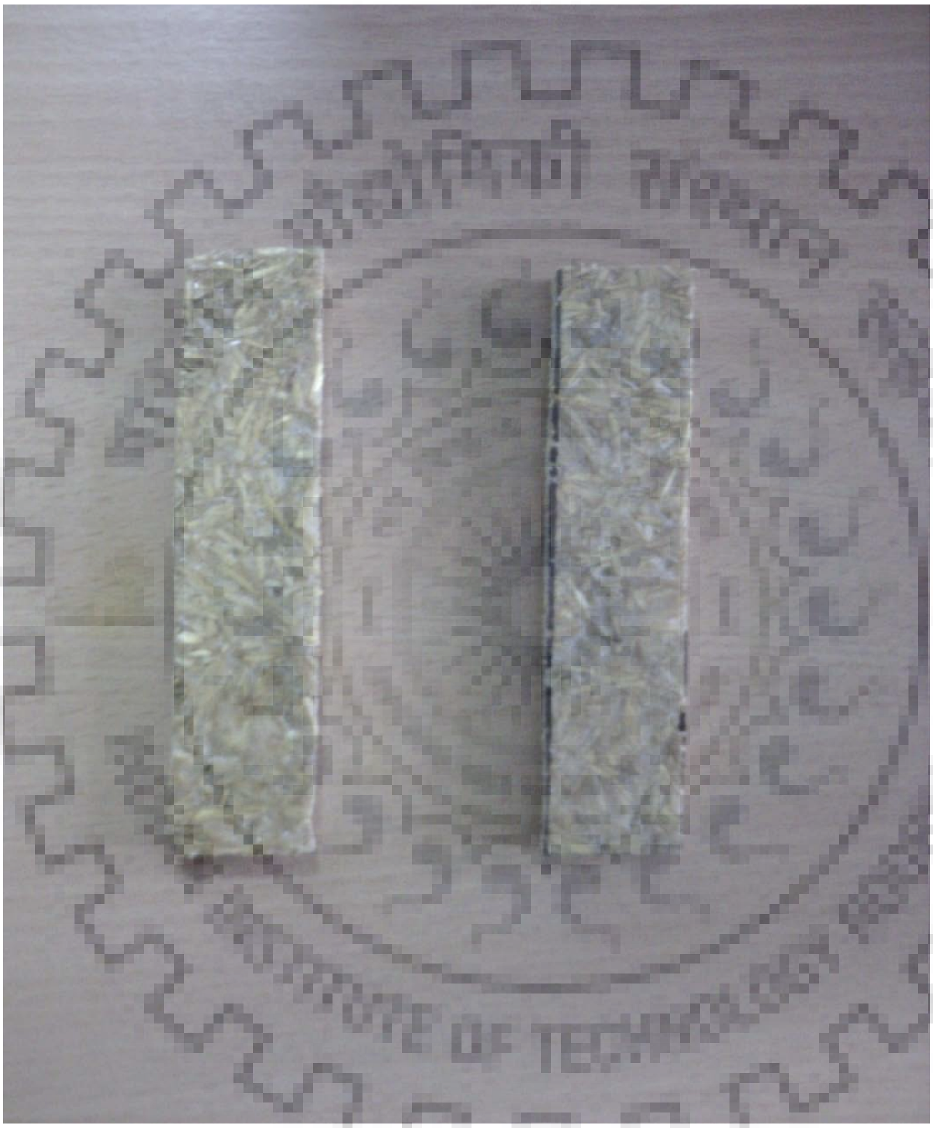


**Figure 3.8: Specimen for a flexural test**

**Figure 3.9: Specimen bar rests on two supports**

### 3.3.1.3 Impact test

The impact test is performed to assess shock absorbing capacity of materials subjected to suddenly apply the load. The impact strength of the material is associated with the toughness of the material. It is the ability of the material to absorb the applied energy. It can be defined as the ability to resist the fracture under the stress, which is applied at high impact.

The impact strength is calculated using an impact testing machine (TINIUS OLSEN Model impact 104) and the hammer having 4 kg weight (Figure 3.10). Izod impact strength is determined using ASTM standard D256. The test specimen is having a dimension 63.5 mm in length and 12.7 mm in the breadth of the material which is cut from a composite sheet of the respective material and shown in figure 3.11.

Procedure to conduct impact test

- The specimen should be tightened vertically in the grips of the machine.
- The dimension of the specimen given to the machine and lock the striking hammer in its position.

- Now, press down the pendulum release over lever so that the hammer strikes the test specimen and breaks it.
- Finally display of machine shows value of impact strength.

**Figure 3.10: Impact testing machine (TINIUS OLSEN Model impact 104)**

**Figure 3.11: Specimen for an impact test**

## 3.3.2 Thermal analysis

Thermal analysis of the composite materials is as important as the mechanical and chemical analysis. Fiber content, surface treatment, crystallinity, molecular orientation, surface and bonding between the fiber & polymer matrix phase have a considerable effect on the thermal properties of the composite material. These tests are essential for the quality control and characterization of these materials. These are three main thermal analysis of method of composite material are as follows:

### 3.3.2.1 Thermo Gravimetric Analysis (TGA)

In TGA analysis, the variation in the respective sample weight is determined while the sample is heated at a constant rate of temperature or constant rate of time under air or inert gas atmosphere. This is a very effective quantitative analysis of thermal reaction accompanying by weight changes due to the evaporation, dehydration, and degradation.

### 3.3.2.2 Differential Thermal Gravimetric (DTG)

In DTA analysis, the differential of temperature plotted against time, or against temperature. This technique gives knowledge about the temperature at which high rate of degradation take place.

#### 3.3.2.3 Differential Scanning Calorimetry (DSC)

Differential scanning deal with thermal transitions of the composite that may be either first or second order. On the application of heat, thermal transition takes place in polymer composite. The first order transition affects the changes in heat capacity and latent heat of the material. Second order transition affects only changes in heat capacity. The plot can be drawn between heat flow vs temperature.

All thermograms are taken from SII 6300 EXSTAR in a nitrogen atmosphere (flow rate 200ml/min) at a constant heating rate of 10 °C/min from ambient to 800 °C temperature. Composite weighing between 8 and 12 mg is used.

### 3.3.3 Water absorption analysis

Performance of natural fiber composites is dependent on factors such as property of the portions and their interface compatibility. One of the main disadvantages of natural fibers used in composite is that it absorbs more moisture compared to neat polymer resin. Many researchers observed that cellulose and hemicellulose are mainly responsible for the more water uptake of natural fibers.

Water absorption property of respective composites specimen is calculated using ASTM D570. The test specimen is having dimension 76.2 mm long and 25.4 mm wide of the material which is cut from a composite sheet of the respective material as shown in figure 3.12. The percentage of the water absorption in the composites is calculated by the weight difference between the sample immersed in distill water and dry sample, use the following equation

$$\% \mathrm{M} = \frac{\mathrm{MF}-\mathrm{MI}}{\mathrm{MI}} * 100$$

Where,

% M is water absorption,

MF is the weight after immersion in water,

MI is the dry initial weight.

Procedure to conduct water absorption test

- Specimen should be free from dust, oil and other impurity.
- The test specimen to be immersed in the container for a given period of time under standard condition.

- Proper weighing of respective specimen at same interval.
- Put the determined value in the respective formula and plot a curve between water uptake vs time period.

**Figure 3.12: Specimen for a water absorption test**

### 3.3.4 Scanning Electron Microscope (SEM)

A scanning electron microscope (SEM) is a type of electron microscope that develops images of a sample by scanning it with a focused beam of electrons. The electrons interact with the atoms present in the given sample, producing several signals that can be detected and that contain data about the sample's surface topography and composition.

SEM morphology provides the information about the change occurs on the surface of the material. This study is conducted using LEO 435 VP with following specifications: acceleration voltage up to 30kV, magnification range (10X-300,000X), detection mode (Secondary and backscattered electron) and use of printer and camera to record images. The test sample is mounted on apparatus after fusing with silver gel and coating with gold to avoid electrical charging during examination period. The line diagram of SEM is shown in figure 3.13.

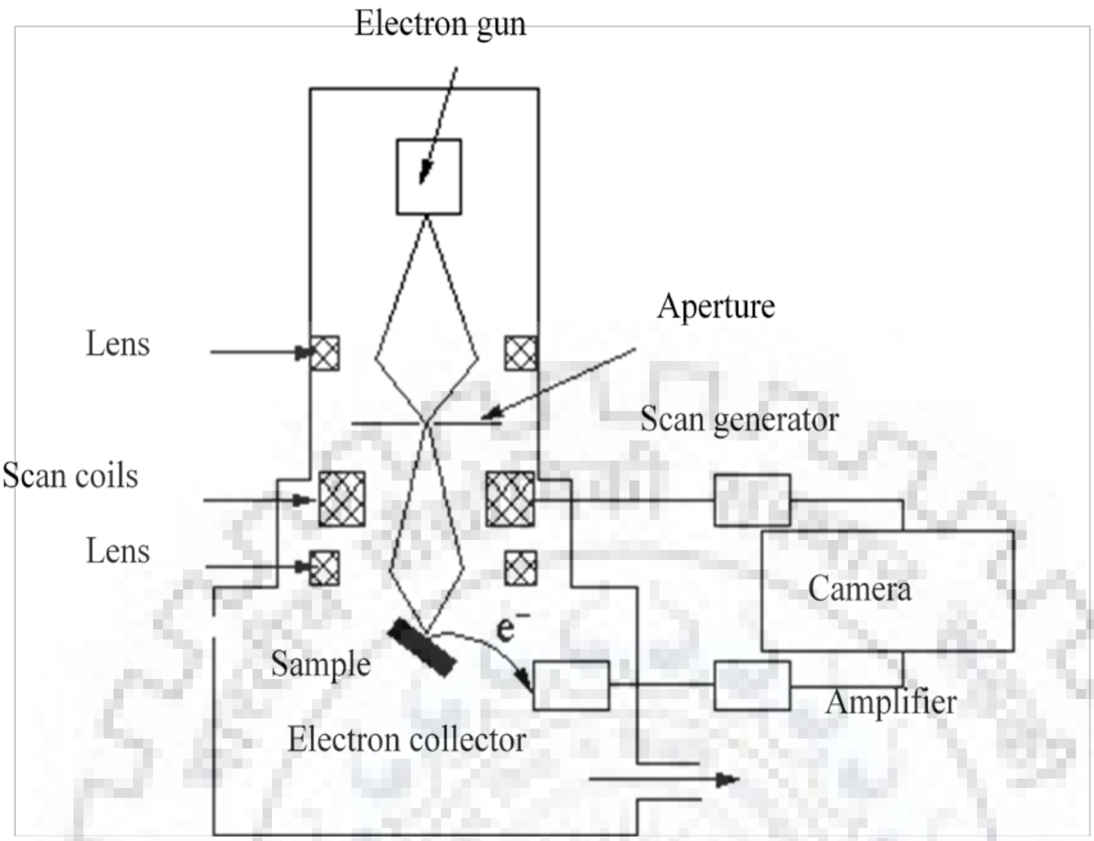


Figure 3.13: Line diagram of Scanning Electron Microscopy (SEM)

### 3.3.5 Fourier Transform Infrared Spectroscopy (FT-IR)

Fourier transform infrared spectroscopy (FT-IR) is a technique which is used to obtain an infrared spectrum of absorption, emission, photoconductivity of a solid, liquid or gas. FT-IR spectrometer picks up spectral data in a wide spectral range at the same time.

It is used to analyze any changes in the functional group of the cellulose fibers after chemical treatment. Fiber sample and KBr are mixed in the ratio of 1:10 is pressed into a disk for (FT-IR NICOLET 6700) spectroscopy measurement. The spectra are recorded with 32 scans in the frequency range of 4000–400 cm$^{-1}$ with a resolution of 40 cm-1. Line diagram of FT-IR spectrophotometer is shows in figure 3.14.

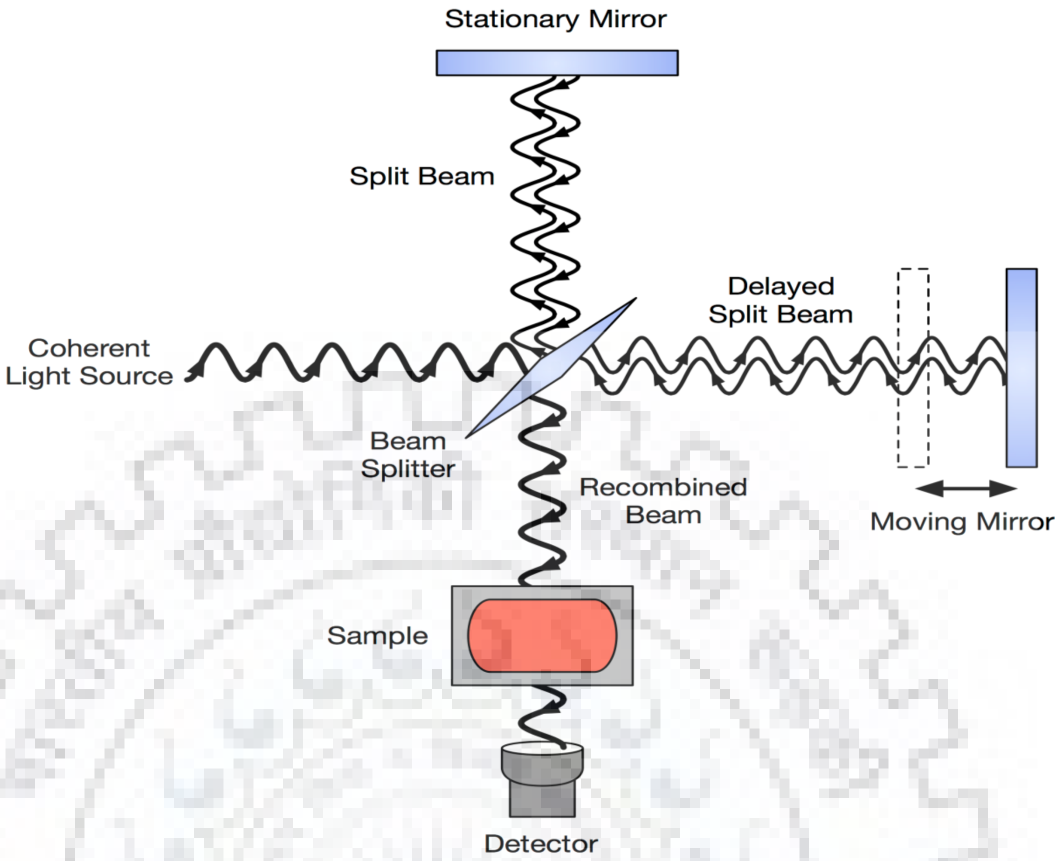


**Figure 3.14: Line diagram of Fourier Transform Infrared Spectroscopy (FT-IR)**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# 4

# RESULTS AND DISCUSSION

## 4.1 EFFECT OF FIBER CONTENT AND CHEMICAL TREATMENT ON MECHANICAL AND WATER ABSORPTION PROPERTIES OF BAGASSE FIBER-REINFORCED EPOXY COMPOSITES

This section deals with the mechanical and water absorption properties of bagasse fiber reinforced epoxy composites. In order to enhance the bonding strength between the fiber and polymer, Bagasse Fiber (BF) is subjected to chemical treatment with 1 % sodium hydroxide followed by 1 % acrylic acid at ambient temperature. This is an order to prompt the high value of mechanical properties and reduces the water absorption properties of the composites. The optimum value of fiber content is analyzed and it has been found that 15 % treated fiber loading gives enhanced mechanical properties in addition reduction in water absorption aspects. The scanning electron microscopy (SEM) was used to observe the surface morphology of the BF.

### 4.1.1 Mechanical analysis

The average values of tensile strength, tensile modulus, flexural strength, flexural modulus and impact strength of BF-epoxy composites are shown in figure 4.1-4.5 respectively. The experimental results show that the tensile, flexural and impact properties are remarkably increased in fiber content varies from 0 to 15 wt %. However, beyond 15 wt % of fiber loading, there is a notable reduction in mechanical properties of composites. The high fiber content in the composite lead to poor fiber-matrix adhesion thereby prompted the formation of microcrack not only at the interface but possesses a non-uniform stress transfer. This is because of agglomeration of fiber within the matrix. For high concentration of fiber (25 and 30 wt %), the polymer matrix is distributed uneventually which lead to the lumping of BF, resulted the poor adhesion at the interface. To improvise this, the chemical treatment process has been carried out. It is observed that the chemical treatment improves the tensile, flexural and impact properties of the composites. All chemically treated BF composites possess higher mechanical

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properties than those of untreated BF composites as shown in the respective figures. The tensile strength of the treated BF composite at the optimum point (15 % BF) content is 22.4 MPa against 18.3 MPa for the untreated one, there is an improvement of 22 % as compared to the untreated BF composite. Similarly, the flexural strength of the treated BF composite is 55.8 MPa against 46.9 MPa for untreated BF composite, which lead to an improvement of 18 % in flexural strength and the impact strength of the treated BF composite is 36.35 J/m against 34 J/m for untreated BF composite, which lead to an improvement of 7 % in impact strength as compared with the untreated fiber at that point (Mittal and Sinha, 2015a). The most important modification achieved with chemical treatment is the disturb the hydrogen bonding which present in the fiber structure, thereby increases the surface roughness. Acrylic acid treatment also modifies the surface of the fiber and makes them more hydrophobic because it coats the OH group of fiber which responsible for their hydrophilic character (Mohanty et al., 2001b).

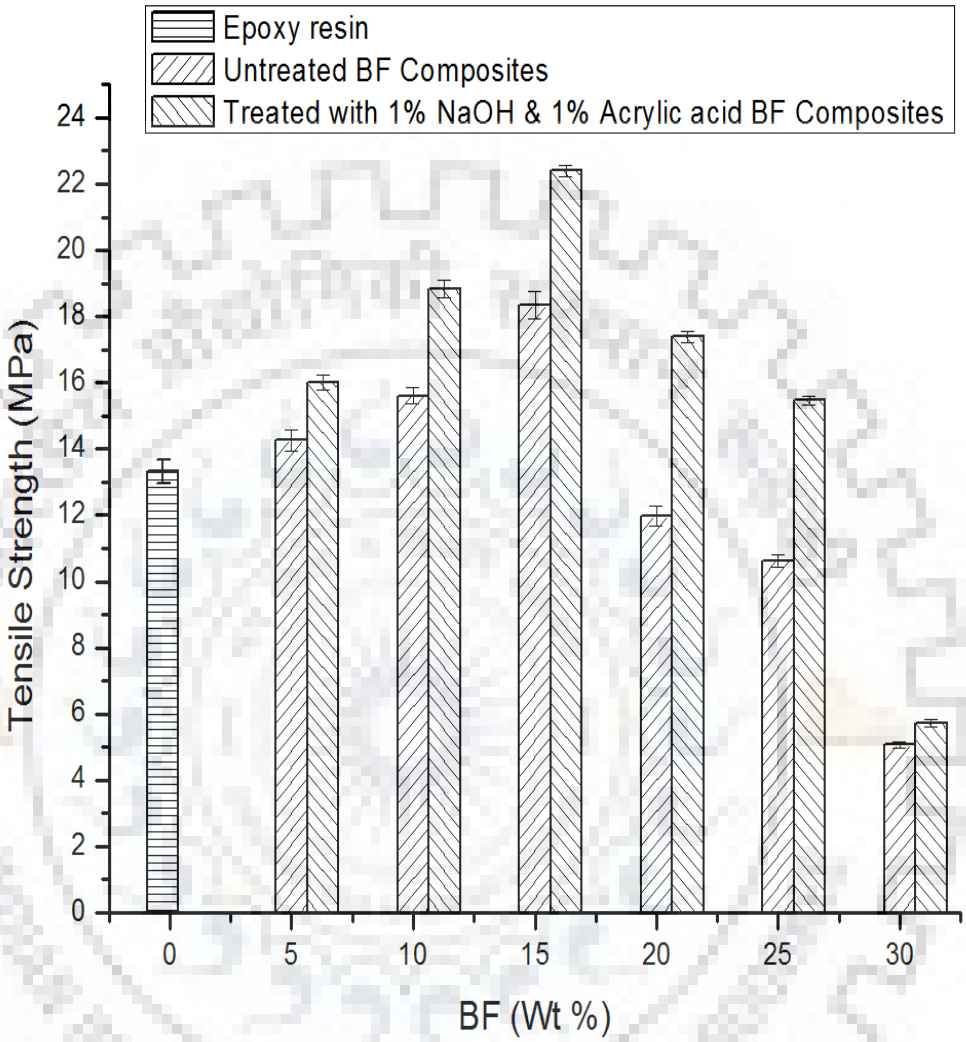


Figure 4.1: Effect of fiber content and chemical treatment on the tensile strength of BF-epoxy composites

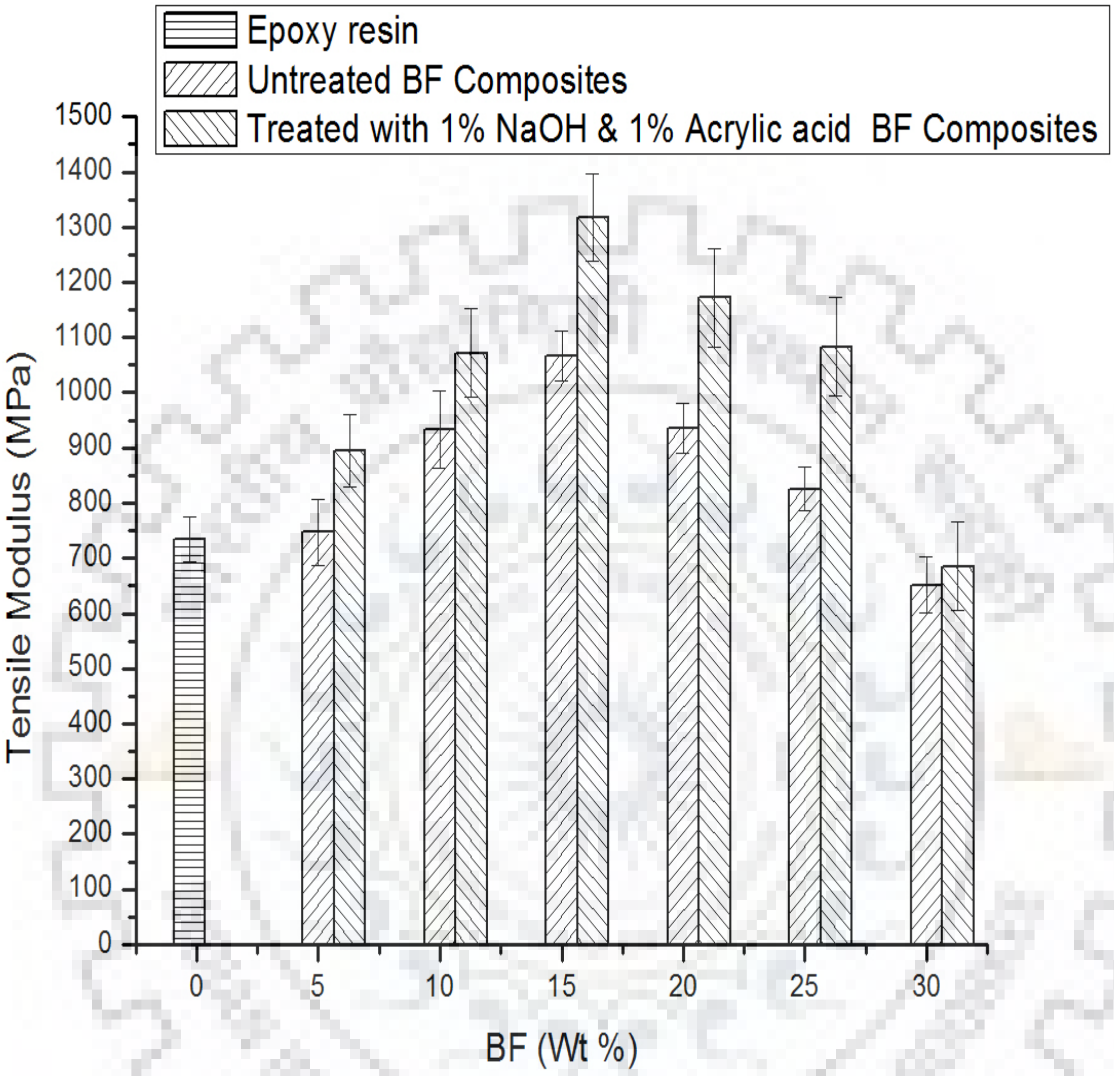


**Figure 4.2: Effect of fiber content and chemical treatment on the tensile modulus of BF-epoxy composites**

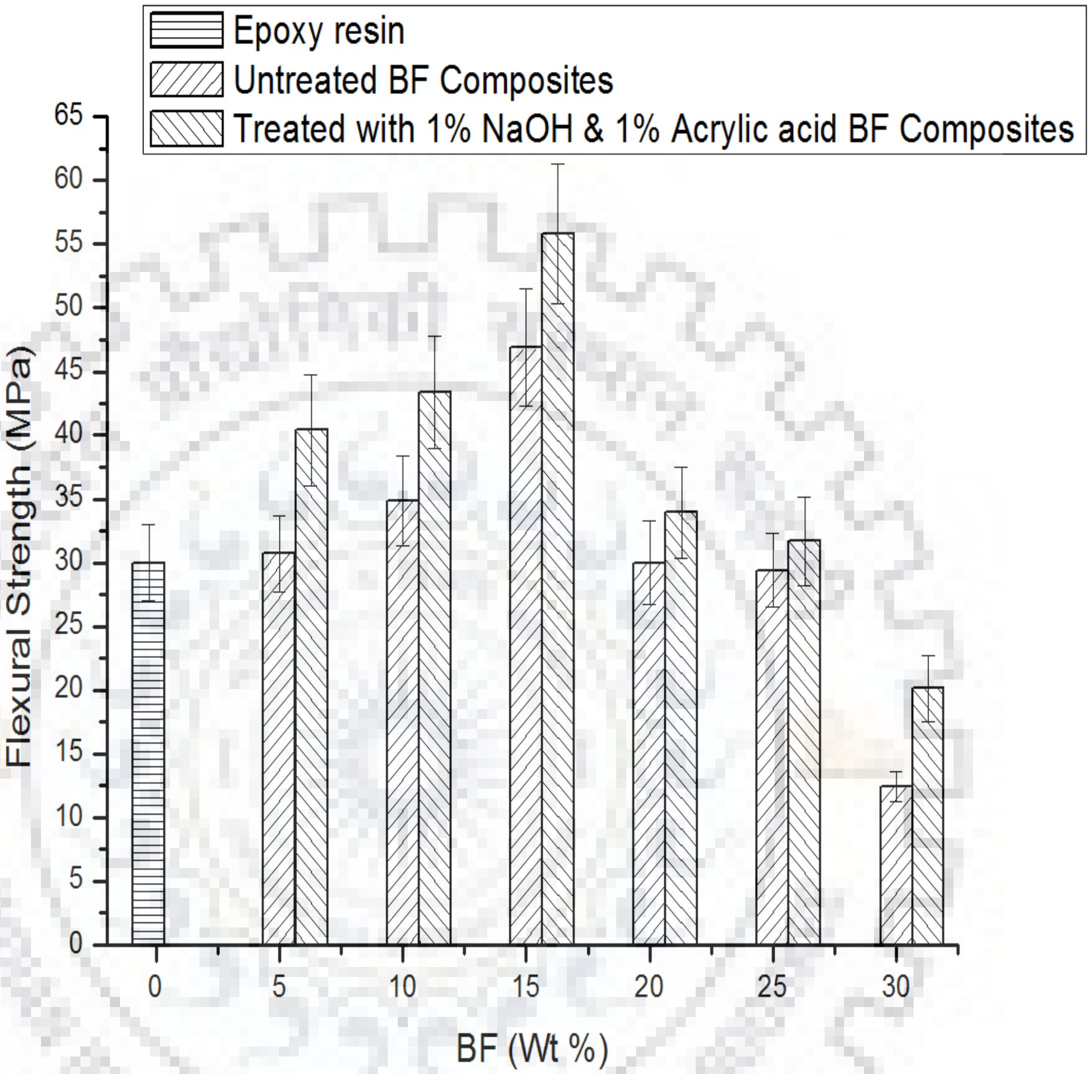


**Figure 4.3: Effect of fiber content and chemical treatment on the flexural strength of BF-epoxy composites**

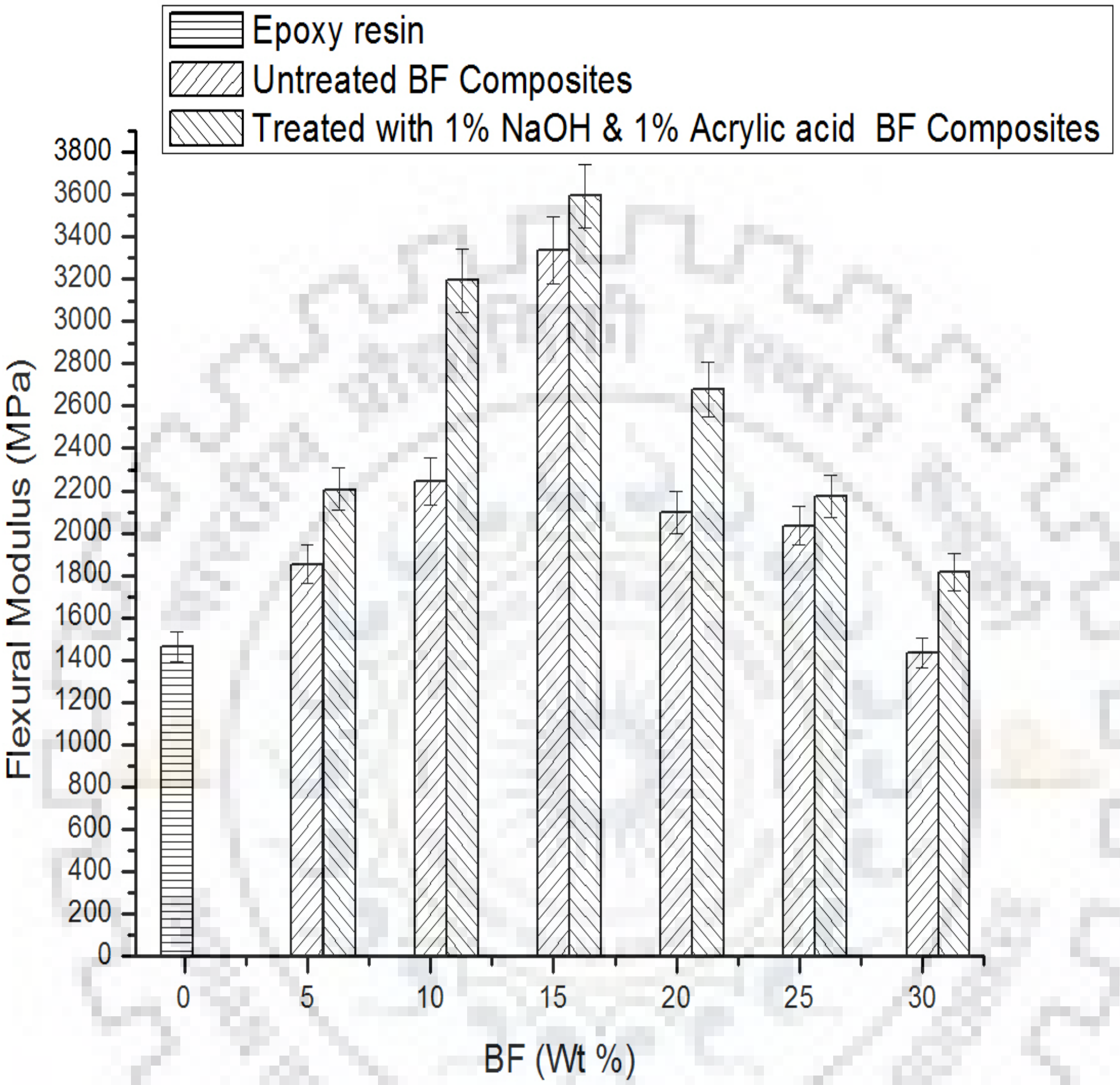


**Figure 4.4: Effect of fiber content and chemical treatment on the flexural modulus of BF-epoxy composites**

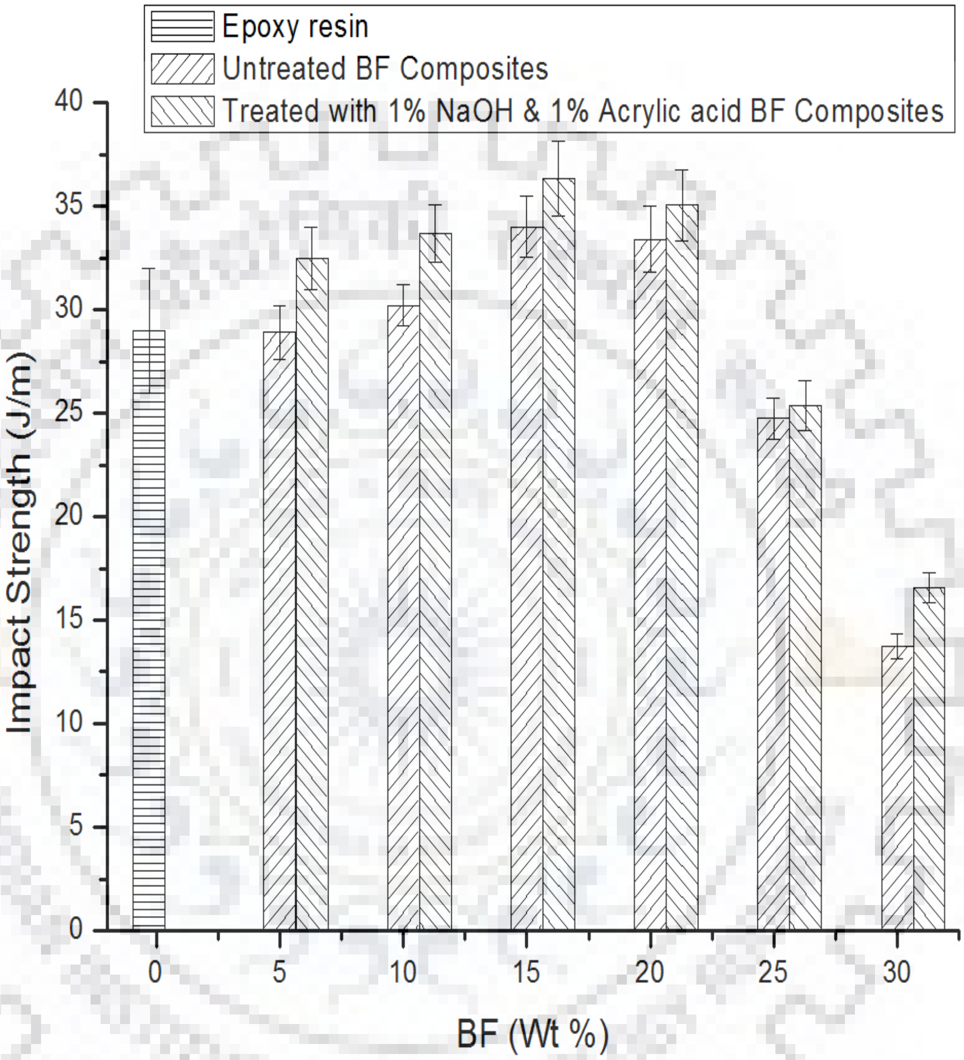
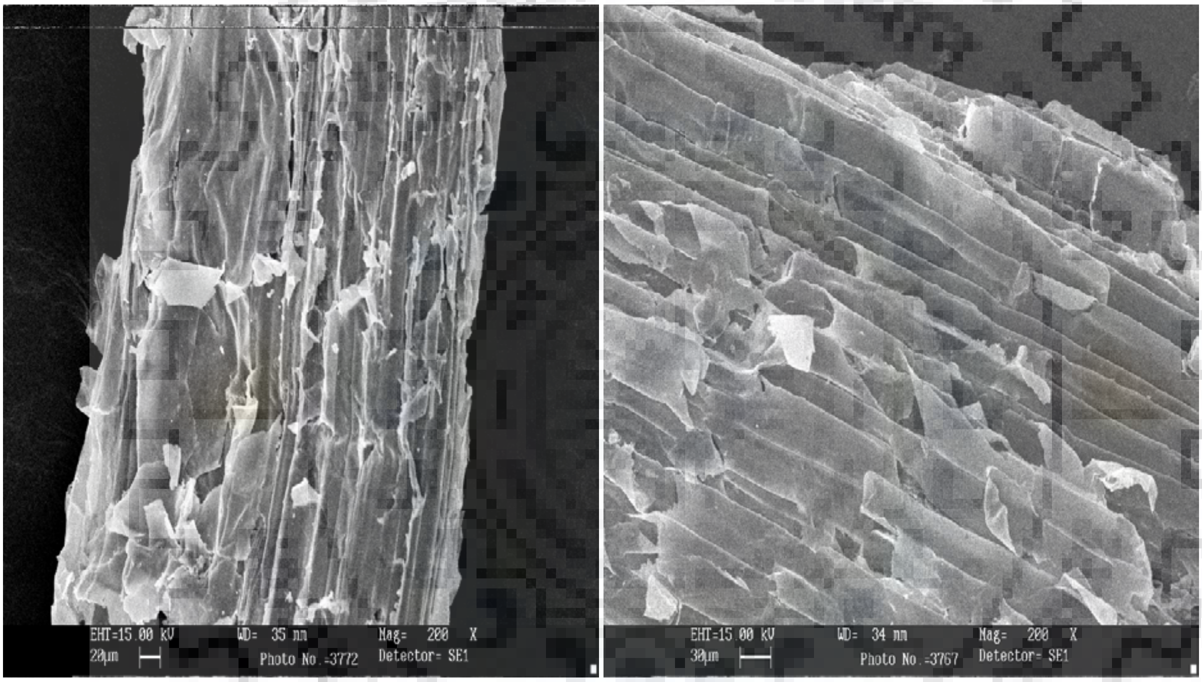


**Figure 4.5: Effect of fiber content and chemical treatment on the impact strength of BF-epoxy composites**

### 4.1.2 Scanning Electron Microscopy (SEM)

In order to analyze the morphological behavior, SEM has been carried out. Figure 4.6(a) represents the untreated fiber having bundles of fibrils and wax present on the surface that reduces the bonding between the fiber and the polymer.

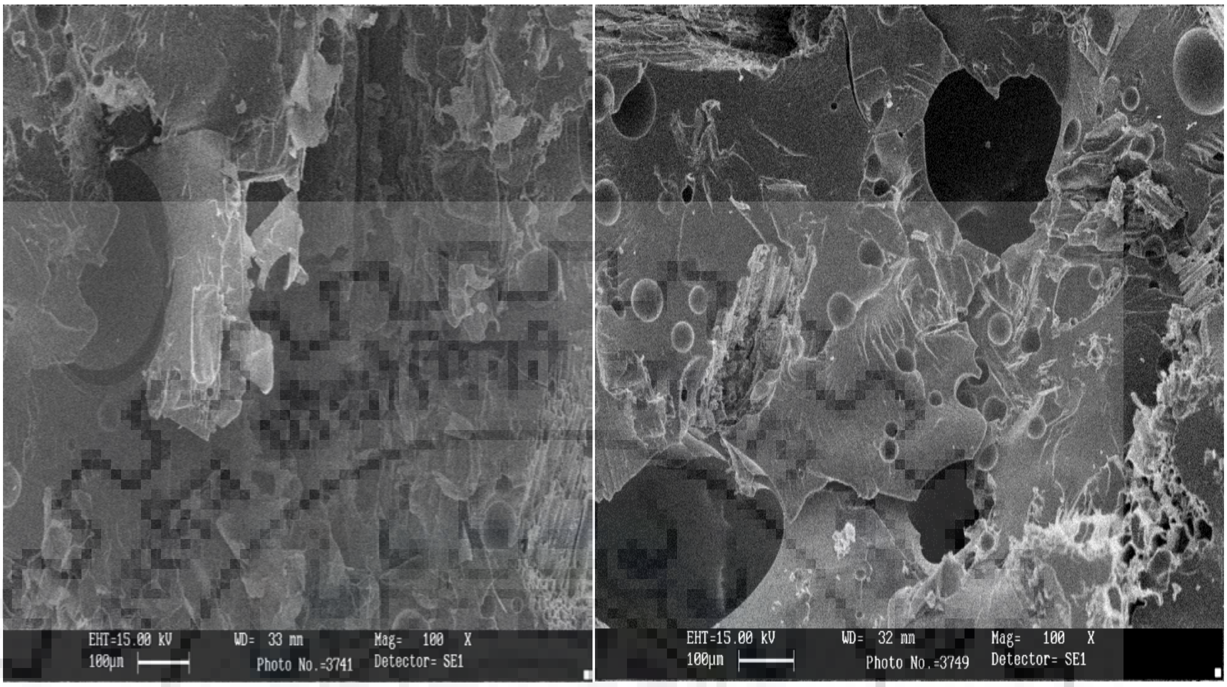
The morphological behavior of treated fiber is represented in figure 4.6(b), after the chemical treatment, the bundle of fibrils is disturbed lead to neat surface of the fiber thereby more surface area available for bonding. Hence, chemically treated fiber is deeply attached to the polymer matrix which provides superior mechanical strength of the composites (Mohanty et al., 2003).



(a) (b)

**Figure 4.6: SEM morphology a) untreated BF b) treated BF with 1% NaOH followed by 1 % acrylic acid**

Figure 4.7(a) and 4.7(b) represents the difference between the morphology of the tensile fractured surface of untreated and the treated fiber composites respectively. It has been observed that the treated BF-epoxy composite microstructure has a deep pit on the fractured surface that enhances contact area of bonding between fiber & polymer matrix, therefore, they provide a better strength than the untreated BF-epoxy composite. It has been observed that the morphological changes in the microstructure are dominant when the treated bagasse fiber particle is added to the polymer matrix this gives superior mechanical properties than the untreated one (Saw and Datta, 2009).



(a) (b)

**Figure 4.7: SEM morphology a) tensile fracture surface of untreated BF (15 wt %) composite b) tensile fracture surface of treated BF (15 wt %) composite**

### 4.1.3 Water absorption analysis

Figure 4.8(a) shows the percentage intake of water absorbs in untreated BF reinforced composites. It is evident that the natural fiber composites intake higher water absorption than pure epoxy resin. By increasing the fiber content, water absorption also increases due to the fact that lignocelluloses material absorbs more water. On the other side, it has been observed that water absorption in the BF composites is decreased by the chemical treatment as shown in figure 4.8(b). At the optimum point (15 % BF) treated BF composites are absorbed 8 % water while 12 % of the untreated BF composite for 10 days. Hence, chemical treatment slightly decreases the water absorption characteristic of the composites.

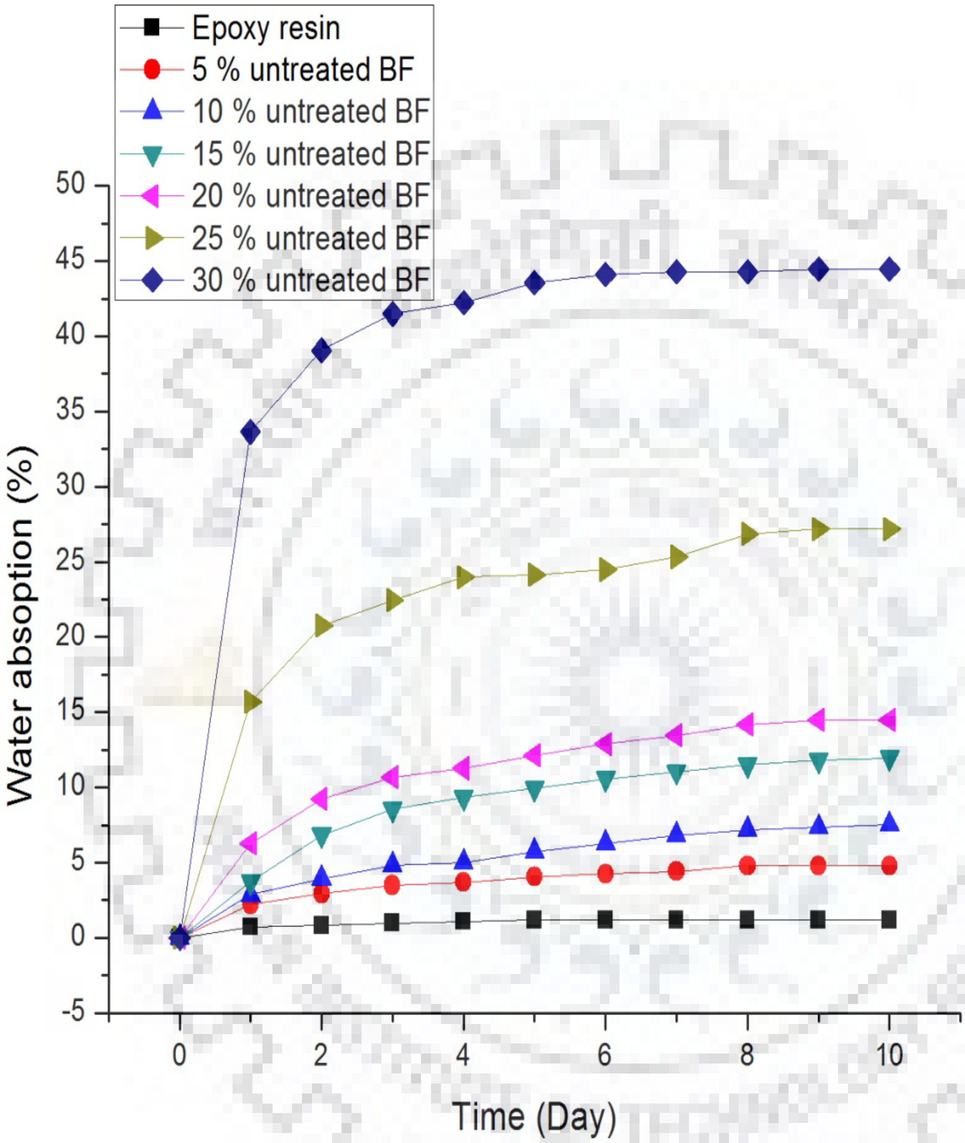


**Figure 4.8(a): Water absorption curve for untreated BF-epoxy composites**

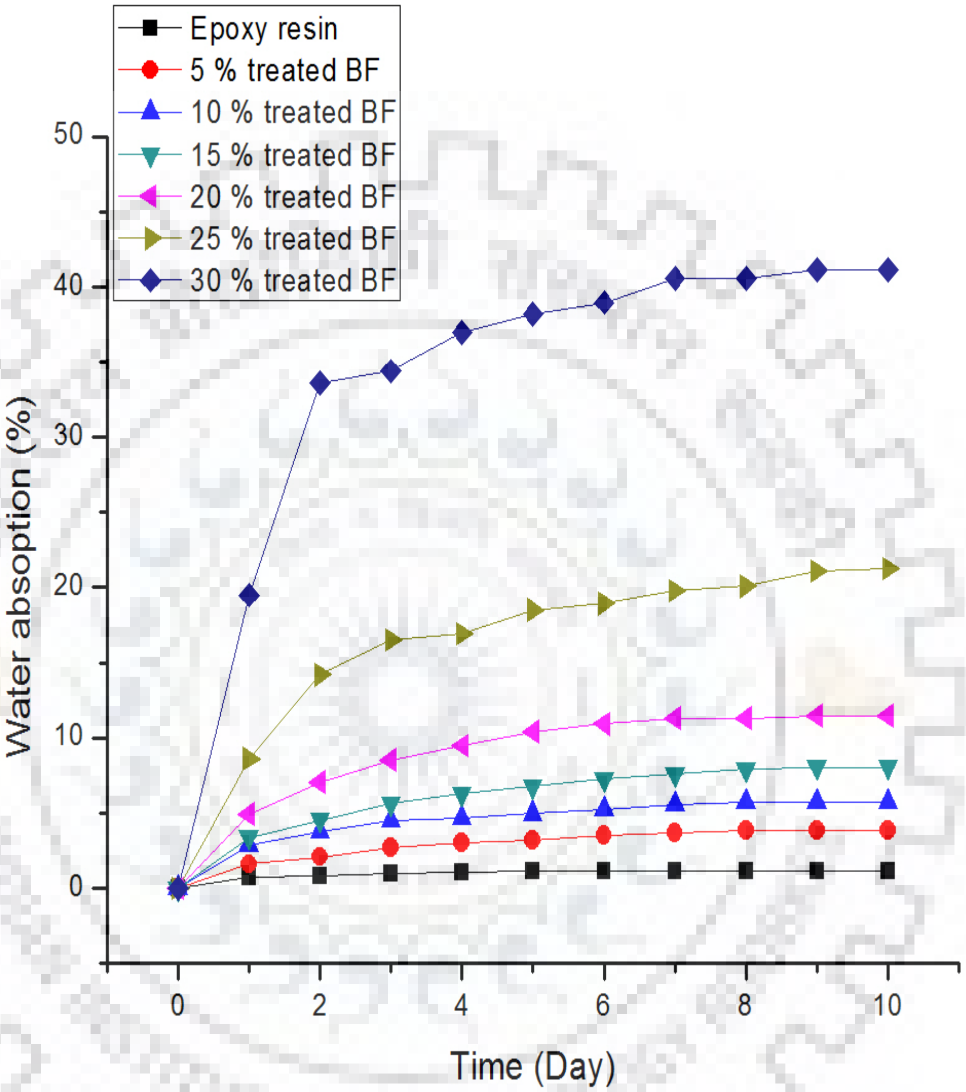


Figure 4.8(b): Water absorption curve for treated BF-epoxy composites

## 4.2 EFFECT OF FIBER CONTENT AND CHEMICAL TREATMENT ON THERMAL PROPERTIES OF BAGASSE FIBER-REINFORCED EPOXY COMPOSITES

In order to analyze the thermal behaviour, Thermo Gravimetric Analysis, Differential Thermal Gravimetric and Differential Scanning Calorimetry has been carried out. Thermal analysis results of treated BF-epoxy composites are compared with untreated one. The chemical treatment of BF induces reasonable changes in the thermal stability of the polymer composites. The slight variation is observed when the treated BF-epoxy composites are compared with the untreated one, which confirms the connection between the presences of the peaks.

### 4.2.1 Thermo Gravimetric Analysis (TGA) and Differential Thermal Gravimetric analysis (DTG)

Thermo gravimetric analysis and Differential thermal gravimetric thermograms of (neat epoxy, untreated BF and treated BF) are shown in figure 4.9 and 4.10 respectively. In TGA thermograms, there are three different zones of degradation. In the first zone (at temperature range 80-100°C) there is a small loss in weight due to the removal of moisture present in the sample. Because of the onset of the thermal decomposition process, a very high loss in weight in the mid zone of degradation and the last zone having negligible weight loss that is called the ultimate thermal degradation.

The TGA behavior of neat epoxy is shown in figure 4.9 (A). A single step decomposition process observed for neat epoxy. It is observed the degradation started at about 200°C. The range of the middle zone is 340-440°C of epoxy that has higher rate of weight loss. From figure 4.10 (A) the higher rate of weight reduction is (0.75 mg/min) observed at about 435°C due to degradation and the degradation of the remaining part of the product for the further degradation observed at 650°C. The residue left after final degradation is 1.1%.

Thermo gravimetric behavior of untreated BF and treated BF is shown in figure 4.9 (B and C) respectively. Untreated and treated BF has 6-7% weight loss observed at 100°C corresponding to the removal of moisture. The middle zone of the thermogram around 260-450 °C are almost the same, corresponding to the degradation of hemicelluloses. From figure 4.10 (B and C) the higher rate of weight reduction is (1.93 mg/min) observed at about 329°C due to

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This part of study has been published by "Mittal, V.; Sinha, S. Effect of chemical treatment on thermal properties of bagasse fiber-reinforced epoxy composite, *Science and Engineering of Composite Materials*, DOI: 10.1515/secm-2014-0434 (**2014**)."

degradation of hemicelluloses (Teng and Wei, 1998) and cellulose for untreated BF shifted to 333°C for treated BF thereafter second decomposition step took place corresponding to the degradation of lignin and cellulose. A middle zone of the treated BF curve has more inclined as compared to untreated BF curve; this is due to low content of lignin. The residue weight of untreated BF found to be 2.4 % and treated BF is 0.7 % due to presence of lignin in BF which is responsible for the char.

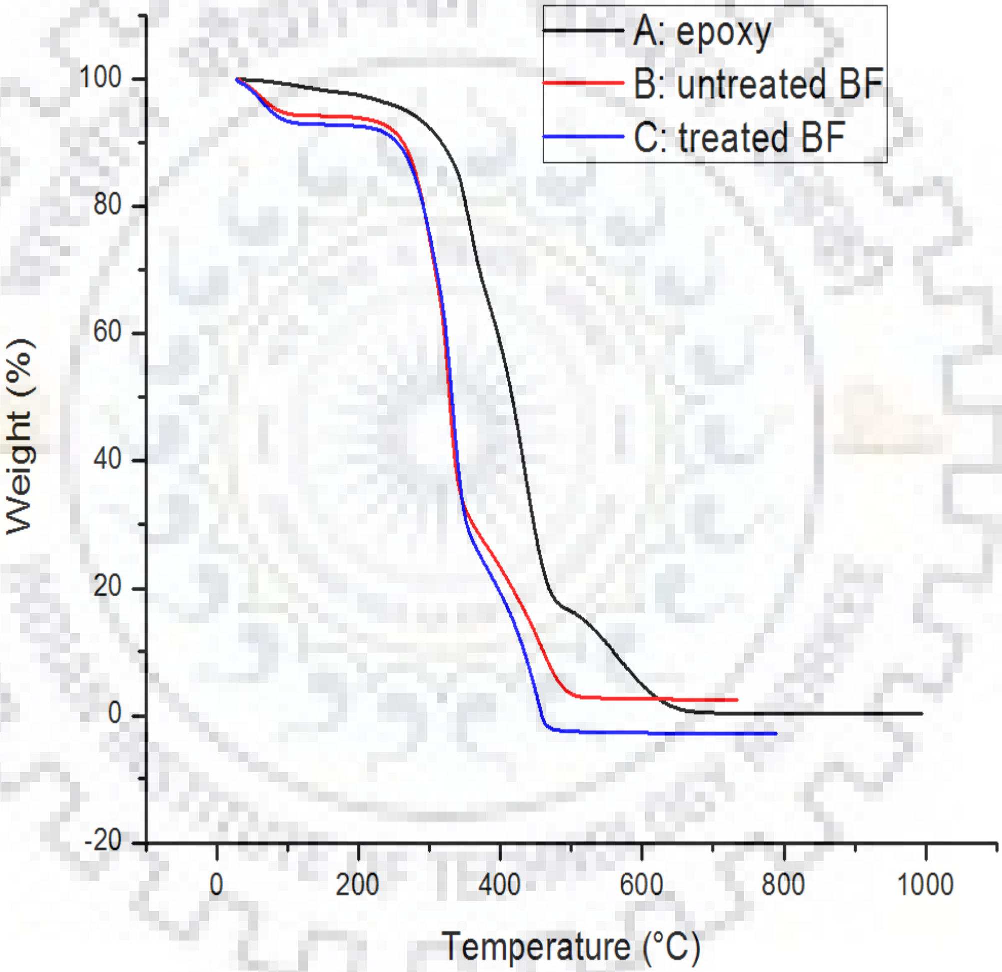

**Figure 4.9: TGA thermogram of epoxy, untreated BF and treated BF**

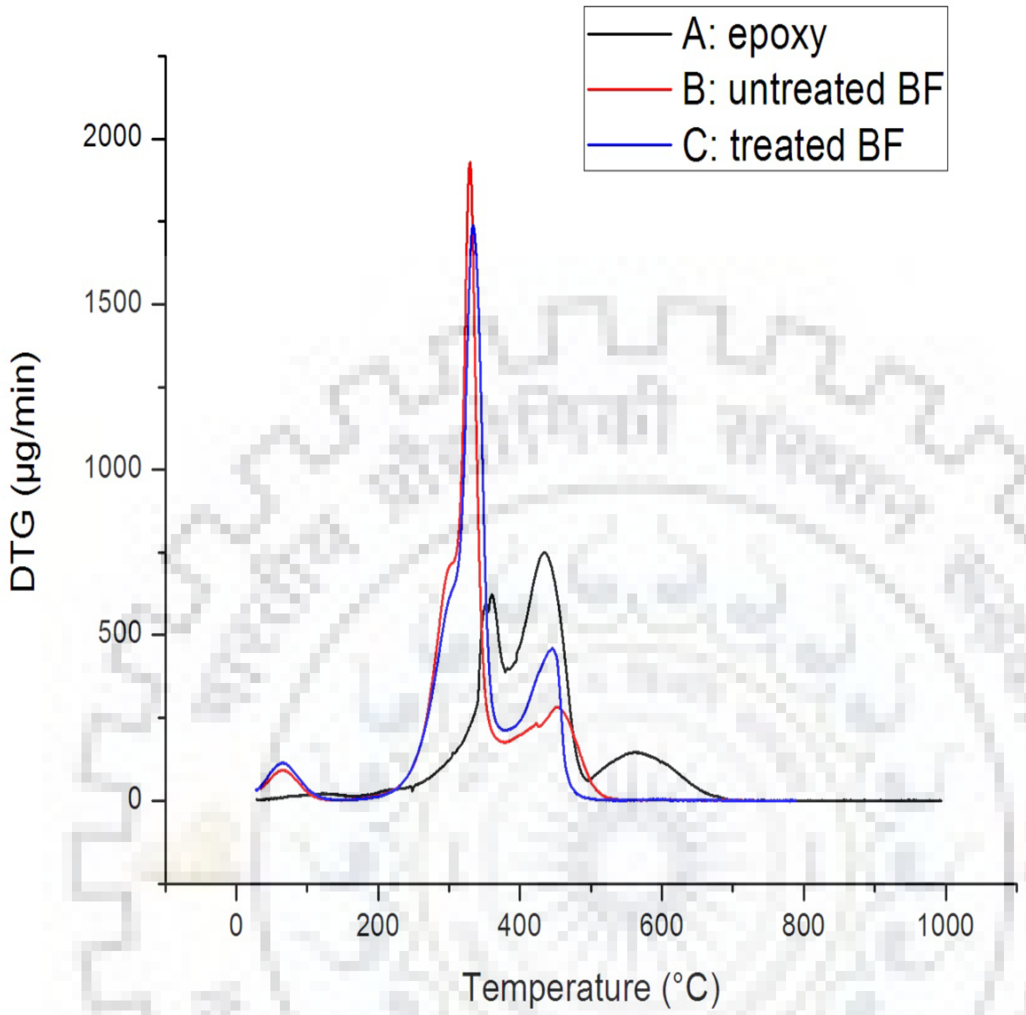

**Figure 4.10: DTG thermogram of epoxy, untreated BF and treated BF**

The TGA behavior of untreated 10 % BF-epoxy composite and treated 10% BF-epoxy composite is shown in figure 4.11 (A and B) respectively. The starting weight reduced of the sample observed at a temperature of 199 °C this is due to the removal of fiber present in the polymer matrix. The range of middle zone is 300-440°C of the respective sample that have much rate of weight loss. From figure 4.12 (A and B) higher rate of weight reduction is (0.74 mg/min) observed at about 359°C for untreated 10 % BF-epoxy composite which is raised to 362°C for treated 10 % BF-epoxy composite. The degradation of the remaining part of the product for the further degradation observed at 477°C for untreated composite which is raised to the 489°C for treated composite thereby, improved the thermal stability of the composite.

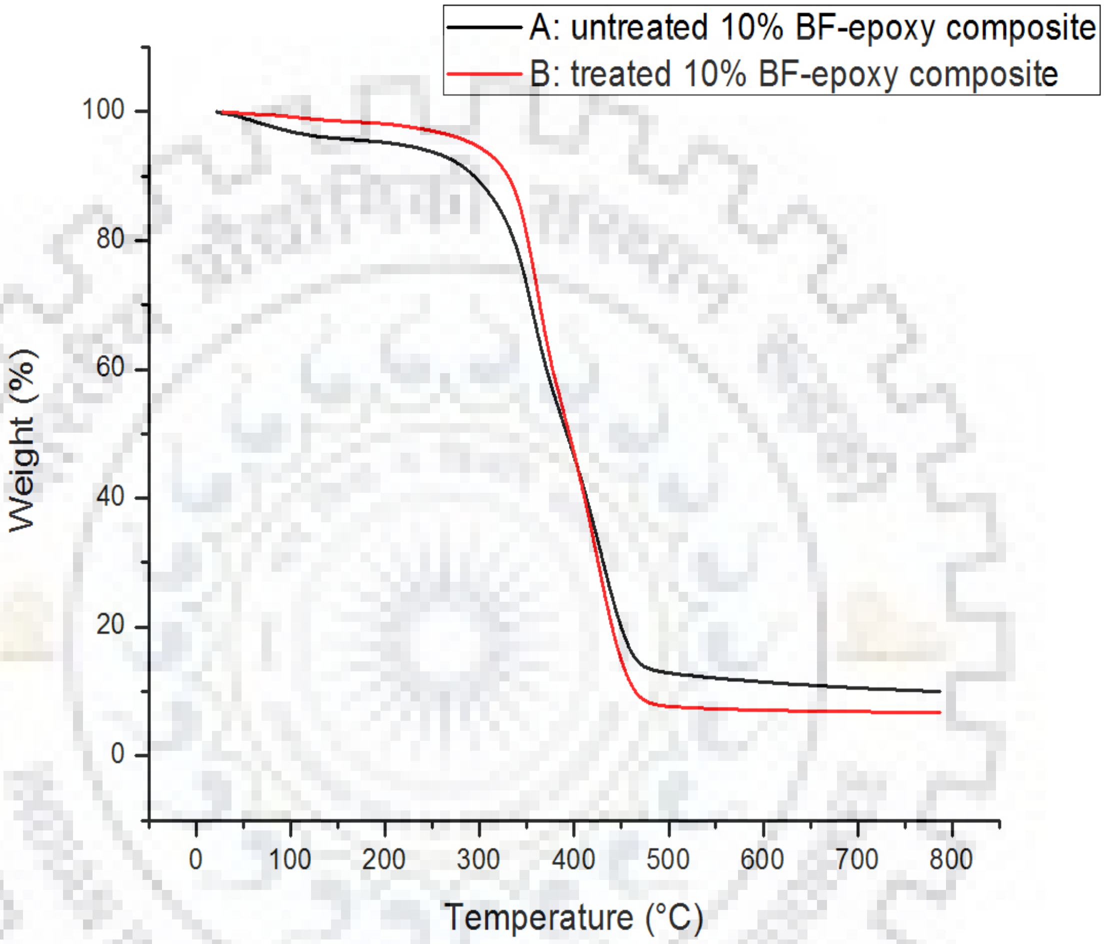

**Figure 4.11: TGA thermogram of untreated and treated 10 % BF-epoxy composites**

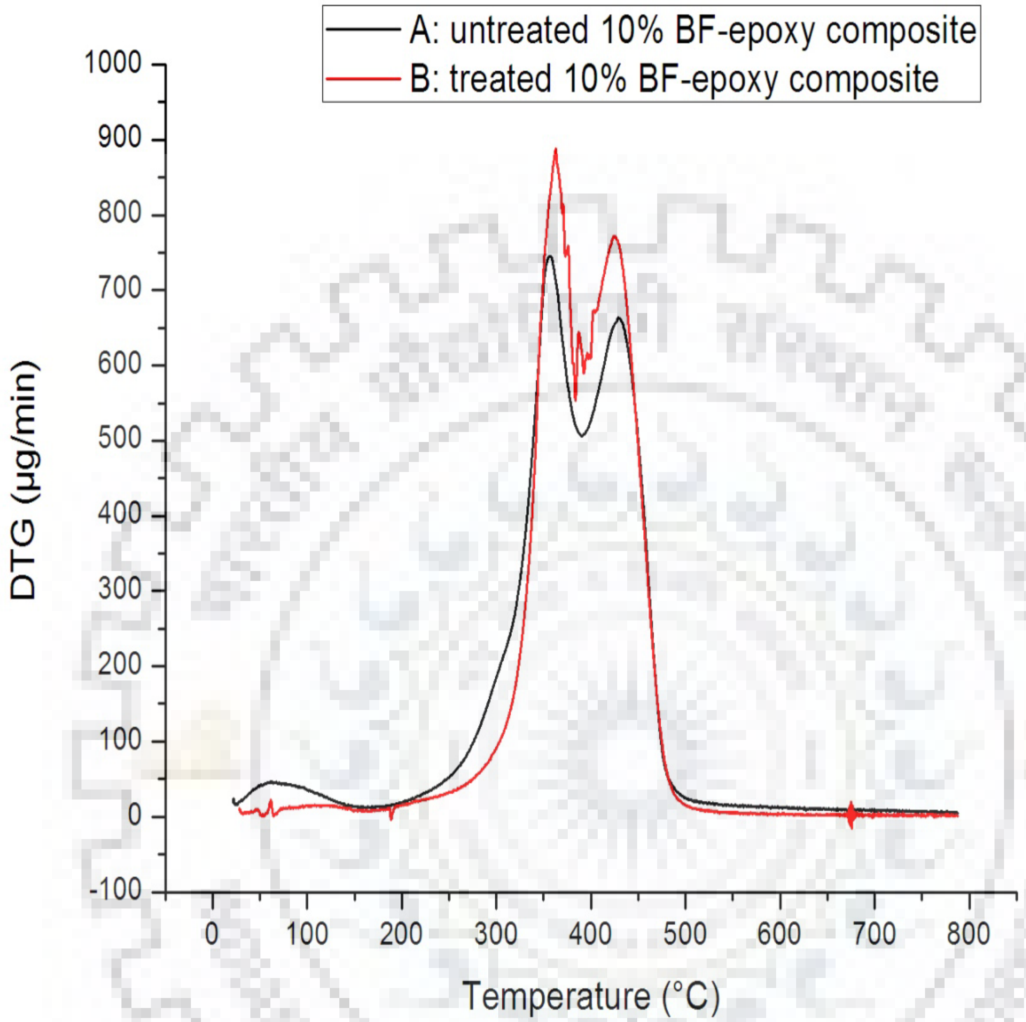

**Figure 4.12: DTG thermogram of untreated and treated 10 % BF-epoxy composites**

The TGA behavior of untreated 20 % BF-epoxy composite and treated 20 % BF-epoxy composite is shown in figure 4.13 (A and B) respectively. The starting weight reduced of the sample observed at a temperature of 195°C corresponds to the removal of fiber present in the polymer matrix. The range of the middle zone is 320-430°C of the respective sample that have excess rate of weight loss. From figure 4.14 (A and B) higher rate of weight reduction is (0.85 mg/min) observed at about 359°C for untreated 20 % BF-epoxy composite down to 356°C for treated 20 % BF-epoxy composite. The degradation of the remaining part of the product for the further degradation is observed at 500°C for untreated composite down to the 483°C for treated composite.

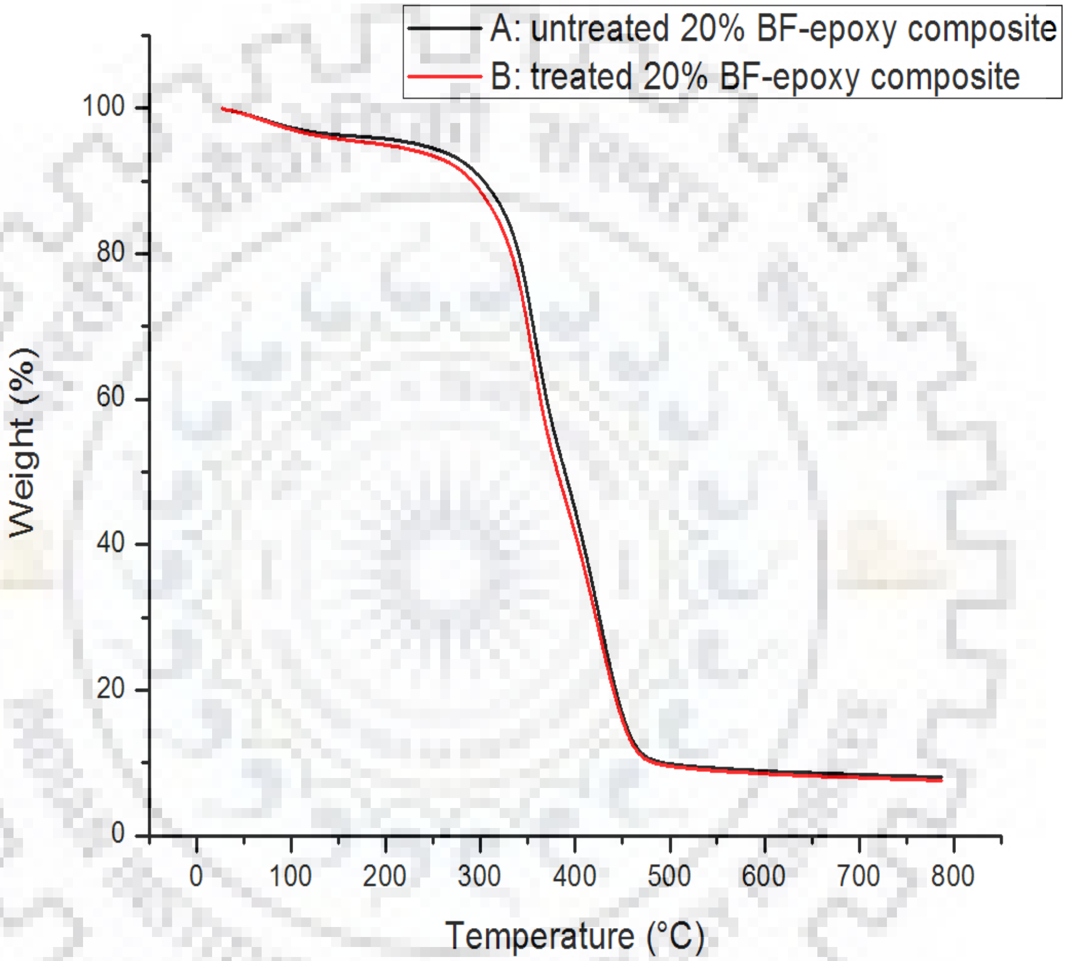

**Figure 4.13: TGA thermogram of untreated and treated 20 % BF-epoxy composites**

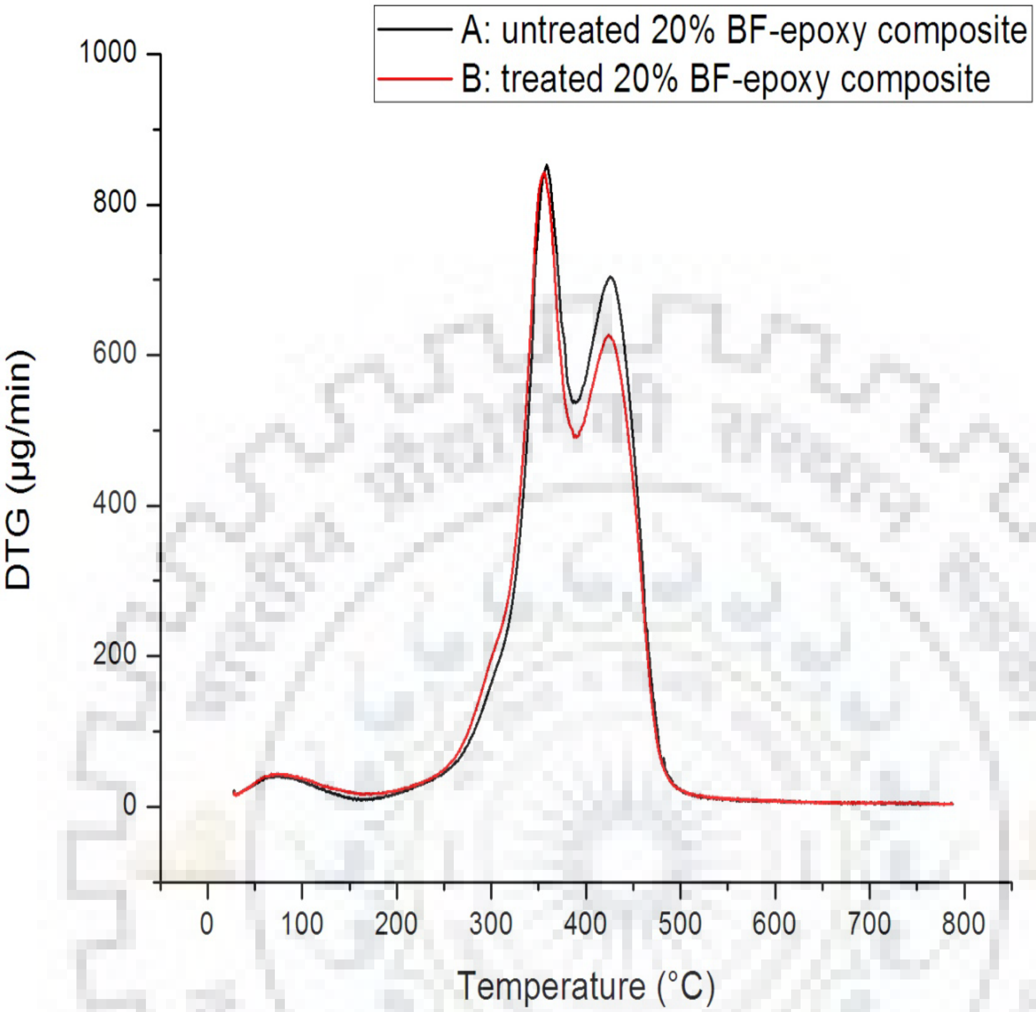

**Figure 4.14: DTG thermogram of untreated and treated 20 % BF-epoxy composites**

The TGA behavior of untreated 30 % BF-epoxy composite and treated 30 % BF-epoxy composite is shown in figure 4.15 (A and B) respectively. The starting weight reduced of the sample observed at a temperature of 195°C corresponds to the removal of fiber present in the polymer matrix. The range of middle zone is 310-440°C of the respective sample that have higher rate of weight loss. From figure 4.16 (A and B) higher rate of weight reduction is (0.84 mg/min) observed at about 354°C for untreated 30 % BF-epoxy composite raised to 356°C for treated 30 % BF-epoxy composite, improved the thermal stability of the composite. The degradation of the remaining part of the product for the further degradation observed at 500°C for untreated composite down to the 484°C for treated composite.

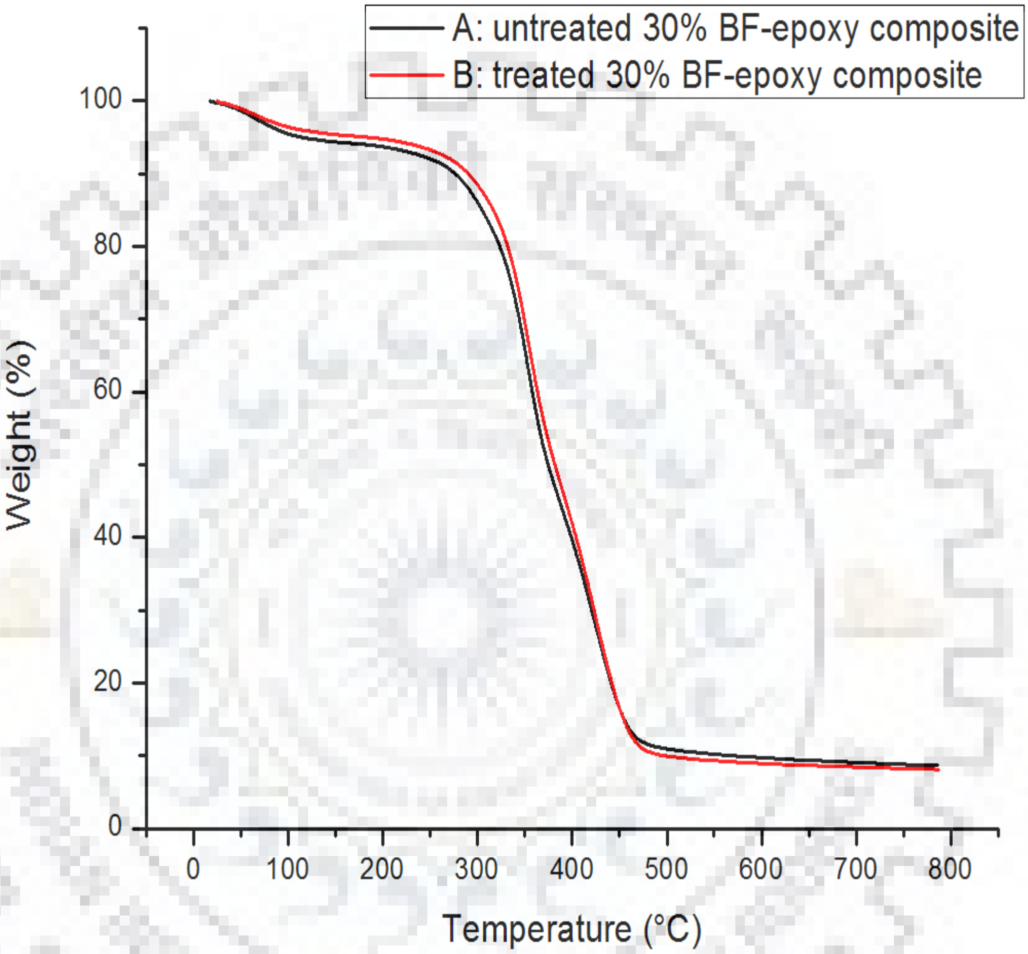

**Figure 4.15: TGA thermogram of untreated and treated 30 % BF-epoxy composites**

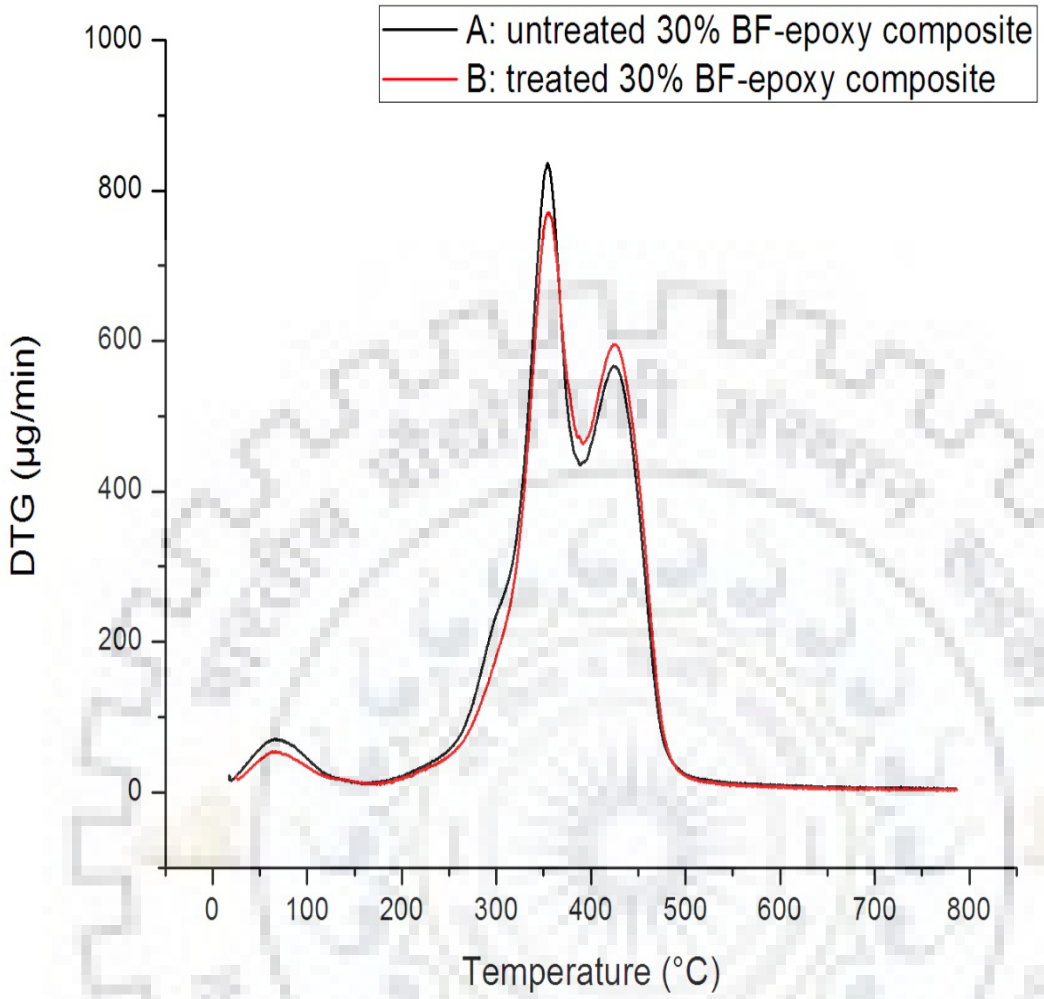

**Figure 4.16: DTG thermogram of untreated and treated 30 % BF-epoxy composites**

The TGA behavior of untreated 40 % BF-epoxy composite and treated 40 % BF-epoxy composite is shown in figure 4.17 (A and B) respectively. The starting weight reduction of the sample is observed at a temperature of 190°C corresponds to the removal of fiber present in the polymer matrix. The range of the middle zone is 310-440°C of the respective sample that have excess rate of weight loss. From figure 4.18 (A and B) high rate of weight reduction is (0.89 mg/min) observed at about 354°C for untreated 40 % BF-epoxy composite raised to 358°C for treated 40 % BF-epoxy composite, improved the thermal stability of the composite. The degradation of the remaining part of the product for the further degradation observed at 500°C for untreated composite down to the 481°C for treated composite (Mittal and Sinha, 2014).

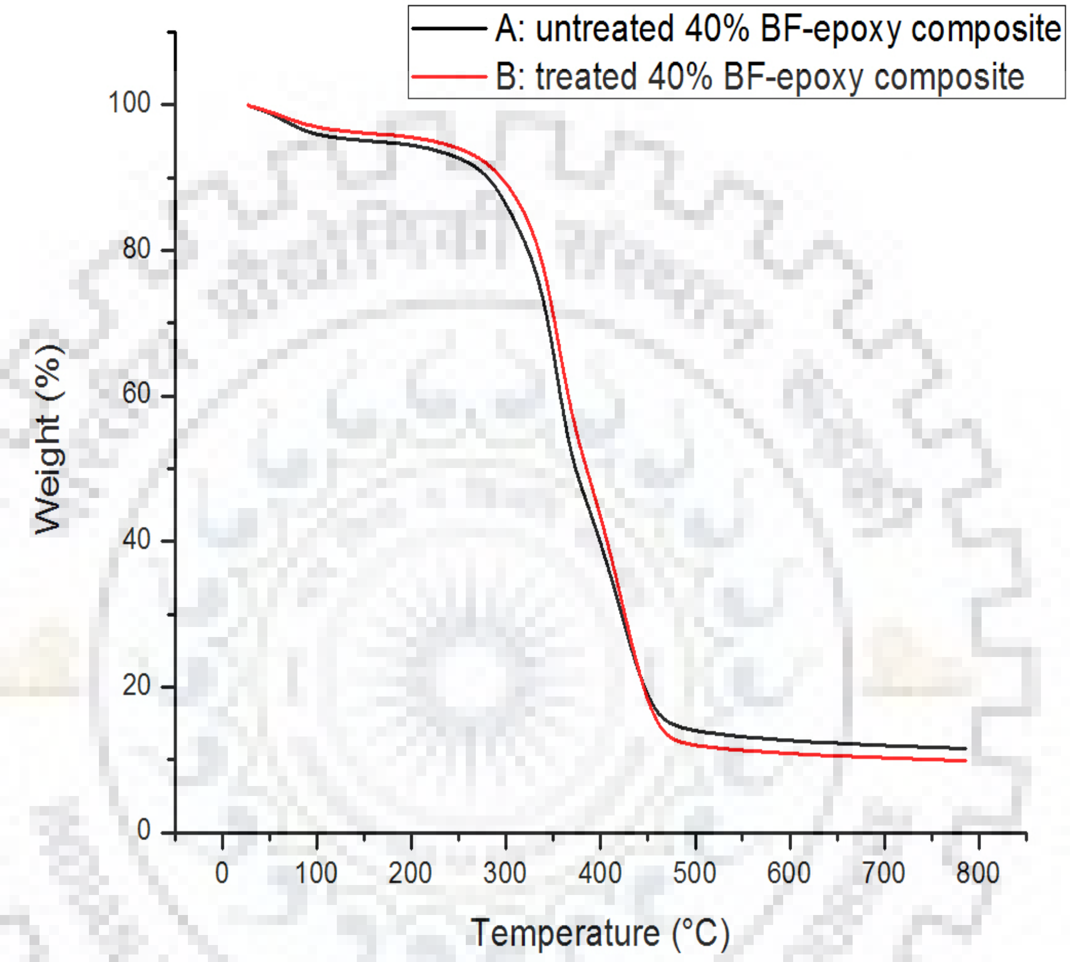

Figure 4.17: TGA thermogram of untreated and treated 40 % BF-epoxy composites

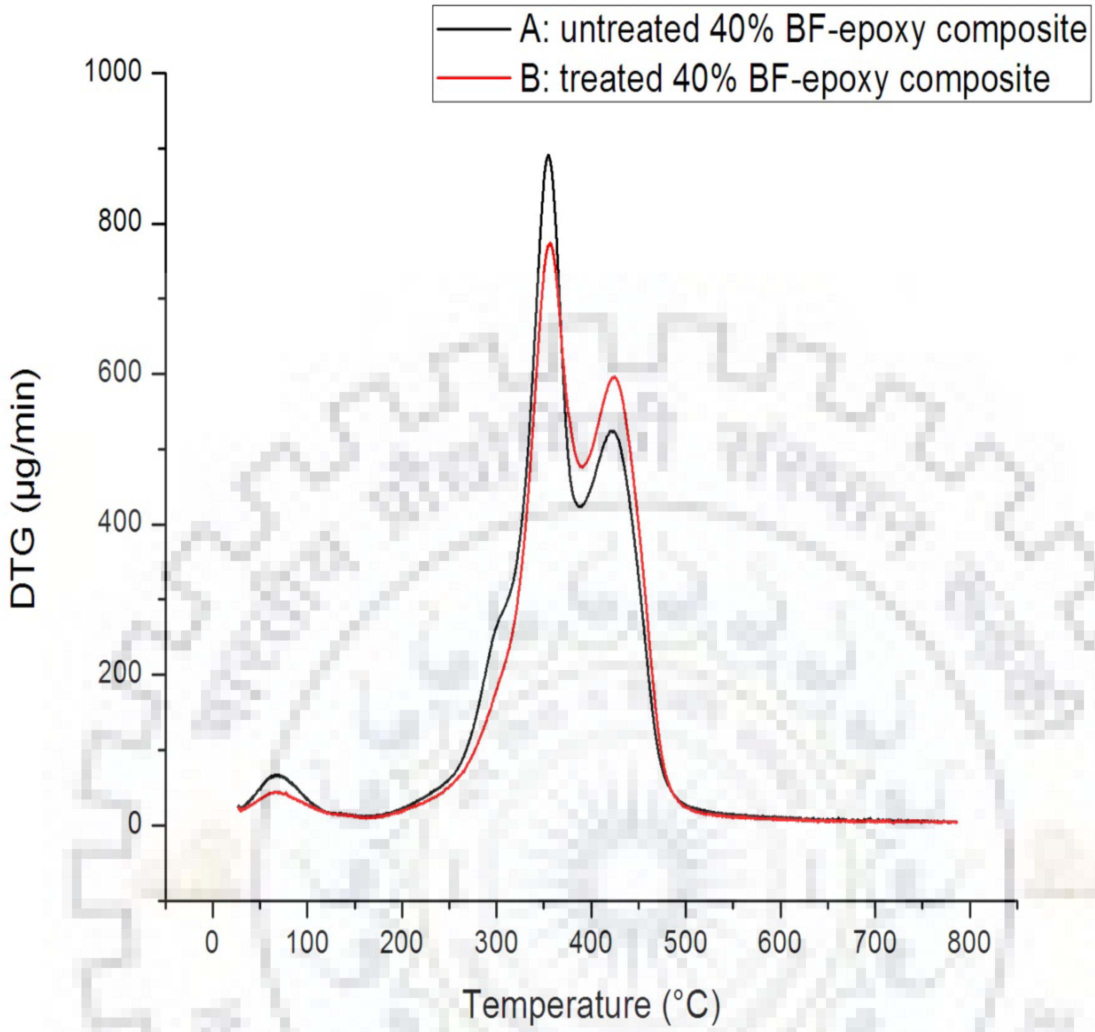

**Figure 4.18: DTG thermogram of untreated and treated 40 % BF-epoxy composites**

### 4.2.2 Differential Scanning Calorimetry (DSC) analysis

The result of DSC thermogram of untreated BF and treated BF is shown in figure 4.19. Both DSC thermogram has distinct exothermic peaks which can be seen in the respective figure. The corresponding ΔH value is given in table 4.3.

From figure 4.19, In DSC thermogram, untreated BF (curve A) has two exothermic peak at 353°C and at 455°C, this is due to the decomposition of hemicelluloses and α-cellulose respectively. In the case of the treated BF (curve B) hemicelluloses peak is shifted to a higher temperature at 364°C, this is because of removal of non-cellulosic matter such as hemicelluloses, pectin, etc. During the chemical treatment of fiber, there is a destruction of the bonding which is present in the fiber might have some influence, shifted the peak. The exothermic peak at 448°C is due to degradation of cellulose in BF and formation of char (Hoareau et al., 2004).

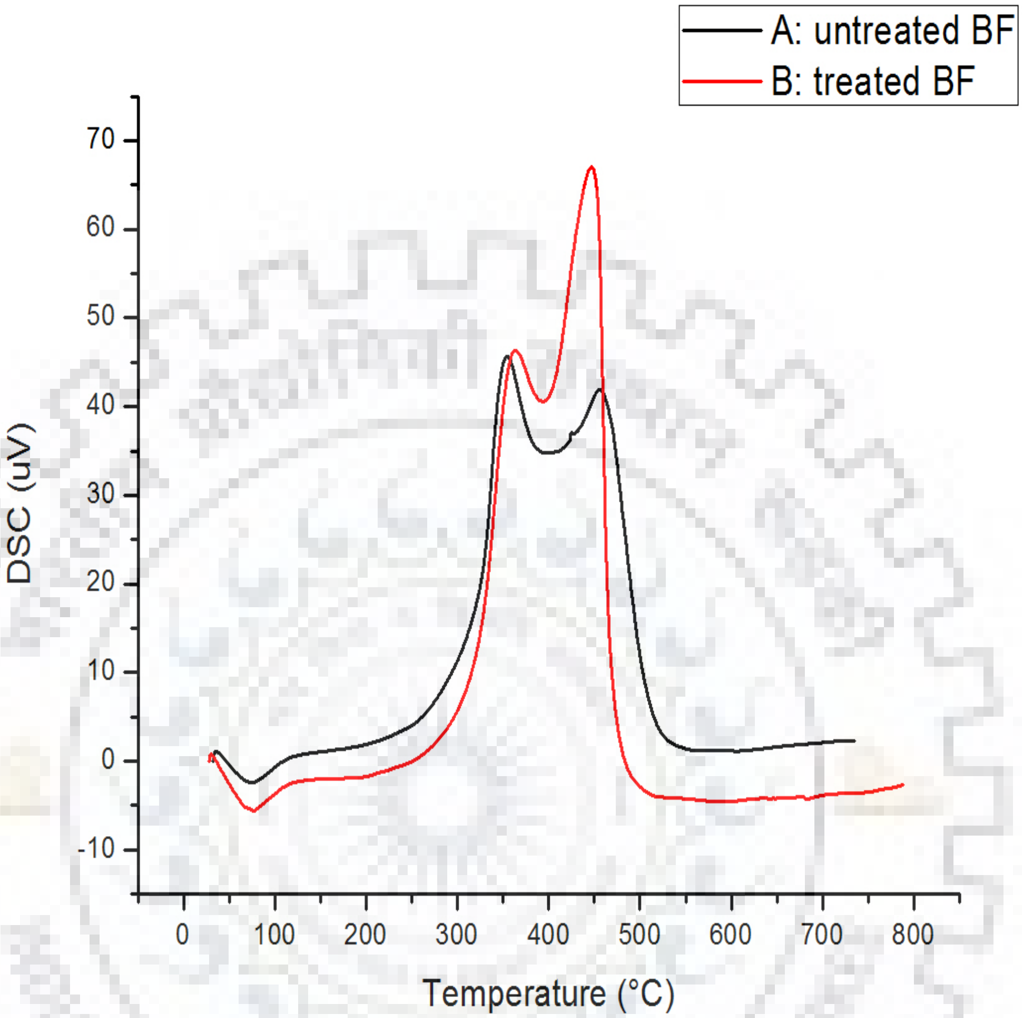

**Figure 4.19: DSC thermogram of untreated BF and treated BF**

**Table 4.1: Result of Differential Scanning Calorimeter (DSC) analysis of untreated BF and treated BF**

| Sample | Peak temperature (°C) | Nature of peak | ΔH (J/mg) |
|---|---|---|---|
| **Untreated BF** | 353 | Exothermic | -3.44 |
|  | 455 | Exothermic | -3.44 |
| **Treated BF** | 364 | Exothermic | -3.66 |
|  | 448 | Exothermic | -3.66 |

## 4.3 FOURIER TRANSFORM INFRARED SPECTROSCOPY (FT-IR) ANALYSIS

Fourier Transform Infrared spectra of the untreated BF and treated BF in the range of 4,000 to 400 cm$^{-1}$ are shown in figure 4.20. In the spectra of untreated BF and treated BF bands observed at 3394–3438 cm$^{-1}$ correspond to the OH stretching vibrations of alcoholic and phenolic OH groups present in starch, cellulose and lignin (Vyas et al., 2013). Both untreated and treated have bands at 2923 cm$^{-1}$ correspond to the C-H stretch alkanes group present in the cellulose and hemicellulose. The untreated BF has the 899 cm$^{-1}$ peak, which shows the presence of the N-H primary amine group and $CH_2$ group (Ouajai and Shanks, 2005; Colom et al., 2003). The peak at 1431 cm$^{-1}$ is related with C-C stretching in aromatic rings and OH in bending shows a presence of lignin (Corrales et al., 2012). The peak at 1509 cm$^{-1}$ arises from the N-O stretching (Dalai et al., 1998). It can be seen that the chemically treated BF graph the absorption peaks are disappearing, which indicates that these functional groups are removed after the chemical treatments, thus lignin concentration, reduced that affect the adhesion between the fiber and polymer surfaces, demonstrating the structure transformation induced by thermal stability.

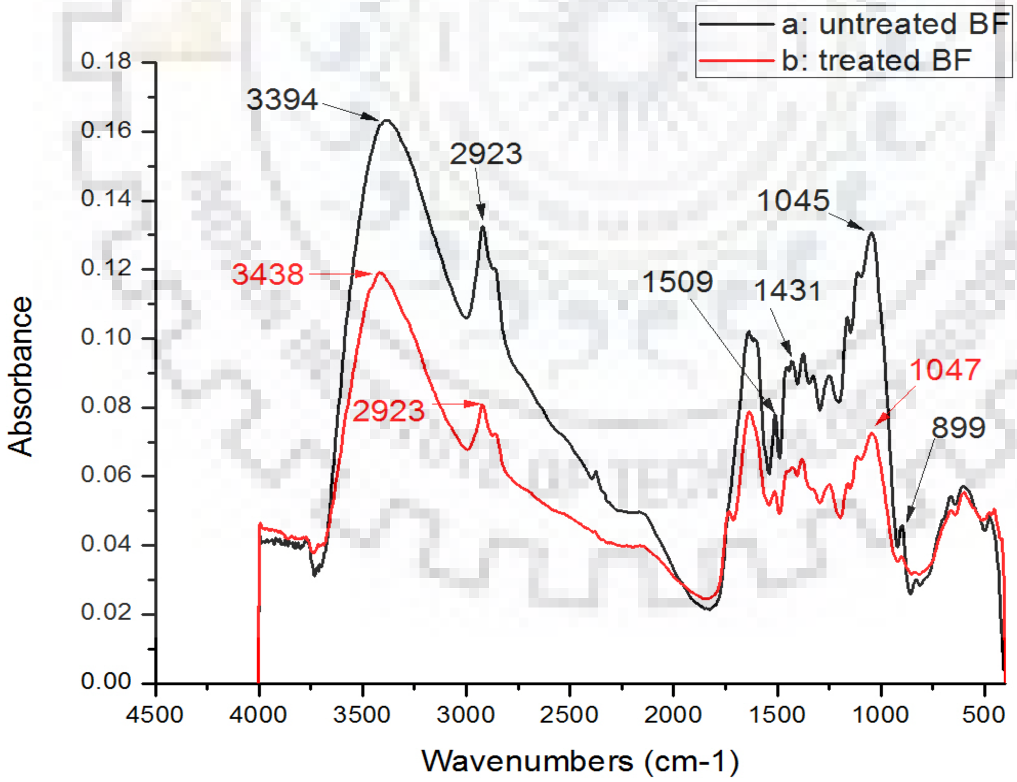

**Figure 4.20: Fourier Transform Infrared Spectroscopy (FT-IR) spectra of (a) untreated BF (b) treated BF**

## 4.4 EFFECT OF FIBER CONTENT AND ALKALI TREATMENT ON MECHANICAL AND WATER ABSORPTION PROPERTIES OF WHEAT STRAW FIBER-REINFORCED EPOXY COMPOSITES

This section deals with the mechanical and water absorption properties of Wheat Straw Fiber (WSF) reinforced epoxy composites. In order to improve the composite characteristic, WSF is treated with three different concentrations of alkali (1, 3 & 5 %). The mechanical and water absorption properties of treated fiber composites are characterized and compared with untreated fiber filled epoxy composites. It has been found that the mechanical properties and water resistance are reduced with an increase in the WSF loading from 5 to 25 wt %. Among the three levels of alkali treatment, the composite made with 3 % alkali treated fiber exhibits superior mechanical properties than the other treated fiber composite, which pointed to an efficient fiber-matrix adhesion. The scanning electron microscopy (SEM) is used to observe the surface morphology of the WSF.

### 4.4.1 Mechanical analysis

Figure 4.21-4.25 showed the effect of WSF loading (5, 10, 15, 20 and 25 weight %) and three levels of concentration (1, 3, 5 %) alkali treatment of fibers on tensile strength, tensile modulus, flexural strength, flexural modulus, an impact strength of the WSF-epoxy composites respectively. It has been observed that the reduction in the mechanical properties of WSF-epoxy composites with an addition of the WSF in neat epoxy resin. This is due to the low strength of the WSF and poor adhesion between the fiber and polymer matrix caused by a cluster of the fiber and resulting that no polymer present in between the fibers.

As seen from figure 4.21 and 4.22, the tensile strength and tensile modulus of the 1 % alkali treated fiber composites at 5 % fiber loading is 12.8 and 945 MPa, respectively, against the tensile strength and tensile modulus of untreated fiber composites is 11.5 and 906 MPa, respectively. It has been observed a betterment of approximately 11 % in tensile strength and 4 % in tensile modulus of the composite made with 1 % NaOH treated fibers. Further, the NaOH concentration raised to 3 %, the tensile strength is changed from 12.8 MPa to 15.8 MPa and the tensile modulus also lifted from 945 MPa to 1315 MPa. Finally, the NaOH

This part of study has been published by " Mittal, V.; Sinha, S. Study the effect of fiber loading and alkali treatment on mechanical and water absorption properties of wheat straw fiber reinforced epoxy composites, *Science and Engineering of Composite Materials*, DOI: 10.1515/secm-2015-0441 (**2015**)."

concentration raised to 5 %, we observed that both tensile strength and tensile modulus decreased.

Similarly, from figure 4.23 and 4.24, the flexural strength and flexural modulus of the 1 % alkali treated fiber composites at 5 % fiber content is 26.5 and 1663 MPa, respectively against the flexural strength and flexural modulus of untreated fiber composites is 23.8 and 1496 MPa, respectively. It has been observed that an enhancement of approximately 11 % in both flexural strength and flexural modulus. Further, the NaOH concentration raised to 3 %, the flexural strength is increased from 26.5 MPa to 35.5 MPa and the flexural modulus also increased from 1663 MPa to 2200 MPa. Lastly, the NaOH concentration raised to 5 %, we observed that both flexural strength and flexural modulus decreased.

From the figure 4.25, WSF-epoxy composites showed low impact strength compared to pure epoxy. The impact strength of neat epoxy is 28.8 J/m, after the addition of 5 % untreated WSF in the polymer, the impact strength of the composites reduced to 25.2 J/m. The impact strength of the 1 % alkali treated fiber composites has remarkably increased from 25.2 to 28.7 J/m. Further NaOH concentration raised to 3 %, the impact strength is lifted from 28.7 J/m to 29.6 J/m. Finally, the NaOH concentration raised to 5 %, then, impact strength jumped to 31.2 J/m.

Similar results had been found by in the case of coir–polyester composites (Rout et al., 2001). As described above, the fibrillation provides a good interfacial adhesion between the fiber polymer matrixes. However, for the 5 % NaOH treated fiber composites, the mechanical properties reduced with the greater fibrillation due to larger fire break lead to stress losses at the interface of the polymer and fiber. The maximum improvement in tensile and flexural properties is found in the 3 % NaOH treated fiber composites, but the impact strength has a high value in the 5 % NaOH treated fiber composites.

However, the effect of various levels of alkali treatment on WSF is observed to increase the above properties of the composites at all the WSF contents investigated. Wheat straw is a natural lignocelluloses fiber. WSF is made up of crystalline cellulose that is surrounded with hemicellulose and lignin. These cells have ended long and overlapping with each other and form the cellular structure. The NaOH reacts with a hydroxyl group which available in the hemicellulose part of the fiber and it contributes to the death of the cellular part of the fiber and thereby the fibers are broken into various filaments parts (Mittal and Sinha, 2015b).

Higher concentration of alkali treatment gives losses the fiber structure that resulted the reducing in the mechanical properties of the composites. Therefore, we don't moved to higher concentration. Using the wheat straw fiber for developing the polymer composites reduced the cost of composites up to 5 to 10 % as compared to the pure polymeric material. We have also study the bagasse fiber-epoxy composites which shows the 43% more tensile strength than the wheat straw composites.

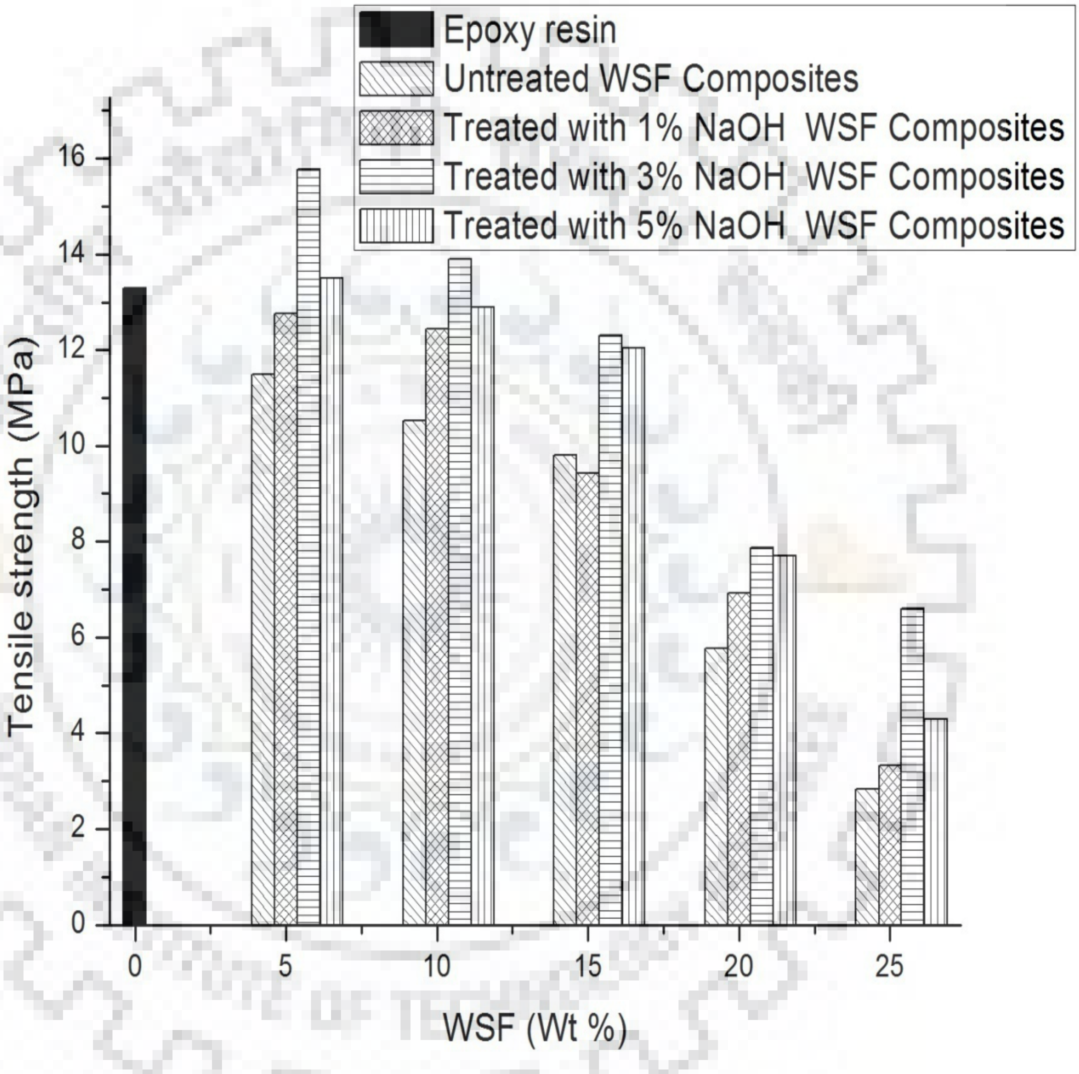

**Figure 4.21: Effect of fiber content and alkali treatment on the tensile strength of WSF-epoxy composites**

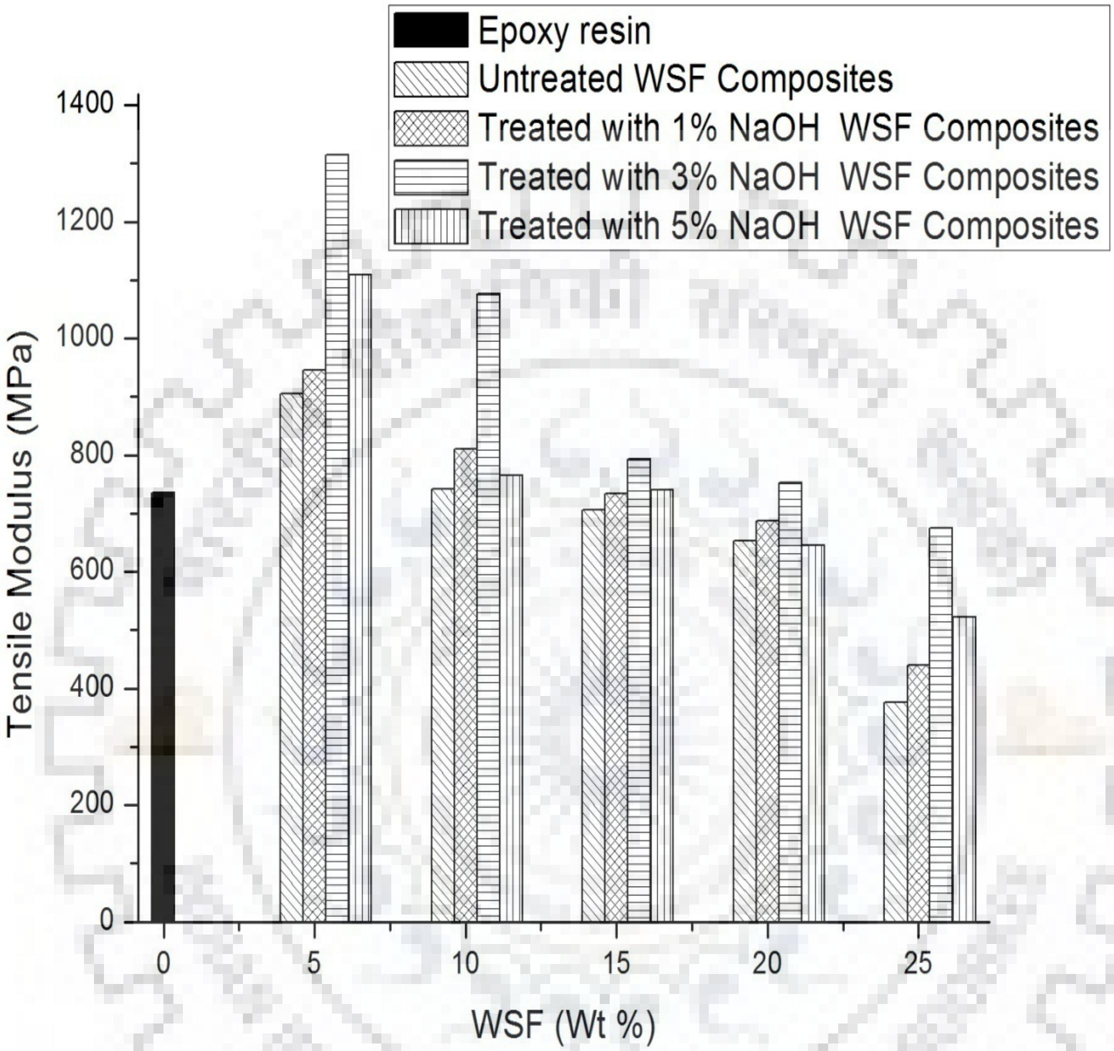

**Figure 4.22: Effect of fiber content and alkali treatment on the tensile modulus of WSF-epoxy composites**

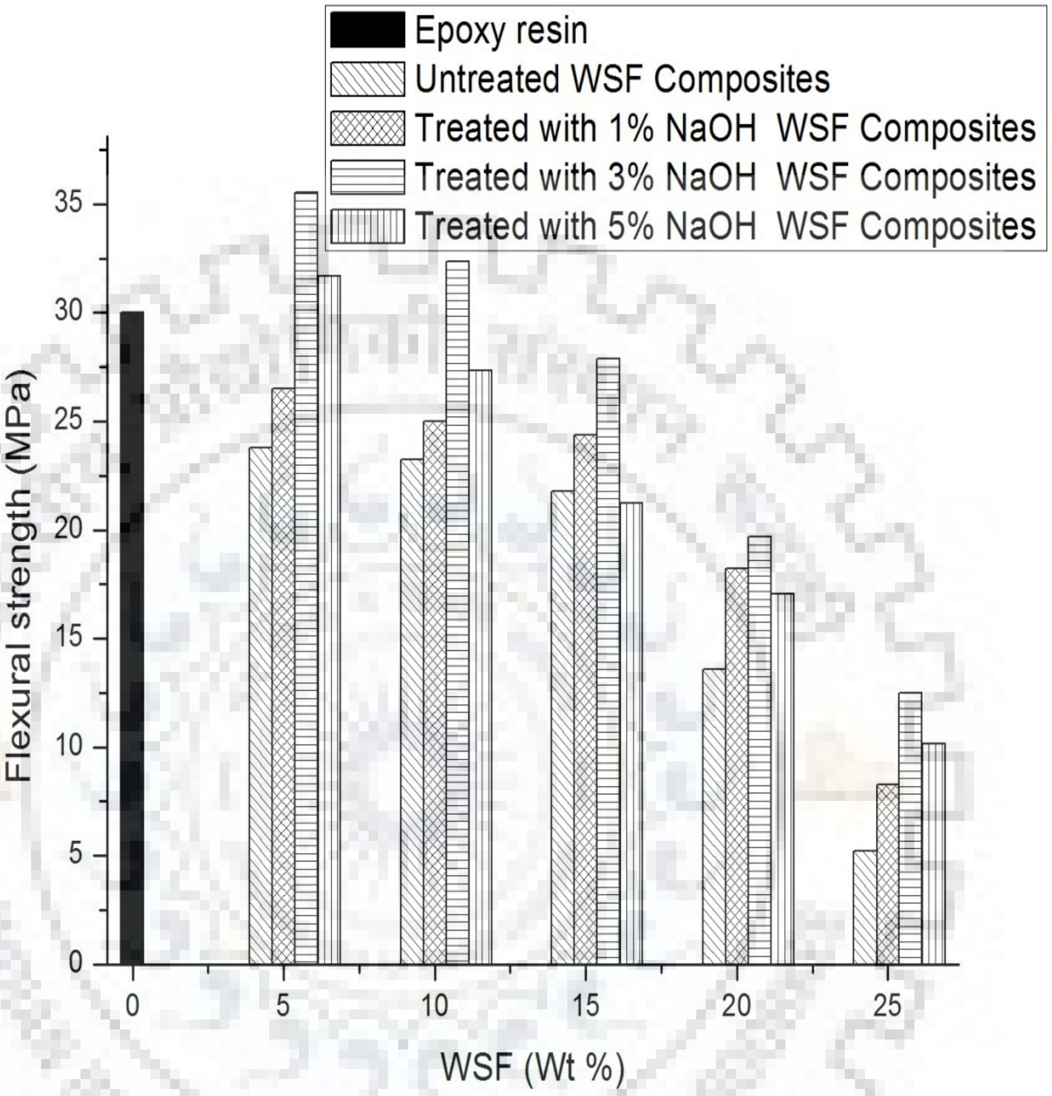

**Figure 4.23: Effect of fiber content and alkali treatment on the flexural strength of WSF-epoxy composites**

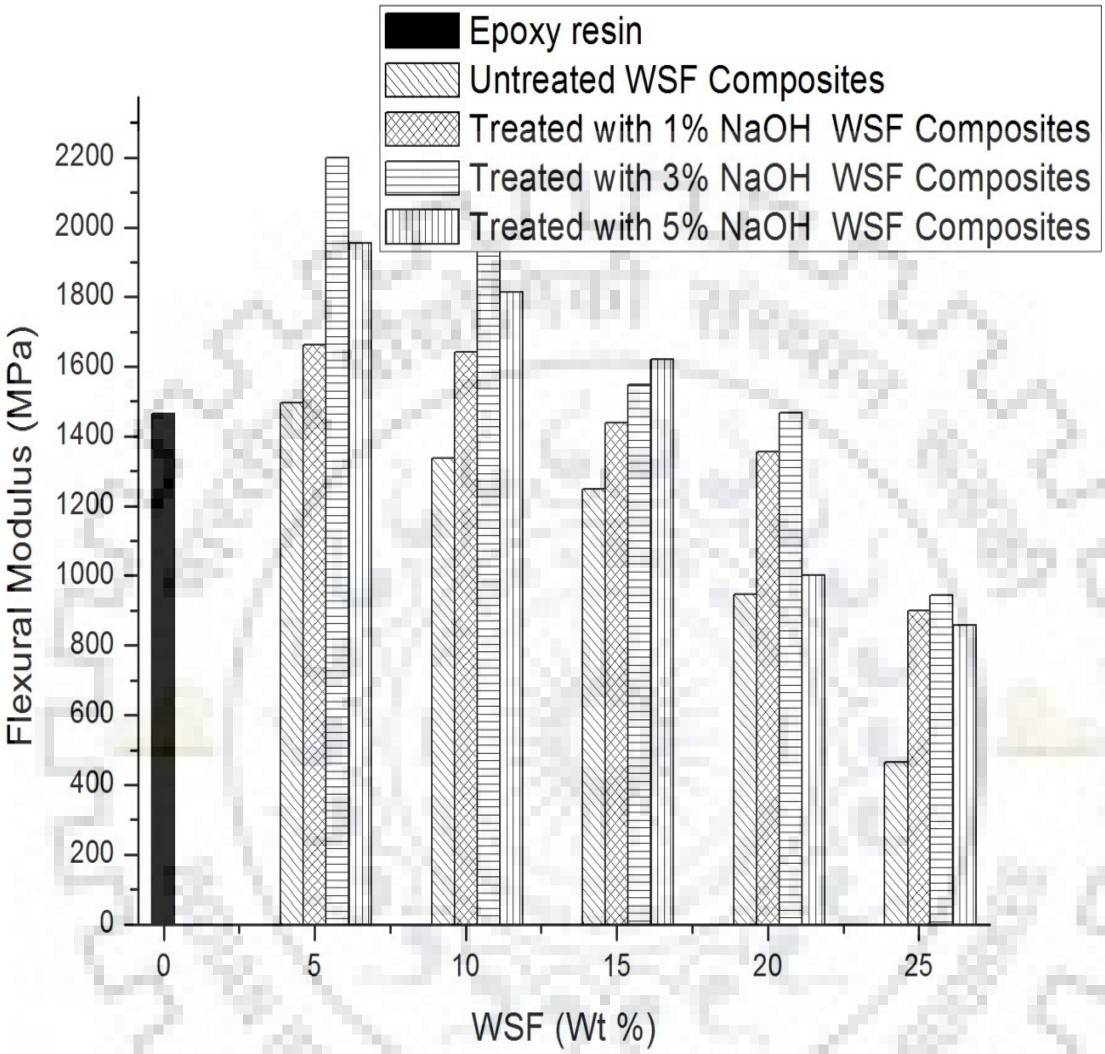

**Figure 4.24: Effect of fiber content and alkali treatment on the flexural modulus of WSF-epoxy composites**

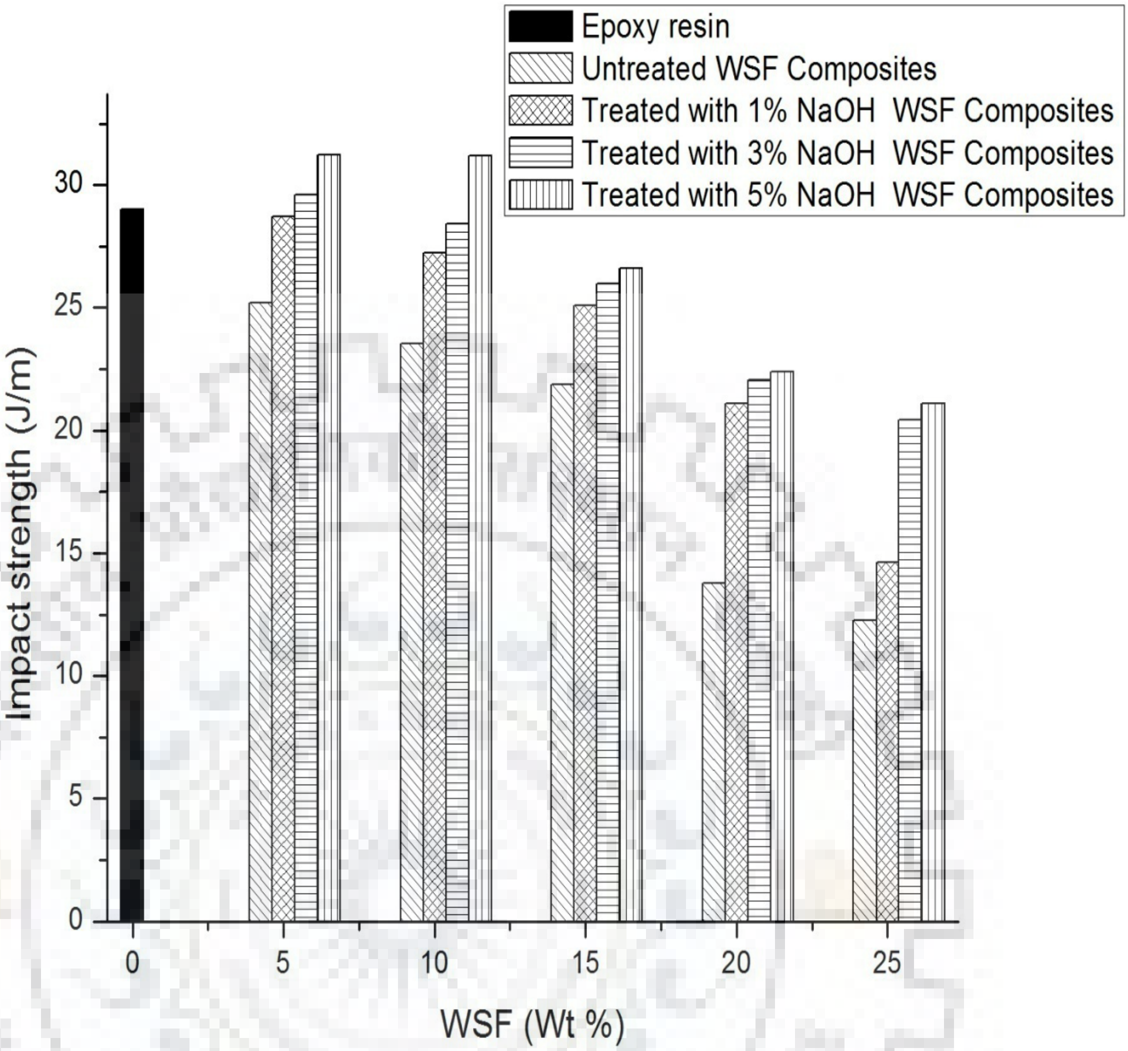

**Figure 4.25: Effect of fiber content and alkali treatment on the impact strength of WSF-epoxy composites**

### 4.4.2 Scanning Electron Microscopy (SEM)

The morphology of the fiber surfaces which are treated by alkali at different levels is shown in figure 4.26 (a), (b), (c) and (d). It is evident that the filaments which present in the untreated fiber are joined together, but spills out after the application of the alkali treatment. This process is termed as branching, which breaks the untreated fiber into more small-scale by the dissipation of the hemicelluloses which present in the fiber. The branching increases the adhesion surface area presents between the fiber and polymer implies strong bonding between them (Bisanda and Ansell, 1991). Hence, alkali treatment helps to improve the interfacial adhesion (Ray et al., 2001).

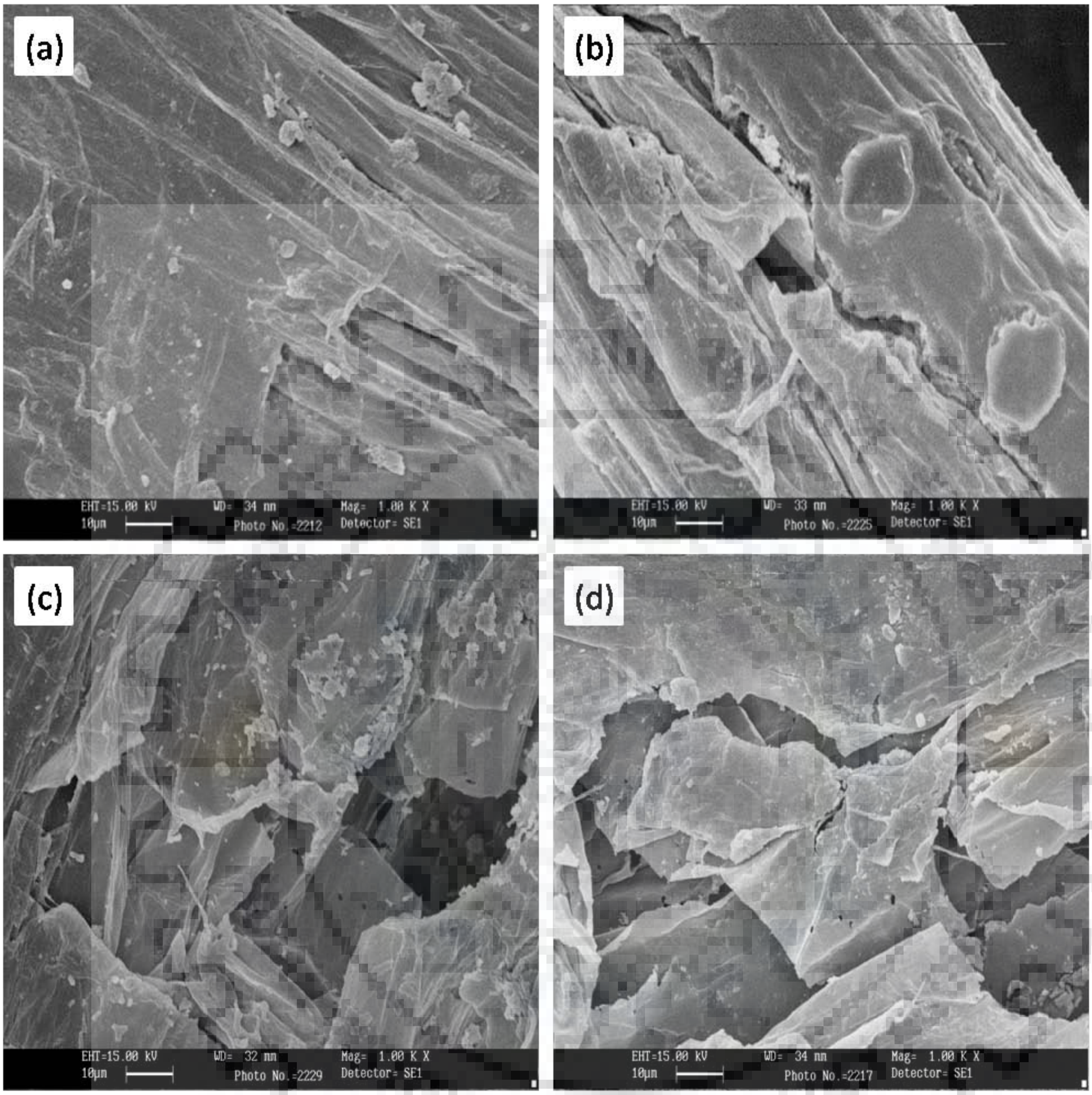

**Figure 4.26: Scanning Electron Microscopy (SEM) micrograph of (a) untreated WSF, (b) WSF treated with 1 % NaOH,(c) WSF treated with 3 % NaOH, (d) WSF treated with 5 % NaOH**

### 4.4.3 Water absorption analysis

Figure 4.27 showed the percentage intake of water absorbed in untreated and various levels of alkali treated WSF-epoxy composites. It is evident that the natural fiber composites intake higher water absorption than pure epoxy resin increases the fiber content water absorption also increases due to the fact that lignocelluloses material absorbs more water.

On the other face, it has been observed that the alkali treatment of WSF decreases the water absorption in the WSF-composites, 5 % NaOH treated WSF composite at the optimum point (5 wt % WSF content) is 3.5 % water absorb while untreated WSF composite is 5 % water absorb for the for 10 days. It has been proved that alkali treatment decreased the water absorption characteristic of the composites.

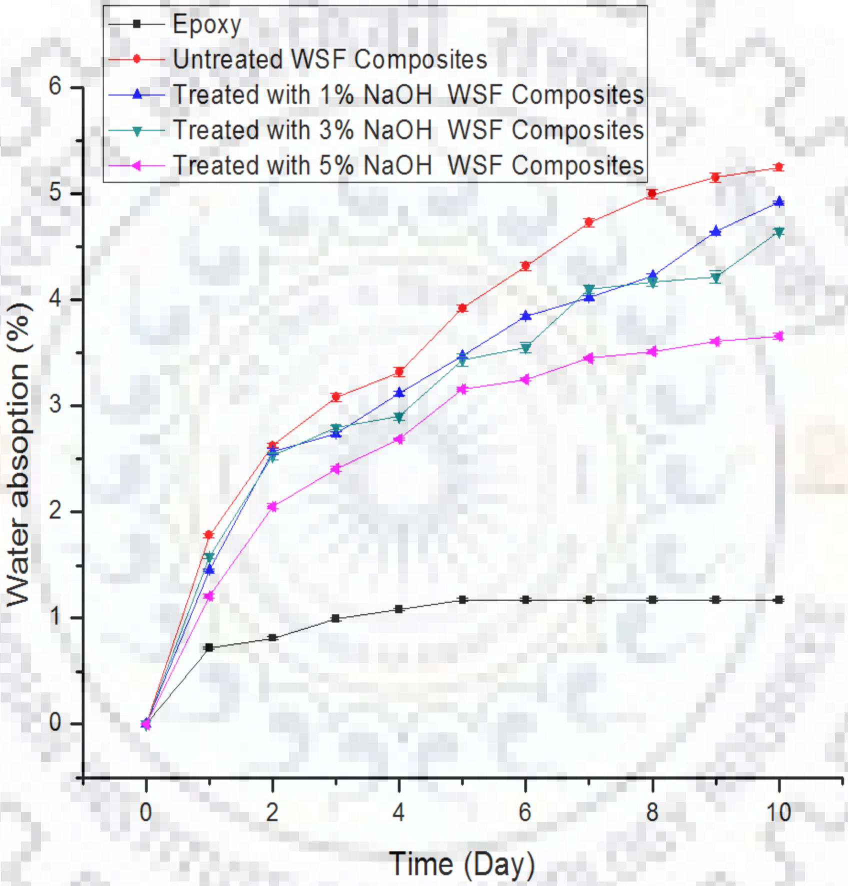

**Figure 4.27: Water absorption curve for the WSF composites at the optimum point (5 wt % WSF content)**

## 4.5 EFFECT OF FIBER CONTENT AND ALKALI TREATMENT ON THERMAL PROPERTIES OF WHEAT STRAW FIBER-REINFORCED EPOXY COMPOSITES

The thermal properties of all the untreated Wheat Straw Fiber (WSF) reinforced epoxy composites and alkali treated WSF reinforced epoxy composites are analyzed by the Thermo Gravimetric Analysis (TGA) and Differential Thermal Gravimetric (DTG). The untreated WSF and alkali treated WSF are also analyzed by the Differential Scanning Calorimetry (DSC). The alkali treatment of WSF induces reasonable changes in the thermal stability of the polymer composites. When untreated WSF composites are compared with the alkali treated composites we have found significant variation in the presence of the peaks.

### 4.5.1 Thermo Gravimetric Analysis (TGA) and Differential Thermal Gravimetric (DTG) analysis

Thermo gravimetric analysis and Differential thermal gravimetric thermograms of (neat epoxy, untreated WSF, alkali treated WSF, untreated WSF-epoxy composites, alkali treated WSF-epoxy composites) are shown in figure 4.28 and 4.29 respectively. In TGA thermograms, we have observed three different zones of degradation. In the first zone at (80-100°C), a small loss in weight is observed due to the removal of moisture content in the sample. Second degradation zone, shows a maximum weight loss which is observed nearly in the range of 200-450°C, this is probably due to the degradation of fiber components. Above 450°C, this is the last zone of thermal degradation also called as ultimate thermal degradation. From DTG thermogram, high rate of thermal degradation temperature of the sample is observed.

The TGA behavior of neat epoxy is shown in figure 4.28 (A). A single step decomposition process is observed for neat epoxy. The degradation started at about 200°C, from figure 4.29 (A) the higher rate of weight reduction (0.75 mg/min) is observed at 435°C due to the degradation. The degradation of the remaining part of the product is seen at 650°C. The residue left after final degradation is 1.1 %.

The TGA behavior of untreated WSF, 1, 3, 5 % alkali treated WSF is shown in figure 4.28 (B, C, D and E) respectively. Untreated and all alkali treated WSF has 6-7 % weight loss which is observed at 100°C corresponding to the removal of moisture. The middle zone of the thermogram around 210-475°C is almost the same, corresponding to the degradation of

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hemicelluloses. From figure 4.29 (B, C, D and E) the higher rate of weight reduction (1.01 mg/min) is observed at 320°C due to degradation of hemicelluloses (Teng and Wei, 1998). and cellulose for untreated WSF shifted to 325°C (1.08 mg/min) for 1 % alkali treated WSF, 325°C (0.95 mg/min) for 3 % alkali treated WSF, further shifted to 331°C (1.26 mg/min) for 5 % alkali treated WSF thereafter second decomposition step took place corresponding to the degradation of lignin and cellulose. The residue weight of untreated WSF is found to be 7.87 %, 1 % alkali treated WSF has 11.29 %, 3 % alkali treated WSF has 10.75 % and 5 % alkali treated WSF has 5.13 %, for due to present of lignin in WSF which is responsible for the char (Hornsby et al., 1997).

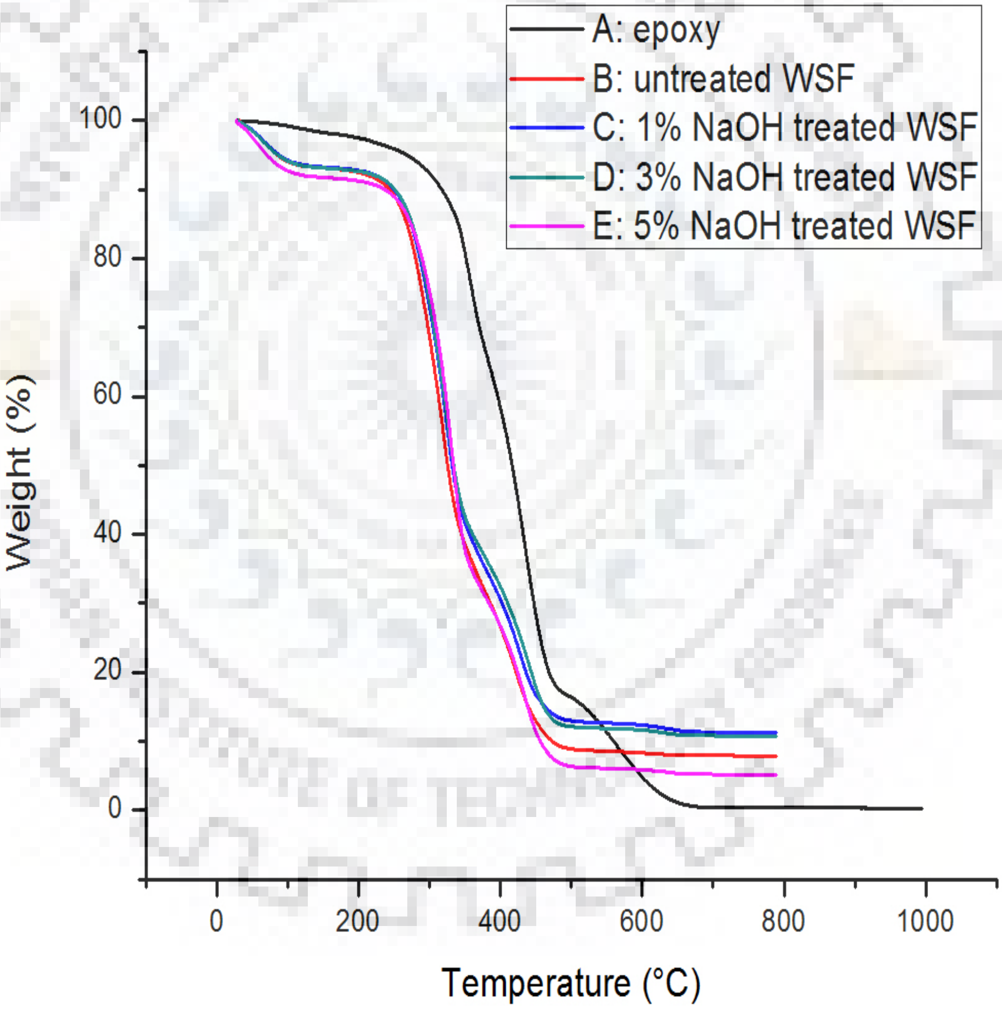

**Figure 4.28: TGA thermogram of epoxy, untreated WSF and all alkali treated WSF**

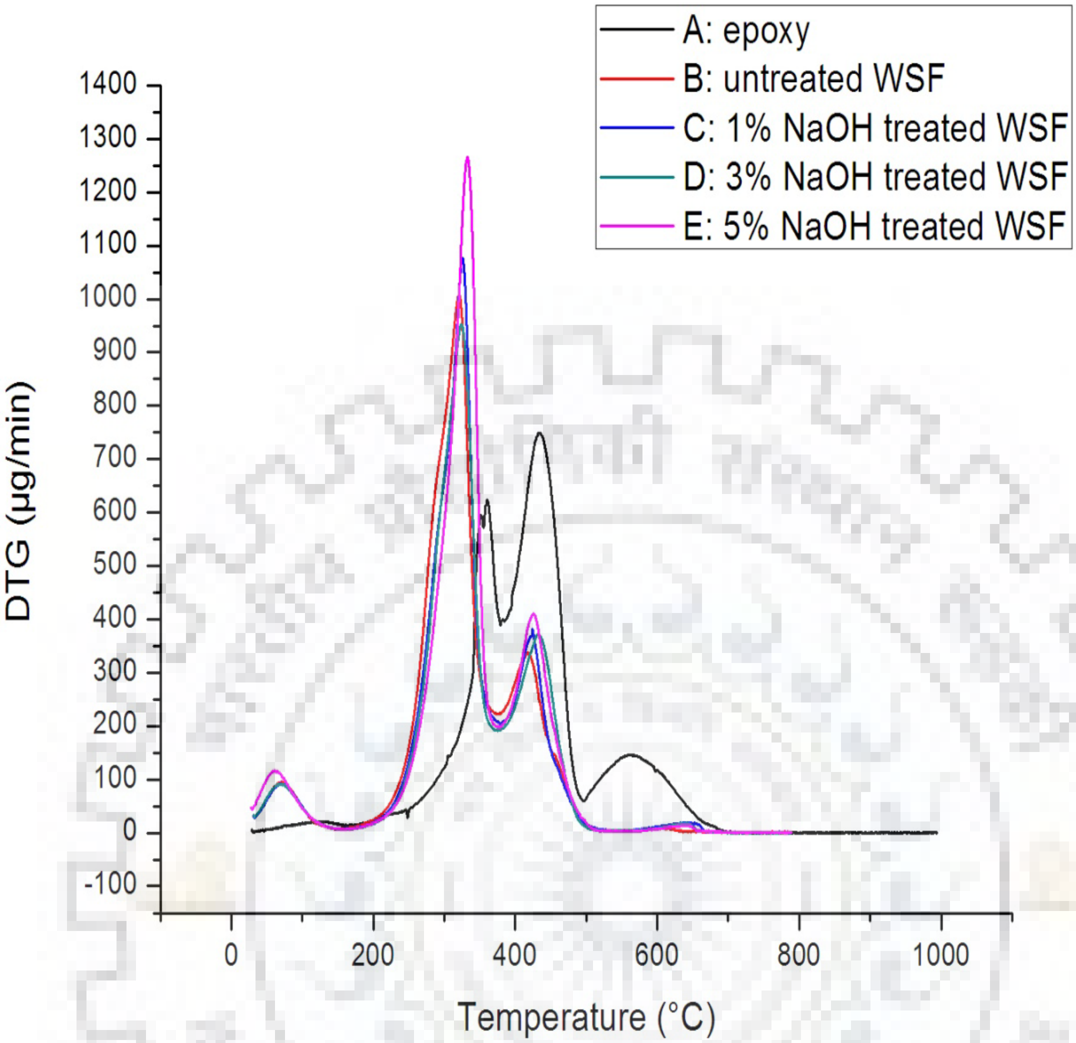

**Figure 4.29: DTG thermogram of epoxy, untreated WSF and all alkali treated WSF**

The TGA behavior of untreated, 1, 3, 5 % alkali treated 10 % WSF-epoxy composite is shown in figure 4.30 (A, B, C and D) respectively. The initial weight loss of the sample is found that at a temperature of 197°C this is due to the fact of the removal of fiber present in the polymer matrix. From figure 4.31(A, B, C, and D), the higher rate of weight reduction for the untreated fiber composite (0.73 mg/min) is observed at 366°C. This peak is shifted to the 365°C for the 1 % alkali treated fiber composite (0.86 mg/min). 3 % alkali treated fiber composite (0.73 mg/min) shows this peak at 359°C, further application of treatment i.e. 5% alkali treated fiber composite (0.9 mg/min) this peak shifted to 362°C. The last zone of the untreated fiber composite for further degradation is observed at 478°C. This point is shifted to be 500°C for 1 % alkali treated composite. 3 % alkali treated composite shows this point at

475°C and 5 % alkali treated composite shows this point at 482°C thus, thermal stability of the composite is improved.

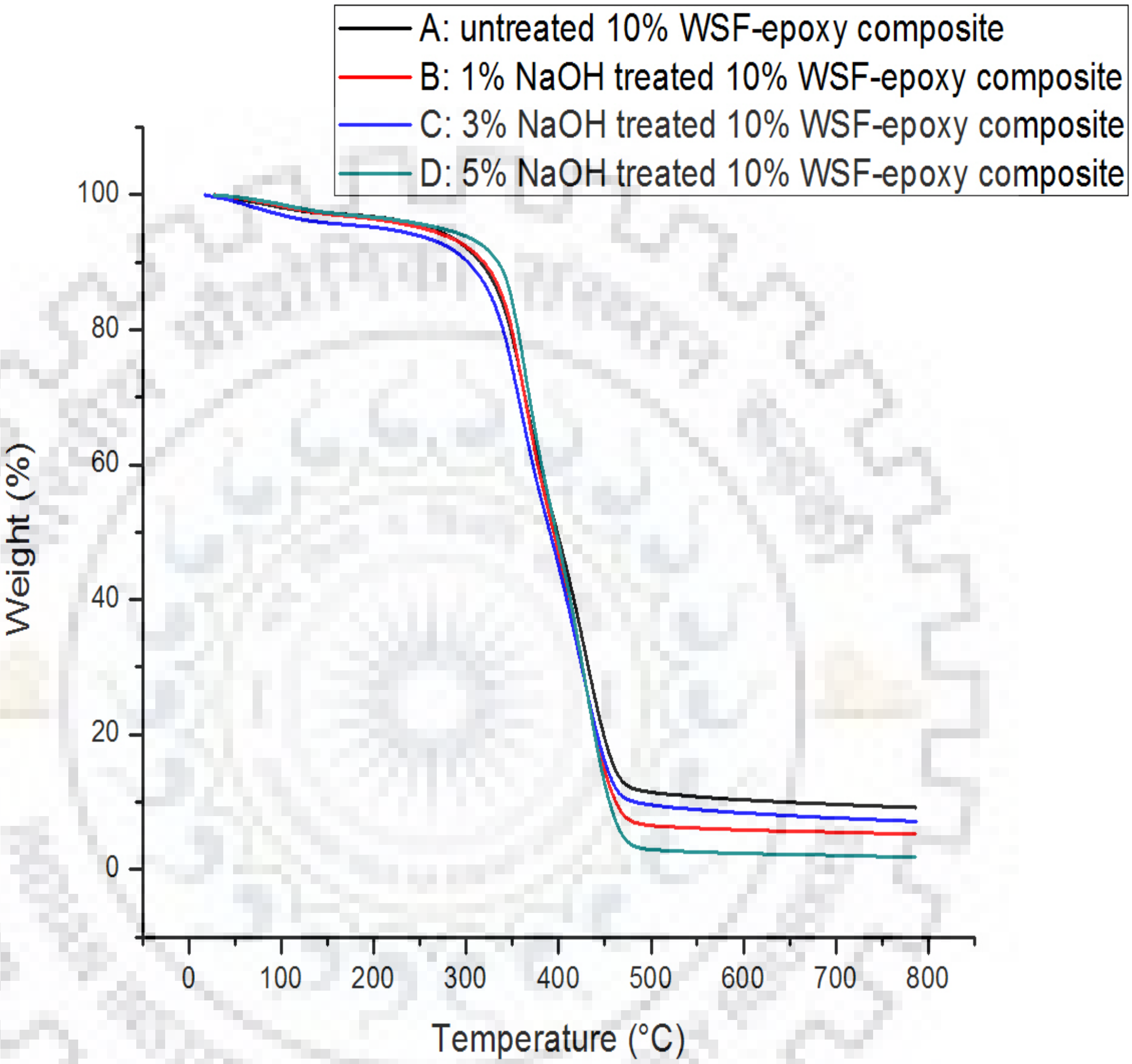

**Figure 4.30: TGA thermogram of untreated and all alkali treated 10 % WSF-epoxy composites**

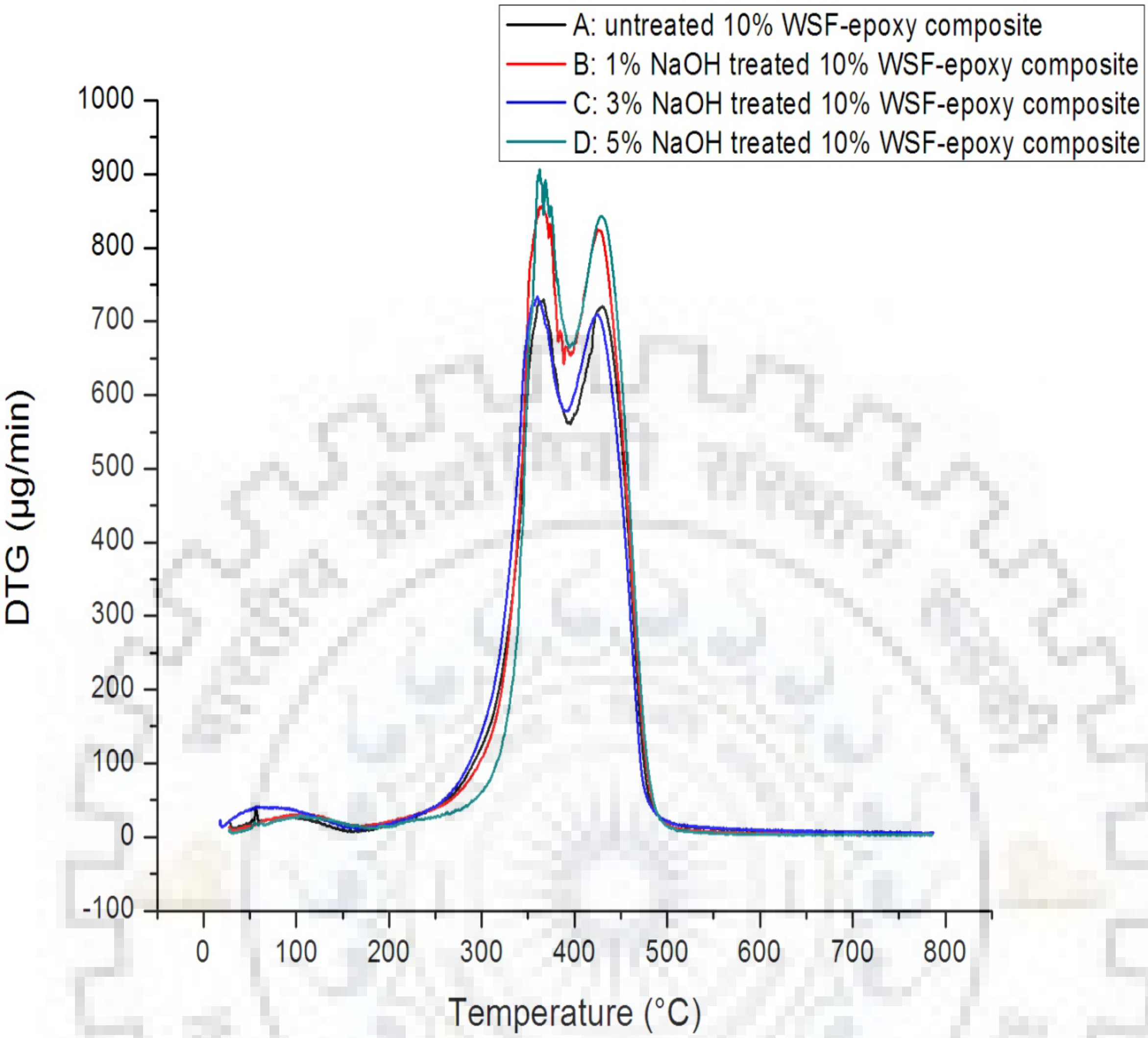

**Figure 4.31: DTG thermogram of untreated and all alkali treated 10 % WSF-epoxy composites**

The TGA behavior of untreated, 1, 3, 5 % alkali treated 20 % WSF-epoxy composite is shown in figure 4.32 (A, B, C and D) respectively. The starting weight reduction of the sample is observed at a temperature of 199°C this is due to the removal of fiber present in the polymer matrix. From figure 4.33 (A, B, C and D) higher rate of weight reduction is observed at 367°C (0.66 mg/min) in untreated fiber composite, this is moved to 368°C (0.76 mg/min) for 1 % alkali treated fiber composite. Further increment of alkali concentration i.e. (3 %), this peak is shifted to 360°C (0.74 mg/min), subsequently moved to 368°C (0.85 mg/min) for 5 % alkali treated composite. The degradation of the remaining part of the product for the further degradation is found to be 477°C for untreated fiber composite, this point is moved to 500°C for 1 % alkali treated composite. 3 % alkali treated composite shows this point at 479°C and 5 % alkali treated composite shows this point at 500°C thus, thermal stability of the composite is improved.

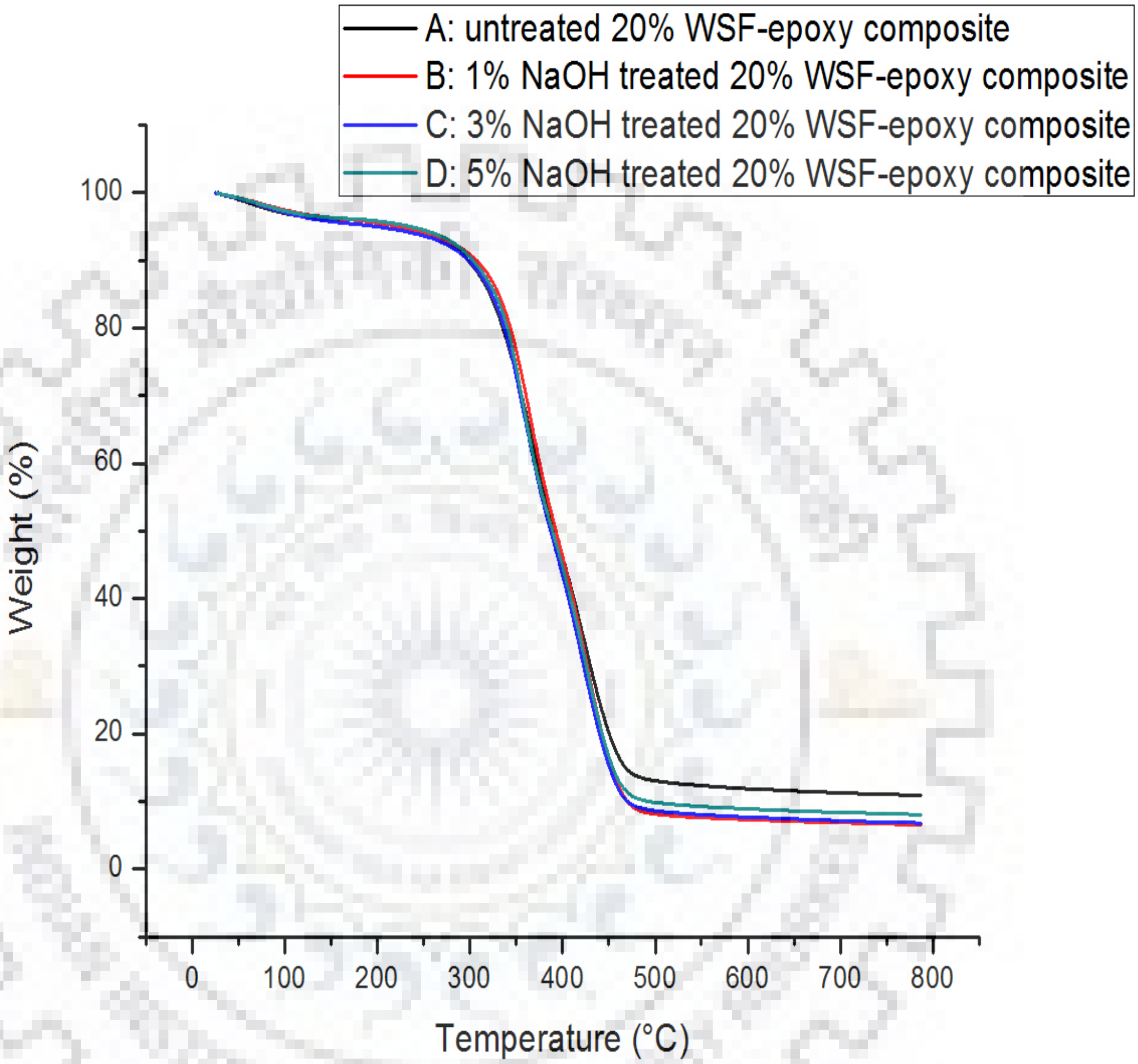

**Figure 4.32: TGA thermogram of untreated and all alkali treated 20 % WSF-epoxy composites**

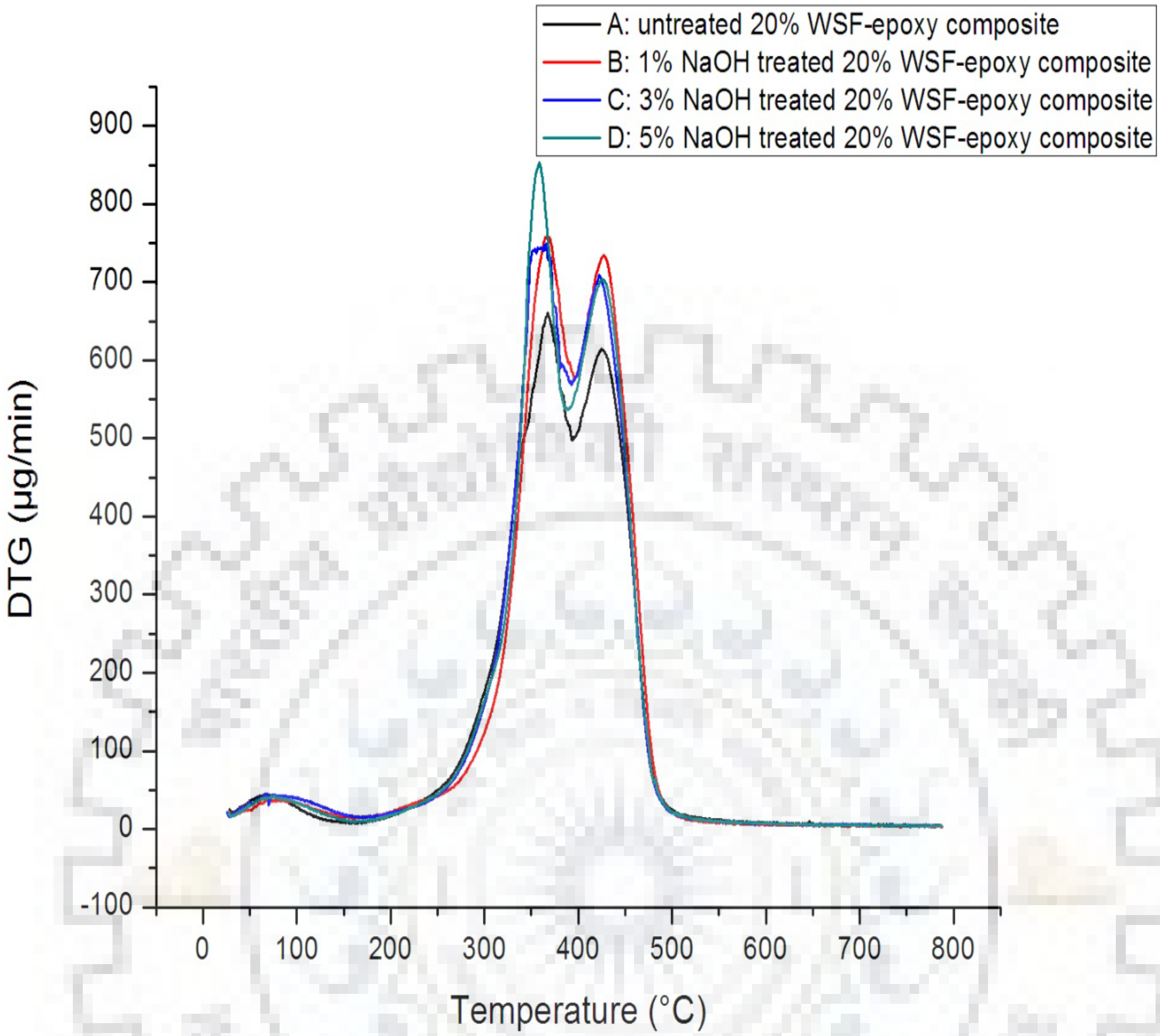

**Figure 4.33: DTG thermogram of untreated and all alkali treated 20 % WSF-epoxy composites**

The TGA behavior of untreated, 1, 3, 5 % alkali treated 30 % WSF-epoxy composite is shown in figure 4.34 (A, B, C and D) respectively. The initial weight loss of the sample is found that at a temperature of 190°C this is due to the fact of the removal of fiber present in the polymer matrix. From figure 4.35(A, B, C, and D), the higher rate of weight reduction for the untreated fiber composite (0.56 mg/min) is observed at 366°C. This peak is shifted to the 354°C for the 1 % alkali treated fiber composite (0.65 mg/min). 3 % alkali treated fiber composite (0.67 mg/min) shows this peak at 350°C, further application of treatment i.e. 5% alkali treated fiber composite (0.84 mg/min) this peak shifted to 354°C. The last zone of the untreated fiber composite and 1 % alkali treated composite for further degradation is observed at a point of 500°C. 3 % alkali treated composite shows this point at 481°C and 5 % alkali treated composite shows this point at 500°C thus, thermal stability of the composite is improved.

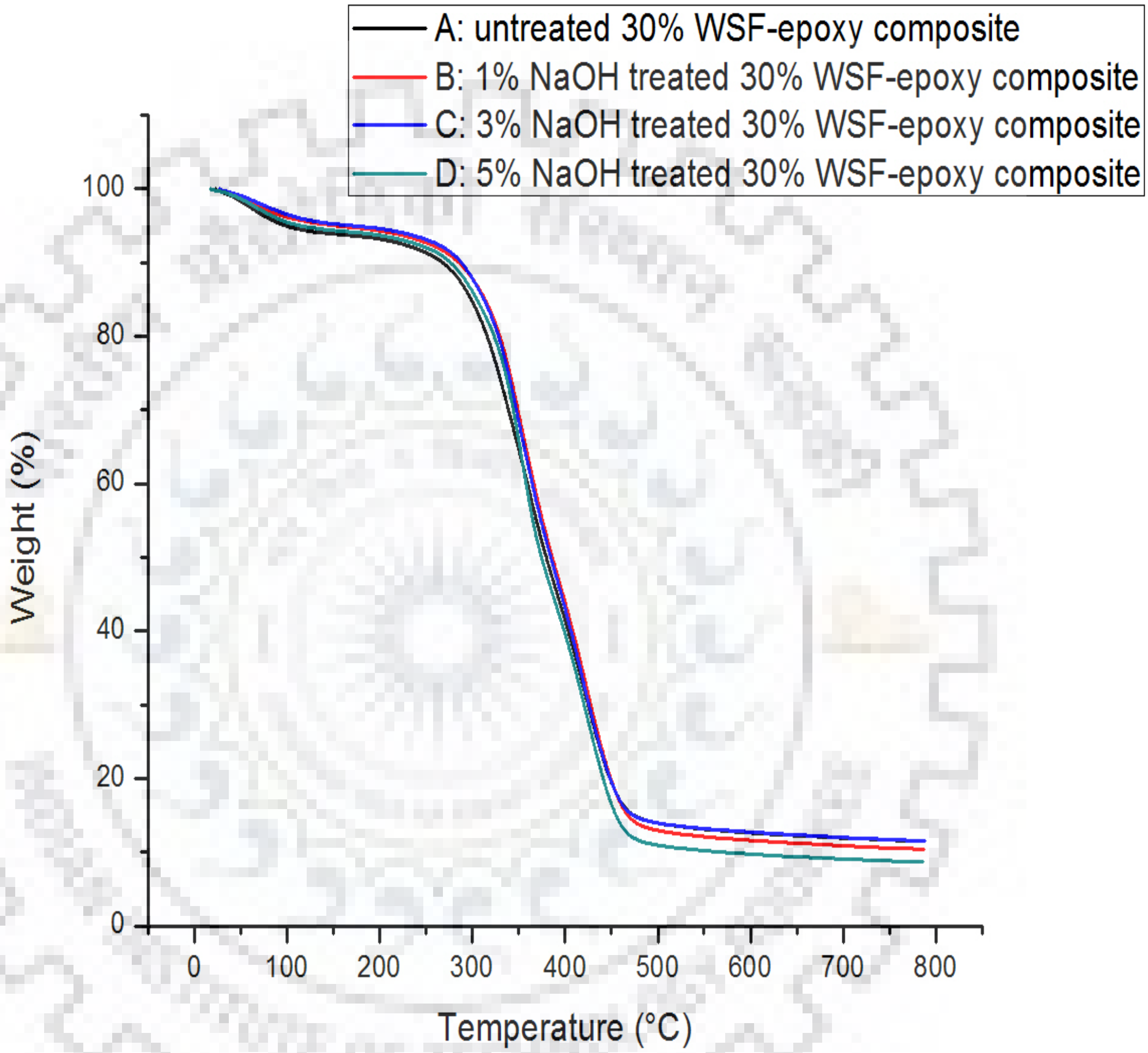

**Figure 4.34: TGA thermogram of untreated and all alkali treated 30 % WSF-epoxy composites**

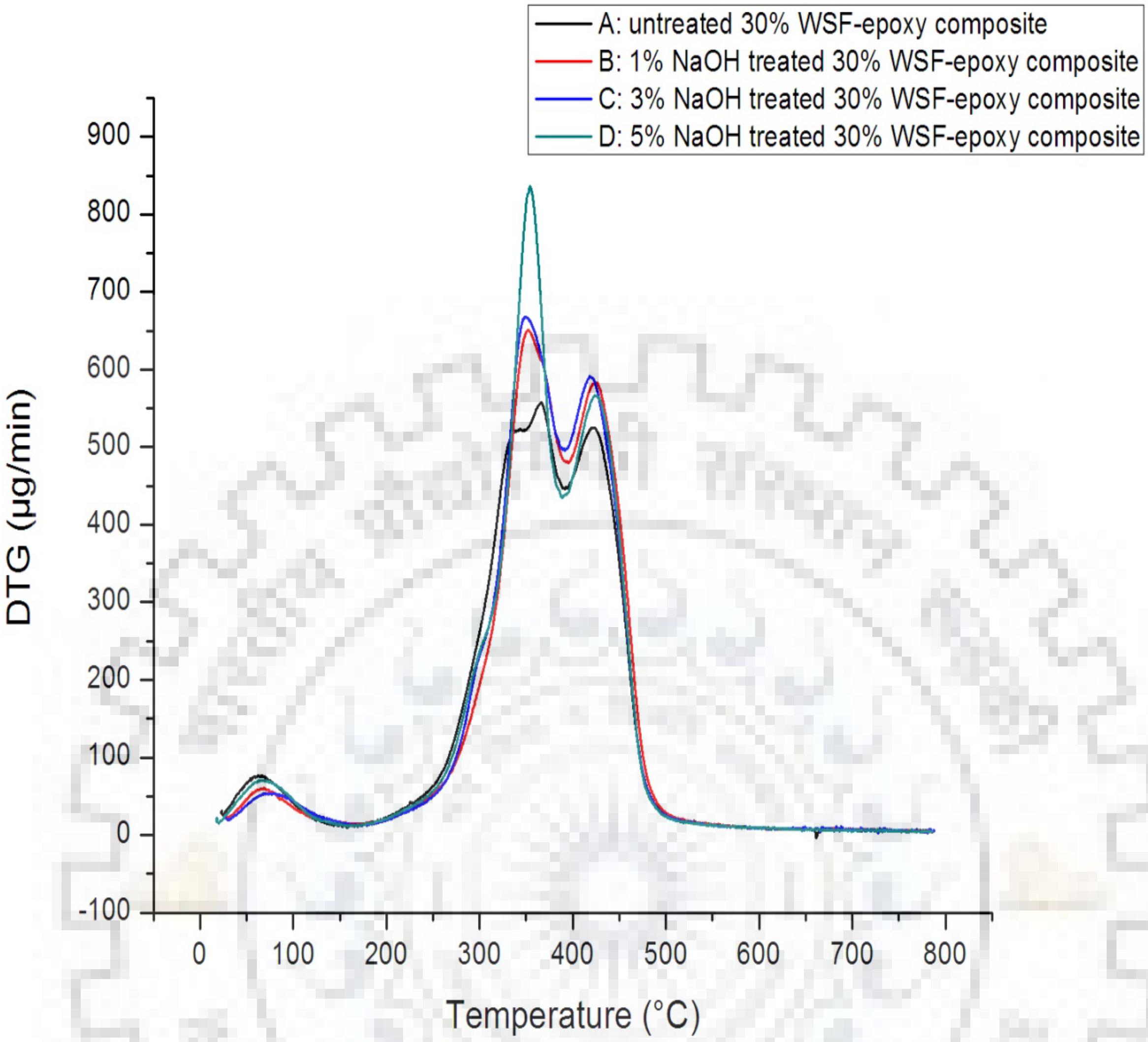

**Figure 4.35: DTG thermogram of untreated and all alkali treated 30 % WSF-epoxy composites**

The TGA behavior of untreated, 1, 3, 5 % alkali treated 40 % WSF-epoxy composite is shown in figure 4.36 (A, B, C and D) respectively. The starting weight reduction of the sample is observed at a temperature of 200°C this is due to the removal of fiber present in the polymer matrix. From figure 4.37 (A, B, C and D) higher rate of weight reduction is observed at 342°C (0.73 mg/min) in untreated fiber composite, this is moved to 347°C (0.71 mg/min) for 1 % alkali treated fiber composite. Further increment of alkali concentration i.e. (3 %), this peak having same level 347°C (0.75 mg/min), subsequently moved to 354°C (0.89 mg/min) for 5 % alkali treated composite. The degradation of the remaining part of the product for the further degradation is found to be 500°C for untreated and 1 % treated fiber composite. 3 % alkali treated composite shows this point at 481°C and 5 % alkali treated composite shows this point at 500°C thus, thermal stability of the composite is improved (Mittal and Sinha, 2016).

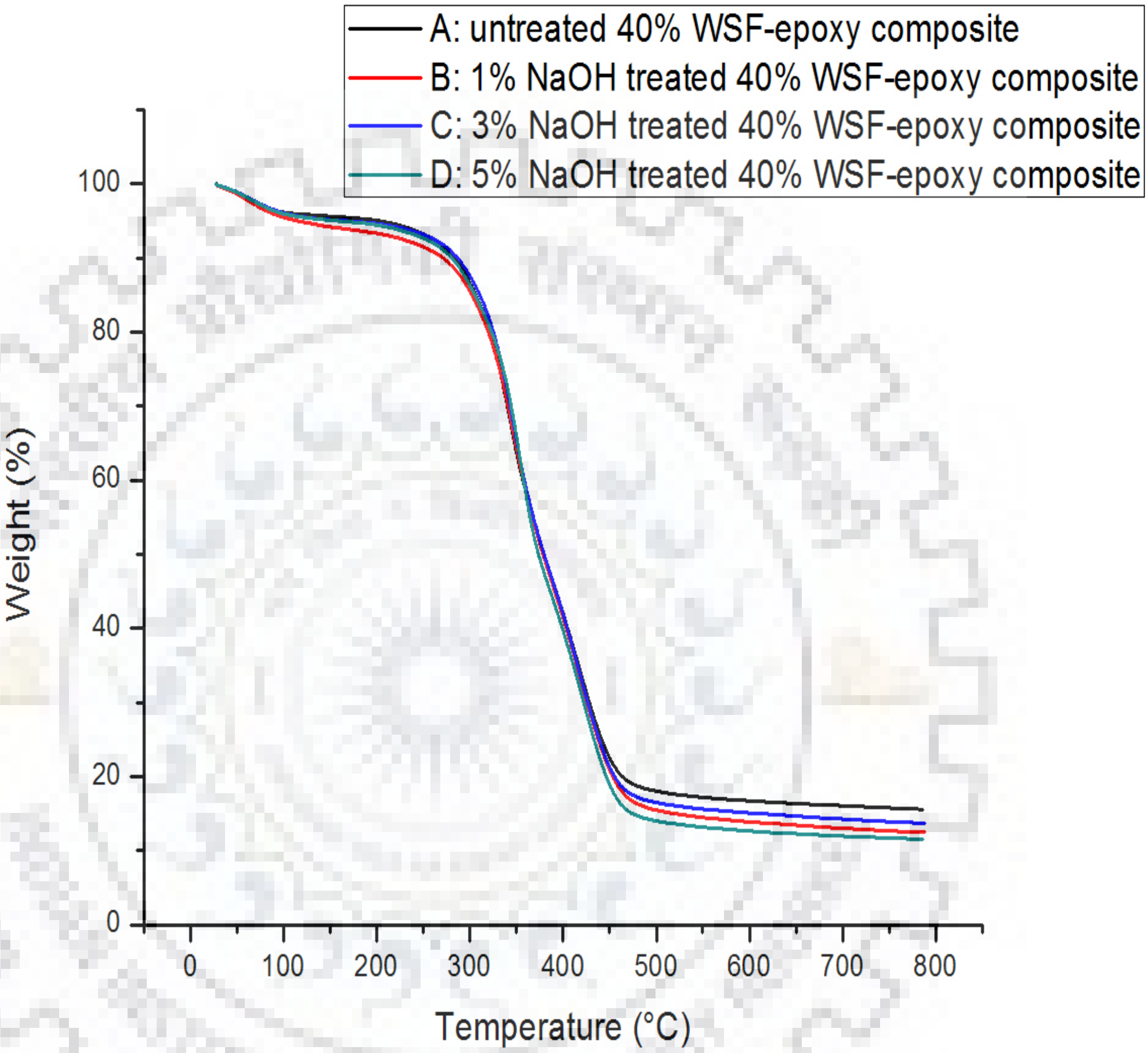

**Figure 4.36: TGA thermogram of untreated and all alkali treated 40 % WSF-epoxy composites**

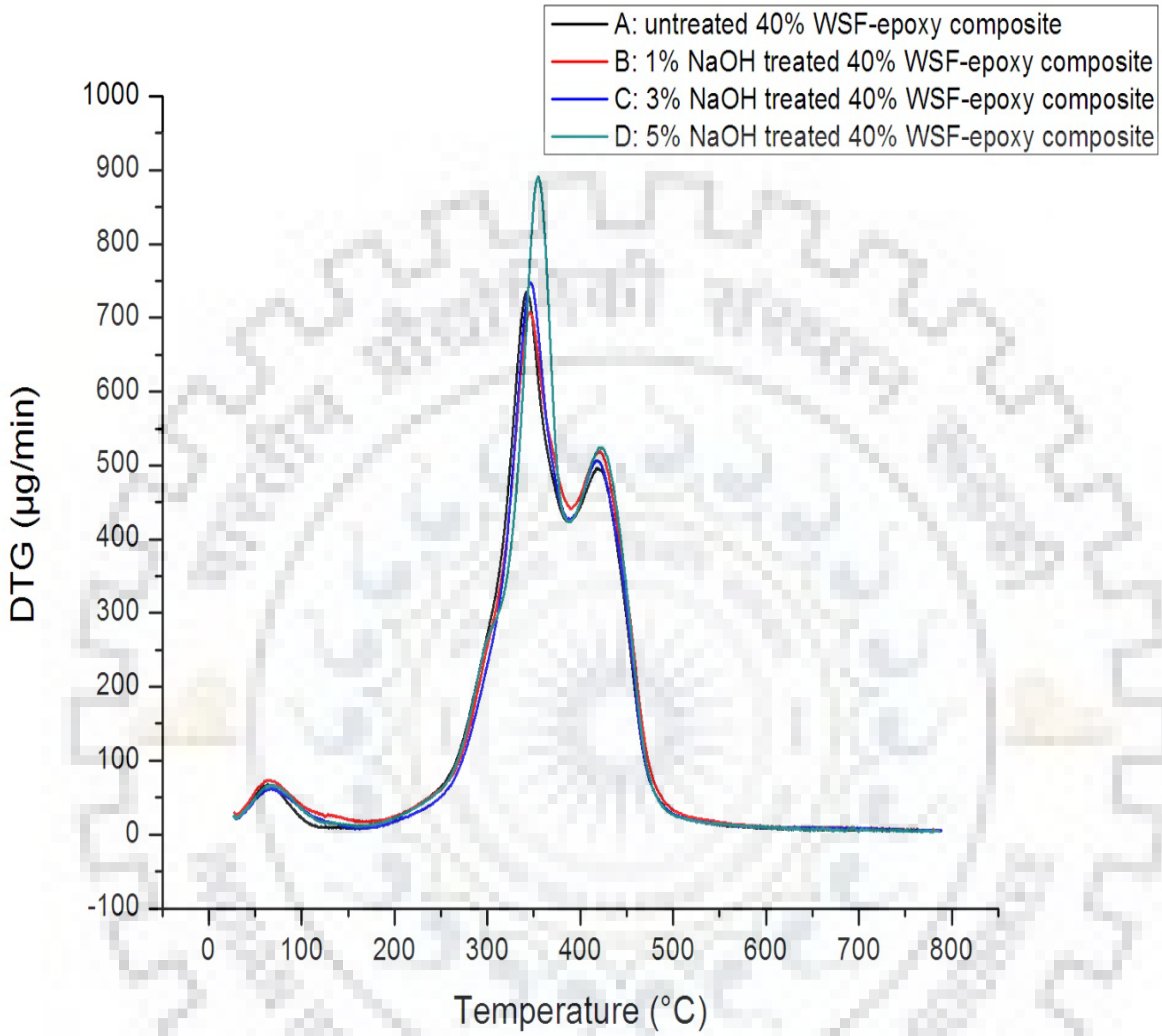

**Figure 4.37: DTG thermogram of untreated and all alkali treated 40 % WSF-epoxy composites**

### 4.5.2 Differential Scanning Calorimetry (DSC) analysis

The observation of DSC thermogram of untreated WSF, 1, 3 and 5 % alkali treated WSF is shown in figure 4.38. DSC thermogram has distinct exothermic peaks which represent heat released from the sample; it can be seen in the figure 4.38. The corresponding ΔH value is given in table 4.4.

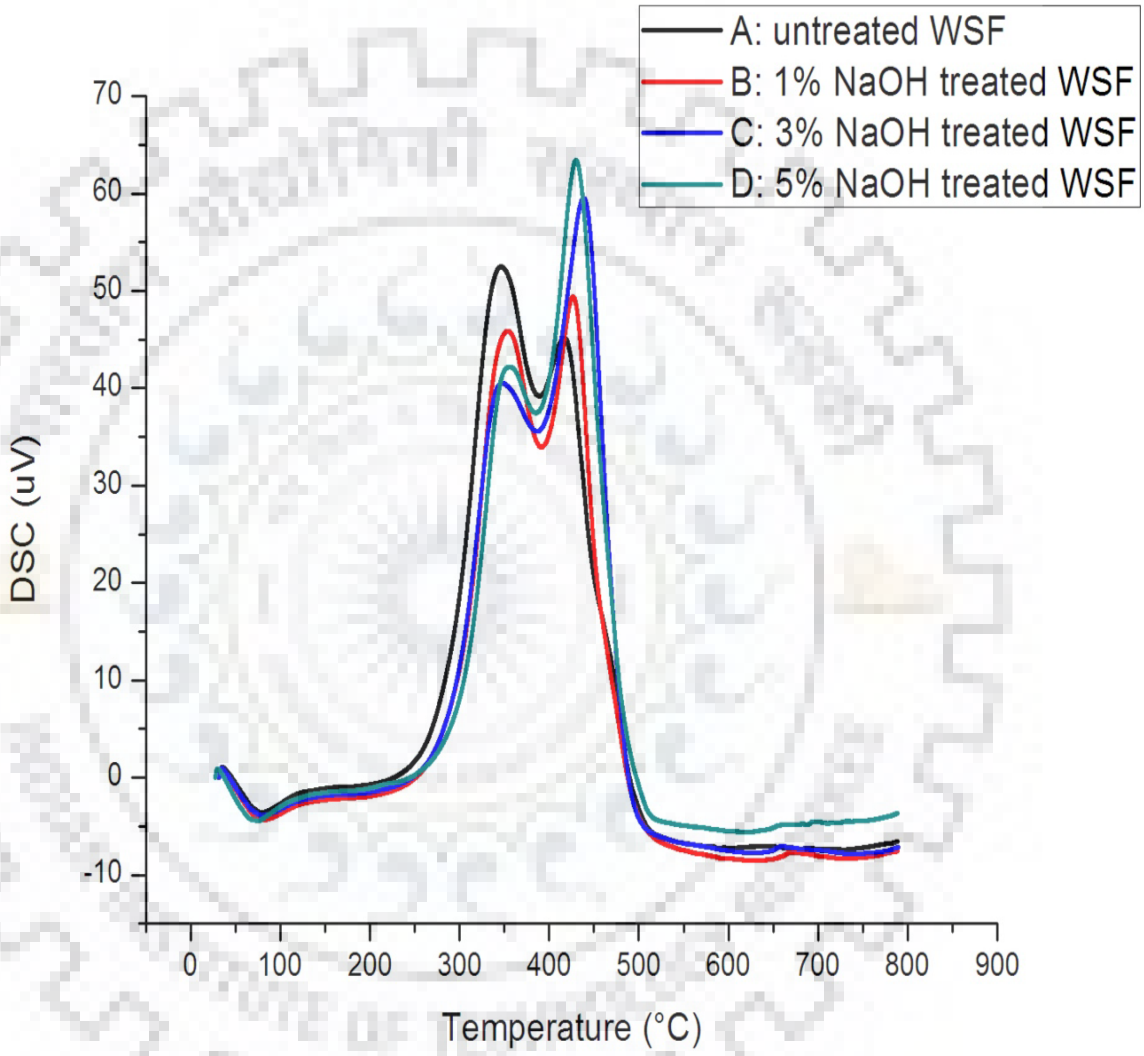

**Figure 4.38: DSC thermogram of untreated and all alkali treated fiber**

**Table 4.2 Result of Differential Scanning Calorimeter (DSC) analysis of untreated WSF and alkali treated WSF**

| Sample | Peak temperature (°C) | Nature of peak | ΔH (J/mg) |
|---|---|---|---|
| **Untreated WSF** | 345 | Exothermic | -4.24 |
| | 417 | Exothermic | -4.24 |
| **1% alkali treated WSF** | 354 | Exothermic | -3.77 |
| | 427 | Exothermic | -3.77 |
| **3% alkali treated WSF** | 345 | Exothermic | -4.19 |
| | 438 | Exothermic | -4.19 |
| **5% alkali treated WSF** | 357 | Exothermic | -3.97 |
| | 429 | Exothermic | -3.97 |

From figure 4.38, the DSC thermogram of the untreated WSF (A) has two exothermic peaks, at 345°C and at 417°C, this is due to the decomposition of hemicelluloses and cellulose present in the WSF. First peak is shifted towards higher temperature 354°C for 1 % alkali treated WSF (B), this peak moved back towards lower temperature 345°C for 3 % alkali treated WSF (C), further treatment of fiber caused the peak shifted to higher temperature 357°C for 5 % alkali treated WSF (D) this is because of removal of non-cellulosic matter such as hemicelluloses, pectin etc. During the application of alkali treatment, fiber losses its original structure and converted into the zones of short microfibril which are joined together with a physical bond this may have some influence, in shifting the peak (Ray et al., 2001). The second exothermic peak of 1 % alkali treated WSF (B) at 427°C this is due to degradation of cellulose in the WSF and formation of char. This peak of 3 % alkali treated WSF (C) is found at 438°C. Further, increment of the alkali concentration i.e. (5% alkali treatment of WSF) peak moved back to temperature 429°C. High alkali concentration is destroying the fibrils of fiber which produces negative effect on the strength of the fiber (Li et al., 2007).

## 4.6 FOURIER TRANSFORM INFRARED SPECTROSCOPY (FT-IR) ANALYSIS

Fourier transform infrared spectroscopy is a practical tool for obtaining rapid information about the structural and chemical changes take place in cellulose, hemicelluloses and lignin concentration caused by various treatments. Figure 4.39 shows the FTIR spectra of untreated and all alkali treated WSF in the range of 4,000 to 400 cm$^{-1}$. Untreated WSF has a peak at 3375 cm$^{-1}$ which relates with the hydrogen-bonded –OH stretching; this may be attributed to vibration present in lignin (Schwanninger et al., 2004; Boricha and Murthy, 2008). The stretching of OH bonded to methyl group shows the hydrophilic nature and C-H stretch observed a signal at 2857.12 cm$^{-1}$. (Bellamy, 1954; Rajesh et al., 2011) The peak at 604 cm$^{-1}$ band may be attributed to the C-alkyl halide stretch. For 1% alkali treated WSF spectra, these peaks are disappeared and observed a new peak 667 cm$^{-1}$ that related to the C-H bond in alkynes (Stewart et al., 1995).

3 % alkali treated WSF has the new band at 2365 cm$^{-1}$ that related to the P-H stretch group. Peak of 605 cm$^{-1}$ is observed that may give the C-alkyl halide stretching, vibration, and all other peaks are same as the previously treated fiber. 5 % alkali treated fiber has the peak band at 656 cm$^{-1}$ that is responsible for C-H bond which disappeared is due cause of time.

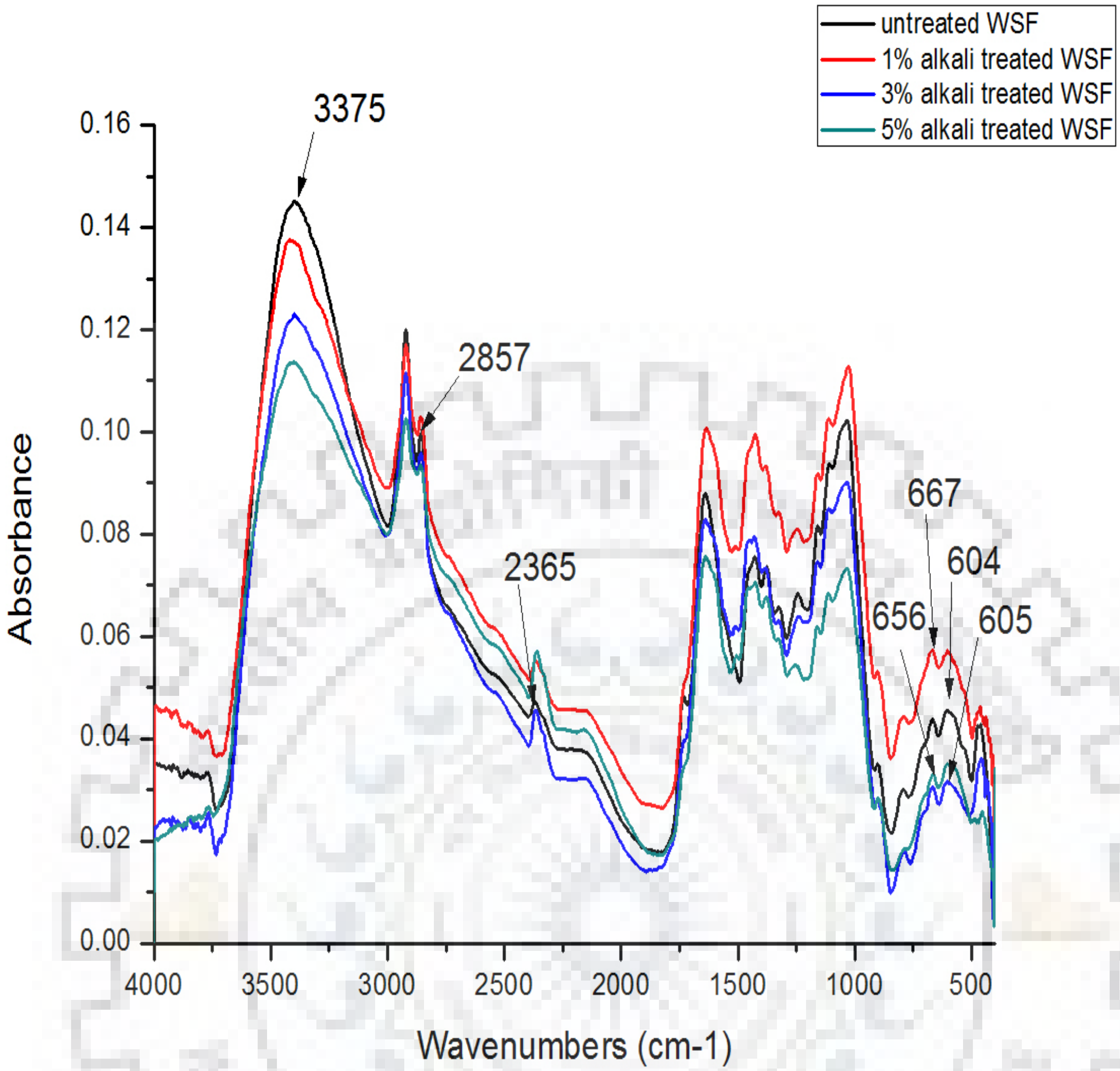

**Figure 4.39: FTIR spectra of untreated and all alkali treated WSF**

## 4.7 EFFECT OF BLEND RATIO OF BAGASSE AND WHEAT STRAW FIBER ON MECHANICAL PROPERTIES OF THE BLENDED EPOXY COMPOSITE

### 4.7.1 Mechanical analysis

The significance of the mechanical properties lies in the estimation of the reinforcing potential of the particular system. However, the mechanical properties can also provide the information about the surface interface of fiber and polymer resin. The determined values of the mechanical properties of blended composites are shown in table 4.3. From the results, it is evident that on increasing weight percentage of BF in WSF-epoxy composite, the mechanical properties also increase; this is probably due to the better inherent properties exhibited by BF. The mechanical properties like tensile strength, flexural strength and flexural modulus are determined to be superior for 20/80 weight ratio of the WSF/BF reinforced epoxy blended composites because BF has high cellulose content than the WSF which provides more micro-fibrils surface for adhesion to the polymer matrix. The alkali treatment breaks these fibrils into various filaments that provide a good interface between the fiber and the polymer matrix (Ray et al., 2001). The superior values of tensile strength, flexural strength, and flexural modulus are 18.9 MPa, 39.1 MPa and 2585 MPa, respectively at 20/80 weight ratio of the WSF/BF, however the superior value of tensile modulus, is observed at 40/60 weight ratio of the WSF/BF reinforced epoxy blended composites, with magnitude of 1378.7 MPa.

Impact strength is defined as the ability of the material to resist fracture under stress applied at high speed. From the results, superior value of the impact strength is observed at 20/80 weight ratio of the WSF/BF reinforced epoxy blended composites with the magnitude of 31 J/m. The impact failure of a composite occurs because of factors like breakage of fiber, low fiber-polymer adhesion and fiber pull out from the resin. The applied load may exceed the fiber/polymer bonding leading to breakage of fiber (Cao et al., 2006). It has been observed that impact strength gradually increases on increasing the bagasse content therefore more content of BF in WSF-epoxy composite enhances the impact properties.

### 4.7.2 Scanning Electron Microscopy (SEM)

It is evident from the SEM morphology that the fractured surfaces of various blended composite specimens, the tensile fracture surface of the weight ratio of WSF/BF (80/20 and 60/40) blended composites has a thick void because fiber is being pulled out from the polymer matrix during the testing as shown in figure 4.40(a) and figure 4.40(b).

On the other side, SEM micrograph of the tensile fracture surface of the weight ratio of WSF/BF (40/60 and 20/80) blended composites shows breakage of fiber attached with the polymer matrix during the test as shown in figure 4.40(c) and figure 4.40(d). This implies that WSF/BF (40/60 and 20/80) blended composites have better interfacial adhesion between the fiber and the polymer matrix, contributing effectively for improvement in mechanical properties.

**Table 4.3: Mechanical properties of WSF/BF epoxy blended composites**

| Weight ratios of WSF/BF | Tensile strength (MPa) | Tensile modulus (MPa) | Flexural strength (MPa) | Flexural modulus (MPa) | Impact strength (J/m) |
|---|---|---|---|---|---|
| **80/20** | 16 | 1178.7 | 32 | 1885.7 | 21.2 |
| **60/40** | 16.6 | 1313.7 | 34.4 | 2107.3 | 22.2 |
| **40/60** | 18 | 1378.7 | 35.1 | 2315.7 | 26.1 |
| **20/80** | 18.9 | 1176 | 39.1 | 2585 | 31 |

### 4.7.3 Water absorption

The water absorption properties of the entire blended composite are calculated in terms of percentage intake of water in the composite specimen (dipped in water at room temperature). The increase in weight percentage is shown in figure 4.41. It has been found that increase of BF content in the WSF-epoxy composite reduces the water absorption properties of the prepared composite. Blended composite with a weight ratio of WSF/BF (80/20) has a 10.7 % of water absorption during the respective examination period. On the other hand, blended composite with a weight ratio of WSF/BF (20/80) showed water absorption up to 8.4 % as BF has more content of lignin than the WSF that provides good resistance to water absorption. (Satyanarayana et al., 1981)

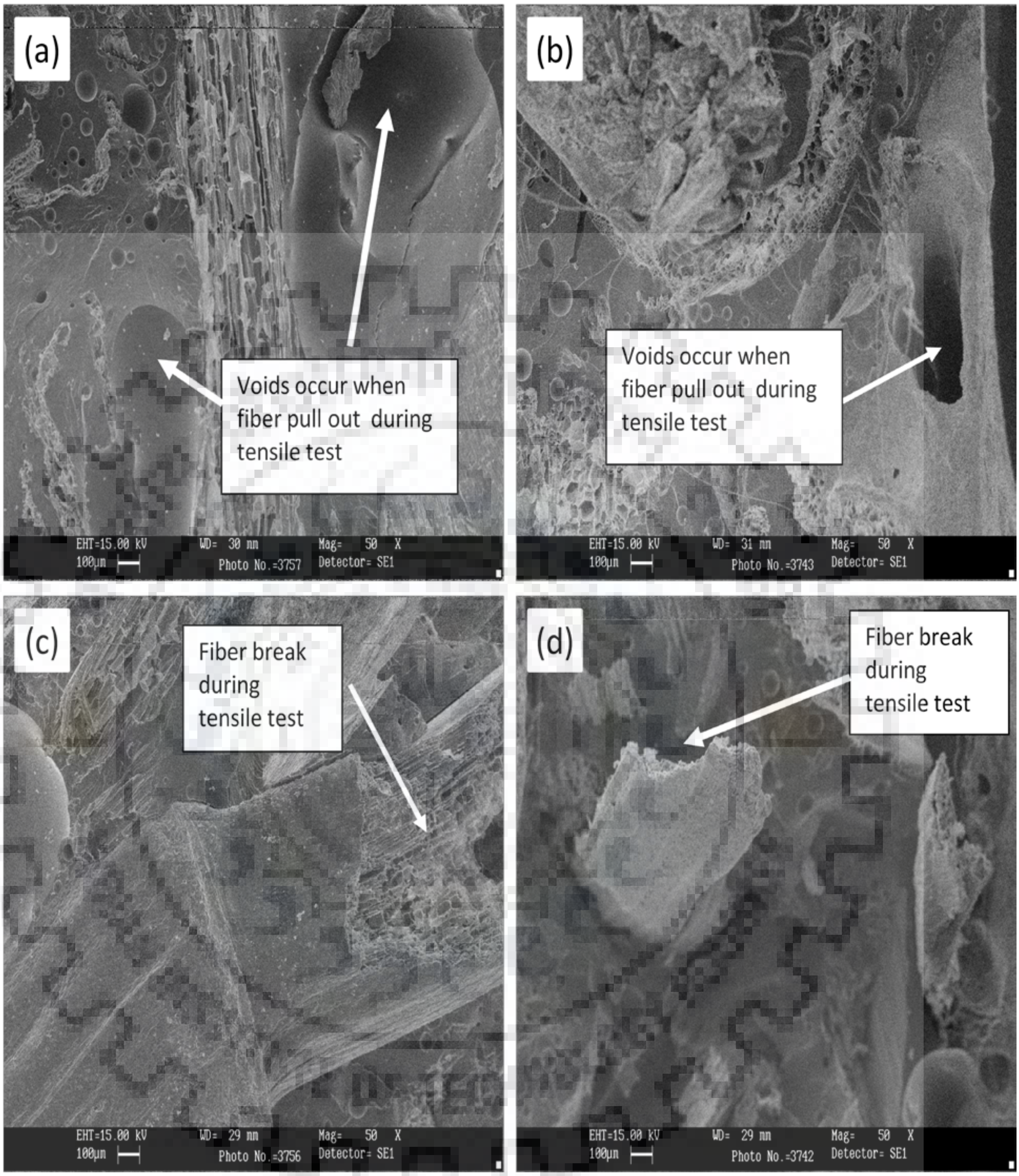

**Figure 4.40: SEM morphology of tensile fractured surface of the weight ratio of all hybrid composite (a) WSF/BF(80/20) (b) WSF/BF(60/40) (c) WSF/BF(40/60) (d) WSF/BF(20/80)**

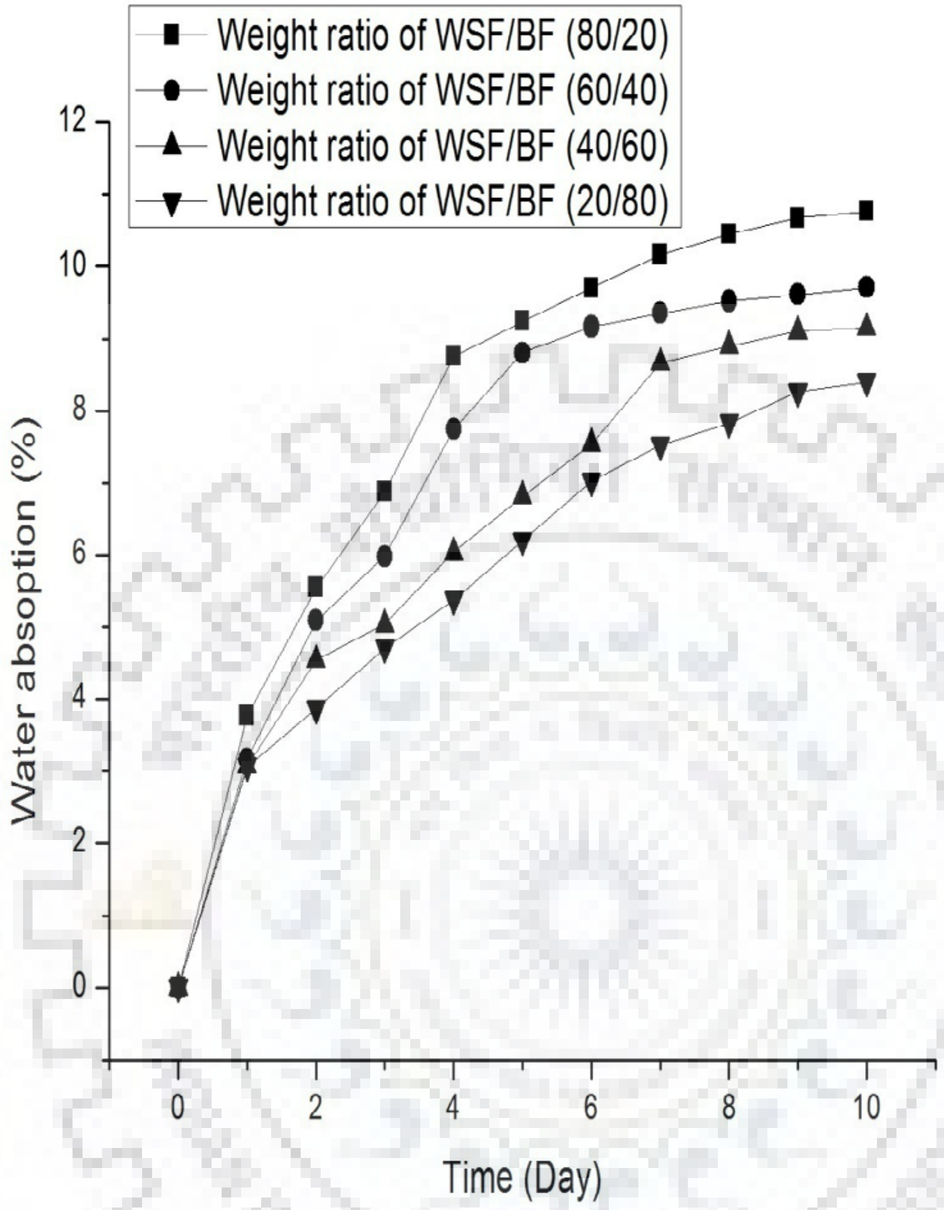

Figure 4.41: Water absorption curve for all (WSF/BF) blended composites

## 4.8 EFFECT OF BLEND RATIO OF BAGASSE AND WHEAT STRAW FIBER ON THERMAL PROPERTIES OF THE BLENDED EPOXY COMPOSITE

### 4.8.1 Thermo Gravimetric Analysis (TGA) and Differential Thermal Gravimetric (DTG) analysis

The thermal properties of all blended composites are analyzed by the TGA and DTG. TGA and DTG thermograms of WSF/BF blended composites are shown in figure 4.42 and figure 4.43 respectively. In TGA thermograms, there are three different zones of degradation. In the first zone, there is a small loss in weight due to the removal of moisture present in the sample. Because of the onset of the thermal decomposition process, a very high loss in weight in the mid zone of degradation and the last zone called the ultimate thermal degradation.

The TGA behavior of weight ratio of WSF/BF (80/20) reinforced epoxy blended composite is shown in figure 4.42 (A). The starting weight reduction of the sample observed at temperatures of 200°C this is due to the removal of fiber present in the polymer matrix. From figure 4.43 (A) higher rate of weight reduction is (0.84 mg/min) observed at 353°C due to degradation of the blended composite. The degradation of the remaining part of the product for the further degradation observed at 637°C.

The TGA behavior of weight ratio of WSF/BF (60/40) reinforced epoxy blended composite is shown in figure 4.42 (B). The starting weight depletion of the sample observed at a temperature of 199°C this is due to the removal of fiber present in the polymer matrix. From figure 4.43(B) higher rate of weight reduction is (0.79 mg/min) observed at 351°C due to degradation of the blended composite. The degradation of the remaining part of the product for the further degradation observed at 649°C. Here the final degradation moved to a higher temperature. It has been observed that blended effect the thermal stability of the composite.

The TGA behavior of weight ratio of WSF/BF (40/60) reinforced epoxy blended composite is shown in figure 4.42 (C). The starting weight depletion of the sample observed at a temperature of 199°C this is due to the removal of fiber present in the polymer matrix. From figure 4.43 (C) higher rate of weight reduction is (0.56 mg/min) observed at 439°C due to degradation of the blended composite. Here the mid-period degradation moved to a higher temperature, improved the thermal stability of the composite. The degradation of the remaining part of the product for the further degradation observed at 648°C.

The TGA behavior of weight ratio of WSF/BF (20/80) reinforced epoxy blended composite is shown in figure 4.42 (D). The starting weight reduced of the sample observed at a temperature of 197°C this is due to the removal of fiber present in the polymer matrix. From figure 4.43 (D) higher rate of weight reduction is (0.66 mg/min) observed at 436°C due to degradation of the blended composite. The degradation of the remaining part of the product for the further degradation observed at 646°C. Here the both mid-period and final degradation moved to lower temperature.

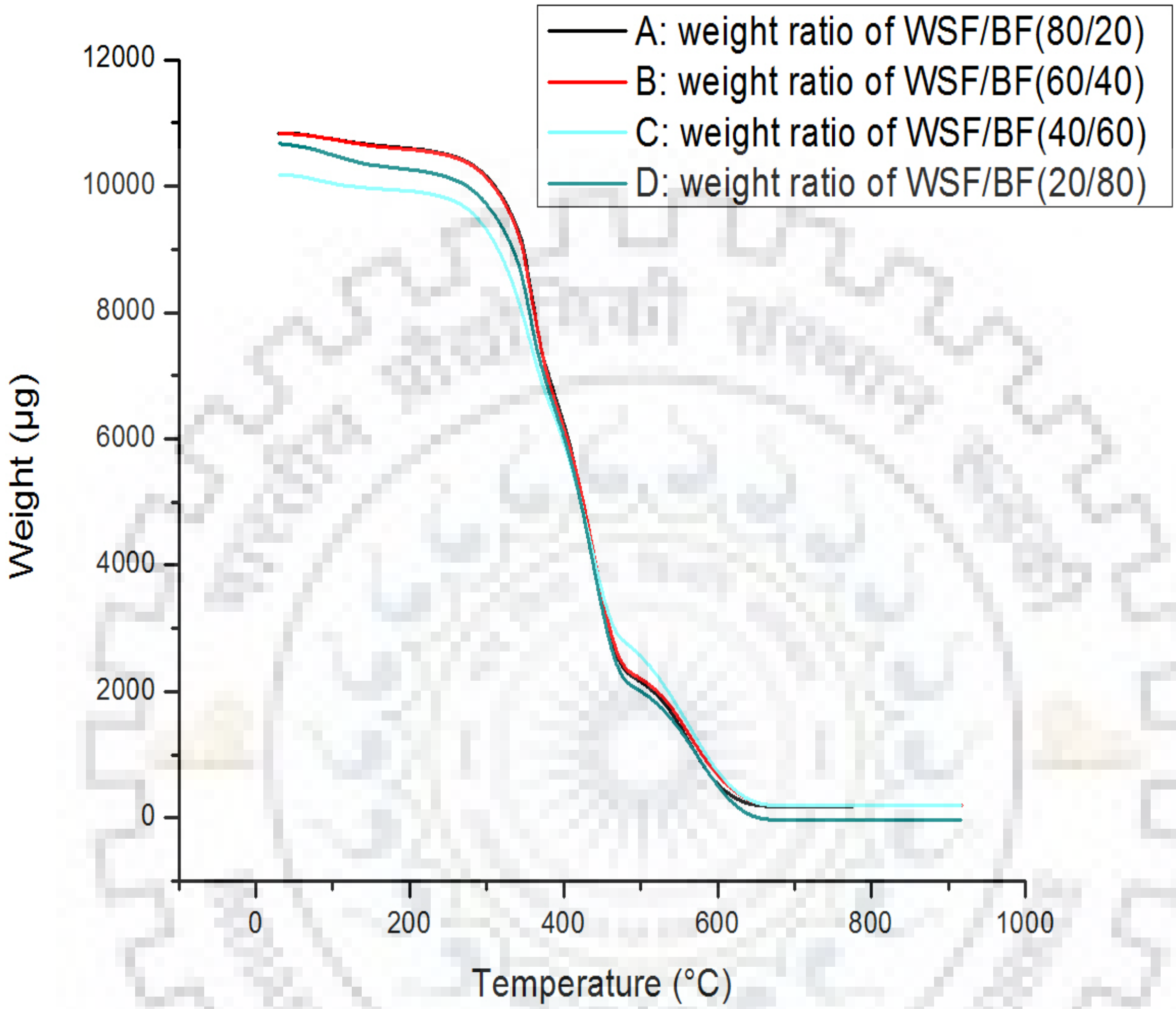

Figure 4.42: TGA curve of all (WSF/BF) blended composites

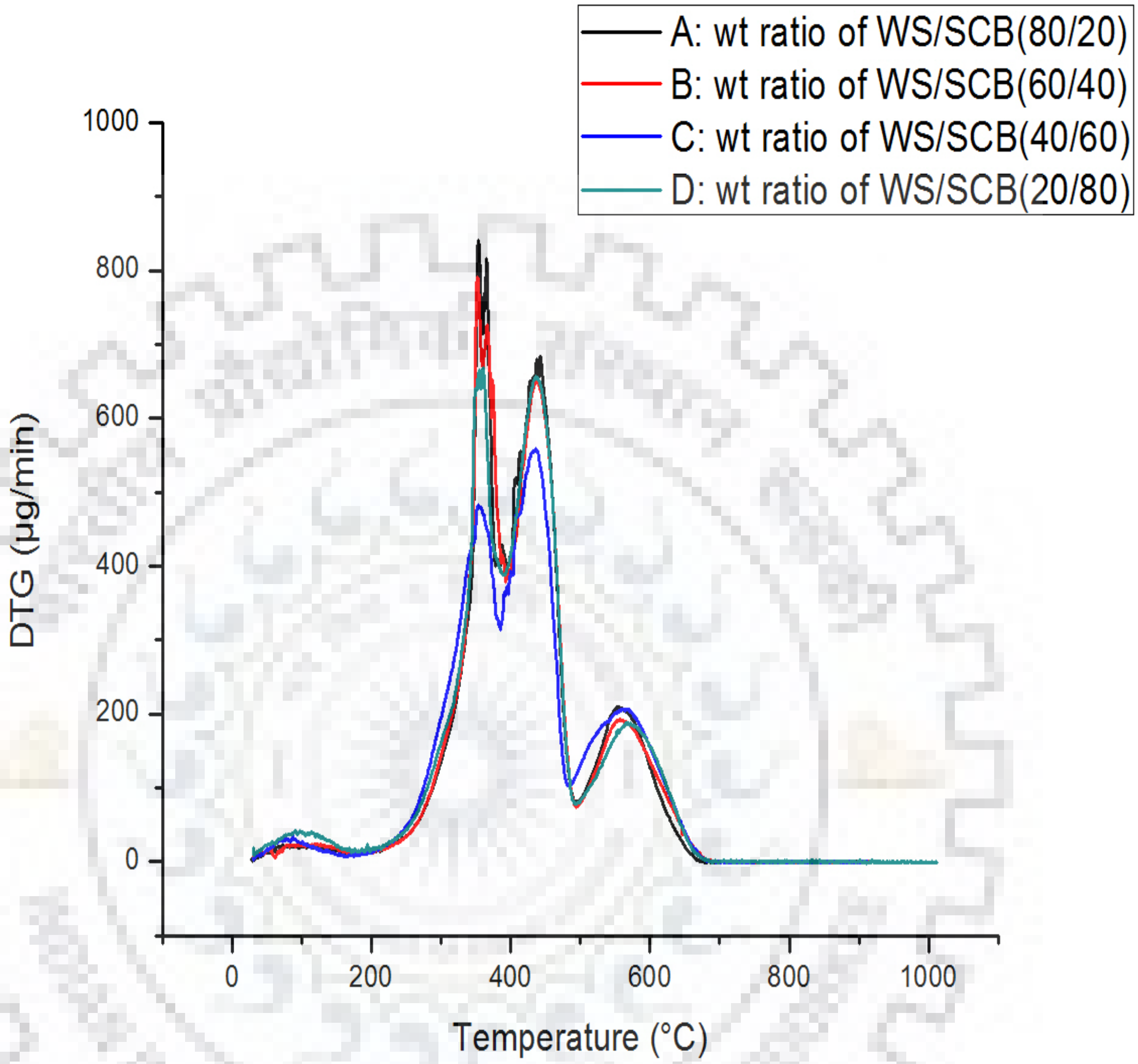

Figure 4.43: DTG curve of all (WSF/BF) blended composites


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# 5

# CONCLUSIONS AND RECOMMENDATIONS

## 5.1 CONCLUSIONS

The main accomplishments and conclusions of the present research work may be summarized as follows:

- Initially, the effect of wt % (5,10,15,20 and 25) of Bagasse Fiber (BF), effect of modifier (1 % NaOH followed by 1 % acrylic acid) on the mechanical and water absorption properties of BF-epoxy composites were compared with untreated BF-epoxy composites. The following conclusions could be drawn from the experimental results, by addition of BF into epoxy matrix the tensile, flexural and impact strength of the composites gradually increases in fiber content from 0 to 15 wt %. However, beyond 15 wt % of fiber loading, there is a notable reduction in mechanical properties of composites. This decrease at high fiber content implied poor fiber-matrix adhesion. It has been observed that treatment of BF with NaOH and acrylic acid improved mechanical properties and an optimum value was attained at 15 % treated fiber loading. At this point it has been observed that 22 % improvement in tensile strength, 18 % improvement in flexural strength, 7 % improvement in impact strength and highly reduced water absorption properties as compared to the untreated fiber composite. From the morphology of Scanning Electron Microscopy (SEM), it can be verified that chemically treated fiber have improved and good adjoin surface than the untreated one. Low cost of BF can be used as an effective reinforcement with a fiber loading of 15 % to prepare good BF-epoxy composites.
- In this part, the effect of fiber content and chemical treatment on the thermal properties of BF reinforced epoxy composites has been studied. From Differential Thermal Gravimetric (DTG) thermograms, it has been observed that the thermal stability of the treated BF composite is superior as compared to the untreated BF composites. It has been found from Differential Scanning Calorimetry (DSC) thermograms that, there is a shifting of the exothermic peaks towards a high temperature of the treated fiber as compared to the untreated one. This proved that the surface structure of the fiber changed after the chemical treatment. It has been reported that treatment of BF with NaOH and acrylic acid improved the thermal stability and an optimum value, attain at

10 % BF reinforced epoxy composite and at that point; it has been observed that 3 % improvement in thermal properties compared to the untreated fiber composite. From the Fourier Transform Infrared Spectroscopy (FT-IR) analysis, it can be seen that some functional groups are removed after the chemical treatments. Thus lignin concentration is reduced, which improves the adhesion between the fiber and polymer surfaces, demonstrating the structure transformation induced by thermal stability.

- The epoxy composites reinforced by the untreated, 1, 3 and 5 % alkali treated Wheat Straw Fiber (WSF) at various (5, 10, 15, 20 and 25) wt % were prepared respectively. Tensile, flexural and impact strength of all WSF-epoxy composites reduced with an addition of fiber in the polymer matrix. All alkali treated WSF/epoxy composites exhibited better mechanical properties than those of untreated WSF/epoxy composites having the same fiber loading. This improvement is because of treatment creates the rougher surfaces which provides a strong bonding between the fiber and polymer matrix. It has been observed that an efficient improvement in tensile strength and flexural strength of composite which prepared with the 3 % NaOH treated fiber and impact strength of composite prepared with the 5 % NaOH treated fiber. Morphological studies revealed that fibrils in the untreated fiber were coupled together. These were split after an application of the alkali treatment thereby, offer more surface available for adhesion between the fiber and polymer that raised the strength of the composites.
- The thermal properties of all composition of untreated and alkali treated WSF reinforced epoxy composites were investigated. From TGA and DTG thermograms, it has been observed that the thermal stability of the high concentration alkali treated WSF/epoxy composite is 3 % superior to the untreated and another level of alkali treated fiber composite. It has been found that from DSC thermograms, there is a shifting of the exothermic peaks towards a higher temperature of the treated fiber as compared to the untreated one. FT-IR measurement of the fibers revealed that the partial removal of hemicelluloses and lignin due to the alkali treatment of the WSF.
- Finally, mechanical, thermal and water absorption properties of WSF and BF reinforced epoxy blended composites has been studied. It has been observed that mechanical properties such as tensile strength, flexural strength, impact strength and water absorption are superior for the weight ratio of WSF/BF (20/80) reinforced epoxy blended composites. The thermal property are superior for the weight ratio of WSF/BF (80/20) reinforced epoxy blended composites. It has been found that the addition of bagasse fiber in the wheat straw-epoxy composites, the 18 % increase in tensile

strength, a 22 % increase in flexural and 46 % increase in impact strength achieved. On comparing the tensile strength of the WSF/BF (with the weight ratio 20/80) reinforced epoxy composite with palmyra/polyester composite (Dabade et al., 2006) the former is found to have 45.49 % more tensile strength. Comparison with woven banana/epoxy composite (Sapuan et al., 2006) showed the tensile strength of the WSF/BF (20/80) reinforced epoxy composite to be greater by 13.78 %. Similarly, impact strength of the WSF/BF (with the weight ratio 20/80) reinforced epoxy composite is 70.04 % higher than jute/banana blended epoxy composite (Boopalan et al., 2013). Upon analysis through SEM of fracture surface, WSF/BF (with the weight ratio 80/20 and 60/40) reinforced epoxy blended composites are found to have voids due to fiber being pull-out, WSF/BF (with the weight ratio 40/60 and 20/80) reinforced epoxy blended composites showed break up of fiber during testing due to superior bonding between the fiber and the polymer.

## 5.2 RECOMMENDATIONS

- In the present work acrylic acid treatment was applied to the bagasse fiber and found that it has positive effect on the mechanical and thermal properties of the BF reinforced epoxy composite. There are several other bio-fibers and surfaces of those can also be modified with this treatment and ultimately leads to improvement of properties , these fibers can also reinforce the different polymer resins at the same time it should not affect the mechanical strength of the composite. Mechanical and thermal properties of composites can also be studied by using the coupling agent and varying the fiber length.

# REFERENCES

Acharya, S.K.; Mishra, P.; Mehar, S.K. Effect of surface treatment on the mechanical properties of bagasse fiber reinforced polymer composite. Bio Resources, **2011,** 6(3), 3155-3165.

Agrawal, R.; Saxena, N.S.; Sharma, K.B.; Thomas, S.; Sreekala M.S. Activation energy and crystallization kinetics of untreated and treated oil palm fiber reinforced phenol formaldehyde composites. Material Science and Engineering A, **2000**, 277(1), 77-82.

Agunsoye, J.O.; Aigbodion, V.S. Bagasse filled recycled polyethylene bio-composites: Morphological and mechanical properties study. Results in Physics, **2013,** 3, 187–194.

Ahmed, K.S.; Khalid, S.S.; Mallinatha, V.; Kumar, S.J.A. Dry sliding wear behavior of SiC/$Al_2O_3$ filled jute/epoxy composites. Materials and Design, **2012**, 36, 306–315.

Alamri, H.; Low, I.M. Mechanical properties and water absorption behaviour of recycled cellulose fibre reinforced epoxy composites. Polymer Testing, **2012a**, 31, 620–628.

Alamri, H.; Low, I.M.. Effect of water absorption on the mechanical properties of n-SiC filled recycled cellulose fibre reinforced epoxy eco-nanocomposites. Polymer Testing, **2012b**, 31, 810–818.

Alamri, H.; Low, I.M.; Alothman, Z. Mechanical, thermal and microstructural characteristics of cellulose fibre reinforced epoxy/organoclay nanocomposites. Composites: Part B, **2012**, 43, 2762–2771.

Alemdar, A.; Sain, M. Biocomposites from wheat straw nanofibers: Morphology, thermal and mechanical properties. Composites Science and Technology, **2008,** 68, 557–565.

Ali, A.; Sanuddin, A.B.; Ezzeddin, S. The effect of aging on Arenga pinnata fiber-reinforced epoxy composite. Materials and Design, **2010,** 31, 3550–3554.

Al-Sultani, K. F. Mechanical Properties Investigation of HDPE Matrix Composites reinforced with Wheat Straw Fibers. The Iraqi Journal For Mechanical And Material Engineering, **2010**, 10(1), 77-87.

Amor, I.B.; Ghallabi, Z.; Kaddami, H; Raihane, M.; Arous, M.; Kallel, A. Experimental study of relaxation process in unidirectional (epoxy/palm tree fiber) composite. Journal of Molecular Liquids, **2010**, 154, 61–68.

Anbukarasi, K.; Kalaiselvam, S. Study of effect of fibre volume and dimension on mechanical, thermal, and water absorption behaviour of luffa reinforced epoxy composites. Materials and Design, **2015**, 66, 321–330.

Arrakhiz, F.Z.; Malha, M.; Bouhfid, R.; Benmoussa, K.; Qaiss, A. Tensile, flexural and torsional properties of chemically treated alfa, coir and bagasse reinforced polypropylene. Composites: Part B, **2013**, 47, 35–41.

Ashoria, A.; Sheshmanib, S.; Farhanic, F. Preparation and characterization of bagasse/HDPE composites using multi-walled carbon nanotubes. Carbohydrate Polymers, **2013**, 92, 865– 871.

ASTM D3039: Standard test method for tensile properties of polymer matrix composite materials.

ASTM D790: Standard test methods for flexural properties of unreinforced and reinforced plastics and electrical insulating materials.

ASTM D256: Standard test methods for determining the izod pendulum impact resistance of plastics.

ASTM D570: Standard test method for water absorption of plastics.

Ataollahi, S.; Taher, S.T.; Eshkoor, R.A.; Ariffin, A.K.; Azhari, C.H. Energy absorption and failure response of silk/epoxy composite square tubes: Experimental. Composites: Part B, **2012**, 43, 542–548.

Avella, M.; Rota, G.L.; Martuscelli, E.; Raimo, M. Poly(3-hydroxybutyrate-co-3-hydroxyvalerate) and wheat straw fibre composites: thermal, mechanical properties and biodegradation behavior. Journal of Materials Science, **2000**, 35, 829– 836.

Azwa, A.Z.; Yousif, B.F. Characteristics of kenaf fibre/epoxy composites subjected to thermal degradation. Polymer Degradation and Stability, **2013**, 98, 2752-2759.

Babaei, I.; Madanipour, M.; Farsi, M.; Farajpoor, A. Physical and mechanical properties of foamed HDPE/wheat straw flour/nanoclay hybrid composite. Composites: Part B, **2014**, 56, 163–170.

Bachtiar, D.; Sapuan, S.M.; Hamdan, M.M. The effect of alkaline treatment on tensile properties of sugar palm fiber reinforced epoxy composites. Materials and Design, **2008**, 29, 1285–1290.

Basuli, U.; Chaki, T.K.; Chattopadhyay, S.; Sabharwal, S. Thermal and Mechanical Properties of Polymer- Nanocomposites Based on Ethylene Methyl Acrylate and Multiwalled Carbon Nanotube. Polymer composites, **2010,** 1168-1178.

Bellamy, L.J. The Infra-red Spectra of Complex Molecules. John Wiley & Sons Inc., New York, **1954**, 1, 83–98.

Beninia, K.C.C.C.; Voorwald, H.J.C.; Cioffi, M.O.H. Mechanical properties of HIPS/sugarcane bagasse fiber composites after accelerated weathering. Procedia Engineering, **2011** 10, 3246–3251.

Benjamin, M.W.; Coles, S.R.; Maggs, S.; Meredith, J.; Kirwan, K. Use of lignin as a compatibiliser in hemp/epoxy composites. Composites Science and Technology, **2011**, 71, 1804–1810.

Berghezan, A. Non-ferrous Materials. Nucleus, **1966**, 8, 5–11.

Bilba, K.; Arsene, M.A.; Ouensanga, A. Sugar cane bagasse fibre reinforced cement composites. Part I. Influence of the botanical components of bagasse on the setting of bagasse/cement composite. Cement & Concrete Composites, **2003**, 25, 91–96.

Bisanda, E.N.T.; Ansell, M.P. The effect of silane treatment on the mechanical and physical properties of sisal-epoxy composites. Composites Science and Technology, **1991**, 41, 165-178.

Biswas, S.; Satapathy, A. A comparative study on erosion characteristics of red mud filled bamboo–epoxy and glass–epoxy composites. Materials and Design, **2010**, 31, 1752–1767.

Bledzki, A.K.; Mamun, A.A.; Lucka-Gabor, M.; Gutowski, V.S. The effects of acetylation on properties of flax fibre and its polypropylene composites. Express Polymer Letters, **2008**, 2, 413–422.

Bledzki, A.K.; Mamun, A.A.; Jaszkiewicz, A.; Erdmann, K. Polypropylene composites with enzyme modified abaca fiber. Composites Science and Technology, **2010a**, 70, 854–60.

Bledzki, A. K.; Mamun, A.A.; Volk, J. Physical, chemical and surface properties of wheat husk, rye husk and soft wood and their polypropylene composites. Composites: Part A, **2010b**, 41, 480–488.

Bonnafous, C.G.; Vasconcellos, D.; Touchard, F.; Arnault, L.C. Experimental and numerical investigation of the interface between epoxy matrix and hemp yarn. Composites: Part A, **2012**, 43, 2046–2058.

Boricha, A.G.; Murthy Z.V.P. Preparation and performance of N,O–carboxymethyl chitosan-polyether sulfone composite nanofiltration membrane in the separation of nickel ions from aqueous solutions. Journal of Applied Polymer Science, **2008**, 110(6), 3596-3605.

Boopalan, M.; Niranjanaa, M; Umapathy, M.J. Study on the mechanical properties and thermal properties of jute and banana fiber reinforced epoxy hybrid composites. Composites: Part B, **2013**, 51, 54-57.

Brugnago, R. J.; Satyanarayana, K.G.; Wypych, F.; Ramos, L.P. The effect of steam explosion on the production of sugarcane bagasse/polyester composites. Composites: Part A, **2011**, 42, 364–370.

Cao, Y.; Shibata, S.; Fukumoto, I. Mechanical properties of biodegradable composites reinforced with bagasse fibre before and after alkali treatments. Composites: Part A, **2006**, 37, 423–429.

Cerqueira, E.F.; Baptista, C.A.R.P.; Mulinari, D.R.; Mechanical behaviour of polypropylene reinforced sugarcane bagasse fibers composites. Procedia Engineering, **2011,** 10, 2046–2051.

Chattopadhyay, P.K.; Basuli, U.; Chattopadhyay, S. Studies on Novel Dual Filler Based Epoxidized Natural Rubber Nanocomposite. Polymer composites, **2010,** 835-846.

Chen, H.; Zhao, Z.; Huang, X.; Patchigolla, K.; Cotton, A.; Oakey, J. Novel Optimized Process for Utilization of CaO-Based Sorbent for Capturing $CO_2$ and $SO_2$ Sequentially. Energy Fuels, **2012**, 26, 5596−5603.

Colom, X.; Carrillo, F.; Nogue´ s, F.; Garriga, P. Structural analysis of photodegraded wood by means of FTIR spectroscopy. Polymer Degradation and Stability, **2003**, 80(3), 543–549.

Coroller, G.; Lefeuvre, A.; Duigou, A.L.; Bourmaud, A.; Ausias, G.; Gaudry, T.; Baley, C. Effect of flax fibers individualization on tensile failure of flax/epoxy unidirectional composite. Composites: Part A, **2013**, 51, 62–70.

Corradini, E.; Ito, E.N.; Marconcini, J.M.; Rios, C.T.; Agnelli, J.A.M.; Mattoso, L.H.C. Interfacial behavior of composites of recycled poly (ethylene terephthalate) and sugarcane bagasse fiber. Polymer Testing, **2009**, 28, 183–187.

Corrales, R.C.N.R.; Mendes, F.M.T.; Perrone, C.C.; Anna, C.S.; De Souza, W.; Abud, Y.; Da Silva Bon, E.P.; Ferreira-Leitão, V. Structural evaluation of sugar cane bagasse steam pretreated in the presence of $CO_2$ and $SO_2$. Biotechnology for Biofuels, **2012**, 5(36), 1-8.

Dabade, B.M.; Ramachandra Reddy, G.; Rajesham, S; Udaykiran, C. Effect of fiber length and fiber weight ratio on tensile properties of sun hemp and palmyra fiber reinforced polyester composites. Journal of Reinforced Plastics Composites, **2006**, 25(16), 1733-1738.

Dalai, A.K.; Sethuraman, R.; Katikaneni, S.P.R.; Idem, R.O. Synthesis and Characterization of Sulfated Titania Solid Acid Catalysts. Industrial and Engineering Chemistry Research, **1998,** 37, 3869-3878.

Denise, C.O.N.; Ferreira, A.S.; Monteiro, S.N.; Aquino, R.C.M.P.; Kestur, S.G. Studies on the characterization of piassava fibers and their epoxy composites. Composites: Part A, **2012**, 43, 353–362.

Deo, C.R.; Ph.D. Dissertation. Preparation and characterization of Polymer matrix composite using natural fiber lantana-camara. Department of mechanical engineering, National institute of technology, Rourkela **2010.**

Dong, C.; Davies, I.J. Flexural properties of wheat straw reinforced polyester composites. American Journal of Materials Science, **2011,** 1(2), 71-75.

Duigou, A.L.; Kervoelen, A.; Grand, A.L.; Nardin, M.; Baley, C. Interfacial properties of flax fibre–epoxy resin systems: Existence of a complex interphase. Composites Science and Technology, **2014**, 100, 152–157.

Elsunni, M.M.; Collier, J.R. Processing of sugar cane rind into nonwoven fiber. Journal of American Society of Sugar Cane Technologists, **1996**, 16, 94– 110.

Eshkoor, R.A.; Oshkovr, S.A.; Sulong, A.B.; Zulkifli, R.; Ariffin, A.K.; Azhari, C.H. Comparative research on the crashworthiness characteristics of woven natural silk/epoxy composite tubes. Materials and Design, **2013,** 47, 248–257.

Eshkoor, R.A.; Ude, A.U.; Oshkovr, S.A.; Sulong, AB.; Zulkifli, R.; Ariffin, A.K.; Azhari, C.H. Failure mechanism of woven natural silk/epoxy rectangular composite tubes under axial quasi-static crushing test using trigger mechanism. International Journal of Impact Engineering, **2014**, 64, 53-61.

Farsi, M. Some of the Mechanical and Thermal Properties of Wheat Straw Filled-PP Composites. Fibers and Polymers, **2012**, 13(4), 515-521.

Faruk, O.; Bledzki, A.K.; Fink, H.P.; Sain, M. Biocomposites reinforced with natural fibers: 2000–2010. Progress in Polymer Science, **2012**, 37(11)1552–1596.

Filho, R.M.B.; Mendes, L.M.; Novack, K.M.; Aprelini, L.O.; Botaro, V.R. Hybrid chipboard panels based on sugarcane bagasse, urea formaldehyde and melamine formaldehyde resin. Industrial Crops and Products, **2011**, 33, 369–373.

Fiore, V.; Bella, G.D.; Valenza, A. The effect of alkaline treatment on mechanical properties of kenaf fibers and their epoxy composites. Composites: Part B, **2015**, 68, 14–21.

Gabr, M.H.; Phong, N.T.; Okubo, K.; Uzawa, K.; Kimpara, I.; Fujii, T. Thermal and mechanical properties of electrospun nano-celullose reinforced epoxy nanocomposites. Polymer Testing, **2014**, 37, 51–58.

Garg, S.; Gupta, S.K. Multiobjective optimization of a free radical bulk polymerization reactor using genetic algorithm. Macromolecular Theory and Simulations, **1999**, 8(1), 46–53.

Garg, S.; Gupta, S.K.; Saraf, D.N. On-line optimization of free radical bulk polymerization reactors in the presence of equipment failure. Journal of Applied Polymer Science, **1999**, 71, 2101-2120.

Gassan, J.; Bledzki, A.K. The Influence of Fiber-surface Treatment on the Mechanical Properties of Jute-polypropylene Composites. Composites Part A, **1997**, 28, 1001-1005.

Gu, Y.; Tan, X.; Yang, Z.; Li, M.; Zhang, Z. Hot compaction and mechanical properties of ramie fabric/epoxy composite fabricated using vacuum assisted resin infusion molding. Materials and Design, **2014**, 56, 852–861.

Guen, M.J.L.; Newman, R.H.; Fernyhough, A.; Staiger, M.P. Tailoring the vibration damping behaviour of flax fibre-reinforced epoxy composite laminates via polyol additions. Composites: Part A, **2014**, 67, 37–43.

Gupta, M.K.; Srivastava, R.K. Tensile and flexural properties of sisal fiber reinforced epoxy composite: a comparison between unidirectional and mat form of fibers. Procedia Material Science, **2014**, 5, 2434 – 2439.

Halvarssona, S.R.; Edlunda, H.K.; Norgrena, M. Properties of medium-density fibreboard (MDF) based on wheat straw and melamine modified urea formaldehyde (UMF) resin. Industrial Crops and Products, **2008**, 28, 37–46.

Hattallia, S.; Benaboura, A.; Ham-Pichavant, F.; Nourmamode, A.; Castel-lan, A. Adding value to alfa grass (Stipa tenacissima L.) soda lignin as phenolic resins. 1. Lignin characterization. Polymer Degradation and Stability, **2002**, 76, 259–64.

Hoareau, W.; Trindade, W.G.; Siegmund, B.; Castellan, A.; Frollini, E. Sugar cane bagasse and curaua lignins oxidized by chlorine dioxide and reacted with furfuryl alcohol: characterization and stability. Polymer Degradation and Stability, **2004**, 86, 567–576.

Hornsby, P.R.; Hinrichsen, E.; Tarverdi, K. Preparation and properties of polypropylene composites reinforced with wheat and flax straw fibres, Part I fibre characterization. Journal of Materials Science, **1997**, 32, 443–449.

Hoyos, C.G.; Vazquez, A. Flexural properties loss of unidirectional epoxy/fique composites immersed in water and alkaline medium for construction application. Composites: Part B, **2012**, 43, 3120–3130.

Huang, Z.; Liang, X.; Hu, H.; Gao, L.; Chen, Y.; Tong, Z. Influence of mechanical activation on the graft copolymerization of sugarcane bagasse and acrylic acid. Polymer Degradation and Stability, **2009**, 94, 1737–1745.

Huang, Z.; Wang, N.; Zhang, Y.; Hu, H.; Luo, Y. Effect of mechanical activation pretreatment on the properties of sugarcane bagasse/poly(vinyl chloride) composites. Composites: Part A, **2012**, 43, 114–120.

Hui, S.; Chaki, T.K,; Chattopadhyay, S. Dynamic and capillary rheology of LDPE-EVA–based thermoplastic elastomer: effect of silica nanofiller. Polymer Composites, **2010**, 378-391.

Jartiz, A.E.; Design, **1965**, 18.

Kandare, E.; Luangtriratana, P.; Kandola, B.K. Fire reaction properties of flax/epoxy laminates and their balsa-core sandwich composites with or without fire protection. Composites: Part B, **2014**, 56, 602–610.

Kaushik, A.; Singh, P.; Kaushik, J. The mechanical properties and chemical resistance of short glass-fiber-reinforced epoxy composites. International Journal of Polymeric Materials, **2006**, 55, 425–440.

Kaushik, A.;, Singh, M.; Verma, G. Green nanocomposites based on thermoplastic starch and steam exploded cellulose nanofibrils from wheat straw. Carbohydrate Polymers, **2010a**, 82, 337–345.

Kaushik, A.; Singh, P.; Kaushik, J. Mechanical and electrical conductivity study on epoxy/graphite composites. Journal of Reinforced Plastics and Composites, **2010b**, 29, 1038-1044.

Kelly, A.; The nature of composite materials, Scientific American Magazine, **1967**, 217(B), 161.

Kuang, X.; Kuang, R.; Zheng, X.; Wang, Z. Mechanical properties and size stability of wheat straw and recycled LDPE composites coupled by waterborne coupling agents. Carbohydrate Polymers, **2010**, 80, 927–933.

Kumar, R.; Kumar, K.; Bhowmik, S.; Optimization of mechanical properties of epoxy based wood dust reinforced green composite using taguchi method. Procedia Materials Science, **2014a,** 5, 688-696.

Kumar, R.; Kumar, K.; Sahoo, P.; Bhowmik, S. Study of mechanical properties of wood dust reinforced epoxy composite. Procedia Materials Science, **2014b**, 6, 551 – 556.

Kumar, S.M.S.; Duraibabu, D.; Subramanian, K. Studies on mechanical, thermal and dynamic mechanical properties of untreated (raw) and treated coconut sheath fiber reinforced epoxy composites. Materials and Design, **2014c,** 59, 63–69.

Kumar, V.; Sinha, S.; Singh, M.S.; Kanungo, B.K.; Biswas, P. Rice husk as reinforcing filler in polypropylene composites. Reviews in Chemical Engineering, **2010**, 26, 41–53.

Kumar, V.; Tyagi, L.; Sinha, S. Wood flour – reinforced plastic composites: a review. Reviews in Chemical Engineering, **2011**, 27, 253–264.

Landro, L.D.; Janszen, G. Composites with hemp reinforcement and bio-based epoxy matrix. Composites: Part B, **2014**, 67, 220–226.

Lee, S.C.; Mariatti, M. The effect of bagasse fibers obtained (from rind and pith component) on the properties of unsaturated polyester composites. Materials Letters, **2008**, 62, 2253–2256.

Lee, S.; Schneider, K.; Schumann, J.; Mogalicherla, A.K.; Pfeifer, P.; Dittmeyer, R. Effect of metal precursor on Cu/ZnO/Al2O3 synthesized by flame spray pyrolysis for direct DME production. Chemical Engineering Science, **2015**, 138, 194–202.

Leman, Z.; Sapuan, S.M.; Saifol, A.M.; Maleque, M.A.; Ahmad, M.M.H.M. Moisture absorption behavior of sugar palm fiber reinforced epoxy composites. Materials and Design, **2008**, 29, 1666–1670.

Li, X.; Panigrahi, S.A.; Tabil, L.G.; Crerar, W.J. Flax fiber-reinforced composites and the effect of chemical treatments on their properties. The Society for Engineering in Agricultural, Food and Biological Systems**. 2004a**, 1–11.

Li, X.; Tabil, L.G.; Panigrahi, S. Chemical treatment of natural fibre for use in natural fibre-reinforced composites: a review. Journal of Polymers and the Environment, **2007**, 15(1), 25–33.

Li, X.H.; Meng, Y.Z.; Wang, S.J.; Rajulu, A.V.; Tjong, S.C. Completely biodegradable composites of poly(propylene carbonate) and short, lignocellulose fabric hildegardiapopulifolia. Journal of Polymer Science: Part B, **2004b**, 42, 666–675.

Liang, S.; Gning, P.B.; Guillaumat, L. Properties evolution of flax/epoxy composites under fatigue loading. International Journal of Fatigue, **2014** 63, 36–45.

Liany, Y.; Tabei, A.; Farsi, M.; Madanipour, M. Effect of Nanoclay and Magnesium Hydroxide on Some Properties of HDPE/Wheat Straw Composites. Fibers and Polymers. **2013**, 14(2) 304-310.

Liu, K.; Zhang, X.; Takagi, H.; Yang, Z.; Wang, D. Effect of chemical treatments on transverse thermal conductivity of unidirectional abaca fiber/epoxy composite. Composites: Part A, **2014**, 66, 227–236.

Liu, Q.; Hughes, M. The fracture behavior and toughness of woven flax fiber reinforced epoxy composites. Composites: Part A**, 2008**, 39, 1644–1652.

Low, I.M.; McGrath, M.; Lawrence, D.; Schmidt, P.; Lane, J.; Latella, B.A.; Simon, K.S. Mechanical and fracture properties of cellulose-fibre-reinforce epoxy laminates. Composites: Part A, **2007**, 38, 963–974.

Lu, T.; Jiang, M.; Jiang, Z.; Hui, D.; Wang, Z.; Zhou, Z. Effect of surface modification of bamboo cellulose fibers on mechanical properties of cellulose/epoxy composites. Composites: Part B, **2013,** 51, 28–34.

Luz, S.M.; Goncalves, A.R.; Jr, A.P.D.A. Mechanical behavior and microstructural analysis of sugarcane bagasse fibers reinforced polypropylene composites. Composites: Part A, **2007**, 38, 1455–1461.

Luz, S.M.; Tio, J.D.; Rocha, G.J.M.; Goncalves, A.R.; Jr, A.P.D.A. Cellulose and cellulignin from sugarcane bagasse reinforced polypropylene composites: Effect of acetylation on mechanical and thermal properties. Composites: Part A**, 2008**, 39, 1362–1369.

Mahjoub, R.; Yatim, J.M.; Sam, A.R.M.; Raftari, M. Characteristics of continuous unidirectional kenaf fiber reinforced epoxy Composites. Materials and Design, **2014**, 64, 640–649.

Majumdar, S.; Mitra, K.; Raha, S. Optimized species growth in epoxy polymerization with real-coded NSGA-II. Polymer, **2005**, 46, 11858–11869.

Marti-Ferrer, F.; Vilaplana, F.; Ribes-Greus, A.; Benedito-Borras, A.; Sanz-Box, C. Flour rice husk as filler in block copolymer polypropylene: effect of different coupling agents. Journal of Applied Polymer Science, **2006**, 99, 1823–1831.

Mishra, P.; Acharya, S.K. Anisotropy abrasive wear behavior of bagasse fiber reinforced polymer composite. International Journal of Engineering Science and Technology, **2010a**, 2(11), 104-112.

Mishra, P.; Acharya, S.K. Solid particle erosion of Bagasse fiber reinforced epoxy composite. International Journal of Physical Sciences, **2010b,** 5(2) 109-115.

Mishra, V.; Biswas, S. Physical and Mechanical Properties of Bi-directional Jute Fiber epoxy. Procedia Engineering, **2013**, 51, 561–566.

Mitra, K.; Majumdar, S.; Raha S. Multiobjective dynamic optimization of a semi-batch epoxy polymerization process. Computers and Chemical Engineering, **2004**, 28, 2583–2594.

Mitra, K. Assessing optimal growth of desired species in epoxy polymerization under uncertainty. Chemical Engineering Journal, **2010**, 162(1), 322-330.

Mittal, V.; Sinha, S. Effect of chemical treatment on thermal properties of bagasse fiber-reinforced epoxy composite, Science and Engineering of Composite Materials, DOI: 10.1515/secm-2014-0434 (**2014**).

Mittal, V.; Sinha, S. Effect of chemical treatment on the mechanical and water absorption properties of bagasse fiber-reinforced epoxy composite, Journal of Polymer Engineering, **2015a**, 35(6), 545-550.

Mittal, V.; Sinha, S. Study the effect of fiber loading and alkali treatment on mechanical and water absorption properties of wheat straw fiber reinforced epoxy composites, Science and Engineering of Composite Materials, DOI: 10.1515/secm-2015-0441 (**2015b**).

Mittal, V.; Sinha, S. Effect of alkali treatment on the thermal properties of wheat straw fiber reinforced epoxy composites, Journal of Composite Materials, DOI 10.1177/0021998316646168 (**2016**).

Mogalicherla, A.K.; Elbashir, N.O. Development of a Kinetic Model for Supercritical Fluids Fischer-Tropsch Synthesis. Energy Fuels, **2011**, 25, 878–889.

Mogalicherla, A.K.; Lee, S.; Pfeifer, P.; Dittmeyer, R.; Drop-on-demand inkjet printing of alumina nanoparticles in rectangular microchannels. Microfluid Nanofluid, **2014**, 16, 655–666.

Mohan, T.P.; Kanny, K. Water barrier properties of nanoclay filled sisal fibre reinforced epoxy composites. Composites: Part A, **2011**, 42, 385–393.

Mohanty, A.K.; Misra, M.; Drzal, L.T. Surface modification of natural fibers and performance of the resulting biocomposites: An overview. Composites Interfaces, **2001a**, 8, 313-343.

Mohanty, A.K.; Mishra, S.; Drzal, L.T.; Misra, M.; Parija, S.; Nayak, S.K.; Tripathy, S.S.; Studies on the mechanical performance of biofiber/glass reinforced polyester hybrid composites. Composites Science and Technology, **2003**, 63, 1377–1385.

Mohanty, A.K.; Nayak, S.K.; Tripathy, S.S.; Misra, M.; Rout, J. The influence of fiber treatment on the performance of coir–polyester composites. Composites Science Technology, **2001b**, 61 1303–1310.

Montano-Leyva, B.; Da Silva, G.G.D.; Gastaldia, E.; Torres-Chávezc, P.; Gontarda, N.; Angellier-Coussya, H. Biocomposites from wheat proteins and fibers: Structure/mechanical properties relationships. Industrial Crops and Products, **2013**, 43, 545–555.

Monteiro, S.N..; Candido, V.S.; Braga, F.O.; Bolzan, L.T.; Weber, R.P.; Drelich, J.W.; Sugarcane bagasse waste in composites for multilayered armor. European Polymer Journal, **2016**, 78, 173-185.

Mulinari, D.R.; Voorwald, H.J.C.; Cioffi, M.O.H.; Da Silva, M.L.C.P.; Da Cruz, T.G.; Saron, C. Sugarcane bagasse cellulose/HDPE composites obtained by extrusion. Composites Science and Technology**,** **2009**, 69, 214–219.

Mulinari, D.R.; Voorwald, H.J.C.; Cioffi, M.O.H.; Rocha, G.J.; Da Silva, M.L.C.P. Surface modification of sugarcane bagasse cellulose and its effect on the mechanical and water absorption properties of sugarcane bagasse cellulose/HDPE composites. Bioresources, **2010**, 5(2), 661-671.

Muralidhar, B.A. Tensile and compressive behaviour of multilayer flax-rib knitted preform reinforced epoxy composites. Materials and Design**, 2013**, 49, 400–405.

Mylsamy, K.; Rajendran, I. The mechanical properties, deformation and thermomechanical properties of alkali treated and untreated Agave continuous fiber reinforced epoxy composites. Materials and Design, **2011a**, 32, 3076–3084.

Mylsamy, K.; Rajendran, I. Influence of alkali treatment and fibre length on mechanical properties of short Agave fibre reinforced epoxy composites. Materials and Design, **2011b**, 32, 4629–4640.

Nanda, S.; Mohanty, P.; Pant, K.K.; Naik, S.; Kozinski, J.A.; Dalai, A.K. Characterization of north american lignocellulosic biomass and biochars in terms of their candidacy for alternate: renewable fuels. Bioenergy Research, **2013a**, 6, 663–677.

Nanda, S.; Dalai, A.K.; Kozinski, J.A. Forestry biomass in a bioenergy perspective. Journal of Science & Technology for Forest Products and Processes, **2013b**, 3(6), 15-26.

Newman, R.H.; Auto-accelerative water damage in an epoxy composite reinforced with plain-weave flax fabric. Composites: Part A, **2009**, 40, 1615–1620.

Newman, R.H.; Clauss, E.C.; Carpenter, J.E.P.; Thumm, A. Epoxy composites reinforced with deacetylated Phormium tenax leaf fibres. Composites: Part A, **2007,** 38, 2164–2170.

Nirmal, U.; Hashim, J.; Low, K.O. Adhesive wear and frictional performance of bamboo fibres reinforced epoxy composite. Tribology International, **2012**, 47, 122–133.

Obele, C.M.; Edith, I.Y.; Raphael, O. Effects of maleic anhydride on the mechanical properties and morphology of wheat straw fibre reinforced polypropylene. Journal of Innovative Research in Engineering and Sciences, **2012**, 3(1), 412-420.

Oshkovr, S.A.; Eshkoor, R.A.; Taher, S.T.; Ariffin, A.K.; Azhari, C.H. Crashworthiness characteristics investigation of silk/epoxy composite square tubes. Composite Structures, **2012,** 94, 2337–2342.

Ouajai, S.; Shanks, R.A. Composition, structure and thermal degradation of hemp cellulose after chemical treatments. Polymer Degradation and Stability, **2005**, 89(2), 327–335.

Pan, M.Z.; Zhou, D.G.; Bousmina, M.; Zhang, S.Y. Effects of wheat straw fiber content and characteristics, and coupling agent concentration on the mechanical properties of wheat straw fiber-polypropylene composites. Journal of Applied Polymer Science, **2009**, 113, 1000–1007.

Pant, K.K.; Kunzru, D. Catalytic Pyrolysis of *n*-Heptane: Kinetics and Modeling. Industrial and Engineering Chemistry Research, **1997**, 36, 2059-2065.

Pant, K.K.; Kunzru, D. Catalytic pyrolysis of methylcyclohexane: kinetics and modeling. Chemical Engineering Journal, **1998**, 70(1), 47-54.

Pant K.K.; Mohanty, P. Biomass, Conversion Routes and Products – An Overview. Transformation of Biomass: Theory to Practice, First Edition. Edited by Andreas Hornung.© 2014 John Wiley & Sons, Ltd. Published **2014**.

Panthapulakkal, S.; Sain, M. Injection molded wheat straw and corn stem filled polypropylene composites. Journal of Polymers and the Environment, **2006**, 14, 265–272.

Panthapulakkal, S.; Zereshkian, A.; Sain, M. Preparation and characterization of wheat straw fibers for reinforcing application in injection molded thermoplastic composites. Bioresource Technology, **2006**, 97, 265–272.

Patchigolla, K.; Oakey, J.E. Design overview of high pressure dense phase $CO_2$ pipeline transport in flow mode. Energy Procedia, **2013**, 37, 3123 – 3130.

Paul, A.; Joseph, K.; Thomas, S. Effect of surface treatments on the electrical properties of low-density polyethylene composites reinforced with short sisal fibers. Composites Science and Technology, **1997**, 57, 67-79.

Pfister, D.P.; Larock, R.C. Green composites from a conjugated linseed oil-based resin and wheat straw. Composites: Part A, **2010** 41, 1279–1288.

Pinto, C.; Da Silva, E.; Arizaga, G.G.C.; Wypych, F.; Ramos, L.P.; Satyanarayana, K.G. Studies of the effect of molding pressure and incorporation of sugarcane bagasse fibers on the structure and properties of poly (hydroxy butyrate). Composites: Part A, **2009**, 40, 573–582.

Raj, R.G.; Kokta, B.V. The effect of processing conditions and binding material on the mechanical properties of bagasse fiber composites. Polymer Journal, **1991**, 27(10), 1121-1123.

Rajesh, S.; Parikh, P.A.; Murthy, Z.V.P. In situ synthesis of inorganic filler filled polyethylene using polyethersulfone supported TiCl4 catalyst system. Journal of Applied Polymer Science, **2011**, 119(5), 2611-2619.

Ray, D.; Sarkar, B.K.; Ranac, A.K.; Rose, N. The mechanical properties of vinylester resin matrix composites reinforced with alkali-treatment jute fibers. Composite: Part A., **2001**, 32, 119-127.

Reddy, C.R.; Sardashti, A.P.; Simon, L.C. Preparation and characterization of polypropylene–wheat straw–clay composites. Composites Science and Technology, **2010**, 70, 1674–1680.

Rodrigues, E.F.; Maia T.F.; Mulinari, D.R. Tensile strength of polyester resin reinforced sugarcane bagasse fibers modified by estherification. Procedia Engineering, **2011**, 10, 2348–2352.

Romli, F.I.; Alias, A.N.; Rafie, A.SM.; Majid, D.L.A.A. Factorial study on the tensile strength of a coir fiber- reinforced epoxy composite. AASRI Procedia, **2012**, 3, 242 – 247.

Rosa. I.M.D.; Santulli, C.; Sarasini, F. Mechanical and thermal characterization of epoxy composites reinforced with random and quasi-unidirectional untreated Phormium tenax leaf fibers. Materials and Design, **2010**, 31, 2397–2405.

Rout, J.; Misra, M.; Tripathy, S.S.; Nayak, S.K.; Mohanty, A.K. The influence of fiber treatment on the performance of coir-polyester composites. Composites Science and Technology, **2001**, 61, 1303-1310.

Samariha, A.; Bastani, A.; Nemati, M.; Kiaei, M.; Nosrati, H.; Farsi, M. Investigation of the mechanical properties of bagasse flour/polypropylene composites. Mechanics of Composite Materials, **2013**, 49(4), 447-454.

Sapuan, S.M.; Leenie, A.; Harimi, M.; Beng, Y.K. Mechanical properties of woven banana fibre reinforced epoxy composite. Materials and Design, **2006**, 27, 689–693.

Satyanarayana, K.G.; Kulkarni, A.G.; Rohatgi, P.K. Potential of natural fibres as a resource for industrial material in Kerala. Journal of Scientific and Industrial Research, **1981**, 40, 222–237.

Saw, S.K.; Datta, C. Thermomechanical properties of jute/bagasse hybrid fiber reinforced epoxy thermoset composites. Bioresources, **2009**, 4(4), 1455-1476.

Schuh, T. G. (2004). Renewable materials for automotive applications. www.ienica.net/fibresseminar/schuh.pdf.

Schwanninger, M.; Rodrigues, J.C.; Pereira, H.; Hinterstoisser, B. Effects of short time vibratory ball milling on the shape of FT-IR spectra of wood and cellulose. Vibrational Spectroscopy, **2004**, 36(1), 23–40.

Scida, D.; Assarar, M.; Poilâne, C.; Ayad, R. Influence of hygrothermal ageing on the damage mechanisms of flax-fiber reinforced epoxy composite. Composites: Part B, **2013**, 48, 51–58.

Sheshmani, S. Effects of extractives on some properties of bagasse/high density polypropylene composite. Carbohydrate Polymers, **2013**, 94, 416– 419.

Shibata, M.; Ozawa, K.; Teramoto, N.; Yosomiya, R.; Takeishi, H. Biocomposites made from short abaca fiber and biodegradable polyester. Macromolecular Materials and Engineering, **2003**, 288, 35–43.

Shibata, M.; Takachiyo, K.; Ozawa, K.; Yosomiya, R.; Takeishi, H. Biodegradable polyester composites reinforced with short abaca fiber. Journal of Applied Polymer Science, **2002**, 85, 129–138.

Shih, Y.F. Mechanical and thermal properties of waste water bamboo husk fiber reinforced epoxy composites. Materials Science and Engineering A, **2007**, 445, 289–295.

Sinha, S.; Agarwal, A.K.; Garg, S. Biodiesel development from rice bran oil: Transesterification process optimization and fuel characterization. Energy Conversion and Management, **2008**, 49, 1248–1257.

Sousa, M.V.D.; Monteiro, S.N.; D'Almeida, J.R.M. Evaluation of pre-treatment, size and molding pressure on flexural mechanical behavior of chopped bagasse–polyester composites. Polymer Testing, **2004,** 23, 253–258.

Sreekumar, P.A. Matrices for natural-fibre reinforced composites. In K. L. Pickering (Ed.), Properties and performance of natural-fibre composite. UK: Brimingham, Woodhead Publication Limited, **2008,** 541.

Srisuwana, S.; Prasoetsophaa, N.; Suppakarna, N.; Chumsamronga, P. The effects of alkalized and silanized woven sisal fibers on mechanical properties of natural rubber modified epoxy resin. Energy Procedia, **2014**, 56, 19–25.

Srivastava, K.; Srivastava, D.; Tripathi, S.K. Studies on the effect of curing agent concentration and type of phenol on various physico-chemical properties of resole and epoxy blends. Journal of Applied Polymer Science, **2008**, 110, 3812–3819.

Stewart, D.; Wilson, H.M.; Hendra, P.J.; Morrison, I.M. Fourier-transform infrared and Raman-spectroscopic study of biochemical and chemical treatments of oak wood (Quercus rubra) and Barley (Hordeum vulgare) Straw. Journal of Agriculture and Food Chemistry, **1995**, 43, 2219–2225.

Subagia, I.D.G.A.; Tijing, L.D.; Kim, Y.; Kim, C.S.; Vista IV, F.P.; Shon, H.K. Mechanical performance of multiscale basalt fiber–epoxy laminates containing tourmaline micro/nano particles. Composites: Part B, **2014,** 58, 611–617.

Suchtelen, V. Product properties: a new application of composite materials, Philips Res**.** Reports**, 1972,** 27**,** 28.

Suddell, B.C.; Evans, W.J. Natural Fiber Composites in Automotive Applications, **2005.**

Talavera, F.J.F.; Guzman, J.A.S.; Richter, H.G.; Duenas, R.S.; Quirarte, J.R. Effect of production variables on bending properties, water absorption and thickness swelling of bagasse/plastic composite boards. Industrial Crops and Products, **2007**, 26, 1–7.

Teel GRT. Natural fiber products and composites [http://www.teelgrt. com; accessed in September, **2015**].

Teng, H.; Wei, Y. Thermo-gravimetric studies on the kinetics of rice hull pyrolysis and the influence of water treatment. Industrial & Engineering Chemistry Research, **1998**, 37, 3806–3811.

Teng, S.; Siegel, G.; Prestgard, M.C.; Wang, W.; Tiwari, A. Synthesis and characterization of copper-infiltrated carbonized wood monoliths for supercapacitor electrodes. Electrochimica Acta, **2015**, 161, 343–350.

Tian, K.; Prestgard, M.; Tiwari, A. A review of recent advances in nonenzymatic glucose sensors. Materials Science and Engineering C, **2014**, 41, 100–118.

Tian, K.; Alex, S.; Siegel, G.; Tiwari, A. Enzymatic glucose sensor based on Au nanoparticle and plant-like ZnO film modified electrode. Materials Science and Engineering C, **2015**, 46, 548–552.

Trana, T.P.T.; Bénézeta, J.C.; Bergeret, A. Rice and einkorn wheat husks reinforced poly(lactic acid) (PLA) biocomposites: Effects of alkaline and silane surface treatments of husks. Industrial Crops and Products, **2014**, 58, 111–124.

Tripathi, G.; Srivastava, D. Toughened cycloaliphatic epoxy resin for demanding thermal applications and surface coatings. Journal of Applied Polymer Science, **2009**, 114, 2769–2776.

Upreti, S.R.; Sundaram, B.S.; Lohi, A. Optimal control determination of MMA polymerization in non-isothermal batch reactor using bifunctional initiator. European Polymer Journal, **2005**, 41(12), 2893–2908.

Van, D.O.M.; Peijs, T. Continous-glass –fiber-reinforced polypropylene composite II. Influence of maleic-anhydride modified polypropylene on fatigue behavior. Composites: Part A, **1998**, 29(3), 227-239.

Vasconcellos, D.S.D.; Sarasini, F.; Touchard, F.; Arnault, L.C.; Pucci, M.; Santulli, C.; Tirillò, J.; Iannace, S.; Sorrentino, L. Influence of low velocity impact on fatigue behaviour of woven hemp fibre reinforced epoxy composites. Composites: Part B, **2014**, 66, 46–57.

Venkateshwaran, N.; Perumal, A.E.; Arunsundaranayagam, D. Fiber surface treatment and its effect on mechanical and visco-elastic behavior of banana/epoxy composite. Materials and Design, **2013,** 47, 151–159.

Vijaykumar, S.; Nilavarasan, T.; Usharani, R.; Karunamoorthy, L. Mechanical and microstructure characterization of coconut spathe fibers and kenaf bast fibers reinforced epoxy polymer matrix composites. Procedia Material Science, **2014**, 5, 2330-2337.

Vilay, V.; Mariatti, M.; Taib, R.M.; Todo, M. Effect of fiber surface treatment and fiber loading on the properties of bagasse fiber–reinforced unsaturated polyester composites. Composites Science and Technology, **2008**, 68, 631–638.

Vyas, A.H.; Jauhari, S.; Murthy, Z.V.P. Chemically Modified Rice Husk Adsorbents, Characterization and Removal of Palladium (II) From Aqueous Solutions. Journal of Dispersion Science and Technology, **2013**, 34(3), 369-380.

White, N.M.; Ansell, M.P.; Straw reinforced polyester composites. Journal of Materials Science, **1993**, 18, 1549–1556.

Wu, J.; Mozaffari, F.E.; Upreti, S. Effect of ozone pretreatment on hydrogen production from barley straw. Bioresource Technology, **2013a**, 144, 344–349.

Wu, J.; Upreti, S.; Mozaffari, F.E. Ozone pretreatment of wheat straw for enhanced biohydrogen production. International Journal of Hydrogen Energy, **2013b**, 38, 10270-10276.

Xiaoqun, M.; Cheng, E.; Wang, D.; Sun, X.S. Physical properties of medium-density wheat straw particleboard using different adhesives. Industrial Crops and Products, **2003,** 18, 47-53.

Xu, Y.; Wu, Q.; Lei, Y.; Yao, F. Creep behavior of bagasse fiber reinforced polymer composites. Bioresource Technology, **2010**, 101, 3280–3286.

Yadav, R.; Srivastava, D. Compatibility, thermal, mechanical and morphological properties of cardanol based epoxidized resin modified with liquid rubber. International Journal of Plastics Technology, **2014**, 18(1), 27–48.

Yan, L.; Chouw, N. Crashworthiness characteristics of flax fibre reinforced epoxy tubes for energy absorption application. Materials and Design, **2013,** 51, 629–640.

Yan, L.; Chouw, N.; Jayaraman, K. Lateral crushing of empty and polyurethane-foam filled natural flax fabric reinforced epoxy composite tubes. Composites: Part B, **2014a**, 63, 15–26.

Yan, L.; Chouw, N.; Jayaraman, K. Effect of triggering and polyurethane foam-filler on axial crushing of natural flax/epoxy composite tubes. Materials and Design, **2014b**, 56, 528–541.

Yongpinga, Y.; Rongronga, Z.; Liqianga, D.; Kavosh, M.; Patchigolla, K.; Oakey, J. Integration and evaluation of a power plant with a CaO-based $CO_2$ capture system. International Journal of Greenhouse Gas Control, **2010**, 4, 603-612.

Yousif, B.F.; Nirmal, U.; Wong, K.J. Three-body abrasion on wear and frictional performance of treated betelnut fiber reinforced epoxy (T-BFRE) composite. Materials and Design, **2010,** 31, 4514–4521.

Yousif, B.F.; Shalwan, A.; Chin, C.W.; Ming, K.C. Flexural properties of treated and untreated kenaf/epoxy composites. Materials and Design, **2012**, 40, 378–385.

Zhang, Y.; Gu, J.; Tan, H.; Di, M.; Zhu L.; Weng, X. Straw based particleboard bonded with composite adhesives. Bioresources, **2011**, 6(1), 464-476.

Zheng, Y.T.; Cao, D.R.; Wang, D.S.; Chen J.J.; Study on the interface modification of bagasse fibre and the mechanical properties of its composite with PVC. Composites: Part A, **2007**, 38, 20–25.

Zou, Y.; Huda, S.; Yang, Y. Lightweight composites from long wheat straw and polypropylene web. Bioresource Technology, **2010**, 101, 2026–2033.

# PUBLICATIONS

## INTERNATIONAL JOURNAL

1. **Mittal, V**.; Sinha, S. Effect of chemical treatment on thermal properties of bagasse fiber-reinforced epoxy composite, *Science and Engineering of Composite Materials*, DOI: 10.1515/secm-2014-0434 (**2014**).
2. **Mittal, V**.; Sinha, S. Effect of chemical treatment on the mechanical and water absorption properties of bagasse fiber-reinforced epoxy composites, *Journal of Polymer Engineering*, **2015**, 35(6), 545-550.
3. **Mittal, V**.; Sinha, S. Study the effect of fiber loading and alkali treatment on mechanical and water absorption properties of wheat straw fiber reinforced epoxy composites, *Science and Engineering of Composite Materials*, DOI: 10.1515/secm-2015-0441 (**2015**).
4. **Mittal, V**.; Sinha, S. Effect of alkali treatment on the thermal properties of wheat straw fiber reinforced epoxy composites, *Journal of Composite Materials*, DOI 10.1177/0021998316646168 **(2016)**.
5. **Mittal, V**.; Saini, R.; Sinha, S. A review on natural fiber-mediated epoxy composites, *Composite Part-B Engineering*, **2016,** 99, 425-435.
6. **Mittal, V**.; Sinha, S. Mechanical, thermal and water absorption properties of wheat straw/bagasse reinforced hybrid polymer composites, *Advances in Polymer Technology*, ADV-02-16-052 under review **(2016)**.

## CONFERENCES

1. **Mittal, V**.; Sinha, S. A review on effect of various chemical treatments of bagasse fiber on properties of bagasse fiber reinforced polymer composite, Paper Presented at the "***Twenty-ninth National Convention of Chemical Engineers and National Seminar on New Composites : The Materials for Chemical and Allied Industries***", held at Udaipur during Oct 4-5, 2013.
2. **Mittal, V**.; Nirupama; Sinha, S. Wheat straw and its composites: A review, Poster presented at ***"CHEMCON-2013"*** organized by 66th Annual session of I.I.Ch.E. held at ICT Mumbai during Dec 27-30, 2013.

3. **<u>Mittal, V</u>**.; Sinha, S. Effect of alkaline treatments of wheat straw fiber on hardness of wheat straw fiber reinforced polymer composite, Poster presented at ***"Third International Conference on Recycling and Reuse of Materials (ICRM 2014)"*** held at Kottayam, Kerela during Apr 11-13, 2014.