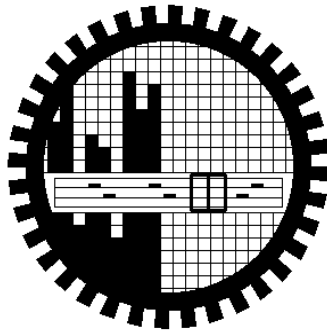


**INVESTIGATION OF MECHANICAL PROPERTIES  
OF JUTE STICK FLOUR REINFORCED  
POLYPROPYLENE COMPOSITES**

**MD. HAFIZUR RAHMAN**



**DEPARTMENT OF MECHANICAL ENGINEERING  
BANGLADESH UNIVERSITY OF ENGINEERING AND TECHNOLOGY  
DHAKA-1000, BANGLADESH**

**December 2011**

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FLOUR REINFORCED POLYPROPYLENE COMPOSITES**

**by**

**Md. Hafizur Rahman**

A thesis submitted for partial fulfillment of the requirements for the degree of  
**MASTER OF SCIENCE IN MECHANICAL ENGINEERING**

**DEPARTMENT OF MECHANICAL ENGINEERING  
BANGLADESH UNIVERSITY OF ENGINEERING AND TECHNOLOGY**

Dhaka-1000, Bangladesh

December 2011

## CERTIFICATE OF APPROVAL

The thesis titled “**INVESTIGATION OF MECHANICAL PROPERTIES OF JUTE STICK FLOUR REINFORCED POLYPROPYLENE COMPOSITES**” submitted by **Md. Hafizur Rahman**, Roll No: 1009102022P, Session: October 2009 has been accepted as satisfactory in partial fulfillment of the requirement for the degree of Master of Science in Mechanical Engineering on 12<sup>th</sup> December, 2011.

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Md. Hafizur Rahman

**DEDICATED  
TO MY  
BELOVED PARENTS**

## ACKNOWLEDGEMENT

The author would like to express his deepest sense of gratitude and thanks to his supervisor **Dr. Md. Afsar Ali**, Professor, Department of Mechanical Engineering, Bangladesh University of Engineering and Technology, Dhaka, for his kind supervision, constant guidance, encouragement, untiring support, and infinite patience throughout the entire course of this research. It was quite impossible to complete this study without his proper guidance.

The author would like to express his sincere gratitude to Dr. Md. Monimul Huque, Professor, Department of Chemistry, BUET, for his valuable suggestions, help, constitutive criticism, and providing the opportunity to work in the laboratory of the Department of Chemistry, BUET.

The author would also like to express his heartiest gratitude to Dr. Md. Abdul Gafur, Senior Research Engineer, BCSIR, Dhaka, for his guidance, suggestion, and help while carrying out experiments in BCSIR laboratories, Dhaka.

The author is thankful to Bangladesh Atomic Energy Commission, Dhaka where the SEM analysis was carried out and SEM photographs were taken.

The author gratefully acknowledges the support of Operators and Attendants of SM Laboratory of Mechanical Engineering Department, Bangladesh University of Engineering and Technology, Dhaka, during conducting experiments.

Finally, the author would like to thank Manabendra Saha, M.Sc. student, Department of Mechanical Engineering, BUET, for his assistance and co-operation during manufacturing of composites.

## **ABSTRACT**

Now a days, natural fiber reinforced polymer matrix composites had caused revolution in the field of materials and received wide attention of the researchers from all over the world because of their outstanding advantages including environment friendliness, biodegradability, recyclability, cost-effectiveness, and comparable physico-mechanical properties. Among various natural fibers, jute sticks(JSs) are potential natural materials which are easily available in Bangladesh at low cost. However, these materials have not yet considered for manufacturing their composites with polymers and exploring their properties. The present study is, therefore, intended to manufacture JS flour reinforced polypropylene (PP) composites with a view to investigating and evaluating their physico-mechanical properties. The JS flour and PP were melt and mixed with a single screw extruder. This mixture was used to manufacture composites with an injection molding machine. Both raw and chemically treated JS fibers were used in manufacturing the composites.

Various mechanical properties such as tensile strength and stiffness, flexural strength and stiffness, Charpy impact strength, and hardness were measured by standard procedures and results were analyzed. Water absorption property was evaluated for 24 hours immersion in distilled water. Scanning electronic microscopic (SEM) images were analyzed to understand the interfacial bonding between JS flour and PP matrix for both the raw and chemically treated fibers. Fourier transform infrared ray (FTIR) analysis was carried out to see the effect of chemical treatment on the change of molecular structures (hydrophilic to hydrophobic) of JS flour.

It was found that almost all the mechanical properties were satisfactorily improved for chemically treated JS-PP composites in comparison with those of raw JS-PP composites. However, water absorption property did not improve because of chemical treatments. Further, the water absorption increases with the increase of fiber loading.



## NOMENCLATURE

<b>SYMBOL / ABBREVIATION</b>	<b>MEANING</b>
$\theta_f$	Angle of rise during sample breaking
$\theta_i$	Angle of fall of pendulum
$\theta_o$	Free angle of rise
$\sigma_f$	Flexural strength
$\lambda$	Wavelength
$A$	Area of the cross-section of specimen below the notch
$b$	Width of beam
$d$	Thickness of beam
$D$	Distance between grips
$e_f$	Energy after breaking of specimen
$e_i$	Initial energy of pendulum
$e_1$	Lost energy per degree of swing
$E_B$	Modulus of elasticity

$G$	Gage length
$K$	Corrected impact energy in breaking the specimen
$L_o$	Overall length
$m$	Slope of the tangent to the initial straight portion of the load-deflection curve
$P$	Maximum load
$R$	Rate of cross head motion
$S$	Support span
$T_g$	Glass transition temperature
$T_m$	Melting point
$t$	Depth of beam
$\bar{\vartheta}$	Wave number
$W_{i,f}$	Weight of composite specimen before and after immersion
$Z$	Rate constant of straining of the outer fiber
ASTM	American Society for Testing and Materials
BCSIR	Bangladesh Council of Scientific and Industrial Research
FTIR	Fourier transform infrared ray

JS	Jute Stick
NF	Natural fiber
NNDMA	N,N-dimethyl-aniline
PP	Polypropylene
SEM	Scanning electronic microscopic
UTM	Universal testing machine

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## INTRODUCTION

### 1.1 INTRODUCTION

Originated from the Latin word ‘compositus’, composite can be defined as a structure or a substance, which is made up by mixing two or more distinct different substances. Generally speaking, any material consisting of two or more components with different properties and distinct boundaries between the components can be referred to as a composite material. From engineering view point, it refers to a complex material, be it organic (such as wood) or inorganic (such as glass fiber), in which two or more distinct yet structurally complementary substances (such as metals, ceramics, glasses and polymers) are combined to produce structural or functional properties not present in any individual components. The idea of combining several components to produce a material with properties that are not attainable with the individual components has been used by human beings for thousands of years. Correspondingly, the majority of natural materials that have emerged as a result of a prolonged evolution process can be treated as composite materials.

Most composites have two constituent materials: a binder or *matrix* and a dispersed phase or *reinforcement* [1]. The reinforcement is usually much stronger and stiffer than the matrix, and gives the composite its good properties. The matrix holds the reinforcements in an orderly pattern. Because the reinforcements are usually discontinuous, the matrix also helps transfer load among the reinforcements. Reinforcements basically come in three forms: *particulate*, *discontinuous fiber*, and *continuous fiber*.

Figure 1.1 illustrates the classification of composites based on reinforcements [1]:

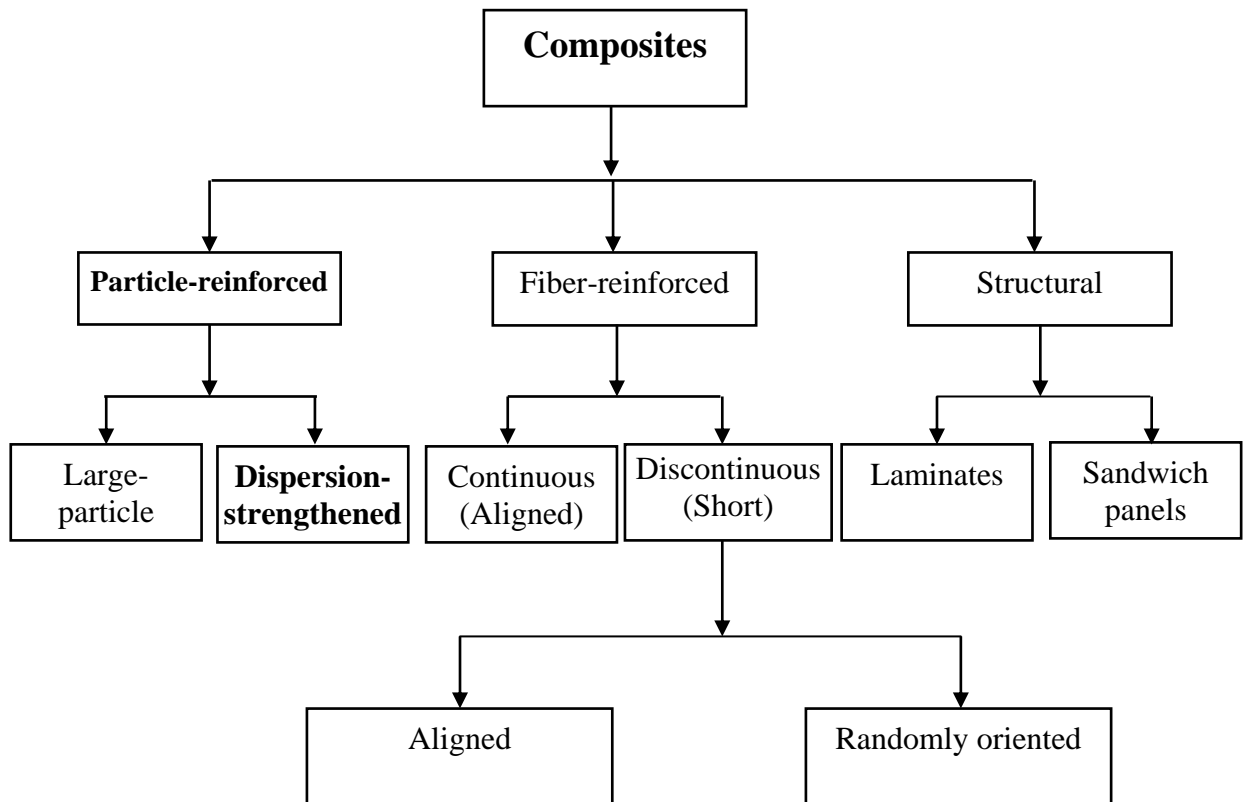


Fig. 1.1: Classification of composites based on reinforcement.

The matrix can be classified as thermoplastic (capable of being separately hardened and softened by the decrease and increase of temperature, respectively) or thermoset (changing into a substantially infusible and insoluble material when cured by the application of heat, or through chemical means) [2].

Typical reinforcements for plastics are various synthetic fibers such as glass, graphite (carbon), boron, inorganic, metallic, and ceramic. These materials are heavy, expensive, and harmful to the environment. Regarding to this environmental concern, now a days, the natural fibers have received significant attention instead of conventional synthetic fibers. One of the natural fiber sources is jute stick (JS), which contains lignocellulosic materials, such as cellulose, hemicelluloses and lignin. Any substance that contains both cellulose and lignin is a lignocellulosic material. Lignocellulosic fibers offer many advantages over these most commonly used synthetic fibers as filler or reinforcement in thermoplastic matrix. The replacement of

inorganic fillers with comparable lignocellulosic fibers provides less weight and decreases the cost without reducing the mechanical properties of the composites [3]. Lignocellulosic materials include wood, agricultural crops such as water hyacinth, jute, kenaf, sisal, etc., agricultural residues such as bagasse, corn stalks, rice husk, etc., and also other plant materials. In general, what is true for wood is also true for lignocellulosic materials even though they may differ in chemical composition and matrix morphology [4]. The use of lignocellulosic fibers as the reinforcing materials in thermoplastics has received a lot of interest due to their low densities, low cost, and non-abrasive nature [5]. The advantages of using lignocellulosic materials also include high specific stiffness and mechanical strength, ease availability, lower cost on unit-volume basis, low hardness, which minimizes the wear of processing equipment, renewability, recyclability, non-hazardousness, biodegradability, and so forth [6,7]. The use of lignocellulosic materials with thermosetting polymeric materials, like phenol- or urea-formaldehyde, in the production of composites has a long history. Disadvantages stemming from the use of thermosets include brittleness, lengthy cure cycles and inability to repair and/ or recycle damaged or scrapped parts. These disadvantages led to the development of the thermoplastic matrix composite system. Compared with thermosets, composites fabricated from thermoplastic materials typically have a longer shelf life, higher strain to failure, are faster to consolidate and can retain the ability to be repaired, reshaped and reused as need arises [8]. The processing temperature of the lignocellulosic fibers in thermoplastics is limited due to fiber degradation at higher temperatures. Thus, the plastics that can be used with it are limited to those with low melting temperatures. In general, no deterioration of properties due to fiber degradation occurs when processing temperatures are maintained below 200<sup>0</sup>C for short periods [4]. These thermoplastics include polypropylene (PP), polystyrene, vinyls, low-density polyethylene (LDPE) and high-density polyethylene (HDPE). The most common polyolefin used to prepare composites is polypropylene (PP), a commodity polymer [9]. Polypropylene provides most of the advantages with regards to economic (price), ecological (recycling behavior) and technical requirements (higher thermal stability like HDPE) [10]. Polypropylene is a semi crystalline polymer and its property and classification depends on its percent of crystallinity. The melting temperature of commercial polypropylene lies in the range 160-170°C, with purified polymer reaching to 176°C

[11]. In this research, JS fibers were used as reinforcing materials to prepare composites.

Bangladesh is an agriculture based country. It produces huge amount of jute every year. Jute is the cheapest lignocellulosic, long vegetable bast fiber and abundantly available here. It was called as 'Golden Fiber' of Bangladesh as it earned huge foreign currency for the economic growth of the country by exporting jute and jute products. Jute was an important foreign exchange earner of the then Pakistan during '60s. Even during '70s, jute was an important commodity of Bangladesh. However, during the '80s, bulk handling techniques and synthetic substitutes entered the market and jute started losing its predominant position in the market [12]. In Bangladesh, jute is traditionally used for making hessian clothes, ropes, gunny bags, shopping bags, backing of carpets, floor mats etc. Nevertheless jute fibers have many drawbacks. Jute fibers are woody, coarse, contain high percentage of lignin and are not spinable to fine fabrics. Its elasticity and compressibility are quite low. The color of jute and jute product changes from yellow to brown on exposure to light. It has poor crease resistance, lack of thermoplasticity and poor dimensional stability which are requirement for manufacturing fabrics. For these drawbacks, jute and traditional jute products are gradually replaced by artificial fiber and synthetic products.

In order to overcome the declining market of jute products, new technologies have been developed for bulk use of jute, as a raw material in the production of high value added and price competitive products. These products for new, alternative and non-traditional use of jute are generally termed as diversified Jute Products [12]. Among the various diversified jute products, jute fiber reinforced composites have high potential for wider use and application. The interest in using jute fibers as reinforcing agent in plastic composites has increased dramatically during the last few years. Production of jute reinforced composites using thermosets and thermoplastics can replace a variety of products including carbon and glass fiber composites.

Although many researchers have experimented with jute fibers reinforced polymer composites, no studies dealing with composites reinforced with jute sticks are available in the literature. So, the aim of the present research is to find out a cost effective technology of using jute stick fiber (JS) as a reinforcing material in



thermoplastic composites. These composites can be used for such products as doors, windows, furniture, gaskets, ceiling tiles, partition boards, automotive interior parts, packaging, molding, etc. Figure 1.2 shown below gives possible uses of different fractions of jute plant [13].

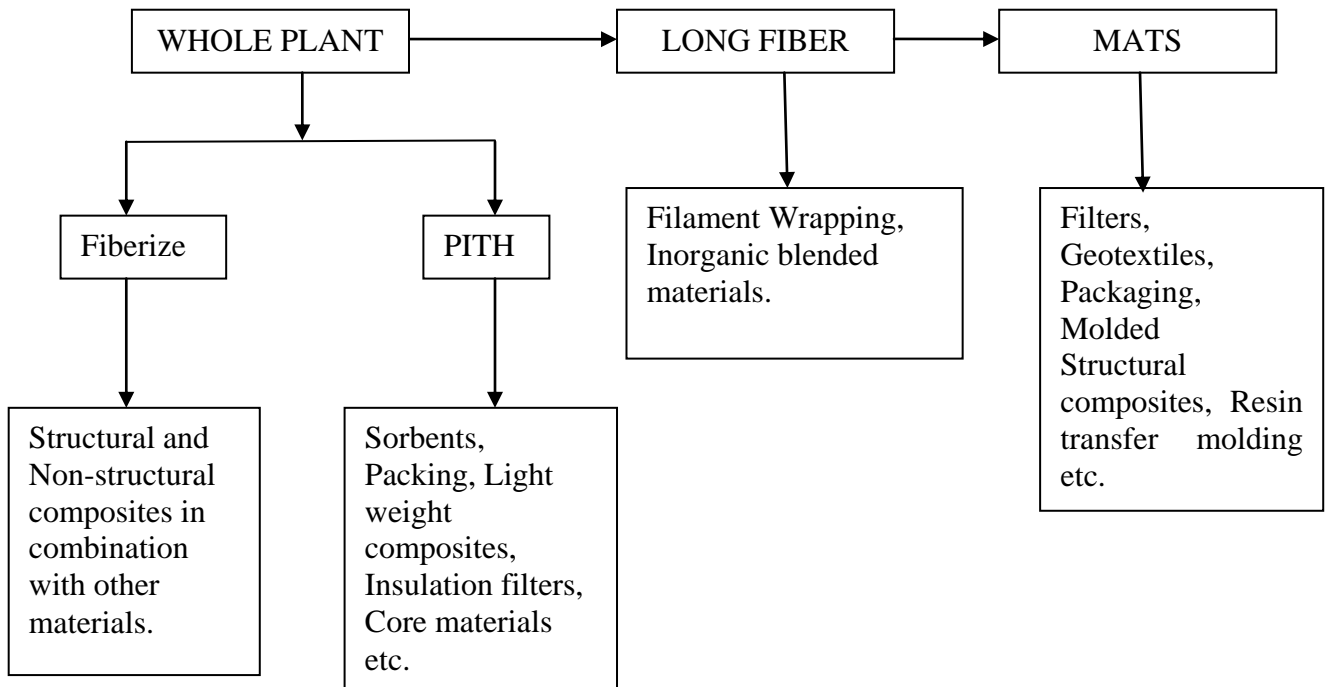


Fig. 1.2: Possible uses of different fractions of Jute Plant.

Natural fiber reinforced composites are inferior to synthetic fiber reinforced composites in tensile strength and modulus, but they exhibit significantly higher elongation, which provides better tolerance to composite damage. However, a major problem of natural fiber reinforced composites is their susceptibility to fungal and insect attack and to degradation by moisture [2]. The high moisture absorption of the natural fibers and their low microbial resistance are disadvantages that need to be considered, particularly during shipment and long-term storage as well as during processing of the composites [14]. In addition, their hydrophilic behavior affects the properties of the fibers themselves as well as the properties of their composites [14]. These limitations in performance of the natural fiber reinforced composites can be greatly improved through chemical modification techniques. Another problem of natural fibers is their tendency to entangle with each other and form fiber agglomerates during processing. The formation of fiber agglomerates prevents

complete fiber dispersion, and affects the properties and appearance of the finish products. The wide polarity differences of the surface of cellulosic fibers (hydrophilic) and thermoplastic matrix (hydrophobic) prevents efficient fiber-matrix bonding [4]. In these respects, surface modification of the fibers with coupling agents is generally needed because it will facilitate the fiber dispersion and induce bond formation between the fibers and the polymer. Coupling agents are able to react chemically (on both ends during processing) with the fibers on one side and the polymer on the other side, thus forming a chemical bridge/ coupling at the interface. Coupling is believed to be the utmost importance for perfect adhesion. Efficient coupling is usually not so simple. The lack of reactive groups in polymers prevents direct coupling of the components [14]. Thus, the modification of the matrix is often required. It is relatively easier for a modified polymer to introduce itself into the composite. Sufficient coupling is often achieved by chemical interaction and inter-diffusion [14]. The nature of the bond formed between a specific coupling agent and the fibers depends mainly on the characteristics of the fiber surface to which the coupling agent is adhered. Lignocellulosic fibers are complex in structure. Since a surface can be characterized by its chemical composition, the investigation on the chemical composition of fiber surface before and after the treatment would be very useful for the study of adhesion. In other words, an understanding of the fundamental mechanism by which the coupling agents form bonds either to the fibers or to the matrix, would be necessary to predict adhesion [15]. In this work, experimental studies on the interfacial adhesion and probable reaction bonding mechanism of chemically treated and untreated jute stick reinforced polypropylene composites by FT-IR spectroscopic and SEM analyses have been carried out. The results of surface modification and their effects on interaction in cellulose-polymer systems may lead to an increased number of applications for cellulose fibers in combination with polymers in our daily life.

## 1.2 OBJECTIVES

The specific objectives of the present research work are to

- 1) Fabricate biodegradable, environment friendly, and low cost JS-PP composites.
- 2) Carry out experiments to investigate different mechanical properties such as tensile and bending strength and stiffness, impact strength, hardness of the composites.
- 3) Investigate the effect of wt% of jute stick flour on mechanical behavior of composites.
- 4) Investigate the water uptake properties of the composites.
- 5) Evaluate the effect of chemical treatment of jute stick on the above mentioned properties of the composites.
- 6) Characterize the interfacial bonding between PP matrix and JS flour in the composites by Fourier Transform Infra Red (FT-IR) and morphological Scanning Electron Microscopy (SEM) analysis.

In this study, commercial isotactic polypropylene was used as the matrix material to prepare JS-PP composites by injection molding. The melting point of PP was found to be 160<sup>0</sup>C. Isotactic structure of PP was confirmed by FTIR spectroscopy. A comparative study on the improvement of physico-mechanical properties of JS flour treated with diazonium salt/alkali has been investigated. The performance of chemically treated JS flour reinforced polypropylene composites comparing with untreated raw JS flour reinforced polypropylene composites have been observed and reported.

## LITERATURE REVIEW

This chapter presents a brief review of literatures related to the composite materials, matrix materials (PP), reinforcing fiber materials (Jute Stick), fabrication of composites, and chemical adjustment of composites that are related to the research work. The details of different chemical treatments of natural fibers and their effects on the properties of composite materials are also discussed.

### 2.1 INTRODUCTION AND CLASSIFICATION OF COMPOSITE MATERIALS

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

Recently, polymer composites have become an important class of engineering materials. The properties of composites are largely influenced by the properties of their constituents and the distribution and interactions among them. The constituents usually interact in a synergistic way, providing properties that are not accounted for by a simple volume-fraction sum of the components. Along with the volume fraction and the distribution of discrete units in the continuous phase, the interfacial area plays an important role in determining the extent of interaction between the reinforcement and the matrix and in this way the final properties of the composite. The fabrication technology of composites, as well as some of their physical properties, is dominated by the chemistry and rheology of the matrix resin and by the type and physical form of the reinforcement [2].

The use of reinforcing agents makes it possible for any thermoset- or thermoplastic-matrix property to be improved or changed to meet varying requirements. Thus most polymer composites have been developed or fabricated to improve mechanical properties such as strength, stiffness, or toughness. The strengthening efficiency of the discontinuous phase plays the most important role in these products, and the strengthening mechanism depends strongly on the geometry of the reinforcements. Therefore, polymer composites can be classified according to geometry of reinforcement materials. The three major classes of polymer composites are fibrous, laminar and particulate. The commonly accepted classification scheme for polymer composites is presented in Figure 2.1 [2].

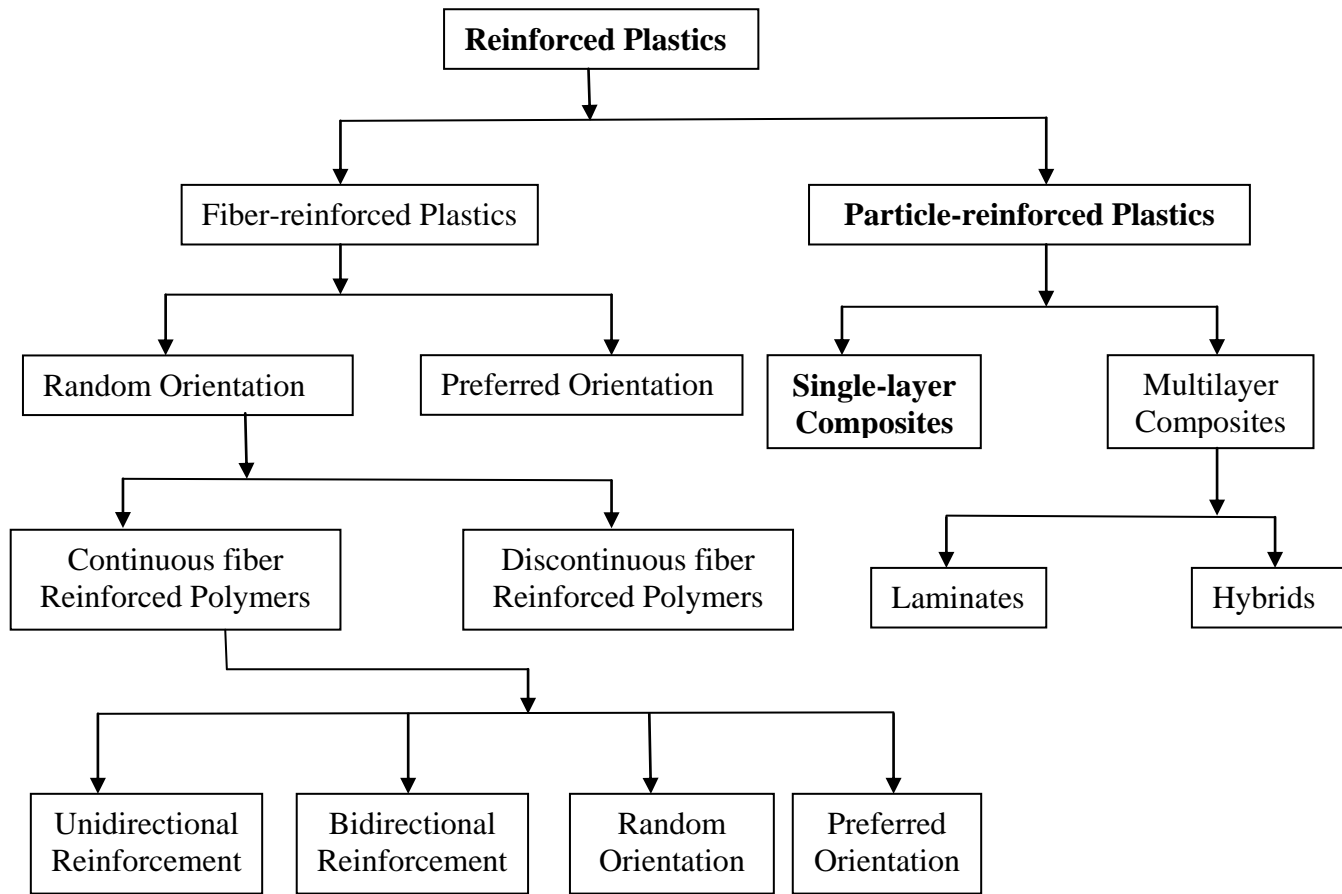


Fig. 2.1: Classification of polymer composites.

### 2.1.1 Matrix materials

The mechanical properties of composites reinforced with lignocellulogic fibers depend on the characteristics of the fibers and the matrix itself as well as on the fiber-matrix interfacial adhesion. The matrix in a polymer composite serves both to maintain the position and orientation of the fibers and to protect them from potentially degrading environments. Polymer matrices may be thermosets or thermoplastics. Thermosetting polymers are rigid, cross-linked materials that degrade rather than melt at high temperatures. These polymers are changed into a substantially infusible and insoluble material when cured by the application of heat or through chemical means. Thermoplastic polymers are linear or branched molecules capable of being separately hardened and softened by the decrease and increase of temperature, respectively. Thermoset-based composites are somewhat less expensive than thermoplastic-based composites, but have lower heat distortion

temperatures and poorer toughness when tested in an interlaminar mode. Thermosetting polymers, includes unsaturated polyesters, epoxies, and phenolic resins whereas thermoplastic polymers are polyamides, polyolefins, polycarbonates and saturated polyesters [8].

Many of the thermoplastic-based composites offer excellent resistance to impact loading and are thereby suitable for use in high performance engineering applications. Other interesting aspects of these composites include the possibility of thermoforming and shaping at elevated temperature and the potential for thermal joining and repair, as well as recycling [8].

### **2.1.2 Thermoplastic polymer**

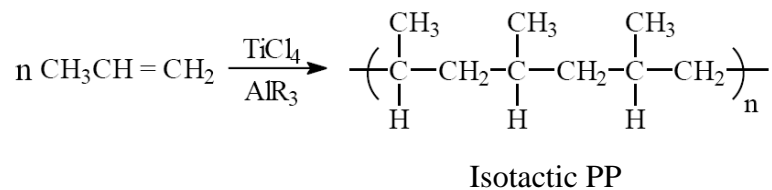
Thermoplastic resins have received considerable attention in the past few years as the matrix material in organic resin-based composites [8]. Thermosetting materials date back to 5000 years, when Egyptians used a straw-reinforcing agent in a clay matrix to form bricks. Compared to thermosetting composites, thermoplastic composites are relatively new development. Although their use in advanced composites is not widespread, thermoplastic composites are used extensively in commercial applications ranging from automobiles to durable goods [8].

Thermoplastic polymers are usually linear molecules with no chemical linkage between the chains of the molecules. The molecules are held together by weak secondary forces, such as Van-der-Waals or hydrogen bonding. These are readily deformed by the application of heat or pressure. Thermoplastic resins can be amorphous (structureless or semicrystalline), in which some of the molecules form an ordered array. A material is usually considered semicrystalline if at least 5% of the polymer is present in the crystalline form. Semicrystalline resins exhibit higher modulus, but amorphous materials are tougher. Amorphous materials are usually more solvent sensitive and can be processed at lower temperature. One of the most important advantages of thermoplastic resins is their toughness that is, high impact strength and fracture resistance, which, unfortunately, is not linearly transmitted into properties of the composite. Other advantages of thermoplastic polymers include long life at room temperature; post formability, that is, thermal reforming; ease to

repair by thermal welding or solvents; and ease of handling, that is, they are not tacky [8]. One of the principal advantages of true thermoplastic polymer is their ability to consolidate or flow at elevated temperature; however, this quality also limits their use at higher temperature. Amorphous materials begin to flow or creep above the glass-transition temperature,  $T_g$ , whereas crystalline resins must be heated above the melting point,  $T_m$ . As a rule of thumb,  $T_g$  is approximately  $2/3 T_m$  (Kelvin temperature). So, crystalline polymers may begin to degrade at the temperatures required for processing. Even a relatively low temperature material, such as polypropylene, which melts between 168 and 175°C, must be heated to approximately 180-190°C to process the composite. In many instances, the processing window is quite narrow owing to a lower temperature limit set by the melting point and an upper temperature limit set by the rate of thermal degradation [8].

### 2.1.3 Polypropylene (PP)

Polypropylene (PP), also known as polypropene, is a thermoplastic polymer. Most commercial polypropylene is isotactic and has an intermediate level of crystallinity between that of low-density polyethylene (LDPE) and high-density polyethylene (HDPE). PP has been in commercial production for the last 40 years following its controlled polymerization by Natta in 1954 [8, 16]. Natta used a Ziegler catalyst consisting of titanium tetrachloride and an aluminum alkyl to produce isotactic polypropylene directly from propylene:



Polypropylene (Figure 2.2) [17] is available with many different reinforcing agents or fillers, such as talc, mica, or calcium carbonate; chopped or continuous fiber. Glass is the most common reinforcing agent used in composites. Many additives have been developed to enhance the thermal stability of polypropylene, to minimize



degradation during processing. One of the most important requirements of the polypropylene used in the manufacture of composites is that, it should be relatively pure and free of residual catalyst. Recent developments to form copolymers of polypropylene and polyethylene have great promise for relatively inexpensive, tough, thermoplastic applications [8].

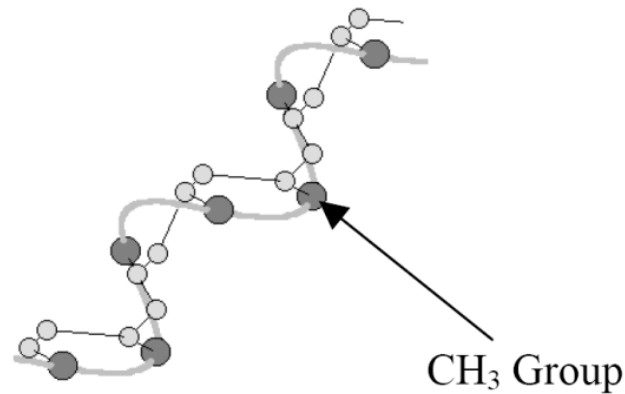
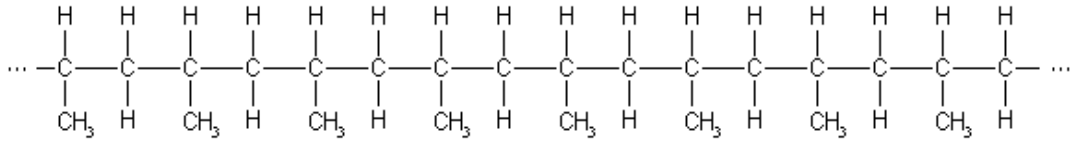


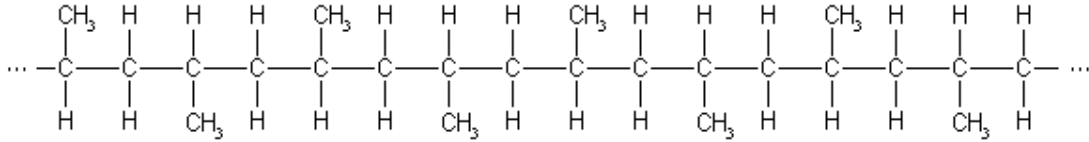
Fig. 2.2: Schematic of structure of polypropylene.

#### 2.1.4 Molecular structure of PP

In 1955 Natta [10] first determined the molecular structures of the various types of PP and introduced the descriptive nomenclature universally used today. Commercial interest lies primarily in highly crystalline PP, together with its further modifications through copolymerization. In isotactic PP (Figure 2.3 (a)), all methyl groups are positioned at the same side with respect to the backbone of the polymer chain without any branching. This is caused by the template type constraints of the stereospecific catalytic site. In syndiotactic configuration (Figure 2.3 (b)), methyl groups are alternatively on either side of the carbon chain. Syndiotactic polymer is of theoretical, but not commercial importance [10].



(a) Isotactic polypropylene; Melting Point = 176<sup>0</sup>C



(b) Syndiotactic polypropylene; Melting Point = 130<sup>0</sup>C

Fig. 2.3: PP chain structures.

### 2.1.5 Crystallization of PP

PP is semi-crystalline, which means it always has two phases, an amorphous and a crystalline one. The useful properties of PP originate from its ability to crystallize and form rigid and tough objects. Highly isotactic polymer, with its regular structure, forms a helical coil having three monomer units per turn. These coils stack together into thin lamellar crystallites which force the chains to fold several times as they emerge and reenter lamellae [10]. Three crystalline forms are known (Table 2.1) of which the  $\alpha$ -form is the most stable. The preparation more stable  $\alpha$ -form is favored at higher temperatures and slower cooling rates. Rapid quenching yields the  $\beta$ -form with a lower density and melting point of 150<sup>0</sup>C. Polymers of lower stereoregularity and random copolymers usually contain low-melting  $\gamma$ -crystallites in addition to the  $\alpha$ -form.

Formation of crystallites from a viscous melt is hindered by chain entanglements and by the need for helices to fold as they close pack into lamellae. This and the formation of nuclei might contribute to the low degree of crystallization even in highly stereoregular polymers. Commercial PP varies from 30% crystallinity in rapidly quenched films to 50-60% in moldings. Crystallinity of PP really enhances up to 70% in purified and annealed samples of very stereoregular polymer. It is to be

pointed out that high helical contents are not synonymous with high crystallinities, which require three-dimensional long-range order. PP is properly regarded as a semicrystalline polymer for these reasons [10]. Crystalline properties of PP are given in Table 2.1.

**Table 2.1: Crystalline properties of PP [10]**

Crystal form	System	Crystal density (20°C), g/cm <sup>3</sup>	Chains per unit cell	Melting Point, °C
Iso $\alpha$	Monoclinic	0.932-0.943	4	171
Iso $\beta$	Pseudo Hexagonal	0.922	9	150
Iso $\gamma$	Triclinic	0.939	1	131
Amorphous	-	0.85	-	-
Syndio	Orthorhombic	0.93	2 (4/1 helix)	138

### 2.1.6 Properties of PP

Polypropylene is a linear hydrocarbon polymer containing little or no unsaturation. It is therefore not surprising that polypropylene and polyethylene have many similarities in their properties [17]. Basic mechanical properties are largely influenced by molecular mass and molecular mass distribution (MMD), chain stereoregularity, and processing conditions, which introduce orientation, structural strain, etc. Various additive packages must be tailored to the application to achieve good and durable performance. Some typical properties of PP are given in Table 2.2. The melting point of commercial PP lies in the range 160-170°C, with purified polymer reaching to 176°C. Within the range of commercial polymers, the greater the amount of isotactic material the greater the crystallinity, and hence, greater the softening point, stiffness, tensile strength, modulus and hardness [17]. The crystalline and nonpolar nature of PP confers good resistance to a wide range of aqueous and polar media, including emulsifier solutions with their strong stress cracking abilities. Powerful oxidizing agents, highly aromatic and chlorinated solvents are too aggressive for safe storage in PP containers [10]. One disadvantage of PP is the susceptibility of its methyl groups to thermo-oxidative degradation [18].

**Table 2.2: Properties of PP [17, 18]**

<b>Properties</b>	<b>Standard Used</b>	<b><math>\alpha</math>-PP</b>	<b><math>\beta</math>-PP</b>
Melting temperature [°C]	-	160	170
Density of crystalline phase [g/cm <sup>3</sup> ]	-	0.936	0.921
Density of amorphous phase [g/cm <sup>3</sup> ]	-	0.858	0.858
Yield stress [MPa]	ASTM D638	36.5	29.5
Tensile modulus [GPa]	ASTM D638	2	1.5
Tensile strength [MPa]	ASTM D638	39.5	44
Elongation [%]	ASTM D638	420	480
Impact strength, notched Izod, J m <sup>-1</sup>	ASTM D256	21	53
Rockwell hardness (R-scale)	ASTM D785	95	90
Izod impact energy [kJ/m <sup>2</sup> ]	-	4000	4520

### 2.1.7 Selection of polypropylene as matrix

Polypropylene (PP) is one of the common substances in modern society. PP is used in almost every sector of our life. But the only problem is that it is not biodegradable. It is creating new environmental problem. On the other hand it makes human life so easy that we can not stop the use of PP. An intermediate strategy may be taken which is to reduce the use of PP. Jute stick flour reinforced PP composite will serve not only the diversified application of JS but also reduction of use of PP. The following factors are responsible for selecting PP as matrix:

- (1) Cheap in price
- (2) Low melting point
- (3) Non adhesive
- (4) Not harmful for the health
- (5) Low density

## 2.2 LIGNOCELLULOSIC FIBERS

Lignocellulosic fibers refer to those agro-based substances which contain cellulose, hemicelluloses, and lignin as their structural molecules. Lignocellulosics include wood, agricultural residues, water plants, grasses, and other plant substances [19]. To better understand the properties of lignocellulosic fiber-reinforced composite materials, it is necessary to know the physical and chemical properties of lignocellulosic fibers. Fibers are classified as [20, 21]

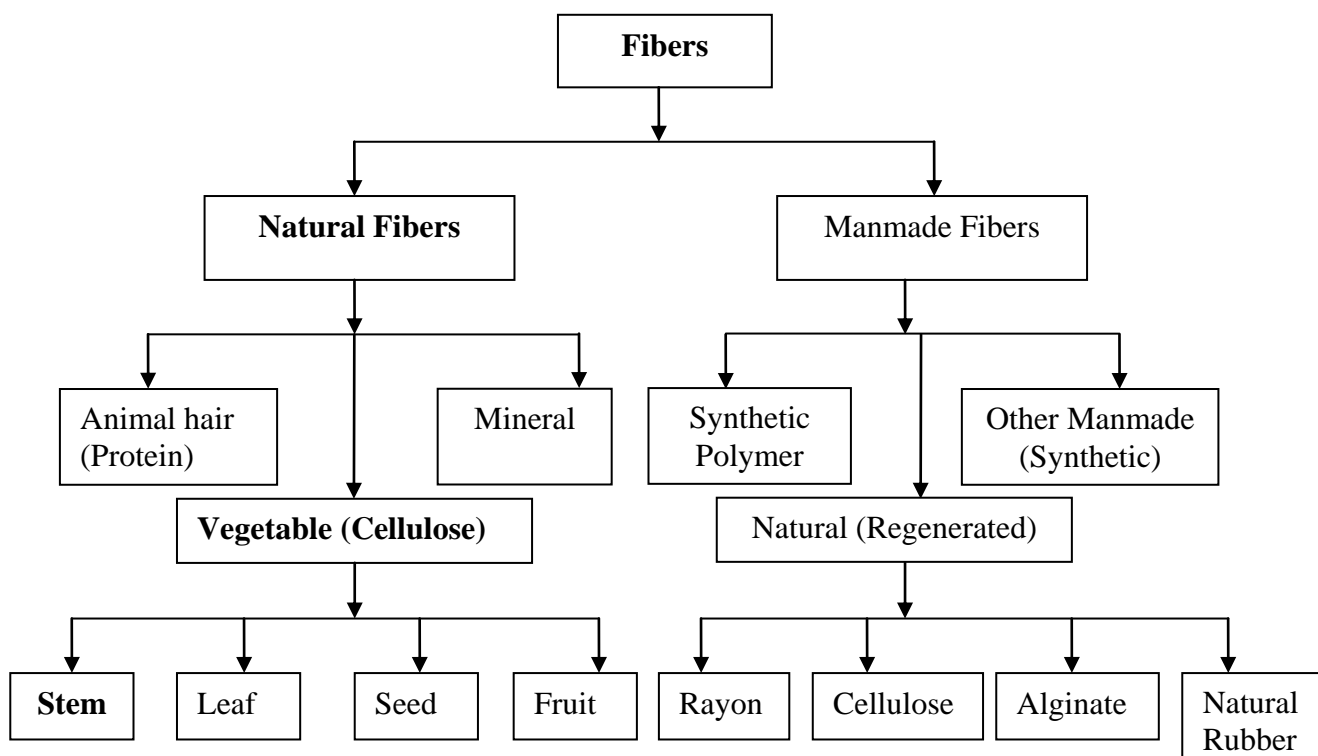


Fig. 2.4: Classifications of fibers.

Agro-based lignocellulosic fibers are also classified on the basis of part of the plant where they store in [13]. Five different classes of fiber are: (1) bast or stem fibers, which are the fibrous bundles in the inner bark of the plant stem running the length of the stem; (2) leaf fibers, which rim the length of leaves; (3) seed-hair fibers; (4) core, pith or stick fibers, which form the low density, spongy inner part of the stem of

certain plants; and (5) all other plant fibers not included above [13]. Examples of bast or stem fibers include jute, flax, hemp, kenaf, ramie, roselle, and urena. Leaf fibers include water hyacinth, banana, sisal, henequen, abaca, pineapple, cantala, caroa, mauritius, and phormium. Seed-hair fibers include coir, cotton, kapok, and milkweed floss. The remaining fibers include roots, leaf segments, flower heads, seed hulls and short stem fiber. While individual single fibers in all of these classes are quite short (except for flax, hemp, ramie, cotton, and kapok), the fibers obtained from inner bark of the plant are quite long. For example, hemp, jute, and kenaf can have fiber bundles as long as 400 cm and abaca, mauritius, and phormium are about half this length. Considering all types of plant fiber, there is a vast array of potential long and short fibers for composite production [13].

### **2.2.1 Jute stick**

Jute is a bast fiber extracted from the stem of *Corchorus capsularis* and *Corchorus olitorius*. In the trade, there are usually two names of jute, White and Tossa. The jute extracted from *Corchorus capsularis* is called White Jute and the jute extracted from *Corchorus olitorius* is called Tossa Jute. It is a major cash crop of India (1.23 million tons), Bangladesh (1 million ton) and Thailand (0.24 million ton). It is a significant fact that for every ton of fiber produced from the jute plant about 2.5 tons of jute sticks are obtained. At present jute sticks are used for thatching roofs, fencing gardens and as a fuel despite their low calorific value. Besides these wastes, other wastes of jute processing are root cutting, caddis and feshwa. Root cutting is the hard portion of the jute fiber which is cut off in the mills to avoid difficulty in processing, caddis is a waste from the weaving process, and feshwa are short fibers which are adhered to jute stick after the removal of long strands of jute fiber by retting when the whole jute plant is subjected to malleating before retting [22].

### **2.2.2 Chemical compositions of jute stick**

The ingredients for both jute fiber and jute stick are mainly alpha cellulose, lignin, hemicelluloses, ash. However, these constituents vary between jute fiber and jute stick. The following table compares the percentages of these components [22]

Table 2.3: Chemical compositions of Jute fiber and Jute stick

Constituent	Jute Fiber (%)	Jute Stick (%)
Alpha Cellulose	61	40.8
Lignin	11.5	23.5
Hemicellulose	24	32.9
Ash	1.6	0.8
Others	1	1.0

### **2.3 LIGNOCELLULOSIC FIBER REINFORCED THERMOPLASTIC COMPOSITES**

There is a long story of the use of lignocellulosic materials with thermosetting polymeric materials, like phenol or urea formaldehyde in the production of composites. The use of lignocellulosics with thermoplastics, however, is a more recent innovation. Lignocellulosic fibers have been incorporated in a wide variety of thermoplastic materials such as polypropylene, polyethylene, polystyrene, poly-vinyl chloride, polyamides. Thermoplastic materials selected for use with lignocellulosic materials must melt at or below the degradation point of the lignocellulosic component, normally 200-220<sup>0</sup>C [19].

#### **2.3.1 Lignocellulosics in combination with other materials**

Combination of lignocellulosic materials and thermoplastic materials can be done in several ways [23]. In one case, thermoplastic materials are simply mixed with bio-based powder or fiber (nut shell powder or wood fiber are presently used) and the mixture are heated. The plastic melts, but the wood fiber and plastic components remain as distinct separate phases. One example of this technology is reinforced thermoplastic composites, which are lightweight, have improved acoustical, and heat reformable properties, and low cost than comparable products made from plastic alone. These advantages make possible the exploration of new processing techniques,

new applications, and new markets in such areas as packaging, furniture, housing and automobiles.

Another way to combine wood fiber and plastics is to use a compatibilizer to make the hydrophobe (plastic) mix better with the hydrophil (wood). The two components remain as separate phases, but if delamination and/ or void formation can be avoided, properties can be improved over those of either separate phase. These types of materials are usually referred to as wood fiber/ plastic blends [23].

A final combination of wood fiber and thermoplastics is in products that can best be described as wood-plastic alloys. In this case, the wood and plastic have become one material and it is not possible to separate them. The formation of wood-plastic alloys is possible through fiber modification and grafting research. This can be done if we consider that bio-based fibers consist of a thermoset polymer (cellulose) in a thermoplastic matrix (lignin and the hemicelluloses). The glass transition temperature (GTT), however, of the thermoplastic matrix is higher than the decomposition temperature of the fiber. If the GTT were lowered through chemical modification, it should be possible to thermoplasticize the lignin and the hemicelluloses at temperatures below decomposition. If a reactive thermoplastic is used it is then reacted with the modified bio-based fiber, it should be possible to form bio-based fiber/ thermoplastic alloys [23]. Rowell et al [24] reported that only the hemicelluloses and lignin have been modified in thermoplasticization using maleic and succinic anhydrides. Matsuda et al [25] also extensively investigated the esterification of wood in order to make a totally thermoplastic material. They esterified wood without a solvent by simply heating wood powder with succinic anhydride in a mixer for 3 h at temperatures higher than 60<sup>0</sup>C.

### **2.3.2 Properties of lignocellulosic fiber reinforced thermoplastic composites**

Before 1980, the concepts of blends and alloys were essentially unknown in the plastics industry. However, blends and alloys have now revolutionized the plastics industry, as they offer new materials with properties that were not achieved before. These materials (blends and alloys, more specially composites) can be tailored for specific end uses. Newer materials composites that have both economic and



environmental benefits are being considered for applications in the automotive, building, furniture, and packaging industries. Mineral fillers and fibers are used frequently in the plastics industry to achieve desired properties or to reduce the cost of the finished product. For example, glass fiber is used to improve the stiffness and strength of plastics, although there are several disadvantages associated with its use. Glass fiber production requires a great deal of energy-processing temperatures 1200°C. They tend to abrade processing equipment and increase the density of the plastic system. Because of these drawbacks, the use of lignocellulosic fibers in thermoplastics has received a lot of interest due to their low densities, low cost, and non-abrasive nature. However, lignocellulosic fibers are polar in nature (hydrophilic) which do not make good bond with non-polar (hydrophobic) plastic leading to weak interfacial shear strength. This characteristic of weak interfacial shear strength was observed in the study of jute and kenaf fibers and polyolefin's matrix that resulted in inefficient composites [4]. The high moisture absorption property of the lignocellulosic fibers and their low microbial resistance along with other disadvantages listed in Table 2.4, need to be considered, particularly during shipment and long-term storage as well as during processing of the composites [9]. One way to get rid of these problems is the proper selection of additives to improve the interaction and adhesion between the fiber and matrix phases.

**Table 2.4: Main advantages and disadvantages of lignocellulosic fibers [9]**

<b>Advantages</b>	<b>Disadvantages</b>
<ul style="list-style-type: none"> <li>➤ Low cost</li> <li>➤ Renewable</li> <li>➤ Low density</li> <li>➤ Nonabrasive</li> <li>➤ Low energy consumption</li> <li>➤ High specific properties</li> <li>➤ High strength and elasticity modulus</li> <li>➤ No skin irritations</li> <li>➤ No residues when incinerated</li> <li>➤ Fast water absorption/ desorption</li> <li>➤ Good thermal conductivity</li> <li>➤ Biodegradability</li> </ul>	<ul style="list-style-type: none"> <li>➤ High moisture adsorption</li> <li>➤ Poor microbial resistance</li> <li>➤ Low thermal resistance</li> <li>➤ Local and seasonal quality variations</li> <li>➤ Demand and supply cycles limited</li> </ul>

Jute sticks and other lignocellulosic materials change dimensions with changing moisture content. The cell wall polymers of lignocellulosic fibers contain hydroxyl and other oxygen-containing groups that attract moisture through hydrogen bonding [26]. This hydrophilic behavior affects the properties of the fibers themselves as well as the properties of their composites. The hydrophilic character of fibers is usually incompatible with hydrophobic matrix material unless a compatibilizer or coupling agent is used. This leads to poor interfacial adhesion between the fiber and matrix as well as poor fiber dispersion.

In general, cellulosic fillers have a higher Young's modulus as compared to commodity thermoplastics, thereby contributing to the higher stiffness of the composites. The increase in the Young's modulus with the addition of cellulosic fibers depends on many factors such as amount of fibers used, orientation of the fibers, interaction and adhesion between the fiber and matrix [27]. In general, dispersing agents and/or coupling agents are necessary for the property enhancement, when fibers are incorporated into thermoplastics. Dispersing agent facilitates the fiber dispersion and improves the interfacial adhesion between the fibers and the

polymer matrix. Grafting of chemical species on to the fiber surface has also been reported to improve the interaction between the fibers and matrix. Although grafting can improve the properties of the composite to a significant extent, this process increases the cost of the system. The use of dispersing agents and/or coupling agents is a cheaper route to improve properties and makes more practical sense for high volume, low cost composite systems [27].

In a natural fiber-thermoplastic composite, the lignocellulosic phase is present in a wide range of diameters and lengths, some in the form of short filaments and others in the form that seem closer to the individual fiber. The high shearing energy of blending the filaments and the polymer in a mixer results in fiber attrition but can also axially separate the filaments into discrete individual fibers. Cellulosic fillers/fibers can be classified under three categories depending on their performance when incorporated to a plastic matrix. Wood flour and other low cost agricultural based flour can be considered as particulate fillers that enhance the tensile and flexural moduli of the composite with little effect on the composite strength. Wood fibers and recycled newspaper fibers have higher aspect ratios and contribute to an increase in the moduli of composite and can also improve the strength of the composite when suitable additives are used to improve stress transfer between the matrix and the fibers. The improvement in modulus is not significantly different than the cellulosic particulate fillers. The specific Young's modulus, specific flexural modulus, the ratio of the composite modulus to the composite specific gravity of composites with natural fibers such as kenaf, Jute are significantly higher than those with wood fibers. The specific moduli (the ratio of the composite modulus to the composite specific gravity) of high fiber volume fraction of bast fibers-PP composites are high and in the range of glass fibers-PP composites. The most efficient natural fibers are those that have a high cellulose content coupled with a low microfibril angle resulting in high filament mechanical properties. A list of mechanical properties of PP composites reinforced with different natural fibers and inorganic substances is given in Table 2.5 [4, 28-29].

**Table 2.5: Comparison of filled PP composites [4, 28-29]**

Filled/Reinforcement of PP	ASTM Standard	None	Kenaf	Jute Fiber	Talc	Ca <sub>2</sub> CO <sub>3</sub>	Glass	Mica
% filler by weight		0	50	50	40	40	40	40
% filler by volume (estimated)		0	39	39	18	18	19	18
Tensile modulus, GPa	D 638	1.7	8.3	7.8	4	3.5	9	7.6
Specific tensile modulus, GPa		1.9	7.8	7.2	3.1	2.8	7.3	6.0
Tensile strength, MPa	D 638	33	68	72	35	25	110	39
Specific tensile strength, MPa		37	58	67	28	20	89	31
Elongation at break, %	D 638	10	2.2	2.3	—	—	2.5	2.3
Flexural strength, MPa	D 790	41	91	99	63	48	131	62
Specific Flexural strength, MPa		46	85	92	50	38	107	49
Flexural modulus, GPa	D 790	1.4	7.8	7.7	4.3	3.1	6.2	6.9
Specific flexural modulus, GPa		1.6	7.3	7.1	3.4	2.5	5.0	5.5
Notched Izod impact-J/m	D 256A	24	32	31	32	32	107	27
Specific gravity		0.9	1.07	1.08	1.27	1.25	1.23	1.26
Water absorption %- 24 hr	D 570	0.02	1.05	—	0.02	0.02	0.06	0.03
Mold (linear) shrinkage cm/cm		0.028	0.003	—	0.01	0.01	0.004	—

### 2.3.3 Effect of coupling agents on the composite properties

Coupling agents improve the adhesion between filler and polymer, preferably via chemical bonds; their use improves the performance of reinforcements, and allows the fiber content to be increased. The reinforcing effect of fibers depends on their

chemistry, shape (fibers, flakes, spheres), and size (fiber length, particle size). The most important reinforcement effects are [30]

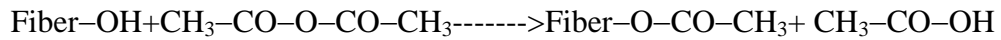
- Increased strength, ultimate elongation, rigidity (modulus of elasticity), and in some cases impact strength.
- Improved heat distortion temperature and dimensional stability, reduced shrinkage, and improved stability of mechanical values at high temperatures and over extended times (fatigue)
- Modification of density (usually increased)
- Improved chemical resistance and lower water absorption
- Better surface quality and surface hardness

A considerable amount of research has been done on the use of Maleic Anhydride (MA) as a coupling agent to improve the compatibility and adhesion between the fibers and matrix using a variety of procedures. It is now evident from the literature that the addition of 0.5-2 wt% maleic anhydride grafted polypropylene (MAPP) to PP matrices significantly improves the adhesion strength of jute fibers and in turn the mechanical properties of composites [30].

#### **2.3.4 Some chemical modifications for property enhancement of lignocellulosic fiber reinforced thermoplastic composites**

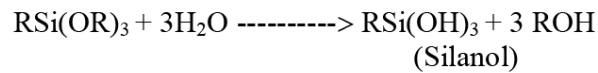
The properties of lignocellulosic materials result from the chemistry of the cell wall components. The basic properties of a fiber can be changed by modifying the basic chemistry of the cell wall polymers. Many chemical reaction systems have been reported for the modification of agro-based fiber. These chemicals include anhydrides (such as phthalic, succinic, maleic, propionic and butyric anhydride etc.), acid chlorides, ketene carboxylic acids, different types of isocyanates, formaldehyde, acetaldehyde, difunctional aldehydes, chloral, phthaldehydic acid, dimethyl sulfate, alkyl chlorides, beta-propiolactone, acrylonitrile, epoxides (such as, ethylene, propylene, butylene) and some difunctional epoxides [31]. By far, the most research has been done on the reaction of acetic anhydride with cell wall polymer hydroxyl groups to give an acetylated fiber. Many different types of lignocellulosic fibers including wood [32-33], bamboo, bagasse, jute, kenaf etc. have been acetylated using a variety of procedures. Although acetylation is not the only chemical modification

procedure that has been shown to improve properties of lignocellulosic materials even then it has been the most studied and are used in many cases [31]. Some researchers [33-36] have reported that acetylation of the lignocellulosic fibers have reduced the hydrophilicity and water absorption behavior of lignocellulosic fibers. The scheme of the reaction of lignocellulosic fibers with acetic anhydride is given below:

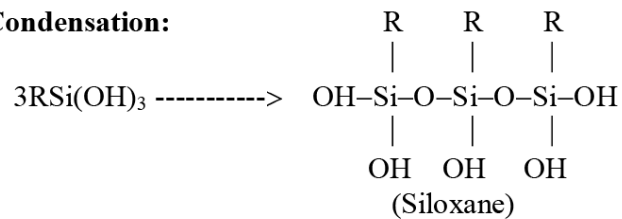


Modification of fiber surface by reacting with *silane* were investigated and reported by some researchers [37, 38]. Silane treatment of fibers led to increase mechanical properties of the composites. The reaction steps in the *silane grafting* of bio-fibers are shown below [38]:

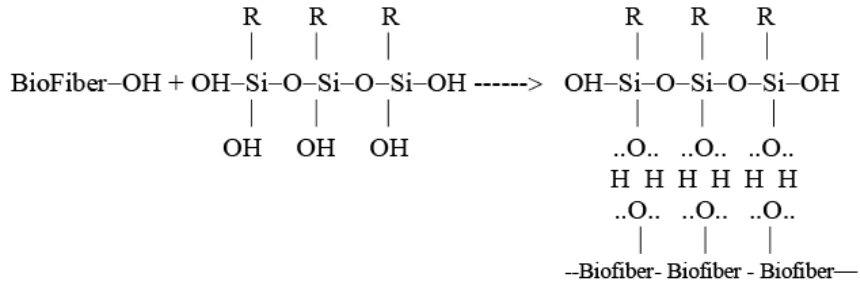
**Hydrolysis:**



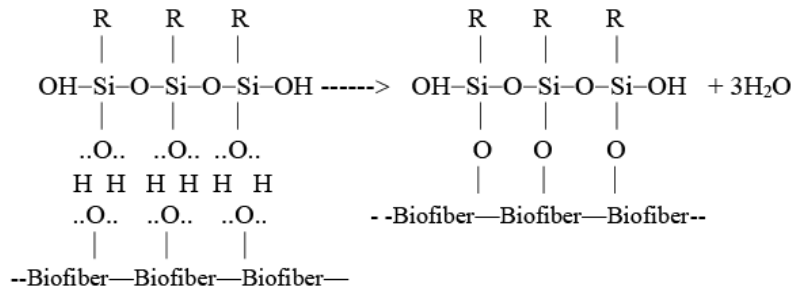
**Condensation:**



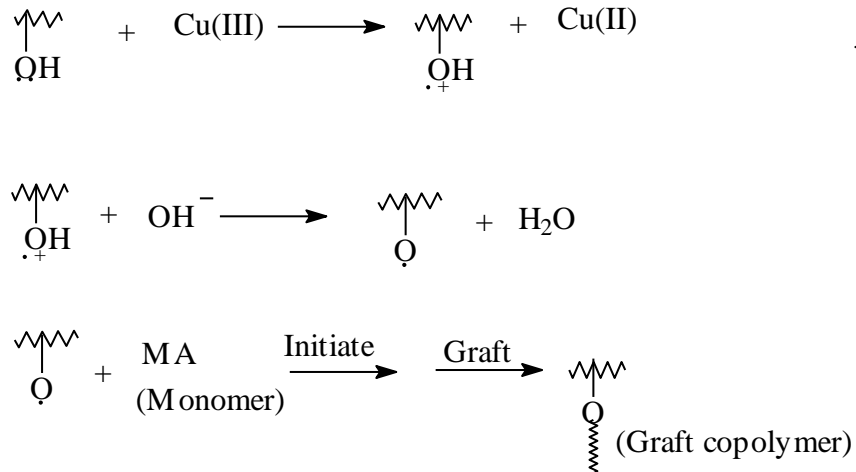
**Hydrogen Bonding:**



**Surface Grafting:**

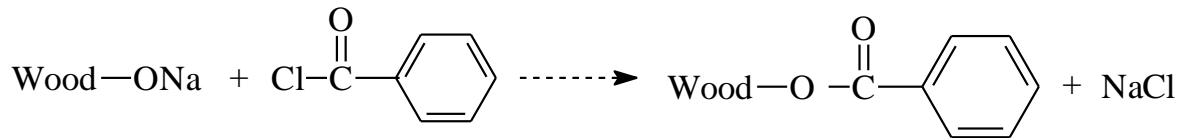
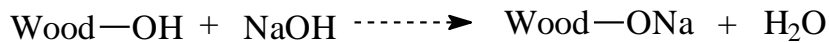


Grafting of polymethyl acrylate (PA) onto pure cellulose by potassium diperiodatocuprate (III) initiating system was carried out by L. Yinghai et al [39] and reported that the thermal stability of grafted product has been improved greatly. The initiation mechanism of grafting reaction is shown below [39]:



Poh et.al [40] carried out the esterification of palm oil wood fibers using benzoyl chloride and reported that benzoylation treatment has improved the thermal stability and changed the supra-structure of the wood fibers which may improve the interfacial

bonding of the lignocellulosic material and thermoplastic materials. The chemical reactions involved are shown below [40]:



Many other researches on chemical modifications have been reported to modify wood, kenaf, sisal, flax, banana, rice husk powder, etc. lignocellulosic fibers using various additives (i.e. alkali, silane, potassium permanganate, sodium chlorite, and acrylic acid treatments) by various procedures [41-54].

### 2.3.5 Composite manufacturing processes

There are two general divisions of composites manufacturing processes: **open molding** and **closed molding**. With open molding, the gel coat and laminate are exposed to the atmosphere during the fabrication process. In closed molding, the composite is processed in a two-sided mold set, or within a vacuum bag. There are a variety of processing methods under the heading of open and closed molding categories:

#### Open molding

- Hand Lay-Up
- Spray-up
- Filament Winding

#### Closed molding

- Compression molding
- Pultrusion
- Reinforced Reaction Injection Molding (RRIM)



- Resin Transfer Molding (RTM)
- Vacuum Bag Molding
- Vacuum Infusion Processing
- Centrifugal Casting
- Continuous Lamination

Among the above mentioned manufacturing methods, the followings are widely used in practice:

- Compression molding
- Extrusion molding
- Injection molding
- Hand molding

Selection of a manufacturing process depends on a number of factors including cost, materials, composite size, and volume of production.

### **2.3.6 Recent works on NF composites**

In the preceding sections, various aspects of natural fibers and polymer matrix have been outlined elaborately. These include sources of natural fibers, their types, structure, major ingredients of natural fibers, surface modification methods, coupling agents, and manufacturing methods of composites. In this article, some recent studies on natural fiber composites are discussed briefly with a view to understanding the current trend of the discipline.

Clemons et al. investigated the microstructure of injection-molded polypropylene reinforced with cellulose fiber and reported that there was little fiber orientation at low fiber contents and a layered structure arosed at high fiber contents [55]. They also reported that dynamic fracture toughness (Charpy impact test) increased with cellulose content and with orientation of fibers perpendicular to the crack direction.

Kokta reported the effect of compounding, molding and fiber length on the properties of wood fiber reinforced thermoplastic composites [56]. He reported that

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

Saad et al. reported on the empty fruit bunch (EFB) fiber reinforced polypropylene (PP) composites [57]. They modified the EFB fibers with maleic anhydride (MAH) before preparing the composites. They observed that MAH-treated fiber reinforced PP composites produce higher tensile and better dimensional stability properties.

Tay et al. investigated on the effect of chemical (epichlorohydrin) loading on the epichlorohydrin modified oil palm empty fruit bunch (EFB) fiber reinforced polyurethane composites (PU) [58]. They reported that composites made with EFB modified with EPH to 8% weight percent gain (WPG) produced higher tensile properties than the others. They expressed that higher degree of modification could cause disruption of crystalline region to the EFB.

Oksman and studied and reported on the mechanical properties and morphology of polypropylene (PP)-wood flour (WF) composites with different impact modifiers and maleated polypropylene (MAPP) as a compatibilizer [59]. They used three types of elastomers as impact modifiers and observed improved impact strength for PP-WF composites. Addition of maleated elastomers gave the greatest improvements in impact strength of the composites. MAPP didn't affect the impact properties of the composites but showed better tensile strength.

Ismail et al. reported the effect of PPMaH and LICA on properties of paper sludge filled PP/EPDM composites [60]. They observed the improved compatibility between sludge paper and PP/EPDM matrix by the addition of PPMaH as a compatibilizer and LICA as a coupling agent. Consequently, the tensile strength, modulus and water absorption resistance of sludge paper filled PP/EPDM composites with PPMaH and LICA showed better results than untreated composites.

Li et al. reported on biodegradable composites prepared by poly (propylene carbonate) (PPC) reinforced with short *Hildegardia populifolia* natural fibers [61]. They observed the effect of alkali treated fibers comparing with untreated fibers on

the composites and reported that better bonding and approximately similar mechanical properties were obtained for both alkali-treated and untreated *Hildegardia populifolia* fiber reinforced PPC composites. They also reported that tensile strength and tensile modulus of the composites increased dramatically with increasing fiber content but elongation at break decreased sharply with increasing fiber content.

Takemura reported on the effect of water absorption on static and creep properties for jute fiber reinforced composite (JFRC) [62]. He mentioned that the static strength and stiffness for JFRC decreases with increased water absorption. He also mentioned that the effect of water absorption is greater at the secondary creep stage than that of the other creep stages.

Sawpan et al. investigated the effect of various chemical treatments (by sodium hydroxide, acetic anhydride, maleic anhydride and silane) on the fiber structure and tensile properties of industrial hemp fibers [63] and reported that the average tensile strength of sodium hydroxide treated fibers slightly increased compared with that of untreated fibers, as a result of increased cellulose crystallinity. They also reported that the average Young's modulus of all treated fibers increased compared with untreated fibers, as a result of densification of fiber cell walls due to the removal of non-cellulosic components during treatment.

Renner et al. investigated the micromechanical deformations in PP/lignocellulosic filler composites and the effect of matrix characteristics on deformation and failure [64] and reported that the dominating micromechanical deformation process changes with matrix properties. They also described that debonding is the dominating process when the adhesion of the components is poor, while matrix yielding and/or filler fracture dominate when adhesion is improved by the introduction of a functionalized polymer.

Ahankari et al. described the mechanical behavior of agro-residue reinforced poly (3-hydroxybutyrate-co-3-hydroxyvalerate), (PHBV) green composites [65], fabricated by incorporating agro-residues as corn straw (CS), soy stalk (SS) and wheat straw (WS) into the bacterial polyester and compare those composites with traditional

polypropylene composites. They reported that the tensile and storage modulus of PHBV was improved by maximum 256% and 308% with the reinforcement of 30 wt% agricultural byproducts to it, and alkali treatment of wheat straw fibers enhanced strain at break and impact strength of PHBV composites by 35%, hardly increasing strength and modulus compared to their untreated counterparts.

Bouza et al investigated the Effect of particle size and a processing aid on the crystallization and melting behavior of isotactic polypropylene (iPP) with red pine wood flour [66], under dynamic and isothermal conditions in the presence of a processing aid with varying filler particle sizes. They have found only small variations in the thermal stability and reported that the wood particles have a heterogeneous nucleating effect on iPP crystallization that reduces the free energy of nucleation and increases the overall crystallization rate to a greater extent in composites with smaller particle size.

Renner et al. investigated the deformation and failure of PP composites reinforced with lignocellulosic fibers [67] with different particle size and aspect ratio in order to determine the effect of these factors on the deformation and failure mechanism as well as on the properties of the composites. They have found that micromechanical deformations change drastically both with decreasing particle size and changing interfacial adhesion and less debonding, fiber pull out and fiber fracture occur in composites containing small particles. They reported that improvement in composite strength is possible only through the optimization of particle size, aspect ratio and the inherent strength of wood.

Reddy et al. investigated the polypropylene hybrid composite consisting of wheat straw and clay as reinforcement materials [68]. They were prepared hybrid composite of constituents of composition as percentages of wheat straw, clay and maleic anhydride grafted polypropylene as a coupling agent, varied in order to investigate their influence on water absorption and flexural properties through melt blending method using a co-rotating twin-screw extruder. They reported that the increase in wheat straw and clay content in a composite increases the flexural modulus and reduces the resistance for water absorption, the increase in PP-MA coupling agent also increases the flexural modulus and resistance for water absorption, and the

addition of clay as additional filler had no significant role on water absorption and flexural properties of the composite.

Zou et al. investigated the lightweight composites from long wheat straw and polypropylene web, [69] and the effect of WS concentration, WS length, and split configuration (half, quarter, and mechanically split) on flexural and tensile properties of the composites. They reported that mechanically split WS–PP composites have 69% higher flexural strength, 39% higher modulus of elasticity, 18% higher impact resistance properties, 69% higher tensile strength and 26% higher Young's modulus by comparing with whole WS–PP composites.

Chen et al. investigated the morphology and properties of hybrid composites based on polypropylene/polylactic acid blend and bamboo fiber [70]. They found that the incorporation of MA-PP into composites brought about beneficial changes in morphology and rheological, related with improved dispersion of PLA and increased bamboo fiber–matrix interactions. They reported that the good rheological, morphological and thermal properties obtained when the ratio of PP/PLA/BF/MA-PP was 48.75/13/35/3.25.

Rezaur Rahman et al. studied and reported on the physico-mechanical properties of jute fiber reinforced polypropylene composites [71]. They found the post-treated jute fiber reinforced specimens yielded better mechanical properties compared to the oxidized and raw ones, and based on fiber loading, 30% fiber reinforced composites had the optimum set of mechanical properties. They proposed that the bonding between the polypropylene matrix and urea treated jute fiber must be increased in order to have improved mechanical properties at higher fiber content.

Sui et al. investigated the thermal and mechanical properties of the polypropylene (PP) composites reinforced using a novel plant fiber [72], sunflower hull sanding dust (SHSD) and compared to an organically modified clay (organo-clay)/PP composite. They reported that mechanical properties of the PP were enhanced with the addition of SHSDs, but compared to the neat PP and organo-clay/PP, the SHSD/PP composites exhibited a relatively decreasing rate of thermal degradation with increase in temperature.

Haque et al. investigated the physical and mechanical properties of raw and treated coir fiber reinforced polypropylene composites [73] of fiber loading (15, 20, 25, 30 and 35 wt %). They found that chemically treated coir fiber reinforced specimens yielded better mechanical properties compared to the raw ones. They reported that 30 wt% fiber reinforced composites had the optimum set of mechanical properties over the other fiber loadings.

Ave'rous et al. investigated the thermal and mechanical behaviour of processed biocomposites (biodegradable composites) by extrusion and injection molding [74]. They looked into the biocomposites thermal behaviour by TGA (thermal degradation) and DSC (transition temperatures, crystallinity). They reported that these materials present good mechanical behaviour due to high filler-matrix compatibility.

Alves et al. investigated the untreated and treated jute fiber composites as candidates to replace glass fibers as reinforcement to produce structural composites with better environmental performance [75]. They conducted environmental characterization by life-cycle assessment method. They reported that jute composites were compared with glass composites and results show that the jute fiber treatments imply a significant increase of the mechanical properties of the composites without damaging their environmental performances.

From the above literature review, it is observed that no research group has conducted any research on fabrication and mechanical characterization of JS-PP composite yet. That is why the present study focuses on the manufacturing and characterizing mechanical properties of JS-PP composites with special attention to the chemical treatments of JS fibers in order to improve the interfacial bonding between the JS flour and PP.

## EXPERIMENTAL

### 3.1 MATERIALS

#### 3.1.1 Polypropylene and Jute stick

In this study, injection molding grade (isotactic) polypropylene (PP) was used as a matrix material. It was manufactured by Linyi Aosen Chemical Co. Ltd., Shandong, China. It has a melt flow rate of 8 g/min and melting point of 165 °C. Melting point and FTIR spectroscopic observations indicated that the used PP was an isotactic [10] polypropylene and of commercial grade. In the literature, it is mentioned that the melting point of commercial grade PP lies in the range of 160-170 °C but Pure PP melts at 176 °C [10].

The jute sticks flour used as reinforcing materials were obtained from a village of Magura, Bangladesh. Jute sticks were cleaned and cut into small pieces. Then they were dried in sunlight. The dried jute sticks were then ground into flour and labeled as jute stick flour.

### 3.1.2 Chemicals

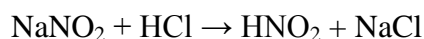
The list of chemicals used in this study and their suppliers name are given below:

Chemicals	Name of Suppliers
Hydrochloric acid (HCl)	Merck, Germany
Sodium nitrite (NaNO <sub>2</sub> )	Merck, Germany
Sodium hydroxide (NaOH)	Merck, India
Sodium bicarbonate (NaHCO <sub>3</sub> )	Merck, India
Aniline (C <sub>6</sub> H <sub>6</sub> NH <sub>2</sub> )	Merck, Germany
N,N-dimethyl aniline [C <sub>6</sub> H <sub>5</sub> N(CH <sub>3</sub> ) <sub>2</sub> ]	Merck, Germany
Benzene diazonium chloride (C <sub>6</sub> H <sub>5</sub> N <sub>2</sub> Cl)	Prepared in laboratory

#### 3.1.2.1 Preparation of benzene diazonium chloride

According to the standard method of diazotization [76], benzene diazonium chloride solution was prepared in two different ways: (i) using aniline and (ii) using N, N, Dimethyl Aniline( NNDMA).

In the first case, nitrous acid (HNO<sub>2</sub>) was prepared from the reaction of sodium nitrite (NaNO<sub>2</sub>) and hydrochloric acid (HCl) in which temperature was maintained in the range of 0- 5 °C. The reaction is shown below.



In the nitrous acid obtained from the above reaction, some HCl remains in dissolved state. Next, nitrous acid (with dissolved HCl) is used to prepare benzene diazonium chloride from the reaction of aniline (C<sub>6</sub>H<sub>5</sub>NH<sub>2</sub>) as shown below.





Temperature was maintained in the range of 0-5 °C in the above reaction too.

In the second case, benzene diazonium chloride solution was prepared following exactly the same procedure as outlined above except that NNDMA was used instead of aniline in the second reaction.

### **3.2 CHEMICAL TREATMENT OF JUTE STICK FLOUR**

In this study, four types of fiber treatment were performed. These are described below.

#### **3.2.1 Treatment by benzene diazonium chloride (prepared from aniline)/sodium hydroxide**

At first, 25 gm of sodium hydroxide (in powder form) was mixed with 100 ml of distilled water in a beaker to prepare sodium hydroxide solution at a temperature below 5°C. Then, JS flour was submerged into benzene diazonium chloride solution at about 5°C in an ice bath with continuous stirring for 10 minutes. Note that benzene diazonium chloride should be used immediately after its preparation in order to avoid oxidation. In order to neutralize acidic diazonium chloride solution, sodium hydroxide solution was poured slowly into the bath and kept 15 minutes below 5°C temperature. JS flour was finally taken out, washed with distilled water, and dried in open air.

#### **3.2.2 Treatment by benzene diazonium chloride (prepared from aniline)/sodium bicarbonate**

At first, 25 gm of sodium bicarbonate (in powder form) was mixed with 100 ml of distilled water in a beaker to prepare sodium bicarbonate solution at a temperature below 5°C. Then, JS flour was submerged into benzene diazonium chloride solution at about 5°C in an ice bath with continuous stirring for 10 minutes. Note that benzene diazonium chloride should be used immediately after its preparation in order to avoid oxidation. In order to neutralize acidic diazonium chloride solution, sodium

bicarbonate solution was poured slowly into the bath and kept 15 minutes below 5°C temperature. JS flour was finally taken out, washed with distilled water, and dried in open air.

### **3.2.3 Treatment by benzene diazonium chloride (prepared from NNDMA)/sodium hydroxide**

This treatment procedure is exactly same as article 3.2.1 except benzene diazonium chloride solution was prepared from NNDMA.

### **3.2.4 Treatment by benzene diazonium chloride (prepared from NNDMA)/sodium bicarbonate**

This treatment procedure is exactly same as article 3.2.2 except benzene diazonium chloride solution was prepared from NNDMA.

## **3.3 FABRICATION OF COMPOSITE**

JS-PP composites were prepared using raw and chemically treated JS flour by following the procedure described below.

The raw and chemically treated JS flour was dried in an oven at 100 °C for 3 hours. At first, JS flour was manually mixed thoroughly with PP granule at various weight proportions. Different weight proportions of JS flour and PP are shown in Table 3.1.

**Table 3.1: Relative amounts of JS Flour and PP matrix**

JS Flour (%)		PP (%)	Composites
Raw	0	100	100 wt % PP
	15	85	15 wt % JS-PP
	20	80	20 wt % JS-PP
	25	75	25 wt % JS-PP
	30	70	30 wt % JS-PP
	35	65	35 wt % JS-PP
Treated	15	85	15 wt % JS-PP
	20	80	20 wt % JS-PP
	25	75	25 wt % JS-PP
	30	70	30 wt % JS-PP
	35	65	35 wt % JS-PP

The JS flour and PP were then melt-mixed by passing them through a single screw extruder machine (Figure 3.1). The processing temperature of the extruder was controlled at 165°C. The melt-mixed JS-PP were delivered from the extruder in the form of small rod of small diameter as shown in Fig.3.2. Then, the rod shaped melt-mixed JS-PP were cut into small pieces of 15-20 cm length by a scissor.



Fig. 3.1: Extruder machine.



Fig. 3.2: Rod shaped melt-mixed JS-PP.

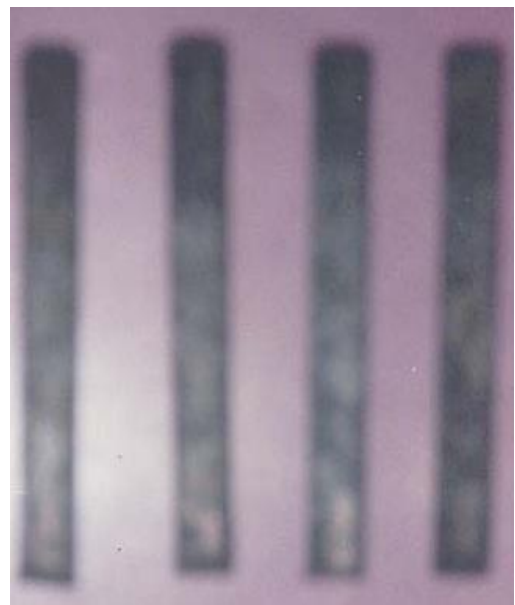
The small pieces of melt-mixed JS-PP were then fed into an injection molding machine (Fig.3.3) in order to prepare composite specimens directly as per ASTM standard. The injection molding temperature was maintained to be 165°C. The injection-molding machine was vertical and operated manually. The tensile test specimens and bending test specimens were fabricated by using corresponding die in the injection molding machine. The photographs of tensile and bending test specimens are shown in Fig. 3.4.



Fig. 3.3: Injection molding machine.



(a)



(b)

Fig. 3.4: Test specimens; (a) tensile, (b) bending.

### 3.3.1 Block diagram of fabrication of composite specimens

The whole process of JS-PP composite manufacturing method is outlined in the following block diagram (Fig. 3.5).

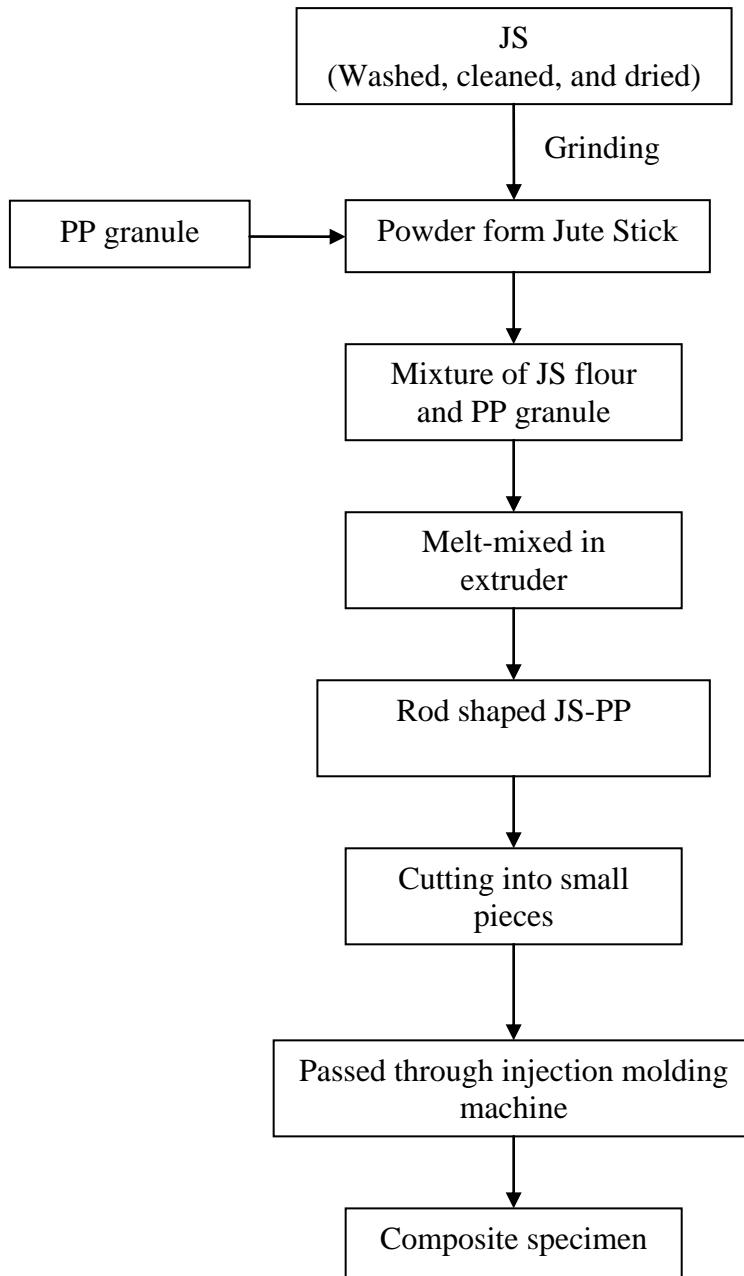


Fig. 3.5: Block diagram of JS-PP composite manufacturing process.

### 3.4 MECHANICAL PROPERTIES OF COMPOSITES

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

#### 3.4.1 Tensile test

The static tensile test of the composites were carried out in an universal tensile testing machine of model: Hounsfield UTM (H10KS), capacity: 10KN, Ogawa Seiki C. Ltd., Japan (Fig. 3.6) at a cross head speed of 5 mm/min. Tensile tests were conducted following ASTM D 638-01 standard [77] and each test was performed until the failure of the specimen.



Fig. 3.6: Universal testing machine UTM (H10KS).

Dumbbell-shaped injection molded composite specimens were used as test specimen without having any kind of machining. Dimensions of tensile test specimens are shown in Fig. 3.7. Three to five specimens of each composition were tested and the average values were reported. The load vs. elongation curves and stress vs. strain curves were obtained directly by using the QMAT software in a computer interfaced with the Hounsfield UTM machine. The tensile strength and Young's modulus were calculated automatically by the QMAT software while the tests were being performed on the composite specimens. Different dimensions of the specimens were:  $W = 10$  mm,  $G = 50$  mm,  $D = 105$  mm,  $L_o = 148$  mm,  $b = 20$  mm, and  $t = 4.1$  mm.

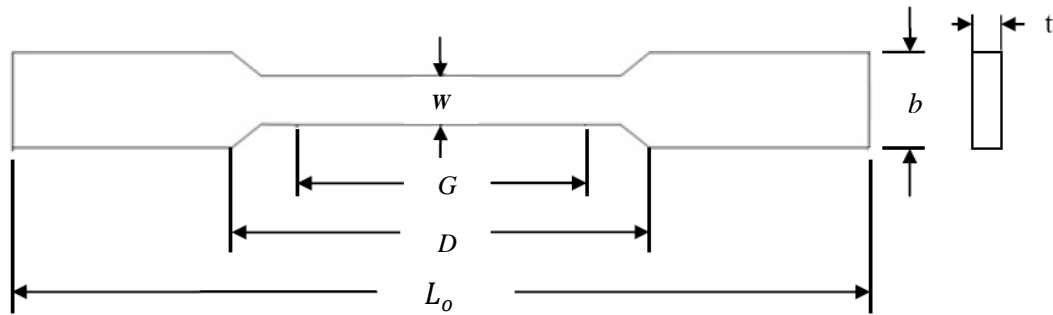


Fig. 3.7: Dimensions of tensile test specimen.

### 3.4.2 Three point flexural test

The static flexural tests of the composites were carried out by the same machine that was used for the tensile test only by changing the attachment. Set-up of the three point flexural test and dimension of the specimen are shown in Fig. 3.8. Different dimensions of flexural test specimen are:  $L=100$  mm,  $b =10$  mm, and  $d =4.1$ mm. Flexural tests were conducted following the ASTM D 790-00 standard [78] at a cross head speed determined by the following equation:

$$R = ZS^2/6d \quad (3.1)$$

where

$R$  = rate of cross head motion = 1.749 mm/ min

$d$  = depth of beam = 4.1 mm

$S$  = support span =  $16d = 16 \times 4.1$  mm = 65.6 mm

$Z$  = rate constant of straining of the outer fiber = 0.01 [78]



Injection molded test specimens (Fig. 3.8) were used directly to perform the flexural test and the other operating conditions were similar to that of the tensile test. Three specimens of each composition were tested and the average values were reported. The flexural strength ( $\sigma_f$ ) and flexural modulus ( $E_B$ ) were obtained by using QMAT software in a computer interfaced with the Hounsfield UTM machine. The following two equations were used in the QMAT software to calculate the flexural strength  $\sigma_f$  and flexural modulus  $E_B$ , respectively [78].

$$\text{Flexural strength, } \sigma_f = 3PS/2bd^2 \quad (3.2)$$

$$\text{Flexural modulus, } E_b = S^3m/4bd^3 \quad (3.3)$$

Where

$P$  = maximum load, N

$m$  = slope of the tangent to the initial straight portion of the load-deflection curve, N/mm.

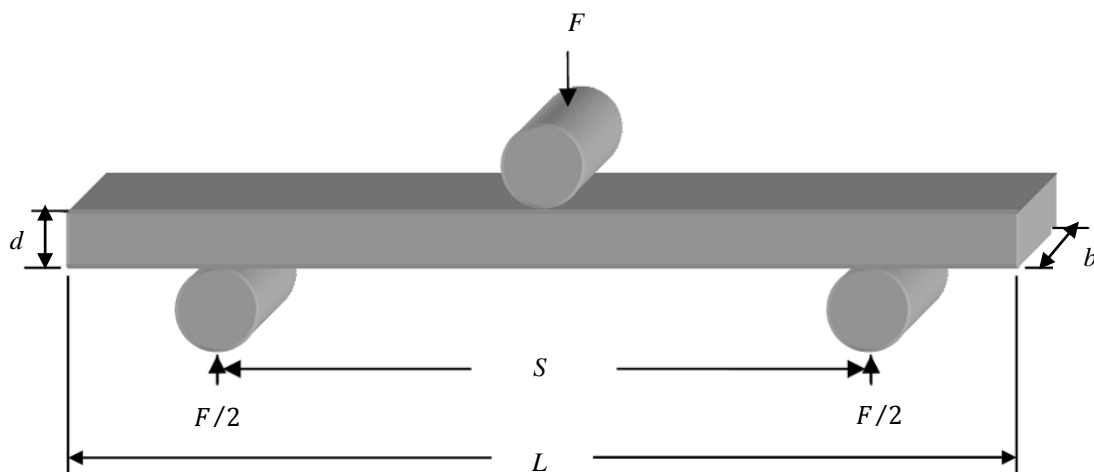


Fig. 3.8: Set-up of three point flexural test and dimension of specimen.

### 3.4.3 Charpy impact test

The dynamic Charpy impact tests of the composites were done according to the ASTM D 6110-97 standard [79]. Figure 3.9 shows the photograph of Universal Impact Testing Machine (Type: TIT-30, Tokyo Testing Machine MFG. Co. Ltd.,

Japan). This machine was used to perform Charpy impact tests of all the samples. Weight of the hammer of this machine was 20.54 kg.



Fig. 3.9: Pendulum type impact test machine.

The Charpy impact test specimens were injection-molded without having any kind of machining on them. Notched specimens were used. The design of the notch cut is shown in Fig. 3.10. Notching of the test specimens was done on a milling machine. In this experiment, the width ( $b$ ) and depth ( $d$ ) were 10 mm and 4.1 mm, respectively. Notch cut was made on the shorter side of 4.1 mm.

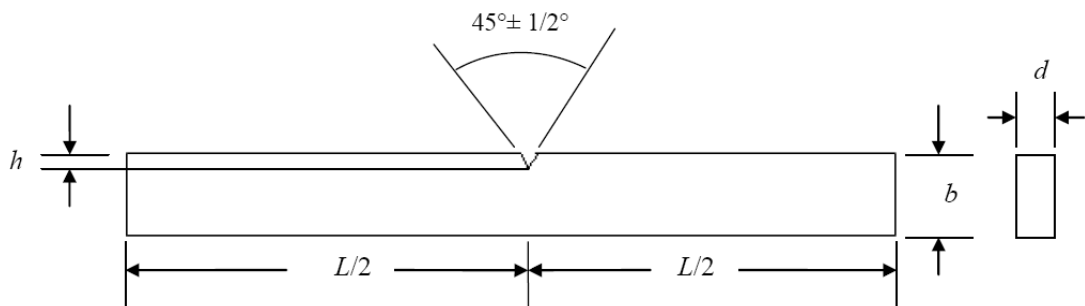


Fig. 3.10: Dimensions of simple beam, Charpy type, impact test specimen.

The impact tests were performed after 32 hours of notching. The width ( $b$ ), depth ( $d$ ), length ( $L$ ), and notch depth ( $h$ ) of the specimen were 10mm, 4.1 mm, 79 mm, and 2 mm, respectively. According to the test method, the specimen was set as a simple supported beam with the notch directly opposite to the impact point. The specimen was broken by a single strike of the pendulum with the impact point midway between the supports and directly opposite to the notch. The results were calculated in terms of energy absorbed per unit of specimen width from the following equations:

$$\text{The notch impact strength, } I = K/A \quad (3.4)$$

where

$A$ = Area of the cross-section of specimen below the notch = 32.8 mm<sup>2</sup>

$K$ = Corrected impact energy in breaking the specimen =  $[(e_i - e_f) - e_1(\theta_i + \theta_f)]$

$e_i$ = Initial energy of pendulum =  $wr(1 - \cos \theta_i)$

$\theta_i$ = Angle of fall of pendulum = 140°

$e_f$ = Energy after breaking the specimen =  $wr(1 - \cos \theta_f)$

$\theta_f$ = Angle of rise during sample breaking = 138.55°

$e_1$ = Lost energy per degree of swing =  $wr(\cos \theta_0 - \cos \theta_i)/(\theta_i + \theta_o)$

$\theta_o$ = Free angle of rise = 139.6°

#### 3.4.4 Hardness test

Rockwell hardness test of the composites were carried out following ASTM D785-93 [80]. A Rockwell hardness number is a number derived from the net increase in depth impression as the load on an indenter increased from a fixed minor load to a major load and then returned to a minor load. Procure A was followed using R scale with a 12.7 mm diameter indenter and 590 N major load. Five measurements were taken for each type of specimen and the results were averaged.

### 3.5 ANALYSIS OF CHEMICAL STRUCTURE OF JS FLOUR

JS flour was characterized by FTIR spectroscopy as stated below:

Fourier transform infrared spectroscopy (FTIR) is a technique which is used to obtain an infrared spectrum of absorption, emission, or photoconductivity of a solid, liquid or gas. An FTIR spectrometer simultaneously collects spectral data in a wide spectral range.

A schematic absorption spectrum is given in Fig. 3.11. The absorption spectrum is a plot of absorption of energy (radiation) against its wavelength ( $\lambda$ ).

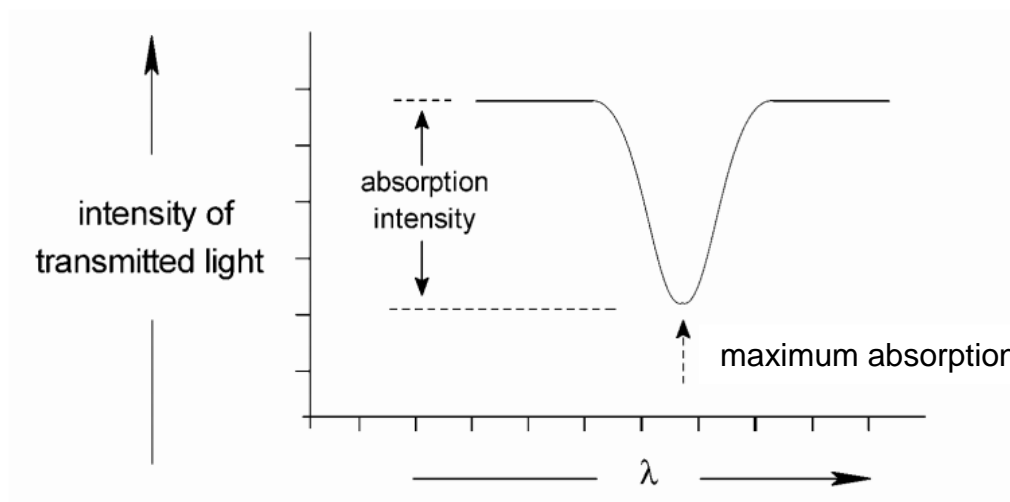


Fig. 3.11: Schematic diagram of absorption spectrum.

An absorption band can be characterized primarily by the wavelength at which maximum absorption occurs. Infrared absorption spectra are calibrated in wavelengths expressed in micrometers ( $\mu\text{m}$ ) or, in frequency-related wave numbers ( $\text{cm}^{-1}$ ) which are reciprocals of wavelengths:

$$\text{wave number, } \bar{\nu} (\text{cm}^{-1}) = \frac{1 \times 10^4}{\text{wavelength (in } \mu\text{m)}} \quad (3.5)$$

Infrared absorption intensities are rarely described quantitatively, except for the general classifications of *s* (strong), *m* (medium) or *w* (weak). The transitions responsible for IR bands are due to molecular vibrations, i.e. to periodic motions

involving stretching or bending of bonds. Polar bonds are associated with strong IR absorption while symmetrical bonds may not absorb at all. Clearly the vibrational frequency, i.e. the position of the IR bands in the spectrum, depends on the nature of the bond.

In this study, the infrared spectrum of the raw JS flour, and chemically treated JS flour were recorded by a Shimadzu FTIR-8101 spectrophotometer. The samples pellets for FTIR spectroscopy were prepared as follows:

Approximately 0.5 mg of powdered samples (Jute stick) was mixed thoroughly with approximately 100 mg of dried powdered potassium bromide in a small agate mortar pestle. The mixture was taken in a die of specific dimensions. Pellets were made by applying pressure on the mixture with the help of a plunger. FTIR tests were carried out on thin specimen and results are presented in the results and discussion section.

### **3.6 SCANNING ELECTRON MICROSCOPY (SEM)**

SEM was employed to study the tensile and flexural fracture surfaces of composite samples of 35 wt% fiber content. They were examined using a JSM-5510 SEM analyzer from JEOL Co. Ltd., Japan. This test was carried out to determine the dispersion of fibers in the matrix, adhesion between fiber and matrix, and to detect the presence of any micro defect in the composite.

The SEM analysis was carried out at Bangladesh Atomic Energy Commission, Dhaka. The photographs are presented in the results and discussion section.

### 3.7 WATER ABSORPTION TEST OF COMPOSITES

In order to evaluate the water absorption property of the composites, flexural test specimens were cut from each category of samples having dimensions of 39 mm × 10 mm × 4.1 mm. The samples were dried in an oven at 105°C for 2 hrs, cooled in a desiccator using silica gel and immediately weighed to the accuracy of ±0.0001 gm using Denver Instron balance. The dried and weighed samples were immersed in distilled water for about 24 hrs at room temperature as described in ASTM D570-99 standard [81]. The samples were taken out from water and excess water on the surface of the samples was removed by using a soft cloth. Then the weight of the samples was taken again. Three replicate specimens were tested and the average results of the three tests were presented. The percentage increase in weight during immersion was calculated as follows [81]:

$$\text{Increase in weight} = \frac{(W_f - W_i)}{(W_i)} \times 100 \% \quad (3.6)$$

where

$W_f$  = weight after 24 hrs immersion in distilled water

$W_i$  = initial weight before immersion.

### RESULTS AND DISCUSSION

In this study, physico-mechanical properties of jute stick flour reinforced polypropylene composites have been studied. Following different types of composites have been fabricated, (i) raw JS-PP composites, (ii) benzene diazonium-chloride (by using aniline)/ sodium hydroxide treated JS-PP composites, (iii) benzene diazonium-chloride (by using aniline)/ sodium bicarbonate treated JS-PP composites, (iv) benzene diazonium-chloride (by using NNDMA)/ sodium hydroxide treated JS-PP composites, and (v) benzene diazonium-chloride (by using NNDMA)/ sodium bicarbonate treated JS-PP composites. Before preparing the composites, JS sticks were chemically treated with an aim to have better coupling with polypropylene (PP). The structural changes of JS due to chemical treatments were confirmed by FTIR-spectra. Raw and treated JS flour were used separately to prepare mixture of JS and PP in the form of rods of small diameter by using a single screw extruder. To study the physico-mechanical properties, sample specimens of specific size and shape had been prepared from the above mixture by using an injection molding machine. Tensile strength, tensile modulus, flexural strength, flexural modulus, charpy impact strength, and hardness of the prepared composites were measured by using universal testing machines. The results are presented in the following sections.

## 4.1 FTIR SPECTROSCOPIC ANALYSIS OF JUTE STICK FLOUR

### 4.1.1 Raw JS flour

The hydroxyl group in the raw JS is responsible for high water absorption and weak interfacial bond between the JS and PP matrix. There are actually three hydroxyl groups present in a cellulose unit (Fig. 4.1) of JS flour. One is a primary hydroxyl group at C<sub>6</sub> and the other two are secondary hydroxyl groups at C<sub>2</sub> and C<sub>3</sub>. The FTIR spectroscopic analysis of the raw JS (Fig. 4.2) confirms this phenomenon.

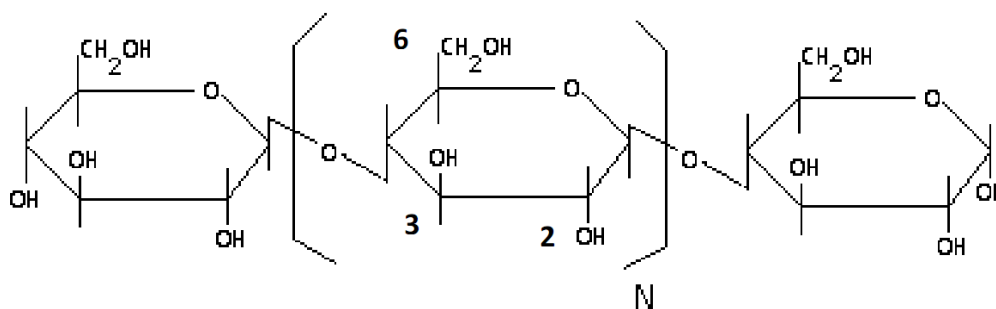


Fig. 4.1: Cellulose structure of JS

The peak assignments of the absorption bands corresponding to various groups are summarized in Table 4.1, which are quite similar to those available in literatures [61, 82-84].

**Table 4.1: FTIR spectral data of raw (untreated) JS flour**

Position (cm <sup>-1</sup> )	Assignment
~ 3639.4-3421.5	Strong free -OH band from the cellulose, hemicellulose and lignin of jute stick
~ 2912.3	C-H stretching band associated with methyl and methylene groups
~1730-1620.1	C=C aromatic in-plane ring stretching from the cellulose and lignin
~ 1504.4-1450.4	In-plane -OH bend of cellulose
~ 1163-1051.1	C-O-R stretching band of ether group
~ 896.8	C -O-C bond in plane, symmetric



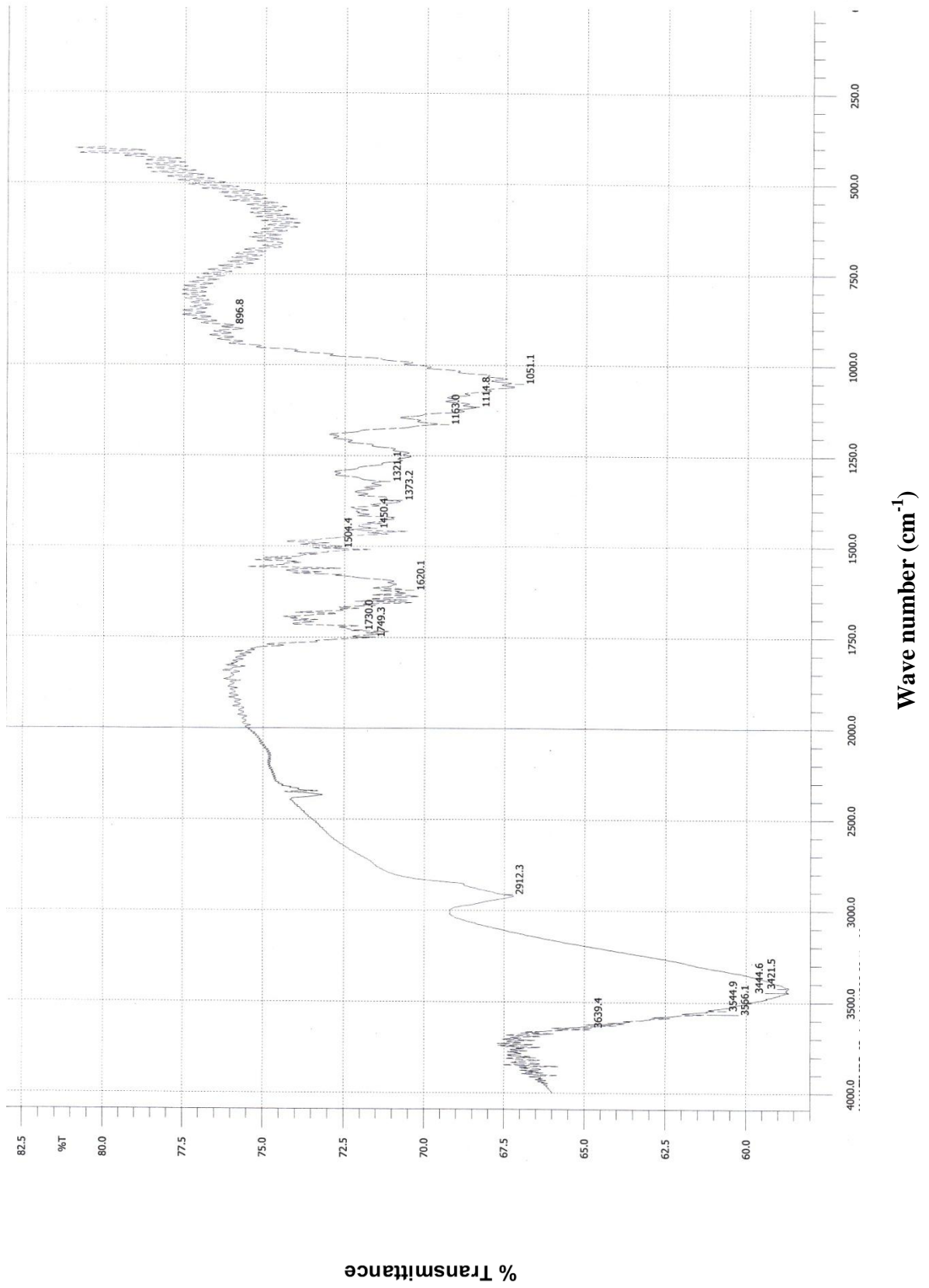
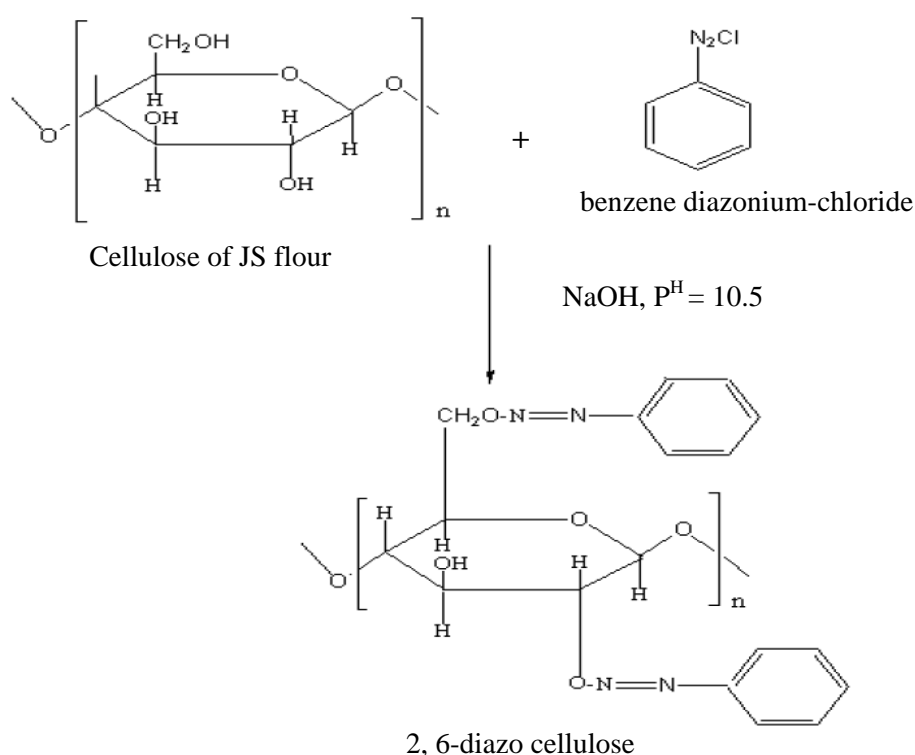


Fig. 4.2: FTIR spectrum of raw JS flour.

#### 4.1.2 Benzene diazonium-chloride (by using aniline)/ sodium hydroxide treated JS flour

In order to improve mechanical properties of the composites, jute stick flour was chemically treated using benzene diazonium salt (by using aniline). The hydroxyl group (-OH) in the raw JS is responsible for high water absorption and weak interfacial bond between the JS and PP matrix. The diazonium salt breaks the OH group of cellulose of JS flour during the reaction. This converts the two hydroxyl groups into diazo groups and results in an azo product, 2, 6-diazo cellulose, as illustrated below [85].



The FTIR spectrum (Fig. 4.3) of benzene diazonium-chloride (by using aniline) and sodium hydroxide treated JS flour confirms this phenomenon. The IR spectrum clearly shows the presence of the characteristic band of the free -OH group in the region of  $3419.6 - 3396.4 \text{ cm}^{-1}$  but the band (-OH) intensity has become weaker than raw JS flour due to chemical treatment. The IR spectrum also shows the presence of the characteristics band of N=N stretching near  $1375.2 \text{ cm}^{-1}$ , an absorption band of NO group near  $1633.6 \text{ cm}^{-1}$ , and a C-O stretching band at the region of  $1240.1 - 1060.8 \text{ cm}^{-1}$ .

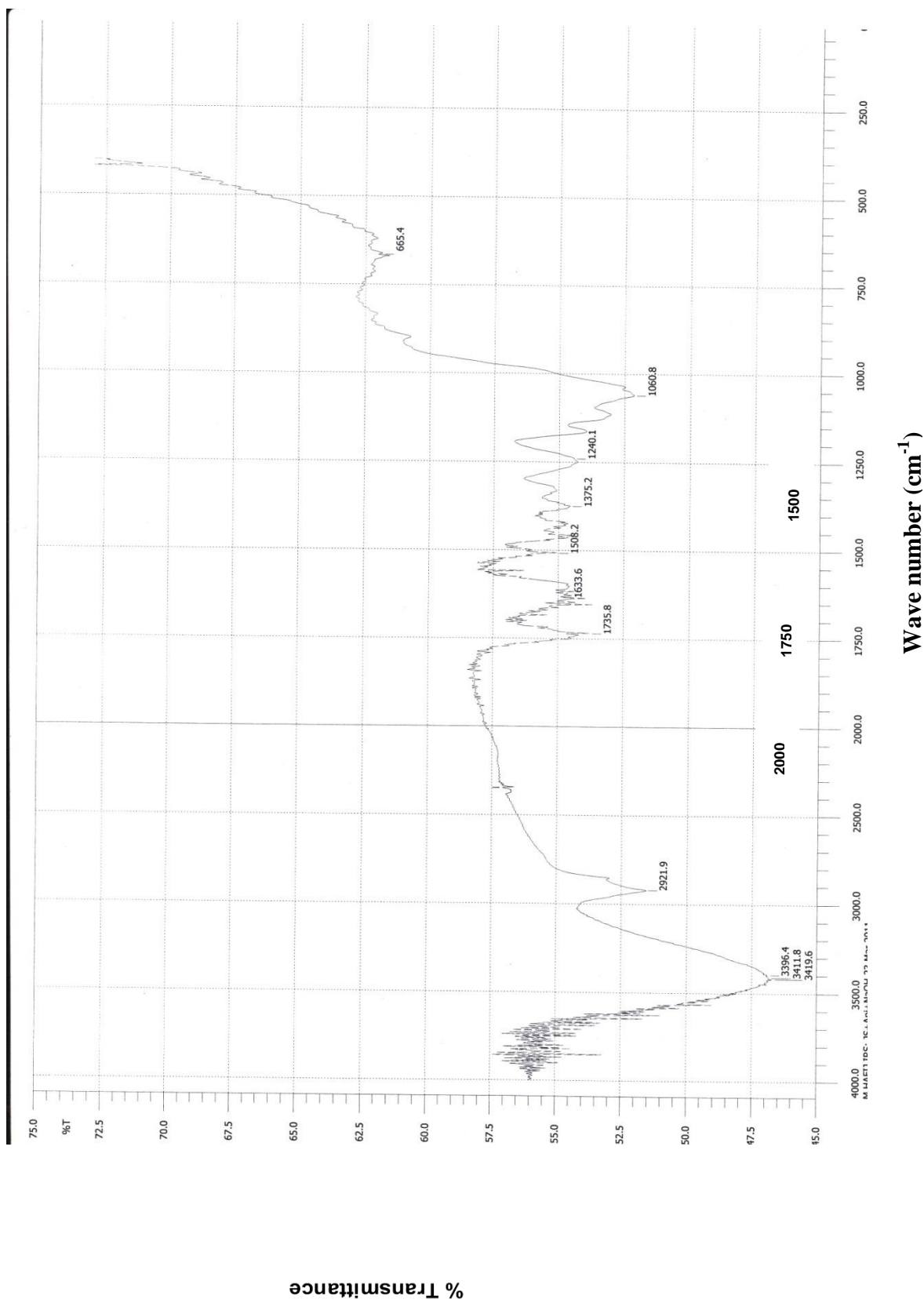


Fig. 4.3: FTIR spectrum of benzene diazonium -chloride (by using aniline) /sodium hydroxide treated JS flour.

#### **4.1.3 Benzene diazonium-chloride (by using aniline)/ sodium bicarbonate treated JS flour**

The FTIR spectrum of benzene diazonium-chloride (by using aniline)/sodium bicarbonate treated JS flour is shown in Fig. 4.4. The IR spectrum of treated JS flour clearly shows the strong characteristic bands of diazonium salt (N=N) at the region of  $1417.6\text{ cm}^{-1}$  due to the formation of bonding with cellulose of JS flour, and consequently weakens the characteristics band of in-plane -OH group of cellulose. The IR spectrum also shows the medium characteristics band with aldehyde group at the region of  $2912.3\text{ cm}^{-1}$  due to C-H stretching. The IR spectrum also shows the presence of the characteristics absorption band of NO group near  $1633.6\text{ cm}^{-1}$ , and a C-O stretching band at the region of  $1051.1\text{--}1238.2\text{ cm}^{-1}$ .

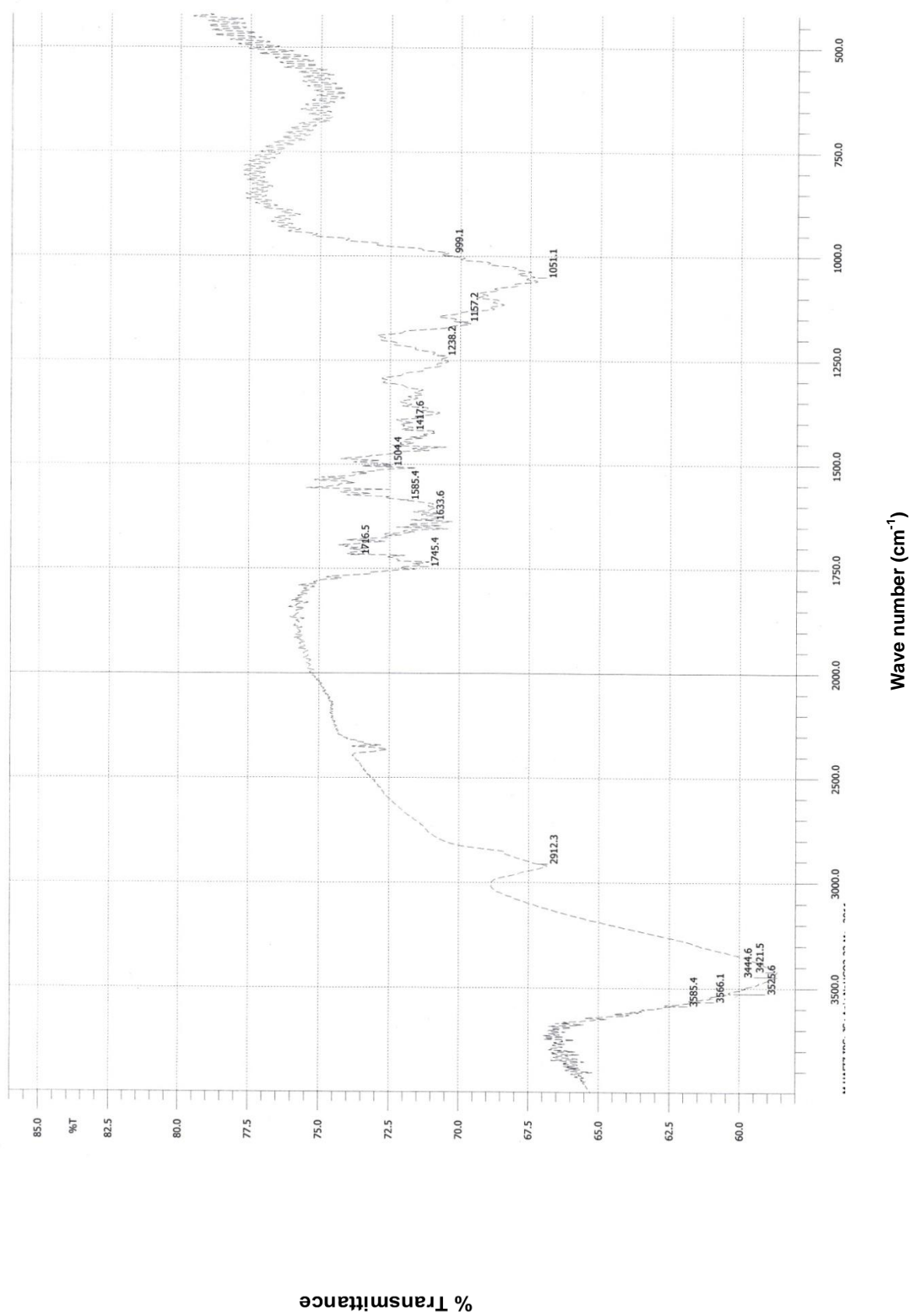


Fig. 4.4: FTIR spectrum of benzene diazonium -chloride (by using aniline) /sodium bicarbonate treated JS flour.

#### **4.1.4 Benzene diazonium-chloride (by using NNDMA)/ sodium hydroxide treated JS flour**

The FTIR spectrum of benzene diazonium-chloride (by using NNDMA)/ sodium hydroxide treated JS flour is shown in Fig. 4.5. The IR spectrum clearly shows the presence of characteristic band of the free –OH group in the region of 3651 – 3423.4  $\text{cm}^{-1}$ . The IR spectrum also shows the characteristics band with methyl group near 2921.9  $\text{cm}^{-1}$  due to C-H stretching and near 1652.9 to 1735.8  $\text{cm}^{-1}$  due to carbonyl stretching. Treated JS flour also shows the presence of the characteristics band of N=N stretching at the region of 1373.2–1330.8  $\text{cm}^{-1}$ , an absorption band of NO group near 1591.2  $\text{cm}^{-1}$ , and a C–O stretching band at the region of 1247.8–1055  $\text{cm}^{-1}$ .

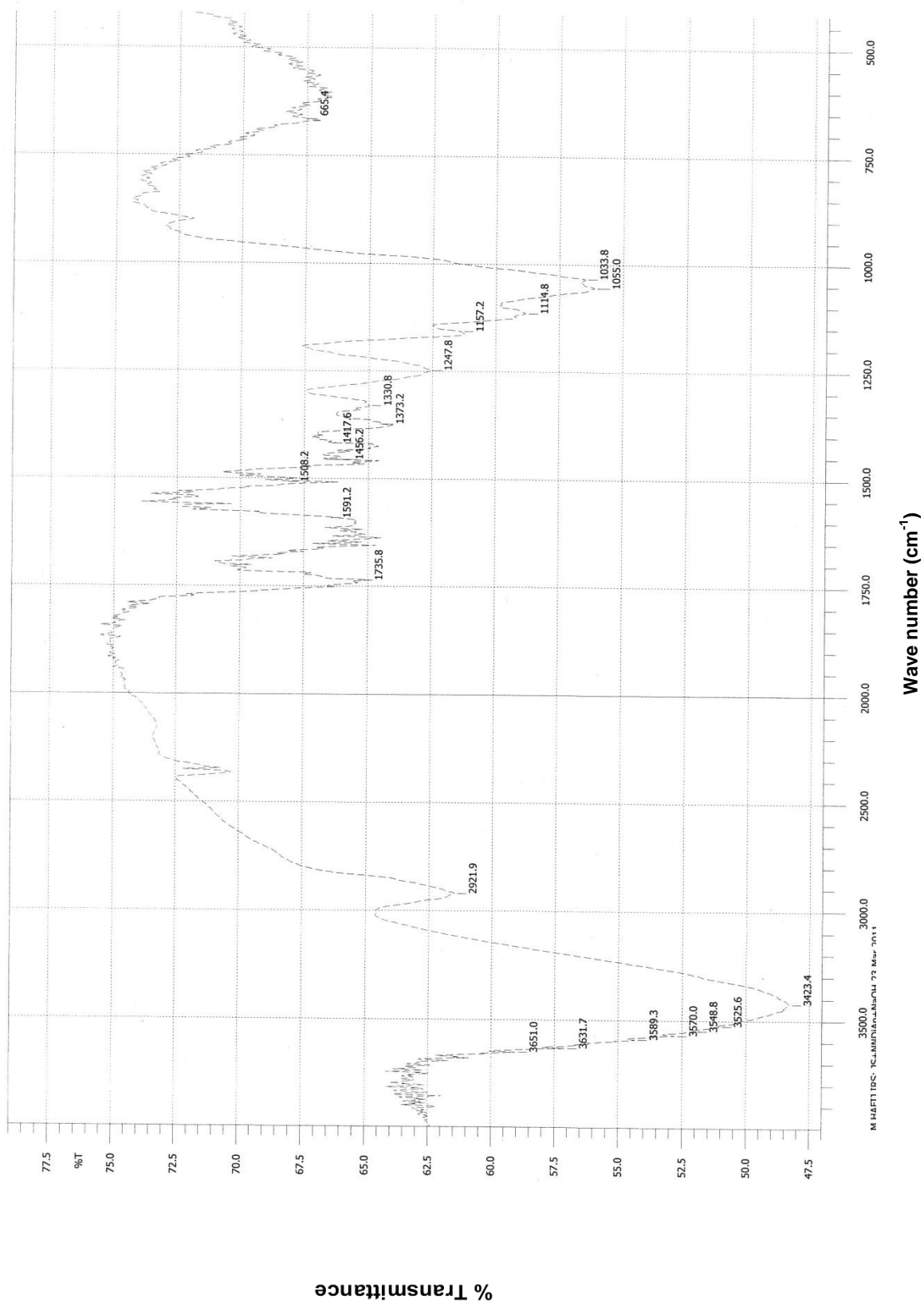


Fig. 4.5: FTIR spectrum of benzene diazonium -chloride (by using NNDMA) /sodium hydroxide treated JS flour.

#### **4.1.5 Benzene diazonium-chloride (by using NNDMA)/ sodium bicarbonate treated JS flour**

The FTIR spectrum of benzene diazonium-chloride (by using NNDMA)/ sodium bicarbonate treated JS flour is shown in Fig. 4.6. The IR spectrum of treated JS flour clearly shows that the free -OH groups of cellulose in the region of 3564.2 – 3444.6  $\text{cm}^{-1}$  have become weaker due to the chemical treatment. Treated JS show the absorption near 1639.4  $\text{cm}^{-1}$  due to the carbonyl aldehyde of dialdehyde cellulose. The IR spectrum also shows that the characteristic band of in-plane -OH group of cellulose has become weaker at the region of 1456.2  $\text{cm}^{-1}$  due to N=N stretching of diazonium salt near 1247.8  $\text{cm}^{-1}$ . The IR spectrum clearly shows the characteristic band with methyl group at the region of 2921.9  $\text{cm}^{-1}$  due to C-H stretching and near the region of 1739.7  $\text{cm}^{-1}$  due to carbonyl stretching.



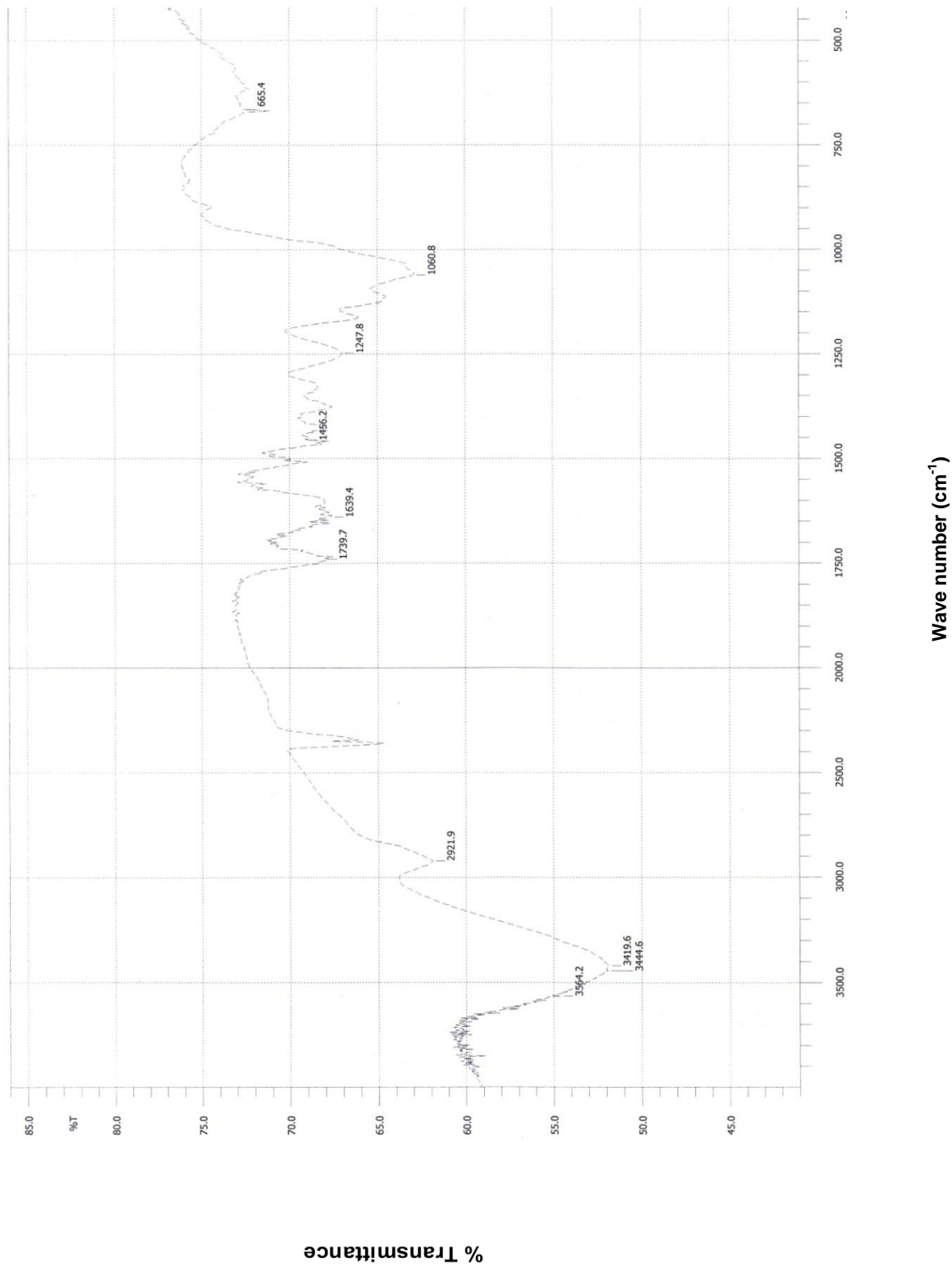


Fig. 4.6: FTIR spectrum of benzene diazonium -chloride (by using NNDMA) /sodium bicarbonate treated JS flour.

## **4.2. ANALYSIS OF PHYSICO-MECHANICAL PROPERTIES OF JS-PP COMPOSITES**

As mentioned earlier JS reinforced PP composites were prepared by using both the raw and chemically treated JS flour. Tensile strength, tensile modulus, flexural strength, flexural modulus, charpy impact strength, hardness, and water absorption of the raw and treated JS flour reinforced PP composites have been determined by following the ASTM standards as described in the experimental sections. The results obtained in this study are presented below.

### **4.2.1 Tensile properties**

The importance of natural fiber reinforced composites of polymeric materials comes from the substantial improvement of strength and modulus that offers a possibility of composites in practical applications. The tensile strengths of the raw and chemically treated JS-PP composites at different fiber loadings are shown in Fig. 4.7.

In general, for both raw and treated JS flour, the tensile strength increases with the fiber loading until it reaches to a maximum value and then decreases. The same trend was observed for other NF-PP composites [86, 87] in the past. This is because the incorporation of JS flour leads to uniform and effective stress transfer across the composite. But after a particular fiber loading (30%), tensile strength of the composites shows dropping nature. JS flour was treated in four different ways. It is observed from the Fig. 4.7, for all the cases, that the composites have higher tensile strength than raw JS-PP composite. The treatment of JS flour with benzene diazonium-chloride (by using aniline)/ sodium hydroxide is advisable when tensile strength is the main concern.

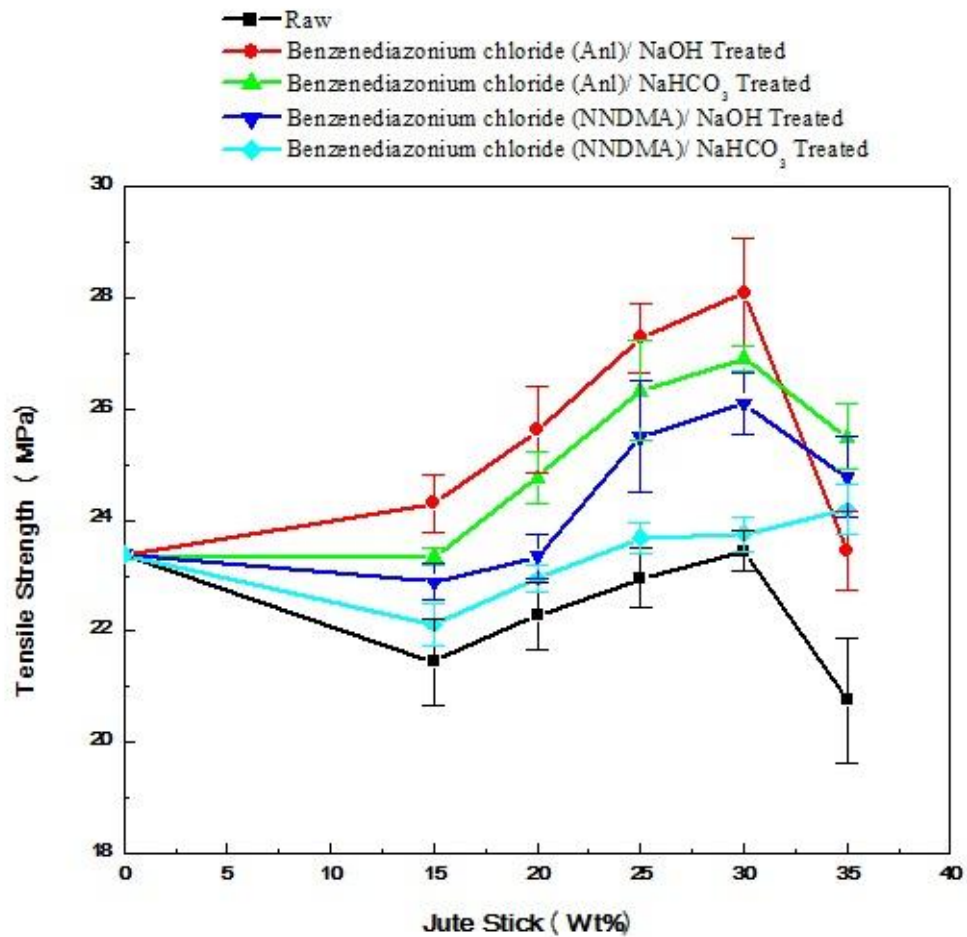


Fig. 4.7: Tensile strength as a function of JS flour loading.

However, tensile modulus, which is an indication of load bearing capacity, also increases with JS weight fraction as seen from the Fig. 4.8. Tensile modulus is primarily dependant on JS weight fraction and not on physical structure of fibers [88]. For all the cases, the modulus shows increasing trend until 35%, up to which the experiments were carried out.

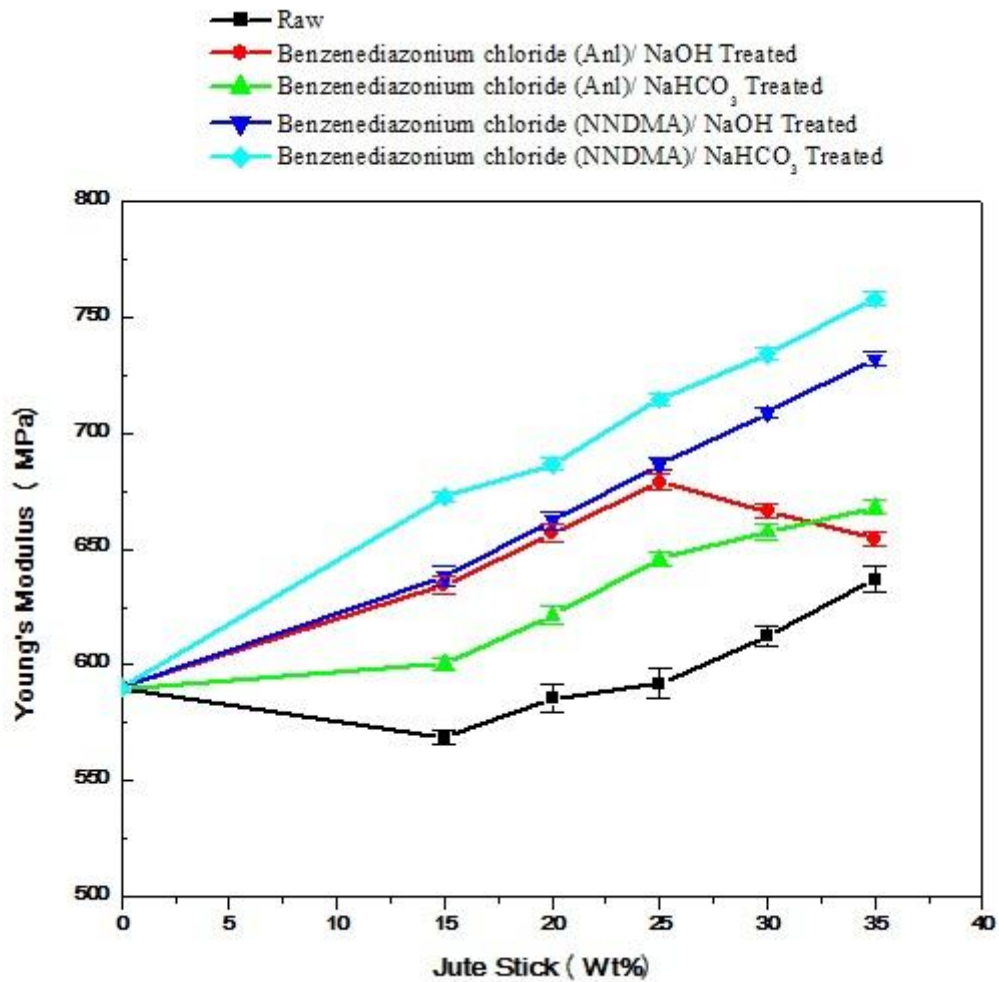


Fig. 4.8: Variation of the Young's modulus as a function of JS flour loading.

#### 4.2.2 Flexural properties

The flexural properties of treated and raw JS flour reinforced PP composites with respect to different fiber loadings are illustrated in Fig. 4.9 and 4.10, respectively. It is essential to find out the optimum fiber loading to achieve maximum property. For raw JS-PP composites, the flexural strengths showed increasing tendency up to 25 wt% of fiber loading and then strengths decreased with increasing fiber loading. So it may be said that 25 wt% is the optimum fiber loading for raw JS-PP composites, in terms of flexural strength.

Due to the chemical treatment of JS flour, fiber-matrix adhesion was improved. In fact the chemical treatment, converted  $-OH$  group of JS flour into  $O=N=N-$  group. This converted group contains double ( $-N=N-$ ) bond. As a result, hydrophilic nature of JS flour has significantly reduced, imparting favorable interfacial adhesion between the fiber and the matrix. Thus, the flexural strengths were improved for treated JS-PP composites corresponding to treatment with benzene diazonium-chloride. The highest value of flexural strength was observed for 30 wt% benzene diazonium-chloride (by using Aniline)/ sodium bicarbonate treated JS-PP composite, which was 46.11 MPa and much higher than that of the untreated JS-PP composites.

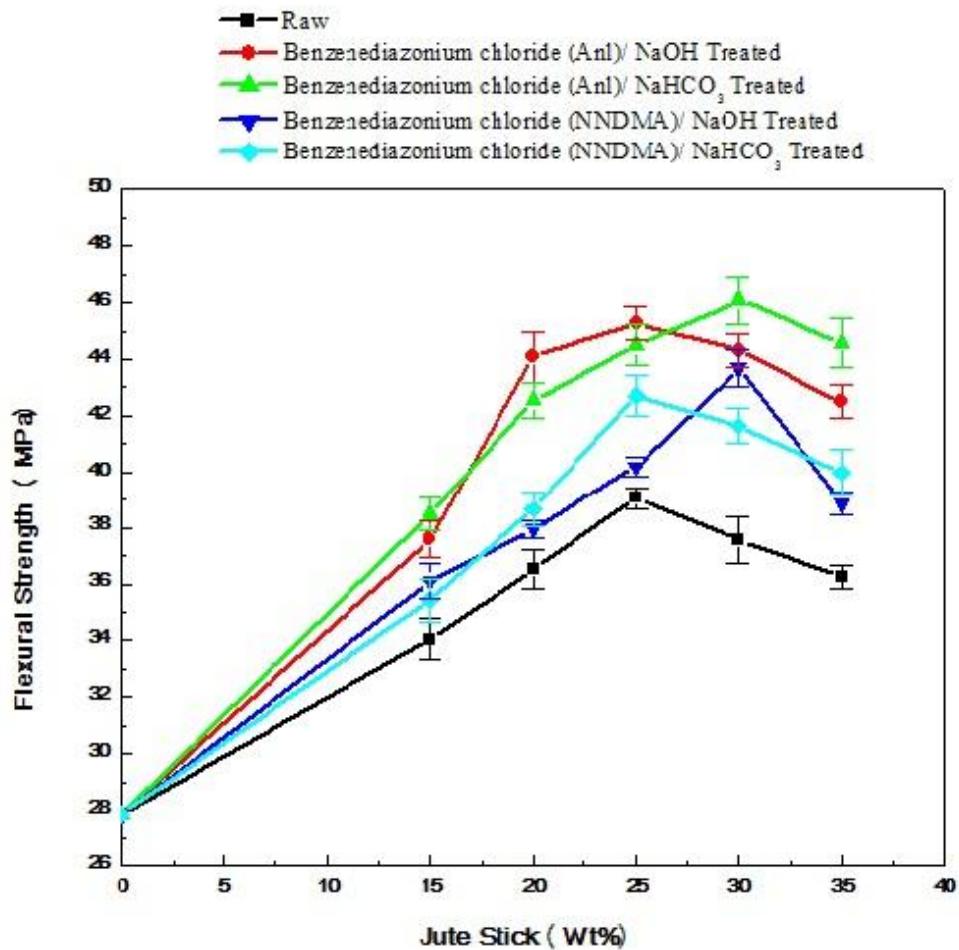


Fig. 4.9: Flexural strength of JS-PP composites as a function of fiber loading.

From Fig. 4.10, it is observed that the flexural modulus increases with fiber loading in accordance with other studies in literatures [34-38, 57-61]. In general, the flexural modulus of the treated JS-PP composites exhibits better characteristics in comparison

with that of raw JS-PP composites over the entire range of fiber loading. The flexural modulus of benzene diazonium-chloride (by using NNDMA)/ sodium hydroxide treated JS-PP composites is found to be maximum (2.31 GPa) for 35 wt% fiber loading compared to that of the untreated one (1.94 GPa) for 15 wt% fiber loading. This corresponds to 19.07% increase in the flexural modulus, which is attributed to the good interfacial bonding.

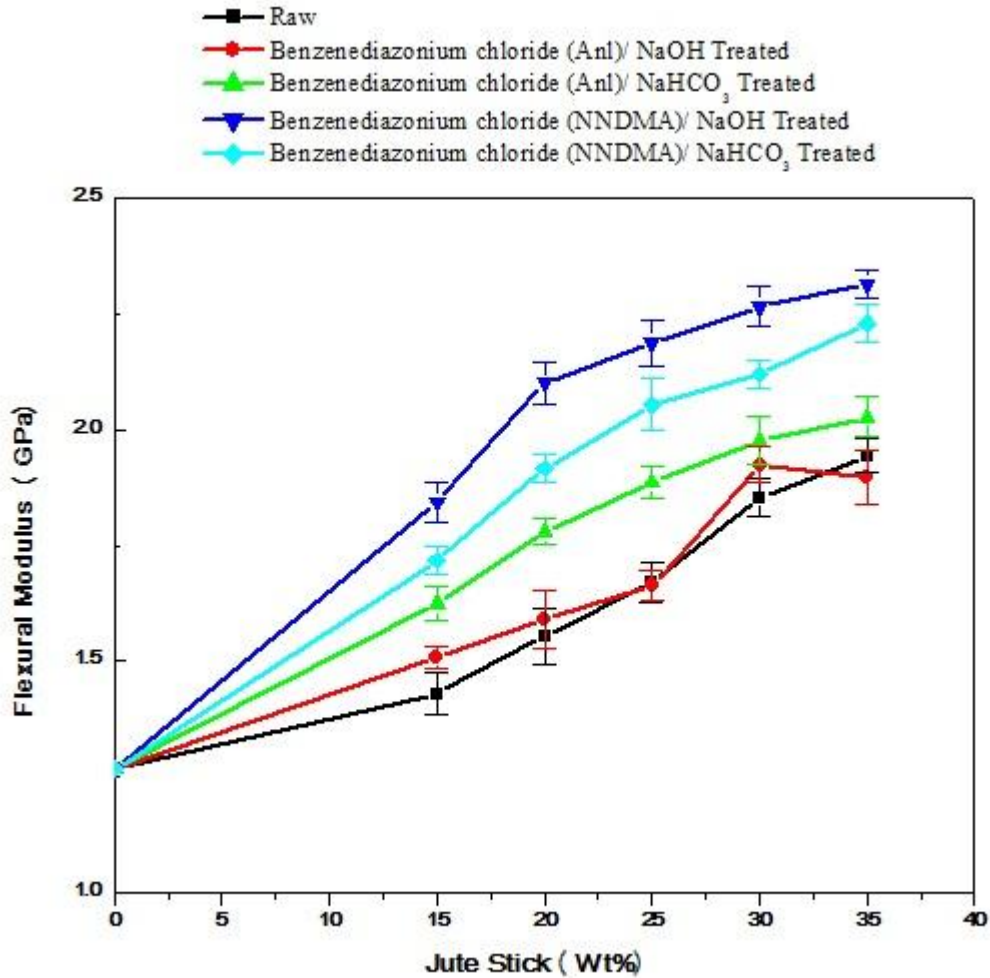


Fig. 4.10: Flexural modulus of JS-PP composites as a function of fiber loading.

#### 4.2.3 Charpy impact strength

The comparative charpy impact strength with fiber loading for both raw JS-PP composites and chemically treated JS-PP composites is shown in Fig. 4.11. The Charpy impact test is a standardized high strain-rate test which determines the amount of energy absorbed by a material during fracture. This absorbed energy is a

measure of a given material's toughness and acts as a tool to study brittle-ductile transition. It is observed from Fig.4.11 that the notched Charpy impact strength shows similar trend as flexural strength properties, i.e. impact properties increase with fiber loading in accordance with other similar studies in the literatures [34-38, 57-61].

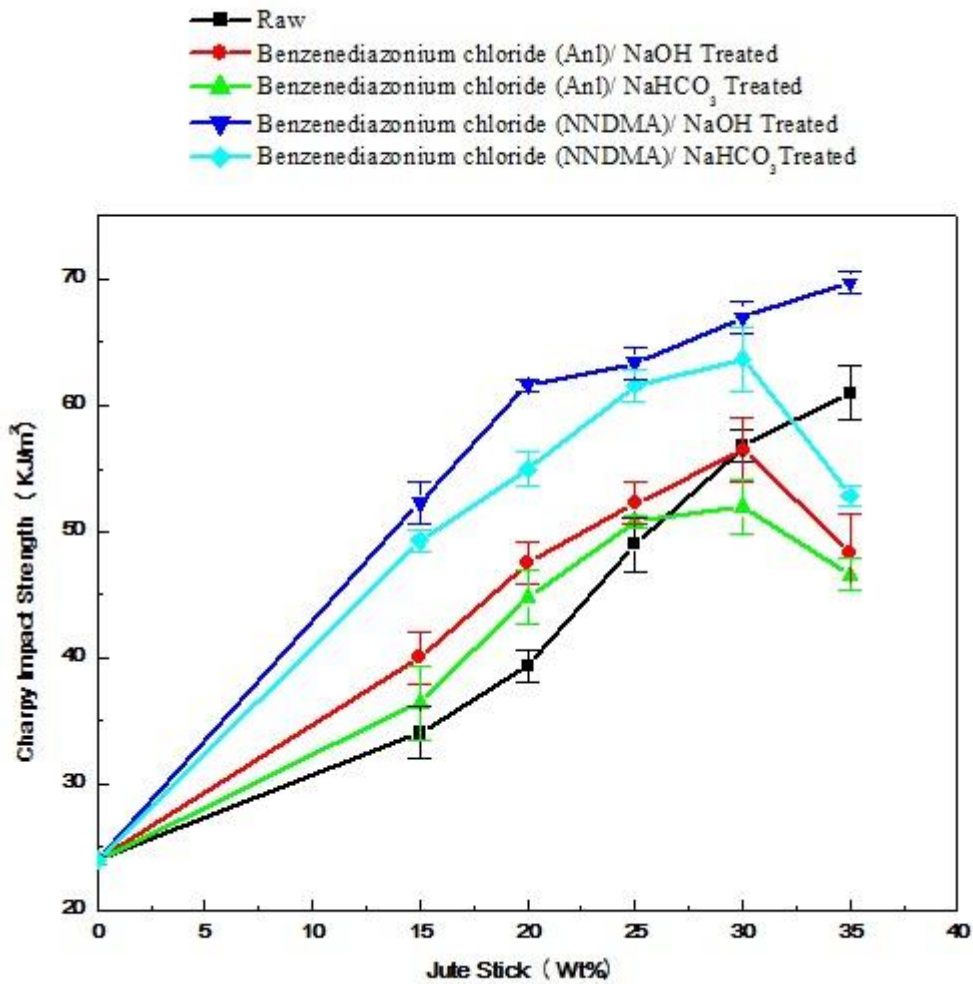


Fig. 4.11: Impact strength of JS-PP composites as a function of fiber loading.

The highest value of impact strength is observed for 35 wt% of fiber loading corresponding to benzene diazonium-chloride (using NNDMA)/sodium hydroxide treated JS-PP composites. One of the factors of impact failure of composite is fiber pull out. With increase in the fiber loading, bigger force is required to pull out the fibers. This consequently increased the impact strength. The maximum value of impact strength of the treated JS-PP composites is found to be 69.70 KJ/m<sup>2</sup> which is

greater than that of the untreated JS-PP composites with maximum value of 60.98 KJ/m<sup>2</sup>. This corresponds to 14.30%, increase in the impact strength.

#### **4.2.4 Hardness results**

Fig. 4.12 shows the hardness of various composites at different fiber loading. The average Rockwell hardness increased with the chemical treatment of JS flour. This is due to the decrease of flexibility and increase of stiffness of composites. The highest value of hardness, obtained for 30 wt% of fiber loading corresponding to benzene diazonium-chloride (using NNDMA)/sodium bicarbonate treated JS-PP composites, is found to be 87.67 Rockwell (R) which is greater than that of the raw JS-PP composites with maximum value of 84 Rockwell (R). This corresponds to 4.37% increase in the Rockwell hardness. This could be attributed to both dispersion of the fiber into the matrix with minimization of voids and strong interfacial bonding between the fiber and matrix.



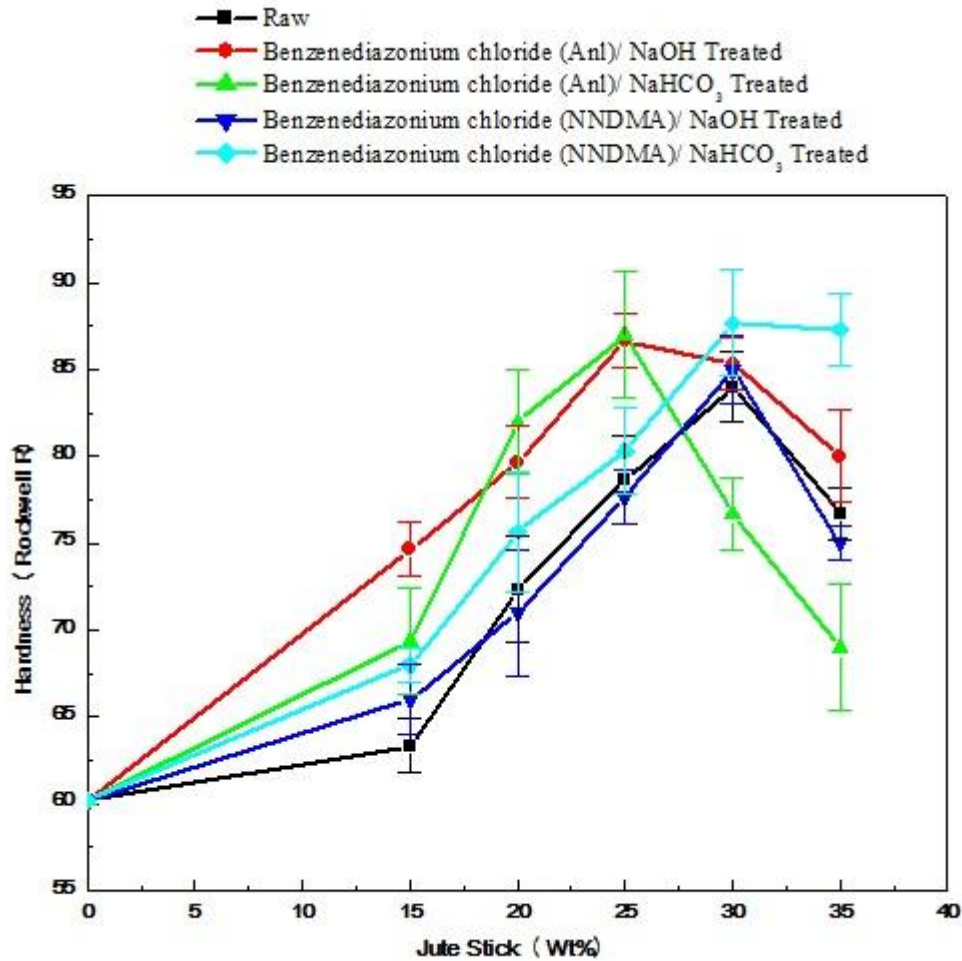


Fig. 4.12: Variation of Rockwell (R) hardness at different fiber loading.

#### 4.2.5 Water absorption characteristics

Water absorption of JS flour reinforced PP composites is important characteristic that determines terminal applications of these materials [27]. Water absorption could lead to a decrease in some of the properties and needs to be considered when selected for applications. It is difficult to eliminate entirely the absorption of moisture from the composites without using expensive surface barriers on the composite surface. Water absorption in lignocellulosic fibers enriched composites can lead to build up of moisture in the fiber cell wall and also in the fiber-matrix interphase region. Moisture built up in the cell wall could result in fiber swelling and the concerned dimension stability cannot be ignored [27].

Water absorption characteristics of the JS-PP composites against fiber loading are shown in Fig. 4.13. Water absorption (%) increases with fiber loading [89, 90, 27]. Chemically treated JS flour reinforced PP composites had higher water content compared to the raw JS-PP composites after 24 hrs immersion in distilled water at room temperature. Thus, although the chemical treatment increased the mechanical properties of the composites, it was unable to decrease the water absorption of the composites.

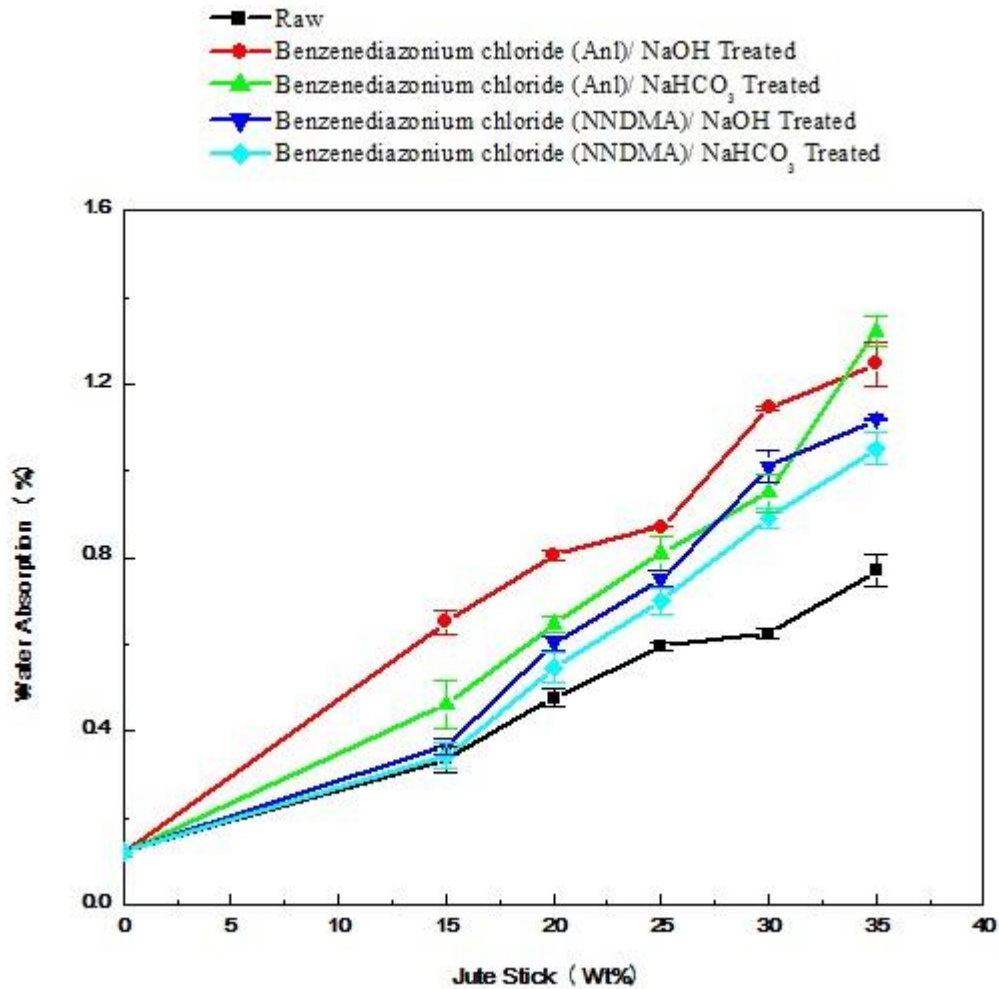


Fig. 4.13: Variation of water absorption as a function of fiber loading.

The highest value of water absorption for benzene diazonium-chloride (using Aniline)/ sodium bicarbonate treated JS-PP composites is found to be 1.32% whereas for raw JS-PP composites, its value is only 0.77%.

### 4.3 SEM ANALYSIS

The SEM morphology of the fracture surfaces of tensile and flexural test samples shows the phase information reflecting the reasons why the mechanical properties of the composites fabricated under different conditions of the fibers are different. The SEM image of JS-PP composites are shown in Fig. 4.14 – 4.23 for the 35 wt% fiber loading of raw and chemically treated JS-PP composites.

#### 4.3.1 Raw JS – PP composite

Fig. 4.14 and Fig. 4.15 illustrate the SEM micrographs of fracture surface of tension and flexural specimens, respectively. It is observed from both the figures (Figs. 4.14 and 4.15), the fiber surfaces have very clear boundary or interface indicating no strong bonding between JS flour and PP matrix. Also, there are some black holes which imply the fiber pull-out.

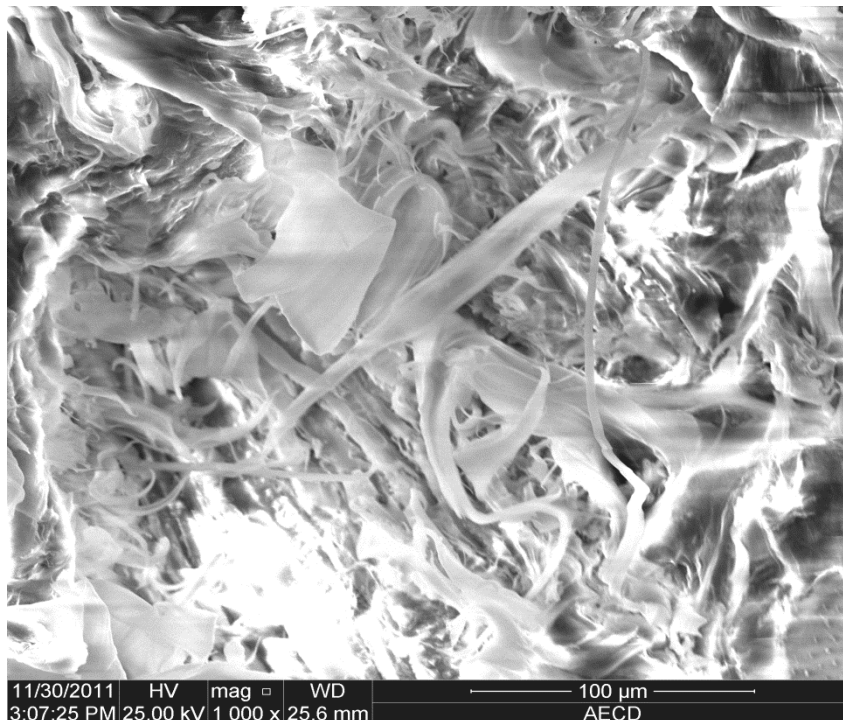


Fig. 4.14: SEM micrograph (fracture surface of tension specimen) of 35 wt% raw JS-PP composite.

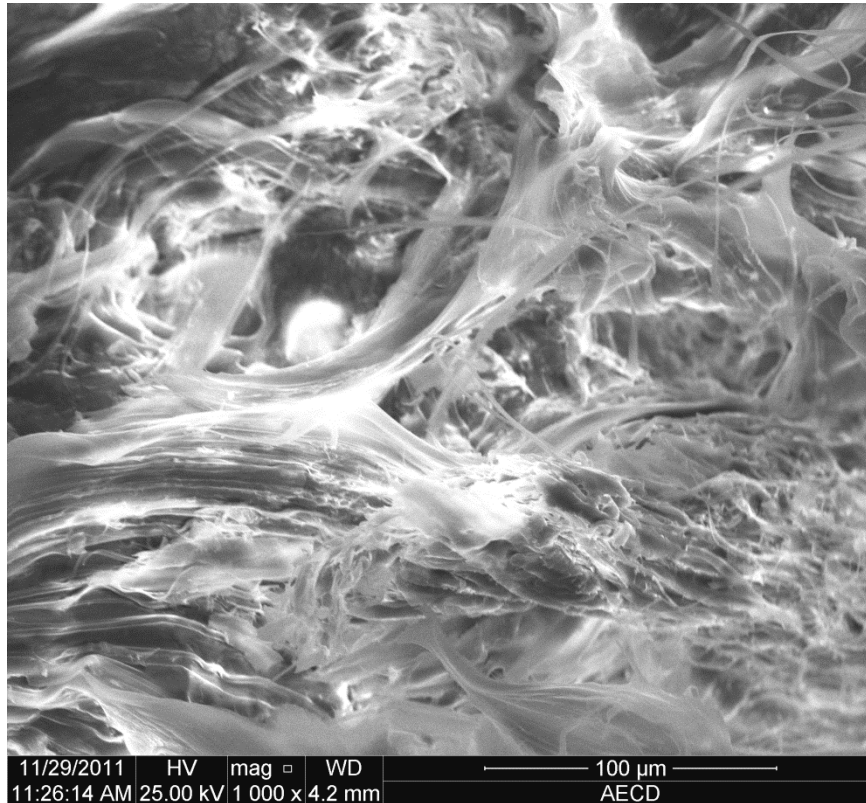


Fig. 4.15: SEM micrograph (fracture surface of flexural specimen) of 35 wt% raw JS-PP composite.

#### **4.3.2 Benzene diazonium-chloride (by using aniline)/ sodium hydroxide treated JS flour reinforced PP composite**

Figures 4.16 and 4.17 illustrate the SEM micrographs of fracture surface of tension and flexural specimens, respectively. The composite consists of benzene diazonium-chloride (by using aniline)/sodium hydroxide treatment of JS flour with 35 wt% fiber loading. Fiber pullout and de-bonding reduced significantly and the adhesion between fiber and matrix also improved which can be understood from the fact that there is no sharp interface between JS and PP matrix. This happens as a result of the formation of ester linkages between PP and OH groups of cellulose. The improvement of adhesion between fiber and matrix is verified by the higher tensile strength and flexural strength of the chemically treated composites with compared to raw JS-PP composites.

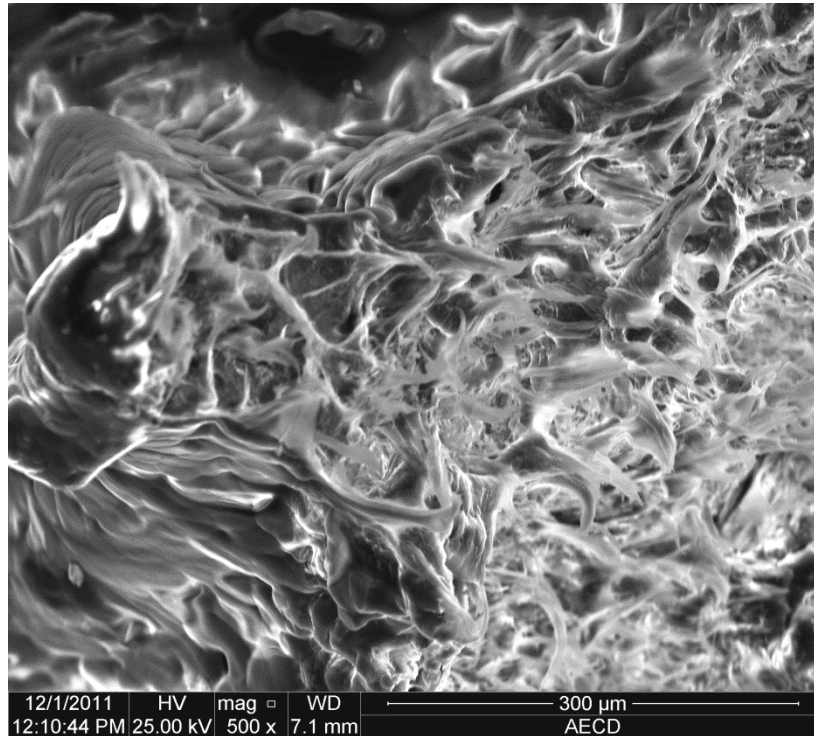


Fig. 4.16: SEM micrograph (fracture surface of tension specimen) of 35 wt% benzene diazonium-chloride (by using aniline)/sodium hydroxide treated JS-PP composite.

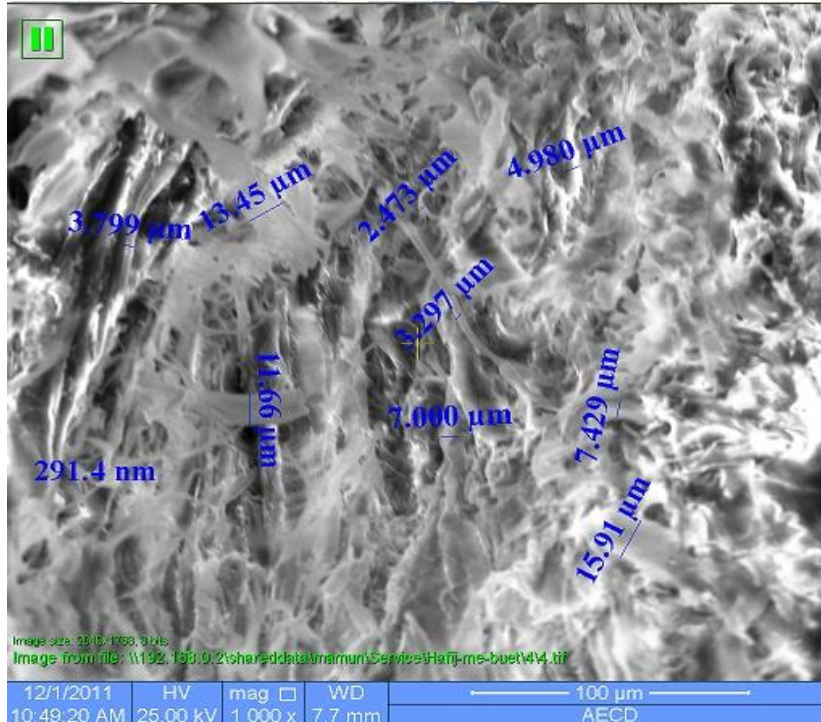


Fig. 4.17: SEM micrograph (fracture surface of flexural specimen) of 35 wt% benzene diazonium-chloride (by using aniline)/sodium hydroxide treated JS-PP composite.



### 4.3.3 Benzene diazonium-chloride (by using aniline)/ sodium bicarbonate treated JS flour reinforced PP composite

The SEM images of the benzene diazonium-chloride (by using aniline)/ sodium bicarbonate treated JS reinforced PP composites with 35 wt% of fiber loading are shown in Fig. 4.18 – 4.19 that show the influence of benzene diazonium-chloride (by using aniline) and sodium bicarbonate treatment of JS flour on the microstructures. It is observed that the adhesion between fiber and matrix is strong and fiber pullout reduced significantly. Apparently the interfacial adhesion of the treated fiber composites is much better than that of untreated fiber composites.

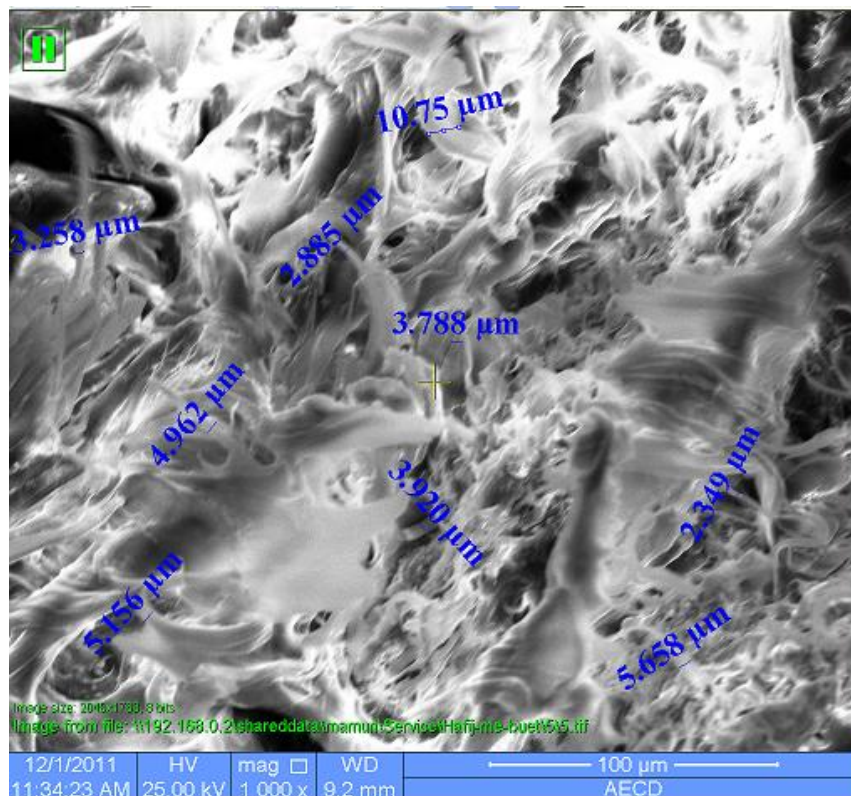


Fig. 4.18: SEM micrograph (fracture surface of tension specimen) of 35 wt% benzene diazonium-chloride (by using aniline)/sodium bicarbonate treated JS-PP composite.

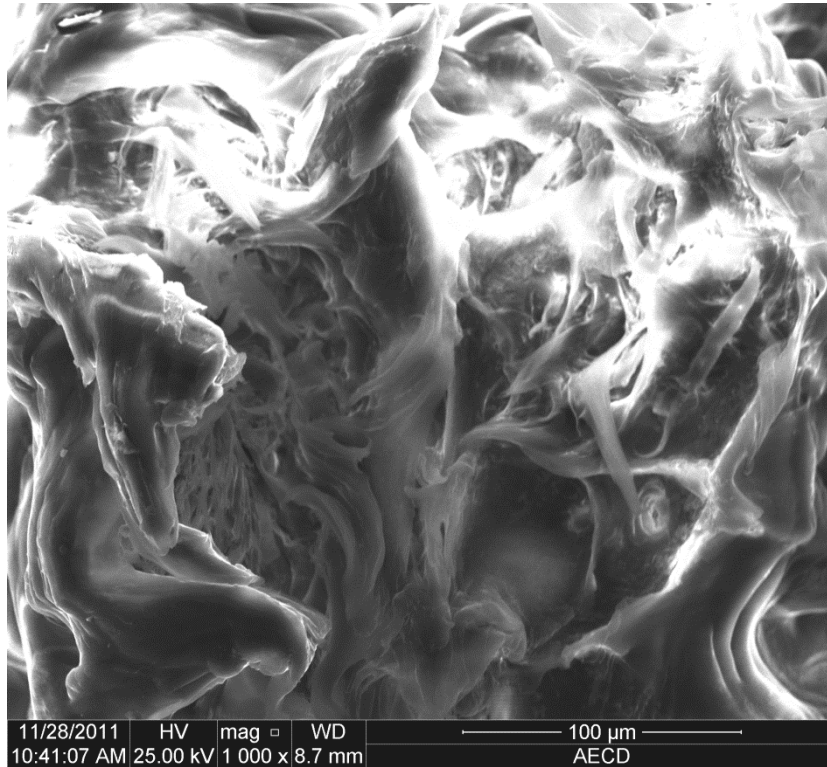


Fig. 4.19: SEM micrograph (fracture surface of flexural specimen) of 35 wt% benzene diazonium-chloride (by using aniline)/sodium bicarbonate treated JS-PP composite.

#### **4.3.4 Benzene diazonium-chloride (by using NNDMA)/sodium hydroxide treated JS flour reinforced PP composite**

The SEM images of the 35 wt% fiber loaded benzene diazonium-chloride (by using NNDMA)/sodium hydroxide treated JS flour reinforced PP composites are shown in Figs. 4.20– 4.21 that represents the influence of benzene diazonium-chloride (by using NNDMA)/sodium hydroxide treatment of JS flour on the microstructures. There is significant reduction of fiber pullout and improvement of adhesion between fiber and matrix due to chemical treatment of fibers, which was the result of the formation of ester linkages between PP and OH groups of cellulose, was attested by the experimental results of tensile test and flexural test.

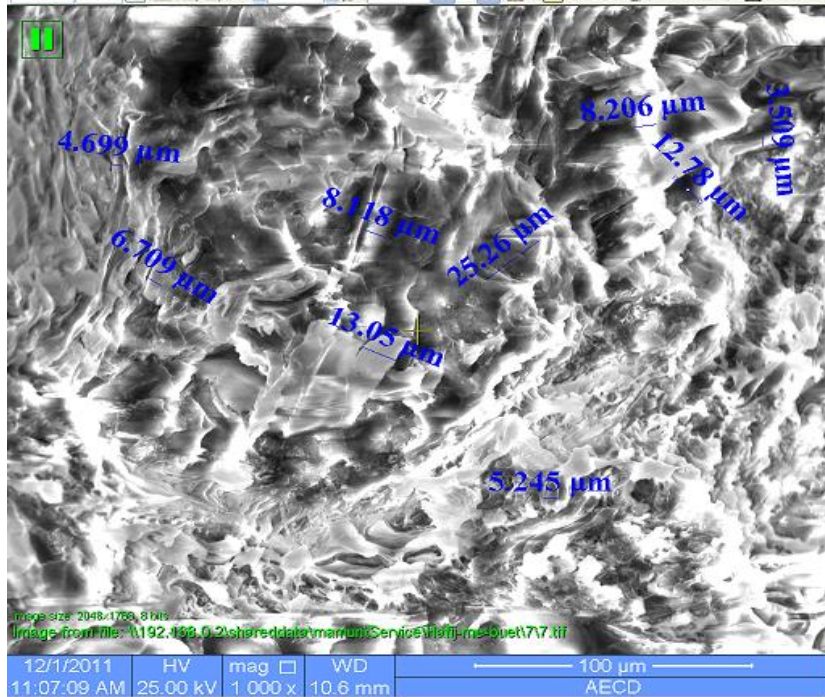


Fig. 4.20: SEM micrograph (fracture surface of tension specimen) of 35 wt-% benzene diazonium-chloride (by using NNDMA)/ sodium hydroxide treated JS-PP composite.

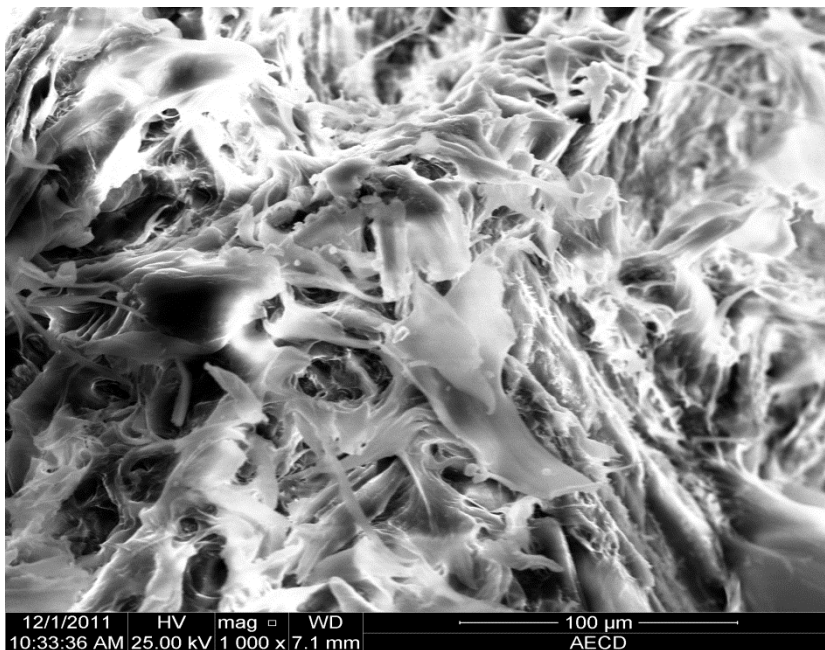


Fig. 4.21: SEM micrograph (fracture surface of flexural specimen) of 35 wt% benzene diazonium-chloride (by using NNDMA)/sodium hydroxide treated JS-PP composite.



#### 4.3.5 Benzene diazonium-chloride (by using NNDMA)/sodium bicarbonate treated JS flour reinforced PP composite

The SEM images of the 35 wt% fiber loaded benzene diazonium-chloride (by using NNDMA)/sodium bicarbonate treated JS flour reinforced PP composites are shown in Fig. 4.22 – 4.23. The Figures represent the influence of benzene diazonium-chloride (by using NNDMA)/ sodium bicarbonate treatment of JS flour on the microstructures. Fiber pullout reduced significantly and the adhesion between fiber and matrix improved significantly due to chemical treatment of fibers, which was the result of the formation of ester linkages between PP and OH groups of cellulose.

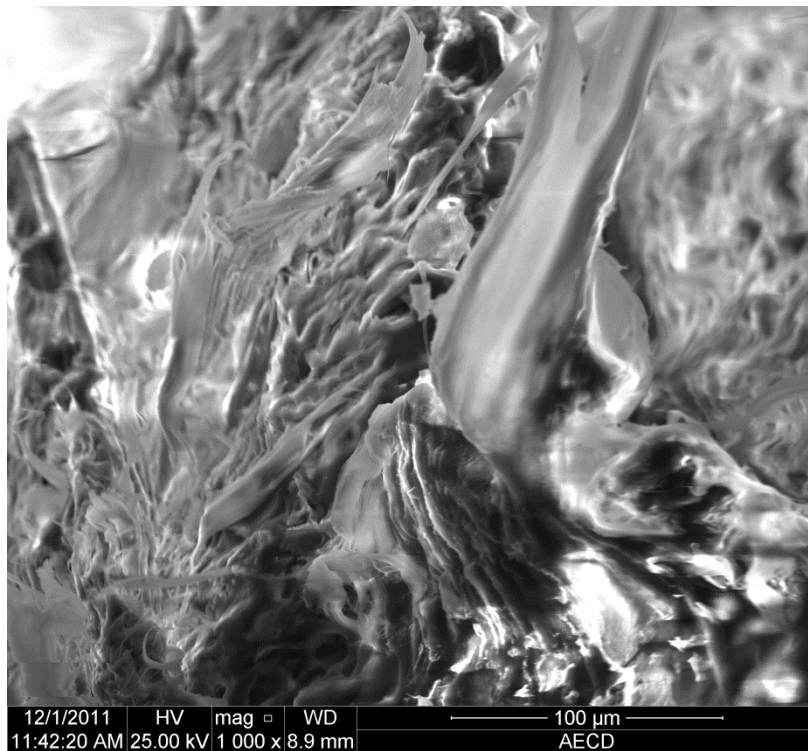


Fig.4.22: SEM micrograph (fractured surface of tension specimen) of 35 wt% benzene diazonium-chloride (by using NNDMA)/sodium bicarbonate treated JS-PP composite.

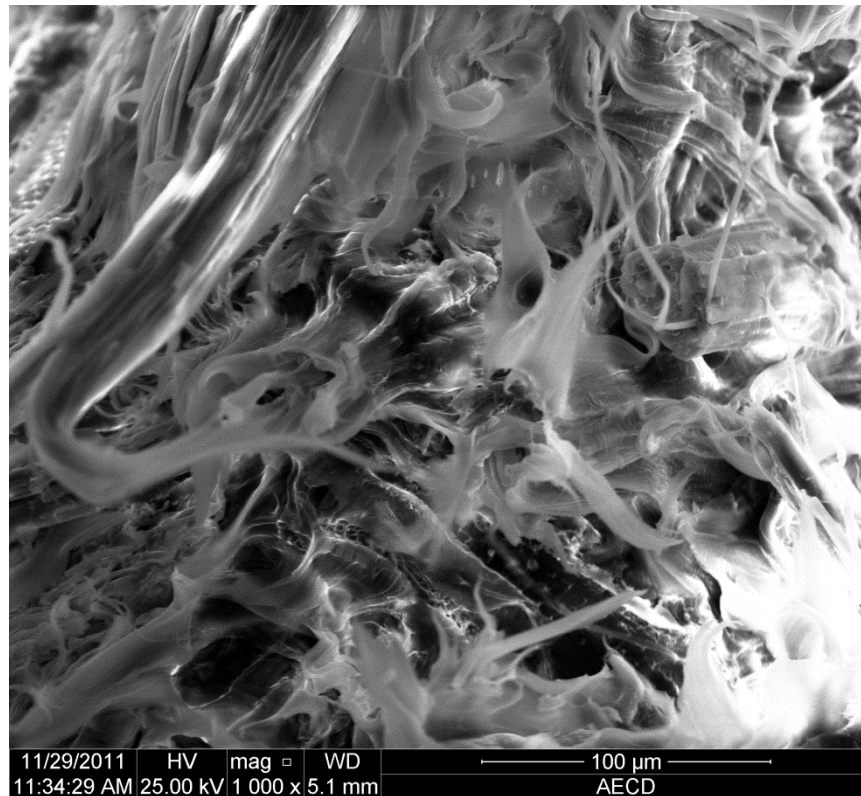


Fig. 4.23: SEM micrograph (fracture surface of flexural specimen) of 35 wt% benzene diazonium-chloride (by using NNDMA)/ sodium bicarbonate treated JS-PP composite.

#### **4.4 LIMITATIONS**

It is well-known that the properties of composites somewhat depend on manufacturing processes and conditions. For fully automatic machines, manufacturing conditions can be precisely controlled and consistency in the properties of composites can be ensured. However, in our laboratory, all the machines including the single screw extruder and the injection molding machine were manually operated and backdated. Therefore, the manufacturing conditions could not be controlled precisely for all the lots of composite specimens. Among various limitations, the following are considered to be mentioned:

- (i) The temperature of different sections of the single screw extruder was not possible to monitor and control.
- (ii) The injection molding machine was manually operated and old, so uniform pressure and speed were not possible to maintain for all the lots of specimens.

#### **4.5 COST ANALYSIS**

The sole purpose of conducting cost analysis is generally to determine the true costs of each of the products under analysis. We can utilize this knowledge to identify and prioritize cost-saving opportunities, to price the product (for paying beneficiaries) at a level that covers the true costs, and to compare the cost of the product with existing one.

Table 4.2 represents a simple analysis of cost of JS-PP composites in comparison with glass-PP or carbon-PP composites.

**Table 4.2: Cost analysis of JS-PP composites**

Sl. No.	Items	JS-PP composites	Glass(or Carbon)-PP composites	Benefits for using JS-PP
01	Raw reinforcing materials cost (per kg)	100/=	\$08 = 592/= [91]	83.10%
02	Processing of reinforcing materials (per kg)	250/=	\$09 = 666/= [92]	62.46%
03	Handling cost of reinforcing materials (per kg)	5/=	\$0.5 = 37/= [93]	86.48%
04	PP (per kg)	160/=	160/=	0%
05	Manufacturing cost of composites (per kg with 15-35 wt% fiber loading)	500/=	\$09 = 666/= [93]	23.07%
06	Packaging and carrying cost of composites (for 200 specimens of size 5" ×2" ×1" )	25/=	\$5 = 370/= [92]	93.24%
Total cost of composites		1040/=	2491/=	58.24%

From the above analysis, it is seen that using of jute sticks as reinforcing materials ensures a financial benefit of around 58.24% in comparison with glass (or carbon) fiber composites. Thus it can be concluded that natural fiber composites have the advantage of low-cost in addition to other properties of interest.

## CONCLUSIONS AND RECOMMENDATIONS

### 5.1 CONCLUSIONS

The specific aims of this study were to produce biodegradable (at least partially) polymer composites and develop chemical technique of improving mechanical properties of lignocellulosic fiber-plastic composites. Another aim of this study was to look for better use of jute stick which may economically help the poor farmers of Bangladesh.

In this study, JS-PP composites were fabricated by using a single screw extruder and an injection molding machine. Five different categories of composites were manufactured. These are i) raw JS-PP composites, (ii) benzene diazonium-chloride (by using aniline)/ sodium hydroxide treated JS-PP composites, (iii) benzene diazonium-chloride (by using aniline)/ sodium bicarbonate treated JS-PP composites, (iv) benzene diazonium-chloride (by using NNDMA)/ sodium hydroxide treated JS-PP composites, and (v) benzene diazonium-chloride (by using NNDMA)/ sodium bicarbonate treated JS-PP composites. Again for each category of composites, different wt% of fibers was used to manufacture the composites. From the analysis of the results, following salient conclusions can be made:

- The tensile strength of the composites increased with an increase in the JS flour loading. However, the 30% benzene diazonium-chloride (by using aniline)/ sodium hydroxide treated JS flour reinforced PP composites exhibited better tensile properties than raw JS-PP composites.

- The Young's modulus, flexural strength, flexural modulus, charpy impact strength, and hardness of the raw JS-PP composites increased with an increase in the flour loading. In general, all of these properties were much better for treated JS-PP composites in comparison with raw JS-PP composites.
- The chemical treatments of JS flour increased the interfacial bonding between the fibers and matrix. For this reason, above properties of composites were improved due to chemical treatments. However, water absorption property was not improved because of chemical treatments.
- Among the four chemical treatment process, treatment with benzene diazonium-chloride (by using aniline)/ NaOH is found to be the best in terms of tensile strength. Again, treatment with benzene diazonium-chloride (by using aniline)/ NaHCO<sub>3</sub> is found to be the best in terms of flexural strength. Similarly, treatment with benzene diazonium-chloride (by using NNDMA)/ NaOH is found to be the best in terms of impact strength. Thus, It can be said that a particular chemical treatment process is not good for all properties of composites.

Finally, although water absorption property of JS-PP composites degraded because of the reinforcement with JS flour, other properties improved to a certain extent. This ensures the net benefits of the composites of interest of this study. Thus, the use of treated JS in JS-PP composites, will reduce the environmental problem caused by dumping waste polypropylene as well as it will reduce the degradation of fertility of soil due to the biodegradable nature of JS and high percentage of nitrogen content in treated JS. So, it can be concluded that chemical modification of jute sticks by various ways may improve the quality of JS-PP composites which can be considered as alternatives to costlier wood and can find applications in furnitures, door panels, and others.

## **5.2 RECOMMENDATIONS FOR FUTURE WORK**

To explore the possibility of further improvement in the properties of JS-PP composites, it is recommended:

- To analyze the effect of JS particle size.
- To investigate the effect of other chemical treatment methods of JS flour.
- To fabricate the JS-PP composites by other manufacturing methods.
- To investigate the effect of JS type on JS-PP composites.
- To investigate the effect of JS flour loading in the range of 1 to 14% in order to find out the optimization of flour loading which will offer a chance to optimize between mechanical properties and water uptake properties

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