# PHYSICO-MECHANICAL PROPERTIES OF
# TYPHA ANGUSTATA (ELEPHANT GRASS) FIBER REINFORCED
# THERMOPLASTIC COMPOSITES

**BY**

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**A**

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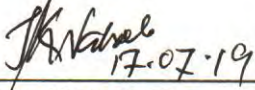
**BUET, DHAKA-1000**

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# DEPARTMENT OF CHEMISTRY

# BANGLADESH UNIVERSITY OF ENGINEERING AND TECHNOLOGY

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## THESIS ACCEPTANCE LETTER

The thesis titled **"Physico-mechanical properties of typha angustata (elephant grass) fiber reinforced thermoplastic composites"** Submitted by Walid Bin Kader. Roll No: 0416032606F, Registration No: 0416032606, Session: April-2016 has been accepted as satisfactory in partial fulfilment of the requirement for the degree of Master of Science (M.Sc) in Organic Chemistry on July 17, 2019.

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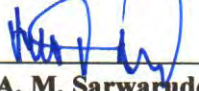   BUET, Dhaka-1000, Bangladesh.

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# Dedicated To

*My Beloved Parents*

*&*

*Honorable  Supervisor*

**STUDENT'S DECLARATION**

I hereby certify that this material, which I now submit for assessment on the programme of study leading to the award of M.Sc is entirely my own work, and I also declare this thesis or any part of it has not been submitted elsewhere for the award of any degree or diploma.

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# Acknowledgement

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# List of Abbreviation

| Elaboration | Abbreviation |
| --- | --- |
| Betel Nut Husk Fiber | BNHF |
| Low Density Polyethylene | LDPE |
| Polystyrene | PS |
| Attenuated Total Reflection | ATR |
| Fourier Transform Infrared | FTIR |
| Scanning Electron Microscope | SEM |
| Thermo Gravimetric Analysis | TGA |
| Differential Scanning Calorimetric Analysis | DSC |

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# ABSTRACT

Natural fibers have been found to be excellent reinforcing materials for preparing polymer matrix-based composites. In the present study both raw and surface modified polystyrene matrix-based composites were prepared in order to explore the effect of reinforcement on the mechanical properties of the matrix. Due to increased environmental concerns natural fibers have recently attracted a great attention as reinforcement for the synthesis of fiber reinforced polymer matrix-based composites and these composites have an added advantage of being cheap, easily available, nonabrasive, easy to process, high specific strength and biodegradable in nature. Among various polymer matrices, thermoplastics are mostly used in manufacture of plastic/ wood composites. Polystyrene is one of the most important thermoplastics which is in great demand due to its transparency, fluidity and good electrical insulation properties. In the past decade, natural fiber-based composites have been utilized as door panels, seat backs, head liners, dash boards, and interior parts of car. The usage of composites is not only limited to automobile parts but it also extends to current generation military aircraft. These advance materials have the potential to contribute to aircraft development, since one-third of the structural mass of aircraft is made from fiber reinforced polymer composites. Among many natural fibers elephant grass fiber has a great prospective in the composite field, because it is cheap, available in large quantities. Its botanical name is Typha Angustata. Elephant grass (Typha Angustata) fiber is used as new natural fiber reinforcement and polystyrene as matrix. Typha Angustata fibers (TAF) were extracted and used to prepare sodium hydroxide treated and untreated TAF reinforced polystyrene composites. Composites were prepared with randomly oriented fibers with different proportions of fibers and matrix ratio. Different types of compression molded TAF-polystyrene composite were prepared using different weight percent of treated and untreated TAF with compositions 5, 15, 20, 25 and 30 wt. % in polystyrene. The composites were characterized by FT-IR and SEM analysis and their physico-mechanical and thermal properties were analyzed by tensile test, TGA and DSC analysis. Mechanical properties tensile strength, elongation at break, impact strength was measured. Improved mechanical properties were obtained for all the treated fiber reinforced composites over untreated TAF reinforced plastics composites. Scanning Electron Microscopy (SEM) were taken for all composites to investigate the interfacial adhesion and bonding between fiber and matrices. Morphological analyses showed the evidence of stronger interfacial adhesion between the fibers and matrices in the all treated TAF - polymer composites than the untreated TAF polymer composites. Thermal properties of prepared composites were examined by TGA and DSC analysis. Almost same thermal properties were obtained for all treated and untreated TAF reinforced polymer composites. Water absorption test were carried out for all composite to investigate the effect of chemical treatment on the moisture absorption properties which have direct effect on dimensional stability of the composites. According to the FTIR spectroscopic results the positive chemical treatment removed an amount of lignin and other impurities, produced dialdehyde cellulose in TAF. Water absorption tests and SEM observation results revealed that the composites with treated TAF absorbed the less water and good uniformity between fiber and matrix than composites with untreated TAF. It was found that 10 wt. % treated fiber reinforced composites have higher tensile strength than all other composites.

# CHAPTER 1: INTRODUCTION

## 1.1 Introduction

The word 'composite' means a substance, which is made up by mixing two or more distinct different substances. In most cases mixing is done by physical process. In a few cases it is also done partly by chemical reaction. Polymer composites consist of one or more discontinuous phases embedded in a continuous-phase polymer matrix. The discontinuous phase is usually harder and stronger than the continuous phase, and is called reinforcement, The matrix can be classified as thermoplastic (capable of being separately hardened and softened by the decrease and increase of temperature respectively) or as thermoset (changing into a substantially infusible and insoluble material when cured by the application of heat, or through chemical means) [1].

Typical reinforcements for plastics are various synthetic fibers such as glass, graphite (carbon), boron, organic, metallic and ceramic. These materials are heavy, expensive and harmful to the environment. Lignocellulosic fibers offer many advantages over these most commonly used synthetic fibers as filler reinforcement in thermoplastic matrix. The replacement of inorganic fillers with comparable lignocellulosic fibers provides less weight and decreases the cost without reducing the mechanical properties of the composites [2] any substance that contains both cellulose and lignin is a lignocellulosic material. Lignocellulosic materials include wood, agriculture crops like jute, kenaf, sisal etc. the use of lignocellulosic fibers as the reinforced materials in thermoplastics has received a lot of interest due to their low densities, low cost and non-abrasive nature [3]. The advantages of using lignocellulosic materials also include high specific stiffness and mechanical strength,[4] ease availability, lower cost on unit-volume basis, low hardness, which minimizes the wear of processing equipment, renewability, recyclability, non-hazardousness, biodegradability and so forth [5,6]. Compared with thermosets, composites fabricated from thermoplastic materials typically have a longer shelf life, higher strain to failure, faster to consolidate and retain the ability to be repaired, reshaped and reused as need arises [7-9]. These thermoplastics include polypropylene (PP), polystyrene, vinyl's, low-density polyethylene (LDPE) and high-density polyethylene (HDPE). Recent research on the use of natural fibers suggests that these fibers have high potential for being used as reinforcing fillers in thermoplastics. Experimental results indicate that natural fiber reinforced polypropylene (PP) composites have significant advantages over conventional inorganic filled/reinforced PP systems for certain application [3]. In these researches low density polyethylene (LDPE) polymer used as the matrix material to prepare compression molded natural fiber reinforced LDPE composites.

Many researchers, scientists and engineers have done rigorous research to develop the biodegradable and sustainable composites using natural fiber [1]. Synthetic fiber shows impressive mechanical, thermal properties and durability but when we think about its disposal processing it produces huge amount of black smoke and bad smell. To overcome the problem, the importance of natural fiber-based composites is demanding to make the world green. Natural fibers have made a remarkable impact as a potential substitute for conventional synthetic fibers like aramid and glass fiber over a past few

decades. Because of their mechanical properties, good thermal insulation properties, low density, nonabrasive nature, easy availability from restorable sources, cheaper prices and recyclability of natural fiber reinforced polymer composites have attracted the composite industry, both for structural and nonstructural applications [2].

In this research work, *Typha angustata* fiber was used as reinforcing materials with thermoplastics to make composites. *Typha Angustata* is a perennial plant and is an aquatic weed and is widely grown in waterlogged areas and it belongs to the family *Typhaceae* in the plant kingdom [3]. It is highly available in Bangladesh as waste fiber. Composites are used in various applications such as households, packaging, aerospace, sports and automobile industries by replacing metallic components due to their inherent light weight and immune to corrosion [10, 11].

However, natural fiber gives rise to incompatibility between fiber and polymer in composites due to hydrophilic nature. Natural fibers are composed of cellulose, hemicellulose, lignin, pectin, wax and ash. Chemical treatment of natural fibers can solve the limitations due to hydrophilic nature by purifying the fiber surface, chemically modify the surface, prevent the moisture absorption and gives the better compatibility between fiber and polymer in composites [3]. Siregar*et al.* Investigated and reported on effect of alkali (NaOH) treatment on the mechanical properties of pineapple leaf fiber (PALF) reinforced high impact polystyrene (HIPS) composites [4]. They obtained highest mechanical properties value for the short PALF treatment with NaOH concentration of 4%. Ponnukrishnan *et al.* investigated on effect of the alkali (NaOH) treatment on tensile properties of Typha domingensis reinforced polyester and reported increase in the strength of composite with the increase of fiber content in the polyester matrix [5]. Tanvir sultana *et al.* worked on effect on the properties of low-density polyethylene composites reinforced with treated and untreated *Typha angustata* fibers. They reported that alkali (NaOH) treated based composites notably gave better compatibilities than untreated fiber-based composites [6]. In the recent years, it has been noticed that not much appreciable researches have been carried out on the optimized surface treatment, manufacturing techniques and product application using the *Typha angustata* fibers. So there are more scope to develop economically and ecologically superior engineering material by utilizing these *Typha angustata* fibers and its composites [6].

In this research work, *Typha angustata* fiber was treated with sodium hydroxide to improve its adhesion with polystyrene. The vision of this research work is to find out the effects of treated and untreated *Typha angustata* fiber loading on the physico-mechanical properties *Typha angustata* fiber- polystyrene composites.

## 1.2 Objective

In the literature lot of work has been found on natural fiber-polystyrene and natural fiber-low density polyethylene composites. However, very limited works have been reported on pre- and post-treatment together on the natural fiber-polystyrene and natural fiber-low density polyethylene composites. The aim and objective of the present work is to investigate and prepare some improved compression molded *Typha angustata* – polystyrene composite and *Typha angustata* – LDPE composite materials by chemically pretreatment of *Typha angustata* fiber.

The specific aims and objectives of this research are:

1. Collection of raw materials, i.e.; polystyrene, Low density polyethylene (LDPE), good quality *Typha angustata* fiber and the chemicals required for *Typha angustata* fibers treatment.
2. To modify raw *Typha angustata* fibers by sodium hydroxide and sodium meta per iodate oxidation process which will make the fiber more active by decreasing its hydrophilic nature.
3. Structural and morphological characterization of treated *Typha angustata* fibers by FT-IR and SEM analyses.
4. To prepare *Typha angustata* – polystyrene and *Typha angustata* – LDPE composite materials followed up by blender mixing and then compression molding process using both the treated and untreated *Typha angustata* fiber.
5. Evaluation of mechanical properties for all the prepared composites such as tensile strength, tensile modulus, elongation at break, flexural strength and flexural modulus etc.
6. To investigate the effect of chemical treatment of *Typha angustata* fibers on the performance of treated *Typha angustata* reinforced polystyrene and LDPE composites comparing with the untreated *Typha angustata* reinforced polystyrene and LDPE composites.
7. To find out the properties of the composites according to fiber loading variation.
8. To characterize the interfacial bonding between polystyrene matrix and fiber, LDPE matrix and fiber in the composites by morphological SEM analyses.
9. To increase the water repellent properties of the *Typha angustata* – polystyrene composites and *Typha angustata* – LDPE composites.

# CHAPTER 2: LITERATURE REVIEW

This chapter offers a brief review on the literature related to the composite's materials, raw materials used in this research work (polystyrene and *Typha angustata,* LDPE and *Typha angustata*), fabrication of composites and physico-mechanical properties of the composites. The details of chemical treatments of natural fibers and their effects on the properties of composite materials are also discussed.

## 2.1 Introduction and classification of composite materials

The term composite materials are used to describe microscopic combinations of two or more materials. The fundamental goal in the production and application of composite materials to achieve a performance from the composite that is not available from the separate constituents or from other materials. The concept of improved performance is broad and includes increased strength or reinforcement of one material by the addition of another material [8]. The modern interest in composite materials can be traced in the development of Bakelite, or phenolic resin, which was synthesized by Backland a Belgium scientist, in 1906 [8]. Bakelite was a hard, brittle material that had a very few mechanical applications of its own. However, the addition of a filler, (the earliest applications used short cellulose fibers) yielded Bakelite molding compounds that were very strong, tough and found several applications in mass-produced automobile components. The wood flour additives improved Bakelite's processibility, physical, chemical and electrical properties as well as reducing its cost [8].

Polymer composites are now an important class of engineering materials. The properties of composites are largely influenced by the properties of their constituents and the distribution and interactions among them. The constituents usually interact in a synergistic way, providing properties that are not accounted by a simple volume fraction sum of the components. Along with the volume fraction and the distribution of discrete units in the discontinuous phase, the interfacial area plays an important role in determining the extent of interaction between the reinforcement and the matrix and in this way the final properties of the composite. The fabrication technology of composites, as well as some of their physical properties, is dominated by the chemistry and rheology of the matrix resin and by the type and physical form of the reinforcement [1].

The use of reinforcing agents makes it possible for any thermoset- or thermoplastic-matrix property to be improved or changed to meet varying requirements. Thus, most polymer composites have been developed or fabricated to improve mechanical properties such as strength, stiffness or toughness. The strengthening efficiency of the discontinuous phase plays the most important role in these products and the strengthening mechanism depends strongly on the geometry of the reinforcements. Therefore, polymer composites can be classified according to geometry of reinforcement materials. The three major classes of polymer composites are fibrous, laminar and particulate. The commonly accepted classification scheme for polymer composites are represented in figure 2.1 [1].

```
                    ┌─────────────────────┐
                    │ Reinforced Plastics │
                    └─────────────────────┘
                              │
              ┌───────────────┴───────────────┐
    ┌──────────────────┐          ┌──────────────────┐
    │ Fiber Reinforced │          │ Particle Reinforced │
    │     Plastics     │          │     Polymers      │
    └──────────────────┘          └──────────────────┘
         │                              │
    ┌────┴─────┐              ┌─────────┴─────────┐
┌─────────┐ ┌──────────┐ ┌──────────────┐ ┌──────────────┐
│ Random  │ │ Preferred│ │ Single-layer │ │  Multilayer  │
│Orientation││Orientation││  Composites  │ │  Composites  │
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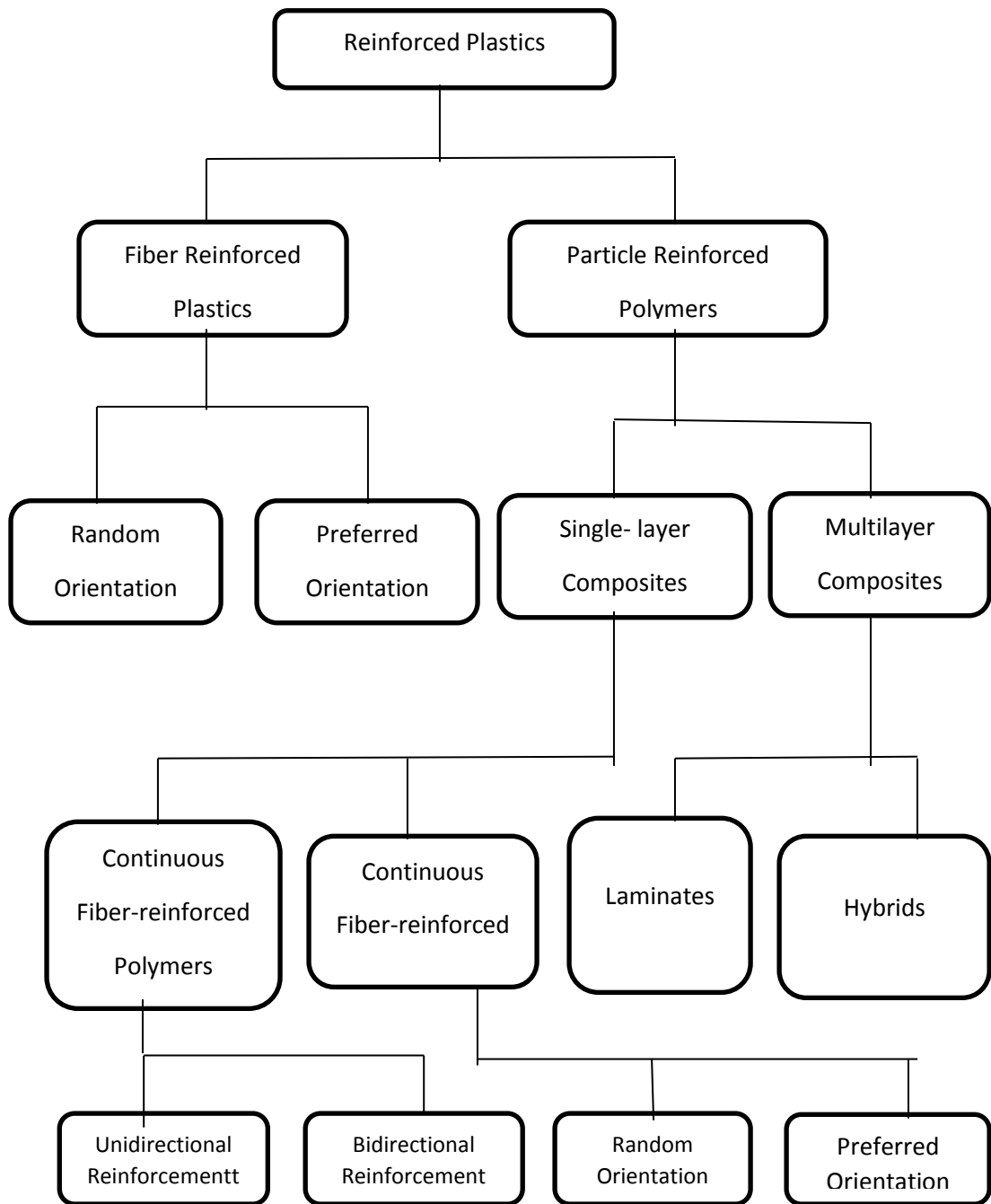
Figure 2.1: Classification of Polymer composites.

Single-layer Composites branches to:
- Continuous Fiber-reinforced Polymers
- Continuous Fiber-reinforced
- Laminates
- Hybrids

Continuous Fiber-reinforced Polymers branches to:
- Unidirectional Reinforcementt
- Bidirectional Reinforcement

Continuous Fiber-reinforced branches to:
- Random Orientation
- Preferred Orientation

## 2.1.1 Plastics and composite materials

Plastics and composite materials are to be found in practically all areas of everyday life – in car dashboards, as insulates in buildings, or as lightweight components in aircraft for example. Composites are gaining significance through the increased use of lightweight design processes in a wide range of applications. The integration of new materials, extended functions, and intelligent systems into future products opens up new

opportunities for various branches of industry such as building construction, mechanical engineering, road transport, and aviation, along with new possibilities for the intelligent fusion of functionality, weight reduction, and aesthetics; this all has the potential to conserve materials and energy resources and to protect the environment. Functionalized lightweight applications such as these are usually only feasible with the use of new high-tech materials and critical substances. Savings in the usage phase (lower energy requirements, longer lifespan) are frequently offset by more intricate production processes (more expensive, more detrimental to the environment) and in many cases by still unresolved disposal issues (incineration instead of high-quality recycling). The ecological and economic suitability of these materials can only be assessed under consideration of the interplay of all these factors in the overall system, since their high complexity precludes overgeneralization. Analysis of the entire life cycle is crucial in order to make statements about overall ecological impact and costs. The Life Cycle Engineering Department has at its disposal a unique fund of knowledge and many years of experience in the field of the life cycle-related ecological assessment of conventional and high-performance materials, application scenarios, and recycling processes of today and tomorrow. This is a substantial constituent of strategically oriented, ecologically and economically appropriate research and development work.

## 2.1.2 Plastic

A plastic is a material that can change its shape. Many things are made of plastics, usually because making them the right shape is easy. There are many types of plastic. Some can be shaped only when they are freshly made; then they become hard. Others are thermoplastic and can be softened by heating them. These plastics can be used for 3D printing, because the plastic will become soft enough to form into different shapes, and then become hard again when they cool down. Most plastics are man-made; they do not occur in nature. The process of making plastics is usually quite complicated. Most of the materials that are called plastic are polymers. Polymers are long chains of atoms bonded to each other. In most plastics, the long chain is a chain of carbon atoms with other atoms attached to them. The different atoms and the shape and length of the chains change how the plastic looks and works. People experimented with plastics based on natural polymers for a very long time. Alexander Parks, an English inventor (1813-1890), created early form of plastic in 1855. It was hard but flexible and transparent. He called it "Parkesine" but now it is called "Celluloid. "Old plastics are usually thrown away and put in landfills. Some plastics release toxic fumes when heated. Some plastics are recycled so they won't become waste. Some plastics have other materials added to them, like glass, because they make the plastic stronger and stiffer. Plastics are mostly petrochemicals, made from natural gas or from petroleum, a type of oil. Chemical engineers refine the petroleum which goes through a heating process. It develops ethylene and propylene, which are the chemical building blocks for many plastics. These chemicals are then combined with other chemicals to produce a polymer. Today, some of these plastics are also being made without oil. Instead, other sources like *plants* and bacteria are used to make the plastic.

These plastics are called bio plastics. They are useful because they can be eaten by other bacteria instead of throwing them away.

## 2.1.3 Composite Materials

A composite material can be defined as a combination of two or more materials that results in better properties than those of the individual components used alone. In contrast to metallic alloys, each material retains its separate chemical, physical, and mechanical properties. The two constituents are reinforcement and a matrix. The main advantages of composite materials are their high strength and stiffness, combined with low density, when compared with bulk materials, allowing for a weight reduction in the finished part. The reinforcing phase provides the strength and stiffness. In most cases, the reinforcement is harder, stronger, and stiffer than the matrix. The reinforcement is usually a fiber or a particulate. Particulate composites have dimensions that are approximately equal in all directions. They may be spherical, platelets, or any other regular or irregular geometry. Particulate composites tend to be much weaker and less stiff than continuous fiber composites, but they are usually much less expensive. Particulate reinforced composites usually contain less reinforcement (up to 40 to 50 volume percent) due to processing difficulties and brittleness. A fiber has a length that is much greater than its diameter. The length-to-diameter (l/d) ratio is known as the aspect ratio and can vary greatly. Continuous fibers have long aspect ratios, while discontinuous fibers have short aspect ratios. Continuous-fiber composites normally have a preferred orientation, while discontinuous fibers generally have a random orientation. Examples of continuous reinforcements include unidirectional, woven cloth, and helical winding (Fig. 2.1.3.1 a), while examples of discontinuous reinforcements are chopped fibers and random mat (Fig.2.1.3.1 b). Continuous-fiber composites are often made into laminates by stacking single. Sheets of continuous fibers in different orientations to obtain the desired strength and stiffness properties with fiber volumes as high as 60 to 70 percent. Fibers produce high-strength composites because of their small diameter; they contain far fewer defects (normally surface defects) compared to the material produced in bulk. As a general rule, the smaller the diameter of the fiber, the higher its strength, but often the cost increases as the diameter becomes smaller. In addition, smaller-diameter high-strength fibers have greater flexibility and are more amenable to fabrication processes such as weaving or forming over radii. Typical fibers include glass, aramid, and carbon, which may be continuous or discontinuous. The continuous phase is the matrix, which is a polymer, metal, or ceramic. Polymers have low strength and stiffness, metals have intermediate strength and stiffness but high ductility, and ceramics have high strength and stiffness but are brittle. The matrix (continuous phase) performs several critical functions, including maintaining the fibers in the proper orientation and spacing and protecting them from abrasion and the environment.

**Continuous**

Unidirectional (UD)

Cloth

Roving

0°

0°/90° (Woven)

±30° Helical
Filament Wound

(a)

**Discontinuous**

Chopped

Mat

(b)
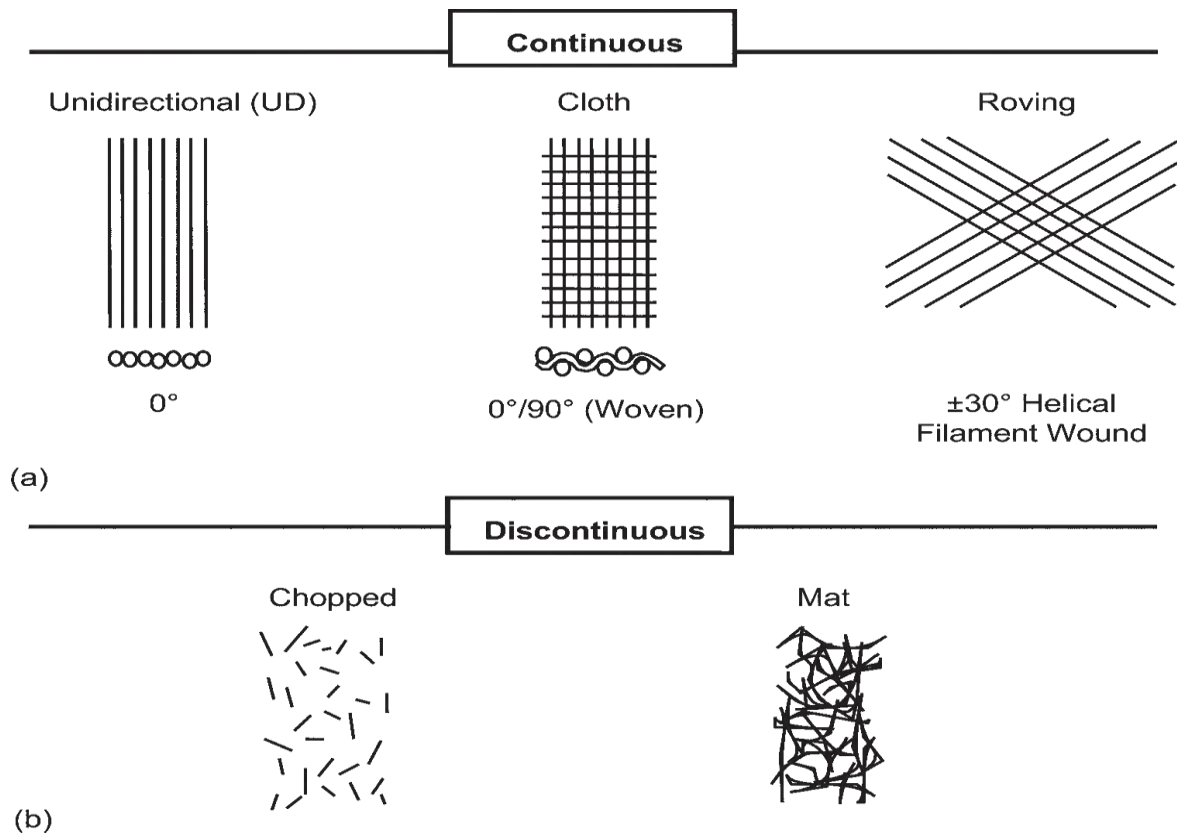
**Fig. 2.1.3.1 typical reinforcement types**

In polymer and metal matrix composites that form a strong bond between the fiber and the matrix, the matrix transmits loads from the matrix to the fibers through shear loading at the interface. In ceramic matrix composites, the objective is often to increase the toughness rather than the strength and stiffness; therefore, a low interfacial strength bond is desirable. The type and quantity of the reinforcement determine the final properties. Figure 2.1.3.2 shows that the highest strength and modulus are obtained with continuous-fiber composites. There is a practical limit of about 70 volume percent reinforcement that can be added to form a composite. At higher percentages, there is too little matrix to support the fibers effectively. The theoretical strength of discontinuous-fiber composites can approach that of continuous-fiber composites if their aspect ratios are great enough and they are aligned, but it is difficult in practice to maintain good alignment with discontinuous fibers. Discontinuous-fiber composites are normally somewhat random in alignment, which dramatically reduces their strength and modulus. However, discontinuous-fiber composites are generally much less costly than continuous-fiber composites. Therefore, continuous-fiber composites are used where higher strength and stiffness are required (but at a higher cost), and discontinuous-fiber composites are used where cost is the main driver and strength and stiffness are less important. Both the reinforcement type and the matrix affect processing
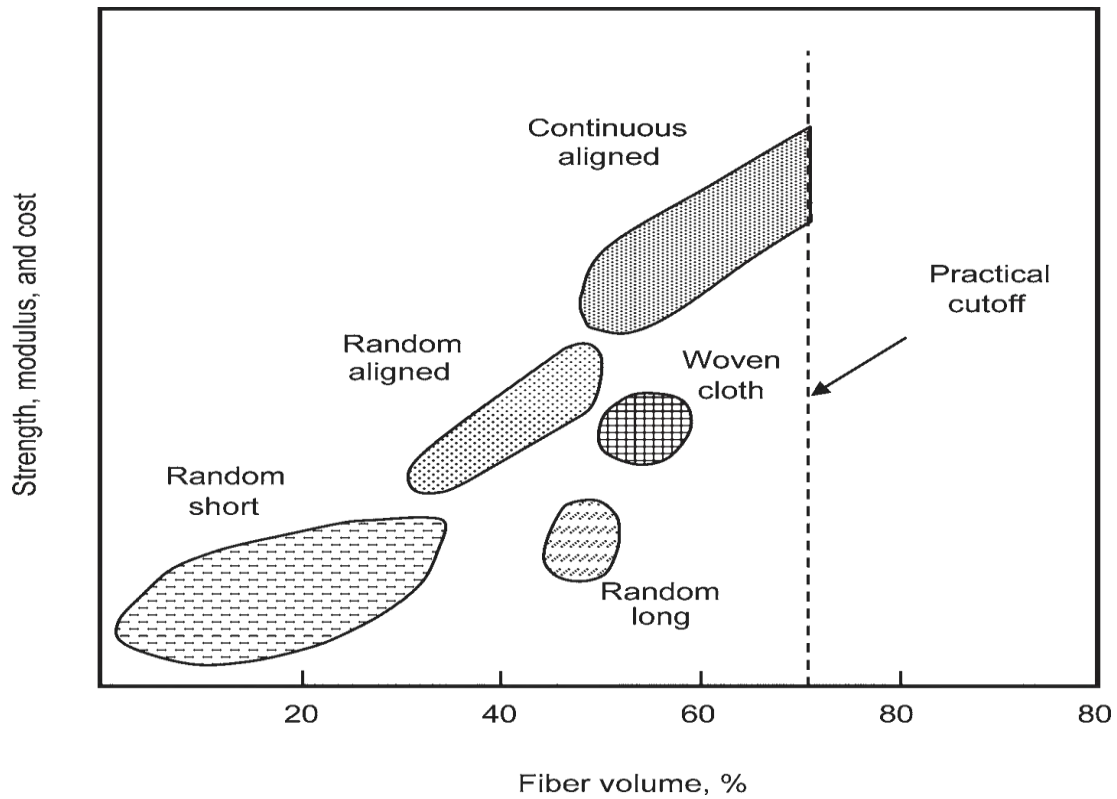
**Fig. 2.1.3.2** Influence of reinforcement type and quantity on composite performance

The major processing routes for polymer matrix composites are shown in Fig. 1.3. Two types of polymer matrices are shown: thermosets and thermoplastics. A thermoset starts as a low-viscosity resin that reacts and cures during processing, forming an intractable solid. A thermoplastic is a high-viscosity resin that is processed by heating it above its melting temperature. Because a thermoset resin sets up and cures during processing, it cannot be reprocessed by reheating. By comparison, a thermoplastic can be reheated above its melting temperature for additional processing. There are processes for both classes of resins that are more amenable to discontinuous fibers and others that are more amenable to continuous fibers. In general, because metal and ceramic matrix composites require very high temperatures and sometimes high pressures for processing, they are normally much more expensive than polymer matrix composites. However, they have much better thermal stability, a requirement in applications where the composite is exposed to high temperatures. This book will deal with both continuous and discontinuous polymer, metal, and ceramic matrix composites, with an emphasis on continuous- fiber, high-performance polymer composites.
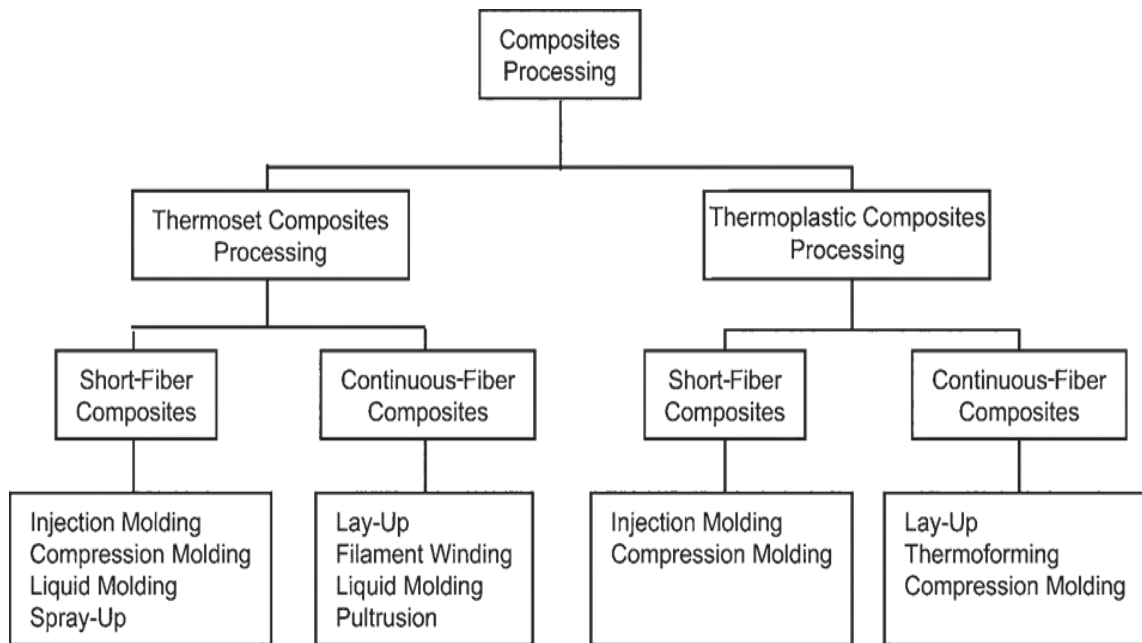
**Fig.2.1.3.3** Major Polymer matrix composite fabrication processes

There is no really, adequate definition of a composite material but the preceding decision give some indication of the scope of the subject. In term of the approach to be adopted here, there are main points to be included in definition of acceptable composite materials for use in structural application-

i)      It consists of two or more physically distained and mechanically separable materials.

ii)     It can be made by mixing the separate in such a way that the dispersion of one material in the other can be done in a controlled way to achieve optimum properties.

iii)    The properties are superior and possibly unique in some specific respects to the properties of the individual components.

## 2.1.4 Properties of Composites Materials

**High strength to weight ratio**

Fiber composites are extremely strong for their weight. By refining the laminate many characteristics can be enhanced. A common laminate of say 3 mm Chopped strand mat is quite flexible compared to say a 3 mm ply. However, it will bend a long way more than the ply before yielding. Stiffness should not be confused with Strength. A carbon fiber laminate on the other hand, will have a stiffness of many times that of mild steel of the same thickness, increased ultimate strength, yet only be less than 1/4 of its weight.

**Lightweight**

A standard Fiber glass laminate has a specific gravity in the region of 1.5, compared to Alloy of 2.7 or steel of 7.8. When you then start looking at Carbon laminates, strengths can be many times that of steel, but only a fraction of the weight. A DVD case lid was produced using carbon fiber to reduce the case's overall weight so that it could be carried as cabin baggage whilst traveling, and for improved security. It was used by support crew for the All Blacks during their 1999 Rugby World Cup campaign.

**Fire resistance**

The ability for composites to withstand fire has been steadily improving over the years. There is two types of systems to be considered:

Fire Retardant - Are self-extinguishing laminates, usually made with chlorinated resins and additives such as Antimony trioxide. These release $CO_2$ when burning so when the flame source is removed, the self-extinguish.

Fire Resistant - More difficult and made with the likes of Phenolic Resins. These are difficult to use, are cured with formaldehyde, and require a high degree of post curing to achieve true fire resistance.

Other materials are also becoming more readily available to be used as in tumescent layers, which expand and blanket the surface, preventing spread of flame. There is paint on coating usually applied to the back of the product laminate, plus a thin fiber film to go under the Gel coat giving the outer surface a blanketing coat as well.

Fiberglass Developments Ltd produces a Fire Door as part of our SteridorTM range. Use of special Phenolic resin has allowed us to create the only fully tested Composite door in Australasia. Fire rated by BRANZ to 4 hours, this door is also approved by MAF as meeting all their Hygiene requirements.

**Electrical properties**

Fiberglass Developments Ltd produced the Insulator Support straps for the Tranz Rail main trunk electrification. The straps, although only 4mm thick, meet the required loads of 22kN, as well as easily meeting insulation requirements.

**Chemical & weathering resistance**

Composite products have good weathering properties and resist the attack of a wide range of chemicals. This depends almost entirely on the resin used in manufacture, but by careful selection resistance to all but the most extreme conditions can be achieved. Because of this, composites are used in the manufacture of chemical storage tanks, pipes, chimneys and ducts, boat hulls and vehicle bodies.

FDL manufactured architectural panels for the construction of the Auckland Marine Rescue Centre. Composite panels were chosen because of their ability to withstand salty sea side conditions without corrosion.

**Color**

Almost any shade of any color can be incorporated into the product during manufacture by pigmenting the gelcoat used. Costs are therefore reduced by no further finishing or painting. Soluble dyes can be used if a translucent product is desired.

We do not however, recommend dark colors. These produce excessive heat on the surface which can lead to the surface deteriorating and showing print through, where the Resin matrix cures more and shrinks, bringing the fibers to the surface. In extreme cases delamination can occur.

**Translucency**

Polyester resins are widely used to manufacture translucent moldings and sheets. Light transmission of up to 85% can be achieved.

**Design flexibility**

Because of the versatility of composites, product design is only limited by your imagination.

**Low thermal conductivity**

Fiberglass Developments has been involved in the development and production of specialized meat containers which maintain prime cuts of chilled meat at the correct temperature for Export markets. They are manufactured using the RTM process, with special reinforcing and foam inserts.

**Manufacturing economy**

Fiberglass Developments produces several models of fuel pump covers for Fuel quip. Fiberglass is an ideal material for producing items of this type for many reasons, including being very economical. Because of its versatile properties, fiberglass can be used in many varied applications.

## 2.1.5 Classification of Composite:

Generally, there are two kind of composite materials:

  i)      Natural composite materials
  ii)     Synthetic composite materials

Again, Broad classification of composite materials:

  i)      Natural composite materials: Wood, Bone, Bamboo, Muscle and other tissue.
  ii)     Micro composite materials: Metallic alloys, e.g. steels, Toughened thermoplastics, e.g. impact polystyrene, ABS, sheet molding compounds, reinforced thermoplastics.
  iii)    Macro composite materials (Engineering products): Galvanized steel, reinforced concrete beams and Helicopter blade skis.

## 2.1.6 Classification of Micro Composite materials:

1) Continuous fibers in matrix: aligned, random.

2) Short fibers in matrix: aligned, random.

3) Particulates (Spheres, plates, ellipsoids, irregular, hollow or solid) in matrix.

4) Dispersion strengthened, as for 3 above, with particle size $< 10^{-8}$m.

5) Lamellas structures.

6) Skeletal or inter penetrating networks.

7) Multicomponent, fibers, particles etc.

Again, Composite materials can be classified based on reinforcement following way-

  i)      Fibrous
  ii)     Laminar
  iii)    Particulate

According to composite Committee of German Society for Metallurgy the following groups comprise the composite materials.

  a)  **Laminated composite**: Metallic foils and foil like structures respectively as well as thin ribbons are imbedded into matrix, or the composite is built up from layers

of various materials or by layers made of laminates (i.e. foil, ribbons, sheet, etc.) and the matrix.

b) **Material with surface layers**: In contrast to the ideal materials these layers are very thin. They are applied by different method and include points. The serve for corrosion and oxidation protections and improvement of wear and impart other physical properties to the material surface.

c) **Fiber composite**: Refractory whisker or polycrystalline fibers are imbedded into a matrix for improvement of mechanical properties.

d) **Particle composite**: This group encompasses a wide range of materials. It includes for instance the embedding of nonmetallic inorganic particles for improvement of mechanical properties of matters and also materials which consists of nonmetallic inorganic particles bound by a small metallic binder phase.

## 2.1.7 Application of Composite Materials

Composites are one of the widely used materials because of their adaptability to different situations and the relative case of combination with other materials to serve specific purposes and exhibit desirable properties. Composites are highly efficient to make the parts and structure of aircrafts. We found the characteristics of the composite material make it very suitable material for aerospace industry. Composites like carbon fiber, carbon epoxy, and glass epoxy are very light and high strength which is mostly used in aircraft industries. In addition, our study takes the first step to highlight the uses of composite material to manufacture the different parts of aircraft's

In surface transportation, reinforced plastics are the kind of composites used because of their huge size. They provide sample scope and receptiveness ''to design changes, materials and processes. Their stiffness and cost effectiveness offered apart from easy availability of raw materials make them the obvious choice for applications in surface transportation in heavy transport vehicles, the composites are used in processing of component parts with cost effectiveness. Good reproductively, resilience handling by semi-skilled workers is the basic requirements may not justify the savings obtained in terms of weight visa vehicle production, carbon fibers reinforced epoxides have been used in racing cars and recently for the safety of cars[5]. Polyester resin with suitable fillers and reinforcements were the first applications of composites inroad transportation. The choice was dictated by properties like low cost, ease in designing and production of parts etc. Using a variety of reinforcements, polyester has continued to be used in improving the system and other application.

Each year, fiber-reinforced polymer composites (FRPs) find their way into hundreds of new applications, from golf clubs and tennis rackets to jet skis, aircraft, missiles and

spacecraft. FRPs offer designers an increasing array of potential uses as a material and system solution. At the same time, composite cost trends are highly favorable, especially when the total cost of fabrication is considered.

Fiber-reinforced plastics – or fiber-reinforced polymers are they are also known – are well-suited to any design that necessitates weight saving, precision engineering, predetermined tolerances and simplification of parts in production and operation. Since FRP typically have a low weight and high strength, with good fatigue, impact and compression properties, making them attractive to a number of trades, including the automotive, aerospace and construction industries.

### Aerospace

Carbon-fiber-reinforced plastics are strong and light, with a high tensile strength, chemical resistance, stiffness, tolerance to temperature and low thermal expansion. Although they can be expensive to manufacture, they are often found wherever a high strength-to-weight ratio is required – in aerospace, ship-building, and the automotive industry for example. The rudder of the Airbus A310 is composed of carbon-FRP, which offers a 25% reduction in weight compared to aluminum sheeting. There is also a 95% reduction in the number of components as parts are combined into simpler molded parts, which are cheaper, quicker and easier to make than cast aluminum or steel objects, and uphold or even surpass tolerance and material strengths. The Airbus A35 XWB, for example, is 52% carbon-FRP, including its wing spars and components of the fuselage. Such measures offer an overall reduction in production and operations costs – the economy of parts results in lower production costs and weight savings, which leads to fuels savings and lowers the cost of flying the plane.

### Civil Engineering

Carbon-FRPs are popular in civil engineering to strengthen concrete, masonry, steel, cast iron and timber structured either by retrofitting existing structures to improve their strength or as an alternative reinforcing material to steel. The principle use of carbon-FRP is in retrofitting structures to improve and increase their load capacity and repair damage – bridges that carry far more traffic than there were ever intended to, for example. Carbon-FRP can be wrapped around certain areas of a structure, a column, for instance, to enhance the shear strength of its reinforced concrete. Aluminum windows, doors and facades are also thermally insulated using plastics made of a glass-fiber-reinforced polyamide.

### Automotive Industry

Carbon-FRPs are also attractive to the automotive industry where their high cost is lessened by the material's unsurpassed strength-to-weight ratio. The lighter weight material replaces metal in the body panels of high-end cars and supercars, making the vehicles not only lighter but more fuel efficient too. Glass-fiber-reinforced plastics, specifically glass-fiber-reinforced PA 66, are also used in the automotive industry in

engine intake manifolds - the part of an engine that supplies the fuel/air mixture to the cylinders. Here, the FRP offers up to 60% reduction in weight over a cast aluminum manifold, along with an improved surface quality and aerodynamics. There is also a decrease in the number of components as parts are combined into simpler molds.

Glass-FRPs are also found in gas and clutch pedals, replacing stamped aluminum. Pedals can be molded as a single unit by combining the pedal and mechanical linkages, again streamlining production and operation. The fibers can also be oriented to reinforce again specific stresses, thereby increasing durability and safety [12].

**Sporting and Consumer Goods**

Carbon-RFPs are also making their way into sporting goods such as squash, tennis and badminton racquets; sport kite spars; hockey sticks; bike frames; fishing rods and surfboards. Paralympian Jonnie Peacock also uses a carbon FRP blade for running (and dancing in 2017 on Strictly Come Dancing), and high-performance drones often consist of carbon-FRP bodies, as do other remote-controlled vehicles and aircraft components – helicopter rotor blades for example. The material's high strength-to-weight ratio makes it ideal for such sporting uses.

Other uses for carbon-FRP include in musical instruments – violin bows and guitar picks, for example, or the whole instrument in some cases! It is also found in firearms where it has replaced certain metal, wood and fiberglass components, and in lightweight poles such as tripods and tent poles [12].

**Other Uses**

Fiber-reinforced plastics have impressive electrical properties and a high-grade environmental resistance, along with good thermal insulation, structural integrity, fire hardiness, UV radiation stability and resistance to chemicals and corrosives. Those reinforced with glass are ideal for the power industry as they have no magnetic field and are resistant to electrical sparks. Plastics reinforced with aramids - a class of synthetic polyamide formed from aromatic or ring-shaped monomers – demonstrate robust heat resistance and exceptional strength and thermals stability. As a result, they are utilized in bullet-proof and fire-resistant clothing [12].

## 2.2 Matrix materials

A fiber-reinforced composite (FRC) is a high-performance composite material made up of three components - the fibers as the discontinuous or dispersed phase, the matrix acts as the continuous phase, and the fine interphase region or the interface. The matrix is basically a homogeneous and monolithic material in which a fiber system of a composite is embedded. It is completely continuous. The matrix provides a medium for binding and holding reinforcements together into a solid. It offers protection to the reinforcements from environmental damage, serves to transfer load, and provides finish, texture, color, durability and functionality.

**Types of Composite Matrix Materials**

There are three main types of composite matrix materials:

**Ceramic matrix: -** Ceramic matrix composites (CMCs) are a subgroup of composite materials. They consist of ceramic fibers embedded in a ceramic matrix, thus forming a ceramic fiber reinforced ceramic (CFRC) material. The matrix and fibers can consist of any ceramic material. CMC materials were designed to overcome the major disadvantages such as low fracture toughness, brittleness, and limited thermal shock resistance, faced by the traditional technical ceramics.

**Metal matrix: -** Metal matrix composites (MMCs) are composite materials that contain at least two constituent parts – a metal and another material or a different metal. The metal matrix is reinforced with the other material to improve strength and wear. Where three or more constituent parts are present, it is called a hybrid composite. In structural applications, the matrix is usually composed of a lighter metal such as magnesium, titanium, or aluminum. In high temperature applications, cobalt and cobalt-nickel alloy matrices are common. Typical MMC's manufacturing is basically divided into three types: solid, liquid, and vapor. Continuous carbon, silicon carbide, or ceramic fibers are some of the materials that can be embedded in a metallic matrix material. MMCs are fire resistant, operate in a wide range of temperatures, do not absorb moisture, and possess better electrical and thermal conductivity. They have also found applications to be resistant to radiation damage, and to not suffer from outgassing. Most metals and alloys make good matrices for composite applications.

**Polymer matrix: -** Polymer matrix composites (PMCs) can be divided into three sub-types, namely, thermoset, thermoplastic, and rubber. Polymer is a large molecule composed of repeating structural units connected by covalent chemical bonds. PMC's consist of a polymer matrix combined with a fibrous reinforcing dispersed phase. They are cheaper with easier fabrication methods. PMC's are less dense than metals or ceramics, can resist atmospheric and other forms of corrosion, and exhibit superior resistance to the conduction of electrical current [13].

## 2.2.1 Thermoplastic Polymer

A thermoplastic, or thermo-softening plastic, is a plastic polymer material that becomes pliable or moldable at a certain elevated temperature and solidifies upon cooling [14, 15]. Most thermoplastics have a high molecular weight. The polymer chains associate by intermolecular forces, which weaken rapidly with increased temperature, yielding a viscous liquid. In this state, thermoplastics may be reshaped and are typically used to produce parts by various polymer processing techniques such as injection molding,

compression molding, calendaring, and extrusion. [16, 17] Thermoplastics differ from thermosetting polymers which form irreversible chemical bonds during the curing process. Thermosets do not melt when heated, but typically decompose and do not reform upon cooling. Above its glass transition temperature and below its melting point, the physical properties of a thermoplastic change drastically without an associated phase change. Some thermoplastics do not fully crystallize below the glass transition temperature, retaining some or all of their amorphous characteristics. Amorphous and semi-amorphous plastics are used when high optical clarity is necessary, as light is scattered strongly by crystallites larger than its wavelength. Amorphous and semi-amorphous plastics are less resistant to chemical attack and environmental stress cracking because they lack a crystalline structure.

Brittleness can be decreased with the addition of plasticizers, which increases the mobility of amorphous chain segments to effectively lower the glass transition temperature. Modification of the polymer through copolymerization or through the addition of non-reactive side chains to monomers before polymerization can also lower it. Before these techniques were employed, plastic automobile parts would often crack when exposed to cold temperatures. These are linear or slightly branched long chain molecules capable of repeatedly softening on heating and hardening on cooling.
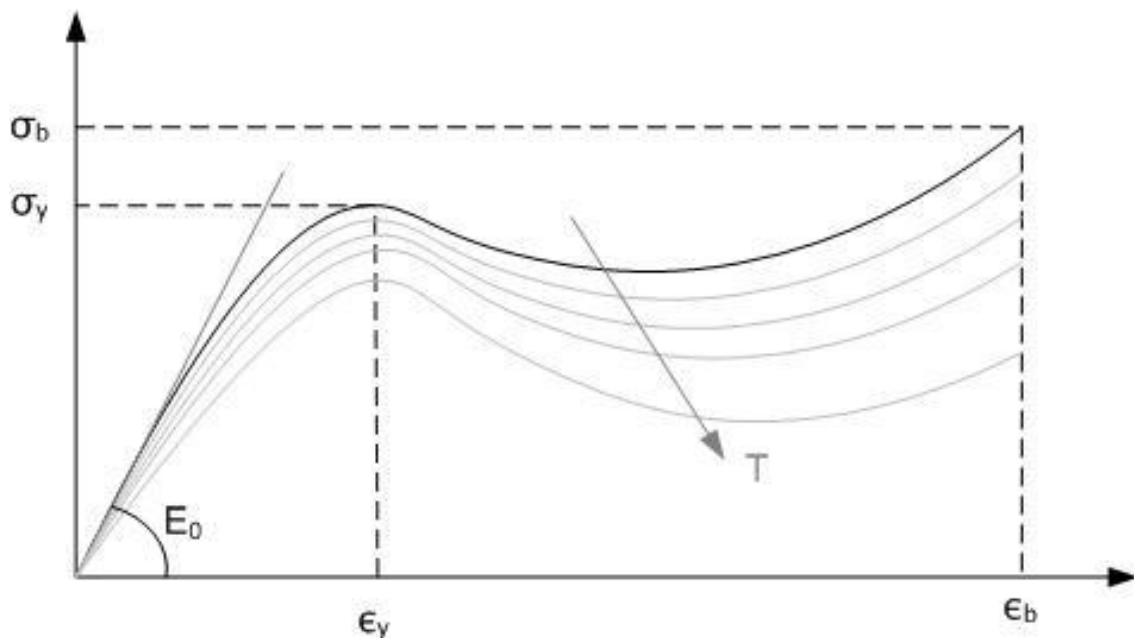


**Fig 2.2.1** Stress-strain graph of a thermoplastic material [14, 15]

## 2.2.2 Low Density Polyethylene (LDPE)

## 2.2.2.1 General Description

Plastic polymers are becoming essential products in industrial markets. Their low price and good aesthetic qualities are important factors determining the rapid growth of manufacturing and usage of polymeric plastics which are generally produced from fossil fuels.

Low-density polyethylene (LDPE) is a thermoplastic made from oil. It was the first grade of polyethylene, produced in 1933 by ICI using a high pressure process via free radical polymerization. During the propagation of polymer chains, branching can occur, this occurs when a chain curls back and bonds to an earlier part of the chain. When this curl breaks, it leaves small chains sprouting from the main carbon backbone. Branched carbon chains cannot line up as close to each other as unbranched chains can. This causes less contact between atoms of different chains, and fewer opportunities for induced or permanent dipoles to occur. A low-density result from the chains being further apart. Lower melting points and tensile strengths are evident, because the intermolecular bonds are weaker and require less energy to break (the only forces possible for non-polar polymers such as polyethylene are dispersion forces which are due to instantaneous dipoles that form as the charge clouds in the molecules fluctuate) In general, LDPE has more branching (on about 2% of the carbon atoms) than HDPE, it is defined by a density range of 0.910 - 0.940 g/cc. It is unreactive at room temperatures, except by strong oxidizing agents, and some solvents cause its swelling, it is quite flexible, and tough to the degree of being almost unbreakable [18] For more than a decade, low density polyethylene (LDPE) has been widely employed in industrial sectors due to its low cost, manufactured easily, long durability to electric properties [19] and long chemical resistibility to most adverse conditions. Various types of LDPE are mainly used as insulating and shielding materials, food-packing films, and as a dispensing bottle. LDPE, also, is used widely as an electrical insulation material because of its low dielectric loss and its superior breakdown strength [20]. QAPCO (Qatar Petrochemical Corporation) is a very well-known company for its production of LDPE with different grades and high qualities and distributes this product all over the world [21]. LDPE is produced by the company from two production lines; each of 180,000MT/year capacity and using tubular and vessel reactor technology.

## 2.2.2.2 Polyethylene

Polyethylene (PE) is a common name for polymers produced in the methane polymerization process. Polyethylene (PE) is a thermoplastic, elastic and non-toxic polymer. It is difficult to ignite and burns with a non-smoky flame. Polyethylene is translucent; it does not solve in all widely used solvents and is resistant to chemical substances such as acids, alkali and saline solutions. Polyethylene (PE) is an inexpensive industrial raw material. In order to improve Polyethylene (PE) properties, it is modified.

Polyethylene or polythene (abbreviated PE; IUPAC name polyethene or poly (methylene)) is the most common plastic. As of 2017, over 100 million tons of polyethylene resins are produced annually, accounting for 34% of the total plastics market [22, 23]. Its primary use is in packaging (plastic bags, plastic films,

geomembranes, containers including bottles, etc.). Many kinds of polyethylene are known, with most having the chemical formula (C2H4)n. PE is usually a mixture of similar polymers of ethylene with various values of n. Polyethylene is a thermoplastic; however, it can become a thermoset plastic when modified (such as cross-linked polyethylene).

### 2.2.2.3 History

Polyethylene was first synthesized by the German chemist Hans von Pechmann, who prepared it by accident in 1898 while investigating diazomethane [24, 25]. When his colleagues Eugen Bamberger and Friedrich Tschirner characterized the white, waxy substance that he had created, they recognized that it contained long –CH2– chains and termed it polyethylene [26].

The first industrially practical polyethylene synthesis (diazomethane is a notoriously unstable substance that is generally avoided in industrial application) was discovered in 1933 by Eric Fawcett and Reginald Gibson, again by accident, at the Imperial Chemical Industries (ICI) works in Northwick, England [27]. Upon applying extremely high pressure (several hundred atmospheres) to a mixture of ethylene and Benz aldehyde they again produced a white, waxy material. Because the reaction had been initiated by trace oxygen contamination in their apparatus, the experiment was, at first, difficult to reproduce. It was not until 1935 that another ICI chemist, Michael Perrin, developed this accident into a reproducible high-pressure synthesis for polyethylene that became the basis for industrial low-density polyethylene (LDPE) production beginning in 1939. Because polyethylene was found to have very low-loss properties at very high frequency radio waves, commercial distribution in Britain was suspended on the outbreak of World War II, secrecy imposed, and the new process was used to produce insulation for UHF and SHF coaxial cables of radar sets. During World War II, further research was done on the ICI process and in 1944 Bakelite Corporation at Sabine, Texas, and Du Pont at Charleston, West Virginia, began large-scale commercial production under license from ICI [28].

The breakthrough landmark in the commercial production of polyethylene began with the development of catalyst that promoted the polymerization at mild temperatures and pressures. The first of these was a chromium trioxide–based catalyst discovered in 1951 by Robert Banks and J. Paul Hogan at Phillips Petroleum [29]. In 1953 the German chemist Karl Ziegler developed a catalytic system based on titanium halides and organoaluminium compounds that worked at even milder conditions than the Phillips catalyst. The Phillips catalyst is less expensive and easier to work with, however, and both methods are heavily used industrially. By the end of the 1950s both the Phillips- and Ziegler-type catalysts were being used for HDPE production. In the 1970s, the Ziegler system was improved by the incorporation of magnesium chloride. Catalytic systems based on soluble catalysts, the metallocenes, were reported in 1976 by Walter Kaminsky

and Hansjörg Sinn. The Ziegler- and metallocene-based catalysts families have proven to be very flexible at copolymerizing ethylene with other olefins and have become the basis for the wide range of polyethylene resins available today, including very low-density polyethylene and linear low-density polyethylene. Such resins, in the form of UHMWPE fibers, have (as of 2005) begun to replace aramids in many high-strength applications.

## 2.2.2.4 Classification of Polyethylene

Polyethylene is classified by its density and branching. Its mechanical properties depend significantly on variables such as the extent and type of branching, the crystal structure, and the molecular weight. There are several types of polyethylene:

- Ultra-high-molecular-weight polyethylene (UHMWPE)
- Ultra-low-molecular-weight polyethylene (ULMWPE or PE-WAX)
- High-molecular-weight polyethylene (HMWPE)
- High-density polyethylene (HDPE)
- High-density cross-linked polyethylene (HDXLPE)
- Cross-linked polyethylene (PEX or XLPE)
- Medium-density polyethylene (MDPE)
- Linear low-density polyethylene (LLDPE)
- Low-density polyethylene (LDPE)
- Very-low-density polyethylene (VLDPE)
- Chlorinated polyethylene (CPE)

With regard to sold volumes, the most important polyethylene grades are HDPE, LLDPE, and LDPE.

**Ultra-high-molecular-weight polyethylene (UHMWPE)**

UHMWPE is polyethylene with a molecular weight numbering in the millions, usually between 3.5 and 7.5 million amu [30]. The high molecular weight makes it a very tough material, but results in less efficient packing of the chains into the crystal structure as evidenced by densities of less than high-density polyethylene (for example, 0.930–0.935 g/cm3). UHMWPE can be made through any catalyst technology, although Ziegler catalysts are most common. Because of its outstanding toughness and its cut, wear, and excellent chemical resistance, UHMWPE is used in a diverse range of applications. These include can- and bottle-handling machine parts, moving parts on weaving machines, bearings, gears, artificial joints, edge protection on ice rinks, steel cable replacements on ships, and butchers' chopping boards. It is commonly used for the construction of articular portions of implants used for hip and knee replacements. As fiber, it competes with aramid in bulletproof vests.

**High-density polyethylene (HDPE)**

HDPE is defined by a density of greater or equal to 0.941 g/cm3. HDPE has a low degree of branching. The mostly linear molecules pack together well, so intermolecular forces are stronger than in highly branched polymers. HDPE can be produced by chromium/silica catalysts, Ziegler–Natta catalysts or metallocene catalysts; by choosing catalysts and reaction conditions, the small amount of branching that does occur can be controlled. These catalysts prefer the formation of free radicals at the ends of the growing polyethylene molecules. They cause new ethylene monomers to add to the ends of the molecules, rather than along the middle, causing the growth of a linear chain. HDPE has high tensile strength. It is used in products and packaging such as milk jugs, detergent bottles, butter tubs, garbage containers, and water pipes. One-third of all toys are manufactured from HDPE. In 2007, the global HDPE consumption reached a volume of more than 30 million tons [30].

**Linear low-density polyethylene (LLDPE)**

LLDPE is defined by a density range of 0.915–0.925 g/cm3. LLDPE is a substantially linear polymer with significant numbers of short branches, commonly made by copolymerization of ethylene with short-chain alpha-olefins (for example, 1-butene, 1-hexene, and 1-octene). LLDPE has higher tensile strength than LDPE, and it exhibits higher impact and puncture resistance than LDPE. Lower thickness (gauge) films can be blown, compared with LDPE, with better environmental stress-cracking resistance, but is not as easy to process. LLDPE is used in packaging, particularly film for bags and sheets. Lower thickness may be used compared to LDPE. It is used for cable coverings, toys, lids, buckets, containers, and pipe. While other applications are available, LLDPE is used predominantly in film applications due to its toughness, flexibility, and relative transparency. Product examples range from agricultural films, Saran wrap, and bubble wrap, to multilayer and composite films. In 2013, the world LLDPE market reached a volume of US$40 billion [31].

**Low-density polyethylene (LDPE)**

LDPE is defined by a density range of 0.910–0.940 g/cm3. LDPE has a high degree of short- and long-chain branching, which means that the chains do not pack into the crystal structure as well. It has, therefore, less strong intermolecular forces as the instantaneous-dipole induced-dipole attraction is less. This results in a lower tensile strength and increased ductility. LDPE is created by free-radical polymerization. The high degree of branching with long chains gives molten LDPE unique and desirable flow properties. LDPE is used for both rigid containers and plastic film applications such as plastic bags and film wrap. In 2013, the global LDPE market had a volume of almost US$33 billion [32]. The radical polymerization process used to make LDPE does not include a catalyst that "supervises" the radical sites on the growing PE chains. (In HDPE synthesis, the radical sites are at the ends of the PE chains, because the catalyst stabilizes their formation at the ends.) Secondary radicals (in the middle of a chain) are more stable than primary radicals (at the end of the chain), and tertiary radicals (at a branch point) are more stable yet. Each time an ethylene monomer is added, it creates a primary radical,

but often these will rearrange to form more stable secondary or tertiary radicals. Addition of ethylene monomers to the secondary or tertiary sites creates branching.

## 2.2.2.5 Properties of LDPE

| Molecular Weight | 400000 gm/mole |
|---|---|
| Density | 0.92 |
| Uses | Thermoplastics, fibers |
| Polymerization | Free radical chain polymerization Ziegler- Natta polymerization Metallocene catalysis Polymerization |
| Morphology | Highly crystalline (linear), highly amorphous (branched) |
| Melting Temperature | $137^0$C |
| Glass transition temperature | $-130^0$C to $-80^0$C |
| Monomer | Ethylene |
| Elongation at break (%) | 400 |
| Tensile modulus (GPa) | 0.25 |
| Tensile Strength (MPa) | 5-25 |

**Table 2.2.2.5 Properties of LDPE**

## 2.2.3 Polystyrene

## 2.2.3.1 General Description

Polystyrene (PS) is a synthetic aromatic hydrocarbon polymer made from the monomer styrene [32]. Polystyrene can be solid or foamed. General-purpose polystyrene is clear, hard, and rather brittle. It is an inexpensive resin per unit weight. It is a rather poor barrier to oxygen and water vapor and has a relatively low melting point [33]. Polystyrene is one of the most widely used plastics, the scale of its production being several million tonnes per year [34]. Polystyrene can be naturally transparent, but can be colored with colorants. Uses include protective packaging (such as packing peanuts and CD and DVD cases), containers, lids, bottles, trays, tumblers, disposable cutlery [35] and in the making of models.

As a thermoplastic polymer, polystyrene is in a solid (glassy) state at room temperature but flows if heated above about 100 °C, its glass transition temperature. It becomes rigid again when cooled. This temperature behavior is exploited for extrusion (as in Styrofoam) and also for molding and vacuum forming, since it can be cast into molds with fine detail.

Polystyrene is slow to biodegrade and is therefore a focus of controversy among environmentalists. It is increasingly abundant as a form of litter in the outdoor environment, particularly along shores and waterways, especially in its foam form, and also in increasing quantities in the Pacific Ocean [36].

## 2.2.3.2 History

Polystyrene was discovered in 1839 by Eduard Simon, an apothecary from Berlin [37] from storax, the resin of the American sweet gum tree Liquidambar styraciflua, he distilled an oily substance, a monomer that he named styrol. Several days later, Simon found that the styrol had thickened into a jelly he dubbed styrol oxide ("Styroloxyd") because he presumed an oxidation. By 1845 Jamaican-born chemist John Buddle Blyth and German chemist August Wilhelm von Hofmann showed that the same transformation of styrol took place in the absence of oxygen [38].They called the product "metastyrol"; analysis showed that it was chemically identical to Simon's Styroloxyd [39] In 1866 Marceline Berthelot correctly identified the formation of metastyrol/Styroloxyd from styrol as a polymerization process [40]. About 80 years later it was realized that heating of styrol starts a chain reaction that produces macromolecules, following the thesis of German organic chemist Hermann Staudinger (1881–1965). This eventually led to the substance receiving its present name, polystyrene. The company I. G. Farben began manufacturing polystyrene in Ludwigshafen, about 1931, hoping it would be a suitable replacement for die-cast zinc in many applications. Success was achieved when they developed a reactor vessel that extruded polystyrene through a heated tube and cutter, producing polystyrene in pellet form. [Citation needed] In 1941, Dow Chemical invented a Styrofoam process [41].

Before 1949, chemical engineer Fritz Stastny (1908–1985) developed pre-expanded PS beads by incorporating aliphatic hydrocarbons, such as pentane. These beads are the raw material for moulding parts or extruding sheets. BASF and Stastny applied for a patent that was issued in 1949. The moulding process was demonstrated at the Kunststoff Messe 1952 in Düsseldorf. Products were named Styropor. The crystal structure of isotactic polystyrene was reported by Giulio Natta [42]. In 1954, the Koppers Company in Pittsburgh, Pennsylvania, developed expanded polystyrene (EPS) foam under the trade name Dylite [43].In 1960, Dart Container, the largest manufacturer of foam cups, shipped their first order [44].

## 2.2.3.3 Structure of Polystyrene

In chemical terms, polystyrene is a long chain hydrocarbon wherein alternating carbon centers are attached to phenyl groups (a derivative of benzene).

Polystyrene's chemical formula is $(C_8H_8)$ n; it contains the chemical elements carbon and hydrogen. The material's properties are determined by short-range van der Waals attractions between polymers chains. Since the molecules consist of thousands of atoms, the cumulative attractive force between the molecules is large. When heated (or deformed at a rapid rate, due to a combination of viscoelastic and thermal insulation

properties), the chains are able to take on a higher degree of conformation and slide past each other. This intermolecular weakness (versus the high intramolecular strength due to the hydrocarbon backbone) confers flexibility and elasticity. The ability of the system to be readily deformed above its glass transition temperature allows polystyrene (and thermoplastic polymers in general) to be readily softened and molded upon heating. Extruded polystyrene is about as strong as unalloyed aluminum but much more flexible and much less dense (1.05 g/cm3 for polystyrene vs. 2.70 g/cm$^3$ for aluminum).
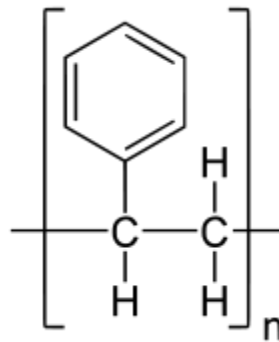
Figure 2.2.3.3 Structure of Polystyrene

## 2.2.3.4 Polymerization

Polystyrene results when styrene monomers interconnect. In the polymerization, the carbon–carbon π bond of the vinyl group is broken and a new carbon–carbon σ bond is formed, attaching to the carbon of another styrene monomer to the chain. The newly formed σ bond is stronger than the π bond that was broken; thus, it is difficult to depolymerize polystyrene. About a few thousand monomers typically comprise a chain of polystyrene, giving a molecular weight of 100,000–400,000.

Each carbon of the backbone has tetrahedral geometry, and those carbons that have a phenyl group (benzene ring) attached are stereo genic. If the backbone were to be laid as a flat elongated zigzag chain, each phenyl group would be tilted forward or backward compared to the plane of the chain. The relative stereo chemical relationship of consecutive phenyl groups determines the tactility, which has an effect on various physical properties of the material. The diastereomer where all of the phenyl groups are on the same side is called isotactic polystyrene, which is not produced commercially.

## 2.3 Lignocellulosic fibers

Lignocellulosic fibers refer to those agro based substances which contain cellulose, hemicellulose and lignin as their structural molecules. Lignocellulosic include wood, agricultural residues, water plants, grasses and other plant substances [45] To better understand the properties of lignocellulosic fiber reinforced composite materials, it is necessary to know the physical and chemical properties of lignocellulosic fibers. Fibers are classified as [10, 11].
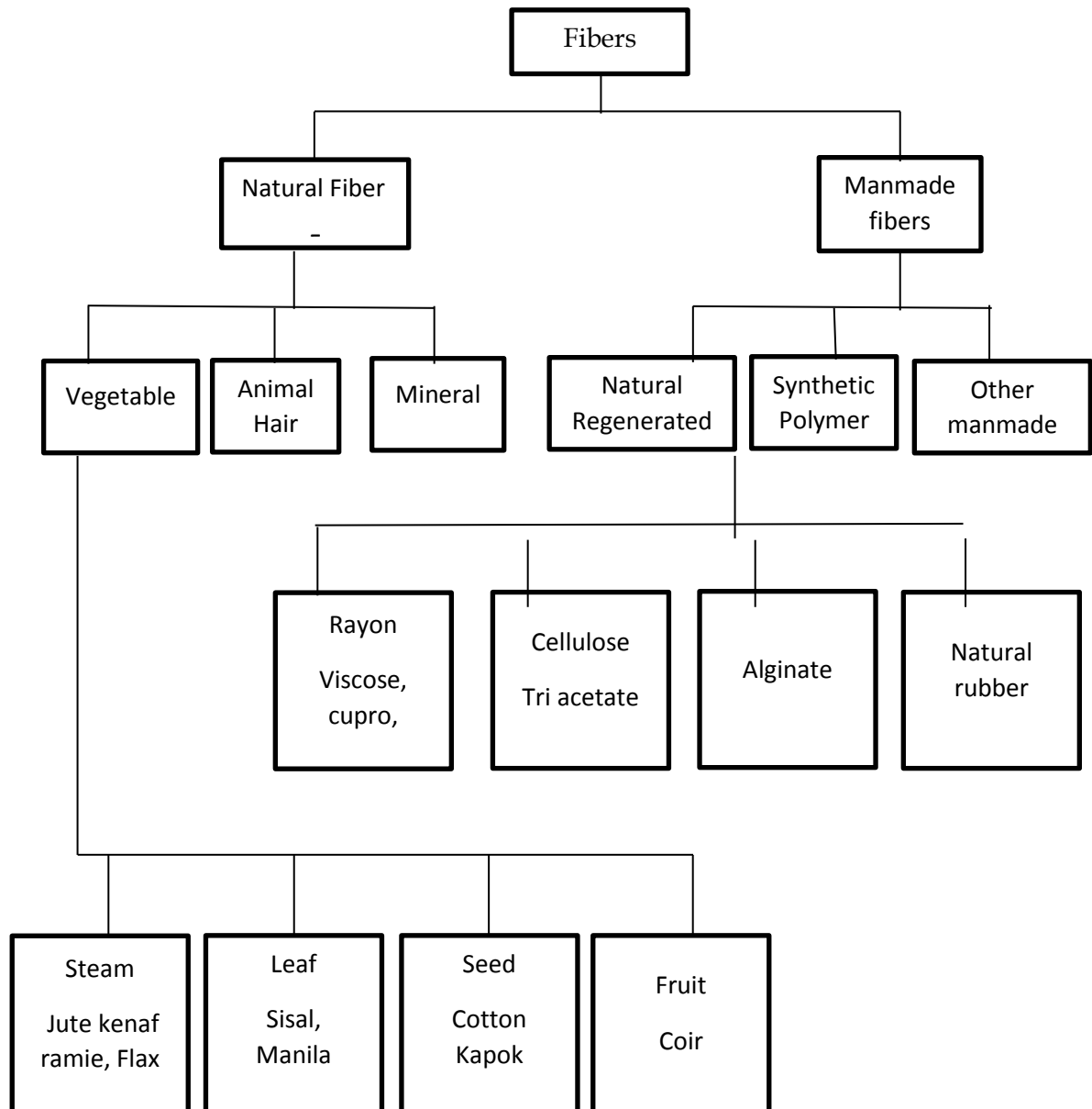
**Figure 2.3: Classifications of fibers [10]**

Agro- based lignocellulosic fibers are also classified on the basis of part of the plant where they store in [46]. Five different classes of fiber are: (1) bast or stem fibers, which are the fibrous bundles in the inner bark of the plant stem running the length of the stem; leaf fibers, which rim the length of leaves; (3) seed-hair fibers; (4) core, pith or stick fibers, which form the low density, spongy inner part of the stem of certain plants and (5) all other plant fibers not included above[15]. Examples of bast or stem fibers include jute, flax, hemp, kenaf, ramie, Roselle and urena. Leaf fibers include banan, sisal, henequen, abaca, pineapple, cantala, caroa, Mauritius and phormium. Seed-hair fibers include coir, cotton, and kapok and milk weed floss. Core fibers represent the centre or pith fibers of

such plants as kenaf and jute and can present over 85 percent of the dry weight of these plants. The remaining fibers include roots, leaf segments, flower heads, seed hulls and short stem fibers. While individual single fibers in all of these classes are quite short (except for flax, hemp, ramie, cotton and kapok). However, the fibers obtained from inner bark of the plant are quite long. For example, hemp, jute and kenaf can have fiber bundles as long as 400 cm and abaca, Mauritius and phormium are about half this length. Considering all types plant fiber, there is a vast array of potential long and short fibers for composite production [46] Table 2.3.1 shows a comparison of properties of natural lignocellulosic fibers and conventional man-made fibers, jute, ramie, flax and sisal are the most commonly used fibers for polymer composites.

Cellulose is the basic structural component of all plant fibers. It is the most important organic compound produced by plants and the most abundant in the biosphere. The cellulose molecule consists of glucose units linked together in long chains, which in turn are linked together in bundles called micro fibrils. The tensile strength of the cellulose micro fibrils is very high. It is the strongest amongst the known material with a theoretically estimated tensile strength of 7.5 GPa or 10, 87,500 pounds per square inch. In the S2 layer the micro fibrils run almost parallel to the fiber axis. With S2 representing about 50% of the cell wall, this gives the fibers a very high tensile strength [47].

Hemicellulose is also found in all plant fibers. Hemicellulose is polysaccharides bonded together in relatively short, branching chains. They are intimately associated with the cellulose micro fibrils, embedding the cellulose in a matrix. Hemicelluloses are very hydrophilic (i.e. containing many sites to which water can readily bond) [47].

Lignin is a Latin word for wood. Lignin is the compound which gives rigidity to the plant. Without lignin, plants could not attain great heights (e.g. trees) or the rigidity found in some annual crops (e.g. straw). Lignin is a three-dimensional polymer with an amorphous structure and a high molecular weight. On the three main constituents in fibers, it is expected that lignin would be the one with least affinity for water. Another important feature of lignin is that it is thermoplastic (i.e. at temperatures around $90^0$C it starts to soften and at temperatures around 170 $^0$C starts to flow) [47]. Lignin in jute makes the fiber resistant to ease microbial attacks, and provides better strength along with hardness and brittleness [48].

## 2.3.1 Properties of natural and synthetic fibers

**Table 2.3.1: Properties of natural and synthetic fibers [49]**

|  | Fibers | Density | Tensile strength | Tensile modulus GPa | Elongation at Break, % | Decomposition Temp $^0$C | Price In Euros/ Kg |
|---|---|---|---|---|---|---|---|
| Natural | Jute | 1.3-1.5 | 187-540 | 3-55 | 1.4-3.1 | 270 | 0.7 |
|  | Ramie |  | 585-900 | 33 | 2.0-3.5 | 260 |  |
|  | Hemp Fibers | 1.4-1.5 | 580-1110 | 3-90 | 1.3-4.7 | 258 |  |
|  | Bamboo |  |  | 17-29 | 3.2 |  |  |
|  | Flax | 1.4 | 250-1000 | 12-100 | 1.3-40 | 280 | 0.15-0.76 |
|  | Sisal | 1.4 | 507-855 | 24 | 2.9 | 270 | 0.7-1.02 |
|  | Cotton | 1.5-1.6 | 350 | 11 | 2-10 |  | 0.35 |
|  | Banana | 1.3 | 791 | 30 | 2.10 |  | 0.7-0.9 |
|  | Kenaf | 1.4 | 930 | 53 |  | 270 |  |
|  | Coconut |  | 544 | 14 |  |  | 0.36-0.45 |
|  | Wood fibers | 0.6 |  | 12 | 2.90 |  | 0.31-0.35 |
|  | Cellulose | 1.2 |  | 2.1 |  |  |  |
|  | Wood flour | 0.6 |  | 10 |  |  |  |
| Synthetic | E glass | 2.5 | 1625-3400 | 72 | 2-5 | 756 | 1.6-2.0 |
|  | C- glass | 2.5 | 2800 | 69 |  | 756 | 9.3-16 |
|  | S-glass | 2.5 | 4600 | 87 |  | 946 | 8.3-20 |
|  | A-glass | 2.5 | 2400 | 68 |  | 696 | 2.5 |
|  | Aramid (Kevlar) | 1.4 | 2380-3100 | 124 |  | 496 | 16-67 |
|  | Carbon Fibers | 1.8-1.9 | 2090-5200 | 525 |  | 3647 | 33-166 |
|  | Zirconia fibers | 5.6 | 700 | 100 |  | 2497 | 41.7 |
|  | Alumina Fibers | 2.8 | 1000 | 100 |  | 1997 |  |

The polymers lignin and hemicellulose are responsible for most of the physical and chemical properties such as: biodegradability, flammability, sensitivity towards moisture, thermo plasticity, degradability by UV- light etc [49] Some structures of the monomeric units for the major polymeric constituents of cellulosic plant fibers are given below: [50]

## 2.4 Lignocelluloses fiber reinforced thermoplastic composites

There is a long story of the use of lignocellulosic materials with thermosetting polymeric materials, like phenol or urea formaldehyde in the production of composites. The use of lignocellulosic with thermoplastics, however, is a more recent innovation. Lignocellulosic fibers have been incorporated in a wide variety of thermoplastic materials such as polypropylene, polyethylene, polystyrene, poly vinyl chloride, polyamides. Thermoplastic materials selected for use with lignocellulosic materials must melt at or below the degradation point of the lignocellulosic component, normally 200-$220^0$C.

## 2.4.1 Lignocellulosic in combination with other materials

Combining of lignocellulosic materials with thermoplastic materials can be done in several ways [51] in one case. Thermoplastic materials are simply mixed with bio-based powder or fiber (nut shell powder or wood fiber are presently used) and the mixture are heated. The plastic melts, but the wood fiber and plastic components remain as distinct separate phases. One example of this technology is reinforced thermoplastic composites, which are light weight, have improved acoustical and heat reform ability properties, and cost less than comparable products made from plastic alone. These advantages make possible the exploration of new processing techniques, new applications, and new markets in such areas as packaging, furniture, housing and automobiles.

A second way to combine wood fiber and plastics is to use a compatibilizer to make the hydrophob (plastic) mix better with the hydrophil (wood). The two components remain as separate phases, but if delamination or void formation can be avoided, properties can be improved over those of either separate phase. These types of materials are usually referred to as wood fiber/ plastic blends [51].

A final combination of wood fiber and thermoplastics is in products that can be best described as wood plastic alloys. In this case the wood and plastic have become one material and it is not possible to separate them. The formation of wood plastic alloys is possible through fiber modification and grafting research. This can be done if we consider that bio-based fibers consist of a thermoset polymer (cellulose) in a thermoplastic matrix (lignin and the hemicellulose). The glass transition temperature (GTT), however, of the thermoplastic matrix is higher than the decomposition temperature of the fiber. If the GTT were lowered through chemical modification, it should be possible to thermo plasticize the lignin and the hemicelluloses at temperatures below decomposition. If a reactive thermoplastic is used it is then reacted with the modified bio-based fiber, it should be possible to form bio-based fiber/ thermoplastic alloys [51]. Rowell *et al* [52] reported that only the hemicellulose and lignin have been modified in thermo plasticization using maleic and succinic anhydrides. Matsuda *et al* [53] also extensively investigated the esterification of wood in order to make a totally thermoplastic material. They esterified wood without a solvent by simply heating wood powder with succinic anhydride in a mixer for 3h at temperatures higher than $60^0$C.

## 2.4.2 Properties of lignocellulosic fiber-thermoplastic composites

Before 1980, the concepts of blends and alloys were essentially unknown in the plastic industry. Blends and alloys have revolutionized the plastic industry, as they offer new materials with properties that were not achieved before. These materials can be tailored for specific end uses. The jute and kenaf industries have the same opportunity to follow this trend and greatly expand markets for new materials based on blends and alloys with other resources. Newer materials and composites that have both economic and environmental benefits are being considered for applications in the automotive, building, furniture and packaging industries. Mineral fillers and fibers are used frequently in the plastic industry to achieve desired properties or to reduce the cost of the finished article. For example, glass fiber is used to improve the stiffness and strength of plastics, although there are several disadvantages associated with its use. Glass fiber production requires a great deal of energy processing temperature can exceed $1200^0$C. They tend to abrade processing equipment and increase the density of the plastic system. The use of lignocellulosic fibers in thermoplastics has received a lot of interest due to their low densities, low cost and nonabrasive nature. The inherent polar and hydrophilic nature of the jute and kenaf fibers and the nonpolar characteristics of the polyolefin lead to difficulties in compounding and result in inefficient composites [3] The high moisture absorption of the lignocellulosic fibers and their low microbial resistance are disadvantages (table 2.7) that need to be considered, particularly during shipment and long term storage as well as during processing of the composites [9] Proper selection of additives is necessary to improve the interaction and adhesion between the fiber and matrix phases. Recent research on the use of jute and kenaf fiber suggests that these fibers have the potential for being used as reinforcing fillers in thermoplastics [3] the low cost and densities and the nonabrasive nature of the fibers allow high filling levels and thereby result in significant cost savings. The primary advantages of using these fibers as additives in plastics are; low densities, low cost, nonabrasive, high filling levels, low energy consumption, high specific properties, renewability, wide distribution, biodegradability and improvement of the rural/ agriculture-based economy [3].

| Advantages | Disadvantages |
|---|---|
| Low cost | High moisture absorption |
| Renewable | Poor microbial resistance |
| Low density | Low thermal resistance |
| Nonabrasive | Local and seasonal quality variations |
| Low energy consumption | Demand and supply cycles limited |

| High specific properties | |
|---|---|
| High strength and elasticity modulus | |
| No skin irritations | |
| No residues when incinerated | |
| Fast water absorption/ desorption | |
| Good thermal conductivity | |
| Biodegradability | |

**Table 2.4.1: Main advantages and disadvantages of lignocellulosic fibers [9]**

Saw dust and other lignocellulosic materials change dimensions with changing moisture content because the cell wall polymers contain hydroxyl and other oxygen-containing groups that attract moisture though hydrogen bonding [54]. This hydrophilic behavior affects the properties of the fiber themselves as well as the properties of their composites. The hydrophilic character of fibers is usually incompatible with hydrophobic matrix materials unless a compatibilizer or coupling agent is used. This leads to poor interfacial adhesion between the fiber and matrix as well as poor fiber dispersion.

In general, Cellulosic fillers and fibers have a higher young's modulus as compared to commodity thermoplastics, thereby contribution to the higher stiffness of the composites. The increase in the Young's modulus with the addition of cellulosic materials depends on many factors such as amount of fiber used, orientation of the fibers, interaction and adhesion between the fiber and matrix [55] In general, dispersing agents and coupling agents, are necessary for the property enhancement, when fibers are incorporated into thermoplastics. Dispersing agents facilitate the fiber dispersion and improve the interfacial adhesion between the fibers and the polymer matrix. Grafting of chemical species on to the fiber surface has also been reported to improve the interaction between the fiber and matrix. Although grafting can improve the properties of the composite to a significant extent, this process increases the material cost of system. The use of dispersing agents and coupling agents is a cheaper route to improve properties and makes more practical sense for high volume, low cost composite system [55].

In a natural fiber-thermoplastic composite the lignocellulosic phase is present in a wide range of diameters and lengths, some in the front of short filaments and other in the form that seem closer to the individual fiber. The high shearing energy of blending the filaments and the polymer in a mixer results in fiber attrition but can also axially separate the filaments into discrete individual fibers. Cellulosic fillers can be classified under three categories depending on their performance when incorporated to a plastic matrix.

Wood flour and other low cost agriculture based flour can be considered as particulate fillers that enhance the tensile and flexural moduli of the composite with little effect on the composite strength. Wood fibers and recycled newspaper fibers have higher aspect ratios and contribute to an increase in the moduli of composite and can also improve the strength of the composite when suitable additives are used to improve stress transfer between the matrix and the fibers. The improvement in modulus is not significantly different than the cellulosic particulate fillers. The most efficient cellulosic additives are some natural fibers such as kenaf, jute, flax etc. The specific Young's moduli, specific flexural modulus, the ratio of the composite modulus to the composite specific gravity of composites with natural fibers such as kenaf are significantly higher than those with wood fibers. The specific moduli (the ratio of the composite modulus to the composite specific gravity) of high fiber volume fraction of bast fibers-PP composites are high and in the range of glass fibers-PP composites. The most efficient natural fibers are those that have a high cellulose content complied with a low micro fibril angle resulting in high filament mechanical properties [55]. A list of mechanical properties of PP composites reinforced by different natural fiber and inorganic substances is given table no: 2.8 [3, 56, 57]

**Table 2.4.2: Comparison of filled LDPE composites [3, 56, 57]**

| Reinforcement of PP | ASTM standard | None | Kenaf | Jute | Talc | $Ca_2CO_3$ | Glass | Mica |
|---|---|---|---|---|---|---|---|---|
| % filler by weight | | 0 | 50 | 50 | 40 | 40 | 40 | 40 |
| % filler by volume (estimated) | | 0 | 39 | 39 | 18 | 18 | 19 | 18 |
| Tensile modulus, GPA | D 638 | 1.7 | 8.3 | 7.8 | 4 | 3.5 | 9 | 7.6 |
| Specific tensile modulus, GPA | | 1.9 | 7.8 | 7.2 | 3.1 | 2.8 | 7.3 | 6.0 |
| Tensile strength, MPa | D 638 | 33 | 68 | 72 | 35 | 25 | 110 | 39 |
| Specific tensile strength, MPa | | 37 | 58 | 67 | 28 | 20 | 89 | 31 |
| Elongation at break, % | D 638 | 10 | 2.2 | 2.3 | _ | _ | 2.5 | 2.3 |
| Flexural strength, MPa | D 790 | 41 | 91 | 99 | 63 | 48 | 131 | 62 |

| | | | | | | | | |
|---|---|---|---|---|---|---|---|---|
| Specific flexural strength, MPa | | 46 | 85 | 92 | 50 | 38 | 107 | 49 |
| Flexural modulus, GPA | D 790 | 1.4 | 7.8 | 7.7 | 4.3 | 3.1 | 6.2 | 6.9 |
| Specific flexural modulus, GPa | | 1.6 | 7.3 | 7.1 | 3.4 | 2.5 | 5.0 | 5.5 |
| Notched Izod impact J/m | D 256A | 24 | 32 | 31 | 32 | 32 | 107 | 27 |
| Specific gravity | | 0.9 | 1.07 | 1.08 | 1.27 | 1.25 | 1.23 | 1.26 |
| Water absorption % 24 hr | D 570 | 0.02 | `1.05 | _ | 0.02 | 0.02 | 0.06 | 0.03 |
| Mold (linear) Shrinkage | | 0.028 | 0.003 | _ | 0.01 | 0.01 | 0.004 | _ |

## 2.4.3 Effect of coupling agents on the composite properties

Coupling agents improve the adhesion between filler and polymer preferably via chemical bonds; their use confers reinforcing properties on inexpensive extenders, improves the performance of reinforcements, and allows the filler content to be increased. The reinforcing effect of fillers depends on their chemistry, shape (fibers, flakes, spheres), and size (fiber length, particle size). The most important reinforcement effects are [58]

1. Increased strength, ultimate elongation, rigidity (modulus of elasticity), and in some cases impact strength.

2. Improved heat distortion temperature and dimensional stability, reduced shrinkage, and improved ~ stability of mechanical values at high temperatures and over extended times (fatigue)

3. Modification of density (usually increased)

4. Improved chemical resistance and lower water absorption

5. Better surface quality and surface hardness

Special fillers are used to obtain compounds with the following properties:

1. Low flammability

2. Electrical conductivity and electromagnetic shielding

3. Radiation and UV shielding
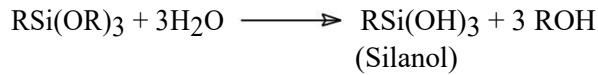
4. Biodegradability

5. Noise suppression

## 2.4.4 Some chemical modification for property enhancement of lignocellulosic fiber reinforced thermoplastic composites

The properties of lignocellulosic materials result from the chemistry of the cell wall components. The basic properties of a fiber can bechanged by modifying the basic chemistry of the cell wall polymers. Many chemical reaction systems have been reported for the modification of agro-fiber. These chemicals include anhydrides (such as phthalic, succinic, malefic, propionic and butyric anhydride etc.), acid chlorides, ketene carboxylic acids, different types of isocyanates, formaldehyde, acetaldehyde, difunctional aldehydes, chloral, phthaldehydic acid, dimethyl sulfate, alkyl chlorides, betapropiolactone, acrylonitrile, epoxides (such as, ethylene, propylene, butylene) and some difunctional epoxides: [59] By far, the most research has been done on the reaction of acetic anhydride with cell wall polymer hydroxyl groups to give an acetylated fiber. Many different types of lignocellulosic fibers including wood 2i-Z2, bamboo, bagasse, jute [60], kenaf etc. have been acetylated using a variety of procedures. Although acetylation is not the only chemical modification procedure that has been shown to improve properties of lignocellulosic materials even then it has been the most studied and are used in many cases [59] some researchers [61-64] have reported that acetylation of the lignocellulosic fibers have reduced the hydrophilicity and water absorption behavior of lignocellulosic fibers. The scheme of the reaction of lignocellulosic fiber with acetic anhydride is given below:

$$\text{Fiber-OH} + CH_3\text{-CO-O-CO-}CH_3 \text{ ----------------> Fiber-O-CO-}CH_3 + CH_3\text{-CO-OH}$$
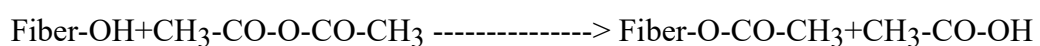
Modification of fiber surface by reacting with silane were investigated and reported by some researchers [65, 66] Silane treatment of fibers led to increase mechanical properties of the composites. The reaction steps in the silane grafting of bio fibers are shown below:

**Hydrolysis:**

$$RSi(OR)_3 + 3H_2O \longrightarrow RSi(OH)_3 + 3\ ROH$$
(Silanol)

**Condensation:**

$$3RSi(OH) \longrightarrow \ \begin{array}{ccc} R & R & R \\ | & | & | \\ OH{-}Si{-}O{-}Si{-}O{-}Si{-}OH \\ | & | & | \\ OH & OH & OH \end{array}$$
(Siloxane)

**Hydrogen Bonding:**

BioFiber OH + OH — Si–O — Si–O –Si –OH  --------▸  (hydrogen bonded siloxane–biofiber structure)

**Surface Grafting:**

(hydrogen bonded siloxane–biofiber structure) --------▸ (grafted siloxane–biofiber structure) + 3H₂
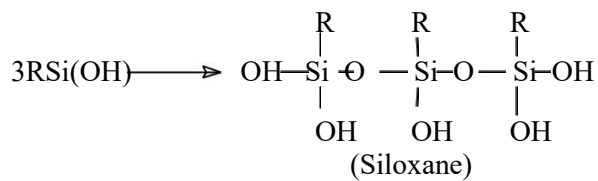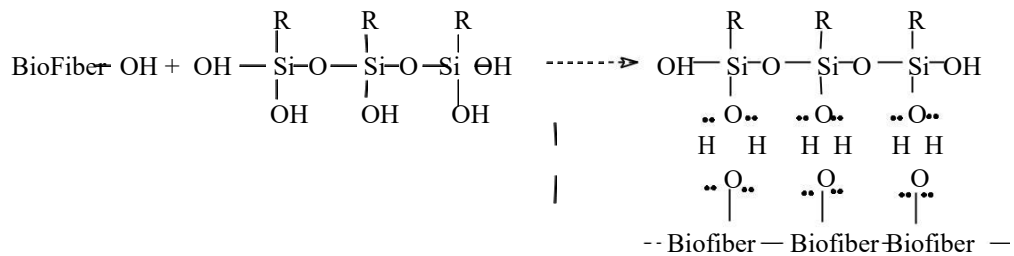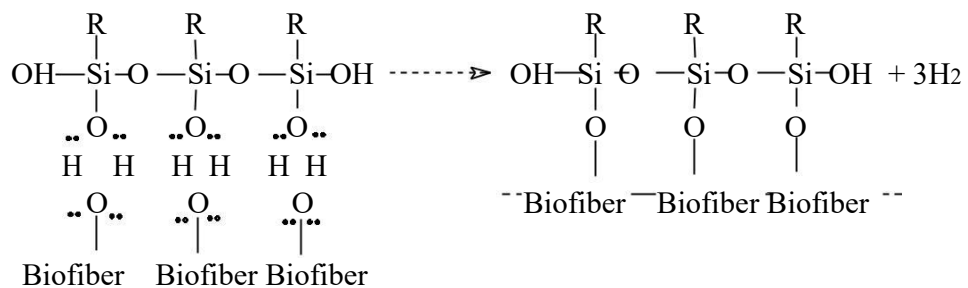
Scheme 2.4.4.1: Reaction steps in the silane grafting of biofibers [65]

Modification of jute fiber with HcMA and the effect of HEMA on the mechanical and thermal properties of jute-polycarbonate composite were investigated and repoted [67]. It has been reported that HEMA deposited on hessian cloth might have quite extensively reacted with the cellulosic backbone of cellulose through graft-copolymerization reaction (scheme 2.4.4.2).
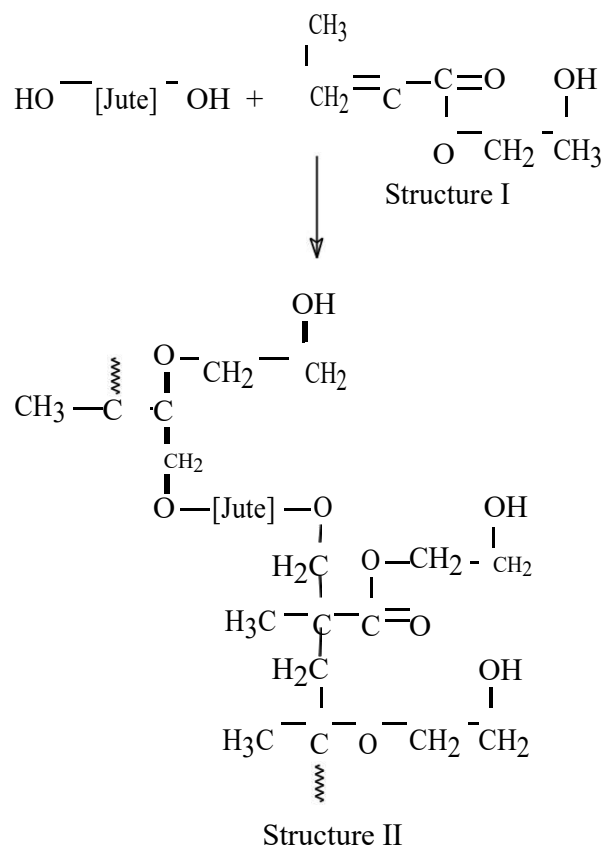
**Scheme 2.4.4.2:** Proposed graft-copolymerization reaction of jute fiber withHEMA[67]

Grafting of polymethyl acrylate (PA) onto pure cellulose by potassium diperiodatocuprate (III) initiating system was carried out by L. Yinghai et al [68] and reported that the thermal stability of grafted product has been improved greatly.
The initiation mechanism of grafting reaction is shown below:
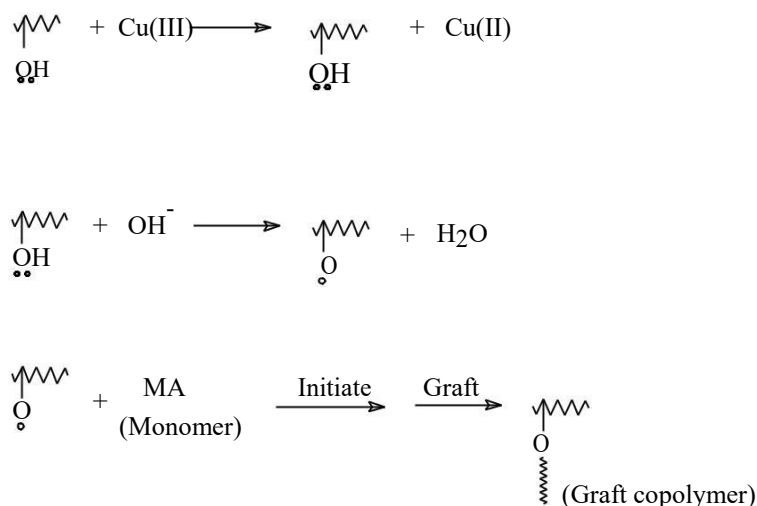
**Scheme 2.4.4.4.3:** Proposed graft-copolymerization of MA onto cellulose [68]

A considerable amount of research has been done on the use of MA as a coupling agent to improve the compatibility and adhesion between the fibers and matrix using a variety of procedures. A proposed method of grafting MA on PP chain is shown in Scheme 2.4.4.4 [65]
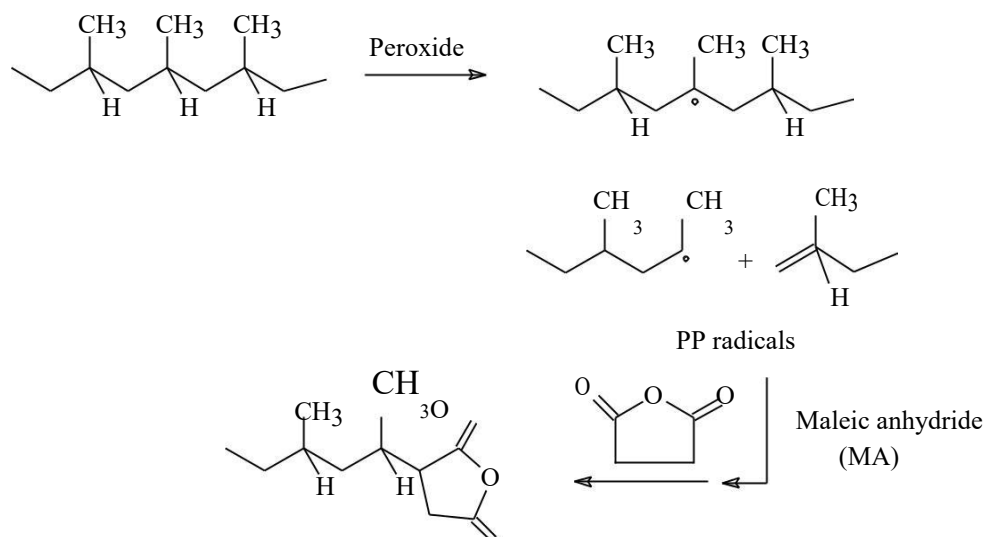


**Scheme 2.4.4.4:** Proposed reaction mechanism of MA on PP chain [65]

It is now evident from the literature that the addition of 0.5-2 wt-% malefic anhydride grafted polypropylene (MAHgPP) to PP matrices significantly improved the adhesion strength of jute fibers and in turn the mechanical properties of composites [69]. A hypothetical structure of coupling of MAHgPP with jute fiber is given below:

2% MAHgPP    MAH Coupling  MAHgPP and PP

layer                interdiffusion zone
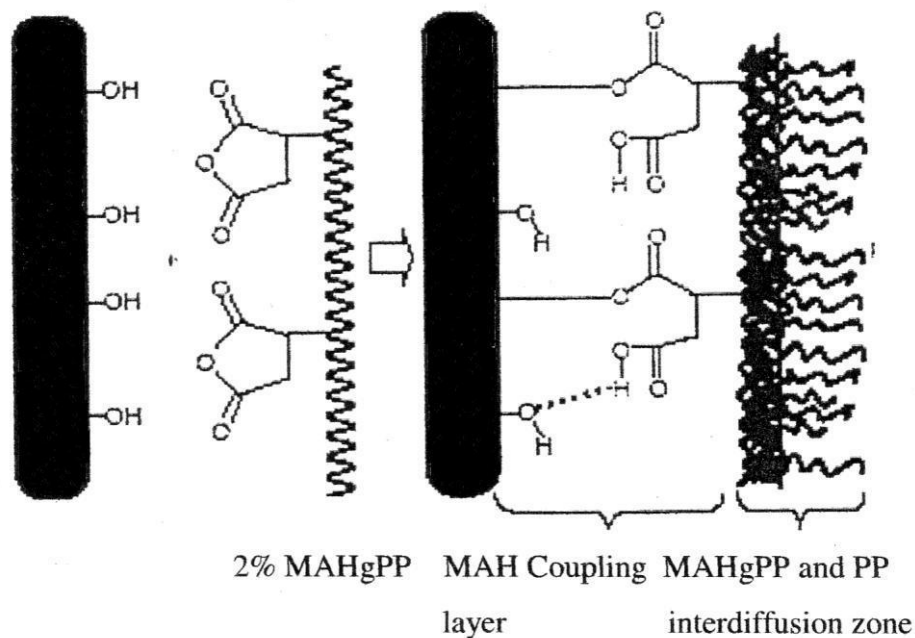
**Scheme 2.6:** Hypothetical structure of MAHgPP coupling with jute fiber at the interface [69]

L. K. Poh *et al* [66] carried out the esterification of palm oil empty fruit bunch (EFB) fibers using benzoyl chloride and reported that benzoylation treatment has improved the thermal stability and changed the suprastructure of the EFB fibers which may improve the interfacial bonding of the lignocellulosic material and thermoplastic materials. The chemical reactions involved are shown below:

Wood — OH +   NaOH ------▷   Wood —ONa  + H O
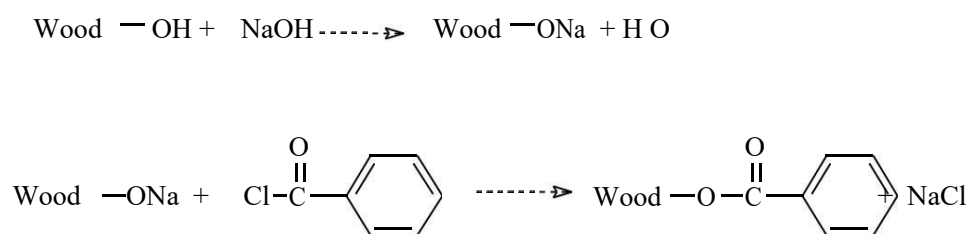


**Scheme    2.7:**    Chemical    reactions    between    EFB    fibersand Benzoyl chloride [70]

Many other researches on chemical modifications have been reported to modify wood, kenaf, sisal, flax, banana, rice husk powder etc. lignocellulosic fibers using various additives (i.e. alkali silane, potassium permanganate, sodium chlorite, and acrylic acid treatments) by various procedures [71-74].

## 2.5 Fiber-Reinforced polymer composite

Technologically, the most important composites are those in which the dispersed phase in the form of a fiber. Design goals of fiber reinforced composites often include high strength and stiffness on a weight basis. These characteristics are expressed in terms of specific modulus parameters, which correspond respectively, to the ratio of tensile strength to specific gravity and modulus of elasticity to specific gravity.

## 2.5.1 Principle of fiber reinforcement

The mechanical properties of a composite can depend on many factors as alluded to in the introduction basically; these factors include the specific properties of the matrix and the reinforcing agents, their respective volume fractions, the shape , size distribution and orientation of the reinforcing phase and the bond between the reinforcement and its matrix. Let us examine in more details the relationships between these factors and their influence on the mechanical behavior of composite materials.

## 2.5.2 Stress Strain relationship

One basic ingredient in the study of the mechanics of deformable bodies is the resistive properties of materials. These properties relate the stresses to the strains and can only be determined by experiment.

One of the simplest tests for determining mechanical properties of a material is the tensile test. In this test, a load is applied along the longitudinal axis of a circular test specimen. The applied load and the resulting elongation of the member are measured.
In many cases, the process is repeated with increased load until the desired load levels are reached or the specimen breaks. Load-deformation data obtained from tensile and/or compressive tests do not give a direct indication of the material behavior, because they depend on the specimen geometry. However, using the relationships we previously discussed, loads and deformations may be converted to stresses and strains.

$$\sigma = \frac{P}{A} \qquad\qquad \varepsilon = \frac{\delta}{L}$$

σ = normal stress on a plane perpendicular to the longitudinal axis of the specimen

P = applied load

A = original cross sectional area

ε = normal strain in the longitudinal

direction δ = change in the specimen's

gage length L = original gage length

The resulting ***stress-strain curve or diagram*** gives a direct indication of the material properties.

### 2.5.3 Composite Manufacturing Processes

There are a number of processes for composite manufacturing. Among them the following are very widely used.

(1)        Compression moulding

(2)        Extrusion moulding

(3)        Injection moulding

(4)        Hand moulding

Taking composite materials as a whole, there are many different material options to be chosen from the areas of resins, fibers and cores, all with their own unique set of properties such as strength, stiffness, toughness, heat resistance, cost, production rate etc. However, the end properties of a composite part produced from these different materials is not only a function of the individual properties of the resin matrix and fiber (and in sandwich structure the core as well), but is also a function of the way in which the materials themselves are designed into the part and also the way in which they are processed.

### 2.5.4 Application of Lignocellulosic Fiber Reinforced Thermoplastic Composites

Natural fibres are now widely used in practical applications, mostly in the packaging and automotive industries [75-85]. In the latter area, the fiber's low density can be fully exploited, resulting in important weight savings and thus reduced fuel consumption. Also, the good thermal and acoustic insulation properties, due to the presence of hollow inside the fibers, are an asset for interior parts. Some other advantages of natural fibers that promote their use in automotive components are the low abrasion on tools and favorable accident performance (less splintering during impact). Also, recyclability of the parts is a major concern nowadays in automotive industry, favoring natural fiber composites over glass fiber reinforced polymers. In this regard, mainly thermal recycling or incineration with energy recovering benefits by natural fiber composites, because of the considerable residue after burning of glass fiber reinforced parts. Some researchers [84] hold the view that natural fiber composites can be reprocessed into parts with a superior performance compared to their recycled glass fiber counterparts, because the

higher flexibility of the natural fibers in comparison with glass fibers would result in less fiber shortening during the recycling processes. However, bearing in mind the fibers vulnerability towards thermal degradation, this statement becomes rather questionable since a reforming process at elevated temperature would eventually harm the natural fibers to a significant extent. Anyhow, a thorough research regarding the quality of recycled natural fiber reinforced polymer parts is still lacking and would be necessary to give a well-founded opinion on this matter.

In spite of all the benefits mentioned above, the applications of natural fiber composites are still limited to semi-structural parts, bearing no high mechanical loads. This is because of the mediocre interface properties between the fibers and a polymer matrix, resulting in low composite performance. Other applications for flax material are particleboards, where flax chives compete with wood particles [87] and paper production [86]. These applications utilize the inferior substances of the flax stem and fiber for a functional purpose, but are examples of cheap filler materials, rather than composite reinforcements. To date, no full load bearing applications for natural fiber composites are known because of the mentioned problem of poor interaction between fibers and matrix. Therefore, pre-treatments of fiber and/or matrix are a prerequisite to obtain qualitative composite parts. This will raise of course the cost of the final product, excluding hereby applications where cost is a key issue.

## 2.6 Mechanical Characterization

Mechanical properties are the characteristics of a material that are displayed when a force is applied to the material. They usually related to the elastic and plastic behavior of the material. Important mechanical properties are hardness, reduction in area, impact, fatigue and creep strengths and wear resistance. Mechanical properties are of foremost importance in selecting materials for structural machine components.

### 2.6.1 Tensile Test

This test is widely used to indicate the strength, ductility and toughness of materials, the test consists in straining a test piece by tensile stress, generally to facture, with a view to determining one or more of the mechanical properties as defined later in this section. The cross-section of the test piece may be circular, square or rectangular. Test pieces are generally made as per standard dimensions. The test pieces are held by

suitable means, for example wedges, screwed holders, shouldered holders etc., as most convenient. It is necessary to take into account the wide variation of actual strain rate which can occur during a tensile test and which may affect the results obtained. The rate of straining during plastic deformation may be measured directly if suitable equipment is available. Some of the common machines used for tensile test are universal testing machine, Hounsfield tesometer, Instron and MTS (Material Testing System). Tensile test can measure many important mechanical properties; such as stress-strain curve, tensile strength, yield strength, elongation, reduction in area, Young's modulus, resilience and toughness

## 2.6.2 Bend Test

The bend test offers a simple, but somewhat crude method of obtaining an index of ductility. The test consists in submitting a straight test piece to plastic deformation by bending without reversing the direction of flexure during the test. The test piece is bent through a large angle or over a pin of fixed diameter, and noting whether or not cracking occurs on the outer surface of the bent piece. Sometimes the angle of bend at which the cracking starts is determined. The severity of test is varied by using different sizes of pins about which the bend is made.

The bend test may be carried out in several ways. In one of the methods, one end of the test piece may be held between two blocks and the test piece bent over one of the blocks which is rounded to the specified radius. The bending force is applied at an appropriate rate permitting free plastic flow of the material. If specified, the ends of the test piece may be brought parallel to each other at a given distance apart.

The bend test is carried out at ambient temperature, unless otherwise specified. Alter bending; the outside of the bent portion should be examined. Generally, the test piece is deemed to have passed the test, if the outer convex surface is free from cracks.

## 2.7 Structural Analysis

## 2.7.1 Fourier Transform Infrared Spectroscopy

The most important source of infra-red light for scanning the spectrum of an organic compound is Nemst glower which consists of a rod of the sintered mixture of the oxides of Zirconium, Ytterium and Erbium. The rod is electrically heated to 1500°C to produce infra-red radiations.

A rod of silicon carbide can also be electrically heated to produce infra-red radiations. To obtain monochromatic light, optical prisms or gratings can be used. For prism material, glass or quartz cannot be used since they absorb strongly through most of the infra-red region. Sodium chloride or certain alkali metal halides are commonly used as cell containers or for prism materials as there are transparent to most of the infra-red region under consideration, Sodium chloride is hygroscopic and is, therefore, protected from the condensation of moisture by working at suitable temperature. Grating gives better resolution than do prisms at high temperatures.

Light from the source is split into two beams. One of the beams is passed through the sample under examination and is called the sample beam. The other beams are called the reference beam. When the beam passed through the sample, it becomes less intense due to the absorption of certain frequencies. Now, there will be a difference in the intensities of the two beams. Let Ia be the intensity of the reference beam and I be the intensities of the beam after interaction with the sample, respectively. $A = \log (I_o/I)$

Also, transmittance T; U $T_o$

Or $A = \log (1/T)$

When the infra-red spectrum of an unknown compound is scanned a large number of information, we can get viz.

(1)        Functional groups present in the compound

(2)        The environment that influence the group frequency

(3)        Type of carbon skeleton

(4)        Type of a compound (aromatic/aliphatic)

## 2.7.2 Scanning Electron Microscope (SEM)

The optical microscope is used for small scale material characterization. As the sophistication of investigations increased, either the Transmission Electron Microscope (TEM) or the Scanning Electron Microscope (SEM) often replaces the microscope. Both of those instruments have superior resolution and depth of Because of its reasonable cost and wide range of application that it provides, the SEM is the preferred instrument used in material studies. The SEM provides the investigator with a highly magnified image of the surface of a material that is very similar to what one would expect if one could actually see the surface visually. The resolution of the SEM can approach a few nm (nanometer) and it can operate at magnifications from about IOX to 300000X. There are various applications of the SEM, such as [88]

1.        Examinations of metallographic ally prepared samples at magnifications well above the useful magnification of the optical microscope

2.        Examination of fractured surfaces and deeply etched surfaces requiring depth of field well beyond that possible by the optical microscope

3.        Evaluation of crystallographic orientation of features on a metallographic ally prepared surface

4.        Evaluation of chemical composition gradients on the surface of bulk samples over distances approaching 1 um

Schematic of a SEM is shown in Figure 2.7.2. In a scanning electron microscope, a source of electron is focused in a vacuum into a fine probe that is passed over the surface of a specimen. A diffusion or turbo molecular pump creates the vacuum while an electron gun provides the source of electrons. A series of lenses are used to de-magnify the spot of electrons on to the specimen surface as the electrons penetrate the surface a number of interactions occur that result in the emission of electrons or photons from the surface. Detectors collect the emitted (output) electrons that are used to modulate the brightness of a cathode ray tube (CRT). Every point that the electron beam strikes on the sample is mapped directly onto a corresponding point on the screen. The collective points that are displayed onto a monitor or else transmitted to a photographic plate

provide an Image of the sample. Samples used in a scanning electron microscope can be of any form as in any solid or liquid having a low vapor pressure. Electrically conductive materials can be prepared using standard metallographic polishing and etching. Non conducting materials are generally coated with a thin layer of carbon gold or gold alloy. Samples must be free from water organic cleaning solutions and remnant oil based films and must be electrically grounded to the holder. Fine samples such as powder are dispersed on an electrically conducting film.
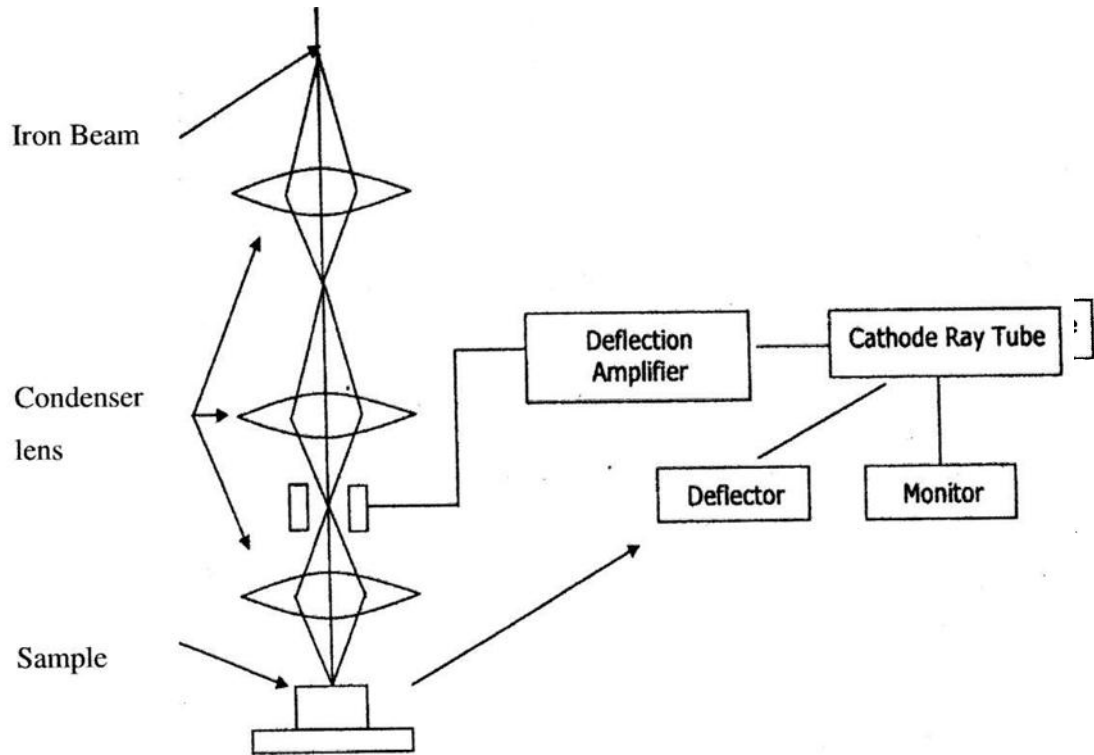


**Figure 2.7.2** Schematic of a scanning electron microscope

## 2.8 Thermal analysis

Thermal analysis includes a group of techniques where some physical property of the sample is monitored under controlled conditions with variation of temperature at a programmed rate. When the mass change is monitored the results, which indicated chemical reactions, are called Thermogravimetry (TG). When heat absorption monitored, the result indicate crystallization, phase change etc. as well as reactions. This is called Differential Thermal Analysis (DTA). Together, they are a powerful method of analysis.

## 2.8.1 Thermo gravimetric Analysis (TGA)

Thermo gravimetric analysis or thermal gravimetric analysis (TGA) is a method of thermal analysis in which changes in physical and chemical properties of materials are measured as a function of increasing temperature (with constant heating rate), or as a function of time (with constant temperature and/or constant mass loss). TGA can provide information about physical phenomena, such as second-order phase transitions, including vaporization, sublimation, absorption, adsorption, and desorption. Likewise, TGA can provide information about chemical phenomena including chemisorption's, desolvation (especially dehydration), decomposition, and solid-gas reactions (e.g., oxidation or reduction).

TGA is commonly used to determine selected characteristics of materials that exhibit either mass loss or gain due to decomposition, oxidation, or loss of volatiles (such as moisture). Common applications of TGA are (1) materials characterization through analysis of characteristic decomposition patterns, (2) studies of degradation mechanisms and reaction kinetics, (3) determination of organic content in a sample, and (4) determination of inorganic (e.g. ash) content in a sample, which may be useful for corroborating predicted material structures or simply used as a chemical analysis. It is an especially useful technique for the study of polymeric materials, including thermoplastics, thermosets, elastomers, composites, plastic films, fibers, coatings and paints.

In research and testing TGA is commonly employed to determine characteristics of materials such as polymers, to determine degradation temperature, absorbed moisture content of materials, the level of inorganic and organic components in materials.

## 2.8.3 Differential Thermo Gravimetric (DTG) Analysis

DTG stands for Differential Thermo Gravimetric analysis. It is not the weight itself rather the first derivative of the sample weight with respect to time at constant temperature or with respect to temperature at constant value of heating is determined then this procedure is termed as DTG.

# CHAPTER 3: LITERATURE SURVEY

A quite good no. of published article in this field have been reviewed. These have been reported in the previous part of this section. Some other recent works have been presented below in brief:

Siregar *et al.* Investigated and reported on effect of alkali (NaOH) treatment on the mechanical properties of pineapple leaf fiber (PALF) reinforced high impact polystyrene (HIPS) composites [89]. They obtained highest mechanical properties value for the short PALF treatment with NaOH concentration of 4%.

Ponnukrishnan *et al.* investigated on effect of the alkali (NaOH) treatment on tensile properties of Typha domingensis reinforced polyester and reported increase in the strength of composite with the increase of fiber content in the polyester matrix [90].

Tanvir sultana *et al.* worked on effect on the properties of low density polyethylene composites reinforced with treated and untreated betel nut husk fibers. They reported that alkali (NaOH) treated based composites notably gave better compatibilities than untreated fiber-based composites [91].

S. Das *et al.* [92] reported on a new process development to improve the dimensional stability of jute composite. They said that steam pretreatment process can minimize the irreversible swelling of jute fibers without any chemical modification of jute fibers.

C. M. Clemons *et al.* [93] investigated the microstructure of injection-molded polypropylene reinforced with cellulose fiber and reported that there was little fiber orientation at low fiber contents and a layered structure arosed at high fiber contents. They also reported that dynamic fracture toughness (Charpy impact test) increased with cellulose content and with orientation of fibers perpendicular to the crack direction.

K. Takemura [84] reported on the effect of water absorption on static and creep properties for jute fiber reinforced composite (JFRC). He mentioned that the static strength and stiffness for JFRC decreases with increased water absorption. He also mentioned that the effect of water absorption is greater at the secondary creep stage than that of the other creep stages.

B. V. Kokta [95] reported the effect of compounding, molding and fiber length on the properties of wood fiber reinforced thermoplastic composites. He reported that when

critical fiber length was achieved the increase of fiber length did not improve composite properties. He also reported that there was practically no difference in resulting properties due to different way of compounding.

X. Li *et al*. [96] reported on biodegradable composites prepared by polypropylene carbonate) (PPC) reinforced with short Hildegardia populifolia natural fibers. They observed the effect of alkali treated fibers comparing with untreated fibers on the composites and reported that better bonding and approximately similar mechanical properties were obtained for both alkali-treated and untreated Hildegardia populifolia fiber reinforced PPC composites. They also reported that tensile strength and tensile modulus of the composites increased dramatically with increasing fiber content but elongation at break decreased sharply with increasing fiber content.

M. J. Saad *et al.* [97] reported on the empty fruit bunch (EFB) fiber reinforced polypropylene (PP) composites. They modified the EFB fibers with malefic anhydride (MAH) before preparing the composites. They observed that MAH-treated fiber reinforced PP; composites produce higher tensile and better dimensional stability properties.

G. S. Tay *et al*. [98] investigated on the effect of chemical (epichlorohydrin) loading on the epichlorohydrin modified oil palm empty fruit bunch (EFB) fiber reinforced polyurethane composites (PU). The reported that composites made with EFB modified with EPH to 8% weight percent gain (WPG) produced higher tensile properties than the others. They expressed that higher degree of modification could cause disruption of crystalline region to the EFB.

# CHAPTER 4: EXPERIMENTAL

## 4.1 Materials

## 4.1.1 Low density polyethylene (LDPE), Polystyrene (PS) and *Typha Angustata* fiber

A commercial grade Polystyrene (PS) and low-density polyethylene (LDPE) were used in this study. Low density polyethylene was manufactured by The Polyolefin Company, Pte. Ltd., Singapore. Melting point of this LDPE was measured and found to be $115^\circ$C. In the literature it is mentioned that the melting point of commercial grade LDPE lies in the range 105-115°C.

Polystyrene (PS) is made into a foam material, called expanded polystyrene (EPS) or extruded polystyrene (XPS), which is valued for its insulating and cushioning properties. Polystyrene is made by stringing together, or polymerizing, styrene, a building-block chemical used in the manufacture of many products. Polystyrene was manufactured by Polystyrene Product Company (Pty) LTD, Randburg, South Africa. Melting point of this LDPE was measured and found to be 240°C [99].

*Typha Angustata* (TA) is a perennial plant and is an aquatic weed and is widely grown in waterlogged areas and is belongs to the family *Typhaceae* in the plant kingdom. It is abundantly available in Bangladesh as waste fibers. The *Typha angustata* fibers were collected from Barishal, Bangladesh.

## 4.1.2 Chemicals

The oxidizing agent sodium hydroxide (NaOH) and sodium meta periodate (NaIO$_4$) were used. To adjust the pH of the solution acetic acid (2%) and sulphuric acid were used. The list of chemicals used in this study and their suppliers name are given below;

| Chemical Used | Name of Suppliers |
|---|---|
| Sodium hydroxide | Loba, India |
| Acetic acid (2%) | Merck, Germany |
| Sulphuric Acid | Merck, India |

## 4.2 Surface modification

## 4.2.1 Surface modification of *Typha angustata* fibers by sodium hydroxide

*Typha angustata* fibers (TAF) were immersed in aqueous solution of sodium hydroxide (6%) (w/w) for one hour at room temperature approximately 25°C and the fibers were

stirred occasionally. Fiber to liquor ratio was 1:20 (w/v). The alkali treated Typha angustata fiber were thoroughly washed in tap water and then immersed in distilled water for 24 hrs. Finally, fibers were washed with distilled water to remove the alkali completely and the pH was measured until it was found neutral. The alkali treated Typha agustata fiber were dried in air first then further dried in an oven to get a constant weight.

## 4.2.2 Chemical treatment of *Typha angustata* fibers with sodium Meta-periodate

The dried *Typha angustata* fibers were clean manually and cut into 1 cm in length, fibers were then washed with distilled water and dried in open air. The air dried fibers were oven dried at 105°C for 5 hours. Sodium meta-periodate solution (0.06M) was prepared in distilled water. Dried fibers were immersed in sodium meta-periodate solution. Fiber to liquor ratio was 1:16 (w/v). The pH of the solution adjusted to 5 by adding sulfuric acid. The reaction was carried out for 4 hours at 70°C. The reaction mixture was stirred occasionally. The mixture was cooled and filtered to isolate oxidized fibers. The fibers were thoroughly washed in tap water and finally washed with distilled water. The washed oxidized fibers were air dried. The air dried fiber was dried in an oven at 105°C for constant weight. These oxidized fibers were used for composite fabrication.

## 4.3 Composite fabrication

Jute-polypropylene composites were prepared using raw *Typha angustata* and chemically oxidized *Typha angustata* following the procedure described below:

The raw and oxidized *Typha angustata* fibers were dried in an oven at 105°C for 6 hours. LDPE polymers were dried in oven for 3 hours at 105°C. Jute fibers were mixed thoroughly with low density polyethylene to prepare composites. LDPE matrix and jute fibers were taken in different weight fractions (Table 4.3).

| Reinforcing material (%) | Polymer Matrix (%) | Composites |
|---|---|---|
| None | LDPE: 100 | 100 wt% LDPE |
| TAF: 05 | LDPE: 95 | 05:95 wt% TAF-LDPE |
| TAF: 10 | LDPE: 90 | 10:90 wt% TAF-LDPE |
| TAF: 15 | LDPE: 85 | 15:85 wt% TAF-LDPE |
| TAF: 20 | LDPE: 80 | 20:80 wt% TAF-LDPE |
| TAF: 25 | LDPE: 75 | 25:75 wt% TAF-LDPE |
| TAF: 30 | LDPE: 70 | 30:70 wt% TAF-LDPE |
| Oxidized TAF: 05 | LDPE: 95 | 05:95 wt% Oxidized TAF-LDPE |
| Oxidized TAF: 10 | LDPE: 90 | 10:90 wt% Oxidized TAF-LDPE |

| | | |
|---|---|---|
| Oxidized TAF: 15 | LDPE: 85 | 15:85 wt% Oxidized TAF-LDPE |
| Oxidized TAF: 20 | LDPE: 80 | 20:80 wt% Oxidized TAF-LDPE |
| Oxidized TAF: 25 | LDPE: 75 | 25:75 wt% Oxidized TAF-LDPE |
| Oxidized TAF: 30 | LDPE: 70 | 30:70 wt% Oxidized TAF-LDPE |

**Table 4.3.1:** Relative amounts of reinforcing materials (TAF) and polymer matrix (LDPE) by weight fraction (%)

Polystyrene polymers were dried in oven for 3 hours at 240°C. *Typha angustata* fibers were mixed thoroughly with low density polyethylene to prepare composites. LDPE matrix *Typha angustata* and fibers were taken in different weight fractions (Table 4.4)

| Reinforcing material (%) | Polymer Matrix (%) | Composites |
|---|---|---|
| None | PS: 100 | 100 wt% PS |
| TAF: 05 | PS: 95 | 05:95 wt% TAF- PS |
| TAF: 10 | PS: 90 | 10:90 wt% TAF- PS |
| TAF: 15 | PS: 85 | 15:85 wt% TAF- PS |
| TAF: 20 | PS: 80 | 20:80 wt% TAF- PS |
| TAF: 25 | PS: 75 | 25:75 wt% TAF- PS |
| TAF: 30 | PS: 70 | 30:70 wt% TAF- PS |
| Oxidized TAF: 05 | PS: 95 | 05:95 wt% Oxidized TAF-PS |
| Oxidized TAF: 10 | PS: 90 | 10:90 wt% Oxidized TAF-PS |
| Oxidized TAF: 15 | PS: 85 | 15:85 wt% Oxidized TAF-PS |
| Oxidized TAF: 20 | PS: 80 | 20:80 wt% Oxidized TAF-PS |
| Oxidized TAF: 25 | PS: 75 | 25:75 wt% Oxidized TAF-PS |
| Oxidized TAF: 30 | PS: 70 | 30:70 wt% Oxidized TAF-PS |

**Table 4.3.2:** Relative amounts of reinforcing materials (TAF) and polymer matrix (PS) by weight fraction (%)

## 4.3.1 Preparation of composites by compression molding

## 4.3.1.1 Weber- Pressen Hydraulic Press

The compression-molding process is a very widely used method for fabrication of articles from polymer materials. Fig.4.4 shows a typical mold employed in this study for compression molding. The mold is made of two halves- the upper and the lower halves. In most cases, the lower half contains a cavity when the mold is closed. The gap between the projected upper half and the cavity in the lower one gives the shape of the molded article.

The mold used in this research work had both the upper and lower halves but had no cavity in any half in the mold. These two halves are completely plane with smooth surface plate, which also acts as heater and cooler simultaneously. In this molding process, one has to make die for casting. A die used in this study had a ring of inside diameter 146 mm and outside diameter 158 mm and have two disc (or plates) on each side, each of 7.5 mm in thickness. This die with the raw samples is placed in between the plates.

Before fabrication of composites treated and untreated betel TAF were dried in an oven at 80°C for 24 hours. LDPE and PS were granulated in a grinder and dried at 80°C for 3 hours. The LDPE and TAF, PS and TAF were dry-blended in a blender for one minute. These mixers were then moulded in a compression moulding machine (Paul-Otto Weber Press Machine) at a molding temperature of 150°C.These procedures were followed for all the composites fabricated in the ratio of composition.

Figure 4.3.1.1: Paul-Otto Weber press Machine

## 4.3.2 Experimental Procedure for Composite Preparation

The TAF-LDPE and TAF-PS composites were prepared by the following ways:

### 4.3.2.1 Mat placing
Mat placing is an important part for this experimentation, because if the TAF and LDPE matrix are not combined homogeneously, the quality of the products obtained will be poor. Considering this, TAF's were carefully placed on LDPE and PS sheets.

### 4.3.2.2 Casting
A special molding device is made by steel to very close tolerance for the molding process. The mixture of TAF and matrix is cast by simply pouring the mixture into the mold and leveling it to the desired thickness. Only slight stamping or hammering on the mold is required for sufficient compaction

### 4.3.2.3 Curing and controlling

On pouring the mat or matrix mixture into the special molding device, sufficient initial pressure of 50 kN is applied at first to get the desired shape and possible homogeneity. The applied pressure is measured by using a pressure gauge, set in the device. Heating is done electrically and the final temperature set at 105°C for PVC and 240°C for PS. Only 30–40 minutes is required to reach the temperature to 105°C for LDPE and 240°C for PS. The temperature was kept for 3 min at final pressure of 100 kN. After completion of heating the initial pressure was set zero and an additional pressure of 100 kN was applied to avoid the formation of voids. This additional pressure was kept for 10–15 minutes.

### 4.3.2.4 Cooling

Cooling is essential throughout the curing operation in order to get the ultimate composites. Cooling was done by tap water through the outer area of the heating plates of the hydraulic press machine. Cooling time was 15 minutes.

### 4.3.2.5 Demolding

When the composite was pressed in the die by the hydraulic press, the composite was obtained intact into the die through fast cooling. As a result, a demolding device is made to separate the composites from the die. Thus, a series of TAF reinforced LDPE and PS composites were prepared with and without sodium hydroxide and sodium meta per iodate treated fibers. Composite prepared by compression molding machine is given below as photograph:



**Fig 4.3.2.5** Composite prepared by compression molding

## 4.4 Characterization of treated *Typha angustata* and prepared composites

The *Typha angustata* fibers (treated and untreated) and composite materials were characterized by FY-IR spectroscopy and Scanning Electron Microscopy (SEM) are stated below:

### 4.4.1 FT-IR Spectroscopy

The infrared spectra of the PP polymer, raw jute fiber, oxidized jute fiber and -30 wt-% fiber reinforced composites were recorded on a Shimadzu FTIR-8101 spectrophotometer. The samples pellets for FT-IR spectroscopy were prepared as follows:

Approximately 0.5 mg of powdered samples was mixed thoroughly with approximately 100 mg of dried powdered potassium bromide in a small agate mortar pestle. The mixture was taken in a die of specific dimensions. Pellets were made by applying vacuum pressure. IR spectra with all information about absorbance were obtained in the printed form. IR spectra obtained this study are presented in the result and discussion section.



**Fig.4.4.1:** A photograph of an FT-IR spectrometer

### 4.4.2 Scanning electron microscopy (SEM)

The fractured surfaces of the tensile test specimens were used to examine by a scanning electron microscopy (JSM-5510, JEOL Co. Ltd., Japan). The SEM analysis was carried out the Department of Glass and Ceramic Engineering, BUET, Dhaka. The photographs are presented in the result and discussion section. Scanning electron microscopy (SEM) was used to observe the microstructure and the surface morphology of the composites [100]. Fig.4.4 shows a SEM apparatus that consists of two linked electron beam devices. It has the following main components: electron gun, condenser lens, objective system (lens, stigmator, scan coils), specimen stage and detector (secondary electron detector,

CRT screen) (Fig.4.4). In a typical SEM, electrons are thermionic ally emitted from a tungsten or lanthanum hexaboride (LaB6) cathode and are accelerated towards an anode; alternatively, electrons can be emitted via field emission.



**Figure 4.4** Photograph of the SEM instrument (model: JSM-5510, JEOL, Co. Ltd, Japan)

Tungsten is used because it has the highest melting point and lowest vapor pressure of all metals, thereby allowing it to be heated for electron emission. The electron beam, which typically has an energy ranging from a few hundred eV to 100 keV, is focused by one or two condenser lenses into a beam with a very fine focal spot sized 1 nm to 5 nm. The beam passes through pairs of scanning coils in the objective lens, which deflect the beam horizontally and vertically so that it scans in a raster fashion over a rectangular area of the sample surface. When the primary electron beam interacts with the sample, the electrons lose energy by repeated scattering and absorption within a teardrop-shaped volume of the specimen known as the interaction volume, which extends from less than 100 nm to around 5 μm into the surface. The size of the interaction volume depends on the beam accelerating voltage, the atomic number of the specimen and the specimen's density. The energy exchange between the electron beam and the sample results in the emission of electrons and electromagnetic radiation which can be detected to produce an image.

Electron Gun

Condenser Lens

Objective Lens

Stigmator

Scan Coils

Aperture

Secondary Electron Detector

Conducting Sample
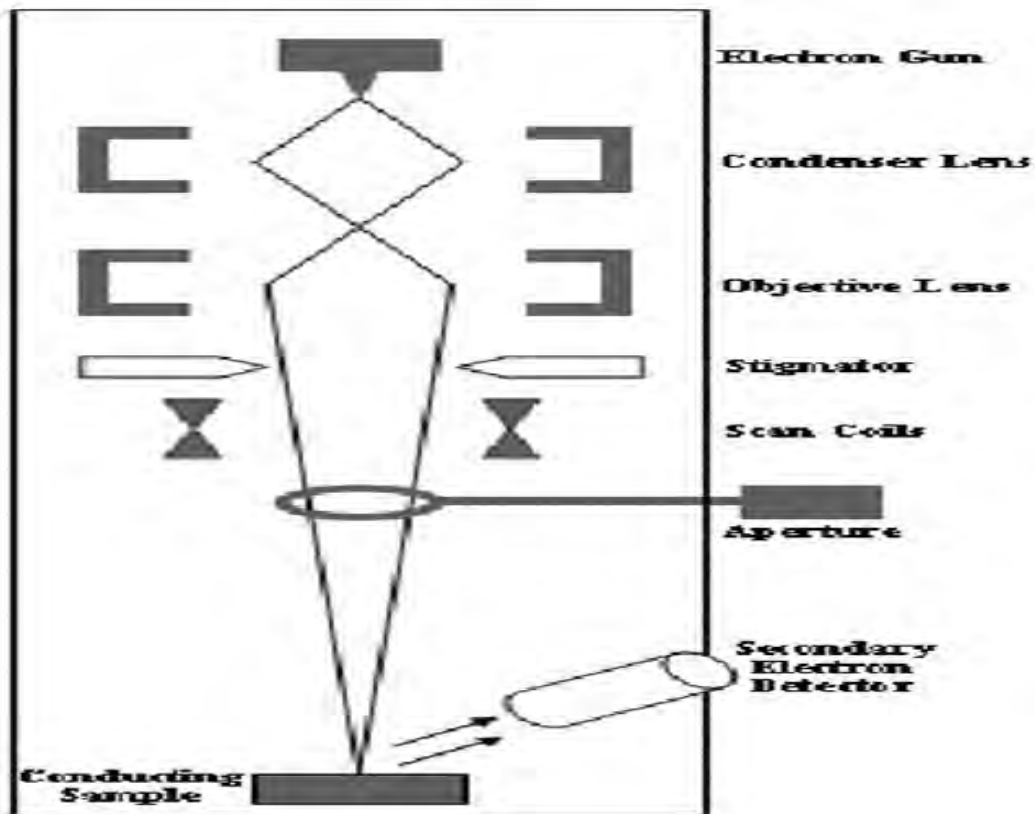
## 4.5 Mechanical properties of the composites

In order to investigate the mechanical properties of the prepared composites the following tests were carried out; (a) tensile (b) three-point flexural (c) charpy impact and (d) hardness. For these tests the appropriate ASTM methods were followed. Specification and picture of the used machines, set-up of the tests, operating conditions and dimension of the test specimens are described below.

## 4.5.1 Tensile test

All the treated and untreated betel nut husk fiber-LDPE composites were used to measure the tensile properties of the composites using a universal tensile testing machine, model: 1410 Titans, capacity: 5 kN, England. Tensile tests were conducted following ASTM D 3039/D 3039 M-00 (2002) standard method and the cross-head speed of the test was 10 mm/min. Each test of the specimen was performed until tensile failure occurred except 100% LDPE composite. Six to ten specimens of each composition were tested and the average values were reported by calculating maximum five values [101] the load vs elongation curves were obtained by the electronic chart recorder of the instrument. The maximum (Peak) load values were also recorded by the instrument, which can be recalled after the completion of the test. The highest load in the tensile test gives the tensile or ultimate strength. The tensile strength $(Qu_r)$ is calculated from the following equation [102]:

Tensile strength, $GUT = W / A_T$

Where W is breaking load and $A_T$ is the cross-sectional area.

Tensile stress and strain values were calculated from the load vs elongation curves. Tensile modulus was determined from the initial slope of the stress-strain curve and then the tensile stress vs strain curves were drawn
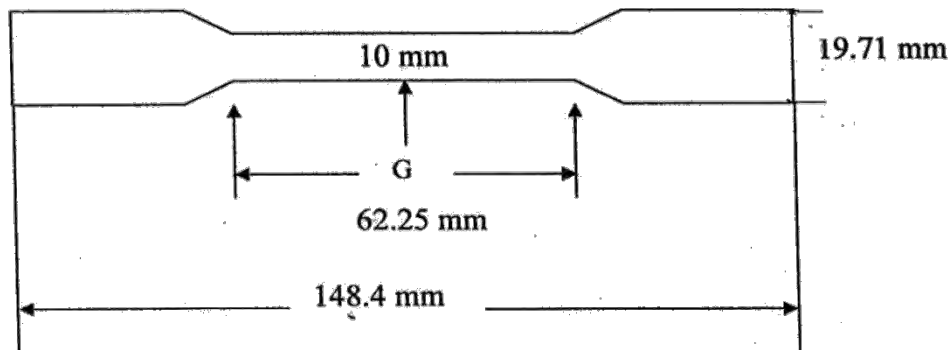


**Figure 4.5.1: Dimension of tensile test specimen**



**Figure 4.5.1.1:** Ultimate Tensile Machine (UTM)

## 4.5.2 Three-point flexural test

The static flexural tests of the composites were carried out by same machine that was used for tensile test only by changing the attachment. Set-up of the three-point flexural test and dimension of the specimen are shown in Fig.4.5.3. Dimension of flexural test specimen is 79 mm length, 10 mm width and 4.1 mm thickness. Flexural tests were conducted following ASTM D 790-00 [103] at a cross head speed of 10 mm/min. Injection moulded test specimens (Fig. 4.5.2) were used directly to perform the flexural

testing operation and the operating conditions were similar to that of the tensile test. Five specimens of each composition were tested and the average values were reported. The load displacement curves were obtained from the electronic chart recorder and the maximum (peak) load values were also recorded by the instrument, which can be recalled after the completion of the test. The flexural strength (QfM) and modulus of elasticity (EB) were calculated by the following equation S [103]:

Flexural strength, QfM = 3 PL / 2bd2
Where,

P=. Maximum load on the load-deflection curve, N L= Support span, 63.7 mm

b= width of beam tested, 10 mm and

d = Thickness of beam tested, 4.1 mm

Modulus of elasticity, EB = Lam / 4 bd3
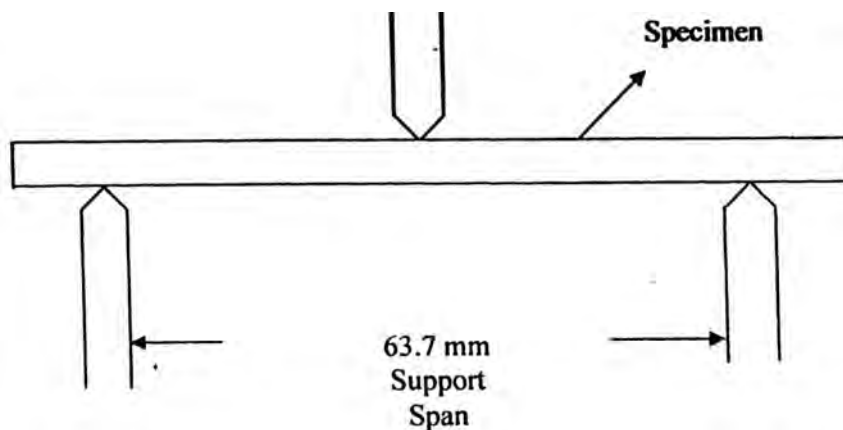


**Figure 4.5.2.1:** Set-up of three point flexural test and dimension of specimen.

Where,

L= Support span, 63.7 mm

b= width of beam tested, 10 mm and

d = Thickness of beam tested, 4.1 mm

**Figure 4.5.2.2:** Tensile and flexural test specimen

## 4.6 Water absorption test of composites

In order to measure the water absorption of the composites flexural tested rectangular specimens were cut from each sample having dimensions of 39 mm x 10 mm x 4.1mm. The samples were dried in an oven at $105^{\circ}$C for 2 h, cooled in a desiccator using silica gel and immediately weighed to the variation of 0.0001 g. Denver instron balance was used for this test. The dried and weighed samples were immersed in distilled water for about 24 h at room temperature and in boiling water for 2 hours as described in ASTM procedure D570-99 (ASTM 2002) [104]. Excess %, water on the surface of the samples was removed by using a soft cloth. Then the weights of the samples were taken. Three replicate specimens were tested and the results were presented as average of the tested specimens. The percentage increase in weight during immersion was calculated as follows [104]:

$$\text{Water absorption (\%)} = \frac{\text{Final Weight} - \text{Original Weight}}{\text{Original Weight}} \times 100$$

# CHAPTER 5: RESULTS AND DISCUSSIONS

In this work, different types *Typha angustata* fiber reinforced polystyrene (PS) composites, low density polyethylene (LDPE) composites (5-30% *Typha angustata*, by weight) have been prepared and their physico-mechanical properties have been investigated. Because of the polar constituents in *Typha angustata* fiber, *Typha Angustata fiber* shows hydrophilic nature, *Typha angustata* fiber and PS composite, *Typha angustata* and LDPE composite are incompatible. Sodium hydroxide and sodium meta per iodate treated oxidized *Typha angustata* fiber have been used to improve *Typha angustata* fiber properties such as dimensional stability or biological resistance. Raw and oxidized *Typha angustata* fibers were used separately to prepare *Typha angustata* fiber polystyrene composites and *Typha angustata* fiber and low density polyethylene composite by using a single screw extruder. To study the mechanical properties, sample specimens of specific size and shape have been prepared by using an injection moulding machine. Tensile strength, tensile modulus, elongation at break, flexural strength, flexural modulus and charpy impact strength of the prepared composites were measured by using Universal Testing Machine (UTM). The prepared composite samples were studied following the same procedures mentioned above. The results are presented in the following sections.

## 5.1 Chemical constituent of *Typha angustata* fiber

*Typha angustata* fiber (TAF) contain cellulose, hemi-cellulose, lignin other constituent in small amount. TAF was extracted and analyzed. The ingredients found in TAF are presented in table-5.1

**Table 5.1: Main chemical constituents of *Typha angustata* fiber**

| 1. Aqueous Extract | 0.5647% |
|---|---|
| 2. Fatty and waxy maters | 2.3796% |
| 3. Pectic maters | 2.91731% |
| 4. Lignin | 15.8653% |
| 5. α-cellulose | 46.0776% |
| 6. Hemi cellulose | 29.1538% |
| 7. Ash | 2.6877% |

## 5.2 FT-IR spectroscopic analysis of *Typha angustata* fiber

FT-IR spectra of raw *Typha angustata*, chemically oxidized *Typha angustata* fiber were taken in an Infra-red Spectrometer (Frontier, PerkinElmer, USA). The spectra and their brief description are presented below:

### 5.2.1 FT-IR spectroscopic analysis of *Typha angustata* fiber

*Typha angustata* fiber is itself a composite material. It mainly consists of cellulose, hemicellulose, and lignin fats etc. So the absorption peaks for different functional groups present in these compounds may shift from their right positions in IR spectra. That is why definite information about different constituents is expected to be obtained from the spectra of raw and chemically treated *Typha angustata* fiber. However, some short change in spectra of *Typha angustata* fiber may be obtained due to chemical treatment on it. From the both FT-IR spectrum it was observed that the absorption band at $1729.22 \text{cm}^{-1}$ of untreated *Typha angustata* fiber was completely disappeared in FT-IR spectrum of alkali treated *Typha angustata fiber*. It indicated that alkali treatment of *Typha angustata* fiber removed the impurities from its molecular structure completely.

The FT-IR spectrum of raw *Typha angustata* fiber is shown in Fig. 5.2.1. The peak assignments of the absorption bands corresponding to various groups are summarized in Table 5.2.1.

**Table 5.2.1: FT-IR spectral data of untreated raw *Typha angustata* fibers [101]**

| Position of bands ($\text{cm}^{-1}$) | Peak assignments |
|---|---|
| ~ 3337.51 | -OH stretching from the cellulose, hemicellulose and lignin |
| ~ 2919.15 | C-H vibration in aromatic ring and alkane |
| ~1729.22 | C=O stretching from the lignin and hemi-cellulose |
| ~ 1604.04 | C=C stretching from aromatic in-plane |
| ~ 1515.66 | C=C stretching in aromatic skeletal ring due to lignin |
| ~ 1423.94 | C-C stretching from aromatic ring |
| ~ 1032.72 | C-O stretching from the lignin |
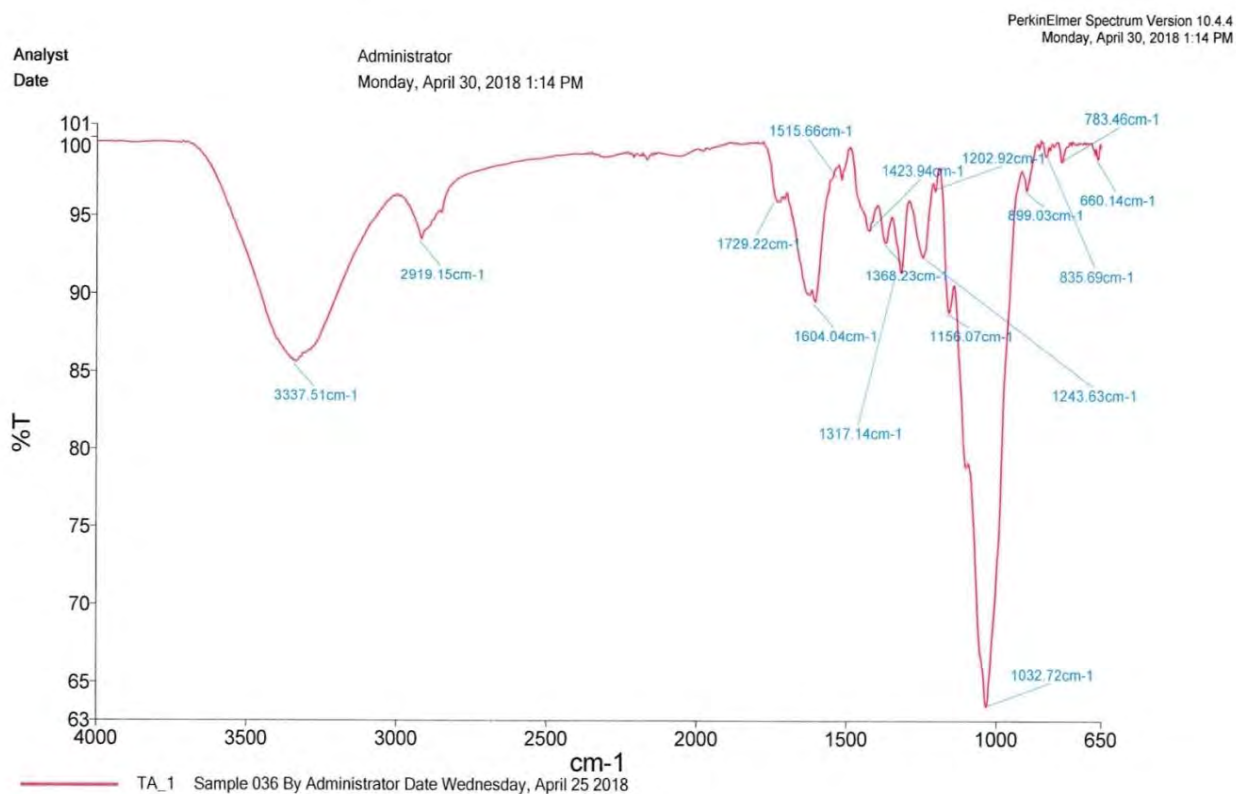
**Figure 5.2.1:** Infrared spectrum of untreated *Typha angustata* fiber

**Table 5.2.2: FT-IR spectral data of sodium hydroxide treated *Typha angustata* fiber [101]**

| Position of bands (cm$^{-1}$) | Peak assignments |
|---|---|
| ~ 3337.03 | -OH stretching from the cellulose, hemi-cellulose and lignin |
| ~ 2918.69 | C-H vibration in aromatic ring and alkane |
| ~ 1606.94 | C=C stretching from aromatic in-plane |
| ~ 1504.75 | C=C stretching in aromatic skeletal ring due to lignin |
| ~ 1422.18 | C-C stretching from aromatic ring |
| ~ 1029.09 | C-O stretching from the lignin |
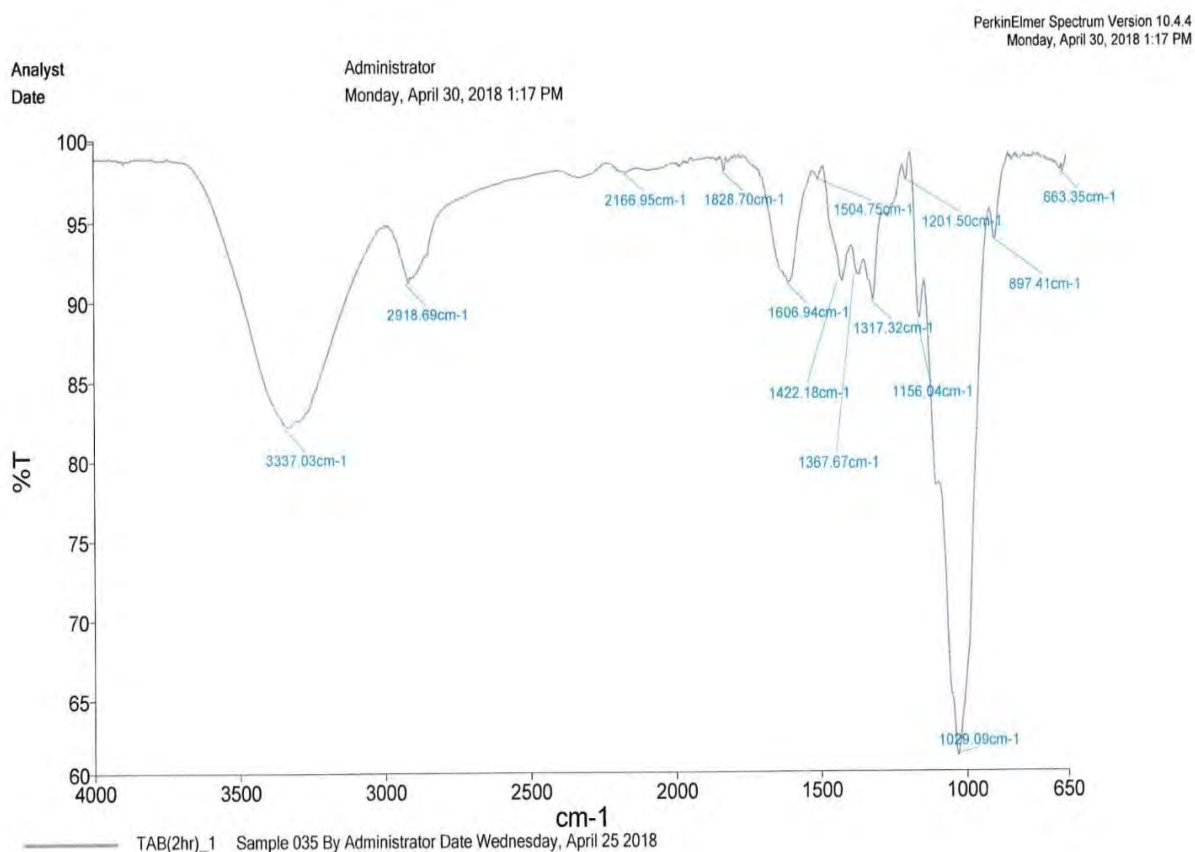
**Figure 5.2.2:** Infrared spectrum of sodium hydroxide treated *Typha angustata* fiber

## 5.3 Surface modification of *Typha angustata* fibers by oxidation reaction

Unbleached raw jute fibers were taken to modify its surface by sodium hydroxide and sodium meta periodate oxidation process to make it more active to the coupling agent.

### 5.3.1 Reaction of cellulose in raw *Typha angustata* fiber with sodium meta per iodate

The reaction of sodium meta per iodate with cellulose in raw *Typha angustata* fibers at 70°C yielded the oxidized product 2,3-dialdehyde cellulose (TAF), as illustrated in Scheme 1. Depending on the type of oxidizing agent and the pH of the reaction medium cellulose (90%) of cotton is oxidized in two products[6]. One is the reducing oxycellulose in which hydroxyl groups have been converted to carbonyl groups or aldehydes and the other is the acidic oxycellulose in which the hydroxyl groups have been converted to carboxyl groups or acids. The oxidation product is dependent on the class of oxidizing agent and the pH of the reaction medium. Three hydroxyl groups are there on cellulose anhydroglucose unit, one is a primary hydroxyl group (at C-6) and other two are secondary hydroxyl groups (at C-2 and C-3). Though primary alcohols are more reactive than secondary alcohol but periodic acid and per iodate salts break the anhydroglucose ring between carbon atoms 2 and 3 and convert the two secondary hydroxyl groups to aldehyde groups

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[105]. On the basis of this fact we have selected the sodium periodate as oxidizing agent to oxidize cellulose (69%) of raw jute to dialdehyde cellulose. The detail of the reaction has been described in the experimental section.

**Cellulose in TAF**

$H^+$ | $70^0$ C, 5h

**Dialdehyde cellulose in TAF**
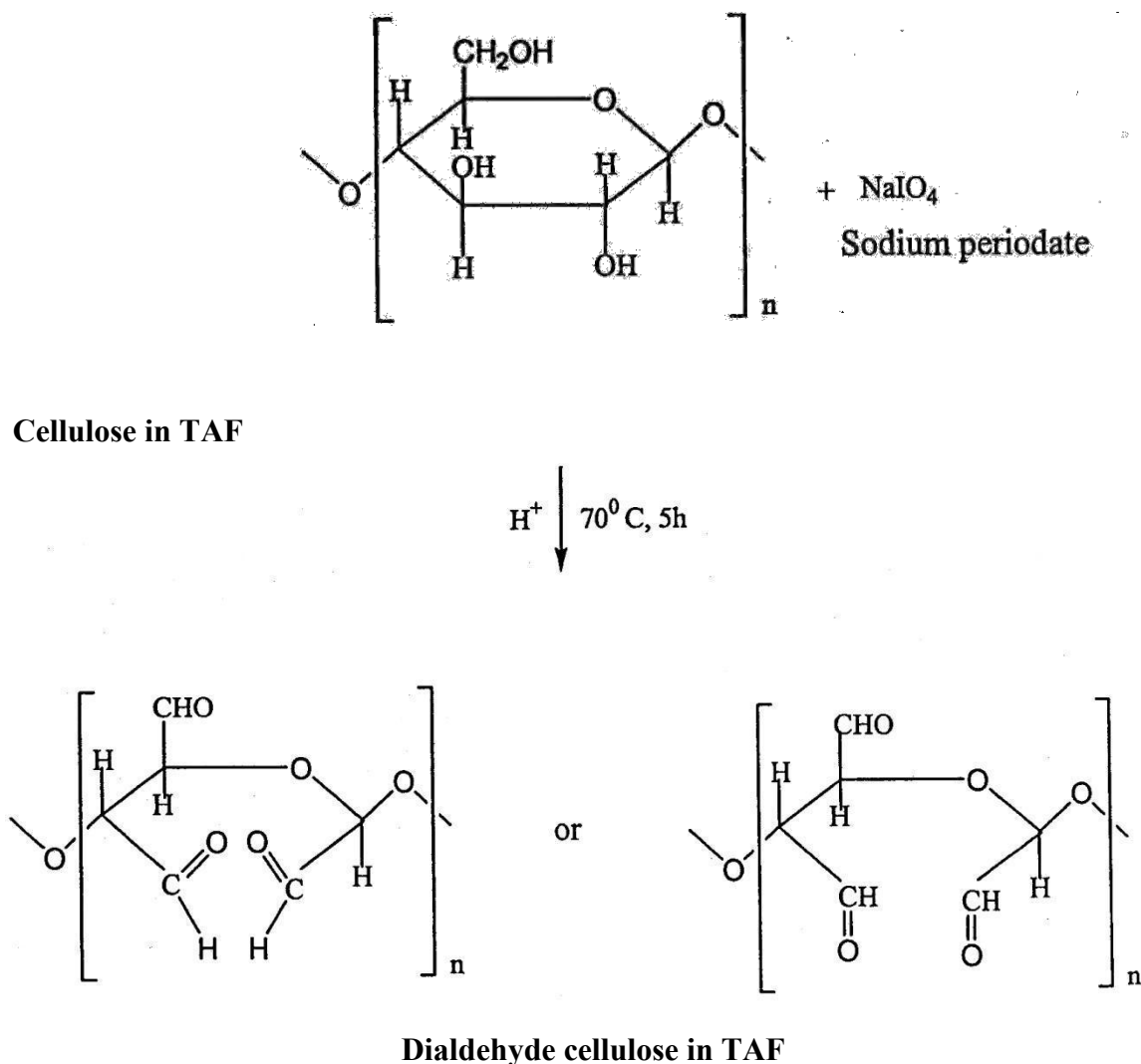
**Scheme 5.3.1:** Oxidation reaction of cellulose in *Typha angustata* with sodium meta periodate

## 5.3.2 FTIR spectroscopic characterization of untreated and alkali treated *Typha angustata* fiber

The characteristic bands of untreated and alkali treated TAF are shown in figure 5.1 and 5.2 respectively. It is cleared from the figures that the absorption band at 1729.22 cm-1 of untreated TAF has disappeared completely in the treated TAF. This indicates that the alkali treatment of *Typha angustata* fibers had removed the impurities, hemicelluloses and part of lignin. The similar FT-IR data was also reported by Tanvir sultana et al in case of alkali treated *Typha angustata* fibers.

## 5.3.3 Interface of untreated *Typha angustata* fiber – polystyrene composite and treated *Typha angustata* fiber-polystyrene composite

A possible hypothetical model of interface of untreated *Typha angustata* fiber surface having hydroxyl groups with the polystyrene matrix and treated *Typha angustata* fiber surface having reactive (- ONa) groups with polystyrene matrix  is shown in Figure 5.3.3.1 and Figure 5.3.3.2.
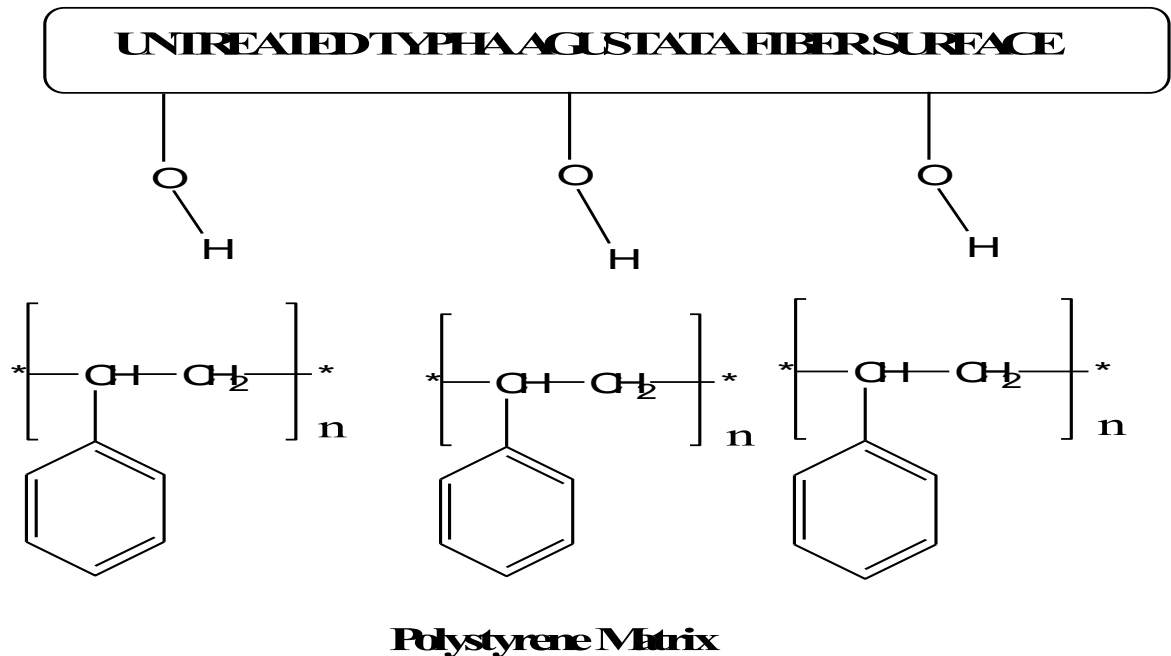


**Figure 5.3.3.1** hypothetical model of untreated *Typha angustata* fiber- polystyrene composite.
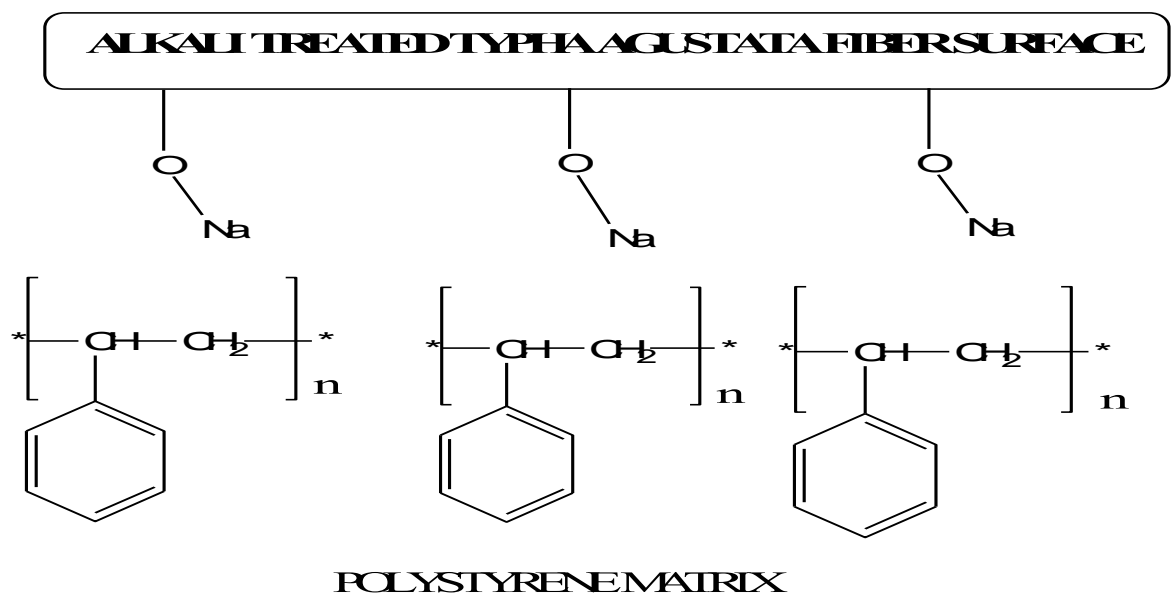


**Figure 5.3.3.2** hypothetical model of treated *Typha angustata* fiber – polystyrene composite.

## 5.4 Morphological (SEM) characterization of raw and oxidized *Typha angustata* fiber reinforced polystyrene composites

The scanning electron microscopy (SEM) images of untreated and treated *Typha angustata* fiber-polystyrene composites are shown in figure 5.4.1, 5.4.2, 5.4.3, 5.4.4, 5.4.5, 5.4.6 and 5.4.7 respectively. From the figure 5.4.3, The SEM image of untreated *Typha angustata* fiber showed big voids and rough surface among the fibers. On the other hand from the figure 5.4.4 sodium meta per iodate treated *Typha angustata* fiber showed almost voids free smooth surface. From the figure 5.4.1 and 5.4.5 it was observed that the SEM image of 20 wt. % untreated TAF-PS composites and 25 wt. % TAF-LDPE composites showed big voids or gap between fibers and matrices. That is why it shows weak uniform dispersion and incompatibility between fibers and matrices. On the other hand from the figure 5.4.2, 5.4.6 and 5.4.7 it was observed that the SEM image of 20 wt. % alkali treated TAF-PS composites and 25 wt. % sodium meta per iodate treated TAF-LDPE composites showed no voids between fibers and matrices. That is why it shows strong uniform dispersion and better compatibility between fibers and matrices. So it may be concluded that alkali treated and sodium meta per iodate treated TA fibers are able to improve the compatibility between the fiber and matrix in the composites.
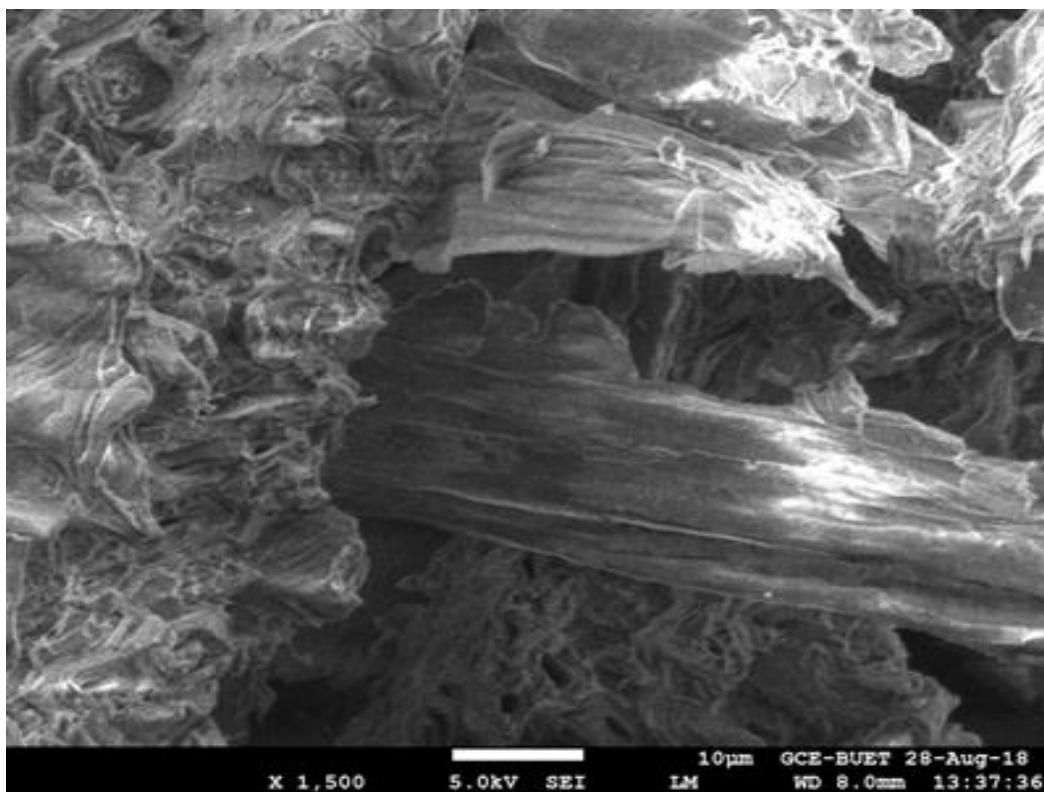


**Figure 5.4.1** SEM image of untreated 20 wt. % TAF - Polystyrene composite.
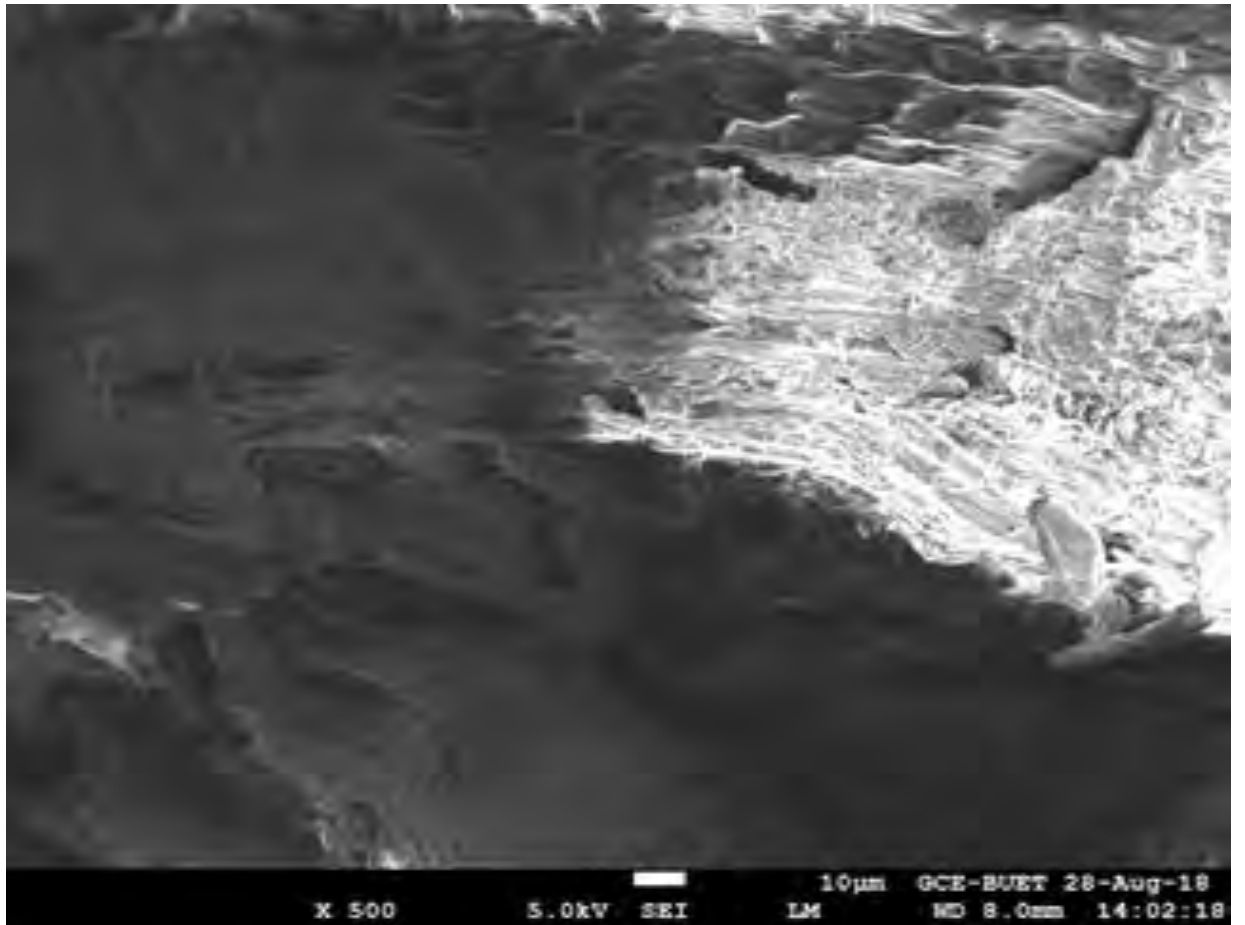
**Figure 5.4.2** SEM image of alkali treated 20 wt. % TAF - Polystyrene composites
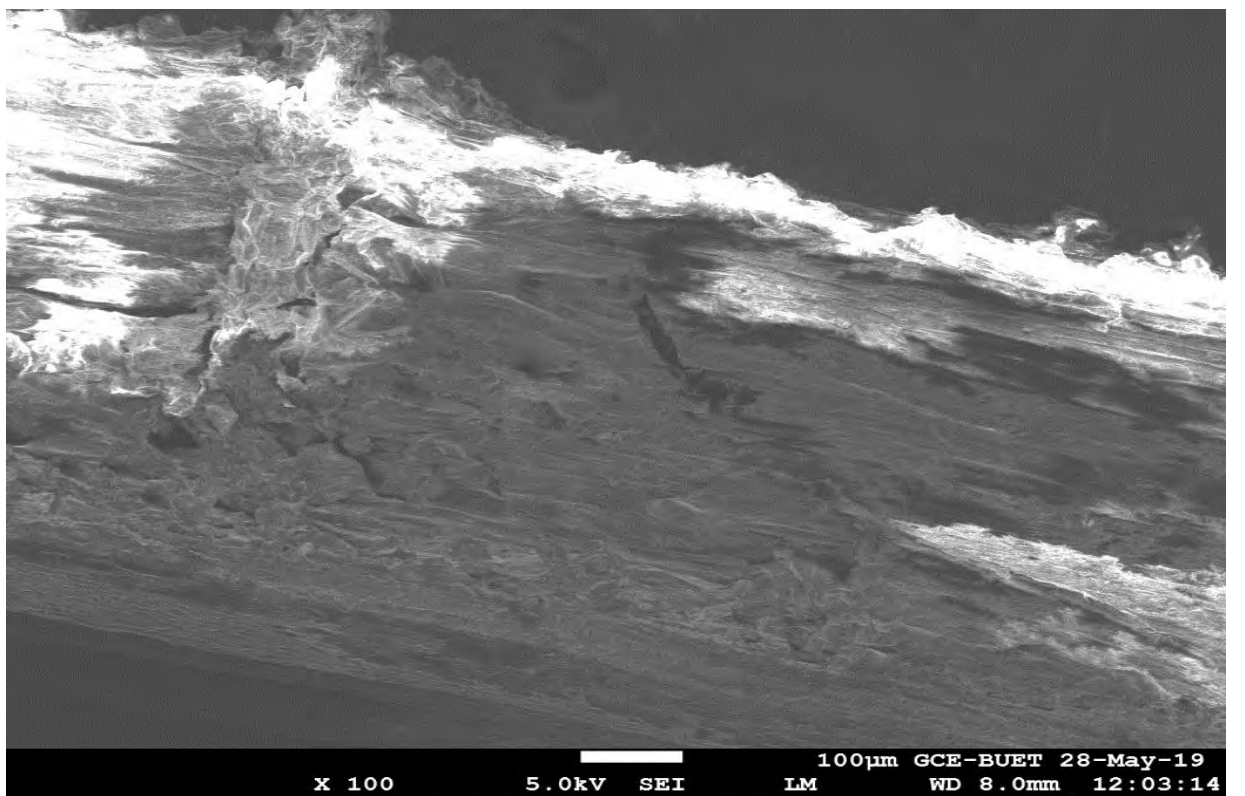


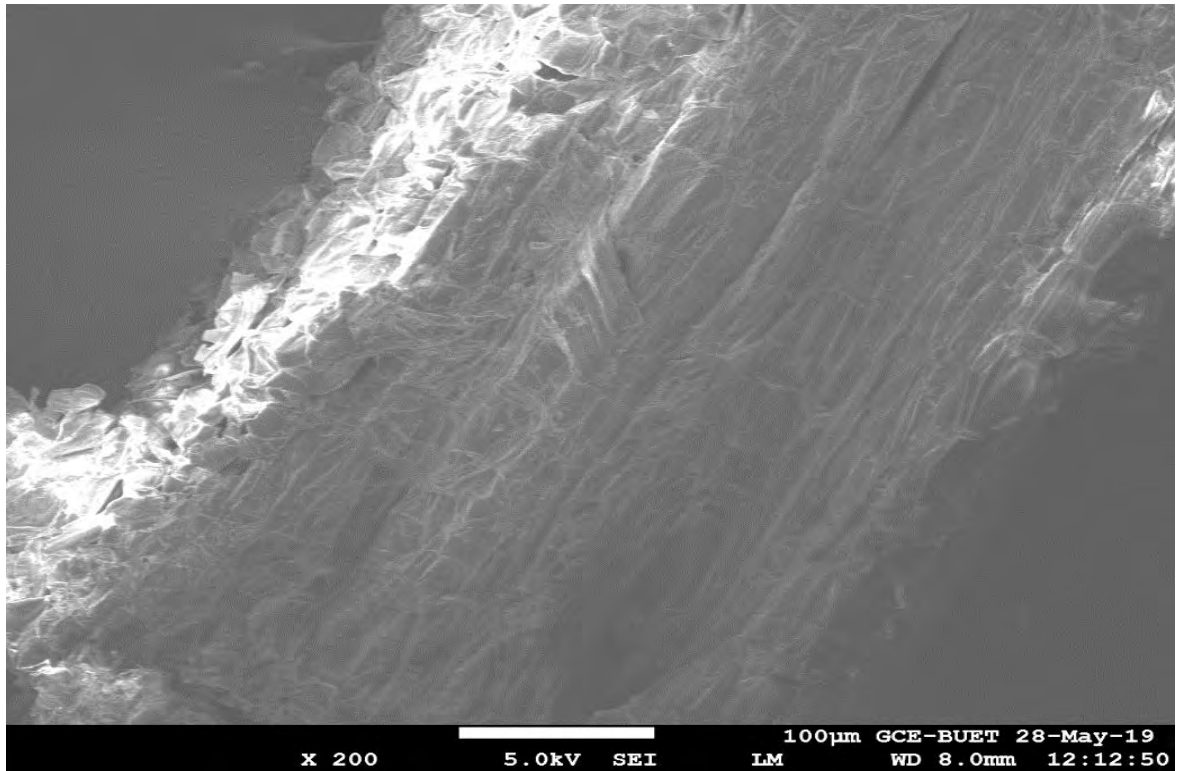**Figure 5.4.3** SEM image of untreated *Typha angustata* fiber

**Figure 5.4.4** SEM image of Meta per iodate treated *Typha angustata* fiber
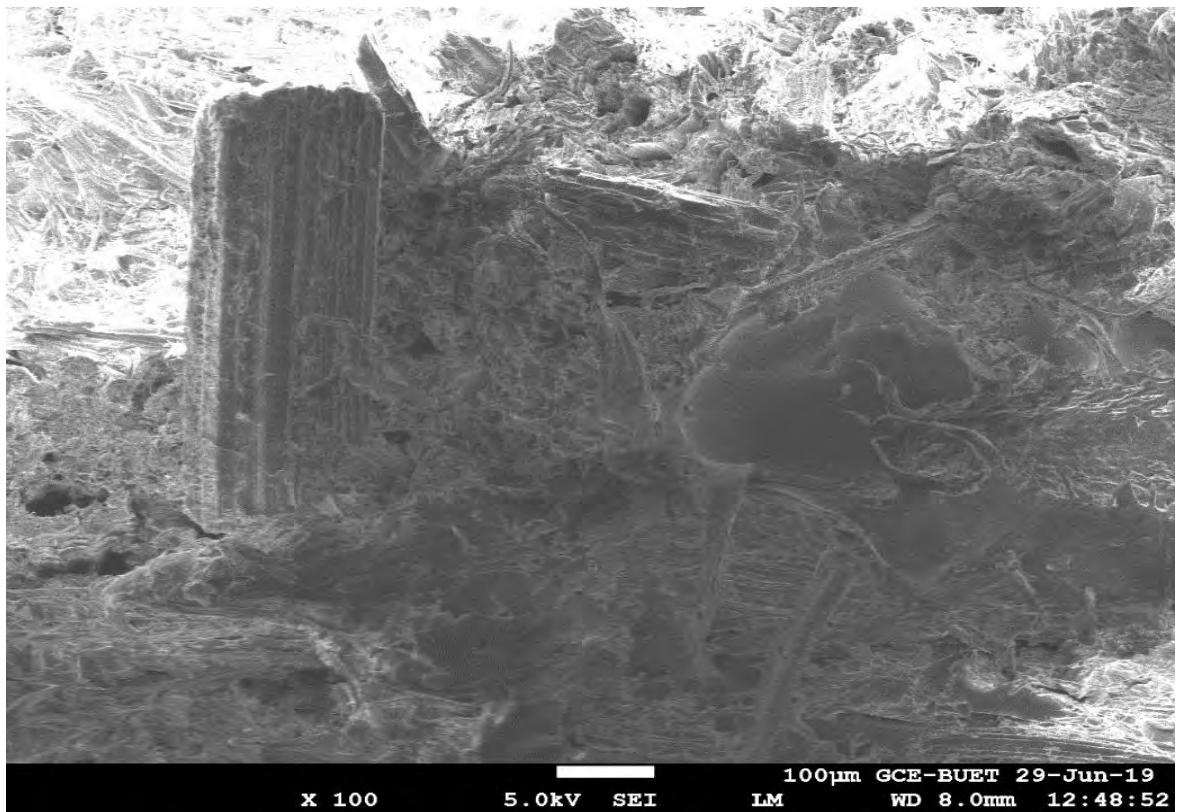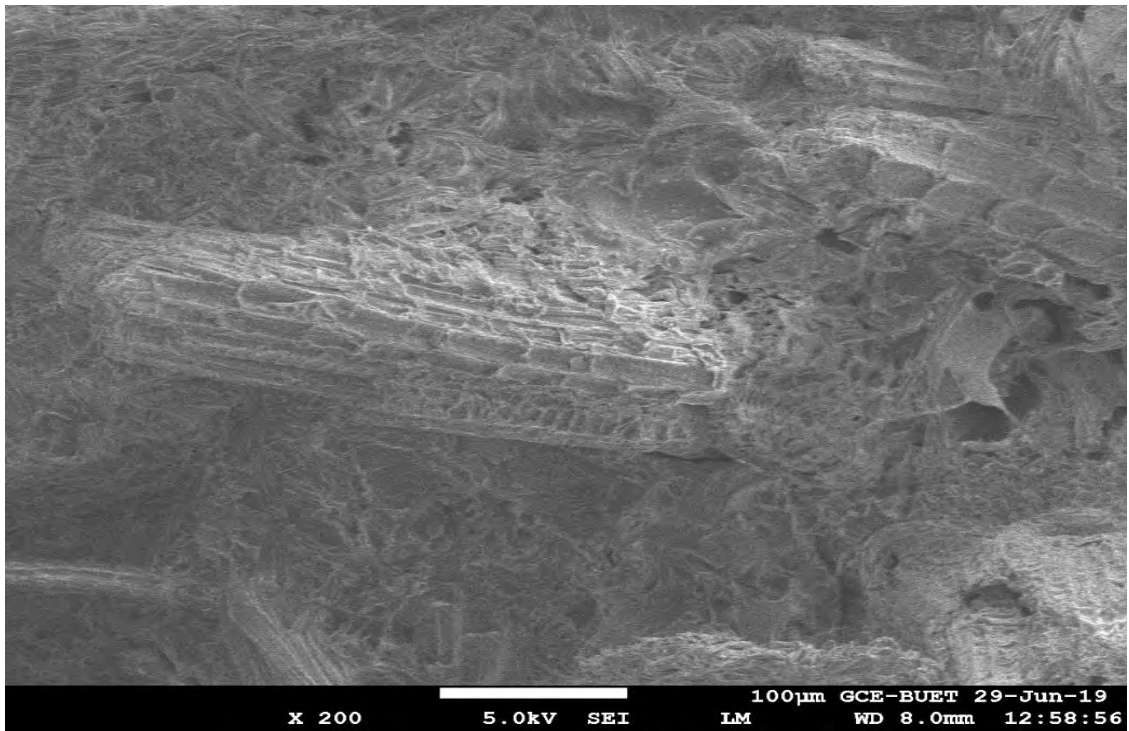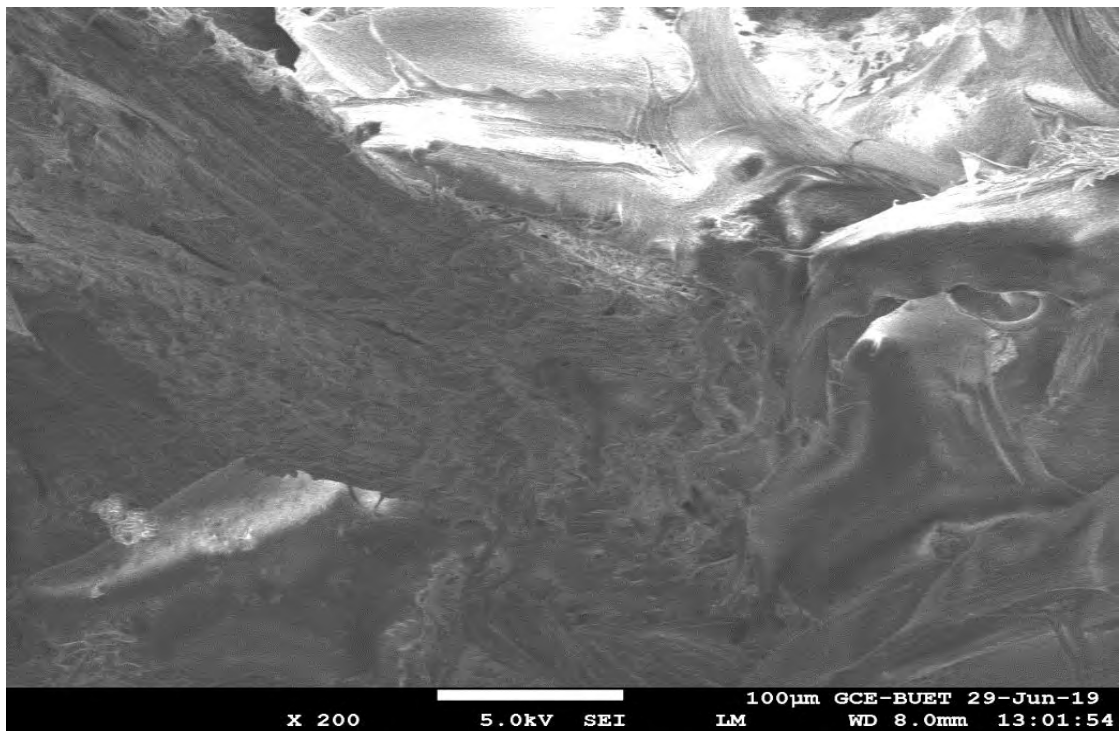


**Figure 5.4.5** SEM image of untreated 25 wt. % TAF - LDPE composites

**Figure 5.4.6** SEM image of alkali treated 25 wt. % TAF - LDPE composites



**Figure 5.4.7** SEM image of Meta per-iodate treated 25 wt. % TAF - LDPE composites

**5.5 Raw and oxidized *Typha angustata* fiber reinforced polystyrene (PS) composites**

As mentioned earlier *Typha angustata* reinforced polystyrene composites were prepared both by using raw and oxidized *Typha angustata* fiber. The composites were characterized by IR-spectroscopic method.

**5.5.1 Mechanical properties of raw and oxidized *Typha angustata* fiber reinforced polystyrene composites**
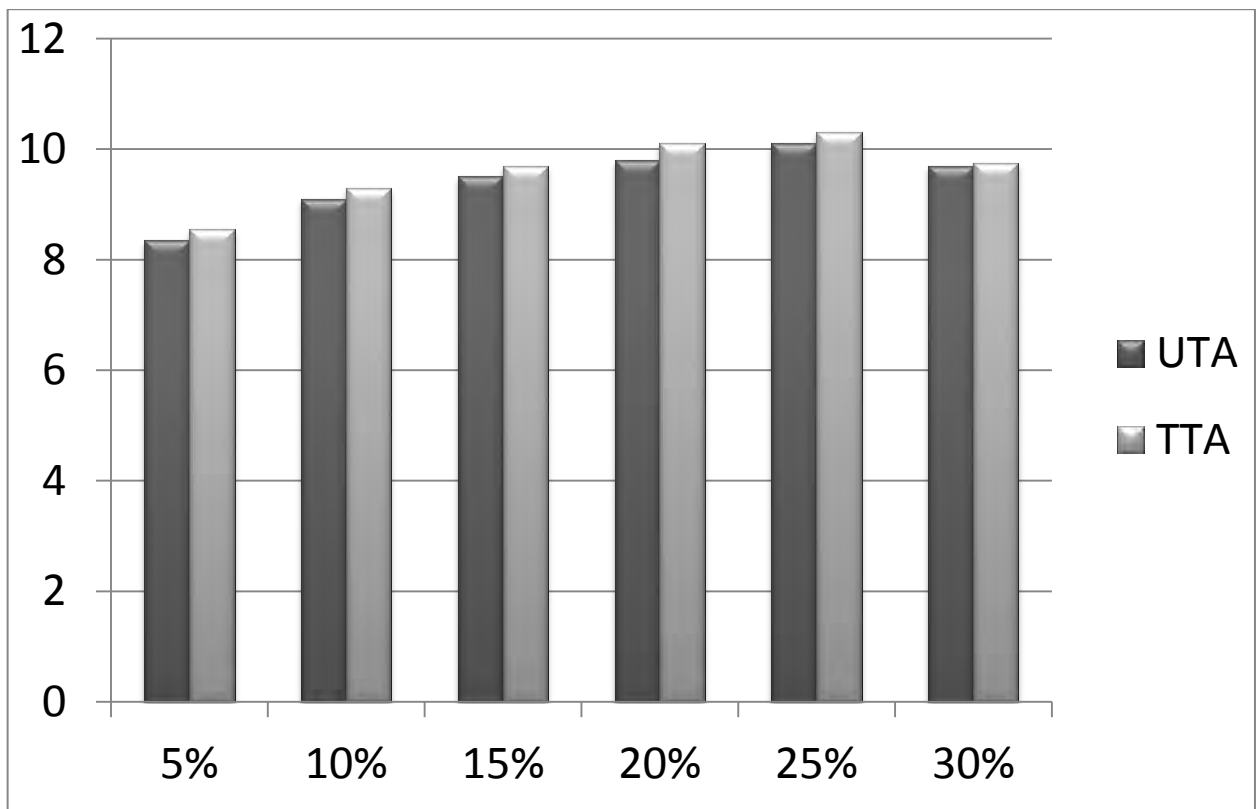
Tensile strength, tensile modulus, elongation at break, flexural strength, flexural modulus of the raw and oxidized *Typha angustata* fiber reinforced polystyrene composites have been determined following the ASTM method described in the experimental sections. The results obtained in this study are presented below.

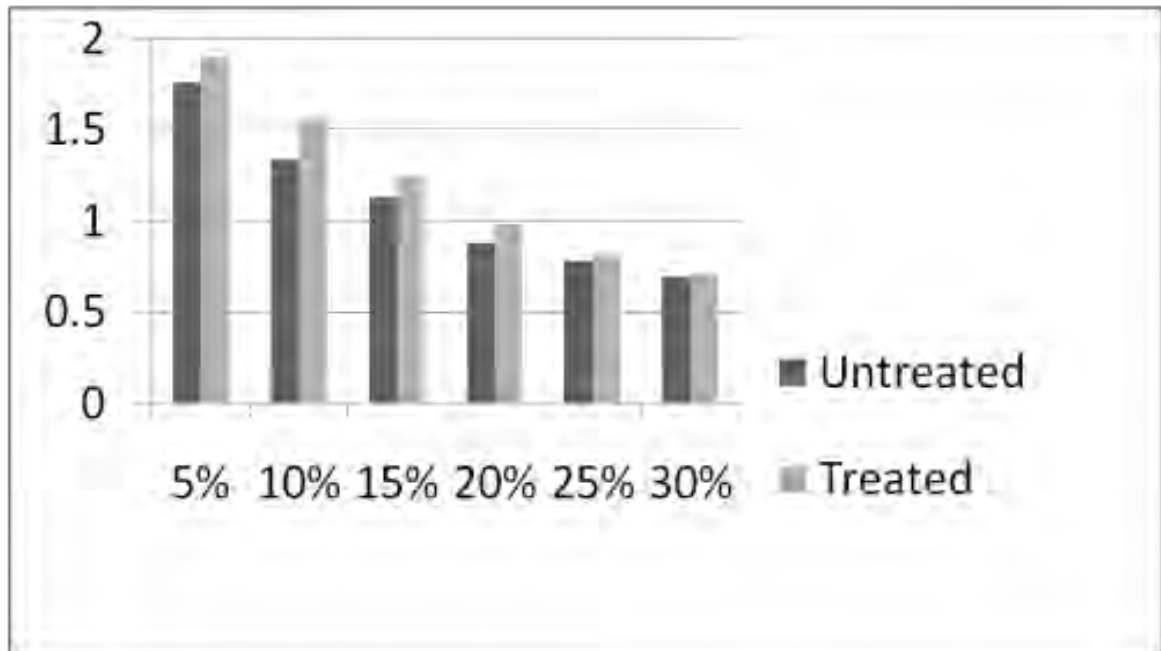**5.5.2 Tensile strength and elongation at break**

With increasing *Typha angustata* fiber loading by weight fraction from 5% (w/w) to 30% (w/w), the tensile strength of the raw and oxidized *Typha angustata* fiber reinforced polystyrene and low-density polyethylene composites are increased Fig: 5.5.2.1. This reduction is possibly due to the lack of stress transfer from the LDPE and polystyrene matrix to *Typha angustata* fibers. Approximately similar results were found for kenaf-PP composites [40] and empty fruit bunch (EFB) fibers-PP omposites[38] in the literature. Tensile strength and tensile modulus of 100 wt% LDPE are 9.30 MPa and 0.14 GPa respectively. The tensile modulus increases with increasing fiber loading (Fig: 5.5.2.2) as compared to 100 wt% LDPE. The higher tensile modulus is found for all oxidized *Typha angustata*-LDPE and *Typha angustata*-PS composites with compared to raw *Typha angustata* – LDPE composites. So stiffness of the oxidized *Typha angustata* –LDPE and *Typha angustata*-PS are higher than raw *Typha angustata*-LDPE and *Typha angustata*- PS composites. Elongation at break decreases with increasing fiber loading (Fig: 5.5.2.3). It is also found that elongation at break of oxidized *Typha angustata*-LDPE and *Typha angustata*- PS composites is higher than that of untreated raw *Typha angustata* – LDPE and *Typha angustata* – PS composites. The tensile strengths of the untreated and treated TAF-Polystyrene composites are shown in figure 5.5.2.1. From the figure it is cleared that the tensile strengths of the composites increased up to 20 wt. % fibers loading and then decreased. The elongations at break of untreated and treated TAF-Polystyrene composites are shown in figure 5.5.2.2. Elongation at break of all composites is decreased with increasing of fiber loading. Tensile properties of all treated TAF-Polystyrene composites are higher than that of untreated TAF-Polystyrene composites. This may be the reason for the better improvement of the fiber-matrix interfacial adhesion in composites which was made by alkali treatment.
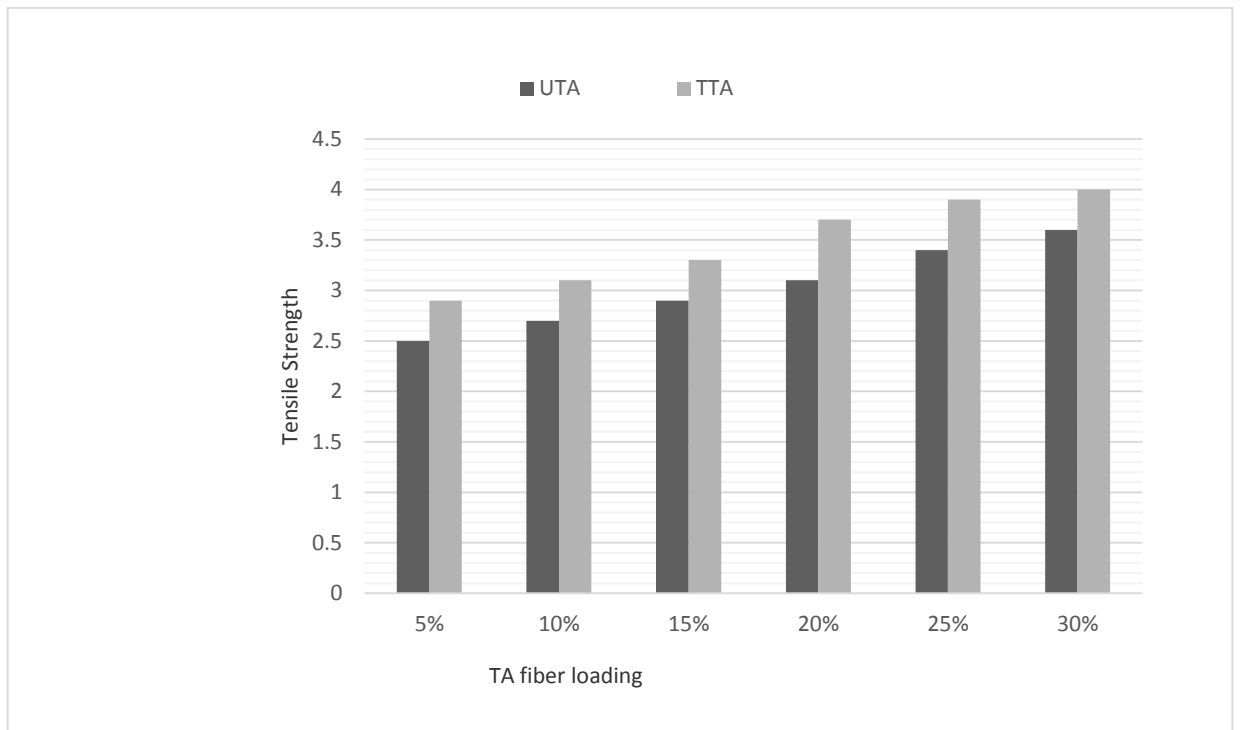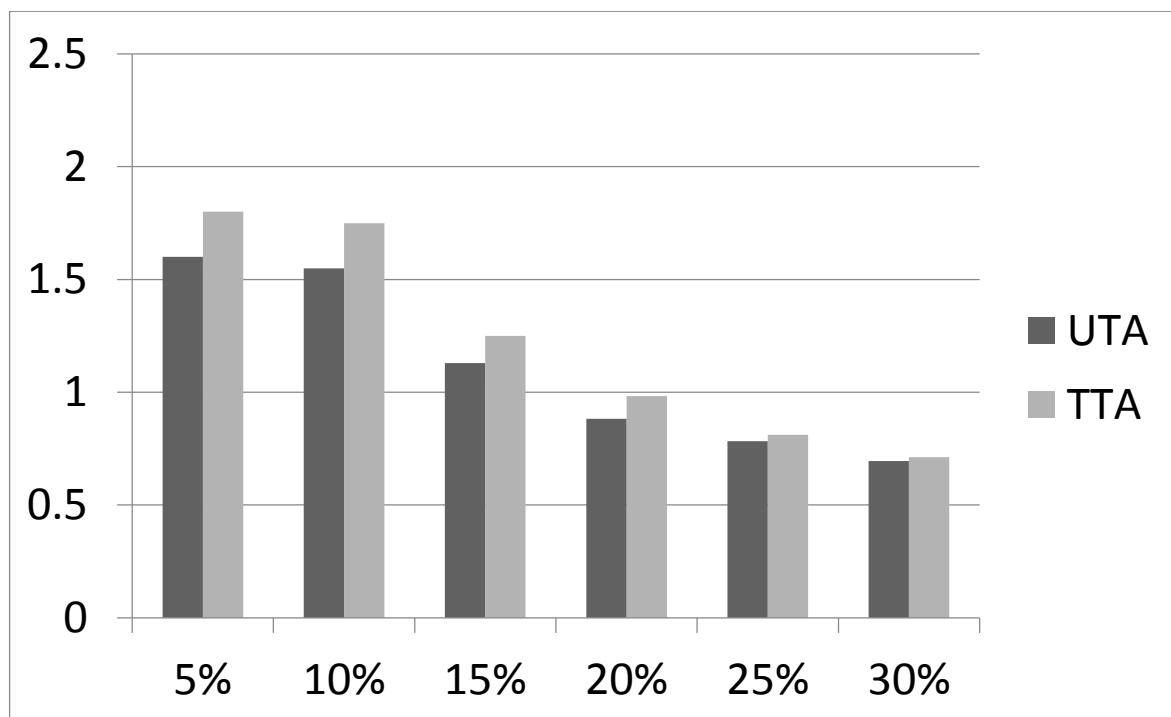
**Figure 5.5.2.1.** Tensile strength (Mpa) vs fiber Loading (wt %) of TAF-PS composites



**Figure 5.5.2.2.** Tensile strength (Mpa) vs fiber Loading (wt %) of TAF-LDPE composites

**Figure 5.5.2.3:** Elongation at break vs fiber loading (wt. %) of TAF-PS composites



**Figure 5.5.2.4:** Elongation at break vs fiber loading (wt. %) of TAF-LDPE composites

## 5.5.3 Flexural Strength and modulus

The flexural strength of composites measures the ability of composites to withstand against the bending forces applied perpendicular to its longitudinal axis. The experimental results on flexural strength of the raw and oxidized *Typha angustata* fiber-Polystyrene composites have been presented in figure 4.5.3.1 against the

percentage (wt) of *Typha angustata* fiber loading. Flexural strength and flexural modulus of polystyrene are 70 MPa and 2.5 GPa respectively. It is observed from the figure 5.5.3.1 that the values of flexural strength for both the composites increase with increasing fiber loading up to 20 wt%, then it starts to decrease at 30 wt% fiber loading as compared to 100 wt% Polystyrene matrices. The experimental values of flexural modulus at different fiber loading have been presented in figure 5.5.3.2. It is observed from the figure that flexural modulus for both raw and NaOH treated *Typha angustata*- Polystyrene composites increases significantly with increasing fiber loading. It is also seen that the flexural strength and modulus of oxidized *Typha angustata* fiber-polystyrene composites have similar fiber content effects like raw *Typha angustata* –polystyrene composites. However, higher values of flexural strength and flexural modulus observed in the case of all oxidized *Typha angustata*-PS composites thanthat of raw Typha angsuata-PS composites. This may be due to the better compatibility of oxidized *Typha angustata* fiber for the formation of *Typha angustata* –PS composites than that of raw *Typha angustata* fiber. The better compatibility may arise from the opening of glucoside ring of cellulose molecules.
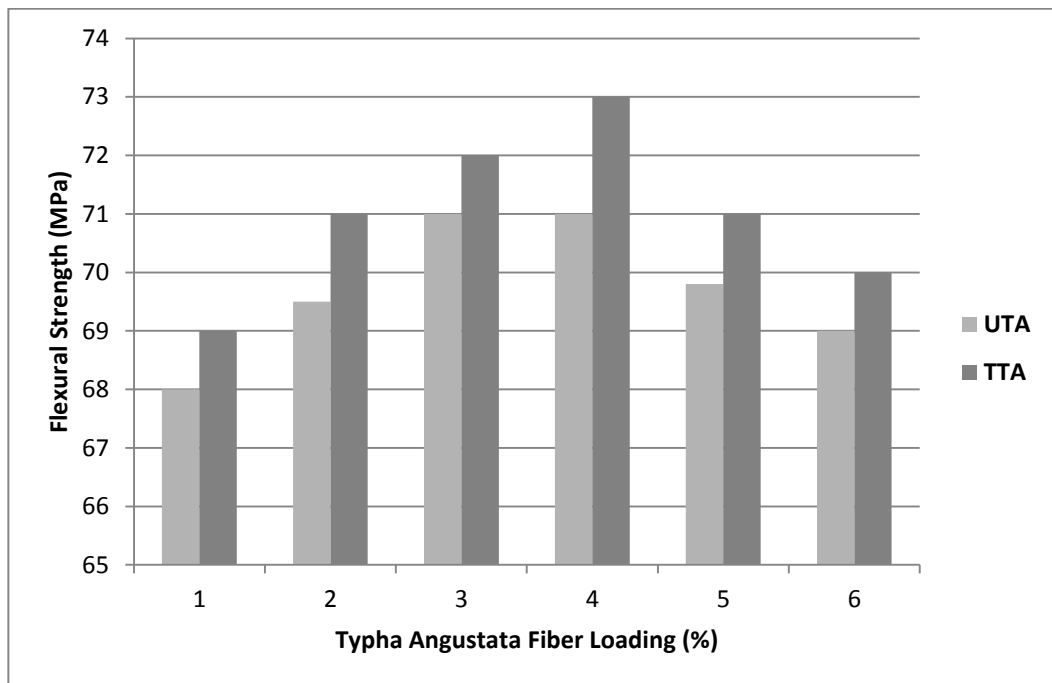


**Figure 5.5.3.1** Flexural strength vs *Typha angustata* fiber content (wt %) for raw and oxidized *Typha angustata*-Polystyrene composites.
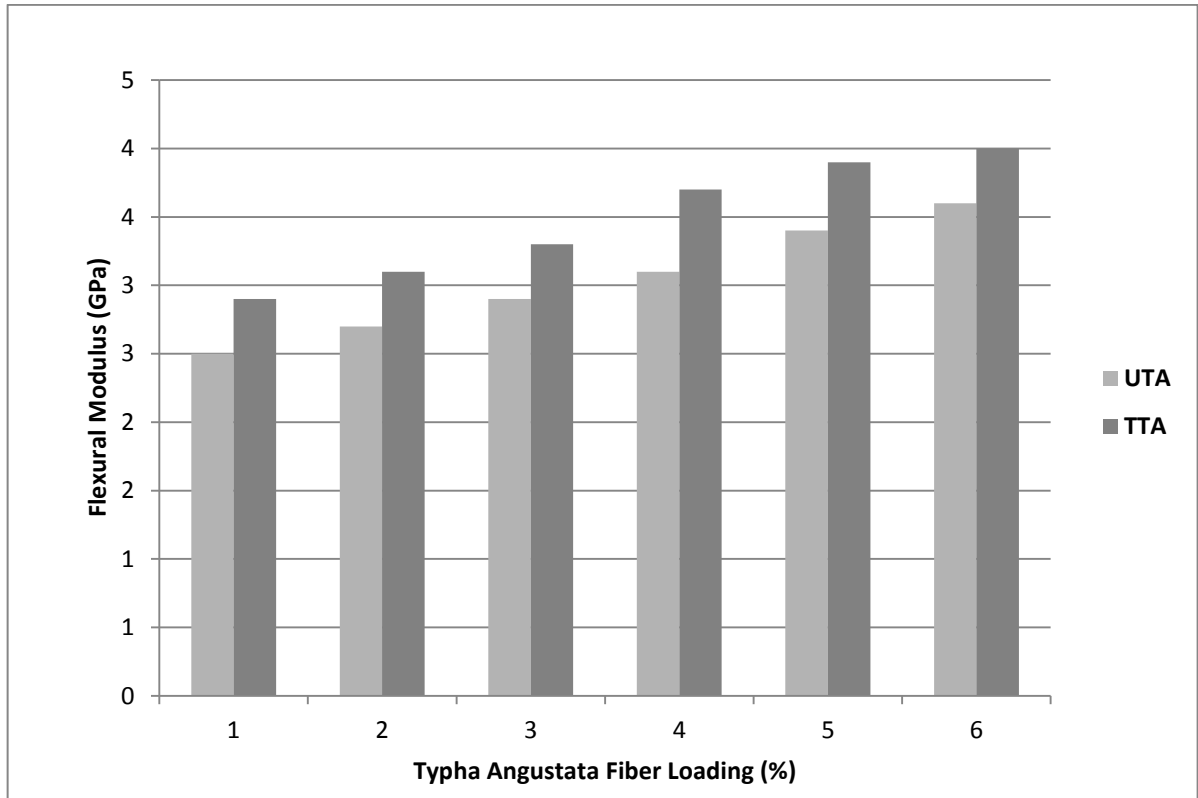
**Figure 5.5.3.2** Flexural modulus vs *Typha angustata* fiber content (wt%) for *Typha angustata*-Polystyrene composites.

## 5.6 Water absorption

Water absorption of lignocellulosic fiber plastic composites are important characteristics that determine the use and applications of these materials [55]. Water absorption could lead to a decrease in some of the properties and needs to be considered when selected for applications. It is difficult to eliminate entirely the absorption of moisture from the composites without using extensive surface barriers on the composite surface. Water absorption in lignocellulosic based composites is mainly due to hydrophilic nature of cellulosic materials. The hydrophilic nature mainly arises from the presence of oil in cellulose, hemicelluloses and lignin molecules which attract moisture. Moisture build up in the cell wall could result in fiber swelling and concerns on the dimension stability cannot be ignored [55].
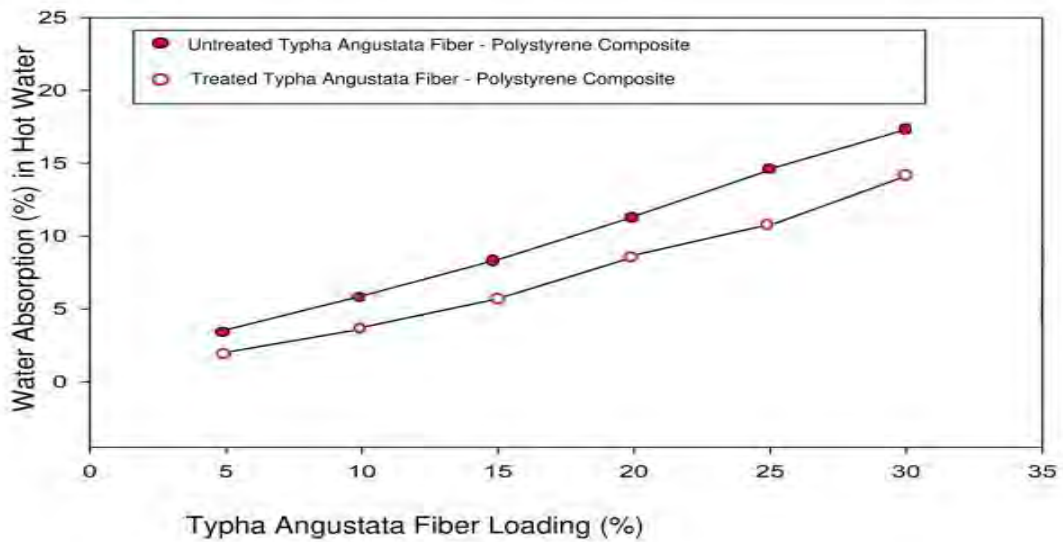
**Figure 5.6.1** Water absorption vs fiber loading (%) curves for untreated and treated *Typha angustata*–Polystyrene composites
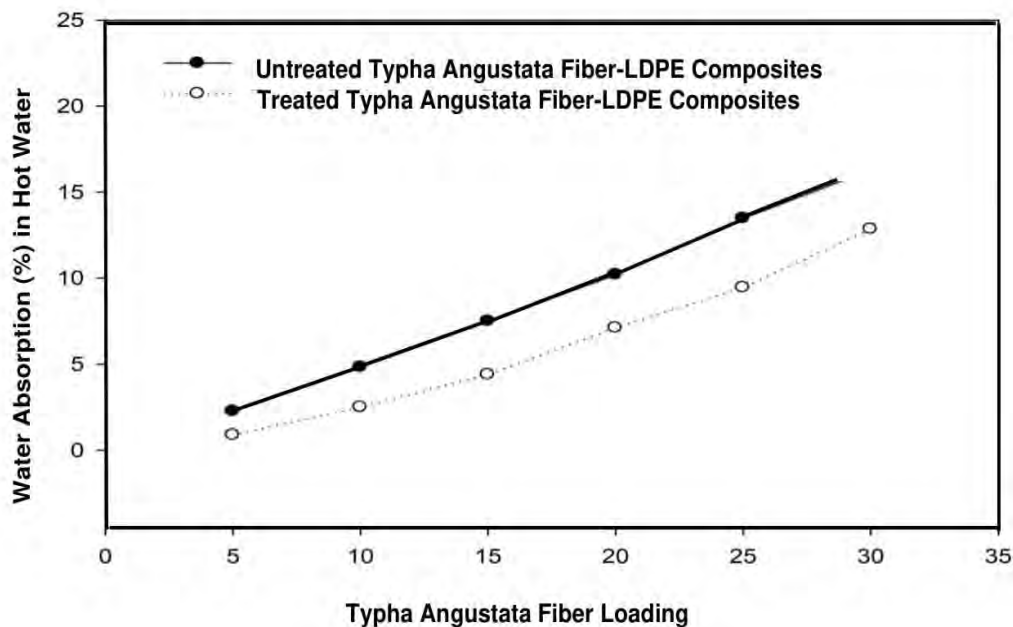


**Figure 5.6.2** Water absorption vs fiber loading (%) curves for untreated and treated *Typha angustata*–LDPE composites

Therefore, chemical modification is needed to reduce the moisture absorption of the fibers results of water absorption obtained in our study are shown in Figure 5.6 it is observed from the figure that water absorption of the composites increases with increasing fiber loading. But water absorption of the oxidized *Typha angustata*-Polystyrene composites is less than that of *Typha angustata*-Polystyrene composites. Cellulose, hemicellulose and lignin of fiber contains hydroxyl group. Hydroxyl group forms hydrogen bond with water molecule. Aldehyde group of oxidized *Typha angustata* is less polar than hydroxyl group. So, hydroxyl group absorbs more water than aldehyde group. As a result, water absorption

of oxidized *Typha angustata*-Polystyrene composites are less than that of untreated *Typha angustata*-Polystyrene composites. Chemical modification of hydroxyl group of cellulose in *Typha angustata* to aldehyde group of dialdehyde cellulose in *Typha angustata* influences to get these improved results. So, dimension stability will be higher for treated *Typha angustata*-Polystyrene composites than untreated *Typha angustata*-Polystyrene composites.

## 5.7 Thermo gravimetric analysis (TGA) and Differential Scanning Calorimetric

(DSC) analysis of TAF-PS composite Thermo-gravimetric analysis and differential scanning calorimetric analysis of the compression moulded PS, 20 wt. % treated and untreated TAF-PS composites were done to examine the degradation attributes of fiber at different stages of treatment. The recorded TGA and DSC thermographs are presented in figure-5.7.1 and figure-5.7.2 respectively. The thermo gravimetric analysis and differential scanning calo-rimetriy of the prepared samples were recorded on a STA 449F3 NETZSCH thermal analysis instrument in the temperature range of (0-900) °C.
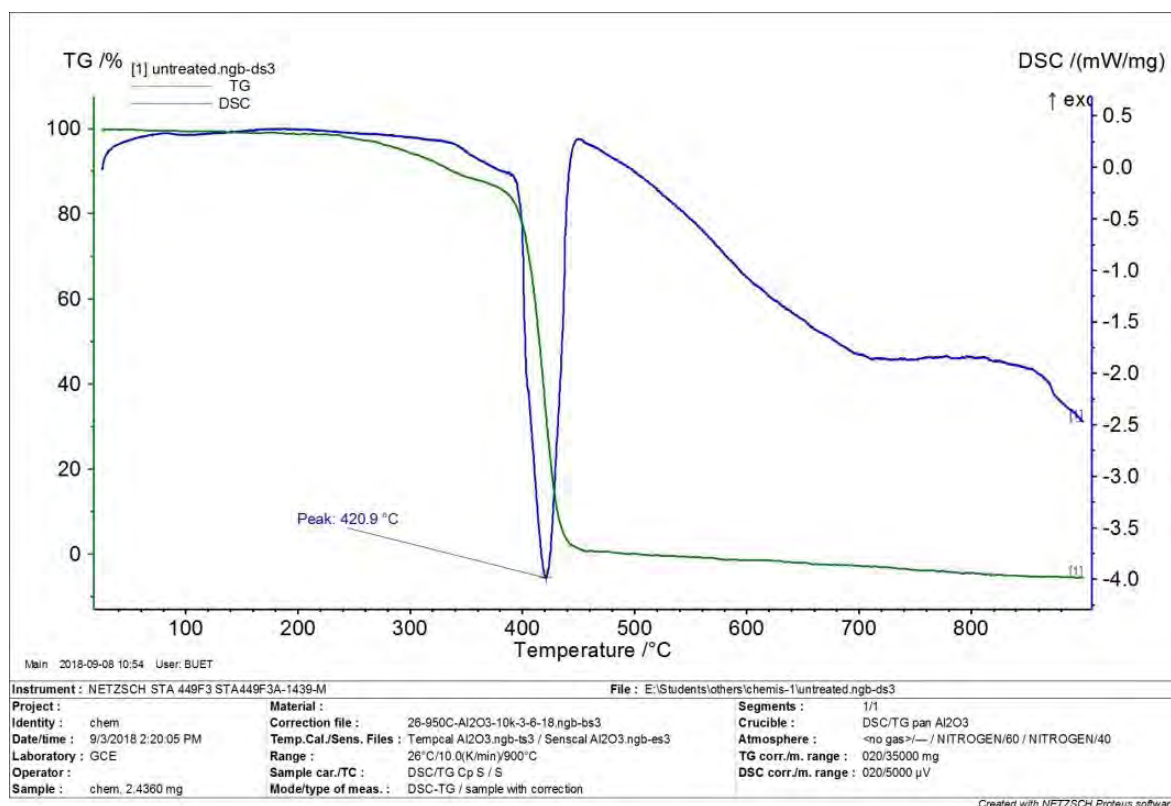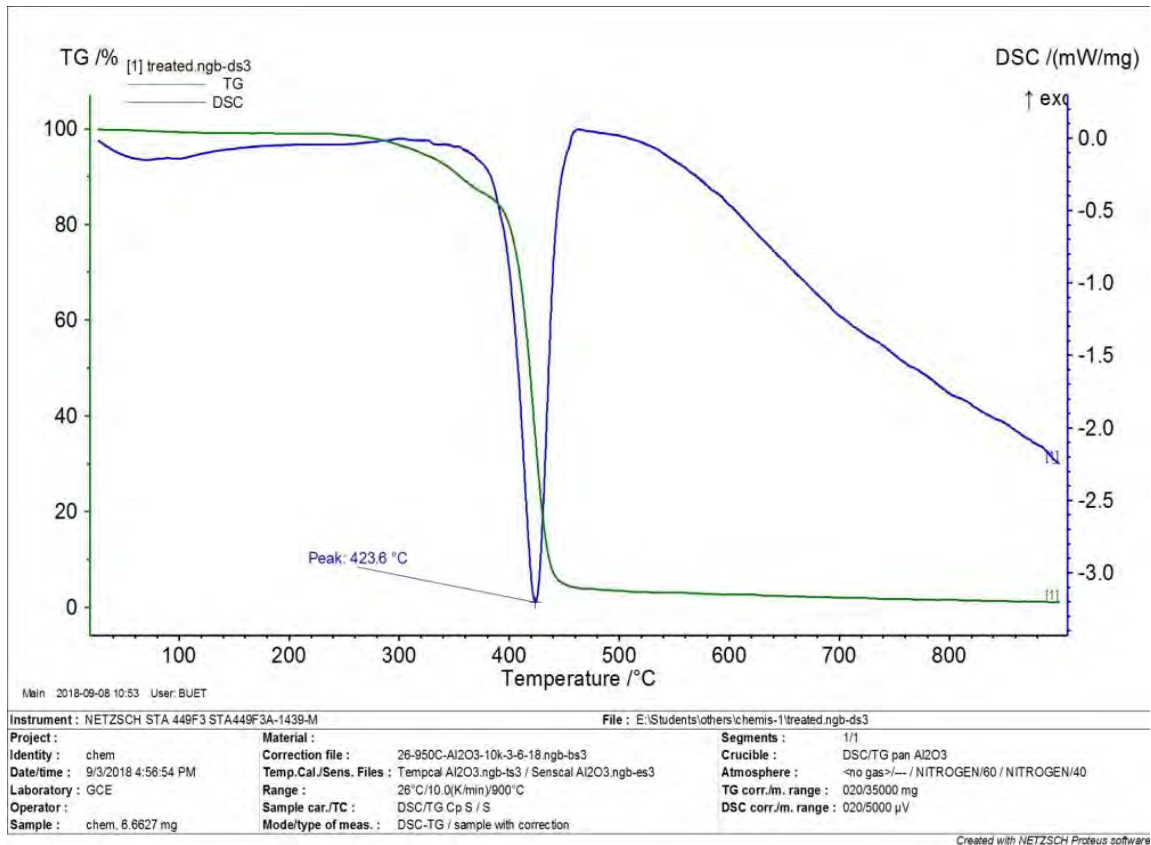


**Figure 5.7.1:** TGA &DSC curves of untreated TAF-PS composite

Degradation start at 360.3ºC and 97.70 % mass change was completed at 454.4 ºC for TAF-PS composite whereas the mass change started at 272.6ºC and 92.86% mass change was completed at 474.9 ºC for the treated TAF-PS composite. The degradation start at 267 ºC and 95.46% mass change was observed at 475.5ºC for TAF-PS composite. DSC is used to identify melting temperature and amount of energy absorbed or release by materials. Tm of untreated TAF-PS composite and treated TAF-PS is 420.9 ºC and 423.6 ºC respectively. Almost same thermal properties were found for treated and untreated TAF-PS composites. Treated TAF enhanced the thermal properties of TAF-PS composites materials.



**Figure 5.7.2:** TGA & DSC curves of alkali treated TAF-PS composite
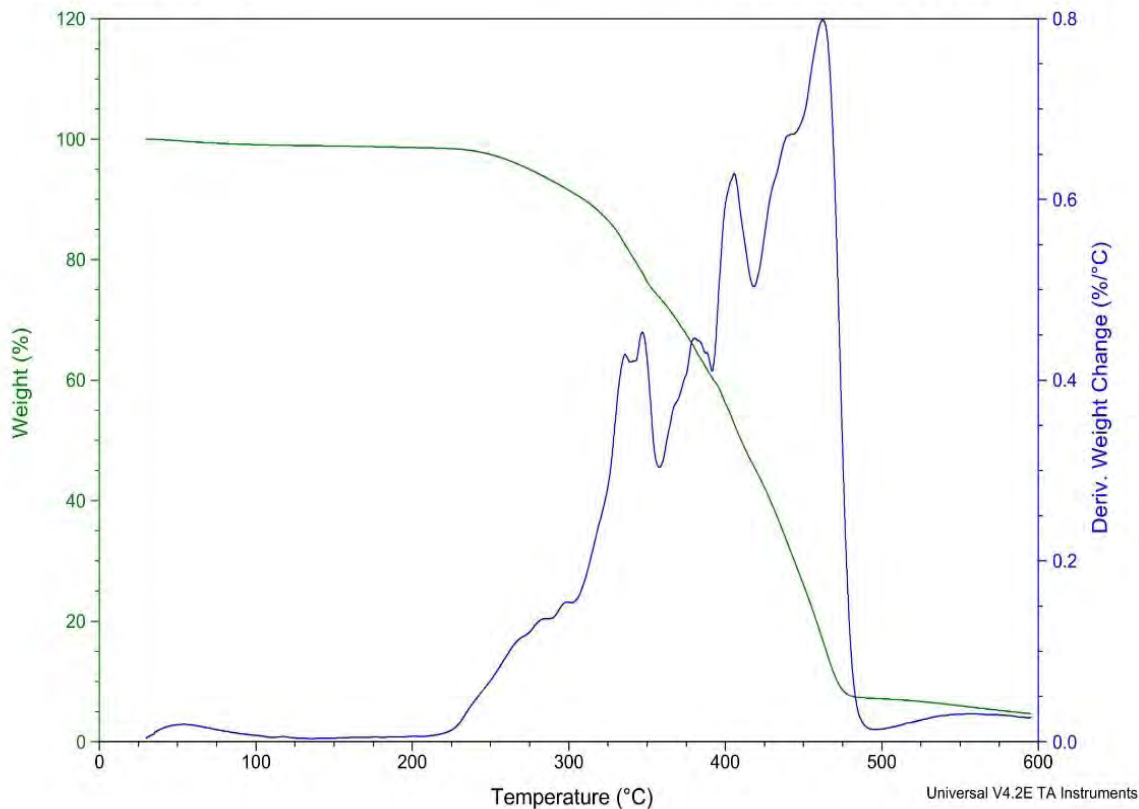
**Figure 5.7.3:** TGA curve of untreated TAF-LDPE composite
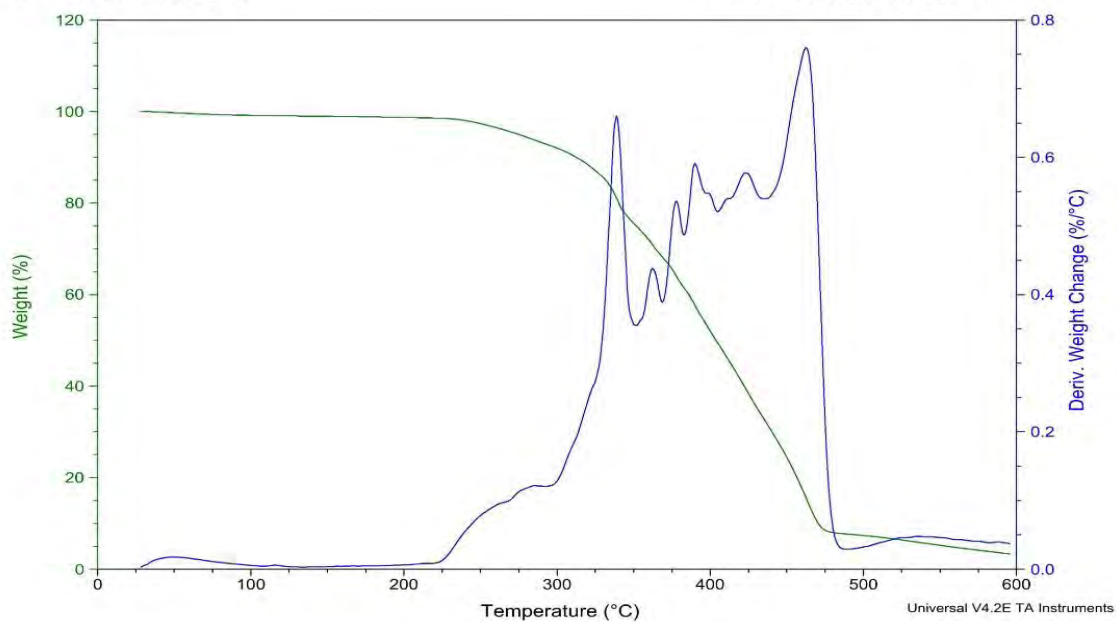


**Figure 5.7.4:** TGA curve of Meta per iodate treated TAF-LDPE composite

**Figure 5.7.5:** TGA curve of alkali treated TAF-LDPE composite



**Figure 5.7.6:** DSC curve of untreated TAF-LDPE composite

**Figure 5.7.7:** DSC curve of Meta per iodate treated TAF-LDPE composite



**Figure 5.7.8:** DSC curve of alkali treated TAF-LDPE composite

# CHAPTER 6: SUMMARY AND CONCLUSION

This study refers to the experimental determination and theoretical interpretation of a series of works on "Physico-Mechanical Properties of Chemically Treated *Typha Angustata* Fibre Reinforced Plastic Composites" The total work of this study is summarized below:
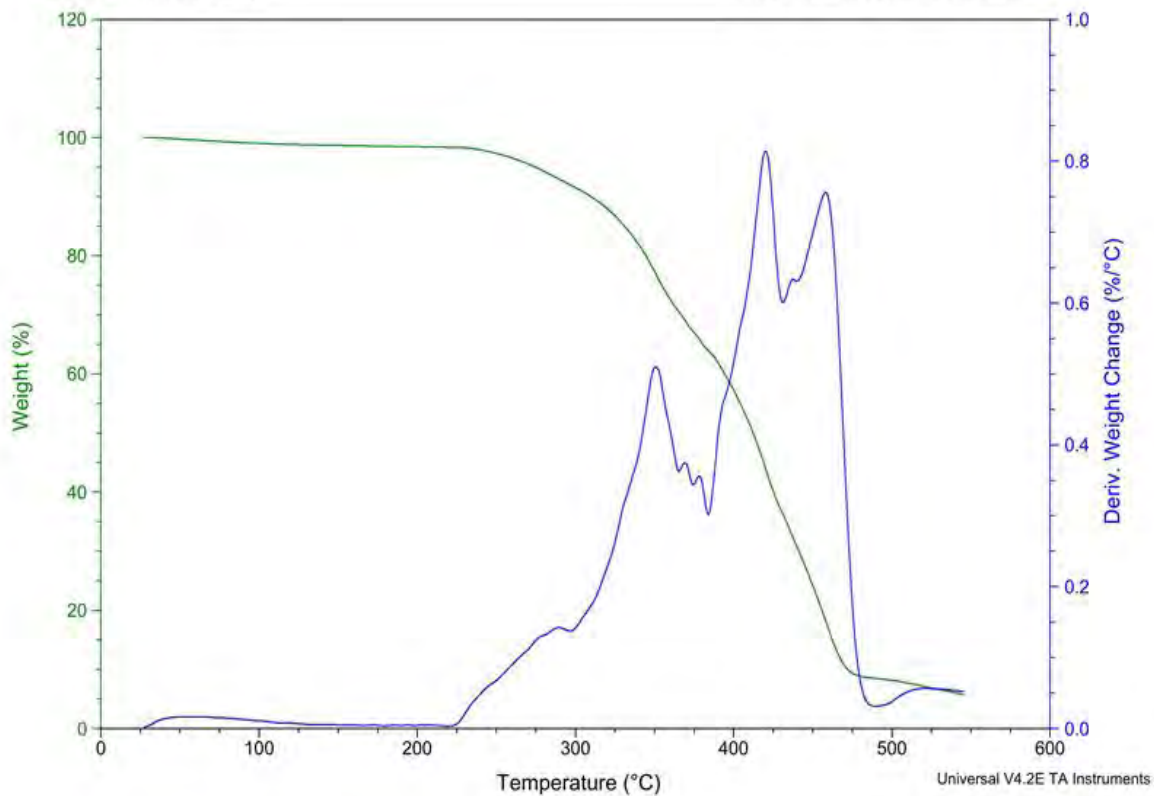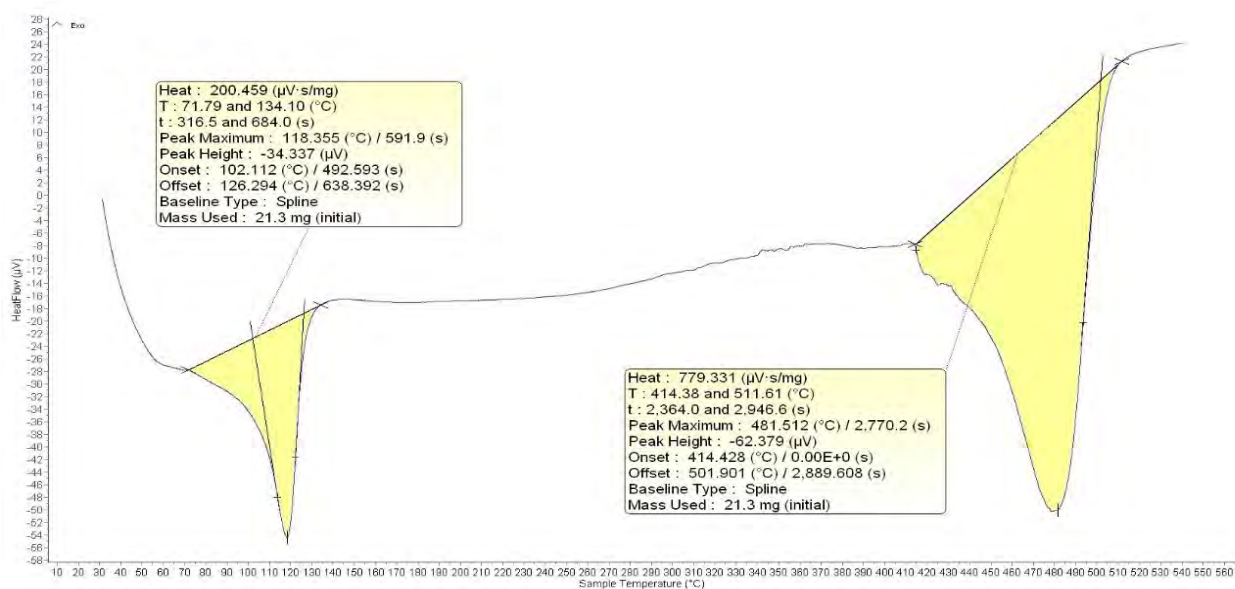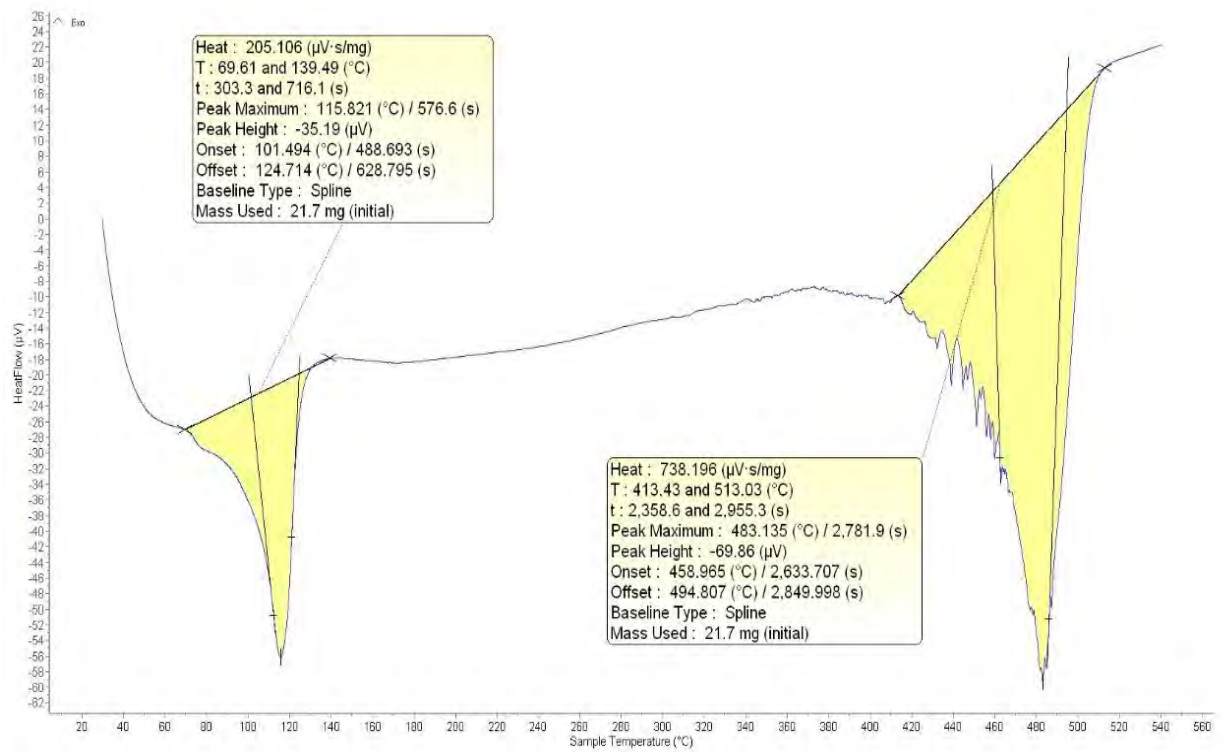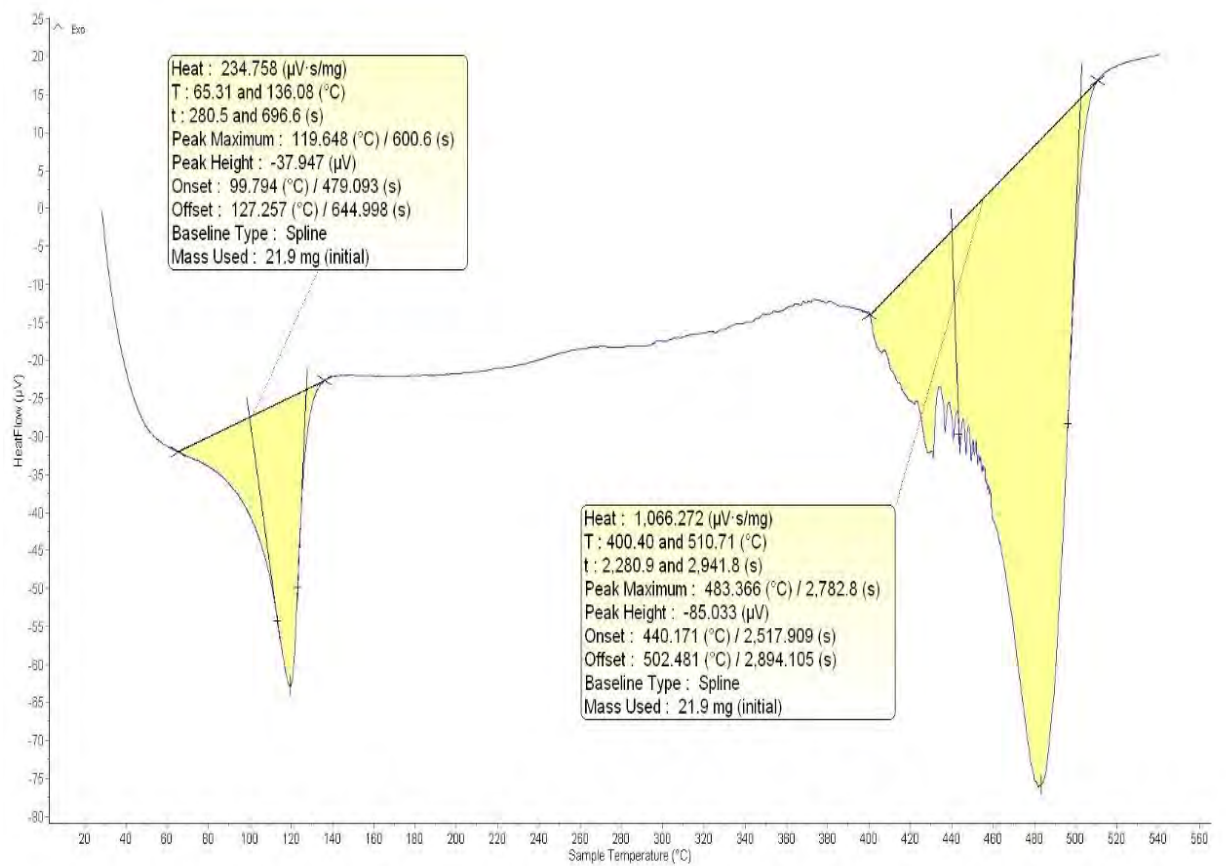
1) Cellulose in *Typha angustata* (TA) fibers can be oxidized at C-2 and C-3 position of anhyroglucose unit to form reducing oxycellulose (dialdehyde) by sodium hydroxide and sodium meta periodate. FT-IR and SEM analyses have been done to characterize the intermolecular interaction and physical structural properties of oxidized *Typha angustata* fibers. Aldehyde group is less polar than hydroxyl group so hydrophilic nature of oxidized *Typha angustata* is reduced than raw *Typha angustata* and it provides better interfacial adhesion between oxidized *Typha angustata* - Polystyrene matrix and *Typha angustata* – LDPE matrix.

2) Tensile modulus, flexural strength and flexural modulus of the raw and oxidized *Typha angustata*-PS and TA-LDPE composites are significantly higher than 100 wt. % Polystyrene and low-density polyethylene (LDPE). Flexural strength of the composites increases up to 20 wt. % fiber loading than unfilled PS and LDPE matrix. Tensile strength and elongation at break of the composites decreases with increasing fiber loading. Tensile modulus and flexural modulus of these fibers loaded composites increases with increasing fiber loading. Tensile strength and flexural strength of oxidized TAF-Ps and TAF-LDPE composites are higher than raw TAF-PS and TAF-LDPE composites respectively. All the mechanical properties of oxidized TAF-PS and TAF-LDPE composites are higher than raw TAF-PS and TAF-LDPE composites. The possible reason for these improvements is due to the absence of intermolecular hydrogen bonding of dialdehyde cellulose in oxidized TAF which improves the adhesion between fiber and LDPE and Polystyrene matrix in the composite interface. As a result oxidized TAF dispersed more uniformly in the composites than raw *Typha angustata* fiber.

3) From the morphological studies, it was observed that the alkali treated TAF-PS and sodium meta per iodate treated TAF-LDPE composites show strong uniform dispersion and better compatibility between fiber and matrix than untreated TAF-PS and TAF-LDPE composites.

4) Water absorption properties of the composites also show better result for oxidized TAF-PS and TAF-LDPE composites than raw TAF-PS and TAF-LDPE composites, which will improve the dimensional stability of the composites.

5) From thermal analysis of the composites it was observed that because of strong interfacial adhesion and dimensional stability between fiber and polymer matrices alkali and sodium

meta per iodate treated *Typha angustata* fiber composites showed the higher thermal properties than untreated *Typha angustata* fiber composites.

From these observations, it can be concluded that natural fiber matrix interaction can be improved by chemical modification of fiber by decreasing their hydrophilic nature which enhances the physico- mechanical properties of the natural fiber reinforced composites.

# CHAPTER 7: REFERENCES

[1]    Ehrenstein, G. W., and Kabelka. J., "Reinforced Plastics, *In Ullmann's Encyclopedia of Industrial Chemistry"*, Vol. 23, pp. 56-60, 1992.

[2]    Matuana, L, M., Balatinez, J. J., Sodhi, R. N. S., and Park, C. B., Surface characterization of esterified cellulosic fibers by XPS and FTIR Spectroscopy, Wood Science and Technology, Vol. 35, pp. 191-201, 2001.

[3]    Rowell, R. M., and Stout, H. P., "Jute and Kenaf, *In handbook of Fiber Science and Technology"*, Vol. 4, Fiber Chemistry, Edited by: M. Lewin and E. M. Pearce, New York, 1985.

[4]    El-Hosseiny, F., and Page, D., The mechanical properties of single wood pulp fibers: Theories of strength, Fibre Sci Technol*,* Vol. 8, pp. 21-31, 1975.

[5]    Woodhams, R. T., Thomas, G., and Rodgers, D. K., "Wood fibers as reinforcing fillers for polyolefin's, Polym. Eng. Sci. Vol. 24, pp. 1166-1171, 1984.

[6]    Zadorecky, P., and Michell, A. J., Future prospects for wood cellulose as reinforcement in organic polymer composites, Polymer Composite, Vol. 10, pp. 69-77, 1990.

[7]    Literature Review: Thermoplastic Composite Materials
       http://wwwscholar.lib.vt.edu/theses/available/etd-1100499
       135025/unrestricted/Etbody.pdf

[8]    Wolf, C. J., Thermoplastics, In KIRK-OTHMER Encyclopedia of Chemical Technology, Fourth Edition, Vol. 7, pp. 256-258, 1998.

[9]    Bismarck, A., Askargorta, I. A. and Springer, J., Surface Characterization of Flax, Hemp and Cellulose Fibers; Surface Properties and the water Uptake Behavior, *Polymer Composites,* Vol.23, pp. 872-894, 2002.

[10]   http://www.jute.org/plant_1.htm

[11]   Kozlowski, R., Potential and diversified uses of green fibers, In 3[rd] International Wood and Natural Fiber Composites Symposium*,* Kassel, Germany, September 19-20, 2000.

[12]   https://www.azom.com/article.aspx?ArticleID=17194

[13]   https://www.azom.com/article.aspx?ArticleID=9814

[14]    "WelcomelgschemistryJusthost.com"(PDF).www.lgschemistry.org.uk, 2018

[15]    Baeurle, S. A., Hotta, A., Gusev, A. A., "On the glassy state of multiphase and pure polymer materials". Polymer. Vol **47**, pp. 6243–6253, 2006.

[16]    Shenoy A. V., and Saini, D. R., Thermoplastic Melt Rheology and Processing, *Marcel Dekker Inc., New York.* 2015.

[17]    Charles, P., Dermott, M., and Aroon, V., Selecting Thermoplastics for Engineering Applications, Marcel Dekker Inc., New York. 2015.

[18]    Low density polyethylene: from Wikimedia the free encyclopedia.

[19]    Alnaimi, S. M., The Optical and Electrical Properties of Low Density Polyethylene Compact Discs in CuSO4 Solution, Journal of the 3rd Magneto Electronic International Symposium (MAGEL-3-La Rochelle-2004), Article in Press.

[20]    Peacock, J. A., Handbook of polyethylene: Structures: Properties, and applications, *Plastics Engineering*, Marcel Dekker Inc, 2000.

[21]    www.qapco.com/English/Plants_Products/GradesandApplications

[22]    "poly(ethylene)_msds"

[23]    Wapler, M. C., Leupold, J., Dragonu, I., Elverfeldt, D. V., Zaitsev, M., and Wallrabe, U., "Magnetic properties of materials for MR engineering, micro-MR and beyond". *JMR*. pp. 233–242, 2014.

[24]    Pechmann, H. V., "Ueber Diazomethan und Nitrosoacylamine", Berichte der Deutschen Chemischen Gesellschaft zu Berlin. Vol. **31**, pp. 2643-2644, 1898.

[25]    Bamberger., and Tschirner, F., "Ueber die Einwirkung von Diazomethan auf β-Arylhydroxylamine", Berichte der Deutschen chemischen Gesellschaft zu Berlin, Vol. 33, pp. 955–959, 1900.

[26]    Bamberger., and Tschirner, F., "Ueber die Einwirkung von Diazomethan auf β-Arylhydroxylamine" *Berichte der Deutschen Gesellschaft zu Berlin*, Vol. 33, pp. 956-957, 1900.

[27]    Iqubal, M. A., "Polysthylene: A crisp overview", Winnington history in the making, pp. 955-959, 2014.

[28]    "Poly – The All Star Plastic", Popular Mechanics, Vol. 91 (1), pp. 125–129, 2014.

[29]    Mathers, H. R., Robert, T., "Review of Phillips Chromium Catalyst for Ethylene Polymerization, Chapter 1, 2010.

[30]    Market Study: Polyethylene – HDPE, *Ceresana Research*, 2012.

[31]    Market Study: Polyethylene – LLDPE, *Ceresana Research,* 2nd Edition, 2015.

[32]    Market Study: Polyethylene – LDPE, 2nd edition, *Ceresana Research*, October 2014. Retrieved 3 February 2015.

[33]    Scheirs, J., Priddy, D., "Modern Styrenic Polymers: Polystyrenes and Styrenic Copolymers", *John Wiley & Sons*, ISBN 978-0-471-49752-3, pp. 3-4, 2003.

[34]    Common Plastic Resins Used in Packaging, Introduction to Plastics Science Teaching Resources. *American Chemistry Council*, 2012.

[35]    Maul, J., Frushour, B. G., Kontoff, J. R., Eichenauer, H., Ott, K.-H., and Schade, C., "Polystyrene and Styrene Copolymers" in Ullmann's Encyclopedia of Industrial Chemistry", *Wiley-VCH*, 2007.

[36]    Kwon, BG., Saido, K., Koizumi, K., Sato, H., Ogawa, N., Chung, SY., Kusui, T., Kodera, Y., and Kogure, K., "Regional distribution of styrene analogues generated from polystyrene degradation along the coastlines of the North-East Pacific Ocean and Hawaii", *Environmental Pollution.* Vol. 188, pp. 45–49, 2014.

[37]    Simon, E., "Ueber den flüssigen Storax (Styrax liquidus)" [On liquid storax (Styrax liquidus)", *Annalen der Chemie*, Vol. 31, pp. 265–277, 1839.

[38]    Blyth, J., Hofmann, Wilh, A., "Ueber das Stryol und einige seiner Zersetzungsproducte" (On styrol and some of its decomposition products)", *Annalen der Chemie und Pharmacie*, Vol. 53 (3), pp. 289–329, 1845.

[39]    Blyth., and Hofmann., "Analyse sowohl als Synthese haben in gleicher Weise dargethan, dass Styrol und die feste glasartige Materie, für welche wir den Namen Metastyrol vorschlagen, dieselbe procentische Zusammensetzung besitzen, pp. 312-313, 1845.

[40]    Berthelot, M., "Sur les caractères de la benzine et du styrolène, comparés avec ceux des autres carbures d'hydrogène" (On the characters of benzene and styrene, compared with those of other hydrocarbons)", *Bulletin de la Société Chimique de Paris,* Vol. 6, pp. 289–298, 1866.

[41]    "Invention of STYROFOAM". Dow Chemical. Archived from the original on 27 October 2012. Retrieved 23 December 2012.

[42]    Natta, G., Corradini, P., Bassi, I. W., Crystal structure of isotactic polystyrene", *Il Nuovo Cimento*. Vol. 15: pp. 68–82, 1960.

[43]    Ferrigno, T.H., Rigid Plastics Foams, 2nd edition. pp. 207-208, 1967.

[44]    Celebrating 50 Years of Excellence in People and Products". Dart Container Corporation. Archived from the original on 4 June 2010. Retrieved 23 December 2012.

[45]    English, B., Chow, P., and Bajwa, D. S., "Processing into composites", *In Paper and Composites from Agro-Based Resources*, Edited by Rowell, R. M., Young R. A., and Rowell, J. K.,

[46]    Rowell, R. M., "A new generation of composite materials from agro-based fiber", *In proceedings of the 3rd International Conference on Frontiers of Polymers and Advanced Materials,* 1995.

[47]    Olesen P. O., and Plackett, D. V., Prospective on the Performance of Natural plant fibers. http://www.ienica.net/fibresseminar/olesen.pdf.

[48]    http://www.jute.org/jute_diversified_prod.htm

[49]    Clemons, C. M., Caulfield D. F., and Giacomin, A. J.," Dynamic Fracture Toughness of Cellulose Fiber- Reinforced Polypropylene: Preliminary Investigation of Microstructural Effects", *Journal of Elastomers and Plastics,* Vol. 31, pp.367-378, 1999.

[50]    http://www.jute.org/Taxonomical%20Characteristics.pdf

[51]    Rowell, R. M., "Opportunity for value-added bio-based composites", *In Pacific Rim Bio-Based Composites Symposium,* pp. 244-252, 1992.

[52]    Rowell, R. M., and Clemons, C. M., "Chemical Modification of wood fiber for thermoplasticity, compatibilization with plastics and dimensional stability", *In proceedings, International Particleboard Composite Materials Symposium,* 1992.

[53]    Matsuda, H., and Ueda, M., "Preparation and utilization of esterified woods bearing carboxyl groups. IV. Plasticization of esterified woods", *Mokuzai Gakkaishi,* Vol. 31, pp. 215-216, 1985.

[54]    Das, S., Saha, A. K.,  Chowdhury, P. K., Basak, R. K., Mitra, B. C., Todd, T., Lang, S., and Rowell, R. M., "Effect of Steam Pretreatment of Jute Fiber on

Dimensional Stability of Jute Composite", *Journal of Applied Polymer Science,* Vol. 76, pp. 269-270, 1993.

[55] Rowell, R. M., Sanadi, A. R., Caulfield, D. F., and Jacobson, R. E., "Utilization of Natural Fibers in Plastic Composites: Problems and Opportunities", *In Lignocellulosic Plastic Comp[osites,* 1997.

[56] "Resin and Compunds", *Modern Plastics Encyclopedia,* McGraw – Hill, New York, pp. 269, 1993.

[57] "Material Design Material Selector Issue", *Thermoplastic Molding Compounds,* Penton Publishing, OH, pp. 184, 1994.

[58] Saad, M. J., Ishak, Z. A. M., and Rozman, H. D., "Empty Fruit Bunch (EFB)-Polypropylene Composites: TheEffect of Chemical Modification of EFB with Mateic Anhydride (MAH) on the Tensile Properties and Dimensional Stability", *In Proceedings USM- Jircas Joint International Symposium,* pp. 209-223, 2001.

[59] Rowell, R, M., "Chemical Modification of Lignocellulosics", *In Proceedings of the international workshop on Frontiers of Surface Modification and Characterization of Lignocellulosic Fibers,* pp. 30-31, 1996.

[60] Andersson, M., and Tillman, A-M., "Acetylationof Jute: Effects on Strength, rot resistance and hydrophobicity", *Journal of Applied Polymer Science,* Vol. 37, pp. 3437-3447, 2003.

[61] Chow, P., Bowers, T. C., Bajwa, D. S., Youngquist, J. A., Muehl, J. H., Stark, N. M., Krzysik, A. M., and Quang, L., "Dimensional stability of composites from plastics and cornstalk fibers", *In Fifth International Conference on Wood-Plastic Composites,* 1999.

[62] Sombatsompop, N., and Chaochanchaikul, K., "Effect of moisture content on mechanical properties, Thermal and structural stability and extrudate texture of poly vinyl chloride/ wood sawdust composites", *Polym. Int.,* Vol. 53, pp. 1210-1218, 2004.

[63] Yachmenev, V. G., Parikh, D. V., and Calamari, T. A., "Thermal Insulation Properties of Biodegradable, Cellulosic Based Nonwoven Composites for Automotive Application", *Journal of Industrial Textiles,* Vol. 31, pp. 283-296, 2002.

[64] Lucka M., and Bledzki, A. K., "Effect of acetylation on the structure and propertiesof flax fiber", *In 3[rd] International Wood and Natural Fiber Composites Symposium,* 2000.

[65]     Karnani, R., Krishnan, M., and Narayan, R., "Biofiber- reinforced polypropylene Composites", *Polymer Engineering and Science,* Vol. 37, pp. 476-483, 1997.

[66]     Takemura, K., "Effect of water absorption on static and creep propereties for jute fiber reinforced composite". http: www.witpress.com, ISBN 1-85321-717-5.

[67]     Khan, M. A., Hassan, M. M., and Drazal, L. T., "Effect of 2- hydroxyl ethyl methacrylate (HEMA) on the mechanical and thermal properties of jute-polycarbonate composite", *Composite: Part A: Applied Science and Manufacturing,* Vol. 36, pp. 71-78, 2005.

[68]     Yinghai, L., Zhanjun, L., Xiaohui, L., and Kuilin, D., "Grafting of poly methyl acrylate onto cellulose by potassium diperdcuprate (III) initiating system", *CJI,* Vol. 5, pp. 18, 2003.

[69]     Kokta, B. V., "Wood Fiber Reinforced Thermoplastics Composites: Effect of Compounding, Molding and Fiber Length on Resulting Properties: Pilot Plant Trials", *In 3$^{rd}$ International Wood and Natural Fiber Composites Symposium,* 2000.

[70]     Poh, L. K., Zakaria, S., Ahmed, S., and Deraman, M., "A study on the thermal properties and superstructure of benzoylated palm empty fruit bunch fibers", *In proceedings USM-JIRCAS Joint International Symposium,* pp. 140-145, 2001.

[71]     Ali, K. M. I., Khan, M. A., and Hussain, M. M., "Wood Plastic Composite with Methylmethacrylate in presence of Novel Additives", *Poly Plast. Technol. Eng.,* Vol. 33, pp. 477-487, 1994.

[72]     Oksman, K. and Clemons, C., "Mechanial Properties and Morphology of Impact Modified Polypropylene-Wood Flour Composites", *Journal of Applied Polymer Science,* Vol. 67, pp. 1503-1513, 1998.

[73]     Ismail, H., Salmah., and Bakar, A. A., "The Effect of PPMaH and LICA On Properties of Paper Sludge Filled PP/EPDM Composites", *In 3$^{rd}$ USM-JIRCAS Joint International Symposium,* 2004.

[74]     Li, X. H., Meng, Y. Z., Wang, S. J., Rajulu, A. V., and Tjong, S. C., "Completely Biodegradable Composites of Polypropylene carbonate and Short, Lignocellulose Fiber Hildegardia populifolia", *Journal of pPolymer Science: Part B: Polymer Physics,* Vol. 42, pp. 666-675, 2004.

[75]     Brouwer, W. D., "Natural Fibre Composites: Where Can Flax Compete with Glass", *Sampe J,* Vol. 36, pp. 18, 2000.

[76] Dahlke, B., Larbig, H., Scherzer, H. D., and Poltrock, R., "Natural Fiber Reinforced Foams Based on Renewable Resources for Automotive Interior Applications", *Journal of Cellulose Plastics*, Vol. 34, pp. 361, 1998.

[77] De Bruijn, J. C. M., "Natural Fibre Mat Thermoplastic Products from a Processor's Point of View", *Applied Composite Materials,* Vol. 7, pp. 415, 2000.

[78] Ellison, G. C., and McNaught, R., "The Use of Natural Fibres in Nonwoven Structures for Applications as Automotive Component Substrates", *Research and Development Report, Ministry of Agriculture Fisheries and Food*, 2000.

[79] Estevez, A. G., Santillanes, J. P., Carvajal, O., Perez, P. S., Dorrego M. E. R., and Fernandez, M., "Polymeric Composites Reinforced with Natural Fiber from Bagasse for Moulding Pieces for Automotive Industry", *In Proc. Atibaia, SP,* pp. 63, 1998.

[80] Karus, M., Kaup, M., and Lohmeyer, D., "Study on Markets and Prices for Natural Fibres", *Nova Institute,* 2000.

[81] Leao, A. L., Rowell R. M., and Tavares, N., "Applications of Natural Fibers in Automotive Industry in Brazil - Thermoforming Process", *In Science and Technology of Polymers and Advanced Materials*, Plenum Press, New York - London, pp. 755, 1998.

[82] Peijs, T., "Composites Turn Green", *e-Polymers,* T_002, 2002.

[83] Schuh, T. G., "Renewable Materials for Automotive Applications, In Proceedings of Natural Fibre Perfomance Forum", 1999.

[84] Papadopoulos, A. N., and Hague, J. R. B., "The Potential for Using Flax (Linum Usitatissimum L) Shiv as a Lignocellulosic Raw Material for Particleboard", *Industrial Crops and Products*, Vol. 17, pp. 143, 2003.

[85] Sain, M., and Fortier, D., "Flax Shives Refining, Chemical Modification and Hydrophobisation for Paper Production", *Industrial Crops and Products*, Vol. 15, pp. 1, 2002.

[86] Sanadi, A. R., Caulfield, D. F., Stark, N. M., and Clemons, C. C., "Thermal and mechanical analysis of lignocellulosic polypropylene composites", *In The Fifth International Conference on Wood Fiber-Plastic Composites,* 1999.

[87] Santulli, C., and Cantwell, W., "Impact Damage Characterization on Jute Reinforced Composites", *Journal of Materials Science and Letters,* Vol.20, pp.471-473, 2001.
www.kluweronline.com/article.asp?PIPS=337022&PDF=1

[88]     Brundle C. R., "Encyclopedia of Materials Characterization, Manning Publications Company", Greenwich, ISBN No. 0750691689, 1992.

[89]     Sireger, J. P., Sapuan, S. M., Rahman, M. Z. A., Zaman, H. M. D. K., "Journal of food, Agriculture & Environment", Vol. 8, pp. 1103– 1108, 2010.

[90]     Ponnukrishnan, P., ChithambaraThanu, M., and Richard, S., "Mechanical Characterization of Typha Domingensis Natural Fiber Reinforced Polyester Composites", ISSN 2328-3491, pp. 241-244. 2014.

[91]     Tanvir, S., Parvin, S. H., Khan, M. W., "International Journal of Advance Research *(IJAR),* Vol. 6, pp. 202-209, 2018.

[92]     Das, S., Saha, A. K., Choudhury, P. K., Basak, R. K., Mitra, B. C., Todd, T., Lang S., and Rowell, R. M., "Effect of Steam Pretreatment of Jute Fiber on Dimensional Stability of Jute Composite", *Journal of Applied Polymer Science*, Vol. 76 pp.1652-1661, 2000.

[93]     Clemons, C. M., Caulfield D. F., and Giacomin, A. J., "Dynamic Fracture Toughness of Cellulose Fiber-Reinforced Polypropylene: Preliminary Investigation of Microstructural Effects", *Journal of Elastomers and Plastics,* Vol. 31, pp.367-378, 1999.

[94]     Takemura, K., "Effect of water absorption on static and creep properties for jute fiber reinforced composite". htpp://www.witpress.com, ISBN 1-85321-717-5, 2002.

[95]     Massimo, B., Elisa, Z., "Effect of Compounding, Molding and Fiber Length on Resulting Properties: Pilot Plant Trials, *In 3rd International Wood and Natural Fibre Composites Symposium,* 2000.

[96]     Li, X. H., Meng, Y. Z., Wang, S. J., Rajulu A. V., and Tjong, S. C., "Completely Biodegradable Composites of Polypropylene carbonate) and Short, Lignocellulose Fiber Hildegardia populifolia", *Journal of Polymer Science: Part B: Polymer Physics,* Vol. 42, pp. 666-675, 2004.

[97]     Saad, M., Ishak Z. A. M., and Rozman, H. D., "Empty Fruit Bunch (EFB)-Polypropylene Composites: The Effect of Chemical Modification of EFB with Malefic Anhydride (MAH) on the Tensile Properties and Dimensional Stability*", In Proceedings USMJIRCAS Joint International Symposium,* pp. 209-223, 2001.

[98] Tay, G. S., Abusamah, A., and Rozman, H. D., "Tensile Properties of Polyurethane (PU) Composites Based on Epichlorohydrin Modified OilPalm Empty Fruit Bunch (EFB)", *In 3rd USM-JIRCAS Joint International Symposium,* 2004.

[99] Wunsch, J.R., "Polystyrene – Synthesis, Production and Applications". *Smithers Rapra Publishing.* ISBN 978-1-85957-191-0, pp. 15, 2012.

[100] Rashdi, A.A.A., Sapuan, S.M., Ahmad, M.M.H.M., Khalina, A., "Water absorption and tensile properties of soil buried kenaf fibre reinforced unsaturated polyester composites (KFRUPC)", *J. Food Agricul. Environ.* Vol. 7, pp. 908-911, 2009.

[101] Tanvir s., shahin s., Husna P. N., Khan, M, W., ''Effects on the properties of low density polyethylene composites reinforced with treated and untreated betel nut husk fibers" *Int.J.Adv.Res,* Vol. 6, pp.202-209, 2017.

[102] "ASTM Standard D 638-01, Standard Test Methods for Tensile Properties of Plastics", *In Annual Book of ASTM Standard,* Vol. 8, 2002.

[103] "ASTM Standard D 790-00, Standard Test Methods for Flexural Properties of Unreinforced and Reinforced Plastics and Electrical Insulating Materials", *In Annual Book of ASTM Standard,* Vol. 8, 2002.

[104] "ASTM Standard D 570-98, Standard Test Method for Water Absorption of Plastics", *In Annual Book of ASTM Standard*, Vol. 8, 2002.

[105] Rowell R. M., and Stout, H. P., "Jute and Kenaf", *In Handbook-of Fiber   Science and Technology,* Volume 4, 1985,
htpp://www.rmmn.orq/documnts/pdf1998/rowel98e.pdf

# CHAPTER 8: APPENDIX

## 8.1 Publication

1. Walid Bin Kader, Tanvir Sultana, Md. Wahab Khan, "Physico-Mechanical Properties of *Typha Angustata* Fiber Reinforced Polystyrene Bio-Composites", *International Journal of Advances Research,* Vol 7, Issue 04, 2019.