

**MANUFACTURING AND CHARACTERIZATION OF BETEL  
NUT AND GLASS FIBER REINFORCED HYBRID  
POLYETHYLENE COMPOSITES**

A Dissertation

by

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Session: April 2013

## **DEDICATION**

*This dissertation is dedicated to my parents*

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The Author

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## **LIST OF ABBREVIATIONS**

<b>FRP</b>	Fiber Reinforced Polymer
<b>PE</b>	Polyethylene
<b>PMC</b>	Polymer Matrix Composite
<b>FTIR</b>	Fourier Transform Infrared Spectroscopy
<b>SEM</b>	Scanning Electron Microscopy
<b>MMC</b>	Metal Matrix Composites
<b>CMC</b>	Ceramic Matrix Composites
<b>CCM</b>	Carbon Carbon Composites
<b>UTS</b>	Ultimate Tensile Strength
<b>TGA</b>	Thermogravimetric Analysis
<b>phr</b>	Parts per hundred

## LIST OF NOTATIONS

$\Sigma$	Ultimate tensile strength
$P_{\max}$	Maximum load
A	Area
R	Strain rate
Z	Rate of straining of the outer fiber
D	Depth of beam
L	Support span
B	Width of beam
D	Elongation
m	Slope of linear portion of stress-strain curve

## ABSTRACT

In the present work, hybrid composites were manufactured by using hot press machine incorporating at four levels of fiber loading (5, 10, 15 and 20%) with fiber ratios of (betel nut: glass=1:1) in polyethylene matrix. Composites were also prepared with betel nut and glass ratio 1:3 and 3:1 for 20% fiber loading. Subsequently, betel nut fiber was alkali treated by 5% NaOH and composites were prepared for treated betel nut and glass at a ratio of 1:3 and also by addition of nano-filler such as TiO<sub>2</sub> and zeolite nano-powder (2phr) for betel nut and glass ratio 1:3. The tensile, flexural, impact and hardness tests were conducted for mechanical characterization of composites. Tensile strength was decreased and young's modulus was increased with fiber loading. On the other hand, all the values of flexural strength, modulus and hardness were increased with fiber loading and filler addition. Interestingly, % elongation at break showed a decreasing trend with the amount of fiber content and filler addition in the composites. Furthermore, impact strength increased with fiber loading and filler content decreased as the glass fiber percentages increased. Notably, mechanical properties enhanced with increasing glass fiber content but incorporation of betel nut fiber with it makes the composite environmentally viable and less expensive. In addition, 5% NaOH treatment of the betel nut fiber provided an improving trend of properties of the composites. However, TiO<sub>2</sub> nano-powder addition makes the properties best among all composites. The Fourier Transform Infrared spectroscopy (FTIR) was carried out of the composites to study the effect of alkali treatment which showed decrease of hemicelluloses content and also for the presence of nano-filler. Scanning Electron Micrograph (SEM) imaging of tensile fracture surfaces of all the composites were carried out to examine the variation in failure modes and to investigate the interfacial adhesion and bonding between fiber and PE matrix, the composite containing titanium nano-powder showed favorable entanglement between fiber and matrix. Thermo Gravimetric Analysis (TGA) showed slight enhancement of thermal stability after alkali treatment.

# **CHAPTER 1**

## **INTRODUCTION**



## INTRODUCTION

A fiber reinforced polymer (FRP) is a composite material consisting of a polymer matrix embedded with high-strength fibers. Now-a-days hybrid FRP composites are receiving considerable attention by the world's scientific community for its capability of providing designers new freedom of tailoring composites and thus achieving properties that cannot be attained in binary systems containing one type of fiber dispersed in a matrix. It has also provided a more cost-effective utilization of expensive fibers by replacing them partially with less expensive fibers. Hybrid composites provide the potential of achieving a balanced pursuit of stiffness, strength and ductility, as well as bending and membrane related mechanical properties with weight savings, reduced notch sensitivity, improved fracture toughness, longer fatigue life and excellent impact resistance [1].

Today's world increasing environmental awareness has promoted designs that are compatible with environment, nontoxic to human body so eco-friendly. Natural fiber reinforced composites are inexpensive and could minimize environmental pollution due to their characteristics bio-degradability [2]. Apart from this, the lignocellulose fibers are lightweight, easily available renewable, non-abrasive, require less energy for processing, reduce the density of furnished products and absorbed CO<sub>2</sub> during their growth [1-3]. The lignocellulosic fibers can be mixed either with thermosetting or thermoplastic polymer matrix to produce composites [3]. On the other hand synthetic glass fibers provide excellent mechanical properties that are not achievable using only natural fiber. Thus incorporation of natural and glass fiber in one matrix to produce hybrid composite provides excellent combination of mechanical properties.

The physical and mechanical properties of lignocellulosic composites largely depend on the type of matrix, content and properties of the reinforcement fillers and filler–matrix interaction. However, the incompatibility between the hydrophilic natural fibers and the hydrophobic matrices leads to undesirable properties of the composites. This interfacial adhesion can be improved by the chemical modification of the fiber surface or the matrix. Apart from those the properties and interfacial characteristics of the composites are significantly influenced by the processing parameters used. Therefore, suitable processing techniques and parameters must be carefully selected in order to yield the optimum composite properties [3].

Polyethylene, often called polythene, is probably the most well-known plastic to the consumer and is used in greater volume worldwide than any other plastic. The total global production of plastics rose from 158 million tonnes to 180 million tonnes per annum between 1998 and 2000 – an increase of 12%. In Western Europe in 2000, polyethylene plastics represented 39% of standard plastics consumed, and totalled 11.7 million tonnes. Future annual demand growth for polyethylene plastics in Western Europe is predicted to be about 6%, with the major application continuing to be packaging. Typically over 50% of packaging plastics are consumed in the food retail and food services sectors. Polyethylene's dominance as a food packaging plastic is due to its relatively low cost, its range of versatile properties, and the ease with which it can be processed into the various packaging forms. Although polyethylene plastics were first produced over 50 years ago, manufacturing and processing developments continue to improve its properties, performance, and food packaging applications.

Polyethylene plastics have the generally advantageous properties of toughness, high tensile strength, and good barrier properties to moisture. A particularly important property of polyethylene plastics, which is due to their relatively low melting point ranges, is the ease with which packaging can be heat-sealed. The barrier properties of polyethylene plastics to oxygen and organic substances are only moderate. These characteristics, along with properties such as clarity and stiffness, vary with the basic polyethylene parameters of density/crystallinity, molecular weight, and molecular weight distribution [4]. Yield strength and stiffness together with thermal and other mechanical properties increase with density/crystallinity. Toughness and tensile strength also increase with molecular weight. The polyethylene's high degree of toughness at low temperatures is due to the very low glass transition temperatures, which are in the range of  $-80^{\circ}\text{C}$  to  $-120^{\circ}\text{C}$ . Clarity generally improves with decreasing density/crystallinity. The relationship between these physical properties and the density/crystallinity allows the polyethylene type to be identified fairly easily. For example, LDPE films tend to be soft and relatively clear, whereas films made from HDPE have a crisp feel and are more opaque [4].

A lot of research work has already been carried out with PE matrix. In one study, LDPE has been reinforced with hemp fibers [5].  $\text{TiO}_2$  nanoparticles was also used for reinforcing PE resin [6]. Epoxy based composite was also reinforced with bagasse-glass fiber and also with betel nut fiber [3,7]. In another study, epoxy matrix was reinforced with jute/E-glass fiber [8]. So combination of both natural and synthetic fiber is a good choice as seen from the previous work. Glass fiber based composite has been also prepared but they are expensive and not environmentally viable. So addition of natural fiber to it makes the composite

environmentally viable and less expensive which is a good option. Among various natural fibers, Betel nut (Areca) fiber is quite available in south Asia and is also cheap in context to the economic condition of Bangladesh. On the other hand, glass fiber consisting of extremely fine filaments of glass that are combined in yarn and woven into fabrics, used in masses as a thermal and acoustical insulator, or embedded in various resins to make boat hulls, fishing rods etc. Glass fibers are used extensively in printed circuit boards, industrial circuit breakers, and conduits for power cables [9]. Now-a-days nanotechnology is a promising approach toward developing advanced materials for future engineering applications. Polymer nano-composites possess better thermal, mechanical and chemical properties over the neat polymer without compromising the density, toughness and processibility [10].

As a result, the main aim of this research is to develop a new hybrid fiber (glass and betel nut) reinforced based polyethylene composite. The developed composite will be able to bear significant strength with less environmental impacts at lower cost. It is very important to replace synthetic fiber (glass) with natural fiber (betel nut) to make it environmentally viable at low cost. By investigating the effect of changing fiber fraction on the mechanical properties of the composites, optimum fiber/matrix ratio will be determined. This research will highlight the production route of an eco-friendly, cost effective hybrid composite combining the properties of a natural (betel nut) and a synthetic fiber (glass) on polyethylene matrix. Besides this, characterization study of the prepared composites will help to understand the structure-property relationship. Another purpose of this research is to characterize both raw and treated betel nut and glass fiber reinforced hybrid polyethylene composite and also the hybridization of the best ratio with addition of nanofillers such as  $\text{TiO}_2$  and zeolite powder. Finding out their morphological, chemical, physical and mechanical characteristics are another target of this research. The major steps of the research project are as follows:

Fabrication of raw betel nut and glass fiber reinforced hybrid polyethylene matrix composites containing different percentages of fibers (e.g. 5%, 10%, 15% and 20%) was done. In all cases ratio of the individual fiber (i.e. betel nut and glass fiber) kept constant (1:1). Fabrication of composites reinforced with betel nut and glass fibers with different ratio of betel nut and glass fiber for fixed percentages of fiber loading was also done. In these cases, optimum ratio was found out for the best combination of properties. Chemical (alkali) treatment of hydrophilic betel nut fibers was also done to enhance its compatibility with hydrophobic polyethylene matrix. Fabrication of composite reinforced with treated betel nut and glass fibers containing fixed wt% fiber was also done for the optimum ratio. Nano-composite was also prepared for this ratio by adding  $\text{TiO}_2$  and zeolite nano-powder (2phr). Mechanical properties of

composites such as tensile strength, young's modulus, flexural strength, modulus, impact strength and hardness were also measured. SEM analysis of tensile fracture surfaces of all types of composites were carried out accordingly. FTIR spectroscopy and TGA analysis of both raw and treated betel nut fiber reinforced composites were also performed.

The thesis is divided into a number of sections consisting literature review, experimental procedure, composites mechanical property characterization, their results and discussion, conclusion and references. In the literature review portion background will be studied. Experimental procedure will highlight the production route. In results and discussion, obtained results will explain with respect to graphs. Finally, the conclusion will provide the outcome or results.

Hopefully these composite can be used in producing mirror casing, paper weights, sailing boats, decks of fishing boats, mail-box, helmet, doors, windows and various parts of car interior together with truck cabins.

**CHAPTER 2**  
**LITERATURE REVIEW**

# LITERATURE REVIEW

Composites and its classification, betel nut fiber, glass fiber and its classification, thermoplastic matrix polyethylene and its classification, feasibility of composite production and its probable applications are discussed in the following section.

## 2.1 Composite

Composite is a multiphase material that is artificially made, as opposed to one that occurs or forms naturally. Composite materials are made from two or more constituent materials with significantly different physical or chemical properties, that when combined, produce a material with characteristics different from the individual components. The individual components remain separate and distinct within the finished structure. Generally, a composite material is composed of reinforcement (fibers, particles, flakes, and/or fillers) embedded in a matrix (polymers, metals, or ceramics). The matrix holds the reinforcement to form the desired shape while the reinforcement improves the overall mechanical properties of the matrix [11]. The numerous unique features of composite materials have led to the widespread adoption and use of it through worldwi many different industries. Below are some of the most important features of composites, and the benefits they provide [12].

- ❖ Composites are incredibly lightweight, a car made from composites weighs 1/4 that of a car made from steel. As a result of which it provides high strength to weight ratio and high stiffness to weight ratio. This amazing performance results in fuel saving and makes it cost effective. This lightweight property has made composites an ineradicable candidate in aerospace industry.
- ❖ The marine industry was one of the first to adopt the use of composites because of their high resistance to various types of chemicals and they do not get corroded in marine environment.
- ❖ Fiber reinforced composites have excellent elastic properties. Bending of metals results in its yielding or dent formation. However, when composites are bent, they want to naturally snap back into place. This feature is ideal for springs, and that is why composites are used in car leaf springs and in the limbs of archery bows.
- ❖ Certain composites, such as composite made with fiberglass, are non-conductive. This is important because often a structure requires combination of strength and non-conductivity of electricity.
- ❖ Composites may be made by a wide range of processes.

- ❖ Composite Laminate patterns and ply buildup in a part can be tailored to give the required mechanical properties in various directions.
- ❖ It is easier to achieve smooth aerodynamic profiles for drag reduction by using composites. Complex double-curvature parts with a smooth surface finish can be made in one manufacturing operation.

## 2.2 Types of Composites

The reinforcement system in a composite material strongly determines the strengthening mechanism in a composite. It is thus convenient to classify composites according to the characteristics of the reinforcement, such as length, orientation etc. and the matrix hold them in their place so there are two classification systems of composite materials. One of them is based on the matrix material (metal, ceramic and polymer) and the second is based on the material structure [1].

### 2.2.1 Classification Based on Reinforced Material

Based on reinforcing material structure, composites are divided into followings as shown in Figure 2.1.

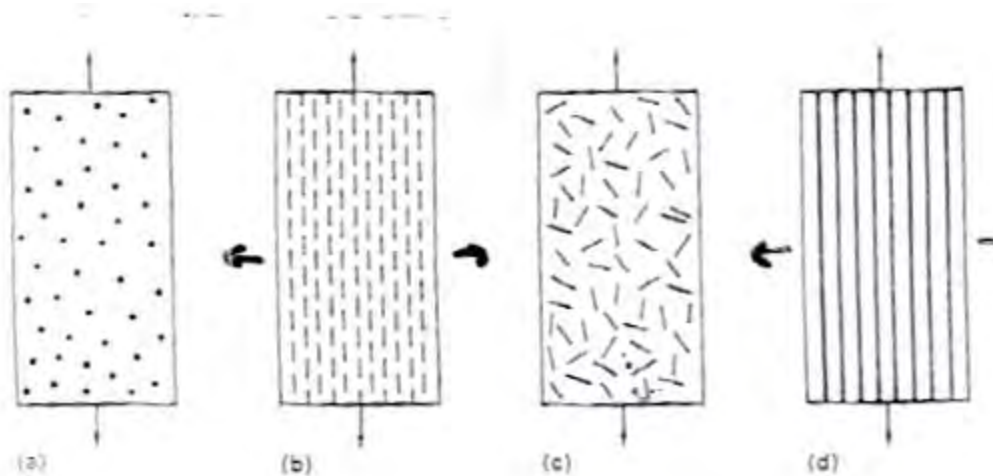


Figure 2.1: Types of composites based on fibers (a) Particulate and random; (b) Discontinuous fibers and unidirectional; (c) Discontinuous fibers and random; (d) Continuous fibers and unidirectional [13].

### Particulate Composites

Particulate composites consist of a matrix reinforced by a dispersed phase in form of particles. The particle diameter is typically in order of a few microns. In this case, the particles carry a major portion of the load. The particles are used to increase the modulus and decrease the ductility of the matrix. Particles

can have virtually any shape, size or configuration. Examples of well-known particulate composites are concrete and particle board.

The composite can be of following types:

- ❖ Composites with random orientation of particles.
- ❖ Composites with preferred orientation of particles. Dispersed phase of these materials consists of two-dimensional flat platelets (flakes), laid parallel to each other.

Particulate composites offer several advantages. They provide reinforcement to the matrix material thereby strengthening the material. The combination of reinforcement and matrix can provide very specific material properties. For example, the inclusion of conductive reinforcements in a plastic can produce plastics that are somewhat conductive. Particulate composites can often use more traditional manufacturing methods such as injection molding which reduces cost [14].

### **Fibrous Composites**

The fiber element performs as the main load-bearing component in fiber reinforced composites. Dispersed phase in form of fibers improves strength, stiffness and fracture toughness of the material, impeding crack growth in the directions normal to the fiber. Effect of the strength increase becomes much more significant when the fibers are arranged in a particular direction (preferred orientation) and a stress is applied along the same direction. Fibers come in three different forms: long, straight continuous fibers that are stacked parallel to each other; short chopped fibers that are randomly distributed through the composite and cloth woven fibers that give durability [15].

### **Short-Fiber Reinforced Composites**

Short-fiber reinforced composites consist of a matrix reinforced by a dispersed phase in form of discontinuous fibers (length  $< 100$ diameter). This type of composites can be of two types:

- ❖ Composites with random orientation of fibers.
- ❖ Composites with preferred orientation of fibers.

### **Long-Fiber Reinforced Composites**

Long-fiber reinforced composites consist of a matrix reinforced by a dispersed phase in form of continuous fibers. Matrix in such materials serves only as a binder of the fibers keeping them in a desired shape and protecting them from mechanical or chemical damages.



- ❖ Unidirectional orientation of fibers.
- ❖ Bidirectional orientation of fibers (woven).

### **Laminate Composites**

Laminate composites consist of layers with different anisotropic orientations or of a matrix reinforced with a dispersed phase in form of sheets. When a fiber reinforced composite consists of several layers with different fiber orientations, it is called multilayer (angle-ply) composite [11].

Laminate composites provide increased mechanical strength in two directions and only in one direction, perpendicular to the preferred orientations of the fibers or sheet, mechanical properties of the material are low.

### **2.2.2 Classification Based on Matrix Material**

Based on matrix material, composites are divided in to followings:

#### **Metal Matrix Composites (MMC)**

Metal matrix composite (MMC) are made by dispersing a reinforcing material (i.e. ceramic (oxides, carbides) or metallic (lead, tungsten, molybdenum) into a metallic matrix [11]. The reinforcement surface can be coated to prevent a chemical reaction with the matrix. For example, carbon fibers are commonly used in aluminum matrix to synthesize composites showing low density and high strength. However, carbon reacts with aluminum to generate a brittle and water-soluble compound  $Al_4C_3$  on the surface of the fiber. To prevent this reaction, the carbon fibers are coated with nickel or titanium boride. The major advantages of MMC's can be listed as follows:

- ❖ High strength even at elevated temperatures
- ❖ High stiffness (modulus of elasticity)
- ❖ High thermal conductivity
- ❖ Excellent abrasion resistance
- ❖ Good creep resistance
- ❖ High wear resistance
- ❖ High thermal stability

#### **Ceramic Matrix Composites (CMC)**

Ceramic matrix composite (CMC) is a material consisting of a ceramic matrix combined with a ceramic (oxides, carbides) dispersed phase. Ceramic matrix composites are reinforced by either continuous (long) fibers or discontinuous (short) fibers [11]. The motivation to develop CMCs was to overcome the

problems associated with the conventional technical ceramics like alumina, silicon carbide, aluminum nitride, silicon nitride or zirconia – they fracture easily under mechanical or thermo-mechanical loads because of cracks initiated by small defects or scratches. The crack resistance is very low like in glass. To increase the crack resistance or fracture toughness, particles (so called mono -crystalline whiskers or platelets) were embedded into the matrix.

Some of the typical properties of Ceramic matrix composites are:

- ❖ High mechanical strength even at high temperatures
- ❖ High thermal shock resistance
- ❖ High stiffness
- ❖ High toughness
- ❖ Low density
- ❖ High corrosion resistance even at high temperatures.

### **Polymer Matrix Composites (PMC)**

Polymer matrix composite (PMC) is the material consisting of a polymer (resin) matrix combined with a fibrous reinforcing dispersed phase. Polymer matrix composites are very popular due to their low cost and simple fabrication methods [12]. They adopt flat, gently curved or sharply sculpted contours with ease, providing manufacturers with design flexibility. In addition, composites offer the opportunity for parts consolidation and lower assembly costs. Polymer-matrix composites provide a stiff, lightweight alternative to steel, aluminum and traditional materials such as wood.

Reinforcement of polymers by strong fibrous network permits fabrication of PMC characterized by the following properties:

- ❖ High tensile strength
- ❖ High stiffness
- ❖ High fracture toughness
- ❖ Good abrasion resistance
- ❖ Good puncture resistance
- ❖ Good corrosion resistance
- ❖ Low cost

However, PMC has disadvantages like low thermal resistance and high coefficient of thermal expansion. Properties of PMCs are determined by properties of the fiber and matrix and also by concentration and orientation of fibers. Polymer composites are used to make very light bicycles that are faster and easier to

handle than standard ones, fishing boats that are resistant to corrosive seawater, and lightweight turbine blades that generate wind power efficiently. New commercial aircraft also contain more composites than their predecessors. A 555-passenger plane recently built by Airbus, for example, consists of 25 percent composite material, while Boeing is designing a new jumbo aircraft that is planned to be more than half polymer composites.

### **Carbon-Carbon Composites (CCMs)**

Carbon fiber-reinforced carbon (carbon-carbon, abbreviated C/C) is a composite material consisting of carbon fiber reinforcement in a matrix of graphite. Carbon-carbon composites are used in very high temperature environments of up to 6000°F and are twenty times stronger and thirty times lighter than graphite fibers. Carbon-carbon is well-suited to structural applications at high temperatures, or where thermal shock resistance and/or a low coefficient of thermal expansion are needed. While it is less brittle than many other ceramics, it lacks impact resistance [13].

### **2.3 The Matrix**

The constituent of fiber composites, the matrix serves two very important functions that is it holds the fibrous phase in place and under an applied force it deforms and distributes the stress to the high modulus fibrous constituent. The choice of a matrix for a structural fiber composite is limited by the requirement that it must have a greater elongation at break than the fiber.

The matrix must transmit the forces to the fibers and change shape as required to accomplish this, placing only tensile loading on the fibers. Apart from this it must protect the fibers from environment and abrasion with each other. Furthermore, during processing the matrix must encapsulate the fibrous phase without excessive shrinkage, which can place internal strain on the fibers. The matrix also helps to maintain the distribution of fibers as well as distribution of load evenly between fibers. It enhances some of the properties of the resulting material and structural component (that fiber alone is not able to impart). These properties are such as transverse strength of a lamina and Impact resistance. It also provides better finish to final product [14]. In order to accomplish this transfer of loads and also reduce the chance of failure in the matrix, adhesion to fibers or other reinforcements must be coupled with sufficient matrix shear strength to sustain these loads.

### **2.3.1 Matrix Material**

The four classes of matrix materials are polymers, metals, ceramics and carbon. The properties of the four types differ substantially. These differences have profound effects on the properties of the composites using them. Characteristics of key materials in each class are described below.

#### **Metal Matrix**

The metal matrix of MMC is a monolithic material into which the reinforcement is embedded, and is completely continuous. This means that there is a path through the matrix to any point in the material, unlike two materials sandwiched together. The principal matrix materials for MMCs are aluminium and its alloys. To a lesser extent, magnesium and titanium are also used as they provide a compliant support for reinforcement. For several specialized applications copper, zinc or lead matrix may be employed. In high temperature applications, cobalt and cobalt-nickel alloy matrices are common. These matrices provide higher transfer strength, toughness but are more susceptible to interfacial degradation at the fiber/matrix interface and to corrosion.

#### **Ceramic Matrix**

The key ceramics used as CMC matrices are silicon carbide, alumina, silicon nitride, mullite and various cements. The properties of ceramics, especially strength, are even more process sensitive than those of metals. These types of matrices can be used up to a high temperature range of  $>2000^{\circ}\text{C}$ , provide high elastic modulus and low density. Ceramics are very flaw sensitive, resulting in a decrease in strength with increasing material volume, a phenomenon called "size effect." As a result, there is no single value that describes the tensile strength of ceramics. In fact, because of the very brittle nature of ceramics, it is difficult to measure tensile strength and flexural strength (often called modulus of rupture).

#### **Carbon Matrix**

Carbon is a remarkable material. It includes materials ranging from lubricants to diamonds and structural fibers. The forms of carbon matrices resulting from the various carbon/carbon manufacturing processes tend to be rather weak, brittle materials. This type of matrix provides high heat absorption capacity and chemical inertness. Some forms have very high thermal conductivities. They have exceptional frictional properties at high energy levels and low coefficient of thermal expansion [15].

## **Polymer Matrix**

Polymer-matrix composites use matrices of various thermosets or thermoplastic resin. Thermoplastic soften upon heating and can be reshaped again whereas thermosets becomes cross-linked and do not soften upon heating. Problems with using polymer matrix materials are [16]:

- ❖ Limited temperature range of using.
- ❖ Susceptibility to environmental degradation due to moisture, radiation.
- ❖ Atomic oxygen (in space)
- ❖ Low transverse strength
- ❖ High residual stress due to large mismatch in coefficients of thermal expansion of both fiber and matrix
- ❖ Polymer matrix cannot be used near or above the glass transition temperature.

### **2.3.2 Types of Polymeric Matrix**

A polymeric solid material contains many chemically bonded parts or units that themselves are bonded together to form a solid. It is a chemical compound or mixture of compounds consisting of repeating structural units created through a process of polymerization. The atoms in the repeating molecules are strongly bonded (usually with covalent) and the bonds between molecules are weaker secondary bonds or similar covalent bonds.

Depending on how they are structurally and chemically bonded, plastics can be divided into two classes that are:

- ❖ Thermoplastics
- ❖ Thermosetting plastic (thermosets)

#### **2.3.2.1 Thermoplastics**

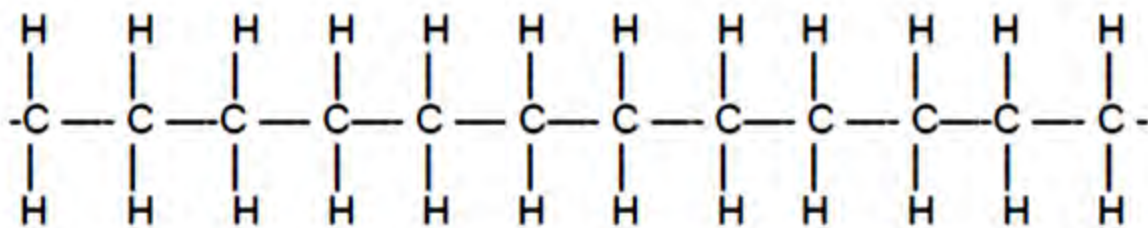
A thermoplastic is a type of plastic made from polymer resins that becomes pliable or moldable above a specific temperature and solidifies upon cooling. When frozen, however, a thermoplastic becomes glass-like and subject to fracture. These materials can be reheated and reformed into new shapes a number of times without significant change in their properties [16-17].

Thermoplastics are usually soft and ductile. Most thermoplastics are high-molecular-weight polymers whose chains associate through weak Vander Waals forces (polyethylene); stronger dipole-dipole interactions and hydrogen bonding (nylon); or even stacking of aromatic rings (polystyrene). Many thermoplastic materials are addition polymers; e.g., vinyl chain-growth polymers such as polypropylene.

Since most thermoplastics have weak Van Der Waal forces, the long molecular chains that make up thermoplastics clump together like lumps of entangled spaghetti. These long molecular chains give thermoplastic polymers the flexibility of candle wax [18]. Thermoplastic material provides high delamination resistance (high toughness), damage tolerance, low toxicity, low moisture absorption and high chemical resistance.

### 2.3.2.2 Polyethylene

Polyethylene is a simple example of a polymer, and is one of the most common and widely used polymers. Polyethylene has a number of different forms with different mechanical properties. The properties of the different forms of polyethylene are governed by branching, crystallinity and most of all molecular weight. Polyethylene is derived from either modifying natural gas (a methane, ethane, propane mix) or from the catalytic cracking of crude oil into gasoline. In a highly purified form, it is piped directly from the refinery to a separate polymerization plant. Here, under the right conditions of temperature, pressure and catalysis, the double bond of the ethylene monomer opens up and many monomers link up to form long chains. In commercial polyethylene, the number of monomer repeat units ranges from 1000 to 10 000 (molecular weight ranges from 28 000 to 280 000)[19].



**Polyethylene**

#### **Molecular Structure of Polyethylene**

The three main types of polyethylene are high density polyethylene (HDPE), low density polyethylene (LDPE), and linear low density polyethylene (LLDPE). These different types are based on the structure of the molecule of the polyethylene, which in turn effects its density, hence the name. The molecules of polyethylene are similar to long strands of spaghetti.

In LDPE some of the carbon atoms are attached to three carbon atoms instead of two, this causes a branch on which other polyethylene chains are attached as shown in Figure 2.2. These branches are relatively

long and intertwine and link with other polyethylene molecules. LDPE is like one long piece of spaghetti, with lots of large pieces of spaghetti attached to it[19].

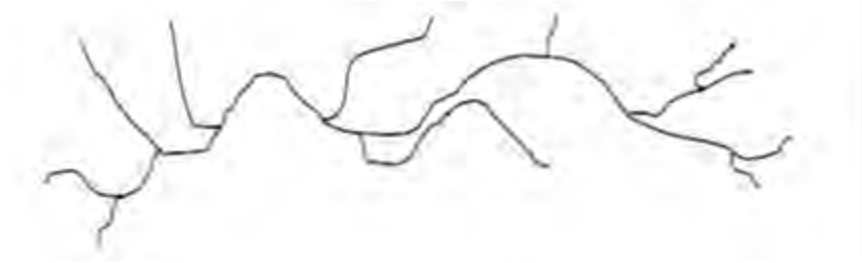


Figure 2.2: LDPE.

HDPE is a more linear molecule where there are very few side chains caused by branching, and these side chains are usually very short compared to LDPE. HDPE is like a normal piece of spaghetti with little bumps on it as shown in Figure 2.3[19].



Figure2.3: HDPE.

LLDPE as the name suggests is a more linear, low density polyethylene molecule. It still has a large number of side branches, however these branches are shorter. The molecules of LLDPE are like spaghetti with lots of very small pieces of spaghetti attached to it as shown in Figure 2.4[19].



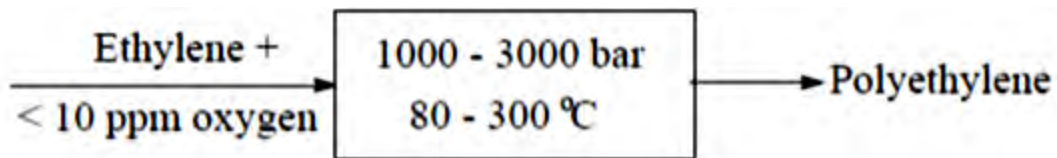
Figure2.4: LLDPE.

## Polyethylene Manufacturing Process

Today, polyethylene manufacturing processes are usually categorized into "high pressure" and "low pressure" operations. The former is generally recognized as producing conventional low density polyethylene (LDPE) while the latter makes high density (HDPE) and linear low density (LLDPE) polyethylene. The difference between these polyethylene processes and types is outlined below.

### High Pressure

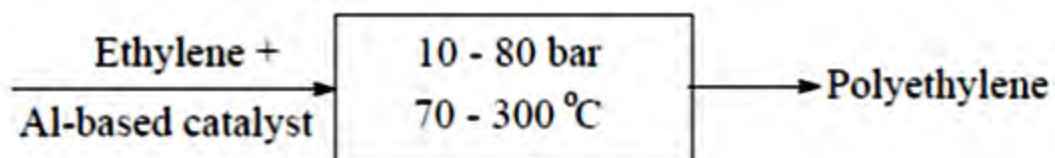
Polyethylene was first produced by the high pressure process by ICI, Britain, in the 1930's. They discovered that ethylene gas could be converted into a white solid by heating it at very high pressures in the presence of minute quantities of oxygen:



The polymerization reaction which occurs is a random one, producing a wide distribution of molecule sizes. By controlling the reaction conditions, it is possible to select the average molecule size (or molecule weight) and the distribution of sizes around this average (molecular weight distribution). The chains are highly branched (at intervals of 20 – 50 carbons). Figure 2.2 shows the molecular weight distribution and long chain branching for LDPE. ICI named their new plastic "polythene" and found that they were able to produce it in a density range of about 0.915 to 0.930 g/cc. It is known today as LDPE and has its single biggest usage in blown film.

### Low Pressure

The initial discovery of LDPE was an accident. So was the discovery of HDPE in 1952. Researchers in Germany and Italy had succeeded in making a new aluminium based catalyst, which permitted the polymerization of ethylene at much lower pressures than the ICI process:





The polyethylene product of this process was found to be much stiffer than any previous with a density range of about 0.940 - 0.970 g/cc. The increased stiffness and density were found to be due to a much lower level of chain branching. The new HDPE was found to be composed of very straight chains of ethylene with a much narrower distribution of molecular weights (or chain lengths) and a potentially very high average chain length. Figure 2.3 shows the molecular weight distribution and short chain branching for HDPE. In the late 1950's, DuPont Canada first applied the low pressure process to the production of LLDPE. LLDPE is made by copolymerizing with a small amount of another monomer, typically butene, hexene or octane as shown in Figure 2.5 [20].

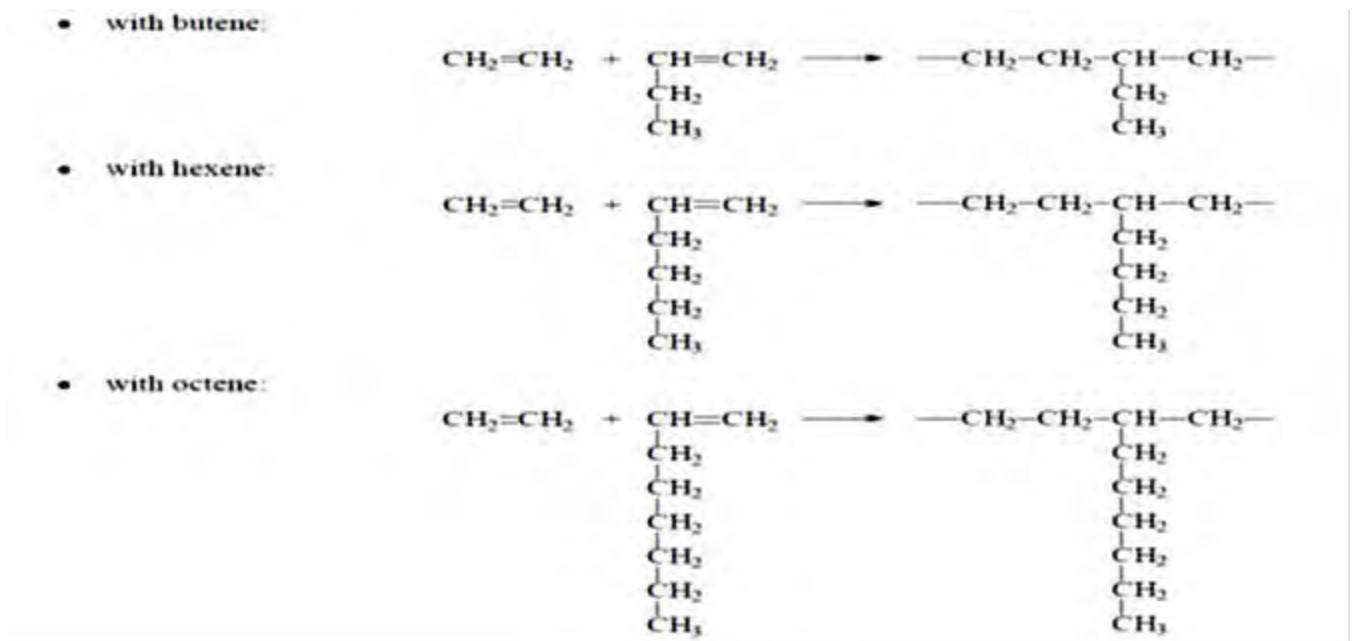


Figure 2.5: Low Pressure process

### Properties of Polyethylene

Making small changes to these molecules brings about the ability to make a huge range of characteristics. The mechanical, chemical, electrical and thermal properties as shown in Table 2.1 can be varied in a huge range of possibilities, which is usually done by changes during manufacture. For all of the types of polyethylene they have the following properties:

- ❖ Polyethylene is an insulator, which means that it does not conduct electricity. In fact polyethylene is one of the best insulating materials available.

- ❖ Polyethylene is chemically stable and inert. This means that they do not readily react with other chemicals.
- ❖ Polyethylene is strong and lightweight.
- ❖ Due to the differences in structure, HDPE is stronger and has a higher melting point than LDPE, with LLDPE generally between the two.

Polyethylene's are easy to distinguish from other plastics because they float in water. Polyethylene provide good resistance to organic solvents, degreasing agents and electrolytic attack. They have a higher impact strength, but lower working temperatures and tensile strengths than polypropylene. They are light in weight, resistant to staining and have low moisture absorption rates.

Table 2.1: Properties of polyethylene [21]

TYPICAL PROPERTIES OF POLYETHYLENE				
	Property	LDPE	HDPE	UHMW
PHYSICAL				
D792	Density(g/cc)	0.92	0.95	0.93
D570	Water Absorption, 24hrs(%)	<0.01	0	0
MECHANICAL				
D638	Tensile Strength(Psi)	1800-2200	4600	3100
D638	Tensile modulus(Psi)	---	---	125000
D638	Tensile Elongation at yield(%)	600	900	---
D790	Flexural Strength(Psi)	---	---	---
D790	Flexural Modulus(Psi)	---	200000	125000
D695	Compressive Strength(Psi)	---	---	2000
D695	Compressive Modulus(Psi)	---	---	---
D785	Hardness, Shore D	D41-D50	D69	D62-D66
D256	IZOD Notched Impact	No Break	3	No Break

### **2.3.2.3 Thermosetting Plastic**

A thermosetting plastic, also known as a thermoset, is polymer material that irreversibly cures. It is a pre-polymer in a soft solid or viscous state that changes irreversibly into an infusible, insoluble polymer network by curing. Curing can be induced by the action of heat or suitable radiation, or both. Thermosetting plastics are made up of lines of molecules which are heavily cross-linked. It creates a rigid molecular structure. They may be heated the first time and shaped but they become permanently stiff and solid. They cannot be reshaped again [16].

Most thermosets consist of a network of carbon atoms covalently bonded to form a rigid solid. Sometimes nitrogen, oxygen, sulfur or other atoms are also covalently bonded into a thermoset network structure. Thermosets are generally stronger than thermoplastics due to their three dimensional network of bonds and better suited to high temperature applications.

### **2.3.2.4 Advantages of Thermoplastics over Thermosets**

As stated earlier, thermoplastics can be repeatedly softened by heating, and hardened by cooling. Thermoplastics possess several advantages over the thermosets, one of the most important being that they do not need storing under refrigeration. They also possess improved damage tolerance, environmental resistance, fire resistance, recyclability and potential for fast processing. Primary reason for the use of thermoplastics is their cost effective processing. Increased use for thermoplastics is also due to three different reasons as stated below [22]:

- ❖ Processing can be faster than that of thermoset composites since no curing reaction is required. Thermoplastic composites require only heating, shaping and cooling.
- ❖ The properties are attractive, in particular, high delamination resistance and damage tolerance, low moisture absorption and the excellent chemical resistance of semi-crystalline polymers.
- ❖ In the light of environmental concerns, thermoplastic composites offer other advantages also. They have low toxicity since they do not contain reactive chemicals (therefore storage life is infinite).
- ❖ Using of thermoplastic provides aesthetically superior finishes and has high impact resistance.

## **2.4 Reinforcement of Composite Material**

The main role of the reinforcement in a composite material is improving the mechanical properties of the neat resin system. Fibers or reinforcements carry load along the length of the fiber to provide strength and

stiffness in one direction. Reinforcements can be oriented to provide tailored properties in the direction of the loads imparted on the end product. All of the different fibers used in composites have different properties and so affect the properties of the composite in different ways. However, individual fibers or fiber bundles can only be used on their own in a few processes such as filament winding. For most other applications, the fibers need to be arranged into some form of sheet, known as a fabric, to make handling possible. Different ways for assembling fibers into sheets and the variety of fiber orientations possible lead to there being many different types of fabrics, each of which has its own characteristics. The four main factors that govern the fiber's contribution are [23]:

- ❖ The basic mechanical properties of the fiber itself.
- ❖ The surface interaction of fiber and resin.
- ❖ The amount of fiber in the composite.
- ❖ The orientation of the fibers in the composite.

#### **2.4.1 Natural Fiber Reinforced Polymer Composite**

Natural fiber composite mean a composite material that is reinforced with fibers, particles or platelets from natural or renewable resources. Natural fibers will play a key role in the emerging “green” economy based on energy efficiency. Natural fibers are ecofriendly because of their renewability. They are also carbon neutral. They absorb the same amount of carbon dioxide they produce. During processing, they generate mainly organic wastes and leave residues that can be used to generate electricity or make ecological housing material. And, above all they have the superior quality of biodegradability [2].

Food and Agriculture Organization(FAO) estimated that production of one tone of jute fiber requires 10% of the energy used for the production of one tone of synthetic fibers (since jute is cultivated mainly by small-scale farmers in traditional farming systems, the main energy input is human labor, not fossil fuels). Processing of some natural fibers can lead to high levels of water pollutants, but they consist mostly of biodegradable compounds, in contrast to the persistent chemicals, including heavy metals, released in the effluent from synthetic fiber processing. Natural fibers have good mechanical strength, low weight and low cost. Natural fibers are now-a-days quite an attractive choice in automobile industry. In Europe, car makers are using an estimated 80,000 tons of natural fibers a year to reinforce thermoplastic panels. India has developed composite boards made from coconut fiber that are more resistant to rotting than teak [24].Here, advantages of natural fiber reinforced composites are mentioned below:

- ❖ Reduction in density of products.
- ❖ Acceptable specific strength, toughness and stiffness in comparison with glass fiber reinforced composites.
- ❖ Ease of shaping into complex shapes in a single manufacturing process.
- ❖ Lower energy consumption from fiber growing to finished composites.
- ❖ The manufacturing processes are relatively safe when compared with glass based reinforced composites.
- ❖ Huge possibility of recycling the cuttings and wastage produced during manufacturing and moulding.
- ❖ The production of natural fibers can be started with a low capital investment and with a lower cost.

Among various natural fibers, betel nut (areca) fiber is quite available and cheap in context to the economic condition of Bangladesh.

#### **2.4.2 Natural Fiber vs Synthetic Fiber**

Both natural and synthetic fibers have advantages and drawbacks. Synthetic fiber-reinforced thermoplastic composites have better mechanical properties than the natural fibers, but they are not environmentally friendly. Some advantages of natural fibers are low abrasion resistance, low density, high toughness, acceptable specific strength properties, good thermal properties, enhanced energy recovery, biodegradability and so on [2]. Natural fibers produce composites that offer advantages like renewability of the fibers, good sound abatement capability and improved fuel efficiency. On the other hand, synthetic FRPCs attracted much attention due to their high strength, better durability and moisture resistance properties. The mostly used synthetic fibers in composites are glass, carbon and aramid. Among the synthetic fibers, glass fibers are widely used due to their low-cost (compared to carbon and aramid) and better physico-mechanical properties. Besides, the greatest problem of using such materials is the convenient disposal of themselves, once they have come to the end of their useful life span [25].

### **2.5 Betel Nut and Glass Fiber Reinforced Hybrid Composite**

#### **2.5.1 Hybrid Composites**

The incorporation of several different types of fibers into a single matrix has led to the development of hybrid composites. The behavior of hybrid composites is a weighed sum of the individual components in which there is a more favorable balance between the inherent advantages and disadvantages. Also, using a hybrid composite that contains two or more types of fiber, the advantages of one type of fiber could

complement with what are lacking in the other. As a consequence, a balance in cost and performance can be achieved through proper material design. The properties of a hybrid composite mainly depend upon the fiber content, length of individual fibers, orientation, extent of intermingling of fibers, fiber to matrix bonding and arrangement of both the fibers [26].

Hybrid composite materials have extensive engineering application where strength to weight ratio, low cost and ease of fabrication are required. Hybrid composites provide combination of properties such as tensile modulus, compressive strength and impact strength which cannot be realized in composite materials. In recent times hybrid composites have been established as highly efficient, high performance structural materials and their use is increasing rapidly. Hybrid composites are usually used when longitudinal as well as lateral mechanical performances are required.

There is a steady increase both in the number of applications being found for fiber reinforced plastics and, concurrently, in the variety of fiber/resin systems that are available to designers. As a result these systems provide wide range for designers to tailor the properties of such materials by using hybrid composite to suit specific requirements. In highly specialized situations where limitations such as high cost and brittle fracture behavior are considered secondary to such qualities such as low density, high rigidity and high strength hybrid composites have found wide application. There are many situations in which, for example, a high modulus material is required but in which the catastrophic brittle failure usually associated with such a material would be unacceptable. In the case of a strut member, a high initial modulus followed by limited yielding of the material and accompanied by the smallest possible reduction of load carrying capacity is usually desirable.

Hybrid composites can be made in two separate ways either by intimately mingling the fibers shown in a common matrix, or by laminating alternate layers of each type of composite. In principle several different types of fiber can be incorporated into a hybrid system. But in practice it is likely that a combination of only two types of fiber would be of most [26].

### **2.5.2 Betel Nut Fiber**

The areca nut is the seed of the areca palm (*Areca catechu*), which grows in much of the tropical Pacific, Asia, and parts of east Africa. It is commonly referred to as betel nut, as it is often chewed wrapped in betel leaves. The areca nut is not a true nut, but rather a fruit categorized as a drupe. It is commercially available in dried, cured and fresh forms. When the husk of the fresh fruit is green, the nut inside is soft

enough to be cut with a typical knife. In the ripe fruit, the husk becomes yellow or orange and, as it dries, the fruit inside hardens to a wood-like consistency [27]. The betel nut husk is the fibrous part of the fruit, which equals to approximately 60 to 80 % of total volume and weight of betel nut. The chemical composition and properties of betel nut fiber are shown in Tables 2.2 and 2.3 respectively.

Table 2.2: Chemical composition of betel nut fiber [28]

Component	Weight (%)
Lignin	7.20
$\alpha$ -cellulose	53.20
Hemicellulose	32.98
Pectin	9.2-15.4
Ash	1.05
Waxy materials	0.08
Other materials	3.04

Table 2.3: Comparison of betel nutfiber properties with other natural fibers [29-30]

Properties	Areca Nut	Betel nut fiber	Coir fiber	Banana fiber	Sisal fiber	Flax fiber	Jute fiber
Density(g/cc)	0.7-0.8	0.34	1.15	1.35	1.45	1.4-1.5	1.3-1.45
Diameter(mm)	.396-.476	0.45-0.53	0.1-0.45	0.08-0.25	0.05-0.20		0.025-0.2
Tensile Strength (MPa)	147-322	166.03	106-175	54-754	568-640	500-900	393-773
Young's Modulus (GPa)	1.12-3.16	1.381	4-6	7.7-20	9.4-15.8	50-70	13-26.5
Elongation at break (%)	10.23-13.15	23.21	17-47	10-35	3-7	3.27	1.16-1.5

### 2.5.3 Glass Fiber

A material consisting of extremely fine filaments of glass that are combined in yarn and woven into fabrics, used in masses as a thermal and acoustical insulator, or embedded in various resins to make boat hulls, fishing rods, and the like. Fiberglass materials are popular for their attributes of high strength compared to relatively light weight. Fiberglass really is made of glass, similar to windows or the drinking glasses. The glass is heated until it is molten, then it is forced through superfine holes, creating glass filaments that are very thin – so thin they are better measured in microns.

#### 2.5.3.1 Classification of Glass Fiber

There are different types of glass fibers depending on the composition. According to the requirement, any types of glass fibers can be used. Common classes of glass fibers are mentioned below:

**A-glass** ('A' for alkali-lime) or soda-lime glass

**E-glass** ('E' for electrical) which is alumino-borosilicate glass with less than 1% w/w alkali oxides and is mainly used for glass-reinforced plastics. (max use)

**C-glass** ('C' for chemical resistance) is alkali-lime glass with high B<sub>2</sub>O<sub>3</sub> content; used mainly for staple fibers and insulation.

**D-glass** ('D' for dielectric constant) is a borosilicate glass.

**R-glass** ('R' for reinforcement) is alumino silicate glass without MgO and CaO with high mechanical requirements.

**S-glass** ('S' for stiff) is alumino silicate glass without CaO but with high MgO content with high tensile strength.

**T-glass** ('T' for thermal insulator) is a glass which is resistant to chemical attack and is often used in thermal insulation-grades of blown fiber glass [31].

#### 2.5.3.2 Properties of Glass Fiber

Now-a-days, versatility of glass fiber makes it unique in industrial textile material. The main properties of glass fibers are mentioned as follows:

**Dimensional Stability:** Glass fiber is a dimensionally stable engineering material. Glass fiber does not stretch or shrink after exposure to extremely high or low temperatures. The maximum elongation for "E" glass at break is 4.8% with a 100% elastic recovery when stressed close to its point of rupture.



**Moisture Resistance:** Glass fibers do not absorb moisture or change physically or chemically when exposed to water.

**High Strength:** The high strength-to-weight ratio of glass fiber makes it a superior material in applications where high strength and minimum weight are required. In textile form, this strength can be unidirectional or bidirectional, allowing flexibility in design and cost.

**Fire Resistance:** glass fiber is an inorganic material and will not burn or support combustion. It retains approximately 25% of its initial strength at 1000°F (540°C).

**Chemical Resistance:** Most chemicals have little or no effect on glass fiber. The inorganic glass textile fibers will not mildew or deteriorate. Glass fibers are affected by hydrofluoric, hot phosphoric acids and strong alkaline substances.

**Electrical Properties:** Glass fiber is an excellent material for electrical insulation. The combination of properties such as low moisture absorption, high strength heat resistance and low dielectric constant makes fiber glass fabrics ideal as a reinforcement for printed circuit boards and insulating varnishes [31]. Important properties of betel nut and glass fiber are mentioned in Table 2.4.

Table 2.4: Properties of betel nut and glass fiber [31, 32].

Properties	Betel Nut fiber	Glass fiber
Density(g/cm <sup>3</sup> )	0.34	
Diameter(mm)	0.45-0.53	
Tensile Strength(MPa)	166.03	2000
Young's Modulus(GPa)	1.381-1.93	72.3
Elongation at break[%]	23.21	4.8

## 2.6 Chemical Treatment of Natural Fiber

The main component of natural fiber is cellulose. The elementary unit of cellulose macromolecule is anhydro-d-glucose, which contains 3 hydroxyl group (-OH). These hydroxyl form hydrogen bond inside the molecule itself (intra molecular) and between other cellulose molecules (intermolecular), as well as with other hydroxyl group from the air. Therefore all natural fibers are hydrophilic that is they absorb water [30].

Natural fiber due to its hydrophilic nature, leads to an adhesion problem with hydrophobic nature of the polymer matrix. Hydrophilic character of natural fibers is incompatible with hydrophobic polymer matrix

and has a tendency to form aggregates [2]. Furthermore, it exhibits poor resistance to moisture, which leads to high water absorption, subsequently resulting in poor mechanical properties and dimensional stability of the natural fiber reinforced composites. Therefore, chemical modifications of either natural fiber, polymer matrix or both materials are the alternative solutions to overcome these challenges. The chemical modification is attempted to improve hydrophobic nature of natural fiber, interfacial bonding between matrix and fiber, surface roughness and wettability, and also decrease moisture absorption which leads to the enhancement of mechanical properties of the natural fiber reinforced composites.

Alkaline treatment or mercerization is one of the most used chemical treatments of natural fibers when used to reinforce thermoplastics and thermosets. The important modification done by alkaline treatment is the disruption of hydrogen bonding in the network structure, thereby increasing surface roughness. This treatment removes a certain amount of lignin, wax and oils covering the external surface of the fiber cell wall, depolymerizes cellulose and exposes the short length crystallites [33]. Addition of aqueous sodium hydroxide (NaOH) to natural fiber promotes the ionization of the hydroxyl group to the alkoxide:  
$$\text{Fiber-OH} + \text{NaOH} = \text{Fiber-O}^- \text{Na}^+ + \text{H}_2\text{O}$$

Thus, alkaline processing directly influences the cellulosic fibril, the degree of polymerization and the extraction of lignin and hemicellulosic compounds. It is reported that alkaline treatment has two effects on the fiber:

- ❖ It increases surface roughness resulting in better mechanical interlocking.
- ❖ It increases the amount of cellulose exposed on the fiber surface, thus increasing the number of possible reaction sites.

Consequently, alkaline treatment has a lasting effect on the mechanical behavior of fibers, especially on fiber strength and stiffness.

Apart from alkali treatment various types of chemical treatment have been carried out by other researchers. In one study abaca fiber was treated with benzene diazonium salt [34]. In another study, jute fibers were chemically treated with copper sulfate ( $\text{CuSO}_4$ ) and sodium carbonate ( $\text{Na}_2\text{CO}_3$ ) for rot retardant (RR) treatment, while diammonium hydrogen phosphate ( $\text{NH}_4\text{H}_2\text{PO}_4$ ) and lissapol were used for fire retardant (FR) treatment [35]. In previous research, flax was treated with propyl-trimethoxysilane [36], coir was chemically treated with stearic acid as compatibilizer [37] and jute fiber was chemically modified by using silane coupling agent [38].

## 2.7 Addition of TiO<sub>2</sub> and Zeolite Nano-powder

Various properties of polymeric systems can often be improved with the addition of organic or inorganic fillers. The use of particulate materials for enhancement of polymer properties dates back to the earliest years of the polymer industry. Initially used as extending agents to reduce the cost of polymer-based products, fillers were soon recognized to be an integral component in many applications involving polymers, particularly in reinforcement [39-42]. In spite of the wide spread use of polymer composites throughout the polymer industry, a satisfactory understanding of the fundamental mechanism of the properties of these materials has eluded researchers.

The mechanism of reinforcement in filled polymer composites depends on various factors, including the properties of the polymer and filler, size and shape of the filler (particulate, fibrous, fabric, etc), phase state of the polymer (crystalline, rubbery, etc.), process by which the filled polymer composite is manufactured, and the nature of the interphase between the polymer matrix and the filler. While all these factors, and more, have an effect on the final product and the stress of matrix is partially transferred to the filler. Since, improved strength and stiffness are typically the properties of interest in the final composite, this effort has usually centered on a search for improved adhesion between the filler and polymer matrix. Generally, this search is for a coupling agent [43] or compatibilizer [44] that bridges between the matrix and filler phases by providing improved adhesion. The quest for improved compatibilizers [44] has attracted the talents of many researchers.

However, many of the fillers used, such as inorganic clays or layered silicates are immiscible with the polymer matrix. This leads to aggregation of the filler particles that can seriously hinder the property improvements of the composite. Often, surface modification of the filler particles is carried out to decrease the disfavorable interactions between the filler and the polymer matrix, thusly creating a finer and more homogenous dispersion within the composite material that leads to greater enhancement of the target properties. Though modified filler is suitable for property enhancement, still commercially many fillers used in its original form [45].

On the other hand, titanium being the ninth abundant metal in the earth's crust is known for its toughness, high strength and can be typically thought of as being chemically inert. It is usually available in several kinds of rocks and mineral sands in the oxidized state as Titanium dioxide (TiO<sub>2</sub>) which is a white, solid, non-hazardous, inorganic substance. Titanium dioxide is usually manufactured as a nanomaterial and as a pigment grade, both having similar production processes. Pigment grade TiO<sub>2</sub> (200nm-350nm) are used

where white opacity and brightness are the parameters of interest as pigment grade TiO<sub>2</sub> has excellent light-scattering properties. Nano TiO<sub>2</sub> (less than 100nm is size) provides ultraviolet absorption in spite of appearing transparent and is not inert [46].

Applications of nanoTiO<sub>2</sub> can be classified into two categories. Firstly, application of nano TiO<sub>2</sub> as a catalyst or semiconductor and secondly as ultraviolet light attenuator. As photocatalysts, nano TiO<sub>2</sub> is being used in various engineering and chemistry applications and as semiconductors it is being applied in electronics field. Selective catalytic reduction (SCR) systems which are used in large industrial boilers, combustion plants and stationary or automotive diesel engines convert harmful nitrous oxides (NO<sub>x</sub>) into harmless nitrogen and oxygen. Nano TiO<sub>2</sub> photo catalysts are highly dispersed and are sensitive to visible light and are used in manufacturing self- cleaning surfaces as on glass. Recently, nano TiO<sub>2</sub> is being used in solar cells for its electron transfer properties. Material pollutants being a major concern today, research has been conducted to study the effect of nano TiO<sub>2</sub> on the environment [47].

Another filler material is Zeolite. Zeolites are porous, hydrated aluminosilicates. They may be natural minerals or synthetic materials. The general chemical composition of a zeolite is:



Where M = e.g. Na<sup>+</sup>, K<sup>+</sup>, Li<sup>+</sup>, Ag<sup>+</sup>, NH<sub>4</sub><sup>+</sup>, H<sup>+</sup>, Ca<sup>2+</sup>, Ba<sup>2+</sup>...

Zeolite is a spectrum of inorganic materials known in diverse applications such as molecular sieves, catalysis, and ion exchange materials. But they are also known as a filler in thermoplastic matrices. Crystalline zeolite is a framework of alumino-silicates based on infinitely extending three-dimensional networks of AlO<sub>4</sub> and SiO<sub>4</sub> tetrahedral linked to each other by the sharing of all oxygen's [48]. In this present research, the effect of titanium and zeolite (2phr) nano-powders are investigated by manufacturing composites for optimum ratio.

## 2.8 Review of Previous Research

Composite materials with thermoplastic matrices and a reinforcement of natural fiber is always regarded as an important structural application source of biodegradable and renewable material. A large number of natural fibers like coir, jute, abaca, areca, flax, sisal has always been incorporated in thermoplastic polymeric matrices most commonly polypropylene and polyethylene.

Recently, betel nut fibers are used as a new reinforcing materials by many researchers. Srinivasaet al. valuate physical, flexural and impact properties of composites reinforced by areca fibers with urea formaldehyde, melamine urea formaldehyde and epoxy resins using compression molding technique [27]. Jayamaniet al. Investigated the effect of fiber surface treatment by sodium hydroxide on mechanical, acoustical and thermal properties of betel nut Fiber reinforced polyester composites [49].

Properties of fiber-reinforced PE composites (FRPCs) have been studied by many researchers. Much of the research has concentrated on determining the physical and mechanical properties of fiber reinforced composites and the influence of different parameters on their mechanical behaviour. The results show that a wide range of different properties and performance may be designed by altering various production parameters, such as thickness of the fibers used, relationship between fiber/matrix ratio, polymeric film used and number of layers.

Elinas studied the mechanical properties of hemp fibers with Low density polyethylene and found best property for 5% fiber loading [5]. In another study, Panin studied the effect of hydroxyapatite nanoparticles by  $N^+$  ion implantation to improve tribotechnical properties of UHMWPE[50]. Vu Manh Tuan and Da woonjeong investigated the effect of adding rutile  $TiO_2$  nanoparticles for reinforcing high density polyethylene resin[6]. Ahmed experimented the effect of adding powder walnut shells on the mechanical properties and flame resistance for LDPE [51]. In other study, Pegorettic investigated the tensile mechanical response of PE-clay nano-composites[52]. Runzhou and Xu studied the effect of individual and combined effect of talc and glass fibers on the mechanical and thermal expansion performance of the filled HDPE[53]. Noorunnisakhanam also manufactured and characterized polyethylene based composites [54]. In another study Enriquez, Joseph King Eos Dawn V., et al. studied coir reinforced high density polyethylene composites. In these research stearic acid was used as the compatibilizer [37]. Effects of rice husk filler on the mechanical and thermal properties of liquid natural rubber compatibilized high-density polyethylene/natural rubber blends were investigated by Jamil et al [55].

Hybrid composites are recently getting considerable attention from world's scientific community. Using both natural fiber as reinforcing materials provide a biodegradable material with a favourable balance of properties. Reinforcing with a natural and synthetic fiber provides a favourable balance of both of their properties. Though natural fibers are extremely acceptable for their characteristic renewability and

biodegradability, synthetic fiber are quite exceptional in terms of their extraordinary mechanical properties.

Lot of research work has been already done by using glass fiber as a one of the hybrid fiber. Joseph et al. compared the mechanical properties of phenol formaldehyde composite reinforced with banana fibers and glass fibers [56]. In another study, Khalil et al. studied oil palm fiber/glass hybrid reinforced polyester composites [57]. Researchers also studied flax/glass, carbon/glass, jute/glass and basalt/glass reinforced hybrid composites [58-61]. Reddy et al. studied performance of hybrid betel nut (*Areca Catechu*) short Fiber/*sansevieria Cylindrica* (*Agaveceae*) reinforced epoxy composites [7]. Senthil et al. investigated various combination of nano-materials on different types of matrix [62]. Rohchoon and Jyongisk investigated the effect of stacking sequence of aramid UHMPE hybrid composites by flexural test method [63]. Manush and Singh studied the mechanical properties of bagasse-glass fiber on epoxy composite [3]. Pichireddy and Rao investigated the tensile and flexural strength of glass fiber epoxy composites [11]. In another study, Ban and Hashem studied the effect of glass fiber orientation on the mechanical properties of composites [12]. Ishaya and Josiah studied the effect of OPEBF/banana/glass fiber reinforced unsaturated polyester composites [14]. In one study, Samadhangarad conducted experimental analysis of glass fiber reinforced composites beams [15]. Rahman and Vijayarangari studied the thermal and mechanical properties of woven glass fiber reinforced epoxy composites. They also added carbon nano-tube [16]. Hussain and Pandurangadu also further investigated the machinability of glass fiber based plastic composites [17]. Vinay and Govindaraju also characterized vinyl ester based composites based on glass and carbon fiber [18]. Sanjay and Yogesha reinforced epoxy matrix with jute/E-glass fiber and also investigated their mechanical properties [8]. Mathapati and Shivukumar also did the same type of work [64].

Now-a -days nanostructured fillers are also been used as the way of reinforcement. In one study Luyt. et al. evaluated mechanical and thermal properties of nanostructured wollastonite filler reinforced polypropylene composites [65]. Mohd, et al. studied mechanical and morphological properties of treated tropical wood sawdust polypropylene composites [66]. In one study, Deogonda, Kumar and chawla studied the effect of glass fiber reinforcement on epoxy matrix with the addition of  $TiO_2$  and  $ZnS$  fillers. In another one, Biswas. et al. investigated the structural effects on the tensile and morphological properties of zeolite filled PP derivative composites [48].

## 2.9 Feasibility of Preparing Betel Nut and Glass Fiber Reinforced Hybrid Polyethylene Composites

To give a short summary on the position of FRP composites as a material used in the industry this section was added. First of all a graphic is given on the total market volume of fiber reinforced polymers as shown in Figure 2.6. It is clearly visible that since the 1970's the FRP market volume has almost quintupled. The total volume of FRP composites used is about 5.5 million tons.

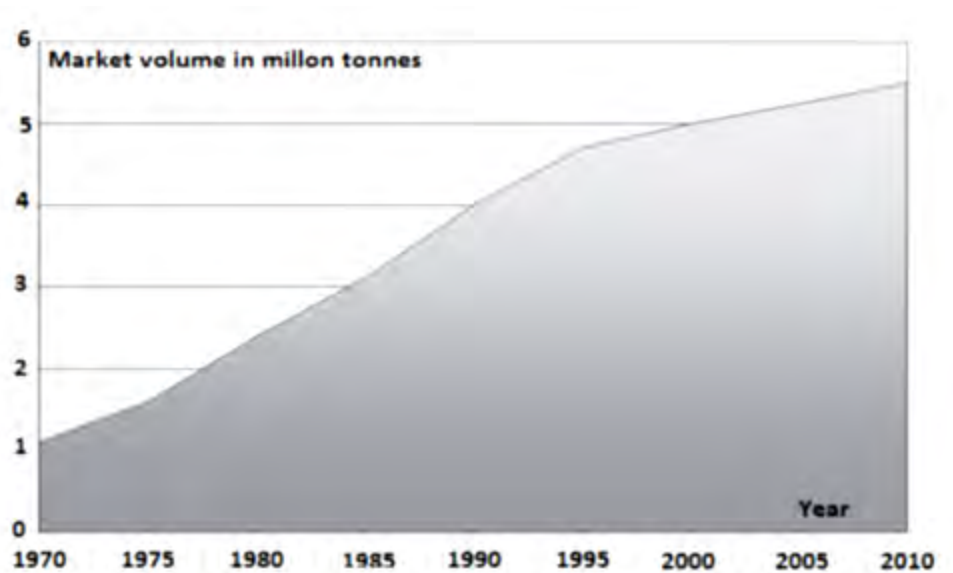


Figure 2.6: Fiber reinforced polymer composites market volume from 1970 to 2010[67]

The idea of using fine glass fibers in weaving processes to produce textile glass was developed by the French physicist Ferchault de Réaumur in the late 18th century. However industrial production of continuous glass fibers only started in 1935 in Newark, Ohio, USA when the company Owens and Corning developed a production method suitable for industrial production. Production in Europe started only 3 years later in Germany. The first industry that used glass fibers was the electrical industry, hence the common name the glass fiber type “E-glass”. Since this the 1930's glass fiber production has undergone a constant evolution of better products and more efficient production.

Glass fibers are the most widely used reinforcements; over 90% of all FRPs are made of glass fiber. The oldest and still most popular form is E-glass or electrical grade glass. Other types of glass fibers are A-glass or alkali glass, C-glass or chemical resistant glass, and the high strength R-glass and S-glass. Under laboratory circumstances glass fibers can resist tensile stresses of about 7000 N/mm<sup>2</sup>, whereas commercial glass fibers reach 2800 to 4800 N/mm<sup>2</sup>[67].

According to a study conducted by Economics of areca nut cultivation in Karnataka, India in 2001 77.80 HA areas of Bangladesh were used for areca cultivation and the total production was 47 tonnes and at 2009 The total area of areca cultivation enhanced to 79 HA and total production increased to 56 tonnes So the productivity increase was 604 kg/HA to 709 kg/HA [68].

According to today's costing glass fiber costs US \$2 -2.5 / Kilogram [69]. So around 160-200 BDT/kg, whereas in local markets betel nut fruit costs around 70 BDT/kg. If a composite sample is prepared weighing 1 kg, then according to the experimental result 20% (betel nut: glass=1:3) reinforced composite provides best results. So requirements of fiber will be 20% of 1 kg that is 200 gm(1:3) ratio means 50gm betel nut and 150 gm glass fiber. 1 betel nut fruit is around 6 gm in weight where recoverable fiber from husk is around 1.5 gm. So 50 gm betel nut fiber requires  $(6*50/1.5= 200\text{gm}$  betel nut fruit. So taking the cost as 70 BDT for 1 kg, the amount requires  $(200*70/1000)$  i.e 14 BDT. According to the price of glass fiber 150 gm costs  $(200*150/1000)$  tk or 30 BDT. 1 kg PE granule costs around 120 BDT so 800 gm means 96 BDT. So 1 kg composite preparation will cost in terms of raw materials  $(96+ 30+14)$  BDT= 140 BDT.

## **2.10 Probable Application of Betel Nut and Glass Fiber Reinforced Hybrid Composites**

Manufacturing companies are in always in constant search of new materials of lower costs and profit margins. Natural fibers are low-cost fibers with high specific properties and low density. Performance characteristics that predestine polymer use in automotive applications include corrosion resistance, low density, good impact toughness and chemical resistance.

A composite of woven jute fabric (burlap) and soybean oil based resin system has been successfully used to develop an I-shaped beam using vacuum assisted resin transfer molding (VARTM) method so this composite can also be used in beam manufacturing [70].

In 1986, a study has been published where it is reported that coir/polyester composites have been used to produce mirror casing, paper weights, projector cover, voltage stabilizer cover, mail-box and helmet and roof [71]. The property of betel nut fiber in many cases are close to coir fiber properties. So this composite will hopefully can be used in those applications.



The widespread application of natural fiber is in automotive industry. Plant fibers are mainly used in the part of car interior and truck cabins. The use of plant fiber based automotive parts such as various panels, shelves, trim parts and brake shoes are attractive for automotive industries worldwide because of its reduction in weight about 10%, energy production of 80% and cost reduction of 5%. For those various part manufacturing this composite can also be considered [72].

The major car manufacturers like Volkswagen, BMW, Mercedes, Ford and Opel now use natural fiber composites in applications. BMW has been using natural materials since the early 1990's in the 3, 5 and 7 series models with up to 24 kg of renewable materials being utilized. In 2001, BMW used 4000 tonnes of natural fibers in the 3 series alone. The combination here is a 80% flax with 20% sisal blend for increased strength and impact resistance. The main application is in the interior door linings and paneling. Wood fibers are also used to enclose the rear side of seat backrests and cotton fibers are utilized as a sound proofing material [72].

Recently, Volvo has started to use soya based foam linings in their seats along with natural fibers. They have also produced a cellulose based cargo floor tray which resulted in improved noise reduction. Hopefully these prepared composite can be used in producing mirror casing, paper weights and sailing boats, decks of fishing boats, mail-box, helmet, doors, windows and various parts of car interior together with truck cabins.

# **CHAPTER 3**

## **EXPERIMENTAL PROCEDURE**

## EXPERIMENTAL PROCEDURE

Standard specimen preparation, chemical treatment of betel nut fiber, composite manufacturing technique and testing of that prepared composites are explained in the following section.

### 3.1 Extraction of Fiber and Collection of Polyethylene

Commercial grade polyethylene (PE) as shown in Figure 3.1, was used in this study as matrix material. Polyethylene granules were collected from the local market. The PE was white in color having a melting point of 115-140°C. Betel nut and glass fibers were collected from a local market and chopped in the size of 3-4 mm, as shown in Figures 3.2 to 3.4.



Figure 3.1: Commercial polyethylene (PE)

The dried betel nut empty fruit was soaked in water for about seven days as shown in Figure 3.2. This soaking process loosens the fiber allowing it to be removed from the fruit easily. This process is called retting. The fibers were removed from the fruit. After drying, the fibers were further separated into an individual state using hand as shown in Figure 3.3.



Figure 3.2: Betel nut fruit



Figure 3.3: Extracted betel nut fiber



Figure 3.4: Glass fiber

### **3.2 Chemical Treatment of Betel Nut Fiber**

Alkali treatment of betel nut fibers was carried out to enhance its compatibility with the polyethylene matrix. Fibers were treated with 5% NaOH. Firstly, 5% NaOH solution was prepared by dissolving sodium hydroxide (NaOH) pellets in distilled water. Betel nut fibers were immersed in the 5% NaOH solution for 10 minutes at room temperature. The treated fiber inside the beaker was neutralized with HCl acid and

washed properly with distilled water. Finally the neutralized fiber was washed with distilled water to eliminate the NaOH residue and oven dried at 70°C for 24 hours and then removed from the oven as shown in Figure 3.5.



Figure 3.5: Alkali treated betel nut fiber

### 3.3 Manufacturing of Composites

At the first stage, hybrid composites of polyethylene matrix reinforced with 5, 10, 15 and 20wt% of fiber was prepared by reinforcing the mixture of betel nut and glass fibers with a ratio of (1:1). After that composites were prepared by kept the fiber percentages 20% with different fiber ratio. Fiber ratio were varied as (betel nut: glass=3:1, 1:1 and 1:3). In another experiment, treated betel nut fibers were prepared for 20% fiber with a ratio of (betel nut: glass=1:3). Composites were also prepared by addition of TiO<sub>2</sub> nano-powder (2phr) and zeolite powder (2phr) for this ratio.

Hybrid composites of polyethylene matrix reinforced with betel nut and glass fibers in a 150mm×150mm×5 mm die was manufactured using a hot press machine having maximum load of 35kN and maximum temperature of 300°C. At first fibers were weighed according to the required weight % needed. Then both fibers were cut into 3-5 mm size in length. Mold surface was cleaned very carefully and mold releasing agent was sprayed over the mould surface properly for the easy removal of the composite after production. In fibers reinforced composite, properly weighed fiber percentage was mixed with each other. After that placing of one layer PE granule and then the mixed fiber was arranged randomly in all direction. After that PE granules were also used for covering the die end. Then the lower part of the die containing arranged lamina was covered with the upper part of the die according to the

indication provided in both part of the die. Then the die was placed in a hot pressing machine (Figure 3.6).

The fiber-matrix mixture was allowed to press at 30kN pressure. The temperature was initially raised to 140°C and hold there for around 12-15 minutes, after that the temperature was raised to 160°C depending on the thickness required. The die was cooled to room temperature, pressure was released and the specimen was carefully withdrawn from the die. The same procedure was applied for preparing composites containing different percentages of fibers and also for different combination. Since the compression temperature was higher than the melting point of PE (140°C), the matrix melted however the fibers (melting point > 220°C) remained intact.



Figure 3.6: Die used for composite manufacturing

### **3.4 Mechanical Characterization of Composites**

#### **3.4.1 Tensile Test**

Tensile tests were conducted according to ASTM D 638-01 [73] using an Instron UTM machine (system Id 3369J8567, Maximum capacity 50KN). Each test was continued until tensile failure. Tensile properties as determined by the test method helps to know the stress of the composite specimen, their fracture characteristics under tensile load and load strain behavior bearing properties of fiber and the matrix materials. Five test specimens were prepared for each testing and the average values are considered.

Test specimens of required shape were cut from the composite part as shown in Figure 3.7. The specimen dimension was given according to the ASTM D 638-01[73] as shown in Figure 3.8.



Figure 3.7: Photograph of a tensile test specimen

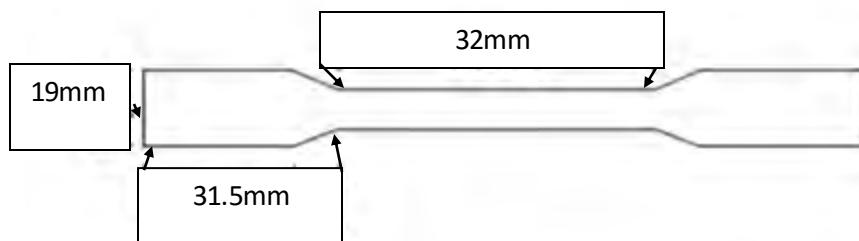


Figure 3.8: Tensile test specimen dimension

At first, dimensions of the specimens were taken .The test machine was calibrated. Strain rate was taken 4 mm/min. The ultimate tensile strength (UTS) was calculated. After that, tensile modulus was calculated by taking the slope of the stress-strain curve.

### 3.4.2 Flexural Test

The flexural test is generally performed on composites for evaluation in which tensile, compressive and shear stress act simultaneously. It says that, “the depth of support span shall be 16 times the depth of beam. Specimen width shall not exceed one fourth of the support span for specimens greater than 3.2 mm in depth”.

Total length of the specimen is taken as  $\{SP \text{ (length of support span= } 16d) + (20-30 \text{ mm})\}$



Figure 3.9: Photograph of a flexure test specimen

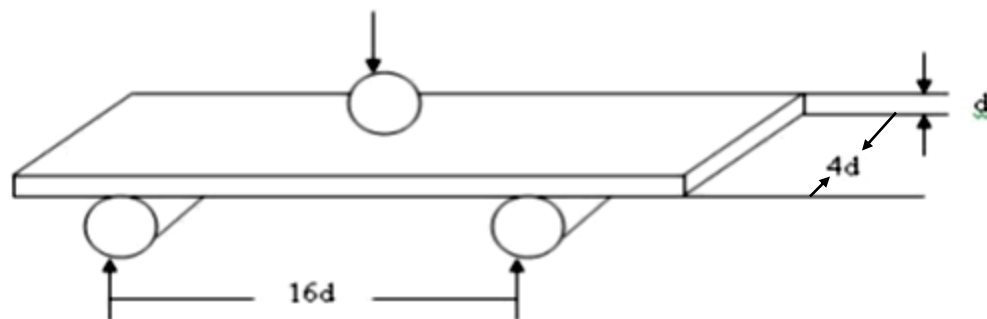


Figure 3.10: Flexure test specimen dimension

Test specimens of required shape were cut from the composite part as shown in Figure 3.9. The specimen dimension was given according to the ASTM D 790-00 [74] as shown in Figure 3.10. Then all the dimensions were measured. After that the rate of strain rate was calculated using the following formula,

$$R = 0.05 * L^2 / 6$$

Where, R = rate of strain rate mm/min, d = depth of beam, mm.

The loading nose and supports were aligned in such a way so that the axis of the cylindrical surfaces was parallel and the loading nose was midway between the supports. The load was applied to the specimen at the specified cross head motion and simultaneously load deflection data was taken. The test was terminated when rupture occurred at the outer surface of the test specimen.

The flexural stress was calculated by means of the following equation:

$$\sigma_f = 3PL / 2bd^2$$



Where  $\sigma_f$  = stress in the outer fibers at midpoint, MPa, P = load, N, L = support span, mm, b = width of beam, mm, d = depth of beam, mm.

Flexural Modulus,

$$E_B = L^3 m / 4bd^3$$

Where, m = the gradient (i.e., slope) of the initial straight-line portion of the load deflection curve.

Flexural strain is calculated according to the equation:

$$\epsilon_{3pB} = 6Dd / 2L^2 * 100$$

Where, D = elongation, mm.

### 3.4.3 Impact Test

The advantages of composite materials are numerous and well documented. Composite materials are often used in environments in which they will suffer from impact damage. For example, damage can occur from a hammer being dropped on a composite pipe or from a bullet striking composite armor. Since impact damage resistance is such an important property for composite materials. Charpy impact testing has been used for many years to test the impact toughness of various metals. The dynamic charpy impact test of the composite was conducted using an impact tester MT 3016 according to ASTM D 6110-97[33]. The test specimen is shown in figure 3.11.



Figure 3.11: Impact test specimen

### 3.4.4 Hardness Test

The hardness of the composite was measured using a shore hardness testing machine as shown in Figure 3.12. Shore Durometer is one of several measures of the hardness of a material. Hardness property of composite was measured using shore A scale.



Figure 3.12: Hardness tester

### 3.4.5 Field Emission Scanning Electron Microscopy (FESEM)

Morphological analysis of the fiber is carried out using SEM. This is the most widely used of the surface analytical techniques. High resolution SEM has proved an invaluable tool for studying surface topography and failure analysis. The technique enables qualitative three dimensional (3-D) imaging of surface features, however, it does not easily lend itself to quantitative surface roughness characterization. This can be overcome by complementing SEM investigations with atomic force microscopy.

The interfacial bonding between the fibers and PE matrix in manufactured composites and tensile fracture surfaces of the same composites were examined using a Scanning Electron Microscope JEOL FESEM analyzer of model no JSM-7600F. The micrographs are presented in the Results and Discussion section.

### 3.4.6 FTIR Spectroscopy

In the present study infra-red spectra of betel nut and glass fiber reinforced hybrid PE composites were recorded on a SHIMADZU spectrophotometer with co-addition of 32 scans. Firstly some powdered samples were collected by scratching of the composite by a knife. Then potassium bromide (KBr) was mixed at a ratio of (KBr: Sample = 100:1) with them in a mortar pestle. The mixture was then taken in a dice of specific dimensions (13 mm dia). The pellet was formed by pressing (mechanical presser at 8 tons of pressure) and was placed on the sample holder and the spectroscopy was carried out.

### 3.4.7 TGA Analysis

In most of the process industries, heat generation becomes unavoidable and good thermal resistance of the composites has attracted the attention of the scientists. Thermo gravimetric analysis is a technique in which the mass of a substance is monitored as a function of temperature or time as the sample specimen is subjected to a controlled temperature programmed in a control atmosphere. It provides important data towards understanding the structure-property relationships and controlling the technology for molecular design with the aim of industrial production of different polymeric materials, especially fiber reinforced composites. Moreover, it is a useful technique to determine the thermal stability of the materials. Additionally, it permits to quantify the amount of possibly damaging, deteriorating volatiles, such as the moisture uptake during a hydrothermal treatment, which can cause deterioration in the composites. In our study, TGA was carried out in an universal V4.2E TA instruments (TGA Q50 V6.4) at a temperature range of 25-800°C, with a constant heating rate of 10°C/sec.

### 3.4.8 Water Absorption Test

The specimens for water absorption test had dimension 39mm×10mm×4.1mm. After oven drying, the weight of all the specimens were taken and recorded. Next, the specimens were immersed in the distilled water according to ASTM D 570-99. After 24 hours, the percentage increase in weight during immersion was calculated using following Equation [75]:

$$\text{Increase in weight, \%} = [(\text{weight}_{\text{wet}} - \text{weight}_{\text{dry}}) / \text{weight}_{\text{dry}}] \times 100$$

**CHAPTER 4**  
**RESULTS AND DISCUSSION**

## RESULTS AND DISCUSSION

FTIR, Mechanical properties, Interfacial characteristics (SEM), Thermal properties (TGA) of the prepared composites are explained in the following section.

### 4.1 FTIR Spectroscopic Analysis Results

The FTIR spectrum of polyethylene is shown in Figure 4.1. In the spectrum the major peaks are found at wavenumber  $2923\text{ cm}^{-1}$ ,  $2852\text{ cm}^{-1}$ ,  $1464\text{ cm}^{-1}$  and  $1367\text{ cm}^{-1}$  corresponds respectively to the  $\text{CH}_2$ (methylene) asymmetric strong stretch,  $\text{CH}_2$  symmetric strong stretch, bending strong deformation and wagging medium deformation. Another important peak is found at  $719\text{ cm}^{-1}$  corresponds to rocking medium deformation [76].

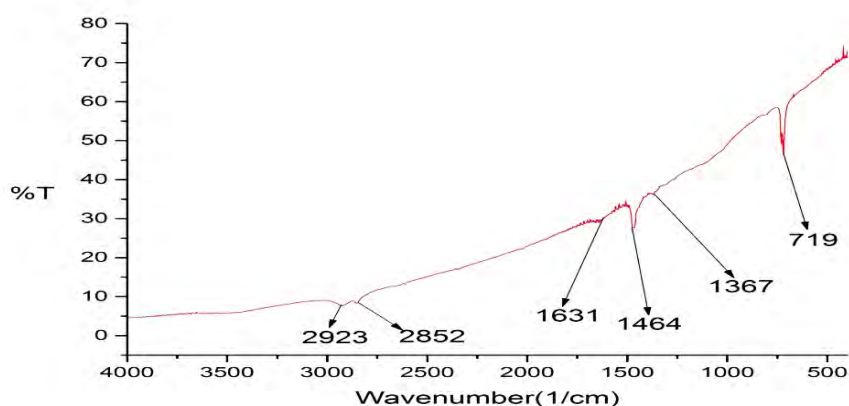


Figure 4.1: FTIR spectrum of polyethylene

FTIR spectra analyses of the prepared composite shows characteristic absorption peak at around  $3477\text{ cm}^{-1}$ , corresponding to the presence of  $-\text{OH}$  group (Figure 4.2). Other peaks are found at  $1633\text{ cm}^{-1}$  for aromatic  $\text{C}=\text{C}$  in plane alkenes (lignin),  $1373\text{ cm}^{-1}$  for  $\text{C}-\text{H}$  bending bond structure of cellulose, hemicelluloses, lignin and  $1107\text{ cm}^{-1}$  for glass fiber in reinforced composites respectively[77].

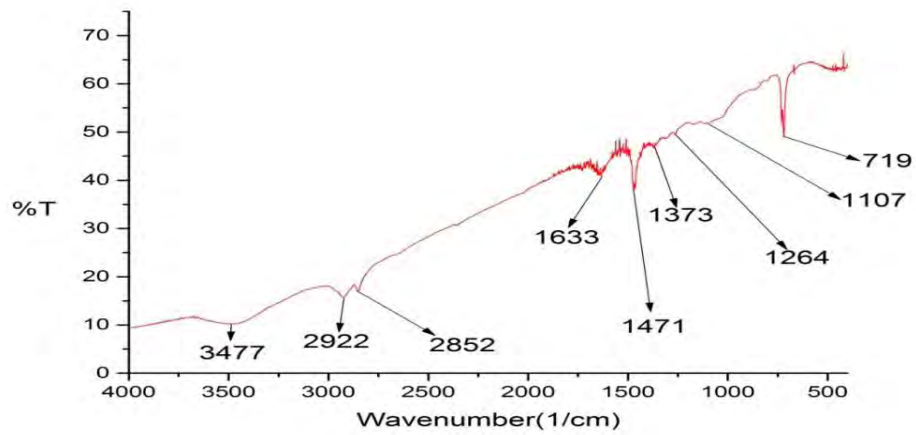


Figure 4.2: FTIR spectrum of 20% untreated betel nut and glass (1:3) fiber reinforced PE composite

A strong sodium hydroxide (NaOH) treatment might remove lignin, hemicelluloses and other alkali soluble compounds from the surface of the fibers to increase the numbers of reactive hydroxyl groups on the fiber surface available for chemical bonding. Thus the alkali treatment could cause an increase of the fiber surface free energy. Moreover, the alkali treatment could make the fiber surface ‘clean’ due to removal of waxes, hemicelluloses, pectin and part of lignin [30]. The removal of these substances enhanced the surface roughness. Therefore, the mechanical interlocking at the interface could be improved. In general, alkali treatment provides good bonding between matrix and fiber.

FTIR spectra analyses of the treated betel nut and glass fiber reinforced PE composite is shown in Figure 4.3. In 5% NaOH treated fiber sample, the characteristic absorption peak at 1729  $\text{cm}^{-1}$  indicates the loss of hemicelluloses. The peak at 1311  $\text{cm}^{-1}$  shows lower intensity which indicates the loss of cellulose, hemicelluloses and lignin respectively. Although the occurrence of majority peaks has not changed, it is noted that the absorption peak around 3477  $\text{cm}^{-1}$ , corresponding to the hydroxyl group in the untreated fiber, has lost its intensity in the spectrum in the alkali treated fiber, indicating the elimination of it by alkali treatment.

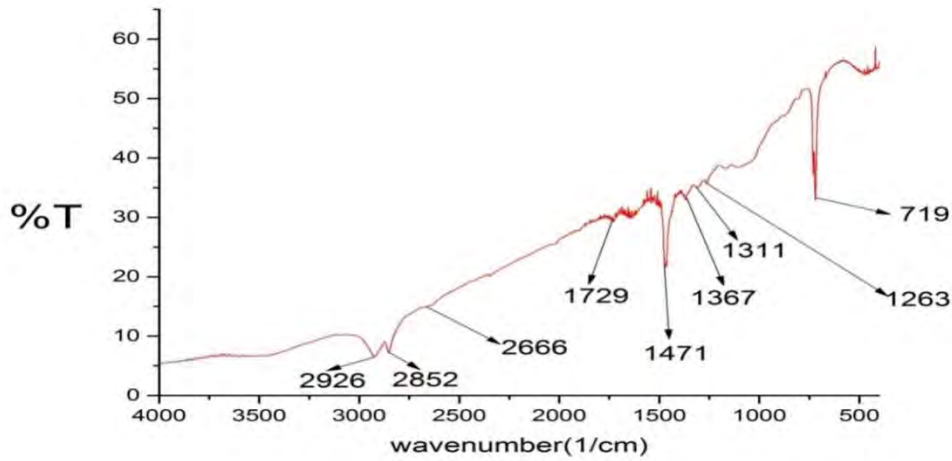


Figure 4.3: FTIR spectrum of 20% treated betel nut and glass (1:3) fiber reinforced PE composite

FTIR spectra analyses of TiO<sub>2</sub> nano-powder added composite shows broad absorption band around 3459 cm<sup>-1</sup> corresponding to the presence of -OH group (Figure 4.4). Another important peak is found at 2353cm<sup>-1</sup> due to the presence of traces amount of CO<sub>2</sub>. Peaks at 667cm<sup>-1</sup> and 467 cm<sup>-1</sup> corresponds to Ti-O, Ti-O-Ti absorption which indicates the presence of TiO<sub>2</sub> nano-powder in the composites. Finally, absorption peak at 1021 cm<sup>-1</sup> corresponds to glass fiber in reinforced composites [78].

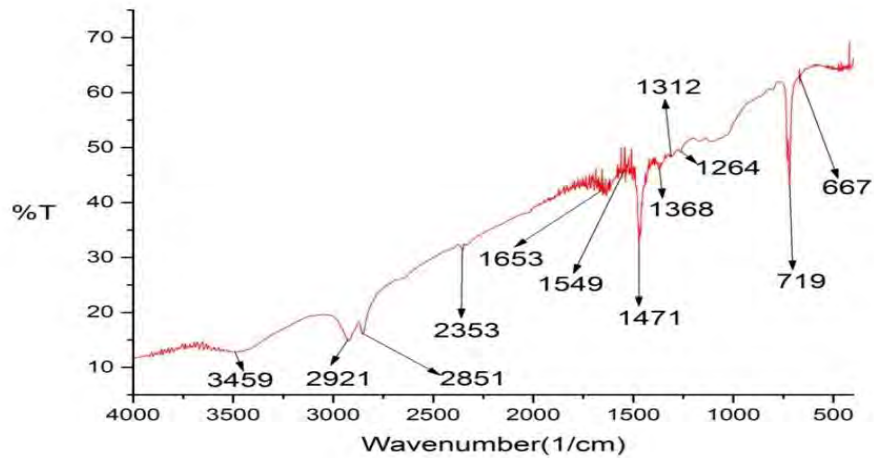


Figure 4.4: FTIR spectrum of 20% untreated betel nut and glass (1:3) fiber reinforced composite with TiO<sub>2</sub> nano-powder (2phr)

FTIR spectrum of zeolite added composite shows 3627  $\text{cm}^{-1}$  peak overlap with peaks in the range 3100-3500  $\text{cm}^{-1}$ . Here, absorption band around 3459  $\text{cm}^{-1}$  indicates the presence of  $-\text{OH}$  group as shown in Figure 4.5. Another important peak is found at 2353  $\text{cm}^{-1}$  due to the presence of traces amount of  $\text{CO}_2$ . Peaks at 1653  $\text{cm}^{-1}$  shows OH bonding vibration. Finally, peaks at 1312  $\text{cm}^{-1}$  and 1264  $\text{cm}^{-1}$  correspond to the presence of glass fiber in reinforced composites respectively [79].

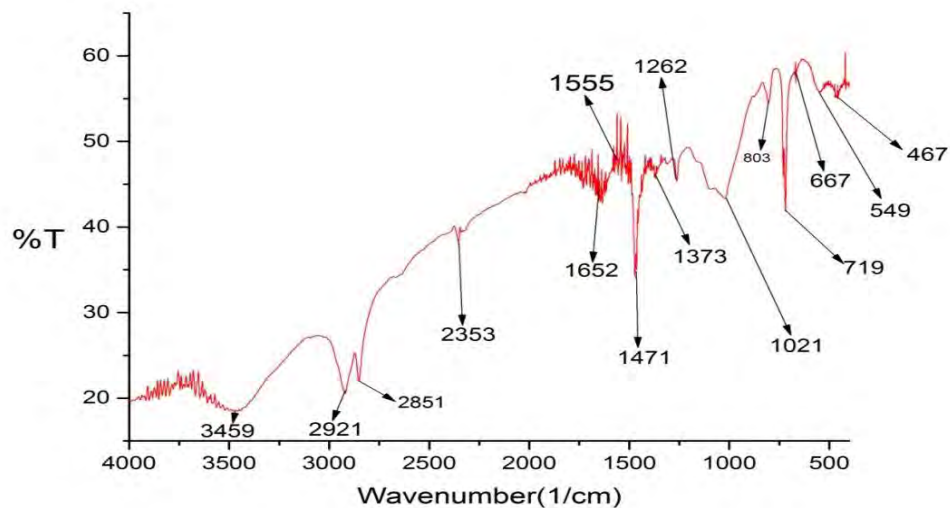


Figure 4.5: FTIR spectrum of 20% untreated betel nut and glass (1:3) fiber reinforced composite with zeolite nano-powder (2phr)

## 4.2 Mechanical Properties

### 4.2.1 Tensile Properties

Tensile properties of the composite with 5, 10, 15 and 20% fiber loading were measured for untreated betel nut and glass fiber ratio (1:1). Initially the tensile strength increased from raw PE to 5% fiber loaded composite and then decreased with increasing fiber percentages. As loading increased, interfacial area between the fiber and matrix increased and became weaker which gradually reduced tensile strength as shown in Figure 4.6. The same trend was also found in previous researcher [30].



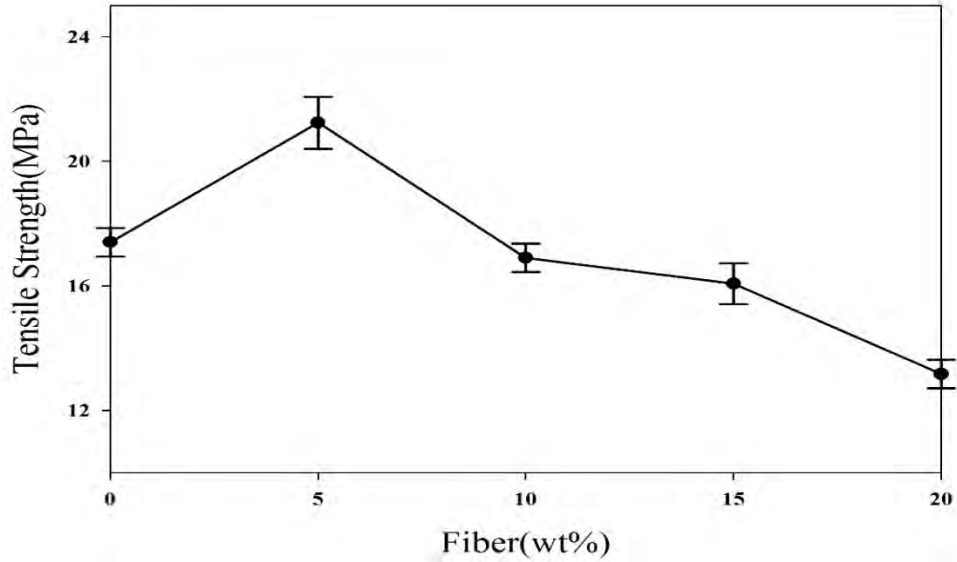


Figure 4.6: Variation of tensile strength against different fiber loading of untreated betel nut and glass (1:1) fiber reinforced PE composite

With increasing in the amount of glass fiber, tensile strength increased as shown in Figure 4.7. The properties of hybrid composites were mainly dependent on the properties of individual reinforcing fibers, orientation and arrangement of fibers, the extent of intermingling of the fibers and fiber-matrix adhesion. In this case, the increase in the strength of the hybrid composites with the incorporation of glass fiber was mainly due to the high tensile strength of glass fiber (2.0 GPa) as compared to the low strength nature of betel nut fiber (166.03MPa) [57].

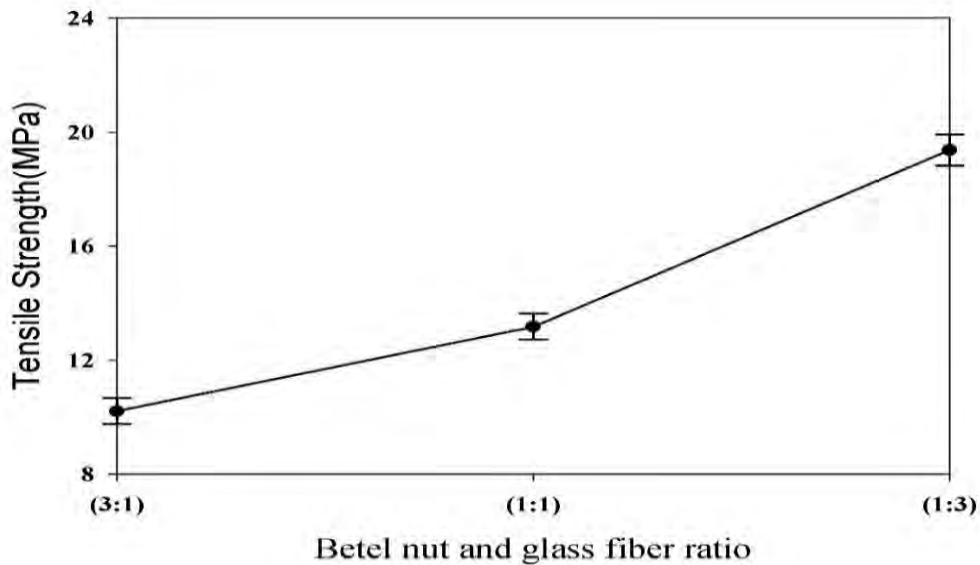


Figure 4.7: Variation of tensile strength at different fiber ratio for 20% fiber reinforced PE composite

It was observed that Young's modulus increased with an increase in fiber loading [56, 80]. With an increase in fiber content, the brittleness of the composite increased and stress/strain curves became steeper. Incorporation of fibers in matrix also creates partially separated micro spaces which obstruct stress propagation between the fiber and the matrix [52]. As the fiber loading increased, the degree of obstruction increased, which in turn increased the stiffness. Furthermore incorporation of fiber into the polymer matrix reduced the matrix mobility, which in turn enhanced the stiffness property [80] as shown in Figure 4.8. The same trend was found by other researchers [30].

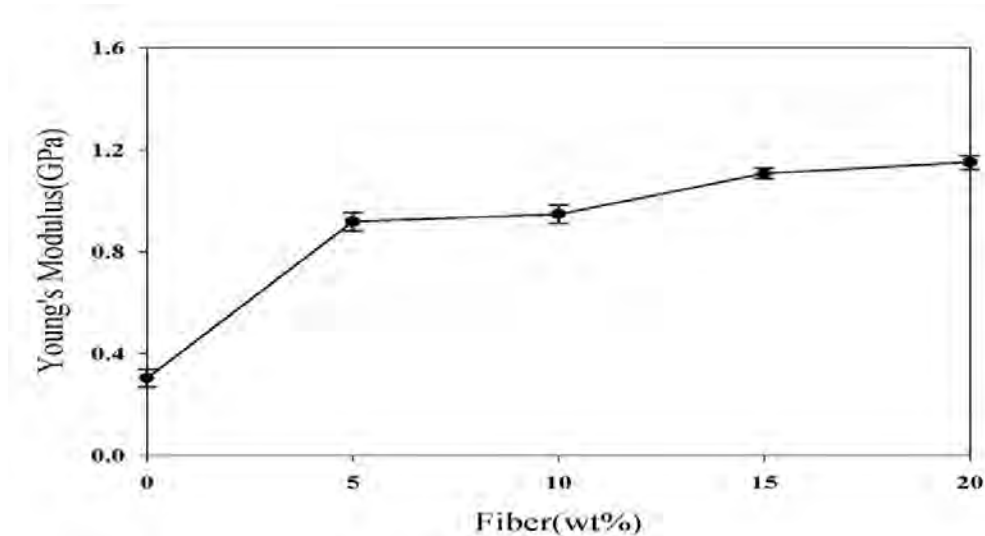


Figure 4.8: Variation of Young's modulus against different fiber loading of untreated betel nut and glass (1:1) fiber reinforced PE composite

The incorporation of glass fiber into betel nut and glass fiber reinforced PE composite increased the modulus values of the hybrid composites as seen in Figure 4.9. The enhancement in the stiffness of these composites is attributed to the higher modulus of glass fiber (72.3 GPa) than that of the betel nut fiber (1.38-1.93 GPa) [57, 81]. It was observed that with an increase in the weight fraction of glass fiber from 25 to 75%, the Young's modulus of hybrid composites increased steadily from 1.08 to 1.23 GPa (13%). As the weight fraction of glass fiber increased, the load bearing capability of the hybrid composites also increased and resulted in improved stiffness.

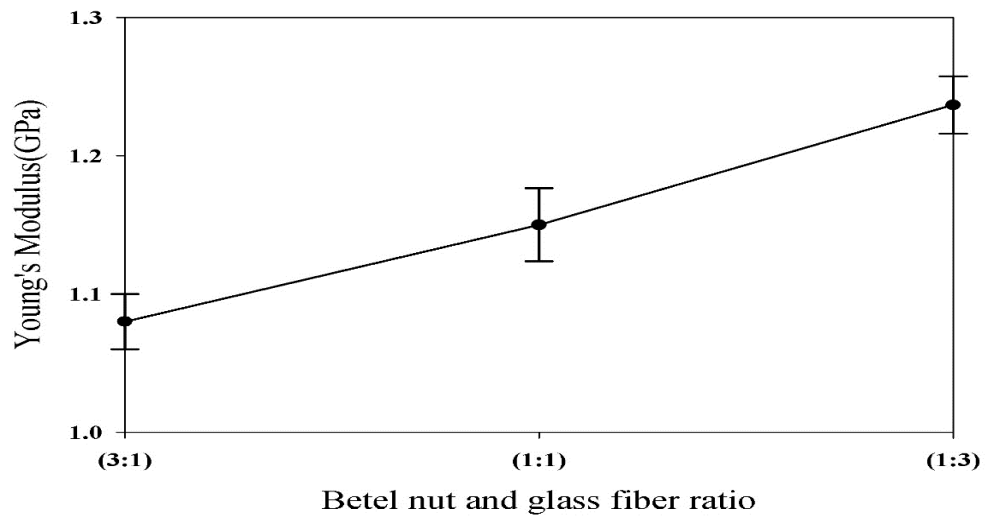


Figure 4.9: Variation of young's modulus at different fiber ratio for 20% fiber reinforced PE composite

Percentage elongation at break decreased with increase in fiber loading as shown in Figure 4.10. This was due to the non-uniform dispersion of fibers that obstructed the mobility or deformability of the matrix, which resulted in the decreasing trend of elongation at break of the composite [82, 83]. Previous work also found the same trend of elongation [30].

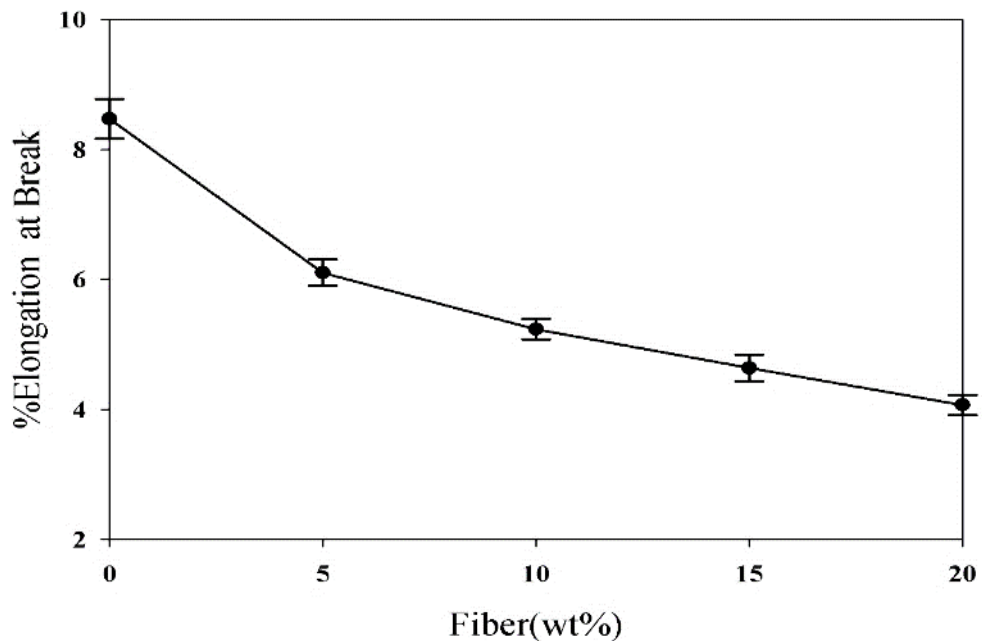


Figure 4.10: Variation of %elongation at break against different fiber loading of untreated betel nut and glass (1:1) fiber reinforced PE composite

In hybrid composites, the properties of the composites are mainly dependent on the modulus and percentage of elongation at break of the individual fibers. Figure 4.11 shows the variation of elongation at break at different betel nut and glass fiber loading. Among the hybrid composites, the incorporation of 75% glass fiber exhibited the lowest elongation at break. This phenomenon was due to the fact that glass fiber individually has low % elongation at break in comparison to betel nut fiber [81].

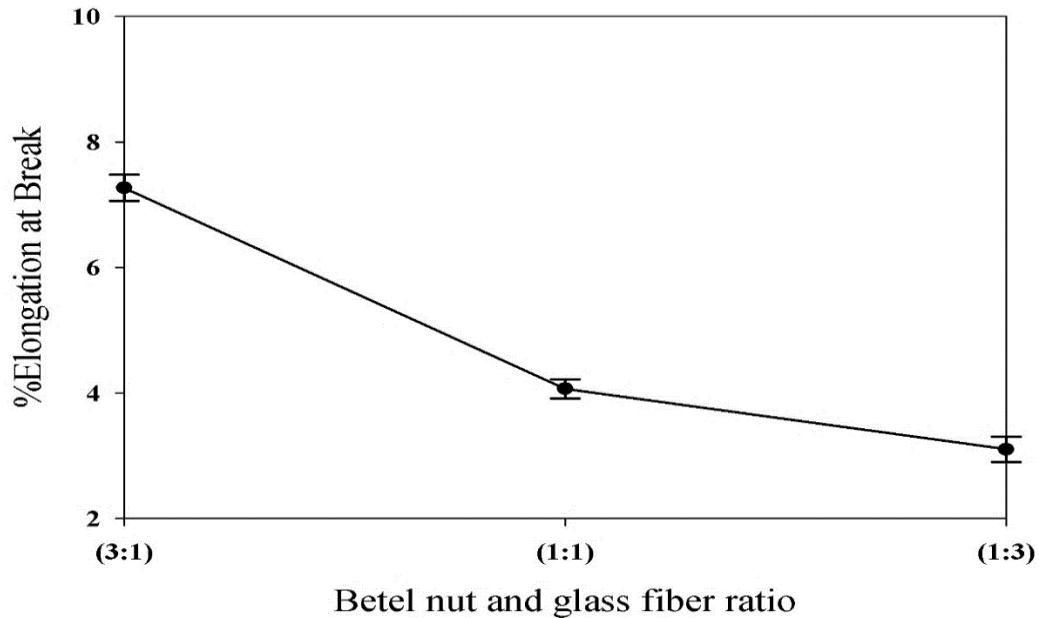


Figure 4.11: Variation of % elongation at break at different fiber ratio for 20% fiber reinforced PE composite

#### 4.2.2 Flexural Properties

The flexural strength and flexural modulus of 5, 10, 15 and 20% betel nut and glass fiber (betel nut: glass=1:1) reinforced hybrid polyethylene composites are shown in Figures 4.12 and 4.13 respectively. From the experimental observation it was found that the flexural strength increased with an increase of fiber loading. This might be due to the lower amount of clustering of fibers which resulted due to the non-uniform dispersion of fibers with increasing of fiber loading. Clustering affects the effective load transfer and reduces the flexural strength [84]. Enhancement of number of fiber loading provides non uniform dispersion of fibers which increases flexural modulus. The increase of flexural strength with fiber loading was also observed in previous research [56, 80]. According to Figure 4.13, the flexural modulus increased with fiber loading. Since both betel nut and glass is high modulus material, higher fiber concentration demands higher stress for the same deformation and increased stress transfer from the matrix to the fiber. Thus this result can be attributed to the incorporation of rigid glass fiber into the soft PE matrix. The same trend was also observed in previous research [30].

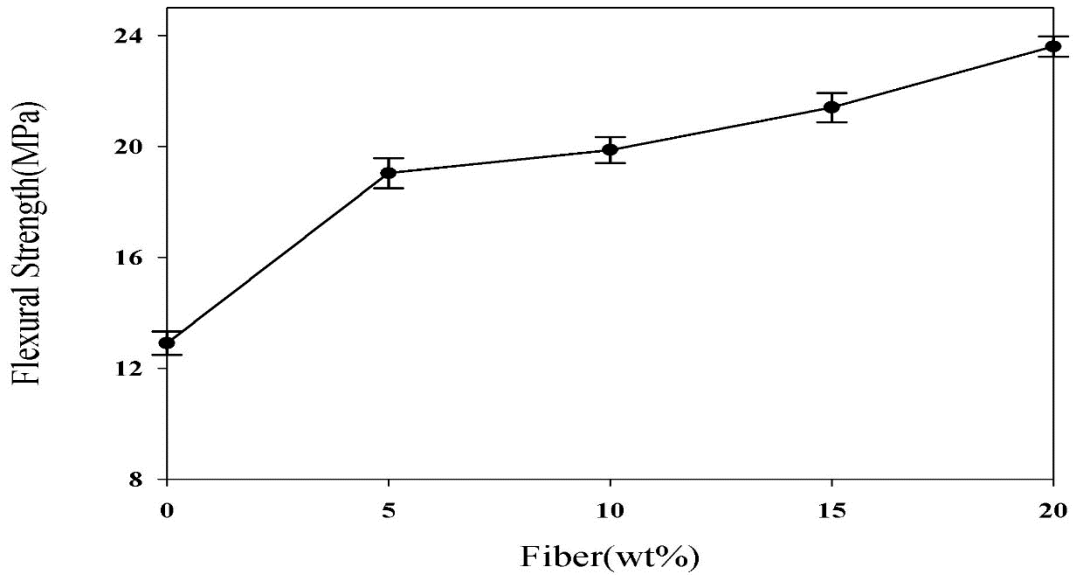


Figure 4.12: Variation of flexural strength against different fiber loading of untreated betel nut and glass (1:1) fiber reinforced PE composite

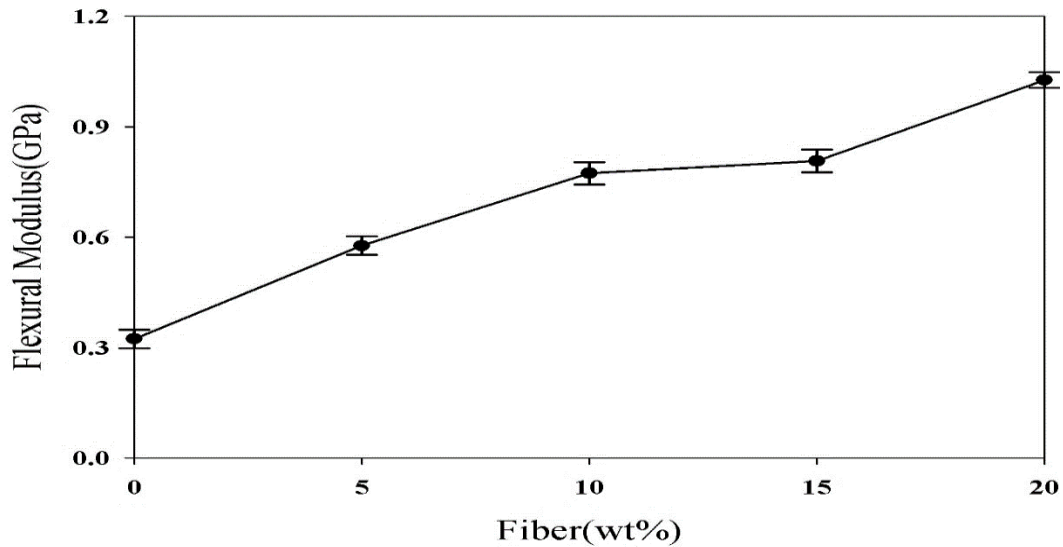


Figure 4.13: Variation of flexural modulus against different fiber loading of untreated betel nut and glass (1:1) fiber reinforced PE composite

The variation of flexural strength and flexural modulus at different ratio of betel nut and glass fiber for 20% fiber reinforced hybrid polyethylene composites are shown in Figures 4.14 and 4.15 respectively. It was observed that the flexural strength and modulus of the hybrid composites had dissimilar trends as the tensile properties where the flexural behavior increased with the addition of glass fiber. In flexural

loading, the composites samples were subjected to compression, tension and shear stresses. In a three-point flexure test, failure occurred due to bending and shearing. The increased flexural strength of the hybrid composites with the loading of glass fiber was mainly due to the increased resistance to shearing of the composites as a result of the inclusion of rigid glass fiber [85].

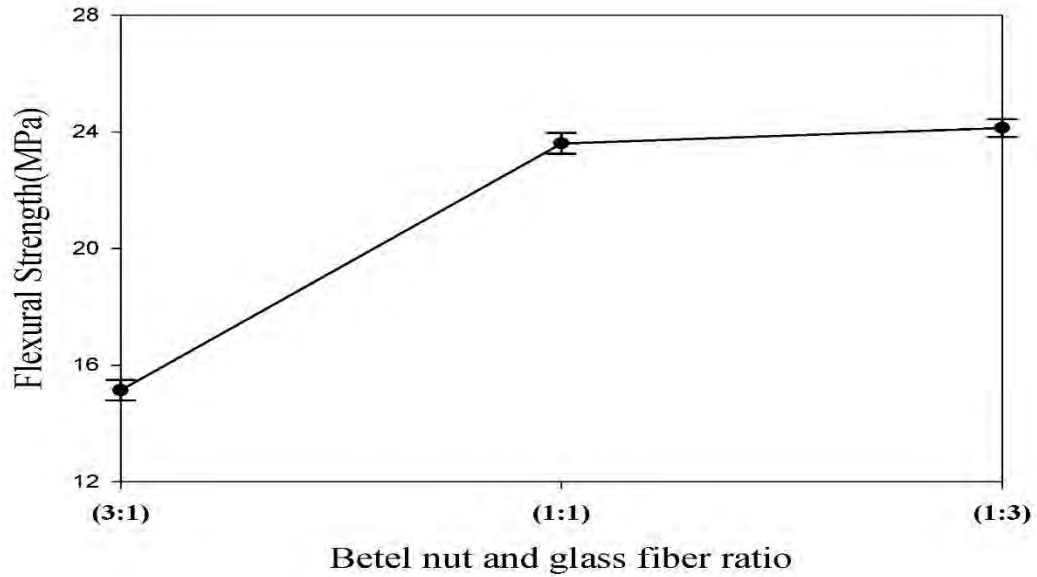


Figure 4.14: Variation of flexural strength at different fiber ratio for 20% fiber reinforced PE composite

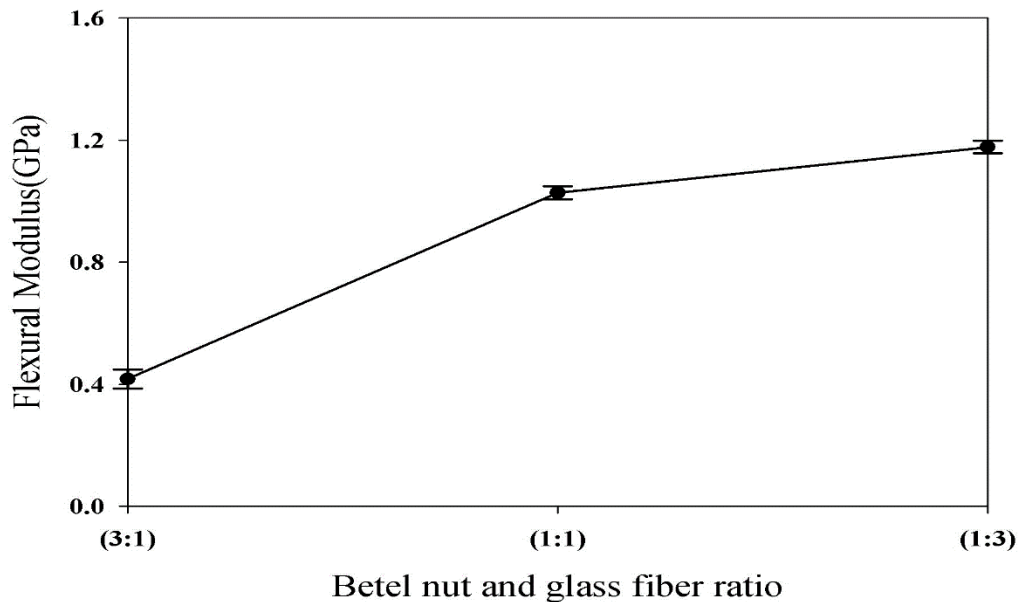


Figure 4.15: Variation of flexural modulus at different fiber ratio for 20% fiber reinforced PE composite

With the enhancement of glass fiber percentage from 25 to 75% the flexural modulus of betel nut and glass fiber reinforced PE hybrid composites increased from 0.42 to 1.17 GPa. This suggests that the flexural modulus of the composites depends largely on the amount of glass fiber, which could be due to the high modulus of glass fiber. The highest flexural modulus was observed in hybrid composites with 75% weight fraction of glass fiber. These were due to the presence of sufficient amount of fiber that provided the effective stress transfer between the fiber and the matrix and also due to the inherent property of glass fiber [57].

### 4.2.3 Hardness

Hardness of a composite depends on the uniform distribution of the fiber into the matrix. Hardness is enhanced when the flexibility of matrix is lowered. The presence of a rigid fiber material into the PE matrix results into more rigid composites. Better dispersion of the fiber into the matrix with minimization of voids between the matrix and the fiber enhanced hardness. Actually incorporation of fiber into the PE matrix has reduced the flexibility of the matrix resulting in more rigid composites. Due to the increase of stiffness of respective composite the hardness of betel nut–glass fiber reinforced hybrid PE composites showed a slight increasing trend with an increase in the fiber content [86]. With the enhancement of fiber loading dispersion of fiber materials increased. As a result, hardness of betel nut-glass fiber reinforced hybrid PE composites has showed a slight increasing trend with an increase in fiber loading [55]. The same trend was also found in literature [30].

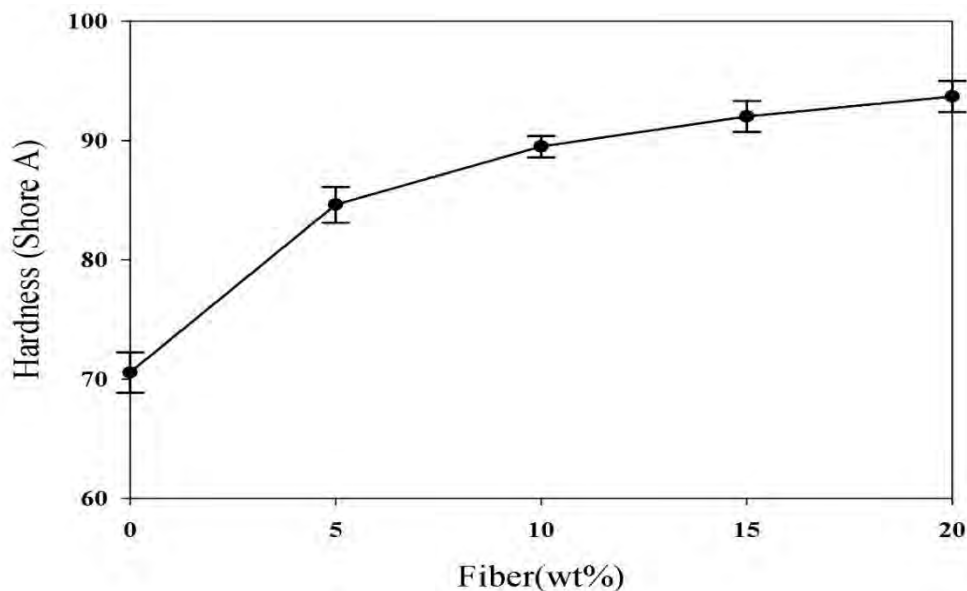


Figure 4.16: Variation of hardness (shore A) against different fiber loading of untreated betel nut and glass (1:1) fiber reinforced PE composite

The shore hardness of betel nut and glass fiber reinforced hybrid polyethylene composites at different fiber ratio is shown in Figure 4.17. Due to the change of rigid glass fiber ratio from 1 to 3, hardness increases from 90.23 to 96.7. Though the enhancement of glass fiber percentage has shown the enhancement of hardness values, the change was quite conspicuous. Enhancement of glass fiber percentage increased the hardness value by only 7.17%.

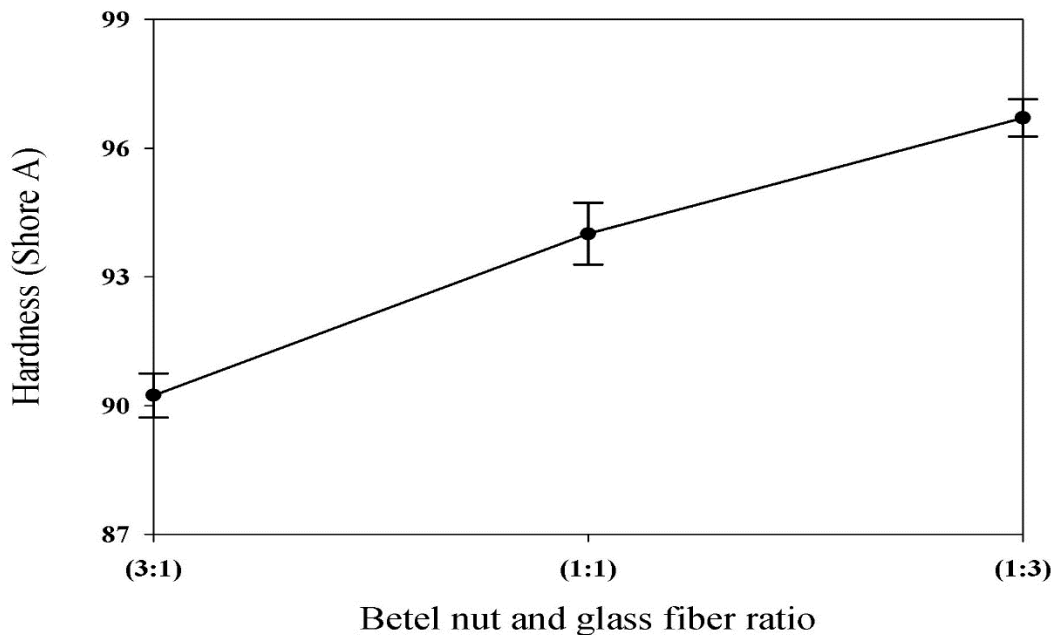


Figure 4.17: Variation of hardness (Shore A) at different fiber ratio for 20% fiber reinforced PE composite

#### 4.2.4 Impact Properties

Variation of the Charpy impact strength with fiber loading for betel nut and glass (betel nut: glass=1:1) fiber reinforced hybrid polyethylene composite is shown in Figure 4.18. The impact strength of the fiber reinforced polymeric composites depends on the nature of the fiber, polymer and fiber-matrix interfacial bonding [24,87]. The impact strength of all composites increased with an increase in fiber loading[13,88-91]. This result suggests that the fiber was capable of absorbing energy because of favorable entanglement of the fiber and matrix. Fiber pull out is found to be an important energy dissipation mechanism in fiber reinforced composites [92]. One of the factors of impact failure of a composite is fiber pull out. With the increase in fiber loading,



stronger force is required to pull out the fibers. This in turn increases the impact strength. Increase of impact strength with fiber loading is also found in previous research [72].

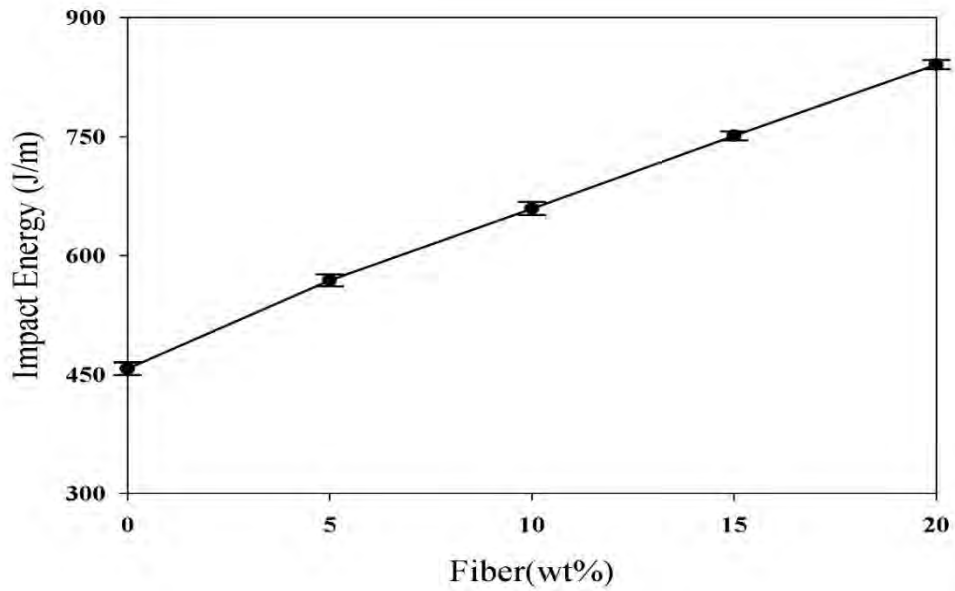


Figure 4.18: Variation of impact energy against different fiber loading of 20% untreated betel nut and glass (1:1) fiber reinforced PE composite

Variation of impact energy at different fiber ratio for 20% fiber loaded composite is shown in Figure 4.19. Incorporation of more glass fiber reduced impact energy because of its brittle nature and makes the overall hybrid composite to a rigid one that is not capable of absorbing more energy due to impact force. As a consequence, increasing glass fiber from 25 to 75 reduced impact energy by 3.64%.

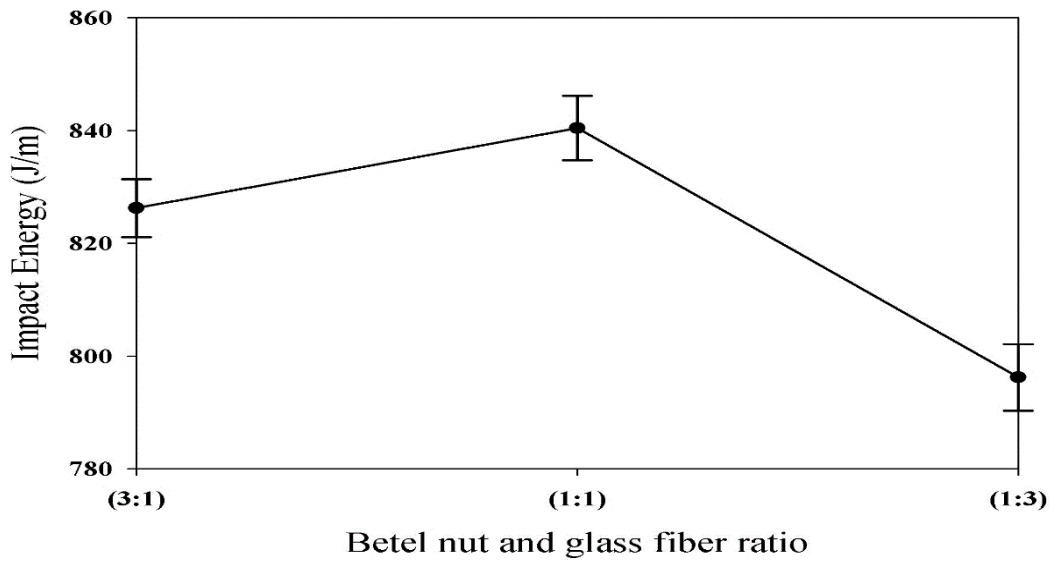


Figure 4.19: Variation of impact strength at different fiber ratio for 20% fiber reinforced PE composite

### 4.3 Water Absorption Test

Water absorption of lignocellulosic fiber composites is another important characteristics that needs to be determined. Water absorption could lead to a decrease in some of the properties and needs to be considered when selected for applications. Cellulose, hemicelluloses and lignin of betel nut fiber contain hydroxyl group. Hydroxyl groups form hydrogen bonds with water molecules. So hydroxyl group absorbs more water. From Figure 4.20, it is cleared that as the fiber loading increased, the water absorption(%) enhanced as natural fiber (betel nut) which is hydrophilic in nature is increased. Results of water absorption in current study for different fiber loading is shown in Figure 4.20.

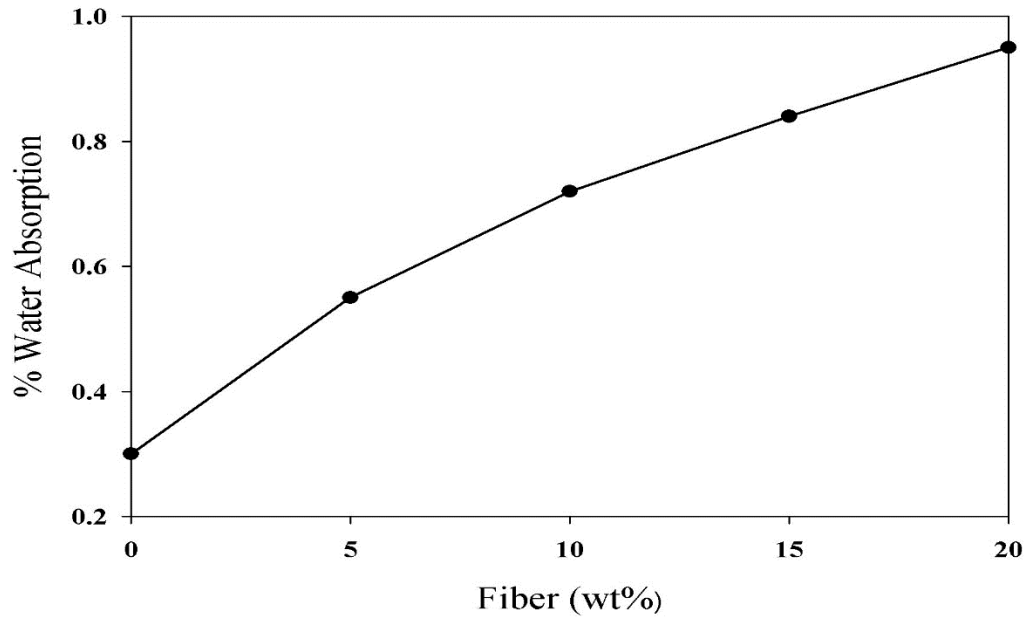


Figure 4.20: Variation of %water absorption against different fiber loading of 20% untreated betel nut and glass (1:1) fiber reinforced PE composite

On the other hand, more betel nut fiber incorporation increased the percentages of water absorption due to the presence of more hydroxyl groups in natural betel nut fiber as shown in Figure 4.21 whereas for less amount of betel nut fiber the percentages reduced.

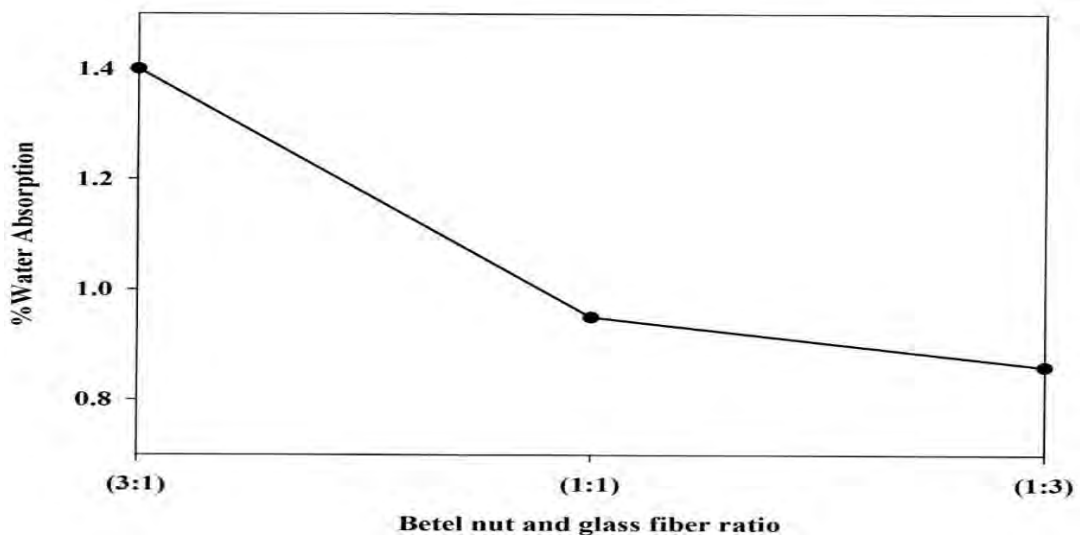


Figure 4.21: Variation of water absorption at different fiber ratio for 20% fiber reinforced PE composite

#### **4.4 Comparative Study**

From the comparative (Figures 4.22 to 4.28) bar graphs it is quite conspicuous that the treated betel nut fiber reinforced composites showed better mechanical properties than untreated fiber reinforced. Untreated fiber reinforced composites provided poor mechanical properties because of the weak interfacial bonding between cellulose based hydrophilic filler (betel nut) and hydrophobic matrix. However, addition of TiO<sub>2</sub> and zeolite nano-powder also enhanced the mechanical properties of composites.

##### **4.4.1 Tensile Properties**

Mercerization (alkali treatment) removed the impurities, waxy materials, lignin as well as hemicellulose and resulted in an increment of cellulose exposed on the fiber surface, thereby increased the number of possible reaction sites. It also enhanced the interfacial bonding by mechanical interlocking due to increase in surface roughness. In addition mercerization improved the fiber wetting by fibrillation, which increased the effective surface area available for the contact with the wet polymer matrix [93]. During alkaline treatment, the inter-fibrillar region was likely to be less dense and less rigid, that made the fibrils rearranged themselves more along the direction of tensile loading. When fibers were stretched, such arrangements among the fibrils would result in better load sharing and hence in higher stress development in the fiber [93, 94]. Alkali treatment also caused loss of crystalline structure of cellulose molecular chains in a micro fibril partly, as a result of which the alignment of the micro fibrils was destroyed and the overall crystallinity was reduced. In addition, removal of certain elements caused easy deformation of the cellular networks. The extensive hydrogen bonding network might be broken and the ordered structural arrangement of cellulose might also lose [95]. An increase in the tensile strength, Young's modulus and elongation at break of the hybrid composites were observed with the incorporation of treated betel nut fiber in place of untreated one for 20% fiber loading at ratio of betel nut:glass=1:3. Due to high dispersion of TiO<sub>2</sub> and zeolite nano-powder, the interaction between polymer matrix and particles increases which tend to enhance the tensile strength.

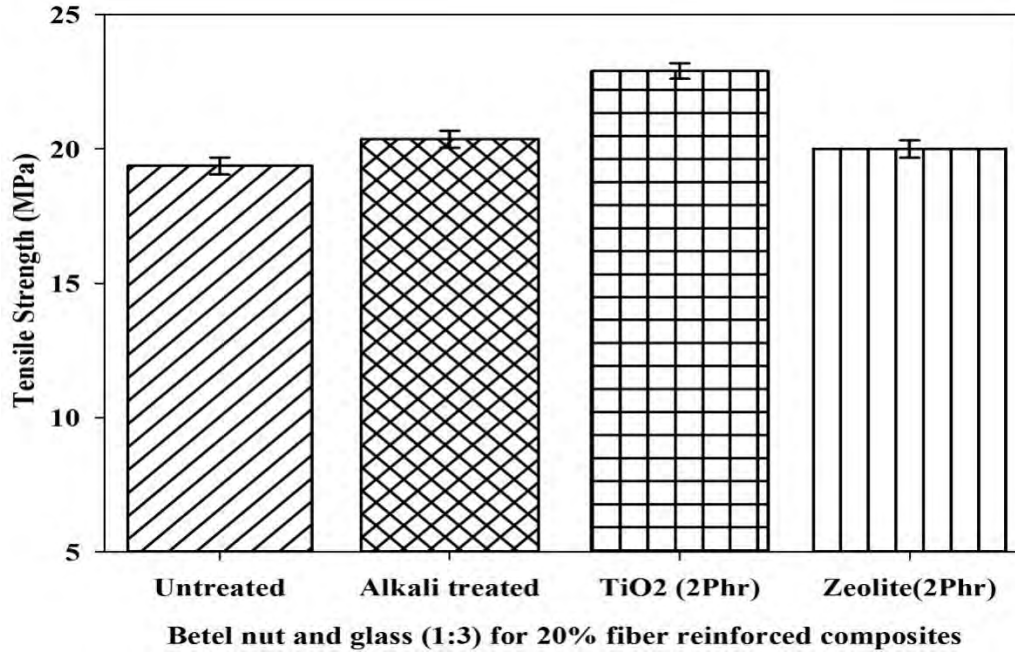


Figure 4.22: Variation of tensile strength for 20% betel nut and glass (1:3) fiber reinforced PE composite

On the other hand, incorporation of rigid particle filler into the polymer matrix increased stiffness and hence increases Young's modulus [96].

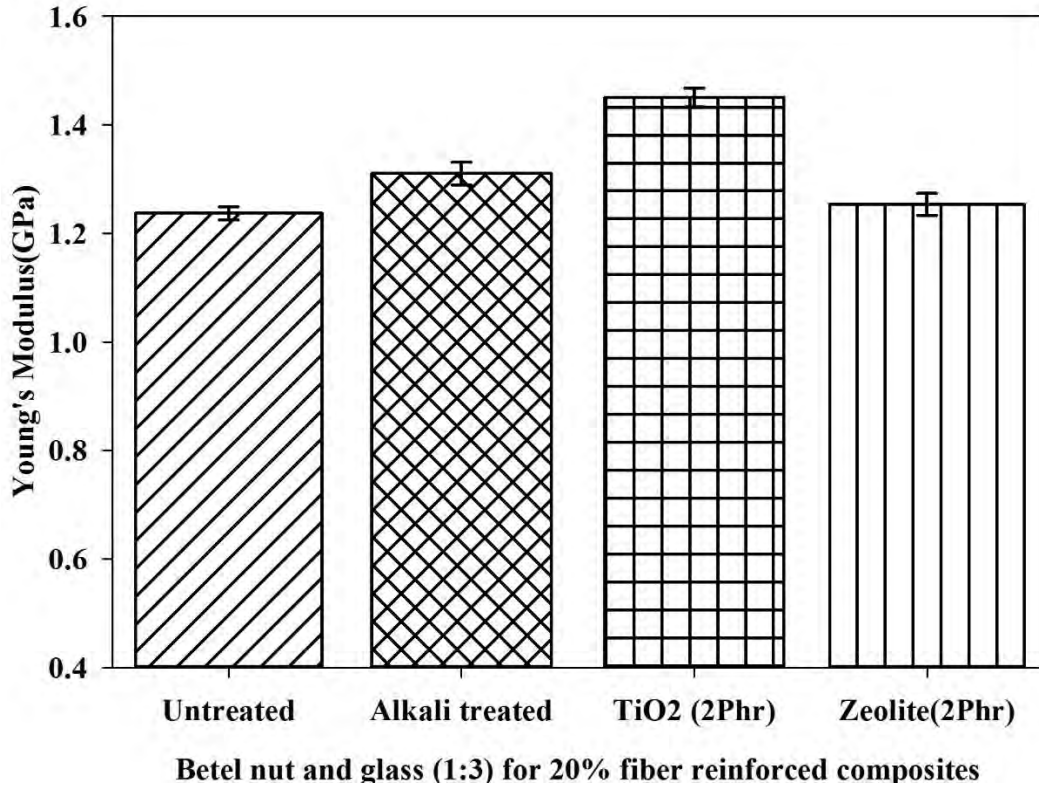


Figure 4.23: Variation of young's modulus for 20% betel nut and glass (1:3) fiber reinforced PE composite

The drop in elongation at break values of the composite with nano-filler content could be explained as the elastic properties of the composite depend on the polymer matrix, which shows brittle behaviour in the presence of the fillers. This is because these fillers restrict the mobility of the polymer, and the higher the filler content the higher the brittleness of the composite.

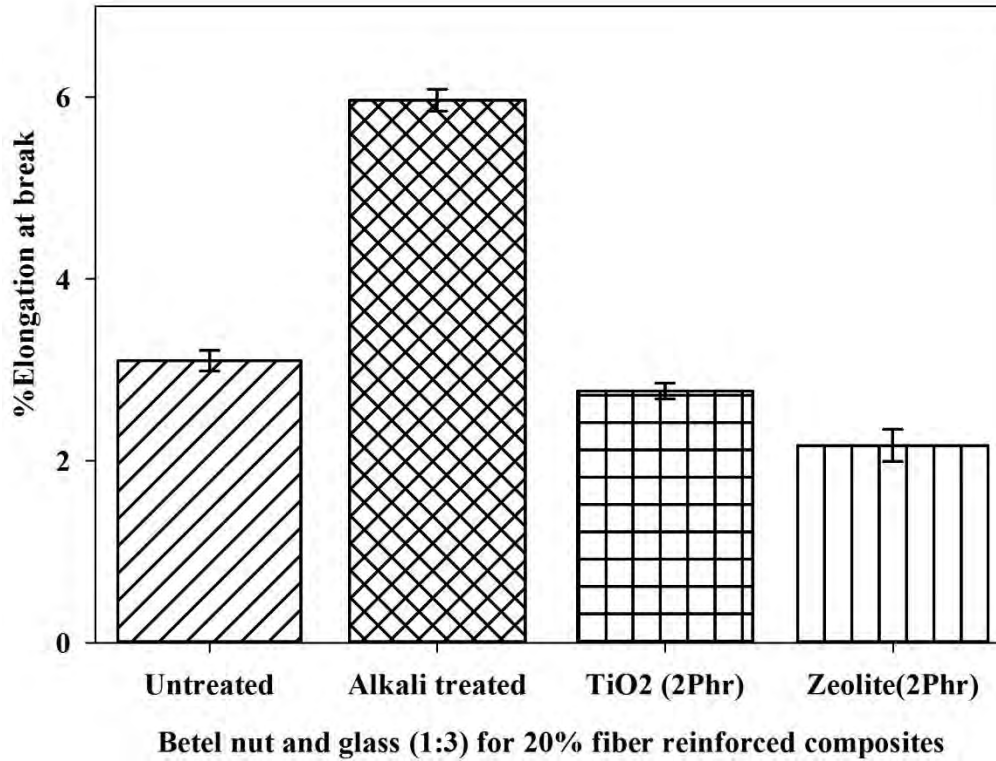


Figure 4.24: Variation of % elongation at break for 20% betel nut and glass (1:3) fiber reinforced PE composite

#### 4.4.2 Flexural Properties

According to the values provided in the Figure 4.23, flexural strength and flexural modulus value of treated betel nut reinforced composite was better than that untreated betel nut reinforced composite. As a consequence of alkali treatment the interfacial interaction between the fiber and matrix were enhanced, which increased the effective available surface area of contact with the matrix, and the possibility of load transfer between the matrix and the reinforcing fibers, this resulted into the increase of flexural strength and as well as modulus [54]. The increase in flexural strength may be due to the plasticizing behaviour of TiO<sub>2</sub> and zeolite in to the matrix [97].

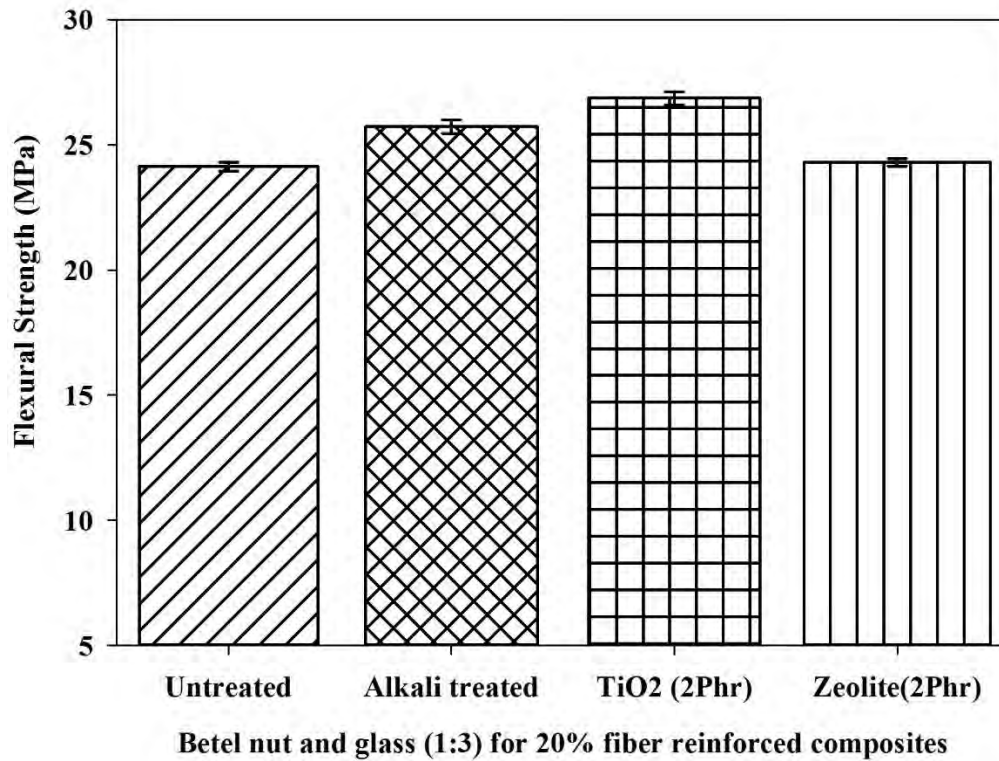


Figure 4.25: Variation of flexural strength for 20% betel nut and glass (1:3) fiber reinforced PE composite

The increase in flexural modulus with the increasing filler content is because the fillers increased the rigidity and stiffness behaviour of the polymer composite [96].



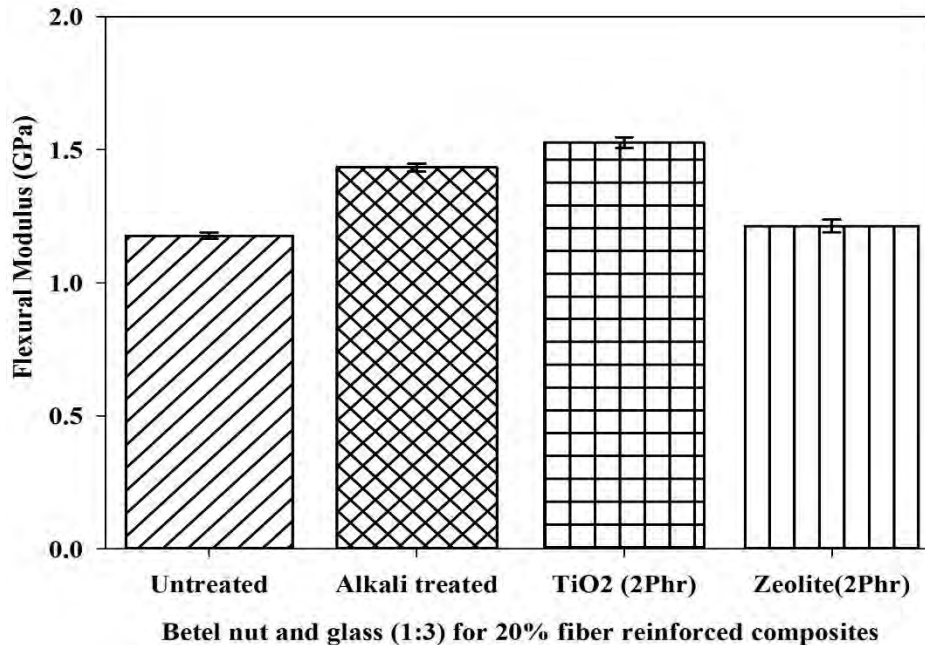


Figure 4.26: Variation of flexural modulus for 20% betel nut and glass (1: 3) fiber reinforced PE composite

#### 4.4.3 Hardness

Comparing results of raw and treated betel nut fiber reinforced composite, it is quite evident that hardness value increased with the incorporation of alkali treated fiber. Actually the alkaline treatment used on the fiber results collapse of the cellular structure due to the removal of cementing material, which leads to a better packing of cellulose chains and results into the reduction of void [95] and as well as better adhesion between the matrix and the filler. In this case, the polymeric matrix phase (PE) and the solid filler phase would be pressed together and touch each other more tightly. Thus the interface can transfer pressure more effectively. This might have resulted in an enhancement of hardness. It is clear that the hardness of the composites increased with increasing filler content. Again, the rigid fillers (TiO<sub>2</sub> and zeolite) increased the hardness of the respective composite as shown in Figure 4.27[96].

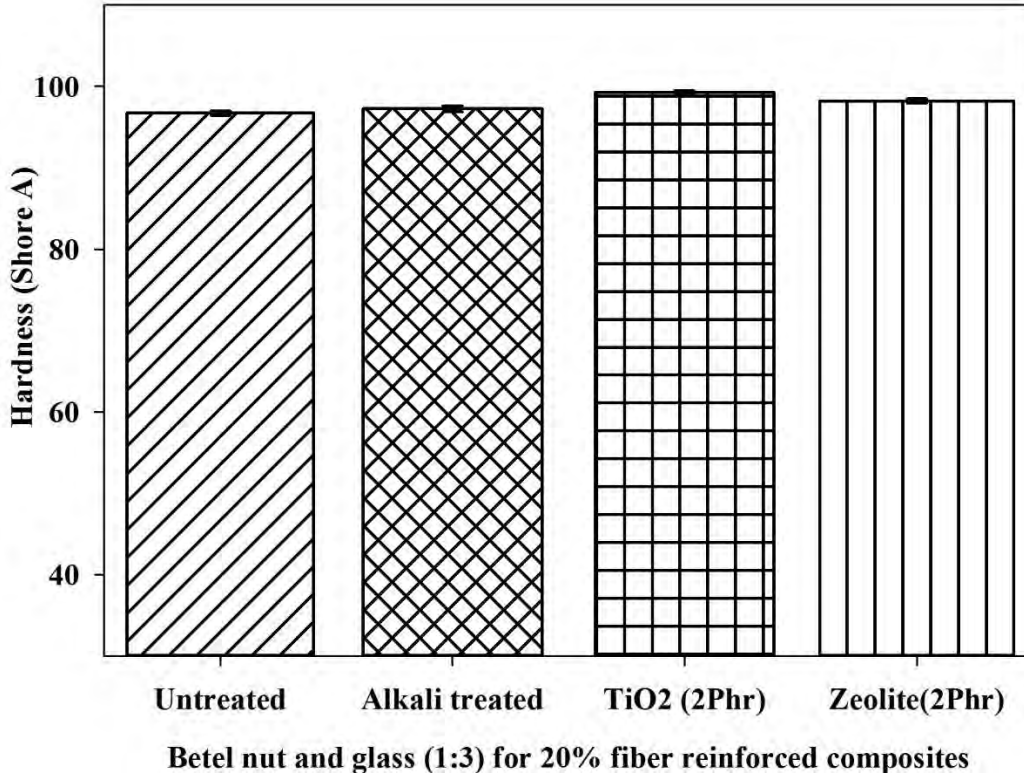


Figure 4.27: Variation of hardness for 20% betel nut and glass (1:3) fiber reinforced PE composite

#### 4.4.4 Impact Properties

The impact strength of the fiber reinforced polymeric composites depends on the nature of the fiber, polymer and fiber-matrix interfacial bonding [24,87]. As presented in Figure 4.28, impact strength of treated betel nut composites increased for fixed fiber loading. This result suggests that the fiber was capable of absorbing energy because of favorable entanglement of the fiber and matrix and this probability increased due to the alkaline treatment. As a consequence, stronger force was required to pull out the fibers. This in turn increases the impact strength [30]. On the other hand, well dispersed nanoparticles (TiO<sub>2</sub> and zeolite) into the matrix tends to increase the impact energy of the composites [97].

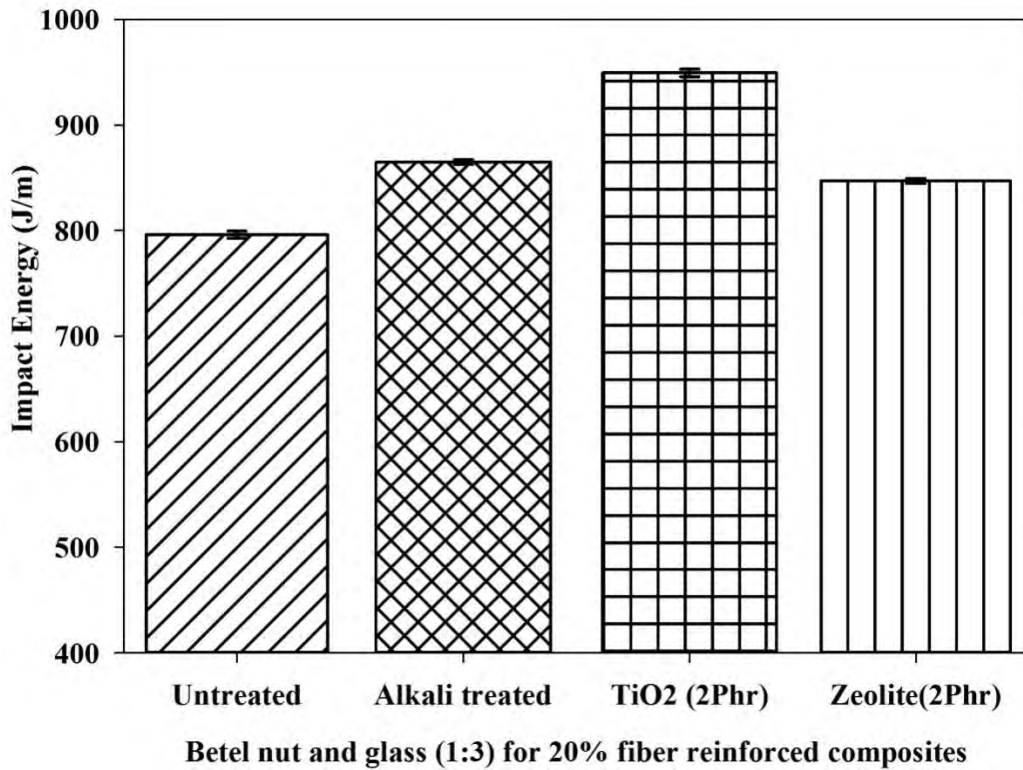


Figure 4.28: Variation of impact energy for 20% betel nut and glass (1:3) fiber reinforced PE composite

#### 4.4.5 Water Absorption Characteristics

Chemical modification is needed to reduce the moisture absorption of the fiber. Cellulose, hemicelluloses and lignin of betel nut fiber contain hydroxyl group. Hydroxyl groups form hydrogen bonds with water molecules. So hydroxyl group absorbs more water. As a result, water absorption of treated betel nut-PE composites were less than that of untreated betel nut reinforced PE composites. So water absorption percentages is higher for more betel nut fiber loaded composites. This is due to the effect of polarity of functional group[75]. On the other hand, due to high hydrophilic nature of TiO<sub>2</sub> and zeolite nano-powder water absorption percentages increased as the filler is added as shown in Figure 4.29.

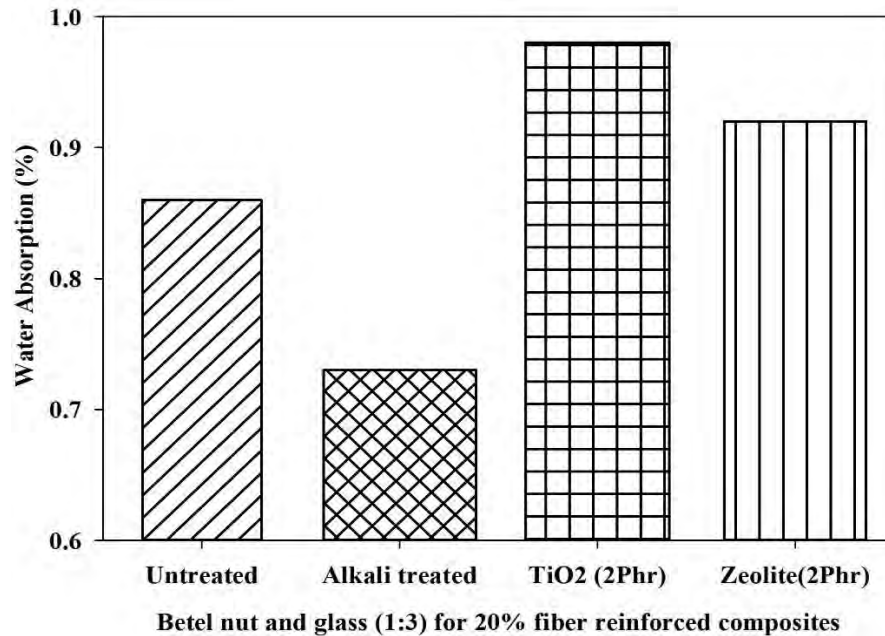


Figure 4.29: Variation of water absorption for 20% betel nut and glass (1:3) fiber reinforced PE composite

#### 4.5 Surface Morphology

The SEM micrograph of untreated betel nut fiber is shown in Figure 4.30, while SEM micrographs of sodium hydroxide (NaOH) treated betel nut fibers are shown in Figures 4.31 to 4.33. From these micrographs it is quite obvious that the alkaline treatment of fiber had effectively cleaned the fiber surface and had increased the fiber surface roughness. The removal of impurity also resulted in separation of fiber strands.

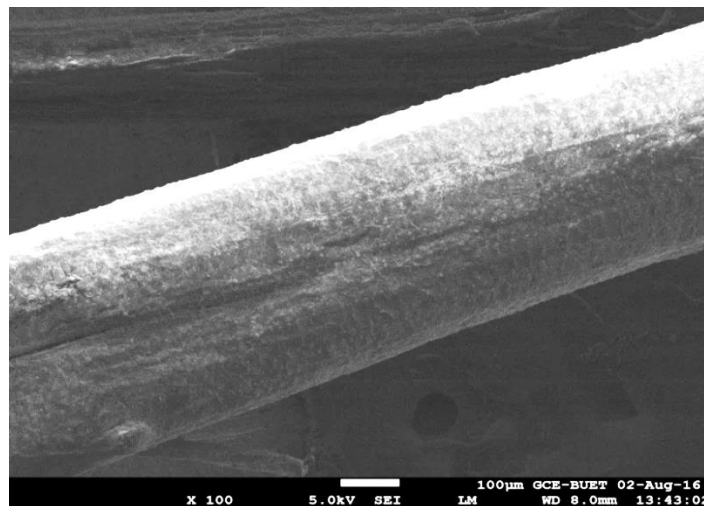


Figure 4.30: SEM micrograph of untreated betel nut fiber.

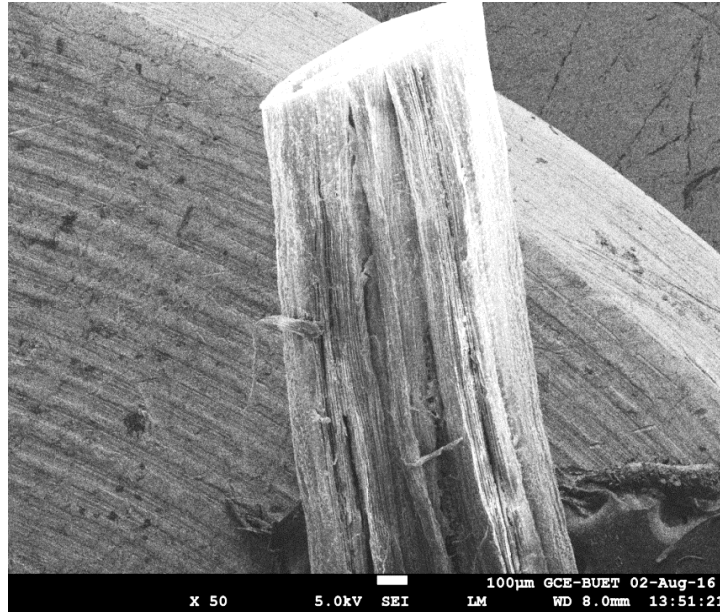


Figure 4.31: SEM micrograph of treated betel nut fiber showing fibrillation

Alkali treatment causes removal of waxy layers from the surface of betel nut fiber, which resulted in pit like pores on to the fiber surface. There are deep pores and also the presence of rough surface is quite evident from Figure 4.33. As a result of which more possible anchoring points for mechanical interlocking. The presence of the waxy layer prohibits the effective interfacial bonding between the natural fiber and polymer matrix. So removal of this low molecular weight components provides better wetting of the fiber surface with the matrix resin. Formation of elongated structure and fibrillated microfibrills are quite evident which provides more exposed area and better reaction sites as shown in Figure 4.31 [34].

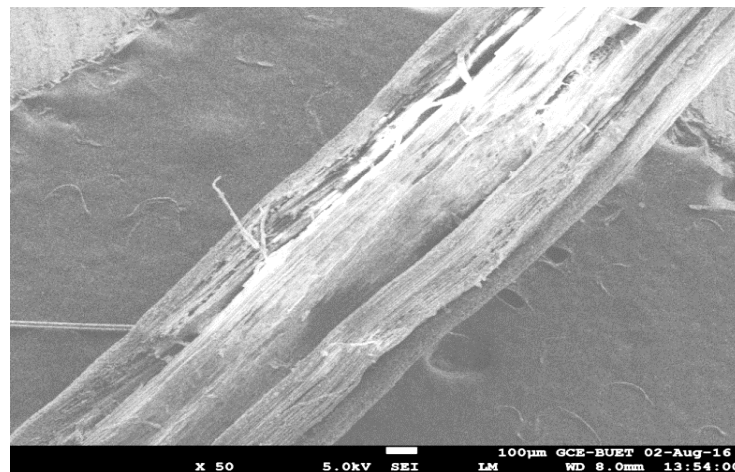


Figure 4.32: SEM micrograph of treated betel nut fiber

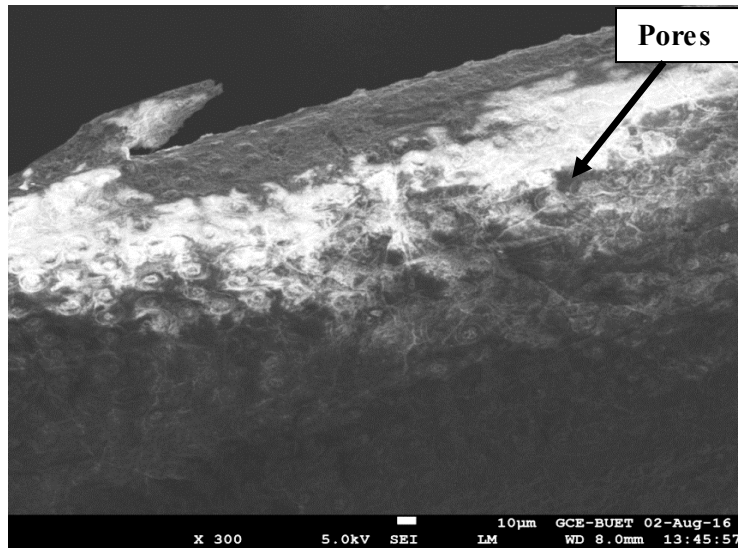


Figure 4.33: SEM micrograph of treated betel nut fiber showing porous surface

The SEM micrograph of glass fiber and tensile fracture surface of polyethylene are shown in Figures 4.34 and 4.35 respectively. The SEM micrographs of glass fiber shows numerous number of fiber bundle.

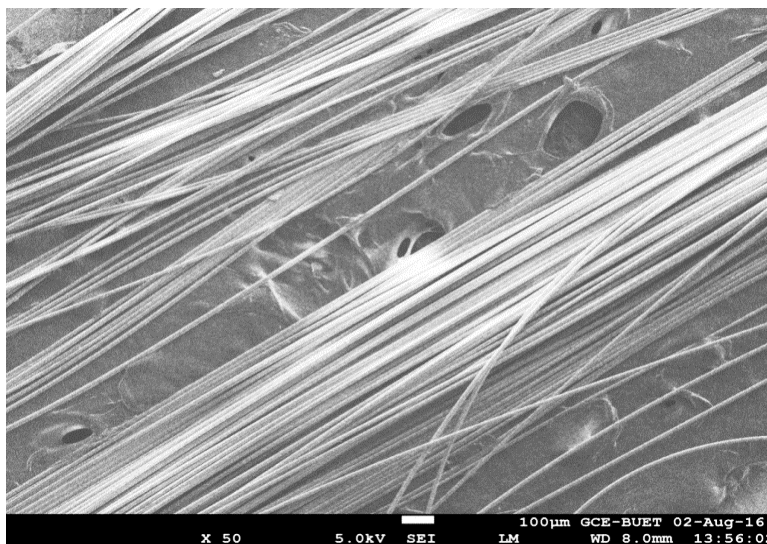


Figure 4.34: SEM micrograph of glass fiber

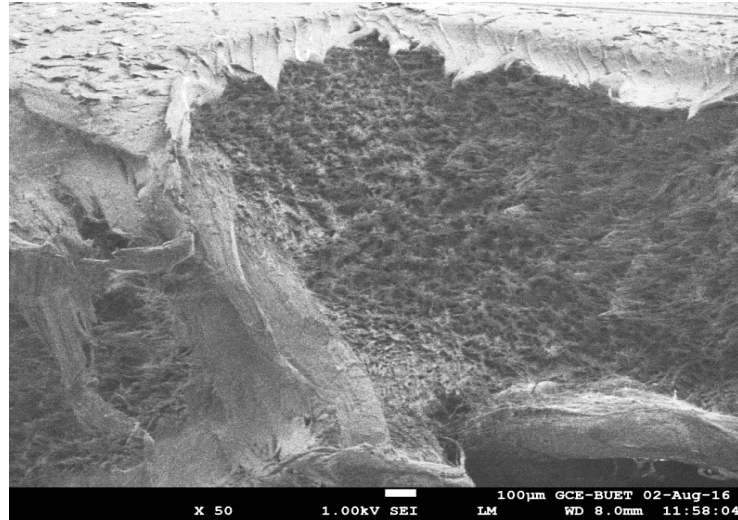


Figure 4.35: SEM micrograph of tensile fracture surface of PE

The tensile fracture surfaces of betel nut and glass fiber reinforced hybrid polyethylene composites are shown in Figures 4.36 to 4.38. The surface morphology provides information which reflects the reasons of variation of mechanical properties of the composites. The SEM micrograph of the betel nut and glass fiber (betel nut: glass=1:1) reinforced composites shows weak interfacial bonding of fiber and matrix, numerous voids as well as agglomeration of the fibers in polyethylene matrix as shown in Figure 4.36.

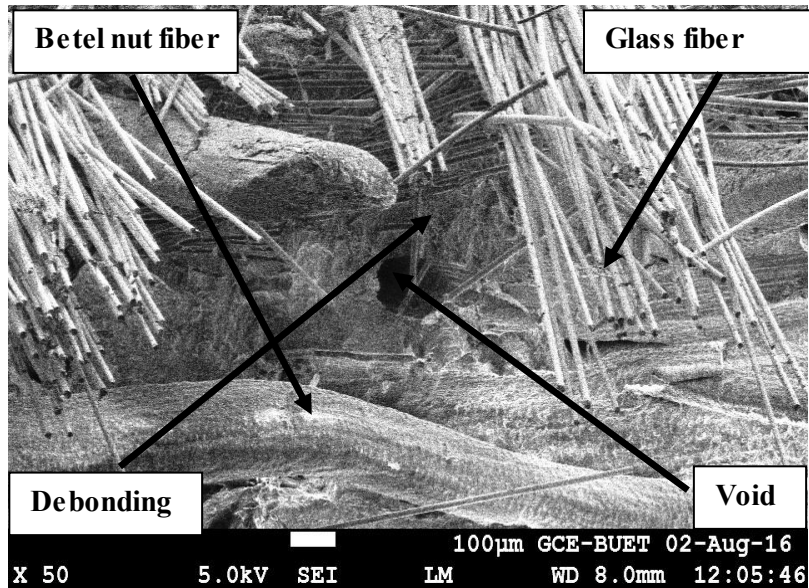


Figure 4.36: SEM micrograph of tensile fracture surface of 20% untreated betel nut and glass (1:1) fiber reinforced PE composite

SEM image of (betel nut: glass=3:1) fiber reinforced PE composite shows voids along with fiber agglomeration and weak interfacial bonding (Figure 4.37).

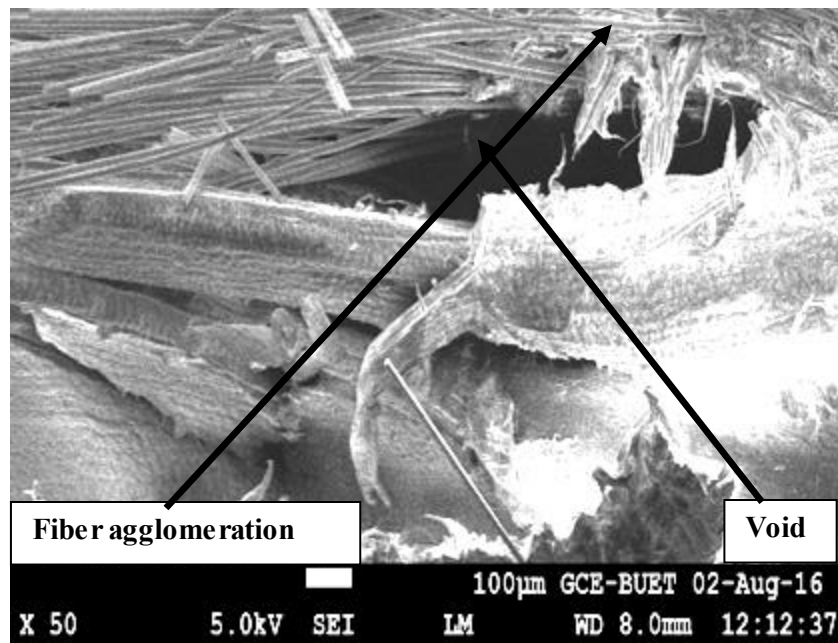


Figure 4.37: SEM micrograph of tensile fracture surface of 20% untreated betel nut and glass (3:1) fiber reinforced PE composite

On the other hand (betel nut: glass=1:3) fiber reinforced PE composites shows better dispersion of the filler into the matrix as clearly seen in the micrograph. Fiber pull-out traces and the agglomeration of the fibers in the matrix is substantially reduced here as shown in Figure 4.38.



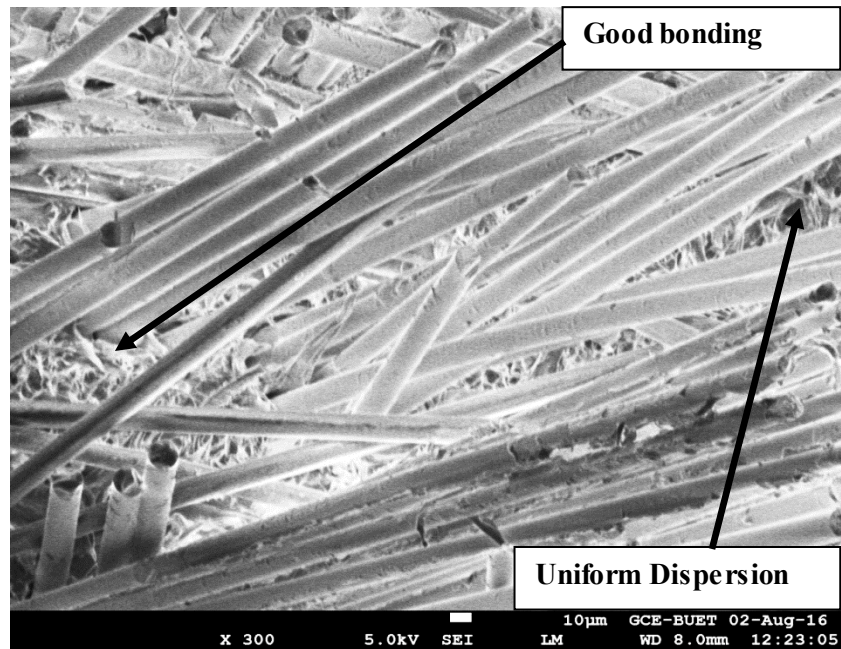


Figure 4.38: SEM micrograph of tensile fracture surface of 20% untreated betel nut and glass (1:3) fiber reinforced PE composite

The tensile fracture surface morphologies of treated betel nut and glass (1:3), betel nut and glass (1:3) with TiO<sub>2</sub> and betel nut and glass (1:3) with zeolite reinforced hybrid polyethylene composites are shown in Figures 4.39, 4.40 and 4.41 respectively. Comparison of SEM micrographs of untreated and treated fiber reinforced composites indicated weak interfacial bonding, whereas sodium hydroxide treated fiber composites showed improved fiber matrix adhesion. Mechanical interlocking involves diffusion of polymer matrix into porous fiber surface. The polymer matrix will flow into the porous fiber surface and the embedded polymer will solidify in the pores and become inextractable[49]. As clearly seen in Figure 4.39 that both fiber pull-out traces and the agglomeration of fiber in the matrix have substantially reduced providing evidence of better dispersion of fiber in matrix in alkali treated fiber reinforced composites [98].

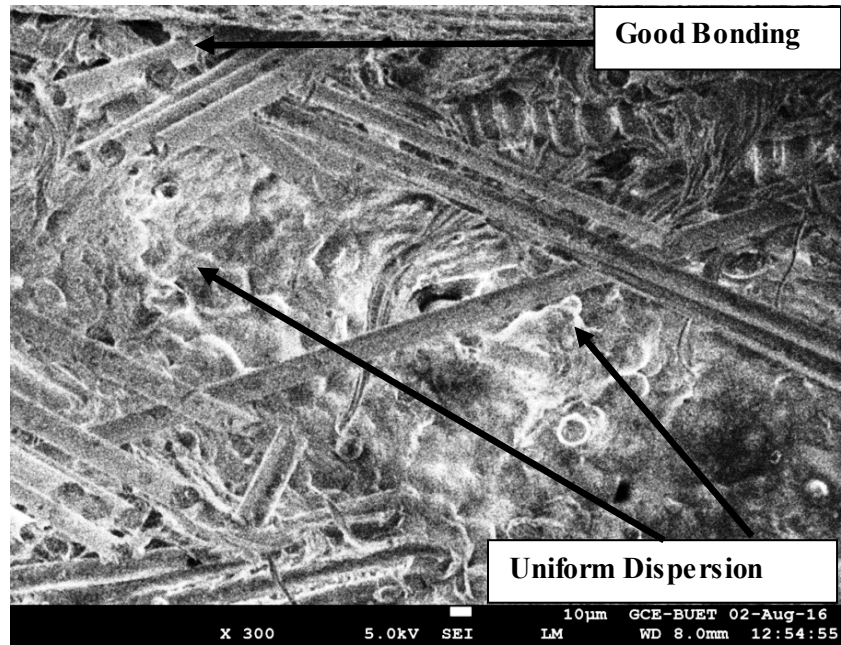


Figure 4.39: SEM micrograph of tensile fracture surface of 20% treated betel nut and glass (1:3) reinforced PE composite

The tensile fracture surface morphologies of 20% betel nut and glass fiber (1:3) reinforced hybrid polyethylene composites with the addition of titania and zeolite nano-powders are shown in Figure 4.40 and Figure 4.42 respectively. From those micrographs the presence of fiber matrix bonding and uniform dispersion of nano-powder filler in the matrix was quite evident. Fiber agglomeration along with voids was also found in the later micrographs than former one.

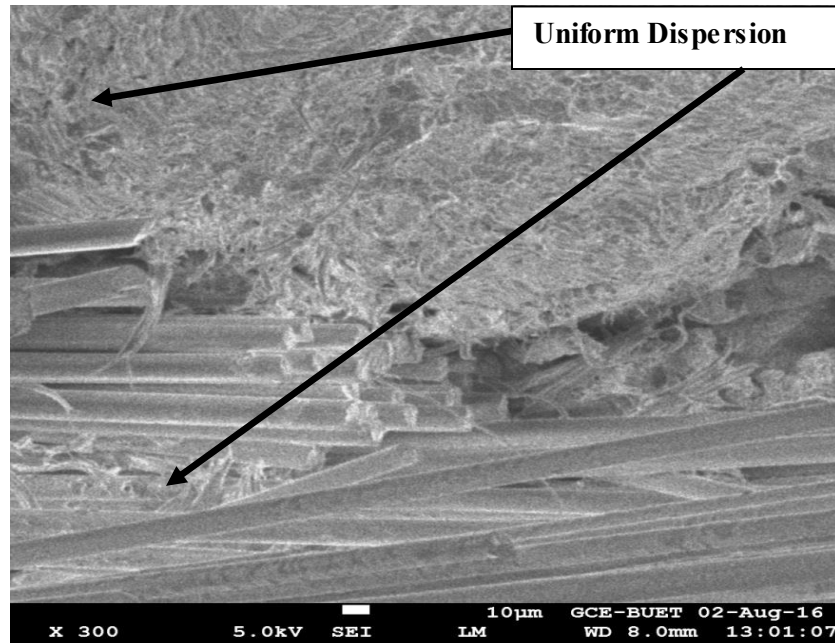


Figure 4.40: SEM micrograph of tensile fracture surface of 20% untreated betel nut and glass (1:3) fiber with TiO<sub>2</sub> nano-powder (2phr) reinforced PE composite

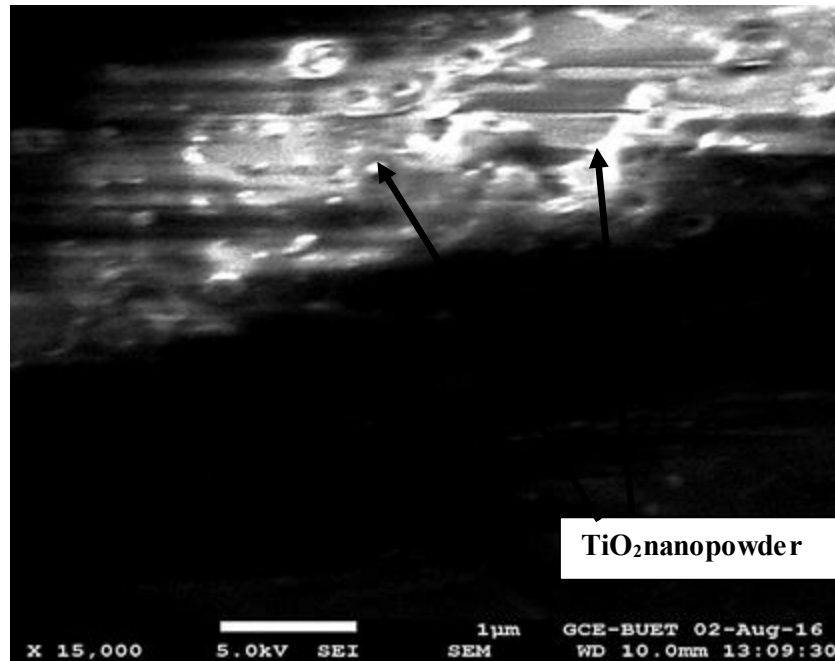


Figure 4.41: SEM micrograph of tensile fracture surface of 20% untreated betel nut and glass (1:3) fiber with TiO<sub>2</sub> nano-powder (2phr) reinforced PE composite showing TiO<sub>2</sub> nano-powder

The SEM micrographs of Figures 4.41 and 4.43 show the presence of TiO<sub>2</sub> and zeolite nano-powder in the respective composites.

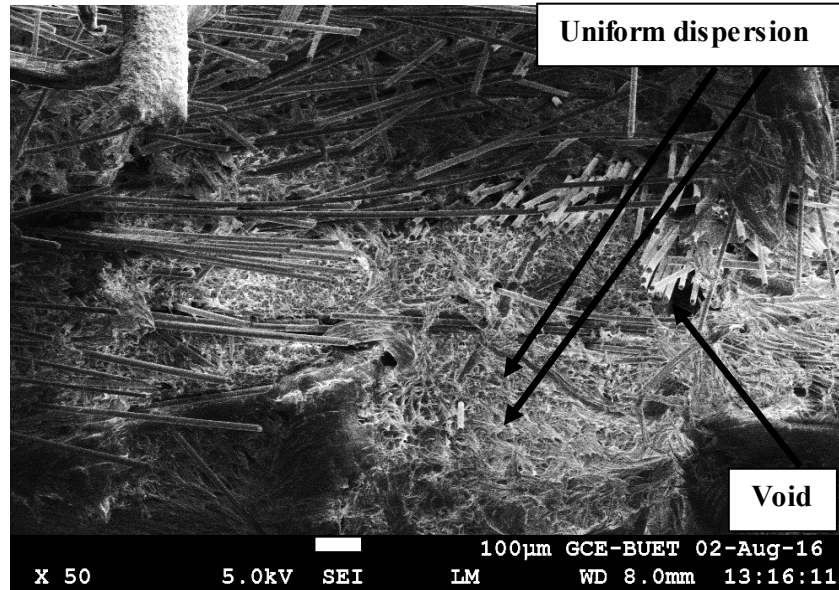


Figure 4.42: SEM micrograph of tensile fracture surface of 20% untreated betel nut and glass (1:3) fiber with zeolite nano-powder (2phr)reinforced PE composite

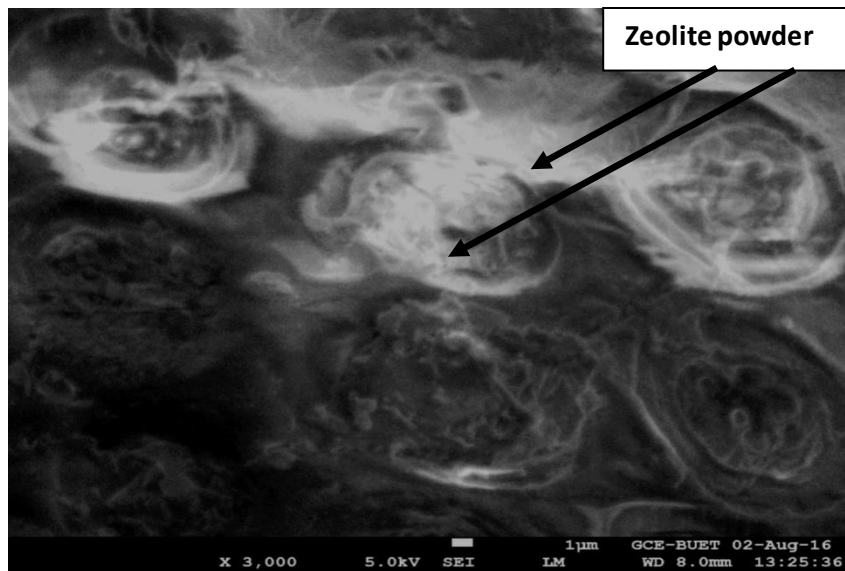


Figure 4.43: SEM micrograph of tensile fracture surface of 20% untreated betel nut and glass (1:3) fiber with zeolite nano-powder(2phr)reinforced PE composite showing zeolite nano-

powder

#### 4.6 Thermo Gravimetric Analysis

The TGA curves of polyethylene (PE) is shown in Figure 4.44. Thermal decomposition starts at around 340°C. In these cases, TGA curves represent one-stage decomposition and therefore represent the thermal stability limit of the composite. The starting of weight loss temperature has been calculated by using extrapolation technique because it is a reproducible temperature calculation and it is specified to be used by ASTM [99].

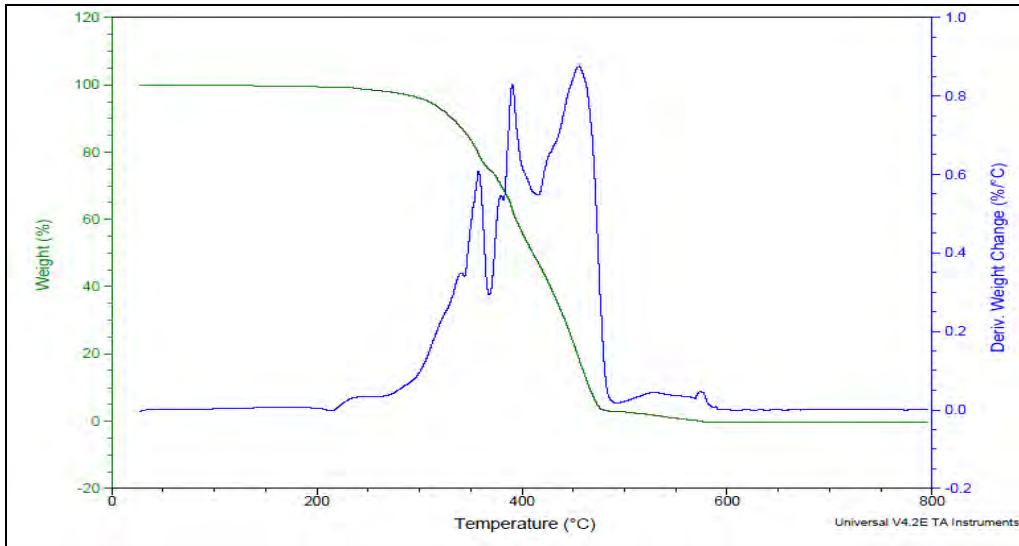


Figure4.44: TGA curve of PE

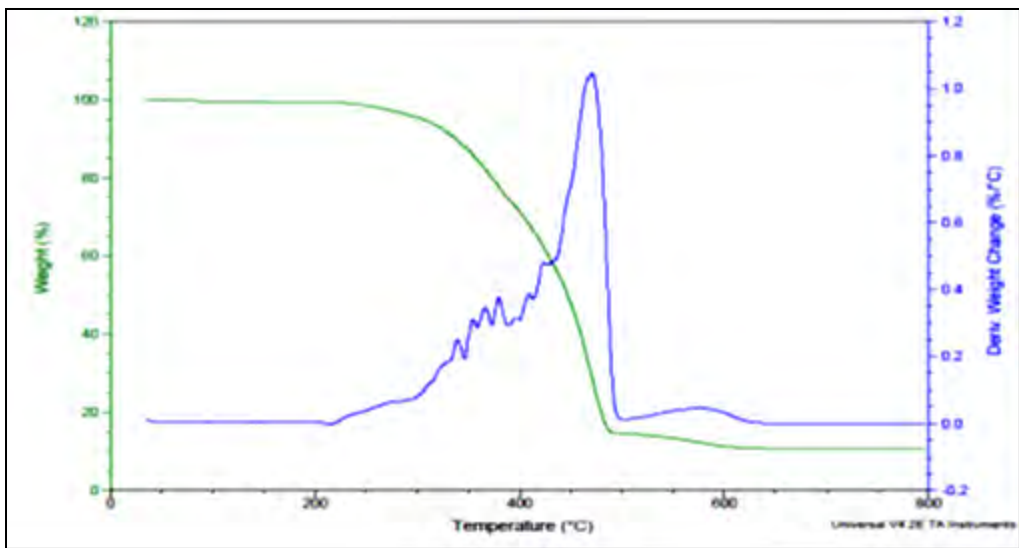


Figure 4.45: TGA curve of 20% untreated betel nut and glass fiber (1:3) reinforced PE composite

For the manufacturing of composites, the thermal stability of the fiber is very important. In present study, alkali treatment has shown little enhancement of thermal stability of the composite. From Figure 4.45, it can be seen that in case of untreated betel nut and glass fiber (1:3) reinforced composite thermal degradation starts at around 330°C. The decomposition starting temperature does not differ much in case of treated betel nut and glass fibers (1:3) reinforced composite which is at 345°C as shown in Figure 4.46. From this it can be concluded that the composites have better thermal stability untreated one which occurs due to the alkaline treatment of the natural fibers. The residual char increased considerably from 9% to 16% in the case of treated fiber reinforced composite. It is proposed that besides removing hemicellulose, NaOH treatment can give rise to a lignin-cellulose complex, therefore the NaOH treated fiber becomes more stable and this has reflected in the increased amount of residual char.

The thermal degradation of hemicelluloses occurred at around 200-260°C. Between 150°C and 300°C, cleavage of  $\alpha$ - and  $\beta$ -aryl-alkyl-ether linkages of lignin occurred. At around 300°C, aliphatic side chains started to split off from the aromatic ring. Finally, the carbon-carbon linkage between lignin structural units was cleaved at 370–400°C. The glycoside linkage of cellulose degrades at around 260°C [100]. The peak of degradation at around 470°C was due to the degradation of polyethylene.

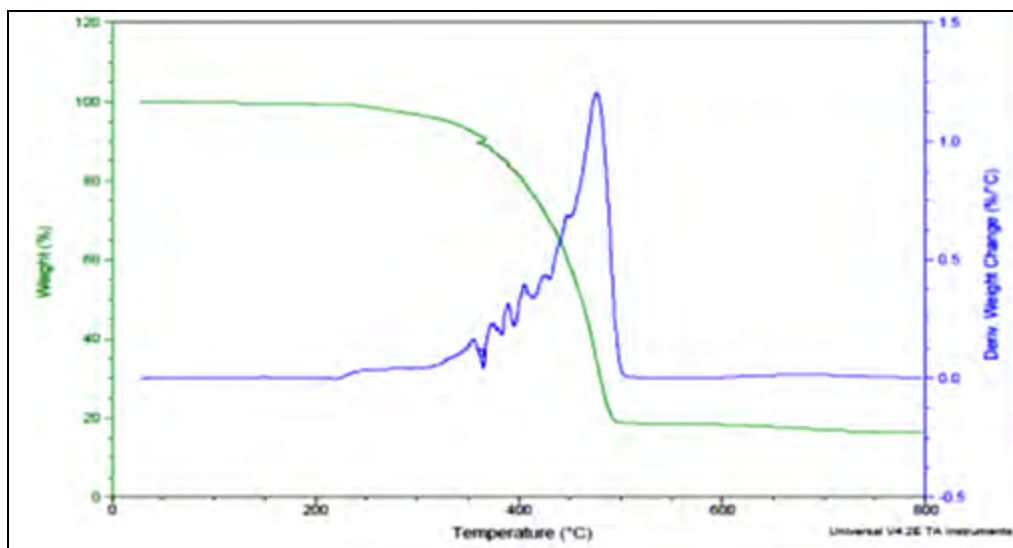


Figure 4.46: TGA curve of 20% treated betel nut and glass (1:3) fiber reinforced PE composite

It is clearly seen that the peak of hemicelluloses in case of untreated fiber reinforced composite was at around 260°C, which got vanished in case of treated reinforced composite due to the considerable removal of hemicelluloses during alkaline treatment. Also the peak of lignin at 370-400°C got lowered in case of treated fiber reinforced composite. In DTG curve of all these composites no peak was observed below 200°C. The major peak at about 320°C is due to the thermal degradation of cellulose which is the main component of betel nut fiber.

The thermal stability of PE decreased by the addition of TiO<sub>2</sub> nano-powder in betel nut and glass fiber (1:3) reinforced polyethylene composites as shown in Figure 4.47. Thermal degradation started in this composite at around 310°C and in the case of nano-composite small amount of residual product was left. It is usually assumed that upon completion of TGA, all carbon has been removed in forms of CO and CO<sub>2</sub> and that all remaining material consists of metal oxides. In case of PE with TiO<sub>2</sub> nano-composites less 2% amount of residual mass was found which is due to the polymer to filler ratio, since the activity in the presence of titania is very high, the filler weight percentages was very low which correspond to less residual char content in the TGA curve [101].

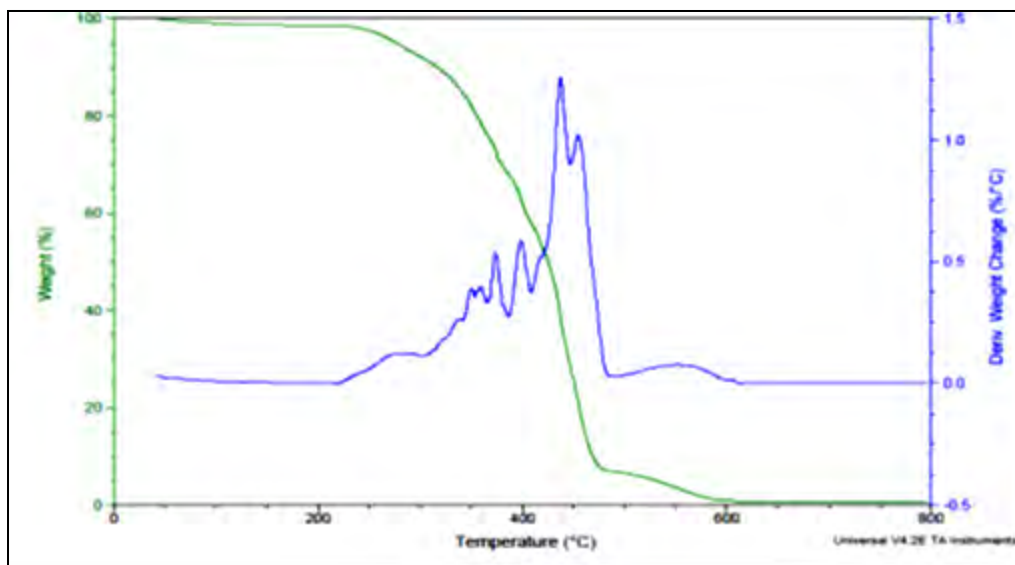


Figure 4.47: TGA curve of 20% untreated betel nut and glass (1:3) fiber with TiO<sub>2</sub> nano-powder reinforced PE composite

On the other hand, the thermal stability of PE increased by the addition of zeolite nano-powder in betel nut and glass (1:3) fiber reinforced polyethylene composites as shown in Figure 4.48. It

starts decomposition around at 370°C. Thermal decomposition occurred in multistage. DTG curve of the composites shows peak at about 370-400°C, which is due to the thermal degradation of lignin, component of betel nut fiber. The peak found at 580°C is due to synthetic glass fiber present in the composites.

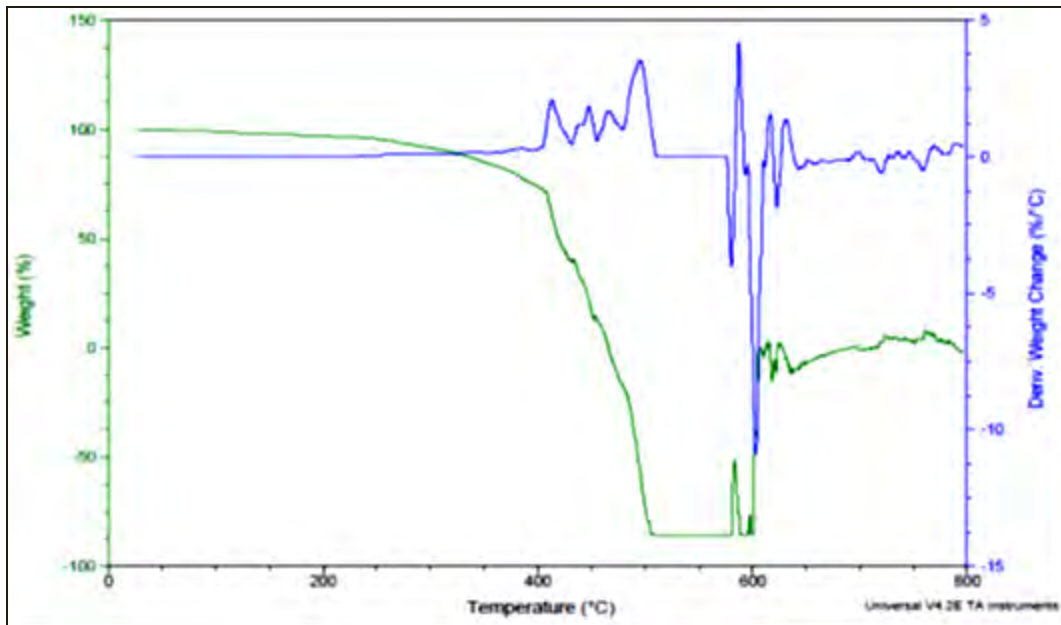


Figure 4.48: TGA curve of 20% untreated betel nut and glass (1:3) fiber with zeolite nanopowder reinforced PE composite



# **CHAPTER 5**

# **CONCLUSION**

## CONCLUSION

In the present research, hybrid composites were fabricated with hot press technique incorporating at four levels of fiber loading 5, 10, 15 and 20% with fiber ratios of (betel nut : glass=1:1) in polyethylene matrix. For 20% fiber loaded composites, fiber ratio were also varied (betel nut: glass=1:3, 1:1 and 3:1). Composites were also prepared with the addition of TiO<sub>2</sub> and zeolite nano-powder for betel nut and glass (1:3). The followings outcome can be concluded from this present research:

- ❖ The tensile strength of the hybrid composites decreased with increasing number of fiber loading. However, 5% alkali treated fiber reinforced composites showed higher tensile strength than raw fiber reinforced composites. In 5% alkali treated fiber containing composites, the enhancement of compatibility of the fiber with PE resulted into better stress transfer from the matrix to the fiber and thus increase in tensile strength in comparison to raw ones occurred. Higher percentages of glass fiber containing composite displayed better tensile properties than the higher betel nut containing one. On the contrary, TiO<sub>2</sub> nano-powder containing composites shows best tensile properties because of high dispersion and uniformity rather than zeolite nano-filled composite.
- ❖ % Elongation at break for fiber reinforced composite decreased with increasing number of fiber content. However 5% NaOH treated fiber reinforced composites showed better value than that of the raw fiber reinforced composite with same volume fraction of fiber. % Elongation at break decreased with the addition of nano-filler in the composites. Composite containing higher percentages of betel nut showed better elongation at break than that of the other systems.
- ❖ It was also observed that Young's modulus increased with the increase of fiber content. 5% NaOH treated fiber reinforced composite exhibited higher Young's modulus than the raw one. Besides TiO<sub>2</sub> nano-powder containing composites shows higher Young's modulus than alkali and zeolite nano-filled composites. Amount of glass fiber enhancement resulted in higher Young's modulus value.
- ❖ Flexural strength and flexural modulus increased with the increase in fiber content. The flexural strength and flexural modulus of 5% alkali treated fiber reinforced composite

was higher than that of the raw one. Higher amount of glass fiber containing composites demonstrated better flexural properties.

- ❖ SEM was carried out to reveal the adhesion between the fiber and the matrix and also the tensile fracture surfaces of different composites. FTIR was carried out to study the effect of alkali treatment which displayed the removal of hemicelluloses and also the presence of nano-filler. TGA was carried out to show the thermal stability limit of the composites and 5% NaOH alkali treated sample shows best result among all.
- ❖ In conclusion it can be said that betel nut and glass fiber can give a better prospect in improvement of hybrid composite and it will be also environmentally viable. Changing their ratio resulted in different combination of properties that could not be obtained using one fiber alone. Above all chemical modification of fiber surface and nano-filler addition can also help to obtain better set of properties.
- ❖ The widespread application of natural fiber is in automotive industry. Plant fibers are mainly used in the part of car interior and truck cabins. Hopefully this composite will be able to use in producing various automotive parts such as various panels, shelves, trim parts and brake shoes.

## **FUTURE WORK**

Development of new composite products from the easily renewable natural materials has a strong potential to deliver novel biodegradable and/or readily recyclable materials suitable for the automotive, packaging industry and all other applications. Hybrid composite reinforced with natural and synthetic fibers provide biodegradable products, easy way of recycling in conjunction with balanced pursuit of properties. In this present research, betel nut and glass fiber was used with polyethylene matrix. Different types of combination can be possible in different ways for manufacturing new composites with respect to its application and the research field in this sector is widespread.

- ❖ In present study only fiber surface modification was carried out by alkali treatment. Apart from alkali treatment large numbers of other chemical treatments like addition of coupling agent are available which can be used for fiber surface modification.
- ❖ Tests such as biodegradability and weathering can be conducted to observe its long time exposure characteristics.
- ❖ To enhance compatibility matrix modification can also be carried out. In this research, composite was prepared by reinforcing fibers but fiber orientation was random. To obtain different combination of properties fiber orientation can be changed.

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