EVALUATION OF DYE TREATED JUTE TEXTILE REINFORCED POLYPROPYLENE COMPOSITES

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(Sharfun Nahar Arju)

DEDICATED

TO

MY BELOVED MOTHER

Jahan Ara Khatun

&

MY LATE FATHER

Md. Moazzem Hossain

TABLE OF CONTENTS

List of Tables

List of Figures

List of Abbreviations of Terms

JRPC	Jute reinforced polymer composite
NFR	Natural fiber reinforced
PP	Polypropylene
LDPE	Low density polyethylene
HDPE	High density polyethylene
MAPE	Maleic anhydride grafted polyethylene
MAPP	Maleic anhydride grafted polypropylene
FTIR	Fourier transform infrared spectroscopy
TGA-	Thermogravimmetric analysis
DTG	Derivative thermo-gravimetry
SEM	Scanning electron microscopy
A	Albafix
AS	Albafix and sodium hydroxide
G	Glytac
GS	Glytac and sodium hydroxide
I	Indosol E-50

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ABSTRACT

Natural fiber reinforced composites have been an emerging area now-a-days in the field of polymer science. The adverse effects of hydrophilic nature of natural fibers on their adhesion to hydrophobic polymer matrices is one of the important issues to be resolved. To achieve this goal with a view to improving the compatibility between the hydrophilic and hydrophobic components, a new approach of chemical modification of jute fabrics has been proposed and adopted. First, chemical modifications of plain jute fabrics with some cationic agents, such as Albafix WFF (poly- diallyl- dimethylammonium chloride), Glytac (Glycidyl trimethyl ammonium chloride), and Indosol-E50 (polyethylene polyamine) in the presence and absence of reactive dye (Drimarene Red K-8b) with or without sodium chloride, sodium carbonate, and sodium hydroxide have been performed following 23 different procedures. Besides, two other conventional treatment procedures were employed to modify the same fabrics for comparing with new approaches. All the treated plain jute fabrics were characterized in terms of water absorbency, FTIR spectra, color measurement, wash fastness, and tensile strength. Mechanical behavior, thermal behavior, surface morphology, and water uptake properties of plain jute fabrics polypropylene composites were evaluated from which the most effective treatment procedure was selected. Then, twill, single jersey, and rib jute fabrics were treated with this treatment procedure. Both treated and untreated jute fabrics of different structures were characterized and analyzed. PP composites of both treated and untreated fabrics of different structures were fabricated and evaluated. Plain jute fabrics treated with the mixture of Albafix WFF and sodium hydroxide and dyed with reactive dye only had 125% higher exhaustion, 95% higher dye uptake and better wash fastness than that of normal dyed jute fabrics. Composites of plain jute fabrics treated with the mixture of Albafix WFF and sodium hydroxide and again dyed with Drimarene Red K8b in the presence of NaCl and Na_2CO_3 had 109% higher tensile strength than that of the composites of untreated fabrics and 88% higher than that of the composites of NaOH treated fabrics. Also, these composites exhibited 49% lower water uptake value than that of untreated jute fabrics PP composites and 28% lower uptake than that of NaOH treated fabrics PP composites. Composites of untreated twill fabrics had 40% higher tensile strength than the composites of untreated plain fabrics. Composites of untreated rib had 58% lower water uptake than that of untreated plain fabric composites.

CHAPTER 1

INTRODUCTION

1.1 Background

The term “Composite materials” may perhaps be simply defined on the basis of the classic definition of a composite material as given in Longman’s dictionary: “something combining the typical or essential characteristics of individuals making up a group”. Thus, commonly speaking a composite material (simply called composite) is a material made from two or more constituent materials with significantly different physical or chemical properties that, when combined, produce a material with characteristics different from the individual components. The individual components remain separate and distinct within the finished structure. The new material may be preferred for many reasons: common examples include materials which are stronger, lighter, or less expensive when compared to traditional materials. In the engineering prospect, 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.

According to above definition, most composites have at least two constituent materials: a binder or matrix and a dispersed phase or reinforcement (Clyne and Hull 1996). Different kinds of polymers such as epoxy, polyester, polypropylene, polyethylene, polyurethane are used as a matrix. Among them polypropylene (PP) is mostly used because of its advantages in safe handling, easy processing and less hazard on the environment. The reinforcements are usually much stronger and stiffer than the matrix, and give the composite their good properties. The matrix holds the reinforcements in an orderly pattern. Composites can be classified from different points of view; textile-reinforced composites are one of the classes. Textile-reinforced composites are those composites which contain fibrous reinforcements in the form of a textile, i.e. in the form of fibers, yarns or fabrics. Here some terms related to textile engineering are described for ready references.

Textile Fibers

Textile fibers have some characteristics which differ from those of normal fibers. Textile fibers can be spun into a yarn or made into a fabric by various methods including weaving and knitting. The essential requirements for textile fibers to be spun into yarn include a length of at least 5 millimeters, flexibility, cohesiveness, and sufficient strength. Other important properties include elasticity, fineness, uniformity, durability, and luster. For example, banana fibers are one kind of fibers but they are not textile fibers. Because they cannot meet the above mentioned requirements. Hence, textile fibers can be defined as the basic entity, either natural or manufactured, which can be twisted into yarns, and then can be used in the production of fabrics.

Yarns

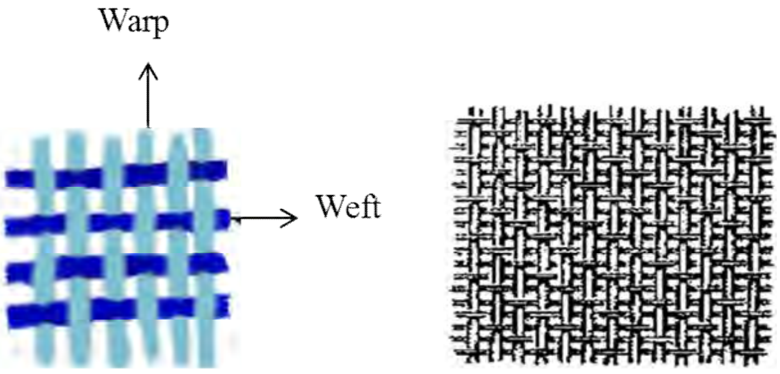
A yarn is a long continuous length of interlocked fibers, suitable for use in the production of textiles, sewing, knitting, weaving, embroidery, and rope making. A yarn is produced by spinning raw fibers.

Fabrics

A fabric is one kind of yarn sheet which is manufactured commonly through weaving or knitting.

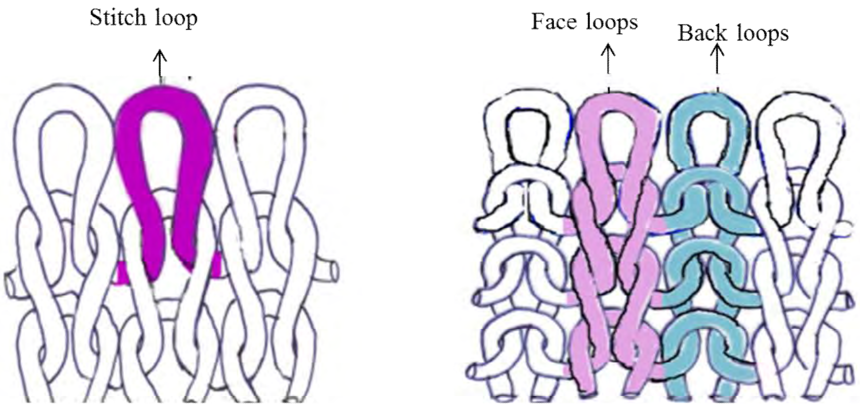
Woven fabrics

A woven fabric is a textile formed by weaving, which is made by the interlacement of two sets of yarns, warp (length wise) and weft (width wise) yarns, on a loom. Plain and twill fabrics are widely used for clothing purpose. In plain weave, the warp and weft yarns are aligned so they form a simple criss-cross pattern. Each weft thread crosses the warp threads by going over one, then under the next, and so on. The next weft thread goes under the warp threads that its neighbor went over, and vice versa (Figure 1.1a). Twill is a type of textile weave with a pattern of diagonal parallel ribs. This is done by passing the weft thread over one or more warp threads and then under two or more warp threads and so on, with a "step" or offset between rows to create the characteristic diagonal pattern (Figure 1.1b). Because of this structure, twill generally drapes well.



(a) Plain (b) Twill

Figure 1.1: Structure of woven fabrics, (a) plain and (b) twill (www.en.wikipedia.org.wiki/weaving).



(a) Single jersey (b) Rib

Figure 1.2: Structure of weft knitted fabrics, (a) single jersey and (b) rib (www.en.wikipedia.org.wiki/Knitting).

Knitted fabrics

When a fabric is made by interloping of one or one set of yarns, all running in the same direction through knitting is called a knitted fabric. At list one or one set of yarns is needed to make a knit fabric. Some knits have their yarns running along the length of the fabric (warp knitted fabric), while others have their yarns running across the width of the fabric (weft knitted fabric). Single jersey and rib are the common weft knitted fabrics. Single jersey is the simplest of all weft knitted constructions produced on machines employing only one set of needles. Hence this construction is termed as plain jersey or plain knit, simplest of all single jersey constructions (Figure 1.2a). In the rib fabrics, a basic stitch is used in weft knitting in which the knitting machines require two sets of needles operating at right angles to each other. Rib knits have a very high degree of elasticity in the crosswise direction (Figure 1.2b).

Yarn count

The yarn count is a numerical expression, which defines its fineness or coarseness. It also expresses weather the yarn is thick or thin. A definition of yarn count is given by textile institute, "count is a number indicating the weight per unit length or the length per unit weight of yarn."

Tex

Yarn count can be measured by two ways, such as direct system (weight/ length) and indirect system (length/ weight). Tex is the unit of yarn count, which is expressed in direct system. The yarn number or count in the Tex system is the weight in grams of 1 Km of yarn.

On the basis of general class of fibers, textile-reinforced composites can be classified into two classes: synthetic fiber composites and natural fiber composites. Synthetic fiber composites are commonly called conventional composites where glass and carbon fibers are widely used fibers because of their advantages in easy processing, better productivity and superior mechanical properties. But their higher density and non-recyclability at the end of their life cycle become significant disadvantages making them unadvisable to use. Manufacturing of these fibers consumes huge energy that is an added threat to the present era of energy crisis. Energy consumption also causes pollution of environments. In addition, disposal of these non-recyclable fibers creates the problem of land filing. Hence, a relatively newer concept is to consider natural fibers as a reinforcing material. Natural fibers provide interesting properties of the final composite especially those related to the environmental issues. Instead, composites are the 21st century materials to meet the stringent demands of lightweight, high strength, and corrosion resistance. Natural fiber reinforced composites, the wonder materials with lightweight, high strength-to-weight ratio and stiffness properties, have come a long way in replacing the conventional materials like metals, ceramics, etc. Now-a-days, one of the highly attractive research lines is the development of natural fiber reinforced composites. The material scientists all over the world focused their attention on natural composites reinforced with jute, sisal, coir, pineapple, etc., primarily to cut down the cost of raw materials.

However, natural fiber composites have some demerits. These 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. Another major problem of natural fiber reinforced composites is their susceptibility to fungal and insect attack and to degradation by moisture (Ehrenstein and Kabelka 2006). The high moisture absorption of the natural fibers and their low microbial resistance are important aspects that need to be considered, particularly during shipment and long-term storage as well as during processing of the composites (Moore and Cervenka 2002). In addition, their hydrophilic behavior affects the properties of the fibers themselves as well as the properties of their composites (Moore and Cervenka 2002). 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 polymer matrix (hydrophobic) prevents efficient fiber-matrix bonding (Rowell and Stout 1985). In these respects, surface modification of the fibers with coupling agents is generally needed because it will facilitate the fiber dispersion and bond formation between the fibers and polymer. Efficient coupling is usually not so simple. The lack of reactive groups in polymers prevents direct coupling of the components (Moore and Cervenka 2002). Thus, a suitable and efficient technique is essential in order to overcome the above mentioned problem.

1.2 Current State of the Research

Among all the natural fibers, jute is abundantly available in tropical countries like Bangladesh and India (Khan et al. 2005). Therefore, it is of particular interest to use jute fibers as reinforcement in polymer matrix. Composites made of jute fibers have high tensile and flexural properties compared to other natural fibers (Hassan et al. 2003). Besides, jute has high cellulose content and low microfibril angle which are the desirable properties of a fiber to be used as reinforcement in polymer matrix (Bogoeva-Gaceva et al. 2007). In spite of the above mentioned advantages, jute fibers like other natural fibers exert some difficulties when they are used as reinforcement in non-polar polymer matrices. These natural fibers are hydrophilic in nature as they are derived from lignocellulose, which contain strongly polarized hydroxyl groups. Hence, these fibers are inherently incompatible with hydrophobic thermoplastics, such as PP. The major limitations of using these fibers as reinforcements in such matrices include poor interfacial adhesion between polar-hydrophilic fiber and nonpolar hydrophobic matrix, and difficulties in mixing due to poor wetting of the fiber with the matrix. To overcome this problem, it has been a fashion to modify these fibers by chemical treatments using some common chemicals such as alkali, methyl acrylate, potassium permanganate, etc.

Many researchers have worked on jute fiber-based polymer composites to impart better fiber-matrix adhesion (Khan et al. 2001; Khan et al. 1999; Sanadi et al. 1995; Karmaker et al. 1996; Joseph et al. 2002; Xue et al. 2007). Most of the researchers (Wei-Ming et al. 2008; Ray et al. 2001; Liu et al. 2007; Stocchi et al. 2007; Kumar et al. 2005; Leonard et al. 2002; Wang et al. 2010; Juganta et al. 2012) have tried to modify the jute fibers by using alkali. Seki in 2009 used siloxane for the above purpose and also performed plasma treatment. Recently, Jahangir et al. (2013) and Zaman et al. (2010a) have performed treatment using potassium permanganate to modify jute fabrics for the improvement of mechanical properties of the composites. Haydaruzzaman et al. (2010) also treated the jute fabrics with different acrylic monomer with radiation and consequently they observed better mechanical properties (Haydar et al. 2012a; Haydar et al. 2012b). Different chemical modifications including permanganate treatment of natural fibers for using in composites were reviewed by Xue et al. (2007). Rahman et al. (2008) used urotropine and Corrales et al. (2007) used oleoyl chloride to enhance the mechanical properties of jute polypropylene composites and acetone treated jute fabrics were used as reinforcement for a thermoset resin by Betiana et al. (2005). Oever and Snijder (2008) used grafted polypropylene and observed the processing and ageing properties of jute fiber reinforced polypropylene (PP) composites. Ghosh and Das (2000) modified jute fiber by some low molecular weight glycols and a polyol under thermal treatment. All of these researchers have used jute either in the form of fibers or plain woven fabrics. Araujo et al. (2008) observed the effect of weft knitted structure of cotton fabrics on polymer matrix composites and reported improved properties of the composites. These modifications have been done for jute either in the form of fiber, yarn or plain woven fabrics. They have not used jute fabrics of other structures, specially knit structure. So, it is necessary to investigate in details the effects of different fabric structures on PP matrix composites.

Apart from the conventional modifications of jute fiber in the field of composites, modification of fibers with reactive dyes is widely used in the discipline of textile dyeing. Reactive dyes are mostly used for dyeing cotton fiber and during dyeing, these dyes react with hydroxyl group of the fiber and form strong covalent bond in presence of alkali. That is why their fixation and fastness properties are very good but their total dye utilization percent is very poor. Because during the dyeing process, hydrolysis reaction takes place, non-reactive oxi-dye form, which is lost for dyeing, and passes into the waste water and finally affects the environment. Only 60-65% dye utilization is attainable even with the use of salt in the normal cotton dyeing systems. In case of jute dyeing, this is 30 to 40 percent. So, a huge amount of dyes are wasted in the normal reactive dyeing system which affects the environment also. Thus, to improve the % dye utilization or to improve the dyeing efficiency such as exhaustion, fixation or wash fastness properties, recently some researchers focus on introducing cationic sites into the cellulosic fibers for better interaction with reactive dye. Recently, Glytac and sodium hydroxide treated jute fibers (Wang et al. 2010) were dyed with reactive dyes in combination with sodium chloride and sodium carbonate. Khan (2007) explained in his paper that the grafted fiber shows greater tensile strength and possesses better color fastness than that of bleached fiber. Physical properties of dyed jute fabrics were observed by Uddin et al. (2007). In these studies, however, attention was focused only on the dyeing performance of modified jute fibers. Bonding and adhesion characteristics of treated jute fibers with polymer were not analyzed.

In the studies mentioned above, some researchers have paid attention to the modification of jute fibers by conventional ways i.e. by using conventional chemicals such as NaOH, potassium permanganate, and different types of acrylic monomer. After modification, they have investigated the effects of modified fibers on the characteristics of their resulting composites. Few researchers (Wang et al. 2010; Khan 2007, Uddin et al. 2007) have focused their attention to the modification of jute fibers by dye followed by the characterization of the modified fibers only. They have not examined the effects of dye modified jute fibers on their resulting composites. Finally, only a very few researchers (Jahangir et al. 2013; Stocchi et al. 2007) have investigated the effects of plain woven jute fabrics on their composites. Thus, it is found that detail studies of dye treatments of jute fabrics of various structures and their composites with PP are still lacking in the literature. Therefore, the present study is intended to focus on these aspects in an attempt to evaluate different physical, thermal, and mechanical properties of jute textiles reinforced PP composites.

1.3 Objectives

As mentioned in the preceding section, detail studies of dye treatments of jute fabrics of various structures and their composites with PP have not been carried out so far. Therefore, main objectives of the present study are to fabricate the jute fabrics of four different structures, two woven (plain and twill) and two weft knitted (single jersey and rib), from untreated jute yarn and modify these jute fabrics in different ways where reactive dye and some cationic agents are specially included. Jute fabrics polypropylene composites are then fabricated by hot press method. Both treated and untreated jute fabrics and their composites are characterized. Some specific objectives of the present study are to

(i) Modify the jute fabrics by using nonconventional chemicals such as Drimarene Red K-8b, Glytac, Albafix WFF, and Indosol E-50.

(ii) Investigate the influence of above modifications of the plain jute fabrics on their properties, such as fabric strength, water absorbency, color strength, exhaustion, fixation, wash fastness, and surface chemistry by using Fourier Transform Infrared Spectroscopy (FTIR).

(iii) Investigate the effects of above treatments on different properties of composites, such as tensile strength, bending strength, impact strength, thermal behavior by Thermo-Gravimetric Analysis (TGA), surface morphology by using Scanning Electron Microscopy (SEM), and water uptake.

(iv) Compare the properties of composites manufactured with fabrics modified by the present and conventional techniques.

(v) Select the most effective treatment procedure based on the mechanical behavior of the treated plain jute fabrics PP composites.

(vi) Modify the twill, single jersey, and rib jute fabrics by the most effective treatment procedure.

(vii) Evaluate and compare the properties of treated and untreated plain, twill, single jersey, and rib jute fabrics PP composites.

CHAPTER 2

LITERATURE REVIEW

This section presents a brief review of literatures associated with composite materials, reinforcing fiber materials, matrix materials, fabrication of composites, and reactive dyes that are related to the present 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 Natural Fiber Based Polymer Composites

The word ‘composite’ means “a substance, which is made up by two or more distinct different substances”. 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 performance from the composite that is not available from the separate constituents. In the field of natural fiber composites, polymers have been the key matrix materials and have replaced many of the conventional materials because of their advantages in ease processing, better productivity and possible cost reduction. Properties of polymers are usually modified using fillers and fibers to suit higher strength and higher modulus requirements. Glass fibers as filler materials have found applications in many fields (Ku et al. 2011; Pickering et al, 2007), but their higher density and non-recyclability at the end of their life cycle become significant disadvantages making them unadvisable to use. On the other hand, natural fibers provide interesting properties of the final composite, especially those related to the environmental issues. Considering the ecological aspects of material selection, replacing synthetic fibers by natural ones is only a first step. Restricting the emission of greenhouse effect causing gases such as CO_2 into the atmosphere and an increasing awareness of the finiteness of fossil energy resources are leading to developing new materials that are entirely based on renewable resources (Essabir et al. 2013a, 2013b; Cao et al. 2006). Some other advantages associated with the use of natural fibers as reinforcement in polymer matrix are their non-abrasive nature, biodegradability, low energy consumption and low cost (Arrakhiz et al. 2012a, 2012b; Malha et al. 2013). Additionally, natural fibers have low density and high specific properties which are comparable to those of traditional reinforcements. Thus, the intrinsic properties of natural fibers can meet the requirements of the current global market especially for the aerospace and automotive industries (Ofomaja et al. 2011). As a result, they have been the potential substitute for non-renewable synthetic fibers. There is a wide variety of different natural fibers, which can be applied as reinforcement or fillers. An organogram with a classification of the various natural fibers which are used as filler in composite is presented in Figure 2.1.

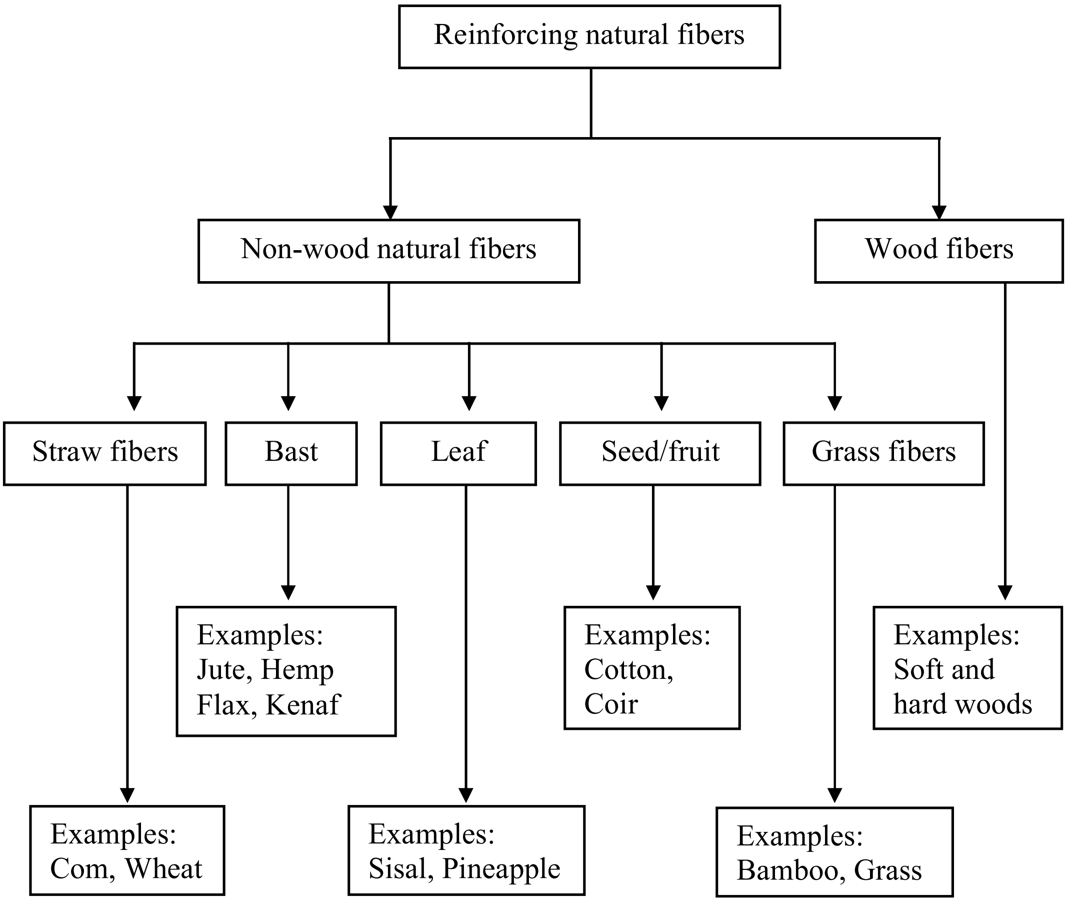


Figure 2.1: Classification of natural fibers which can be used as reinforcement or fillers in polymers (Kozlowski et al. 2009; Mohanty et al. 2002).

The most important types of natural fibers used in composite materials are flax, hemp, jute, kenaf, and sisal due to their properties and availability. Among various natural fibers, jute fibers occupy the second place in terms of world production rate of cellulosic fibers. Jute is an important bast fiber with a number of advantages. Jute has high specific properties, low density, less abrasive behavior to the processing equipment, good dimensional stability and harmlessness. Jute textile is a low cost eco-friendly product and is abundantly available in Bangladesh, easy to transport and has superior drapability and moisture retention capacity. It is being widely used as a natural choice for plant mulching and rural road pavement construction. The biodegradable and low priced jute products merge with the soil after using providing nourishment to the soil. Being made of cellulose, on combustion, jute does not generate toxic gases. Because of its eco-friendly and biodegradable nature, the demand for jute fibers is raising day by day (Chattopadhyay et al.2006; Cai et al. 2000). Consequently, jute fibers have now been the limelight in the field of natural fiber composites. Due to jute's low density combined with relatively higher stiffness and strength, the specific properties of jute fiber can compared to those of glass and some other fibers as mentioned in Table 2.1(Goettler et al. 2007).

Table 2.1: Properties of jute fiber in comparison with other fibers (Goettler et al. 2007)

Fiber	Density (g/cm^3)	Tensile Strength (MPa)	Young's Modulus (GPa)	Elongation at break (%)	Specific Tensile Strength ($MPa/g.cm^{-3}$)	Specific Young's Modulus ($GPa/g.cm^{-3}$)
Jute	1.4	393-773	13-26	1.2-1.5	286-562	9-19
Flax	1.5	345-1100	28	2.7-3.2	230-773	18
Ramie	1.5	400-938	61-128	1.2-3.8	267-625	41-85
Sisal	1.4	468-640	9-22	3-7	323-441	6-15
Coir	1.1	131-175	4-6	15-40	114-152	3-5
E-glass	2.5	2000-3500	70	2.5	800-1400	28
S-glass	2.5	4570	86	2.8	1828	34

Accordingly jute is a natural biodegradable fiber with advantages such as high tensile strength, good dimensional stability, heat and sound insulation properties (2003; Ghosh et al. 2004). Recently, due to the improvement of people's living standards and need for environmental protection, the demand of natural biodegradable and eco-friendly fibers is rising worldwide day by day. Ramie, flax, hemp, and some other vegetable fibers have been used as textile materials, but jute fiber is basically used for traditional purposes such as manufacture of sackings, hessian, carpet backing etc. Taking account of the costliness of ramie and the shortage in sources of flax, and the challenges from the synthetic fibers in the traditional jute products market, if jute could be used to replace ramie and flax partially as textile material, not only the cost could be reduced but also a new market would be provided for jute products (Ghosh et al.2004; Chattopadhyay et al. 2006; Pan et al. 2001).

2.2 Jute Fiber Composites and Their Constituents

2.2.1 Jute fiber

Jute fiber is obtained from two herbaceous annual plants, white Corchorus capsularis (white jute) originating from Asia and Corchorus olitorius (Tossa jute) originating from Africa. Next to cotton, it is the second most common natural fiber, cultivated in the world and extensively grown in Bangladesh, China, India, Indonesia, Brazil (Booth and Price 1989). The jute plant grows six to ten feet in height and has no branches (Figure 2.2). The stem of the jute plant is covered with thick bark, which contains the fibers. In two or three month time, the plants grow up and then are cut, tied up in bundles and kept under water for several days for fermentation. Thus, the stems rot and the fibers from the bark become loose. Then the cultivators pull off the fibers from the bark, wash very carefully and dry them in the sun.


(a) (b)

Figure 2.2: Photograph of (a) jute plants and (b) jute fibers.

Jute is multicelled in structure as shown in Figure 2.3 (Rong et al. 2001). The cell wall of a fiber is made up of a number of layers: the so-called primary wall (the first layer deposited during cell development) and the secondary wall (S), which again is made up of the three layers (S1, S2 and S3). As in all lignocellulosic fibers, these layers mainly contain cellulose, hemicellulose and lignin in varying amounts. The individual fibers are bonded together by a lignin-rich region known as the middle lamella. Cellulose attains highest concentration in the S2 layer (about 50%) and lignin is mostly concentrated in the middle lamella (about 90%) which, in principle, is free of cellulose. The S2 layer is usually by far the thickest layer and dominates the properties of the fibers (Olesen 1999).

Cellulose, a primary component of the fiber, is a linear condensation polymer consisting of Danhydro-glucopyranose units joined together by ß-1, 4-glucosidic bonds (Eichhorn 2001). The long chains of cellulose are linked together in bundles called microfibrils as shown in Figure 2.4 (Joseph et al. 2000; Rowell et al. 2007).

Hemicelluloses are also found in all plant fibers. Chemical structure of hemicellulose is shown in the Figure 2.5 (Rowell et al. 2007). Hemicelluloses are polysaccharides bonded together in relatively short, branching chains. They are intimately associated with the cellulose microfibrils, embedding the cellulose in a matrix. Hemicelluloses are very hydrophilic and have lower molecular masses than both cellulose and lignin. The degree of polymerization is about 50 – 200. The two main types of hemicelluloses

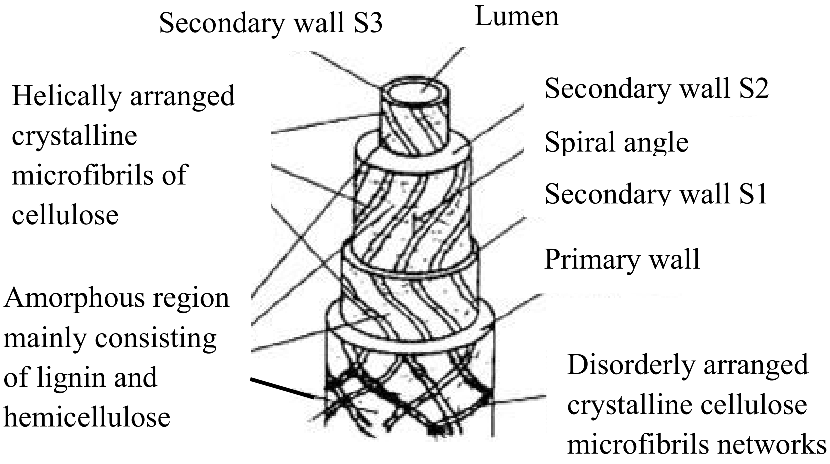


Figure 2.3: Schematic structure of natural fiber showing positional arrangement of its different components (Rong et al. 2001).

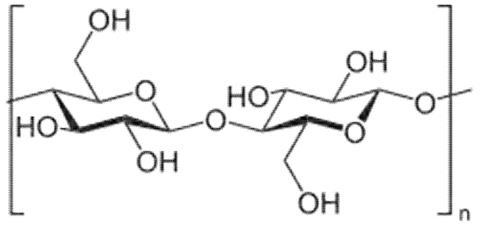
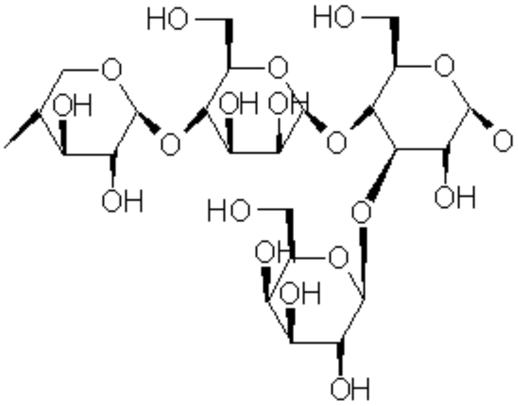


Figure 2.4: Chemical structure of cellulose (Joseph et al. 2000; Rowell et al. 2007).



Xylose - ß(1,4) – Mannose - ß(1,4) - Glucose-
alpha(1,3) - Galactose

Figure 2.5: Chemical structure of hemicellulose (Rowell et al. 2007).

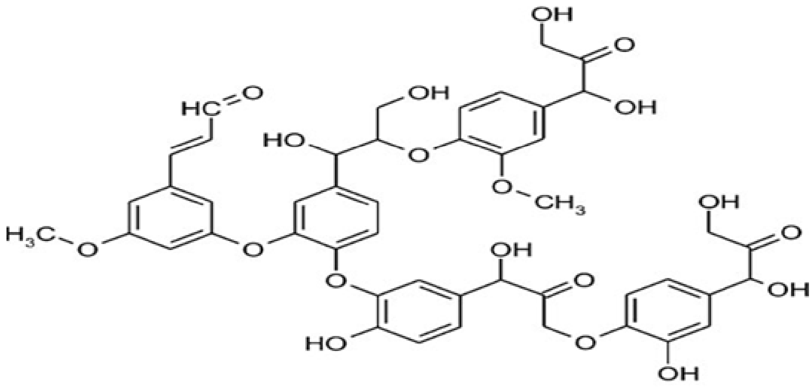


Figure 2.6: Chemical structure of lignin (Goda et al. 2009).

are xylenes and glucomannans. Lignin is a randomly branched polyphenol as shown in Figure 2.6 (Goda et al. 2009), made up of phenyl propane units. It is the most complex polymer among naturally occurring high-molecular-weight materials (Hon 2001) with an amorphous structure. Of the three main constituents in fibers, lignin is expected to be

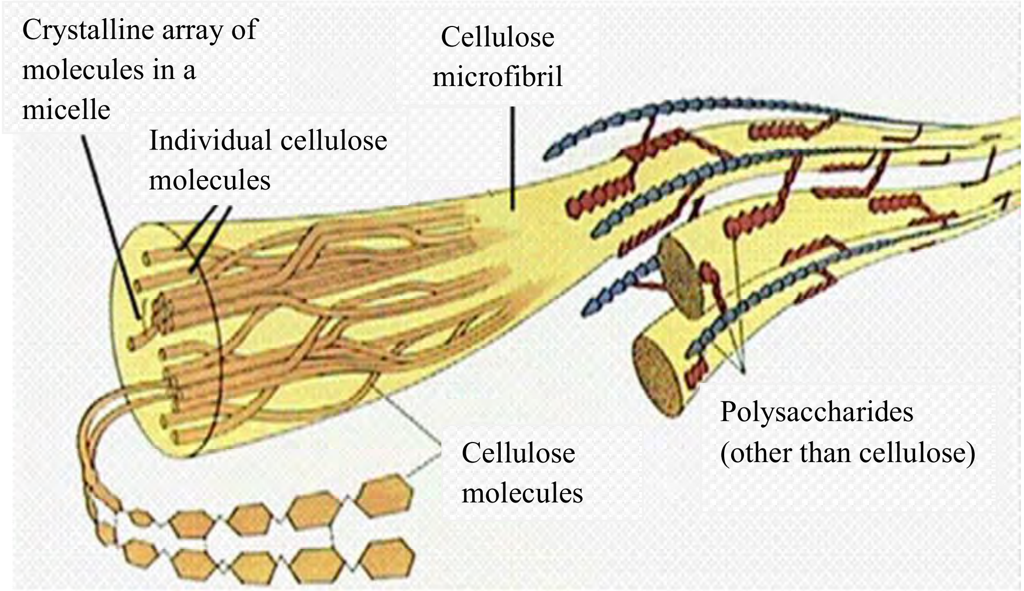


Figure 2.7: Structure of cellulose as it occurs in a plant cell wall (Farabee 2010).

the one with least affinity for water. Another important feature of lignin is that it is thermoplastic (i.e., at temperatures around 90°C it starts to soften and at temperatures around 170°C it starts to flow (Olesen 1999).

The jute fiber is polygonal or oval in cross section with a lumen as shown in Figure 2.7 (Farabee 2010). The structure of the jute fiber is influenced by climatic conditions, age and the fermentation process, which influence also the chemical composition (Bledzki and Gassan 1999). A typical chemical composition of jute fiber is given in Table 2.2 (Mohanty et al. 2000).

The jute fiber possesses moderately high specific strength and stiffness. Therefore, it is suitable as reinforcement in a polymeric resin matrix. However, it exhibits considerable variation in diameter along with the length of individual filaments. The properties of the fiber depend on factors such as size, maturity and processing methods adopted for the extraction of the fiber. Properties such as density, electrical resistivity, ultimate tensile strength and initial modulus are related to the internal structure and chemical composition of fiber (Wang 2004; Mohanty et al. 2001).

Table 2.2: Typical composition of jute fibers (Mohanty et al. 2000)

Substances	Weight percent (%)
Cellulose	61-72
Hemicellulose	13.6-20.4
Pectin	0.2
Lignin	12-13
Moisture content	12.6
Wax	0.5

2.2.2 Physico-chemical and dispersion properties of jute fibers

Crystallinity and Accessibility

The chemical accessibility of cellulose is inversely related to the degree of crystallinity. The accessible areas of cellulose are composed of amorphous areas and the surface of the crystallites. Due of highly ordered arrangement and hydrogen bonding in the crystalline regions, water cannot penetrate or disrupt these areas. However, strong solutions of alkali or other chemicals can swell and sometimes completely disrupt the micellar nature of cellulose.

Swelling

Swelling of cellulose is encountered by different types of solvent. The order of swelling power increases as: organic solvent < water < salts < acids < bases. On swelling, the amorphous areas become more accessible to chemical modification or degradation whereas the crystalline regions of cellulose remain intact.

Acid hydrolysis

Cellulose undergoes acid hydrolysis. The degree of susceptibility depends on amorphous or crystalline regions, concentrations, types of acid, and temperature. The acid hydrolysis is initially quite, but much slower later. This behavior reflects initial rapid attack and reaction in the amorphous zone, followed by a much slower hydrolysis in, or on the surface of, the crystallites. However, it is known that hydrolysis leads to increased crystallinity (Daniel 1985).

Water absorption

Natural fiber composites are susceptible to humidity through absorption of water. Due to presence of polar groups (such as hydroxyl groups) in cellulose components, natural fibers show the tendency to absorb water from its surrounding. Absorbed water can affect mechanical properties of the composites by interacting with polymer matrix, natural fibers and also fiber-matrix interfaces (Sreekumar et al. 2009). Three types of binding water are encountered in the cellulose materials. The most firmly bound water is the water of constitution which is released upon pyrolysis of cellulose. This is covalently bound water, not adsorbed water; less strongly monomolecular bound water which is hydrogen bonded to primary and secondary hydroxyl groups in the amorphous regions and on the surface of the crystallites. The least firmly bound water is the polymolecularly adsorbed water which is referred to as capillary condensed and is best thought of a solid solution of water in the cellulose matrix (Daniel 1985).

Water absorption in the composite can be visualized by three different mechanisms. The first involves diffusion of water molecules inside the micro holes between polymer chains. The second is the capillary transport into the gaps and flaws at the interfaces, due to the incomplete wettability and impregnation during the compounding process. The third is the transport of moisture by micro cracks or other forms of micro damages (Espert et al. 2004; Marom 1986). The way in which natural fiber reinforced composites absorb water depends upon many factors, such as temperature, fiber volume fraction, orientation of reinforcement, fiber nature (permeable or impermeable), area of exposed surface, diffusivity and the amount of surface protection (Springer 1998).

2.2.3 Modification of natural fibers

Cellulose fibers are inherently highly polar and hydrophilic, which make them poorly compatible with commonly used non-polar matrices, such as polyolefin, and subject to loss of mechanical properties upon atmospheric moisture absorption. The hydroxyl groups present in the cellulose backbone may involve in the hydrogen bonding within the cellulose molecules thereby reducing the activity towards the matrix. Surface modifications may activate these groups or can introduce new moieties that can effectively interlock with the matrix. Surface characteristics such as wetting, adhesion, surface tension, porosity etc. can be improved upon modifications. Some treatments of the fibers may lead to major changes in fiber surface roughness. The irregularities of the fiber surface play an important role in the mechanical interlocking at the interface (Joseph et al. 2000). Surface treatments, although having a negative impact on economics, are potentially able to overcome the problem of incompatibility. The appropriate modifications to the components give rise to changes in physical and chemical interactions at the interface. Several processes have been developed to modify fiber surface including chemical treatments, physical treatments, photochemical treatments, surface grafting, etc.

2.2.3.1 Theoretical perspectives of fiber modifications

The fiber surface treatment before introducing the matrix material can modify the interface region and alter the adhesion between the fiber and the matrix.

Alkali (NaOH) treatment

The alkali treatment can cause an increase of the fiber surface free energy. The adsorption of the epoxy resin on the jute fiber surface increases, which is a prerequisite condition for creating the interphases (George et al. 1999). Moreover, the alkali treatment can make the fiber surface 'clean' due to removal of waxes, hemicellulose, pectin and part of lignin. The removal of these substances enhances the surface roughness. Therefore, the mechanical interlocking at the interface could be improved. The following reaction takes place as a result of alkali treatment (Weyenberg 2006).

$$\text{Fiber} - \text{OH} + \text{NaOH} \longrightarrow \text{Fiber} - \text{O}^{-}\text{Na}^{+} + \text{H}_2\text{O}$$

The effect of alkali on cellulose fiber is a swelling reaction, during which the natural crystalline structure of the cellulose relaxes. Native cellulose (i.e. cellulose as it occurs in nature) shows a monoclinic crystalline lattice of cellulose-I, which can be changed into different polymorphous forms through chemical or thermal treatments. The important forms of alkali-cellulose and cellulose-II are shown in Figure 2.8 (Weyenberg 2006).

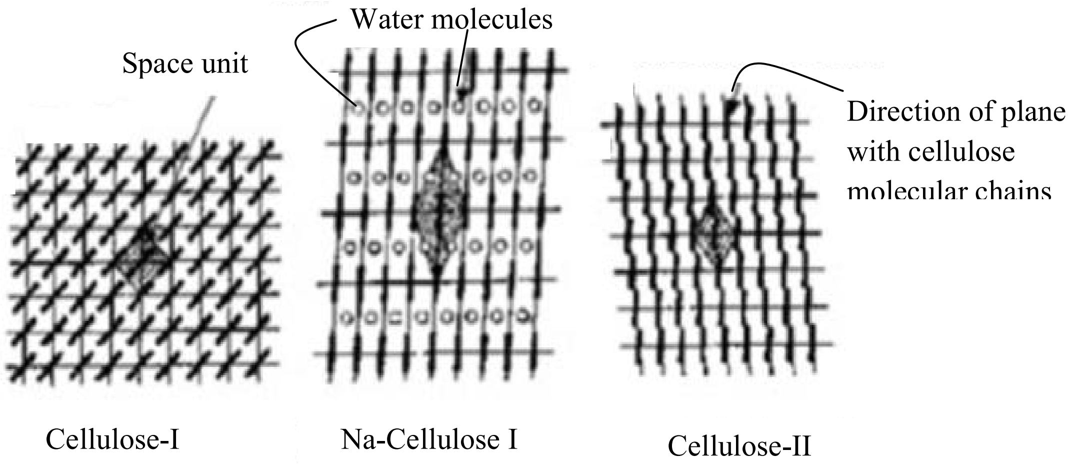


Figure 2.8: Lattice structures of cellulose-I and cellulose-II (Weyenberg 2006).

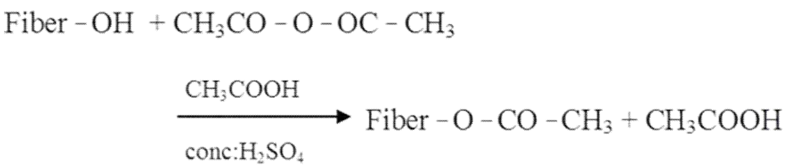
Silane treatment

These chemicals are hydrophilic compounds with different groups added to silicon such that one end will interact with matrix and the other end can react with hydrophilic fiber, which act as a bridge between them. The uptake of silane is very much dependent on a number of factors including hydrolysis time, organofunctionality of silane, temperature, and pH. Alkoxy silanes are able to form bonds with hydroxyl groups. Silanes undergo hydrolysis, condensation, and the bond formation stage. Silanols can form polysiloxane structures by reaction with hydroxyl group of the fibers (Bump 2001).

In addition to the self condensation of silanes and the condensation on the surface of cellulose fiber, amino silane molecules can interact with the OH groups of cellulose via their basic amino groups. Herrera-Franco and coworkers (Herrera-Franco 1997) have studied the influence of different silane coupling agents on the properties of henequen fiber-reinforced polymer composites. The authors used FTIR and XPS spectroscopy to show that the reaction between silanes and cellulose takes place only at temperatures above 70°C.

Acetylation

Acetylation is a rather attractive method of modifying the surface of natural fibers and making it more hydrophobic. It has been shown to reduce swelling of wood in water and has been studied more than any other chemical reaction of lignocellulosic materials. The main principle of the method is to react the hydroxyl groups (OH) of the fiber with acetyl groups (CH_3CO), therefore rendering the fiber surface more hydrophobic. The hydroxyl groups that react are those of the minor constituents of the fiber, i.e. lignin and hemicelluloses, and those of amorphous cellulose (Sjorstrom 1981). The hydroxyl groups in the crystalline regions of the fiber are closely packed with strong interchain bonding, and are inaccessible to chemical reagents. The acetylation of the —OH group in cellulose can be represented as below (Sjorstrom 1981).



$$\text{Fiber}-OH + CH_3CO-O-OC-CH_3 \xrightarrow[\text{conc:}H_2SO_4]{CH_3COOH} \text{Fiber}-O-CO-CH_3 + CH_3COOH$$

Acetylation has been shown to be beneficial in reducing moisture absorption of natural fibers. Reduction of about 50% of moisture uptake for acetylated jute fibers and of up to 65% for acetylated pine fibers has been reported in the literature (Bledzki and Gassan 1999). Acetylation has also been found to improve the interface bonding in flax/polypropylene composites (Zafeiropoulos et al.2002).

2.2.4 Matrix materials

The natural fibers can be used to reinforce both thermosetting and thermoplastic matrices. Thermosetting resins, such as epoxy, polyester, polyurethane, phenolic, etc.

are commonly used today in natural fiber composites, in which composites requiring higher performance applications. They provide sufficient mechanical properties, in particular stiffness and strength, at acceptably low price levels. Compared to compounds based on thermoplastic polymers, thermoset compounds have a superior thermal stability and lower water absorption. However, in the case of the demand for improved recycling and in combination with new long fiber reinforced thermoplastic (LFT) processing, thermoplastic polymers have been expected to substitute the thermoset polymers. Thermoplastic polymers are now becoming popular and widely used as matrix due to their processing advantages. Polypropylene (PP) possesses some outstanding properties like transparency, low density, toughness, surface hardness and sterilizability (Rana et al. 1998). PP is also reasonably economical. So, as a material of construction, jute-PP composite can play a vital role all over the world. The advantages and disadvantages of thermosetting and thermoplastic polymer are given below (Kry 2009).

Thermosets

- Chemical constitution of thermosets is similar to natural fibers.
- Production is lot simpler.
- Thermosets have a highly cross linked chain structure which does not allow the chains to slide and rotate easily.
- Chain structure gives thermosets high strength and good stuffiness properties but ductility reduces, with thermosets generally showing a brittle form of failure.
- Processing of thermoset composites is easy because the initial resin system is in the liquid state.

Thermoplastics

Thermoplastics are comprised of flexible liner chains. Thermoplastics offer many advantages over thermosets polymers. The advantage of the thermoplastic matrix composites are

- Processing cost is lower than thermosets.

- Simple methods such as extrusion injection molding are used for processing these composites.
- They are flexible, tough and exhibit good mechanical properties.
- They generally have a lower weight than conventional thermo sets resulting in a more straightforward and safer handing of the materials.
- Thermoplastics such as polypropylene have the lowest level of moisture absorption.
- Recycling is possible with thermoplastic reinforced natural fiber composites.

The only major disadvantage of using thermoplastics is that they have a different chemical structure compared to natural fibers as they have high non-polar nature, so often a method of improving the interfacial region between the two materials needs to be investigated. However, after assessing the limitation and the benefits of thermoplastic and thermoset in the present study, thermoplastic were chosen in preference to thermoset for natural fiber reinforcement.

Therefore, limited types of thermoplastic can be used in natural fiber composites which are polypropylene (PP) high-density polyethylene (HDPE), low density polyethylene (LDPE) and polyvinyl chloride (PVC) as these have lower melting temperature, generally less than 180°C.

Composites need to be lightweight and therefore require a thermoplastic matrix with low density. Density is often used for the calculation of specific properties which consequently gives better indication of achievable benefits with the material. Polypropylene and low- density polyethylene have the lowest densities and are most suitable for natural fiber. The major disadvantage of the composite is water absorption or migration through polymer to the fiber/matrix interface which results reduction in strength of composites. Polypropylene and low- density polyethylene are most desirable as they have the lowest level of moisture absorption.

2.2.4.1 Selection of polypropylene as matrix

Polypropylene is a linear hydrocarbon polymer. Polypropylene is commonly made from the monomer propylene by a Ziegler-Natta polymerization the results in an isotactic polymer, in which all the methyl groups are on the same side of the chain (Figure 2.9) (Doan 2007). Isotactic polypropylene has good mechanical properties as well as low density. It is a non-polar material. The crystallinity is about 60 - 70%.

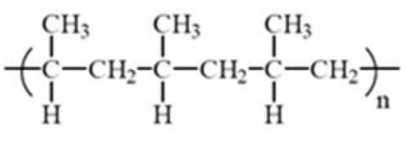


Figure 2.9: Chemical structure of isotactic polypropylene (Doan 2007).

Some typical properties of PP are given in Table 2.3 (Sultana et al. 2007). The melting point of commercial PP lies in the range of 160-170°C, with purified polymer reaching 176° C. Within the range of commercial polymers, the greater the amount of isotactic material the greater the crystalline and hence, greater the softening point, stiffness, tensile strength, modulus and hardness. PP confers good resistance to a wide range of aqueous and polar media, including emulsifier solutions with their strong stress cracking abilities due to their crystalline and nonpolar nature.

Table 2.3: Properties of polypropylene (Sultana et al. 2007)

Parameters	Values
Specific gravity	0.9
Crystallinity,%	60-70
Melting temperature, °C	165-171
Tensile strength, MPa	1.1-1.6
Elongation-to-break,%	100-600
Impact strength, notched lzod, J M^{-1}	21-53
Rockwell hardness (R-scale)	90-95
Heat-deflection temperature, °C, at 455 kPa (66 psi)	225-250

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 cannot stop the use of PP. An intermediate strategy may be taken which is to reduce the use of PP. Jute reinforced PP composite will serve not only the diversified application of wood 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.

2.3 Natural Fibre/Polymer Matrix Interface

A composite material is one in which two or more materials that are different (structure, properties) are combined to form a single structure with identifiable interfaces at multi-scales to achieve properties that are superior to those of its constituents. Fiber reinforced composite materials are composed of fibers and matrix material that binds and protects the fibers. Fiber to matrix adhesion is important in reinforcement. During loading, loads are applied directly to matrix not to the fibers. To achieve excellent mechanical properties, load must be transferred effectively from the matrix to fibers which require good interaction as well as adhesion (means strong and efficient fiber-matrix interface). The fibers are usually the load bearing members, while the polymeric matrix provides transverse integrity and transfers the load onto the fibers. This can be controlled by either surface treatment applied to the fiber or by the use of additives such as coupling agents.

Besides the properties of two main components of fiber and matrix, the fiber/matrix interphase also plays a crucial role for the load transfer. It is not a distinct phase, as the interface does not have a clear boundary. An interphase that is softer than the surrounding polymer would result in lower overall stiffness and strength, but greater resistance to fracture. Instead, an interphase that is stiffer than the surrounding polymer would give the composite less fracture resistance but make it very strong and stiff. The nature of the interface varies with the specific composite system (Jacob et al. 2005). Therefore, in order to design an optimal composite system, it is important to characterize the interface region, including its size, adhesion to the fiber and to the matrix and mechanical behavior (Bump 2001).

2.4 Fabrication Methods of Composites

All natural plant fibers usually have lower mechanical and thermal properties compared to conventional fibers such as glass fibers, carbon fibers and aramid fibers that are commonly used in high strength advanced composites (Netraveli and Chabba 2003). In addition, the performance of the natural fiber composites such as strength and rigidity is strongly dependent on their thermal histories, namely fabrication method and processing conditions, such as time, temperature, and pressure. As a result, it is critical that the fabrication process be selected such that the fiber properties are maintained and not degraded during processing. Generally, the molding temperature for natural fiber based composites is slightly lower than that for conventional glass fiber and carbon fiber reinforced polymer composites because of the lower thermal stability of natural plant fibers at high temperatures. A wide variety of techniques have been used for making natural fiber reinforced composites, for examples: (i) hot-press molding (ii) injection molding (iii) wet lay-up (iv) prepreg method, and (v) vacuum bag/ autoclave molding etc. Hot press molding technique is discussed in details, in the sequel.

Hot press molding

Hot press molding is a conventional and simple method to make fiber reinforced composites. This method is also called ‘compression’ molding method. It is widely used in a production line to obtain thick plate products and in laboratory research to fabricate various prototype composite materials (Lee 1999). Uses of molds allow production of composites with simple shape including curved surfaces. The prepreg is laid inside the mold by hand or mechanically and then hot pressed at a certain pressure in a compression molding machine. With the aid of pressure and heat, the resin is melted and immersed into the reinforcement and cured inside the mold. Compression molding technique drives out the trapped air, which is associated with the fiber bundles, by compressive pressure between the two heating plates.

2.5 Advantages and Disadvantages of Natural Fibers Based Polymer Composites

Advantages

Cellulose fibers possess several potential advantages, namely (i) low density (which makes it possible to obtain lighter composites), (ii) good mechanical properties, (iii) renewability, (iv) abundant at low cost and in a variety of forms (wood, annual agricultural residues, etc.), and (v) modest abrasive character, which ensures a greater longevity of the processing tools. Moreover, cellulose- based composites are easy to recycle at the end of their life cycle, either by reprocessing (thermoplastic matrices) or by energy recovery (combustion of thermoses composites), which does not leave any residue (Belgacem and Gandini 2009).

Disadvantages

The major drawback of cellulose fibers as reinforcement resides in their highly polar and hydrophilic character, which make them poorly compatible with commonly used non-polar matrices, such as polyolefin, and subject to loss of mechanical properties upon atmospheric moisture absorption (Belgacem and Gandini 2009). There are some other limitations to using plant fibers that must be considered in making composite materials. For instance, plant fibers can vary in quality and properties depending on factors such as source, age, processing techniques, climate and other growing conditions (Netraveli and Chabba 2003). Plant fibers become unstable and start to degrade when processed at temperatures above 200°C.

2.6 Applications of Natural Fiber Reinforced Polymer Composites

The leading geographic market in the use of natural fibers-based composites was Europe (Beckermann and Pickering 2008; Torres and Cubillas 2005). The Asian markets have been using natural fibers for many years. For example, jute is a common reinforcement in India. Eckert (2000) presented a statistical data of the major market for natural fibers in the year 2000 in building applications (Figure 2.10) (Bledzki et al. 2002; Karus et al. 2000), whereas other applications have been growing at an increasing rate, especially in the automotive industry. The natural fiber composites can be very cost effective material for following applications:

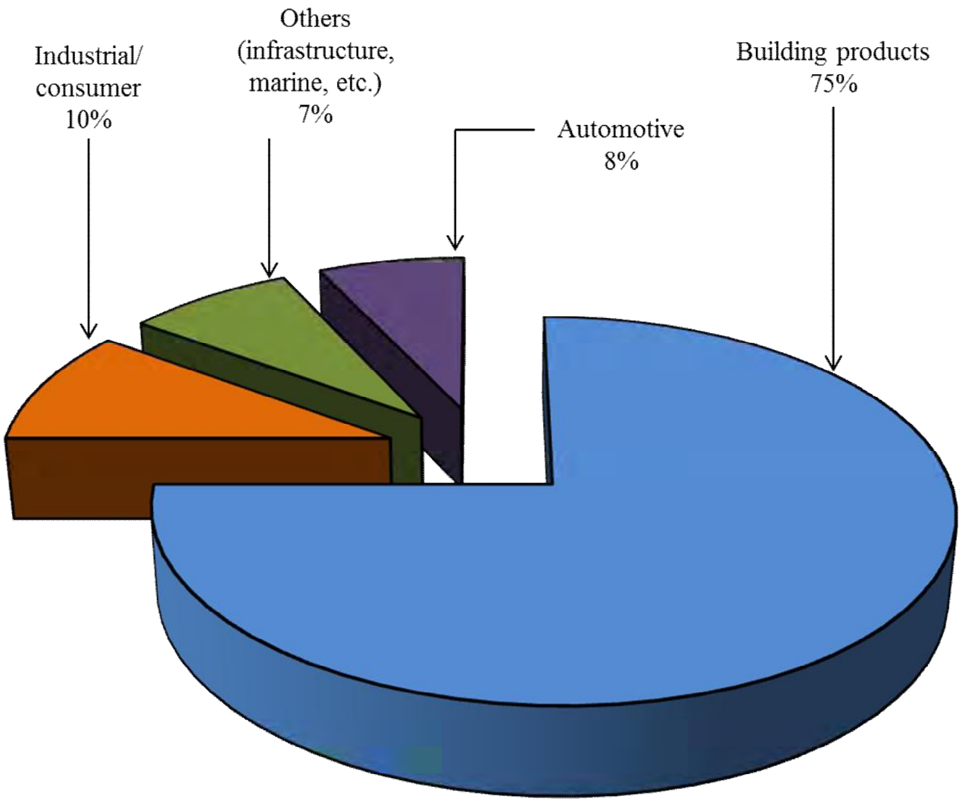


Figure 2.10: North American market demand of natural fiber composites (Bledzki et al. 2002; Karus et al. 2000).

- Building and construction industry: panels for partition and false ceiling, partition boards, wall, floor, window and door frames, roof tiles, mobile or pre-fabricated buildings which can be used in times of natural calamities such as floods, cyclones, earthquakes, etc.
- Storage devices: post-boxes, grain storage silos, bio-gas containers, etc.
- Furniture: chair, table, shower, bath units, etc.

- Electric devices: electrical appliances, pipes, etc.
- Everyday applications: lampshades, suitcases, helmets, etc.
- Transportation: automobile and railway coach interior, boat, etc.

2.7 Reactive Dyes

Dyes are substances capable of coloring fabrics in such a manner that the color cannot be removed by rubbing or washing. Reactive dyes are mostly used for dyeing cotton, and are fixed with cotton fibers forming covalent bonds at temperature ranging between 50-80^{o}C in alkaline environment. During dyeing, these dyes reacts with hydroxyl group of the fiber by either substitution or addition reaction. They react chemically with the fiber being dyed and if correctly applied, cannot be removed by washing or boiling. Mechanism of reactive dye fixation in the cellulosic fiber is shown in Figure 2.11 (Subramanian et al. 2006). The main feature of the dyestuff is its low affinity to cellulose; therefore large amounts of salt are required to force its deposition on the fabric. After this has been achieved, addition of alkali causes the deposited dyes to react with the fiber. Only a successfully concluded reaction guarantees a fast dyeing. During their application, along with dye absorption, dye hydrolysis also takes place.

Reactive dyes are extensively used in the textile industry because of their wide variety of color shades, high wet fastness profiles, simplicity of application, brilliant colors, and nominal energy consumption. The four characteristic features of an exceedingly water-soluble reactive dye molecule's typical structure are the chromophoric group, the reactive system, a bridging group, and one or more solubilizing groups (Shore 1995). They mainly include azo, anthraquinone, triphenodioxazine or copper phthalocyanine chromophores, which contribute to the color and much of the substantivity for cellulose fibers (Broadbent 2001). The reactive system enables the dye to covalently bond with the –OH group in cellulose, with –NH2, –OH or –SH groups in protein fibers and with the –NH2 group in polyamide. Important reactive systems can be classified into two distinct categories, depending on the mechanism of dye-fiber bond formation (nucleophilic substitution and nucleophilic addition) and on the stability of this bond to a subsequent treatment (monofunctional, homo- or hetero-bifunctional and polyfunctional). It is also well-known that during the application of reactive dyes to cellulose fibers under highly alkaline conditions, a competing hydrolysis reaction takes place, originating in the non-reactive oxi-dye form, which is lost for dyeing, and passes into the wastewater (Christie 2001). In all the above cases, only 60-65% dye utilization is attainable even with the use of salt in the normal dyeing systems (Subramanian et al. 2006).

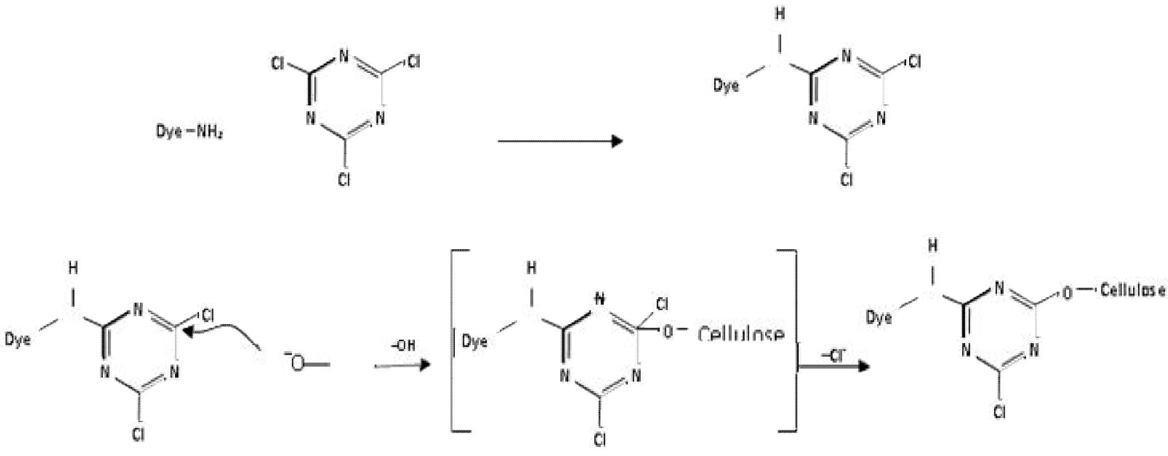


Figure 2.11: Mechanism of reactive dye fixation in the cellulosic fiber (Subramanian et al. 2006).

2.7.1 Coloration of jute fabrics with reactive dyes

Coloration of jute fabric has become essential for all sorts of fabric starting from decorative value added fabrics to attractive packaging material. Due to the eco-friendly and biodegradable nature of jute fiber, the demand for jute fabric is raising worldwide day by day. The coloration of jute fabric with reactive dyes is most suitable for its eco-friendly nature. A number of studies have been done on dyeing of jute with reactive dyes (Chattopadhyay et al. 2003; Bhuiyan et al. 2013; Arju et al. 2015a).

In theory, dyeability of jute fiber is almost similar to cotton, which is generally dyed with direct dyes or reactive dyes, but the results are not very good due to its high crystallinity and high degree of orientation (Liu et al. 2002). For this, many studies had been done to improve the dyeabilities of jute fiber, but most of them focused on the effect of pretreatments, namely scouring and bleaching and chemical compositions content on the dyeabilities of jute fiber (Chattopadhyay et al. 2002; Jeyakodi 2001). Although there is little effects on dye exhaustion and fixation, the environmental problems had not been relieved because the dosage of salt and alkali during the dyeing process had not diminished. Pretreated jute fabrics with the mixture of Albafix WFF and sodium hydroxide dyed with 1% Drimarene Red K8b only had 125% higher exhaustion, 25% higher fixation value than that obtained by normal dyeing, and exhibited good washing fastness as well (Arju et al. 2015a). Furthermore, Arju and Afsar (2016) observed that the dyeing of Glytac (with NaOH) and Indosol E-50 treated jute fibers ensured higher *K/S*, fixation, and better washing fastness and rendered the fiber surface more hydrophobic than those of raw jute fibers.

2.8 Review of the Previous Works

Natural fibers reinforced (NFR) composites are subject of many scientific and research projects as well as many commercial programs (Netraveli and Chabba 2003; Peijs 2003). The performances of NFR composites are influenced mainly by the adhesion between matrix and fiber. The main problem associated with NFR composites is the incompatibility between the hydrophilic natural fibers and the hydrophobic thermoplastic matrices. Matrix or fiber modification is therefore necessary to improve the compatibility between fiber and matrix. In this section, an attempt has been made to review the state of the art of natural fibers reinforced polyolefin composites.

Alkaline treatment

Alkaline treatment or mercerization is one of the most used chemical treatments of natural fibers when used to reinforce thermoplastics and thermosets. The important modification done by alkaline treatment is the disruption of hydrogen bonding in the network structure, thereby increasing surface roughness. This treatment removes a certain amount of lignin, wax and oils covering the external surface of the fiber cell wall, depolymerizes cellulose and exposes the short length crystallites (Mohanty et al. 2001). Addition of aqueous sodium hydroxide (NaOH) to natural fiber promotes the ionization of the hydroxyl group to the alkoxide (Agrawal et al. 2000). Thus, alkaline processing directly influences the cellulosic fibril, the degree of polymerization and the extraction of lignin and hemicellulosic compounds. In alkaline treatment, fibers are immersed in NaOH solution for a given period of time (Ray et al. 2001). Similar treatments were attempted by Morrison et al.2000 to treat flax fiber. They reported that 2% alkali solution for 90s at 200°C and 1.5 MPa pressure was suitable for degumming and defibrillation to individual fibers (Garcia-Jaldon et al.1998). These researchers observed that alkali led to an increase in amorphous for eight hours. The resulting changes in fibers were analyzed by density, Fourier infrared spectroscopic, X-ray, polarized microscopic and scanning electron microscopic techniques. The tensile strength, modulus and density were found maximum at four hours of alkali treatment. The crystallinity of fibers was observed only after eight hours of alkali treatment. The alkali treatment also increased the surface roughness of the fibers. Goda et al. (2006) with his research associates investigated the effect of tensile load application during mercerization of ramie fibers. They observed that mercerized fibers in 15% NaOH solution with load application improved tensile strength as well as fracture strain, as compared to that fiber without load application. Khan et al. (2004) prepared a series of composites with jute, mercerized jute, and man-made cellulose tyre cord yarn (Codenka). The matrix material was a polypropylene/ethylene block copolymer (PP), and a maleic acid anhydride grafted PP (MAPP) was used as a coupling agent. They observed that high strength and excellent impact properties were achieved with pure Cordenka reinforcement. Partial substitution of jute instead of Cordenka leads to enhanced stiffness properties of the composites. Razera and Frollini (2004) prepared composites based on phenolic matrices and jute fiber as reinforcement. Jute fiber was treated with alkali and ionized air. Among the fiber treatments, 5% NaOH solution presented the best results. The ionized air treatment produced an effect similar to the treatment with NaOH. But the combination of the two treatments (ionized air/ NaOH) decreased tensile strength and elongation. Liu and Dai (2007) used film stacking method to improve the mechanical properties of jute/polypropylene composite by treating jute fiber mat with sodium hydroxide and maleic anhydride grafted polypropylene (MAPP). They showed that combination of NaOH and MAPP emulsion is a good adhesion promoter for jute fiber mat/ PP composite.

Oxidation treatment

Oxidation is an effective method to bring about chemical as well as physical changes into the cellulosic materials. The chemical and physical properties of the oxidized products depend on the nature, conditions as well as solvents of oxidizing agents. Surface modification of jute fabrics with oxidizing agents were carried out by different authors (Kabir et al. 2010; Sultana et al. 2007; Tripathy et al. 2001) and the properties of the resulting composites were investigated. Kabir and Sultana with their associates studied on the oxidized jute fibers and mechanical properties of raw jute and oxidized jute based composites were measured. Jute fiber was oxidized with sodium periodate and post-treated with urea in order to increase the compatibility of the jute fiber with the polypropylene matrix. Post-treated jute fiber reinforced specimens yielded better mechanical properties compared to the oxidized and raw ones. Based on fiber loading, 30% fiber reinforced composites had the optimum set of mechanical properties. Tripathy et al. (2001) worked on the interfacial properties of four different forms of jute fibers (sliver, bleached, mercerized and untreated) and polyolefin matrices (LDPE and PP). The jute fibers were bleached with an oxidizing agent (0.7% $NaClO_2$ in aqueous solution) and then treated with 2% aqueous NaOH solution. The fiber-matrix was estimated by means of the critical fiber length and the stress transfer ability parameter. The parameters were obtained by single fiber composite tests. Thermal-mechanical characterization of the fibers was also carried out to evaluate the resistance to processing conditions. It was found that the adhesion was better in jute-PP composites than in LDPE-jute composites. In both cases, the results showed that the sliver jute and the untreated jute had better adhesion to both matrices than had the bleached and the mercerized fibers.

Permanganate treatment

Permanganate is a compound that contains permanganate group MnO_4^-. Permanganate treatment leads to the formation of cellulose radical (Frederick and Norman 2004). Most permanganate treatments are conducted by using potassium permanganate ($KMnO_4$) solution (in acetone) in different concentrations (0.033, 0.0625 and 0.125%) with soaking duration from 1 to 3 minutes after alkaline pre-treatment (Sreekala et al.2000; Sreekala et al. 2002). As a result of permanganate treatment, the hydrophilic tendency of the fibers was reduced, and thus, the water absorption of fiber-reinforced composite decreased. The hydrophilic tendency of fiber decreased as the $KMnO_4$ concentrations increased. But at higher $KMnO_4$ concentrations of 1%, degradation of cellulosic fiber occurred resulting in the formation of polar groups between fiber and matrix.

Peroxide treatment

Peroxide (ROOR) contains the divalent free radical ‘O-O’. Organic peroxides tend to decompose easily to free radicals of the form RO’. RO then reacts with the hydrogen group of the matrix and cellulose fibers. For example, the peroxide initiates free radical reaction between PE matrix and cellulose fibers. The free radicals of PE and cellulose thus formed generate covalent bond between them (Joseph et al. 1996; Paul et al.1997). Benzoyl peroxide (BP, $(C_6H_5CO)_2\ O_2$) and dicumyl peroxide (DCP, $C_6H_5C(CH_3)_2O)_2$) are mostly used in natural fiber surface modifications. In peroxide treatment, fibers are coated with BP or DCP in acetone solution for about 30 min after alkali pre-treatment. Peroxide solution concentration was 6% and saturated solutions of peroxide in acetone were used in the studies of Sreekala et al. (2000). They observed that high temperature was favored for the decomposition of the peroxide; peroxide-treated oil palm fiber-reinforced PF composites could withstand tensile stress to a higher strain level. Joseph et al. (1996) investigated BP and DCP treatment on short sisal fiber reinforced PE composites. The tensile strength values of composites increased with the increase in concentration of peroxide up to a certain level (4% for DCP and 6% for BP) and then remained constant. Khan et al. (2005) used peroxide induced photo-curing technique in preparation of 2-hydroxyethyl methacrylate (HEMA) treated jute fibers-polycarbonate composite. As a result of peroxide treatment, the hydrophilicity of the fibers deceased and the tensile properties of the composites increased (Khan et al. 2005).

Silane treatment

The effect of silane coupling agent on the performance of NFR composites resulted in better interfacial load transfer efficiency, because of the improved adhesion between fiber and matrix (George et al. 1998). Hydrogen and covalent bonding mechanisms could be found in the NF-silane system. The general chemical formula of silane is X_3-Si-R, a multifunctional molecule that reacts at one end (X) with the cellulose fiber surface and the other end, hydrocarbon chain (R) with the polymer phase. The aminopropyltrimethoxy silane with concentration of 1% in a solution of acetone and water (50/50 by volume) for 2h was reportedly used to modify the flax surface (Weyenberg et al. 2003). Rong et al. (2001) soaked sisal fiber in a solution of 2% aminosilane in 95% alcohol for 5 min at a pH value of 4.5-5.5 followed by 30 min air drying for hydrolyzing the coupling agent. Silane solutions in a water and ethanol mixture with concentration of 0.033% and 1% were also carried by Valadez-Gonzalez et al. (1999) and Agrawal et al. (2000) to treat henequen's fibers and oil palm fibers. It was verified that the interaction between the silane coupling agent modified fibers and the matrix was much stronger than that of alkaline treatment, which led to composites with higher tensile strength from silane treated than alkaline-treated fiber. Thermal stability of the composites was also improved after silane treatment.

Acetylation, Benzoylation and Isocyanate treatment

Acetylation of natural fibers is a well-known esterification method causing plasticization of cellulosic fibers. Chemical modification with acetic or propionic anhydride substitutes the hydroxyl groups of cellulose with acetyl groups, so that they become hydrophobic (Hill et al. 1998). Acetylation can increase the dimensional stability of composites as well as improve interfacial adhesion between fiber and matrix (Joseph et al. 2000). Mishra et al. (2003) and Nair et al. (2001) investigated the acetylating of sisal fibers. The treated surface of sisal fiber reportedly became very rough and had a number of voids that provided better mechanical interlocking with the polystyrene matrix. A hypothetical model of interface between the sisal fiber and PS composites has been postulated. The thermal stability of treated fiber composites was found to be higher than that of untreated fiber composite because of better thermal stability of treated fibers and improved fiber-matrix interactions in treated fiber composites. It was also reported that acetylated NFR polyester composites exhibited higher bio-resistance and less tensile strength loss compared to composites with silane treated fiber in biological tests (Khalil and Ismail 2000).

Benzoylation of fiber improves fiber matrix adhesion, thereby considerable increasing the strength of composite, decreasing its water absorption and improving its thermal stability. Benzoyl chloride is most often used in fiber treatment. NaOH and benzoyl chloride treated sisal fibers were analyzed and found that the thermal stability of treated composites were higher than that of untreated fiber composites (Joseph et al. 2000; Nair et al. 2001).

Isocyanate is highly susceptible to reaction with the hydroxyl groups of cellulose and lignin in fibers. Isocyanate is reported to work as a coupling agent used in fiber-reinforced composites. Paul et al. (1997) studied the chemical treatment of cardanol derivative of toluene disocyanate (CTDIC) in sisal fiber-LDPE composites. It was demonstrated that CTDIC reduced the hydrophilic nature of the sisal fiber, thereby enhancing the tensile properties of the sisal fiber-LDPE composites. Geore et al. (1996) treated pineapple leaf fiber with polymethylene-polyphenyl-isocyanate solution at 50°C for 30 min to improve the fiber-matrix interfacial adhesion. Sreekala et al. (2000) reported that TDIC treated oil palm fiber-PF composites had lower tensile strength and Young’s modulus than permanganate-, peroxide- and alkaline-treated fiber-PF composites, but had higher tensile strength and similar Young’s modulus with silane- and acrylic acid-treated fiber-PF composites.

In addition to the above researches of chemical modification of jute fibers, a new treatment procedure was introduced by Arju et al. (2014). Their treatment procedure also improved the adhesion between the treated fibers and polypropylene matrix which was verified by the improved tensile strength and modulus.

Maleated coupling agents

Maleic anhydride grafted polypropylene (MAPP) has been widely used as a coupling agent in NFR polypropylene composites. The treatment of NFs with MAPP copolymer provides covalent bonds across the interface. Through such a treatment, the surface energy of fibers is increased to a level much closer to the surface energy of the matrix. This results in better wettability and higher interfacial adhesion of the fiber (Joseph et al. 2003).

Mohanty et al. (2004) used MAPP as coupling agent for the surface modification of jute fibers. It has been found that a 30% fiber loading with 0.5% MAPP concentration in toluene and 5 min impregnation time with 6 mm average fiber lengths gave the best results. A 72% increase in flexural strength was observed with the treated fibers. In addition to PP matrix, Mishra et al. (2000) reported that maleic anhydride treatment reduced the water absorption to a great extent in banana, hemp and sisal fiber-reinforced novolac composites. Mechanical properties like Young's modulus, flexural modulus, hardness and impact strength of plant fiber-reinforced composites increased after maleic anhydride treatment. Mohanty and Nayak (2006) worked on the mechanical and dynamic mechanical behavior of sisal fiber reinforced HDPE composites. The composites treated with MAPE showed improved properties in comparison with the untreated ones. Dynamic mechanical analysis data showed an increase in the storage modulus of the treated composites. The thermal behavior of the composites studied by TGA and DTG techniques showed an increase in the thermal stability of HDPE matrix with fiber reinforcement and MAPE treatment. Doan et al. (2007) prepared jute/polypropylene composites and studied the thermal, dynamic mechanical and aging behavior of the resulting composites. The investigations were carried out with or without matrix modifier (maleic anhydride grafted polypropylene). Thermal study revealed that thermal stability of the composites was found higher than that of either neat PP or the fiber in both nitrogen and air. Another important finding was that the thermal degradation of PP in air was easier and faster compared to that in nitrogen. They also observed that the thermal stability of the composites with modified fibers was higher than that of composites with non-modified fibers. The modified PP composites absorbed less moisture than unmodified ones. The storage modulus of matrix modified composites showed an improved value compared with non-modified polypropylene at the same fiber content, for as prepared and for aged samples. Joseph and co-workers (2003) studied the thermal and crystallization behavior of sisal/PP composites by thermogravimetry (TG), differential scanning calorimetry (DSC), and polarizing optical microscopy. Sisal fiber was chemically modified with a urethane derivative of polypropylene glycol (PPG), maleic anhydride modified polypropylene (MAPP), and KMnO4 in order to improve the interfacial adhesion between the fiber and matrix. The TG analysis showed that the thermal stability of treated fiber composites was higher compared to untreated system. DSC measurements exhibited an increase in the crystallization temperature and crystallinity upon the addition of fiber to the PP matrix. On increasing the fiber content, the melting peak of the PP component was shifted to higher temperatures suggesting a constrained melting. The researchers suggested that the thickness of the transcrystalline layer formed depended on crystallization temperature and time. The transcrystalline growth rate was slow in the quiescent state. On the other hand, upon the application of stress, transcrystallinity developed quickly. The shear stress at the polymer/fiber interface initiated the nucleation. Fiber surface modification by PPG increased the nucleating ability of sisal fibers to a very small extent.

Photo grafting and photo curing

Photo grafting is a novel technique applied for the surface modifications of NFs by means of radiation source. Radiation is a very convenient method for graft initiation because it allows a considerable degree of control of structural factors of the polymer by careful selection of the dose and dose rate. Photo grafting of different monomers on to cellulosic fibers and polymers improves their characteristics (Khan and Hassan 2004). Jute yarn was modified by photo grafting and photo curing with acrylic monomers (AM). AM was grafted onto jute yarn by UV radiation in order to improve the physico-mechanical properties of jute fibers (Hassan et al. 2003a; Hassan et al 2003b; Hassan et al. 2003c). The effect of radiation time, concentration of monomer on polymer loading (PL) and mechanical properties like tensile strength and elongation at break of the jute yarns were studied. The highest PL (22%) and enhanced tensile strength (95%) of the yarn were observed when the jute yarn was treated in 30% AM in methanol followed by UV radiation for 60 minutes. Jute yarns were also grafted with different types of vinyl monomer such as methyl acrylate (MA), ethylcry1ate (EA), and 2-hydroxyethylacrylate (HEA) with UV radiation (Swapan et al.2003). MA, EA, and HEA produced enhanced tensile strength of 87, 78, and 85%, respectively.

In another investigation, the physico-mechanical properties of jute yarns were improved by UV radiation through graft co-polymerization using a single impregnating monomer 1,6-hexanedioldiacrylate (HDDA) (Khan et al. 2004). Jute yarns demonstrated the enhanced tensile strength (67%) and PL (11%) are achieved with 5% HDDA concentration, 5 minutes soaking time and 4th pass of UV radiation. Coir fibers were also modified with HDDA (Khan et al.2006a) and ethyleneglycoldimethylacrylate (EGDMA) (Khan et al. 2006b) under UV radiation. Alkoxy silane monomer such as 3-(trimethoxysilyl) propylmethacrylate was used to modify jute yarns under UV radiation (Hassan et al. 2003d). Silane concentration in methanol was 5 to 50% and irradiation period ranged from 15 to 300 minutes. The highest PL value (32%) was found at 120 minutes irradiation time and 50% silane concentration. The highest tensile factor was 2.6, which was obtained at 30 minutes irradiation time, 30% silane concentration and 26% PL. However, at highest PL the tensile factor value was 1.91.

Radiation effect on natural fiber reinforced composites (NFR)

A good number of works has been done by the authors (Zaman et al. 2009; Zaman et al. 2010b; Khan et al. 2009) to study the effect of UV and gamma radiations on the properties of NFR composites. Khan et al (2009) prepared various types of jute fiber-PP composites by combination of untreated and pre-treated jute fiber and PP matrix with the aid of gamma-irradiation. Composite prepared with irradiated samples showed the better mechanical properties over the other combinations and irradiation of 500 krad produced the best results.

Urethane acrylate treated jute based composites were prepared using urethane acrylate oligomer (M-1200) in methanol solvent along with photoinitiator (Darocur- 1173) followed by UV radiation (Zaman et al. 2010b). Jute fabrics treated with 70% oligomer, 28% methanol, arid 2% photo initiator followed by UV radiation produced the best mechanical properties of the composites. For further improvement of the mechanical properties of the composites, jute fabrics were first treated with different percentages of methyl methacrylate (MMA) in methanol with photo initiator and cured under UV radiation and composites were fabricated by the oligomer. Urea was incorporated into the MMA formulation and was found to have the potential to improve the mechanical properties of the composites.

In another investigation, Khan et al. (2009) investigated bulk composites and PP sheets under gamma radiation of various doses (250-1000 krad) at the rate of 600 krad/h. It was found that gamma radiation of 500 krad showed better mechanical properties than that of other doses.

Khan et al. (2009) also carried out a comparative study between gamma and UV radiation on the properties of jute based composites. Mechanical properties of irradiated jute fabrics and matrices based composites were found to increase significantly.

Optimized jute fabrics were treated with starch solution of different concentrations for different soaking time and composite made of 0.5% (for UV) and 0.3% (for gamma) starch treated jute fabrics (5 minutes soaking time) showed the best mechanical properties.

Apart from the modification of jute fabrics mentioned above, some authors have also paid attention to the effects of jute fabric structures on their composites. However, only woven structures of jute fabrics were considered to observe the properties of composites. From the comparison of the properties of nonwoven and woven fabric composites, it was observed by Sengupta et al. (2008) that the composites with nonwoven fabrics showed better properties than that of woven fabrics. Araujo et al. (2008) observed the effects of weft knitted structure of cotton fabric on polymer matrix composite and reported improved properties of the composites. In most cases, these researchers used jute in the matrix as a fiber form (Haydar et al. 2012b; Goda et al. 2006 Juganta et al. 2012; Hongqin et al. 2003) or in few cases (Haydaruzzaman et al. 2010 Khan et al. 2005) they used plain woven fabrics. They have not used jute fabrics of other structures, specially knit structure. Recently Arju et al. (2015b) investigated the effects of different fabric structures on the performance of resulting PP composites. They found that composites having twill structure fabrics had the highest value of tensile strength, which was 40% higher than that of composites having plain structure fabrics. Again, water uptake of composites having rib structure fabrics was found to be 58% less than that of the composites having plain structure fabrics.

CHAPTER 3

EXPERIMENTAL

This section is divided into two parts. In the first part, different chemical treatments and test procedures of jute fabrics are discussed for the evaluation of their properties. In the second part, test procedures of composites have been described to evaluate the performance of composites.

3.1 Chemical Treatments of Jute Fabrics

3.1.1 Materials

3.1.1.1 Jute fabrics

For the purpose of selecting the best treatment procedures, 23 different treatments are performed on the plain jute fabrics only. Instead of manufacturing of the plain jute fabrics, these were collected from Babu Bazar, Dhaka, Bangladesh. The specifications of the plain jute fabrics were 10-12 yarns per inch in both the directions (warp and weft) and 279N fabric strength. The specifications of yarns from which the above plain fabrics were manufactured were- number of twist per inch: 4-5, yarn count: 241 tex. It is noted that the same yarns were used in both the directions (warp and weft) of fabrics. After selecting the most effective treatment procedure among 23 treatment procedures, it was applied to the jute fabrics of other different structures including the plain fabrics as well. This time, jute fabrics of different structures were manufactured in the laboratory from the yarns which were collected from New Dhaka Jute Industries Ltd., Kachpur, Dhaka. The specifications of the yarns were the same as mention above, i.e. number of twist per inch: 4-5, yarn count: 241 tex.

3.1.1.2 Chemicals

Albafix WFF, Glytac, Indosol E-50, and Drimarene Red K-8b (reactive dye) were used as modification reagents. Albafix WFF from Huntsman, United States of America, Glytac from Sigma-Aldrich Comp., 3050 Spruce State, ST Louise MO 63103, U.S.A., Indosol E-50 and Drimarene Red K-8b from Clariant, Switzerland and Ecal soap PA (nonionic detergent) from Thailand were bought for the purpose of chemical treatments in this study. In addition, Sodium hydroxide (NaOH), sodium carbonate (Na_2CO_3), and sodium chloride (NaCl) from BDH, (UK) were also used in the present study.

3.1.2 Methods

Jute fabrics were chemically modified with the cationic agents, such as Albafix WFF, Glytac, and Indosol-E50 in the presence and absence of reactive dye with or without sodium chloride (salt), sodium carbonate (soda ash), and sodium hydroxide. To select the most effective chemical treatments, 23 different treatment procedures were followed at optimum chemical concentration to treat the plain jute fabrics as shown in Table 1. The first column of Table 3.1 shows the five major groups of chemicals, which are A, AS, G, GS, and I. These five groups, respectively, represents Albafix WFF, mixture of Albafix WFF and NaOH, Glytac, mixture of Glytac and NaOH, and Indosol E-50. Each group has four or five different treatment procedures. The group A has five different procedures in all of which Albafix WFF is present. Similarly, group AS have four different procedures in all of which mixture of Albafix WFF and NaOH is present. The other groups have similar meanings. All the treatment procedures were carried out with the concentration of Albafix WFF 10g/l, Glytac- 60 g/l, Indosol E-50 4g/l, NaOH - 20g/l, NaCl - 40g/l, and Na_2CO_3 - 10g/l. This concentration of chemicals was selected based on the optimization of jute fabrics characteristics in which jute fabrics were treated by the chemicals of different concentrations and evaluated. The concentration was varied from 10-30g/l for Albafix WFF, 10-70g/l for Glytac, and 1-4g/l for Indosol E-50. Also, 1% Drimarene Red K-8b was used in every procedure. Besides these samples, untreated plain jute fabrics were treated with normal dyeing method, i.e. jute fabrics were treated with the mixture of 1% Drimarene Red K8b, sodium chloride (40g/l) and sodium carbonate (10g/l) at 60°C temperature for 60 minutes. Another treatment (S) in which jute fabrics were treated with 4% sodium hydroxide at 60°C temperature for 60 minutes. All the treatments of Table 3.1 were carried out in a sealed steel container in a laboratory dyeing machine Rota Dyer with the material to liquor ratio of 1:15 and at the temperature of 60°C for 60 minutes. The fabrics were then taken out and washed with water at least three times in order to remove any residual chemicals so that a final pH value of 7 was maintained and then dried in an open air. For wash fastness test, fabrics were washed by placing them in a mixture of Ecal soap – PA (0.50

gm/l) and water at temperature of 95°C for 10 minutes. Afterwards fabrics were hot washed at 80°C for 10 minutes. Finally, fabrics were washed with normal water repeatedly until the pH value of 7 was obtained.

Table 3.1: Different treatment procedures with various chemicals

Chemical group	No. of treatment procedure				
	1	2	3	4	5
A	Treatment with Albafix WFF	Treatment with Albafix WFF + dye	Treatment with Albafix WFF + dye +NaCl +Na_2CO_3	Treatment of A1 treated sample with dye	Treatment of A1 treated sample with dye + NaCl + Na_2CO_3
AS	Treatment with Albafix WFF + NaOH	Treatment with Albafix WFF + NaOH + dye	--	Treatment of AS1 treated sample with dye	Treatment of AS1 treated sample with dye + NaCl + Na_2CO_3
G	Treatment with Glytac	Treatment with Glytac + dye	Treatment with Glytac + dye + NaCl + Na_2CO_3	Treatment of G1 treated sample with dye	Treatment of G1 treated sample with dye + NaCl + Na_2CO_3
GS	Treatment with Glytac + NaOH	Treatment with Glytac + NaOH + dye	--	Treatment of GS1 treated sample with dye	Treatment of GS1 treated sample with dye + NaCl + Na_2CO_3
I	Treatment with Indosol E-50	Treatment with Indosol E-50 + dye	Treatment with Indosol E-50 + dye +NaCl +Na_2CO_3	Treatment of I1 treated sample with dye	Treatment of I1 treated sample with dye+ NaCl + Na_2CO_3

3.1.3 Evaluation of jute fabrics

3.1.3.1 Water absorbency

AATCC/ASTM Test Method TS-018 procedure was followed for water absorbency test of jute fabrics. This test method is designed to measure the water absorbency of jute fabrics by measuring the time required for a drop of water to be completely absorbed by the jute fabric surface. To carry out the water absorbency test, sample is placed over the top of a beaker so that the center is unsupported as shown in Figure 3.1. Then a measured drop of water is placed on the fabrics surface from 1 cm above and time is recorded until the water drop is absorbed completely.

Figure 3.1: Water absorbency test.

3.1.3.2 Fabric strength

ASTM D5035 300 / BS EN ISO 13934-1 test Method was followed for tensile test of jute fabrics. Tensile test of jute fabrics was conducted in length wise direction. There are two usual methods of testing the fabric strength: grab and strip. In grab method, full width of the fabrics is considered for applying the load. In strip method, a sample is cut with a specified width for applying the load. The strip method was used in this study for evaluating the strength of fabrics. Ten samples were prepared and tested using a universal testing machine (model H50 KS-0404, Hounsfield Series S, UK). The load capacity was 1000 N. Fabric strength is a measure of physical force of textiles.

3.1.3.3 Testing of exhaustion

Dyeing exhaustion means how much dye is taken up substantially by the textiles from the dye bath solution. The optical density of the dye solution before and after the dyeing was measured using Shimadzu UV- spectrophotometer (UV-1800, Shimadzu, Japan) at the maximum wavelength of absorbency (λ max) was determined. The dye bath exhaustion percentage (% *E*) was calculated using the following expression.

$$E = \frac{(A_0 - A_1)}{A_0} \times 100\% \tag{3.1}$$

where A_0 = the absorbency at maximum wavelength (λ max) of dye originally in the dye bath.

A_1 = the absorbency at maximum wavelength (λ max) of residual dye after dyeing.

3.1.3.4 Color strength

The color strength or depth of the color was evaluated in terms of *K/S* values. Higher *K/S* value indicates the higher dye uptake or depth of color of the fabrics. The reflectance values of dyed fabrics at all wavelengths were measured by using a Minolta spectrophotometer. The reflectance (*R*) value of dyed fabrics at the maximum wavelength of absorbency (λ max) is found and color strength (*K/S*) is calculated using the built-in software of the computer color matching system. These values are calculated using the following Kubelka Munk equation.

$$K / S = \frac{(1-R)^2}{2R} \tag{3.2}$$

where K = the absorption coefficient of the dyed sample at the wavelength of maximum absorption.

S = the scattering coefficient of the dyed sample at the wavelength of maximum absorption.

3.1.3.5 Determination of fixation

Fixation of dye means that the dye is completely fixed on the dyed fabrics and no more dye can go on to the other fabrics. The percentage of dye fixation (% *F*) was calculated using the following equation.

$$F = \frac{(K/S)_a}{(K/S_b)} \times 100\% \tag{3.3}$$

where $(K/S)_a$ = the color strength with the values after soaping

$(K/S)_b$ = the color strength with the values before soaping.

3.1.3.6 Determination of total dye utilization (%*T*)

The total dye utilization percentage (%*T*) was calculated using the following equation.

$$T = \frac{E \times F}{100}\% \tag{3.4}$$

where E= exhaustion values and F= Fixation values.

3.1.3.7 Testing of wash fastness

Color fastness is the property of a dye to retain its color when the dyed (or printed) textile material is exposed to conditions or agents such as light, perspiration, atmospheric gases, or washing that can remove or destroy the color. Wash fastness means resistance of the material's color to fading by washing. Degree of fastness of color is tested by standard procedures. Textile materials often must meet certain fastness specifications for a particular use. The wash fastness of textile dye is categorized from one to five and the higher the number the better fastness is obtained. The change in color and degree of staining was evaluated visually using geometric grey scale. ISO standards 105-C02 method was followed for wash fatness test. A specimen of 10 × 4 cm was attached with a multifiber fabric strip. Washing solution containing 5g/l soap was taken in the laboratory dyeing machine with a liquor ratio of 1:50. The specimen was treated for 45 minutes at 50 ± 2^{o}C. The specimen was then removed and rinsed in normal water and dried in shadow.

3.1.3.8 FTIR spectroscopy

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. FTIR spectrum is an important record which gives sufficient information about the structure of a compound. An FTIR spectrometer simultaneously collects spectral data in a wide spectral range. The most important region for an organic chemistry is 2.5μm to 15μm in which molecular vibration can be detected and measured in a spectrum.

A schematic absorption spectrum is given in Fig. 3.2. The absorption spectrum is a plot of absorption of energy (radiation) against its wavelength (λ).

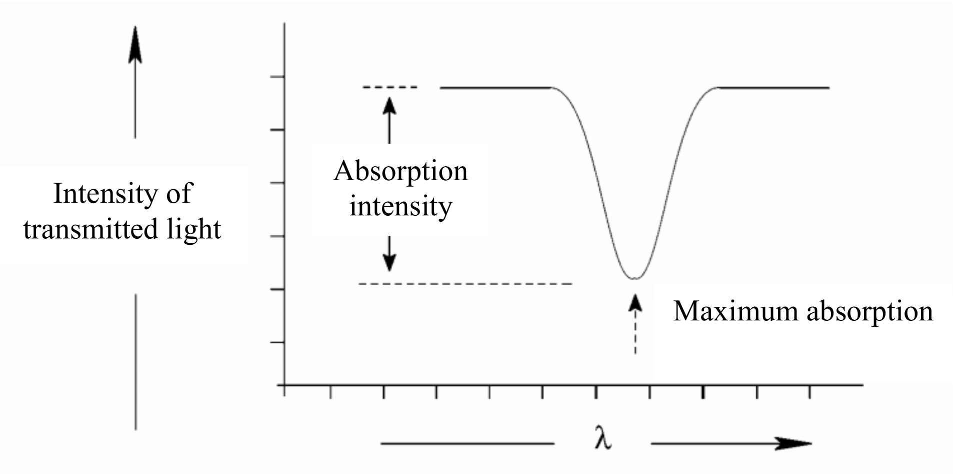


Figure 3.2: Schematic diagram of absorption spectrum.

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 (μm) or, in frequency-related wave numbers (cm^{-1}) which are reciprocals of wavelengths as shown in the following equation.

$$\text{Wave number, } \upsilon\,(\text{cm}^{-1}) = \frac{1\times10^{4}}{\text{wave length (in } \mu\text{m)}} \qquad (3.5)$$

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 untreated jute fibers and chemically treated jute fibers were recorded by an IR Prestige – 21, Shimadzu, Fourier Transform Infrared Spectroscopy (FTIR) instrument at a resolution of 2 cm^{-1}. Average of 30 scans was recorded in absorbance units from 4000 to 400 cm^{-1}.

3.2 Test of Jute Fabrics/PP Composites

3.2.1 Materials

Jute yarns: Untreated jute yarns (Figure 3.3) were collected from New Dhaka Jute Industries to prepare jute fabrics of different structures. Specifications of these yarns were - no. of twist per inch: 4-5, yarn count: 241 tex, strength: about 3.7 kg.

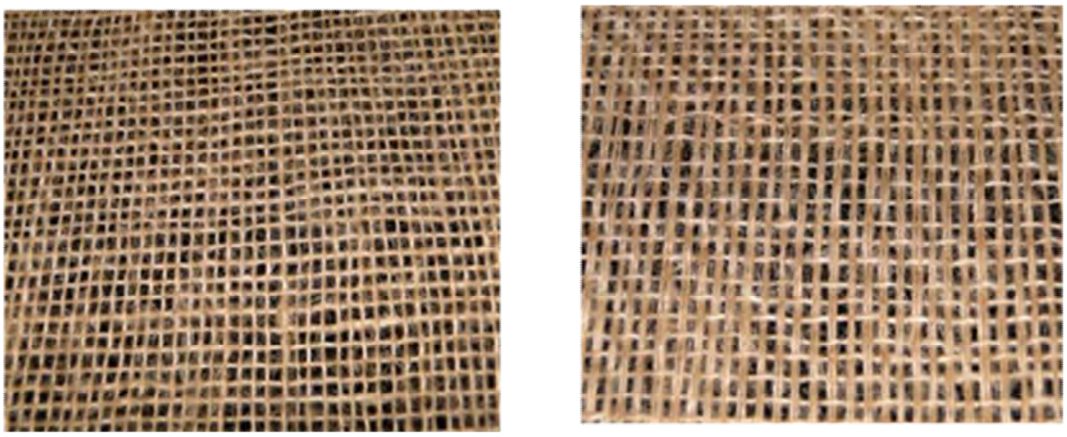
Jute fabrics: Fabrics of four different structures, namely plain, twill, single jersey, and rib fabrics were manufactured as shown in Figure 3.4. In all the cases, above mentioned yarns were used. Parameters of jute fabrics of different structures are shown in Table 3.2. Composites were manufactured with both the treated and untreated jute fabrics.

Table 3.2: Parameters of jute fabrics of different structures

Fabric	Structure	Count of yarn		No of yarn per inch	
		Warp (Tex)	Weft (Tex)	Warp	Weft way
Woven	1/1(plain)	241	241	10-12	10-12
	2/1(twill)	241	241	18-20	9-10
Knitted	single jersey	241	241	9-10	9-10
	rib	241	241	8-10	8-10

Polypropylene: Polypropylene (PP) granules from the polyolefin company SABIC (Saudi Basic Industries Corporation) Riyadh, Saudi Arabia were used as received. In this study, isotactic polypropylene (PP) was used as a matrix material. It had a melt flow rate of 8 g/min and melting point of 165^{o}C. Melting point observations indicated that the used PP was an isotactic 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^{o}C but pure PP melts at 176 ^{o}C (Whiteley et al. 2006).

Figure 3.3: Jute yarns which were used to prepare the jute fabrics of different structures.



(a) Plain

(b) Twill

(c) Single jersey

(d) Rib

Figure 3.4: Close view of plain, twill, single jersey, and rib jute fabrics.

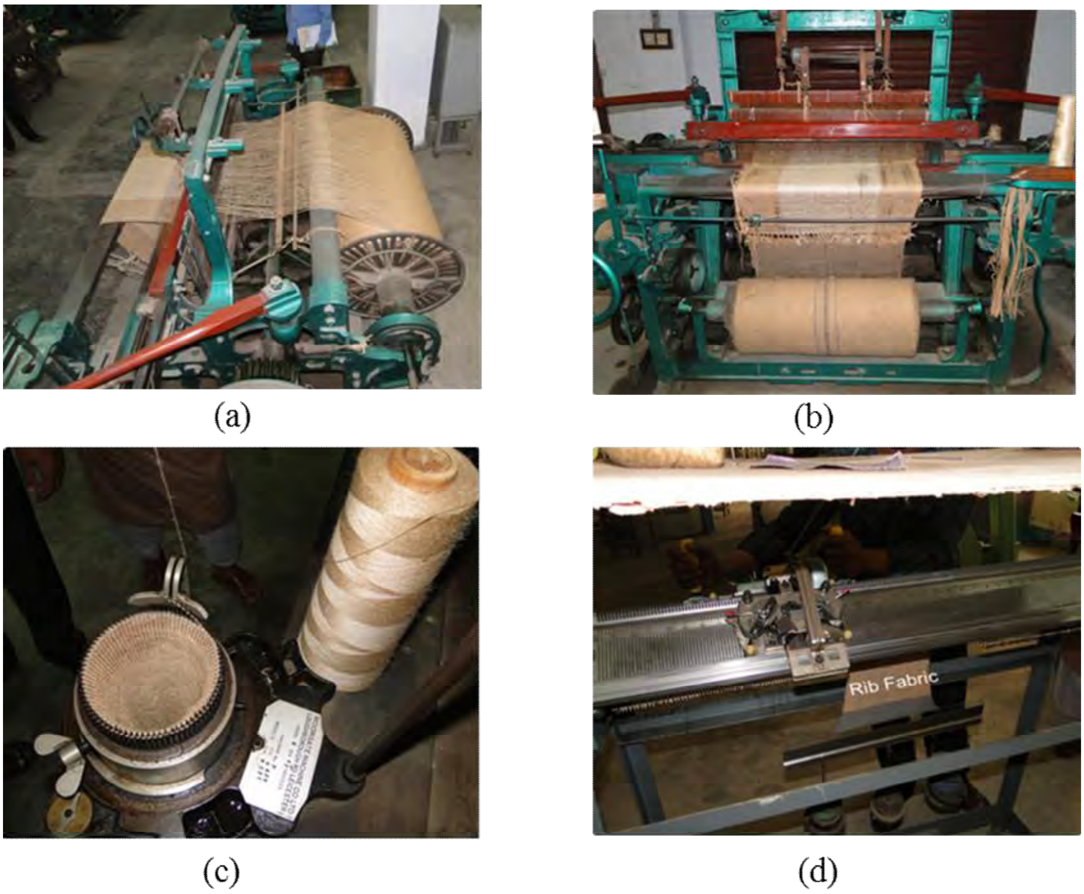

(a) (b) (c) (d)

Figure 3.5: Picture of a (a) loom used for plain fabrics, (b) loom used for twill fabrics, (c) socks circular knitting machine for single jersey fabrics, (d) hand operated flat/ v bed knitting machine for rib fabrics.

3.2.2 Sample preparation

3.2.2.1 Preparation of jute fabrics

Fabric samples were prepared with above mentioned yarns in the shed of Bangladesh University of Textiles (BUTex). For woven fabrics, conventional jute loom and for knitted fabrics, Hand Operated Socks Circular and Hand Operated Flat/ V bed Knitting machines were used (Figure 3.5). Woven fabrics with two different structures such as 1/1 (plain) and 2/1(twill) and also two different structures such as single jersey and rib of knitted fabrics were manufactured as shown in Table 3.2. For knitted fabrics, weft knitted of 1×1 structures were used as the preform for the composites.

Figure 3.6: Extruder (Lab Tech Engineering Company Ltd. (USA)) machine which was used to produce polypropylene sheet.

Figure 3.7: External appearance of (a) polypropylene chips and (b) polypropylene sheet.

3.2.2.2 Preparation of PP sheets

Granules of polypropylene (PP) were made into thin sheets (0.1 - 0.4 mm thickness) using an extruder, Lab Tech Engineering Company Ltd. (USA) (Figure 3.6). Polypropylene chips and polypropylene sheet are shown in Figure 3.7.

3.2.2.3 Fabrication of composites

The PP sheets were cut into small pieces (18×18 cm) and kept in the polyethylene bag until composite fabrication. Carver Laboratory press was used to prepare composites. Jute fabrics were cut into small pieces of 18×18 cm dimension and then placed between two steel plates under normal pressure at 90°C temperature to remove moisture. Composites were prepared by sandwiching one layers of jute fabrics (55±1%) between two layers of pre-weighted PP sheets as shown in Figure 3.8 and pressed at 190°C for 10 min between two steel plates under a pressure of 5×10^{8} N/m^{2} (Khan et al. 2008; Haydaruzzaman et al. 2010) in the Carver Laboratory press machine (Figure 3.9). Then composite containing steel mold was cooled to room temperature using another press (Carver, USA) and cut to the desired size.

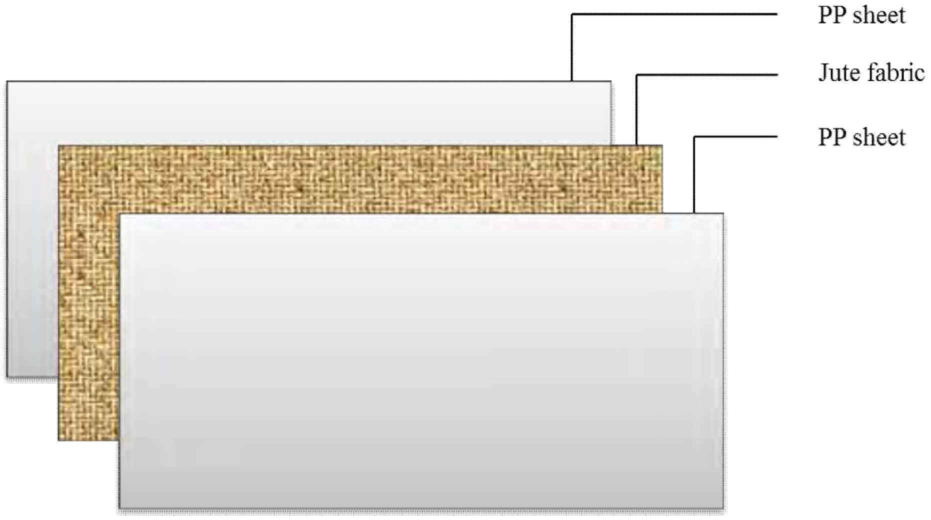


Figure 3.8: Preparation of composite sample.

3.2.3. Mechanical properties of composites

In this study, tensile, impact, and flexural tests were done for jute fabrics/PP composites in view of studying the effects of different treatment procedures and different fabric structures on the performance of resulting composites. Test standards, set-up of the tests, operating conditions, and dimensions of the test specimens are described below.

Figure 3.9: Carver (USA) Laboratory press machine.

3.2.3.1 Tensile properties

Among various mechanical properties of plastic as well as composite materials, tensile properties are probably the most frequently considered, evaluated, and used throughout the industry. These properties are an important indicator of the material's behavior under loading condition. Tensile testing provides these useful data: tensile strength at break (ultimate tensile strength), elongation at break, tensile modulus (Young's modulus). The tensile strength (σ_u) is given by

$$\sigma_u = \frac{F}{bh} \tag{3.6}$$

where F = maximum load,

b = width of the sample,

h = thickness of the sample.

Strain is defined as

$$\varepsilon = \frac{\Delta l}{l_0} \tag{3.7}$$

where Δl = elongation at break,

l_0 = initial gauge length.

The Young's modulus in tension is the slope of the stress vs. strain curve evaluated at small strains, where the response is linear.

Tensile tests were performed according to ASTM D 638 with a universal testing machine (model H50 KS-0404, Hounsfield Series S, UK) at a crosshead speed of 5 mm/min and each test was performed until the failure of the specimen. All the results were taken as the average values of at least five samples. Dog-bone shaped/Dumbbell-shaped composite specimens were used as a test specimen. Dimensions of tensile test specimens are shown in Figure. 3.10. Dimensions of the specimens were: W = 19 mm, G = 50 mm, D = 115 mm, L_0 = 165 mm, b = 20 mm, and h = thickness of the specimen in mm. The tensile strength, Young's modulus and elongation at break were calculated automatically by the QMAT software in a computer interfaced with the Hounsfield UTM machine when the tests were being performed on the composite specimens.

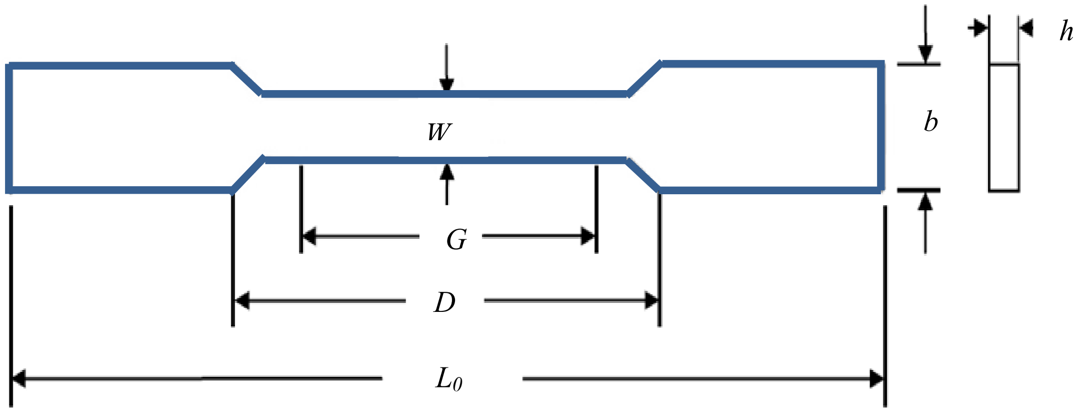


Figure 3.10: Dimensions of tensile test specimen.

3.2.3.2 Flexural properties

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 Figure 3.11. Different dimensions of flexural test specimen are: L=100 mm, b =10 mm, and h =4.1mm. Flexural tests were conducted following the ASTM D 790-00 standard at a cross head speed determined by the following equation.

$$R = ZS^2 / 6h \tag{3.8}$$

where R = rate of cross head motion = 1.749 mm/ min,
h = thickness = 4.1 mm,
S = support span = 16h = 16×4.1mm = 65.6 mm,
Z = rate constant of straining of the outer fiber = 0.01.

To perform the flexural test, 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 (σ_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 σ_f and flexural modulus E_b, respectively.

Flexural strength, $\sigma_f = 3FS / 2bh^2$ (3.9)

Flexural modulus, $E_b = S^3 m / 4bh^3$ (3.10)

where F = maximum load in N,
m = slope of the tangent to the initial straight portion of the load-deflection curve in N/mm.

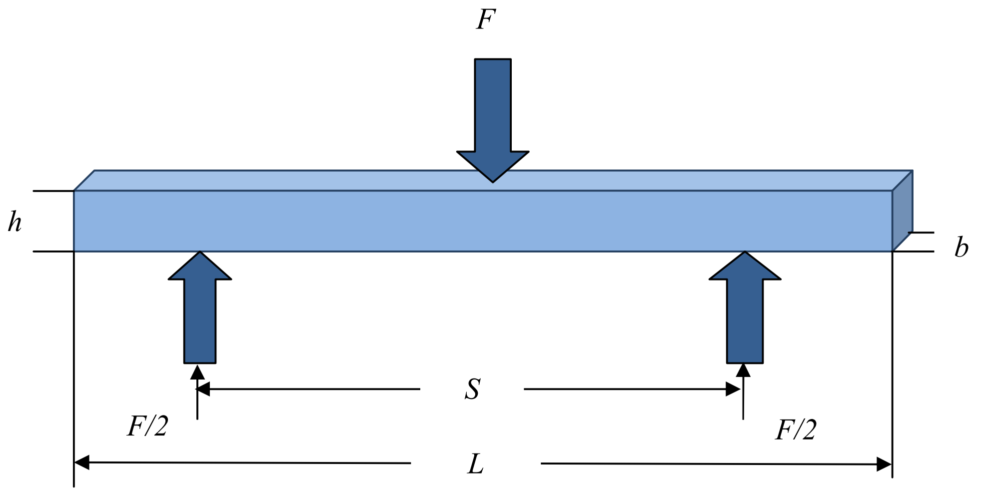


Figure 3.11: Set-up of three point flexural test and dimension of specimen.

3.2.3.3 Impact strength

The impact strength of the composites was determined according to ISO 179/1fU by the impact testing machine (HT-8041B Izod, Pendulum type, Hung Ta Instrument Co. Ltd.) as shown in Figure 3.12. Figure 3.13 shows the test specimen of jute fabrics reinforced polypropylene composites. The dimension of test specimen for the Izod impact test was 80×10×4 mm without notch. Unnotched impact strength (a_{cU}) is calculated by the following equation:

$$a_{cU} = \frac{W}{hb} \times 10^3 \ \text{kJ/m}^2 \tag{3.11}$$

where W = energy in N-m (Joul),

b = width in mm,

h = thickness in mm.

Figure 3.12: The impact tester (HT- 8041B Izod, Pendulum type).

Figure 3.13: Test specimens of PP composites of different fabrics (P- plain, T- twill, S- single jersey, and R- rib) for impact test.

3.2.4 Scanning Electron Microscopy (SEM)

Scanning electron microscopy (SEM) is a method for high-resolution imaging of surfaces. The SEM is an instrument that produces a largely magnified image by using electrons instead of light to form an image. A beam of electrons is produced at the top of the microscope by an electron gun. The electron beam follows a vertical path through the microscope, which is held within a vacuum. The beam travels through electromagnetic fields and lenses, which focus the beam down toward the sample. Once the beam hits the sample, electrons and X-rays are ejected from the sample. Detectors collect these X-rays, backscattered electrons, and secondary electrons and convert them into a signal that is sent to a screen similar to a television screen. This produces the final image.

The morphological properties of jute fabrics reinforced PP composites were observed by using scanning electron microscope. High resolution SEM has proved a precious tool for studying surface topography and failure analysis. In SEM, a highly focused scanning electron beam bombards the surface causing large numbers of secondary electrons to be generated, the intensity of which is governed by surface topography. The method is suitable for all materials, but non conducting materials must be given a thin conductive coating sputtered gold, which can alter or mask the true surface morphology. In this study, the fiber matrix adhesion of the tensile fracture surface of the composites was examined by using a Field Emission Scanning Electron Microscope (model no: JEOL JSM -7600F, serial no: SM1761001050105) at BUET. The micrographs were taken at a magnification of 1000X.

3.2.5 Thermogravimetric Analysis (TGA)

Thermogravimetric Analysis (TGA) is a technique in which the mass of the sample is monitored against time or temperature while the temperature of the sample, in a specified atmosphere, is raised at a specified heating rate. By this technique, the curve of mass loss of a sample as a function of temperature or time, which is called the TGA curve, is plotted. Any reaction of the sample that results in a mass change (loss or gain) is recorded. TGA curve provides information on the thermal reactions of the polymer that occur over a definite temperature range. In order to enhance interpretation of transitions in the TGA curve, the first derivative of mass change against temperature or time is frequently plotted, which is called the derivative thermogravimetric (DTG) curve, to determine the rate of mass change and the occurring temperature of maximum mass loss rate. A derivative weight loss curve can be used to tell the point at which weight loss is most apparent. Any temperature difference between sample and reference is also recorded. The differential temperature is then plotted against time, or against temperature which is called differential thermal analysis (DTA). TGA has been a very favoured technique for the study of flame-retardant polymeric materials in that it provides qualitative and quantitative data on the pyrolysis, oxidation and residue char of polymers upon heating. In general mass losses occur due the moisture desorption, volatiles release from decomposed material and the degradation of polymer. Thermal analysis provides important data to quantify the amount of possibly damaging, deteriorating volatiles, such as the moisture uptake during a hydrothermal treatment, which can cause deterioration in the composites (Joseph et al. 2003).

Thermo gravimetric analysis was carried out on 8-10 mg of untreated (raw) and treated jute fibers at heating rate of 20^{o}C /min in a nitrogen atmosphere using a thermo-gravimetric analyzer, Seiko instruments Inc. (TG/DTA 6300). Raw and treated samples were subjected to TGA/DTA in pure nitrogen gas under constant flow rate of 50 ml/min. Thermal decomposition of each sample occurred in a programmed temperature range of 30^{o}C - 550^{o}C.

3.2.6 Water uptake measurement

The water uptake properties of the composites were carried out according to ASTM D 570-99 standard. The specimens were dried in a vacuum oven at 70^{o}C until a constant weight was attained. The weight of the dry specimens was measured carefully (up to 0.01 g). Then, the samples were immersed in distilled water at room temperature. After that, the specimens were taken out and wiped with filter paper. The sample was weighed again and the water uptake was calculated with the following equation.

$$\text{Absorbed water } (\%) = \frac{(w_2 - w_1)}{w_1} \times 100 \qquad (3.12)$$

where w_1 = weight of the dry specimen,

w_2 = weight of the wet specimen

CHAPTER 4

RESULTS AND DISCUSSION

In this section, experimental results of different properties of matrix, fabrics, and composites are presented and discussed in detail. Firstly, properties of PP matrix, untreated fabrics of four different structures, and their composites are presented. Secondly, effects of 23 different treatments on plain structure fabrics only are discussed. Thirdly, properties of PP composites reinforced with plain structure fabrics treated by 23 different procedures are outlined. Lastly, results of composites of four different types of fabrics treated with the most effective treatment procedure (among 23 treatment procedures) are presented.

4.1 Characterization of PP, Untreated Jute Fabrics, and Their Composites

4.1.1 Mechanical properties of PP matrix

Polypropylene (PP) was used as matrix materials for the preparation of composites. Since, PP exhibits varying properties depending on manufacturer, it advisable to evaluate its properties before preparing the composites. As a result, mechanical properties of PP such as tensile strength and modulus, tensile elongation at break, flexural strength and modulus, and impact strength were evaluated which are presented in Table 4.1.

Table 4.1: Mechanical properties of PP

Tensile strength (MPa)	Tensile modulus (MPa)	Tensile elongation at break (%)	Flexural strength (MPa)	Flexural modulus GPa)	Impact strength (kJ/m^2)
22± 0.2	436 ± 4	385 ± 5	36 ± 1.3	2.4±0.06	4.5 ± 2

4.1.2 Thermal properties of PP matrix

Thermal analysis of PP was carried out by thermo gravimetric and differential thermal analyzer. The thermograms of PP are displayed in Figure 4.1. It is observed that a broad single peak starts at around 291°C (visualized at TG thermogram) with a maximum degradation at 432°C at the rate of 1.3 mg/min (DTG thermogram). This peak corresponds to the rupture of C-C chain bonds along with H- abstraction at the site of rupture (Joseph et al. 2003; Doan 2007). TG thermogram shows that loss of adsorbed water or moisture from PP is 0.6% which indicates that PP is strongly hydrophobic in nature. PP is completely depleted at 488°C with the formation of 13% char residue (TG thermogram). 50% degradation temperature of PP is observed at 417°C (TG data). The endothermic peak at 166°C in DTA thermogram corresponds to the melting point of PP.

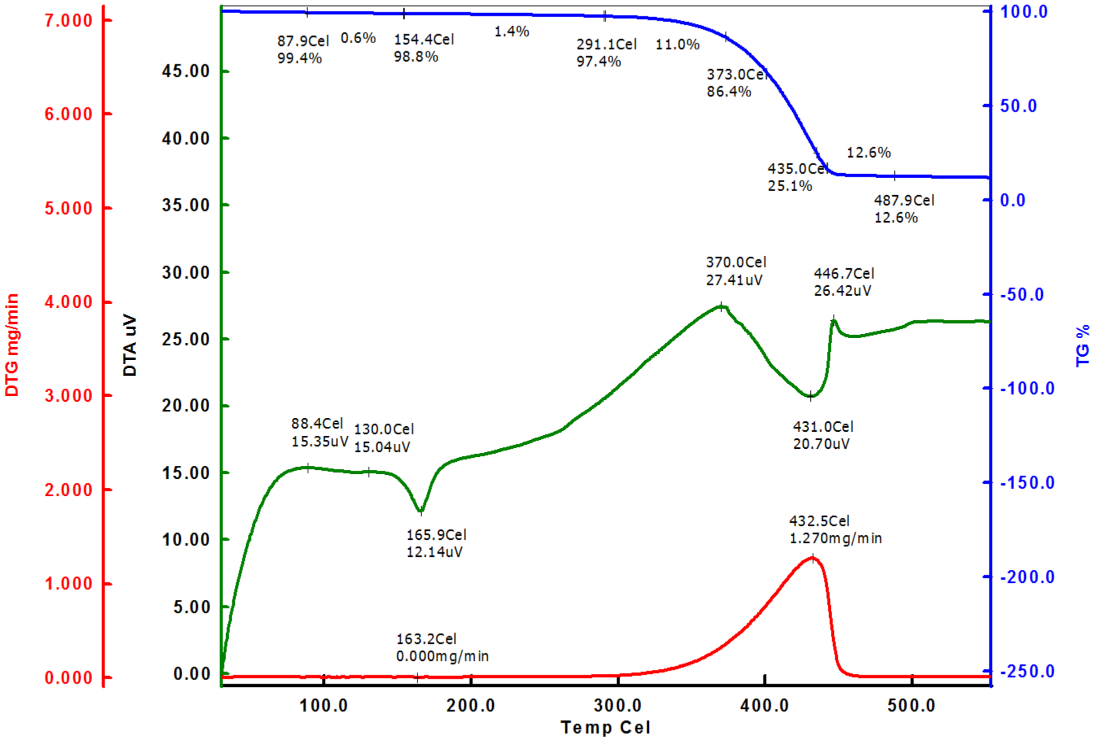


Figure 4.1: Thermograms of PP (Top - TG, middle - DTA, and bottom - DTG).

4.1.3 Fabric strength

Table 4.2 illustrates the effect of fabric structures on fabric strength. It was obviously observed that the fabric structures influenced the tensile strength of the fabrics. Further, it was observed that the twill structure fabrics had the highest tensile strength and lowest percent elongation among all the fabrics. On the other hand, plain structure fabrics had the lowest strength and medium elongation. However, it was noted that the rib structure fabrics had the highest % elongation owing to the fiber orientation of the fabrics. The similar features were reported by researchers in the literature (.Hull 1981; Betiana et al. 2005).

Table 4.2: Tensile properties of jute fabrics of different structures

Fabric structure		Fabric strength Max. force (N)	Elongation %
Woven	Plain	249 ± 3	39 ± 0.3
	Twill	435 ± 5	31 ± 2
Knit	Single jersey	291 ± 3	67 ± 0.5
	Rib	332 ± 3	96 ± 0.8

4.1.4 Thermal properties of fabrics

Thermal analysis of jute fabrics was performed by thermo gravimetric and differential thermal analyzer. The thermograms of jute fabrics are presented in Figure 4.2. DTG thermograms show that decomposition of fabrics occurs as a three step process. The first process below 100°C corresponds to the loss of adsorbed water (6%) of the fabrics. In the second process the decomposition of hemicellulose of jute fabrics occur at 299°C with the rate of 0.3 mg/min (Doan 2007). The strong third peak observed at 377°C (rate 1.70 mg/min) may be attribute o the decomposition of α-cellulose (Doan 2007). The char residue obtained at 449°C was 9% (TG thermogram). The formation of char residues may involve initial physical desorption of water, intramolecular dehydration, formation of carboxyl and carbon-carbon double bonds, cleavage of glycosidic linkage and rupture of C—O and C—C bonds, condensation and, aromatization of carbon atoms from each original pyranose ring to form discrete graphite layers (Doan 2007; Mohanty et al., 2006; Daniel 1985). It is also observed from TG thermogram that onset and 50% degradation temperatures of jute fabrics were 228^{o}C and 364^{o}C, respectively. The investigation reveals that jute fabrics are less thermally stable compared to PP (onset and 50% degradation temperatures were 291^{o}C and 417^{o}C) in nitrogen environment. The similar observation was also reported in the literatures (Doan 2007; Mohanty et al. 2006).

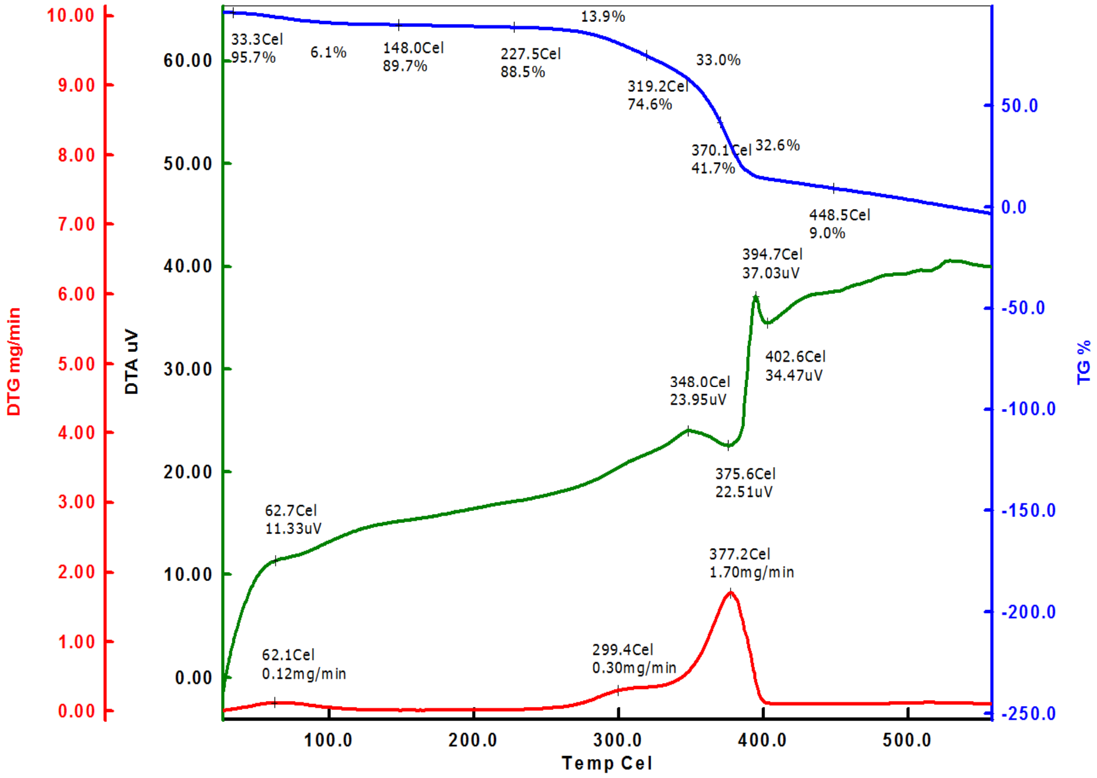


Figure 4.2: Thermograms of jute fabrics (Top - TG, middle - DTA, and bottom - DTG).

4.1.5 Water absorption of fabrics

Water absorption tests were carried out for all the fabrics of four different types as shown in Table 3.2. The test results of these fabrics are shown in Figure 4.3, which indicate the time required for a drop of water to be fully absorbed by the jute fabrics. Observation of Figure 4.3 shows that the rib fabrics were the most hydrophobic and twill fabrics were the least hydrophobic in nature among others, because the rib fabrics exhibited the highest and the twill fabrics exhibited the lowest value of time among all the samples. Further, between two woven structure fabrics (plain and twill), the twill fabrics require less time to absorb water than plain structure fabrics. Again, between two knitted structure fabrics (rib and single jersey), water absorption time is less for single jersey fabrics than that of rib structure fabrics. This is owing to the fact that the surface roughness of these fabrics is opposing with respect to their different structures.

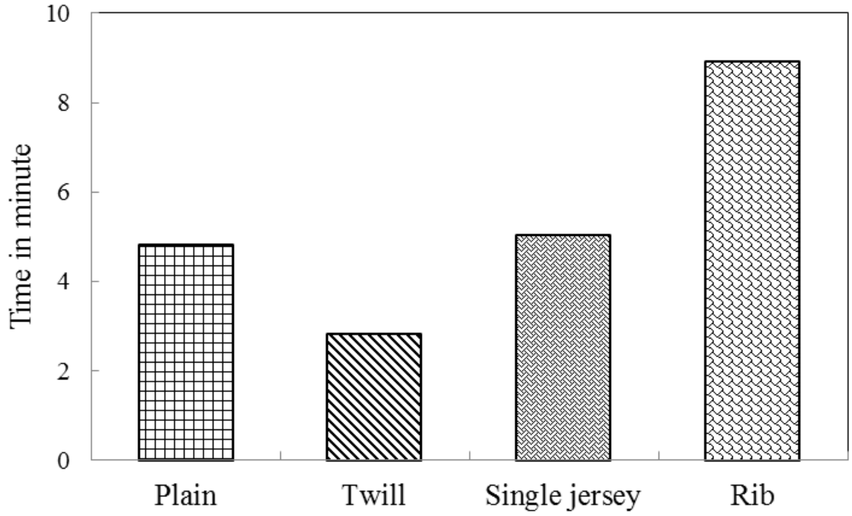


Figure 4.3: Water absorption properties of jute fabrics of different structures.

4.1.6 FTIR analysis of fabrics

The hydroxyl group in the raw (untreated) jute fabrics is responsible for high water absorption and weak interfacial bond between the jute fabrics and PP matrix. There are actually three hydroxyl groups present in a cellulose unit of jute fiber (Figure 4.4). One is a primary hydroxyl group at C_6 and the other two are secondary hydroxyl groups at C_2 and C_3. The FTIR spectroscopic analysis of the raw jute fabric (Figure 4.5) confirms this phenomenon.

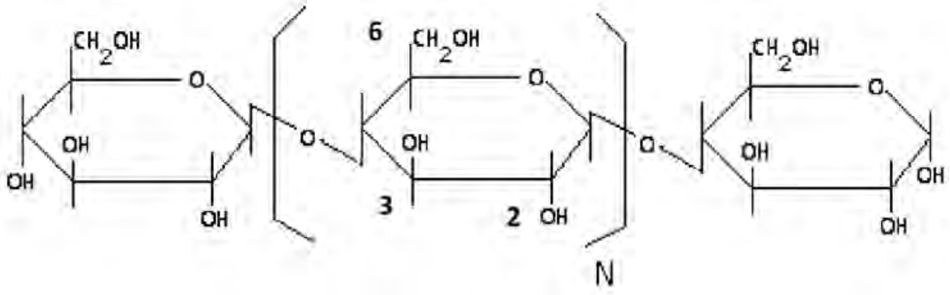


Figure 4.4: Cellulose structure of jute fiber.

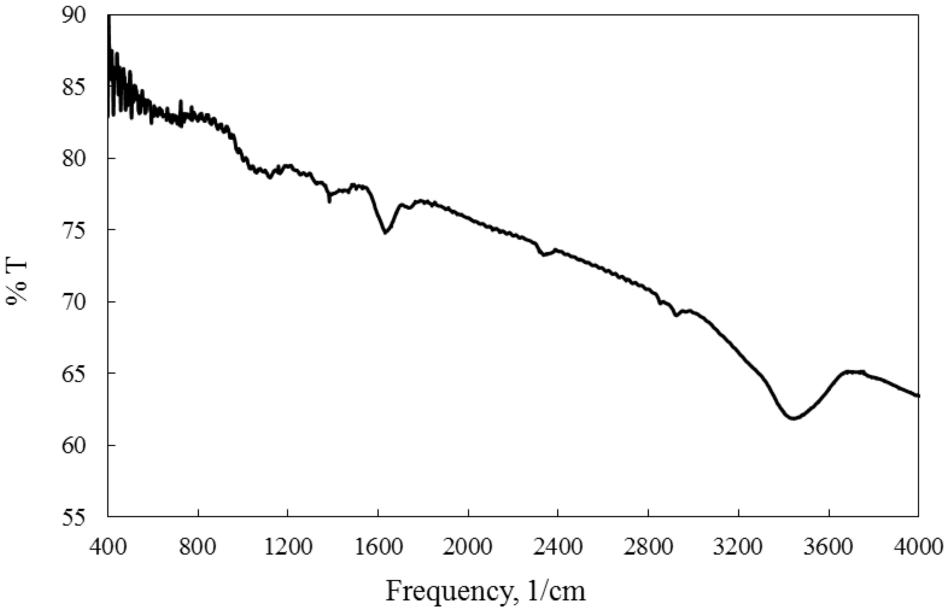


Figure 4.5: FTIR spectra of untreated jute fabrics.

The peak assignments of the absorption bands corresponding to various groups are summarized in Table 4.3 which is quite similar to those available in literatures (Li et al. 2004; Silverstein et al; Garside et al.; Haque et al. 2006).

Table 4.3: FTIR spectral data of untreated jute fiber.

Position (cm^{-1})	Assignment
3639 - 3422	Strong free -OH band from the cellulose, hemicellulose and lignin of jute fiber
~ 2912	C-H stretching band associated with methyl and methylene groups
1730 - 1620	C=C aromatic in-plane ring stretching from the cellulose and lignin
1504 - 1450	In-plane -OH bend of cellulose
1164 -1051	C-O-R stretching band of ether group
~ 897	C-O-C bond in plane, symmetric

4.1.7 Mechanical properties of the composites

Jute fabrics of different structures were used as reinforcement for PP matrix. The structure of jute fabrics play a pivotal role in determining the strength of the fabrics. The yarn quality and number of yarn per inch in both the directions of the fabrics were different. In this study, mechanical properties of the composites were evaluated measured along the longitudinal direction (warp) in all the cases.

4.1.7.1 Tensile properties

The importance of natural fiber reinforced polymer matrix composites comes from the substantial improvement in strength and modulus that offer a possibility of uses of these composites in practical applications. The tensile strength of a composite material is more sensitive to the fiber-matrix interfacial properties, whereas the modulus is dependent on the fiber properties. To improve the tensile strength, a strong interface, low stress concentration, and good fiber orientation are required, whereas fiber concentration, fiber wetting in the matrix phase, and a high fiber aspect ratio influence the tensile modulus (Seki 2009; Kelly et al. 1965). Figure 4.6 illustrates the effects of fabric structures on the tensile properties of the untreated jute fabrics reinforced polypropylene composites. It is observed from Figure 4.6 that the composites of the twill structure fabrics exhibited the highest tensile strength (48 MPa), which is 134% higher than that of plain structure fabric composites. This is due to the fact that the twill structure fabrics had more yarns in the warp (longitudinal) direction than plain structures fabrics for the same weight fraction (55±1%) of fabrics. If normalized in terms of the same number of yarns in the warp direction in both the plain and twill structures, the tensile strength of twill fabrics composites will be 28 MPa which is still 40% higher than that of plain fabric composites. So, it can be said that structure of reinforcing fabrics affects the strength of the final composites. Further, the rib and single jersey fabrics reinforced composites exhibited 46% and 17% higher tensile strength than that of plain structure fabrics composites respectively. This increase in tensile strength may be attributed to the improved interfacial mechanical locking between jute fiber and PP matrix. It is well known that the tensile strength of jute reinforced PP composites depends on the strength of both the constituents and the adhesion between the jute fibers and PP matrix (Cyras et al. 2004). The improved strength might have been achieved due to the different surface structure of the fabrics. So, this structure is advisable when tensile strength is the main concern. Also, tensile modulus of the composites having twill, rib, and single jersey fabrics had the higher value than that of the composite having plain structure fabrics as shown in Figure 4.7. Furthermore, the elongation at break of the composites having single jersey fabrics was significantly higher than that of the composites having plain, twill, and rib structure fabrics (Figure 4.8). Thus, it could be said that this structure was advantageous for the improvement of elongation at break.

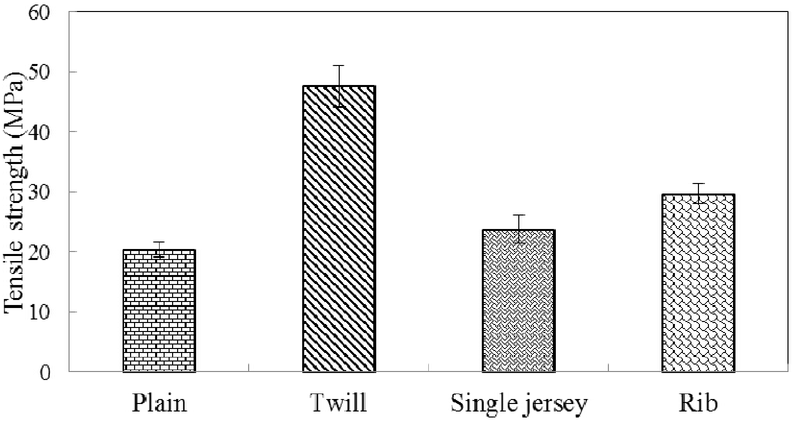


Figure 4.6: Tensile strength of jute fabrics/PP composites.

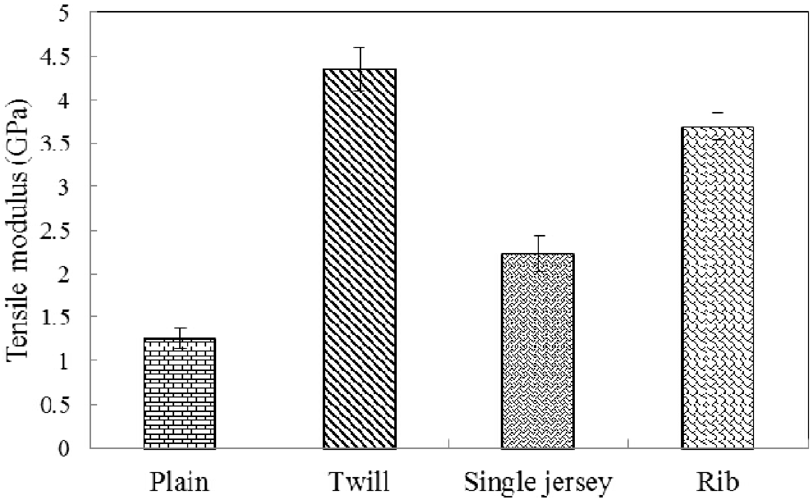


Figure 4.7: Tensile modulus of jute fabrics/PP composites.

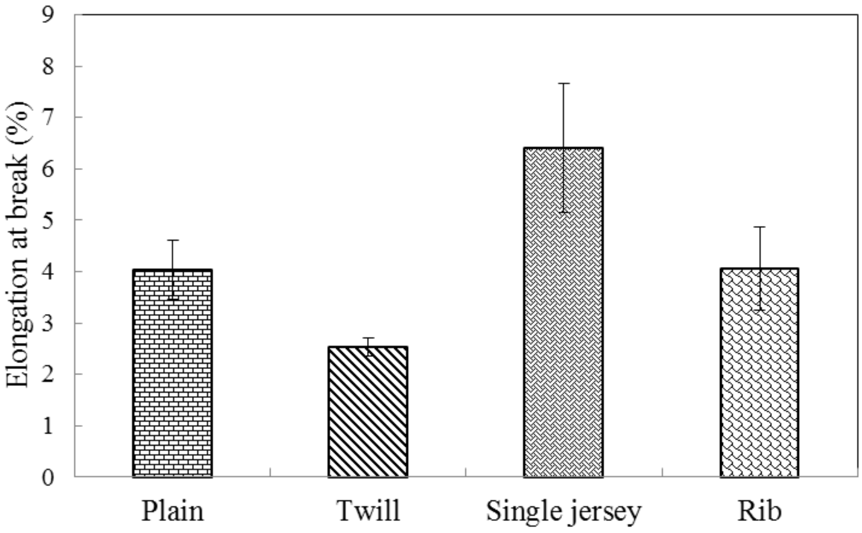


Figure 4.8: Elongation at break of jute fabrics/PP composites.

4.1.7.2 Flexural properties

The flexural properties of composites with different fabric structures are illustrated in Figures 4.9 and 4.10. As seen from Figure 4.9, the highest value of flexural strength was observed for twill fabric composites, which was 59.51 MPa. In general, the flexural strength is much greater than the tensile strength which is due to the failure behavior of the outermost matrix layer. This general trend was also observed for the present case as seen from Figures 4.6 and 4.9. From Figure 4.10, it was observed that the flexural modulus of twill fabrics/PP composites was the maximum (4.76 GPa). Thus, the twill fabrics composites exhibited better flexural strength and modulus compared to the knitted fabrics composites. This could be attributed to the better dispersion of twill fabrics in the PP matrix.

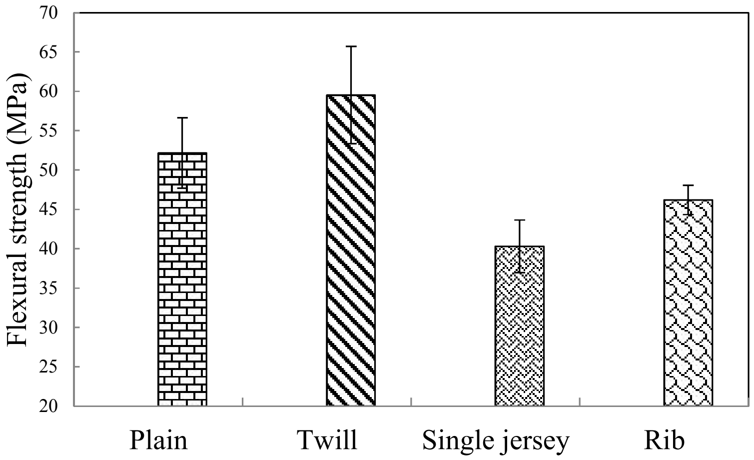


Figure 4.9: Flexural strength of jute fabrics/PP composites.

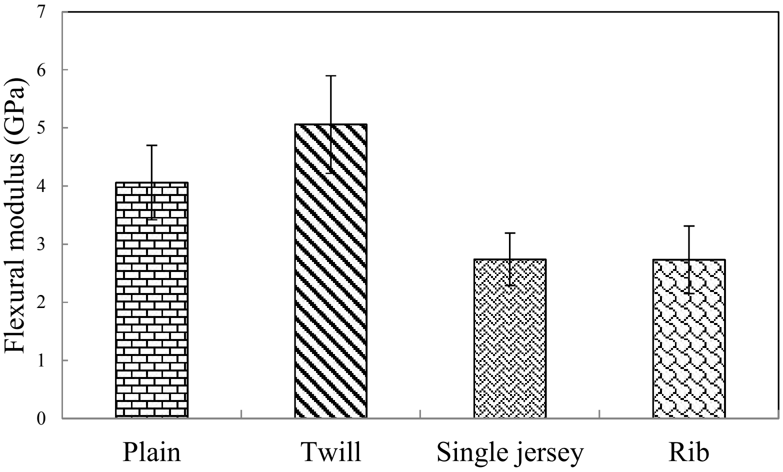


Figure 4.10: Flexural modulus of jute fabrics/PP composites.

4.1.7.3 Impact strength

A comparison of impact strengths of different jute fabric composites is shown in Figure 4.11. Impact strength showed similar trend as tensile strength properties. The highest value of impact strength was observed for twill fabric composites. One of the factors of impact failure of composites is fiber pull out. For twill structure, bigger force is required

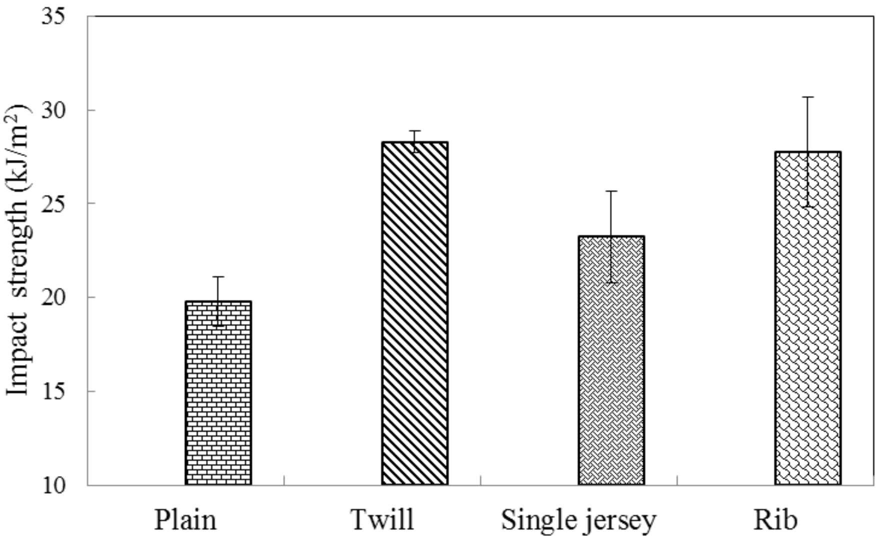


Figure 4.11: Impact strength of jute fabrics/PP composites.

to pull out the fibers. This consequently increased the impact strength. The average value of impact strength of the twill fabric /PP composites was found to be 28 kJ/m^2 which was greater than that of the plain fabric/PP composites having an average value of 20 kJ/m^2. This corresponds to 43% increase in the impact strength. It is known that the cracks in composites can propagate preferentially along the fiber–matrix interfaces or transversely right through the fiber and matrix, depending on the properties of the interface with respect to those of the fiber and matrix. In this case, where the results of the unnotched tests are considered, energy absorption takes place due to a combination of crack initiation and propagation. In the case of twill fabric, the fracture advances with little change in the cracking plane, breaking the fibers instead of pulling them out. On the other hand, when the interface is weak, interface debonding and fiber pull-out, which are the major sources of fracture toughness in composites, can take place.

4.1.8 SEM analysis of composites

The tensile-fracture surfaces of composites were analyzed through SEM to evaluate the degree of interfacial adhesion between jute fibers and PP matrix. The SEM micrographs (1000X) of the fracture surfaces are displayed in Figure 4.12. The micrographs show that there is no or only a little trace of PP matrix on the surfaces of jute fibers, which

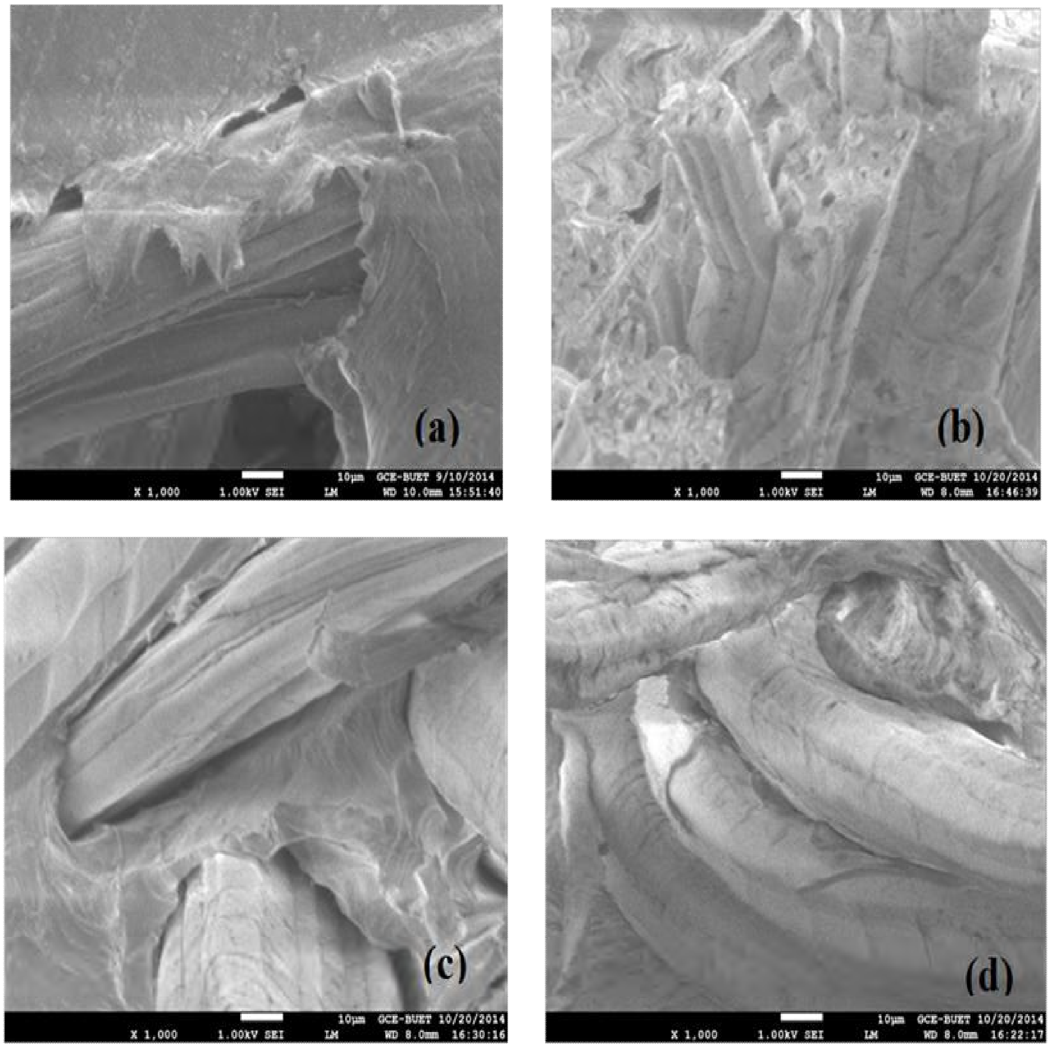


Figure 4.12: SEM micrographs of tensile fracture surfaces of composites (a) Plain, (b) Twill, (c) Single jersey, and (d) Rib fabrics.

indicates no adhesion between fibers and matrix. The gap between fibers and matrix is relatively large for plain and single jersey composites than that for twill and rib composites. This ensures the better mechanical interlocking between fibers and matrix for twill and rib fabrics composites. Consequently, these composites had higher tensile strength than that of plain and single jersey composites as can be seen from Figure 4.6. For poor adhesion and mechanical interlocking, transfer of stress from matrix to fibers could not take place effectively for which strength of resulting composites had not improved.

4.1.9 Thermal properties of composites

Thermal properties of the composites were determined to study the comparative thermal stability of the composites with regard to its component materials. Thermograms of jute fabrics/PP composites are presented in Figure 4.13. The TG thermogram shows that loss of adsorbed water is 4%. It is thought that during processing or testing, water could have been absorbed from the atmosphere if the fibers were not fully wetted by the matrix in the composite (Aziz et al. 2004). The residues obtained for the composite is 8% at 450°C (TG thermogram). In composites, two stage degradations occur: the first stage is responsible for jute fabric and second stage is for PP. The DTG thermogram shows that maximum degradation temperatures (MDT) of the composite for the first and the second stages are 378°C and 449°C, respectively. From these observations it can be

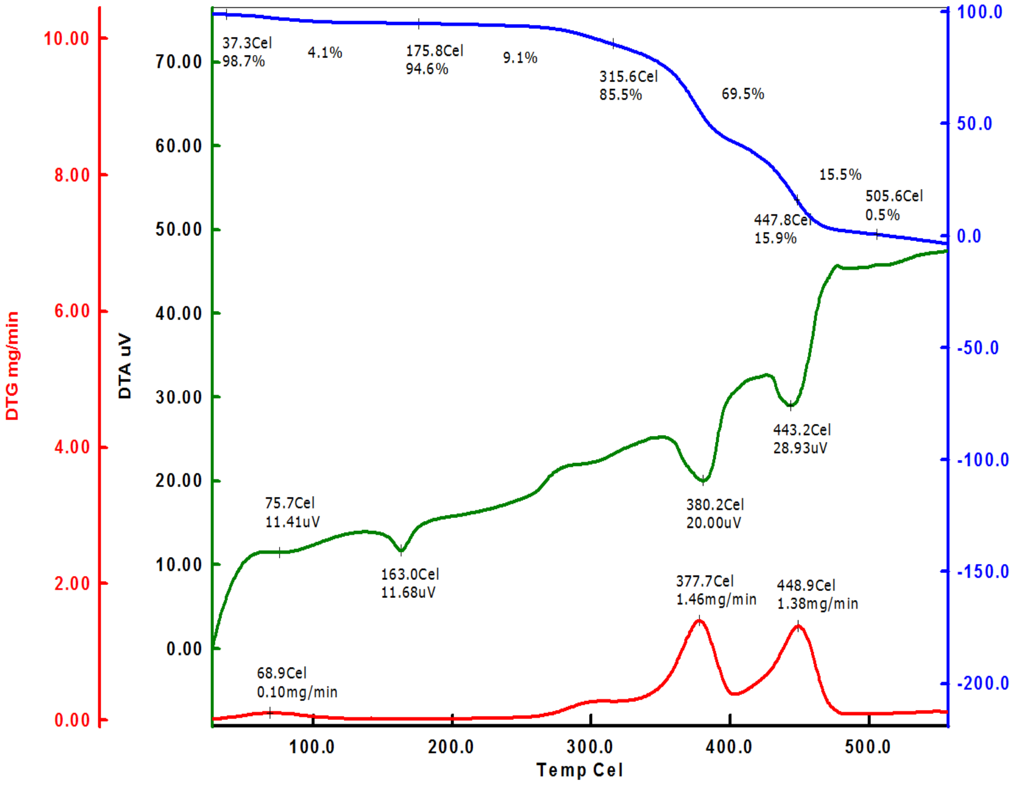


Figure 4.13: Thermograms of jute fabrics/PP composites (Top - TG, middle – DTA, and bottom - DTG).

realized that MDT (377 °C) of untreated jute fabrics (Figure 4.2) remains almost unchanged in the composite (378 °C). At the second stage, the MDT of neat PP (433 °C) as shown in Figure 4.1 is shifted to higher temperature regions by 16 °C in the composite (449 °C). It indicates that composites are more thermally stable compared to PP. Therefore, it can be concluded that thermal analysis of PP and fabrics.

4.1.9 Water uptake properties of composites

Water uptake of jute fabrics reinforced PP composites is an important characteristic that determines terminal applications of these materials (Hull 1981). Water absorption could lead to a decrease in some of the properties and needs to be considered when selected for applications. It is quite difficult to eliminate entirely the absorption of moisture by 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 dimensional stability cannot be ignored (Hull 1981). Water uptake percent of jute fabrics/PP composites having four different structures are shown in Figures 4.14 - 4.16. These results indicate that water uptake significantly depends upon the surface characteristics of the reinforcing material. It shows that twill fabric composites had the highest and rib fabric composites had the lowest water uptake among all the four different types of composites. At 24 hours, water uptake of rib fabric composites is about 58% less in comparison with that of plain fabric composites as seen from Figure 4.15. It is also noticed that water uptake of composites of knitted fabrics differ significantly from the composites of woven fabrics up to time interval of 24 hours (Figure 4.14 and 4.15). Figure 4.16 shows that water uptake for all the composites reaches to the maximum value at around 10 days. After this, all the curves become flat indicating the saturation of composites by water. The lower value of water uptake of the knitted fabric composites is due to the fact that the surface tension of knitted fabrics is higher because of the entrapped air is more in the knitted fabrics than the woven structure fabrics.

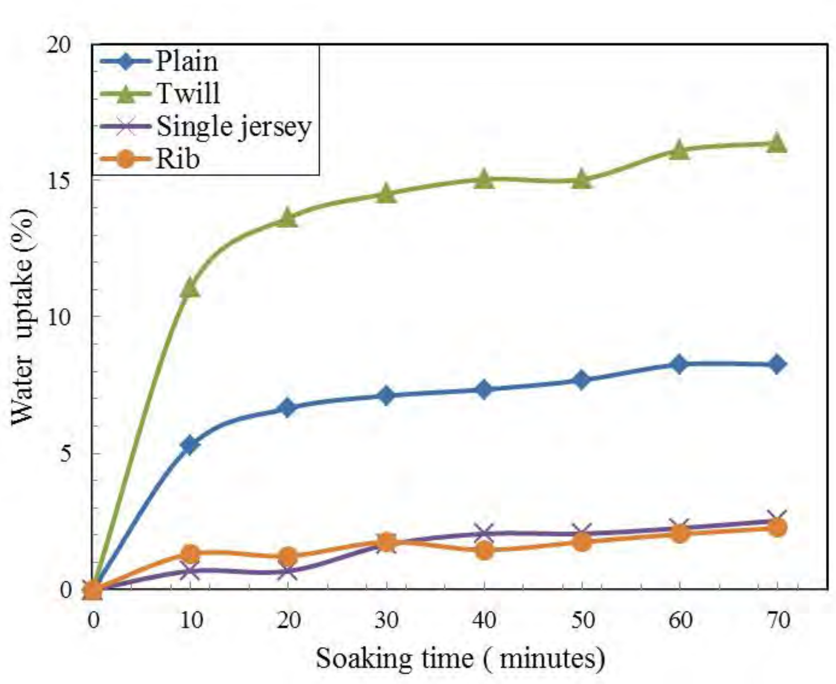


Figure 4.14: Water uptake versus soaking time (minutes) of the jute fabrics/ PP composites.

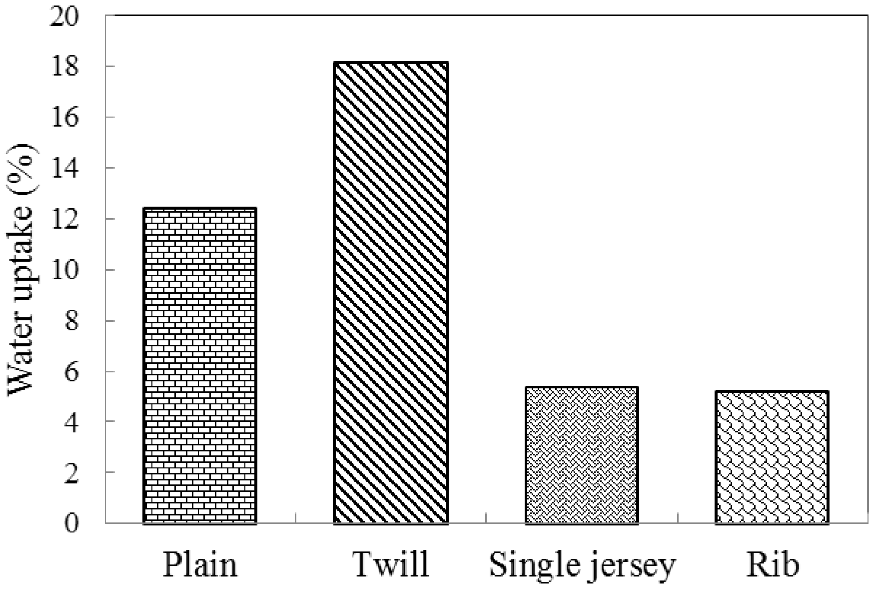


Figure 4.15: 24 hours water uptake of the jute fabrics/PP composites.

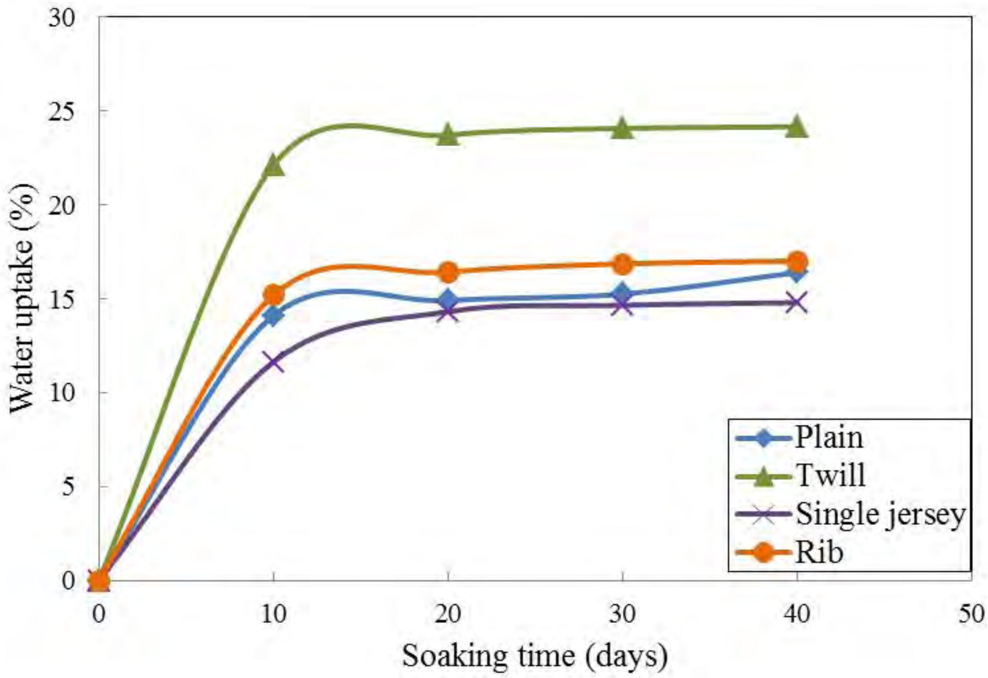


Figure 4.16: Water uptake versus soaking time (days) of the jute fabrics/PP composites.

4.1.11 Conclusions

Performance of jute fabrics reinforced PP composites having four different structures of untreated fabrics have been evaluated and compared. It was found that both the mechanical and water absorption properties of the composites were influenced by the structures of the fabrics. When normalized in terms of the same number of yarns in the warp direction, composites having twill structure fabrics exhibited 40% higher strength than the composites having plain structure fabrics. It was also observed that the elongation at break of the composites containing single jersey fabrics was significantly higher than that of the composites containing plain structure fabrics. Water absorption of weft knitted fabrics reinforced PP composites was much better than that of woven fabrics composites.

4.2 Effects of Treatments on the Properties of Plain Structure Jute Fabrics

All the twenty three treatment procedures of Table 3.1 at optimum chemical concentration had been followed to modify the plain structure jute fabrics for improving the adhesion properties between hydrophilic jute fabrics and hydrophobic polypropylene matrix. For convenience and simplicity, the jute fabrics treated by 23 different procedures are denoted by various symbols. The treatment procedure mentioned in raw one of Table 3.1 uses Albafix WFF along with other chemicals in all the five subcategory of treatments in each column of Table 3.1. In other words, Albafix WFF is common in all the five subcategory of treatments in each column of raw one of Table 3.1. Thus the jute fabrics treated by these five procedures are denoted by JA1, to JA5, where A indicates Albafix WFF. Similarly, the jute fabrics treated by other procedures of raw 2 to 5 of Table 3.1 are denoted in the similar manner. A complete list of symbols of treated jute fabrics is shown in Table 4.4. Some other symbols corresponding to jute fabrics treated by conventional treatment procedures are displayed in Table 4.5. Untreated jute fabrics are denoted by JUn. The samples JUn, JN, and JS are used as control samples in this study for the purpose of comparison.

Table 4.4: Symbols of treated jute fabrics

Symbols of groups of fabrics	Symbols of individual jute fabric				
	1	2	3	4	5
JA	JA1	JA2	JA3	JA4	JA5
JAS	JAS1	JAS2	-	JAS4	JAS5
JG	JG1	JG2	JG3	JG4	JG5
JGS	JGS1	JGS2	-	JGS4	JGS5
JI	JI1	JI2	JI3	JI4	JI5

Table 4.5 Symbols of jute fabrics treated by conventional procedures

Treatment procedures	Symbol of fabrics
Treatment with the mixture of 1% Drimarene Red K8b, NaCl (40g/l), and Na_2CO_3 (10g/l) at 60°C for 60 minutes	JN
Treatment with 4% NaOH at 60°C for 60 minutes	JS

4.2.1 Water absorbency

Water absorbency tests were carried out for all the samples of Table 4.4 and 4.5. The test results of these samples are shown in Figure 4.17 and 4.18. These figures show that all the Indosol treated samples (JI) are the most hydrophobic among all the samples. There was a significant difference of time required to absorb a drop of water between the fabrics JI (J1 to J_{15}) and the untreated fabrics JUn. Further, the fabrics of subcategory 2 (JA2, JAS2, JG2, JGS2, and JI2) exhibited the highest value of time among each corresponding group of samples. In addition, the fabrics of JAS group (except AS5) exhibited the lowest value of time among all the fabrics including the untreated jute fabrics JUn.

Apart from all the JI fabrics, all the samples of GS group show the higher hydrophobic character compared to other groups of samples. The sample JGS2 shows the highest value of time among all including control fabrics. That is the treatment with the mixture of Glytac, sodium hydroxide, and Drimarene Red K8b induces higher hydrophobicity than those treated with the mixture of Glytac and sodium hydroxide (JGS1). Again, comparison of fabrics 1 and 2 of each group shows that fabrics 2 of each respective group have the higher hydrophobic character. Thus, it is inferred that using of Drimarene Red K8b during treatment increases the time to absorb a drop of water by the fabrics.

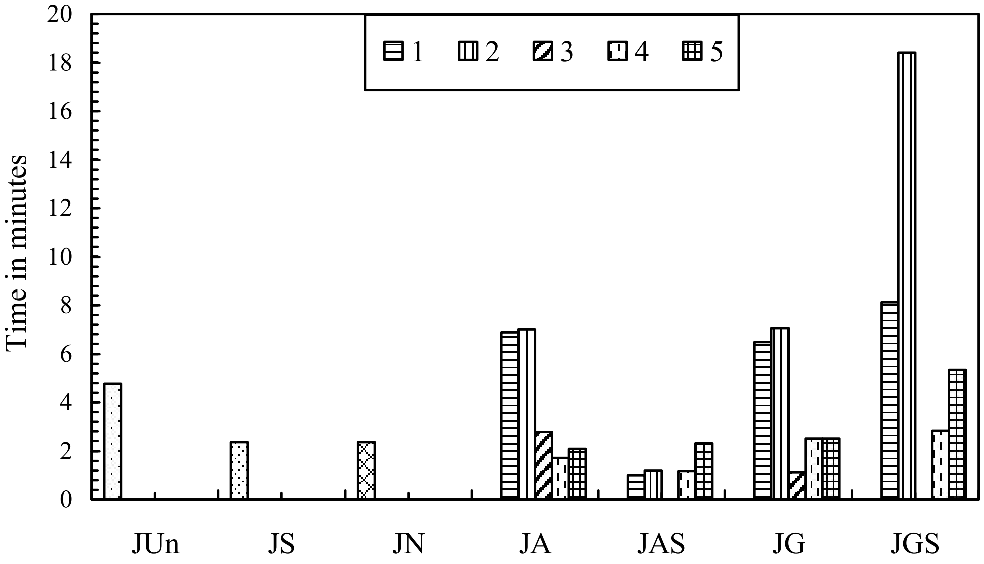


Figure 4.17: Water absorbency properties of different treated and control fabrics.

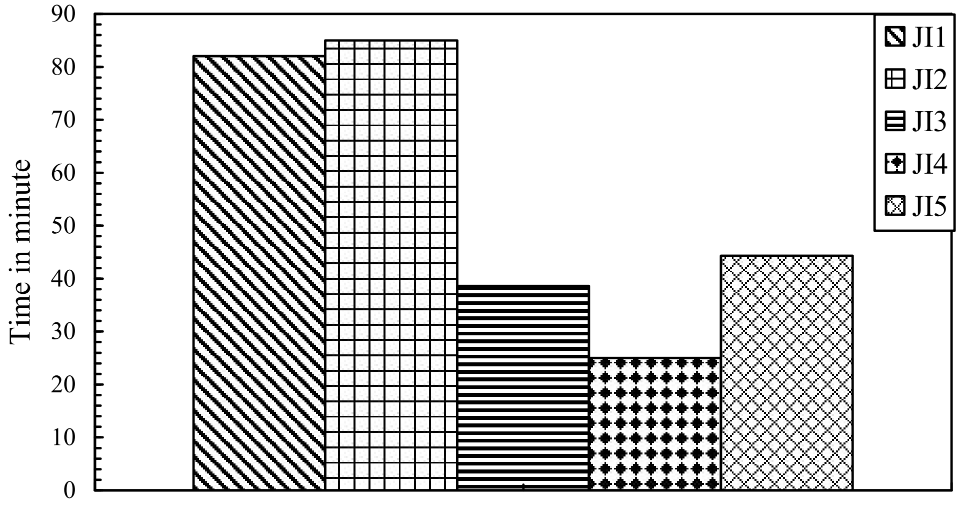


Figure 4.18: Water absorbency properties of JI fabrics.

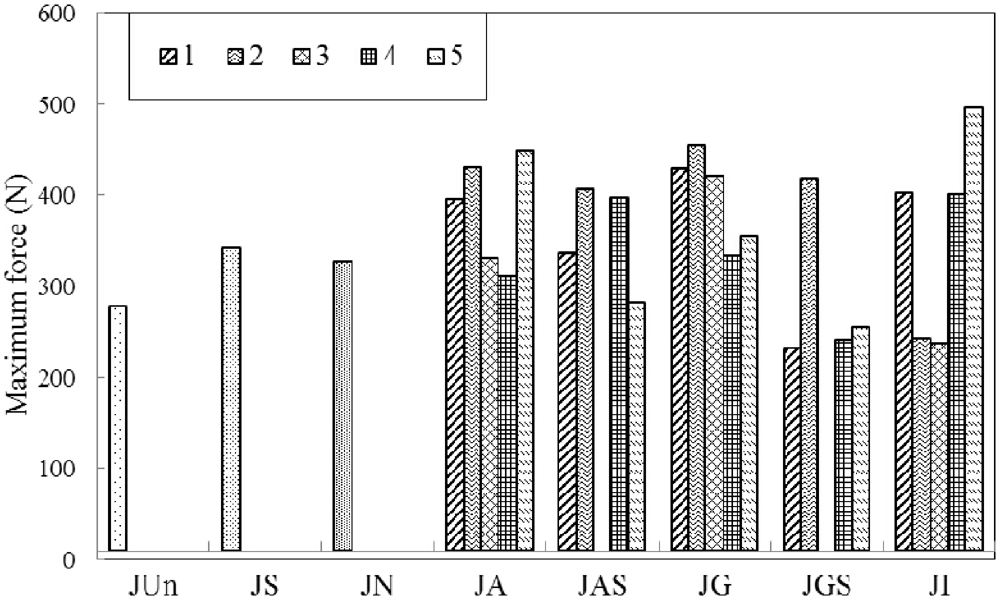


Figure 4.19: Tensile strength of different treated and control fabrics.

4.2.2. Fabric strength

The tensile strength of all the treated and control jute fabrics are shown in terms of maximum force (N) in Figure 4.19. It shows that most of the treated jute fabrics including the two of the control fabrics JS and JN have higher tensile strength than that of the untreated jute fabrics (JUn). Apart from Indosol treated fabrics (JI), the tensile strength of all the treated jute fabrics of subcategory 2 shows the higher tensile strength than that of all the treated fabrics of subcategory 1. That means the fabrics (JA2, JAS2, JG2, and JGS2) treated in the presence of dye exhibit higher strength than that of fabrics (JA1, JAS1, JG1, and JGS1).treated without dye.

4.2.3 FTIR analysis

FTIR analysis was performed for all the treated plain jute fabrics of Tables 4.4 and 4.5 except the fabrics of subcategory 3 in every case. Because, all the fabrics of subcategory 3 and 5 correspond to the treatment with the same chemicals and fabrics of subcategory 3 have the lower color strength (*K/S*) (as will be seen later) compared with the corresponding fabrics of subcategory 5. Thus, fabrics of subcategory 3 have not been considered for FTIR analysis. Figure 4.20 shows the comparison of FTIR results for all

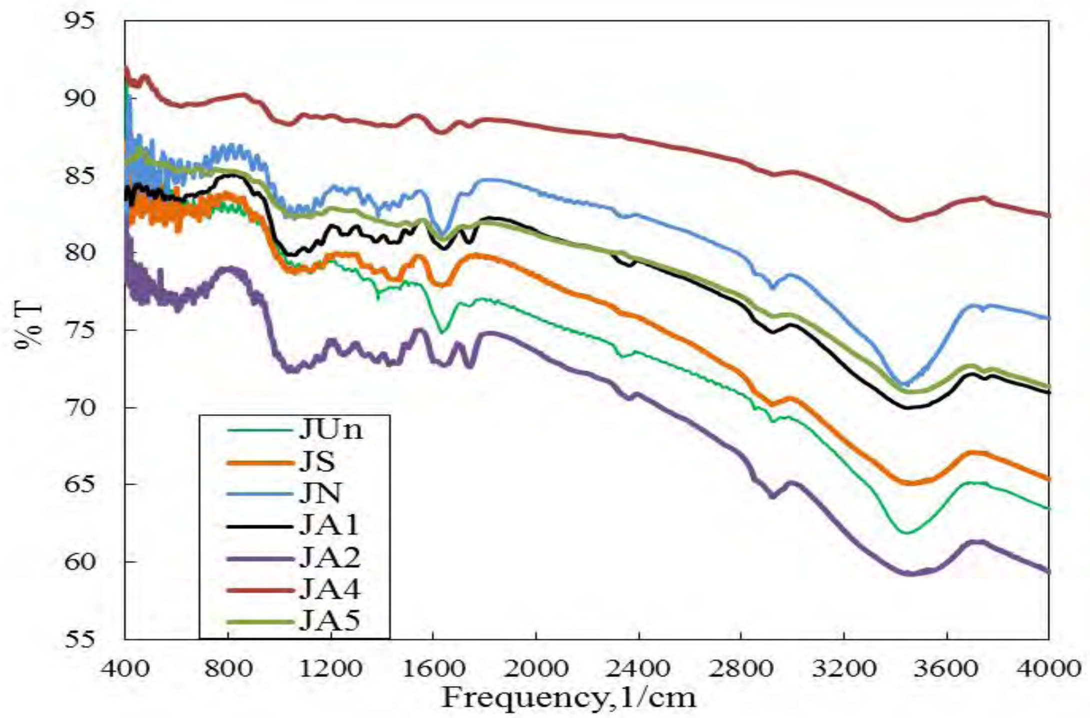


Figure 4.20: FTIR spectra of JA and control fabrics.

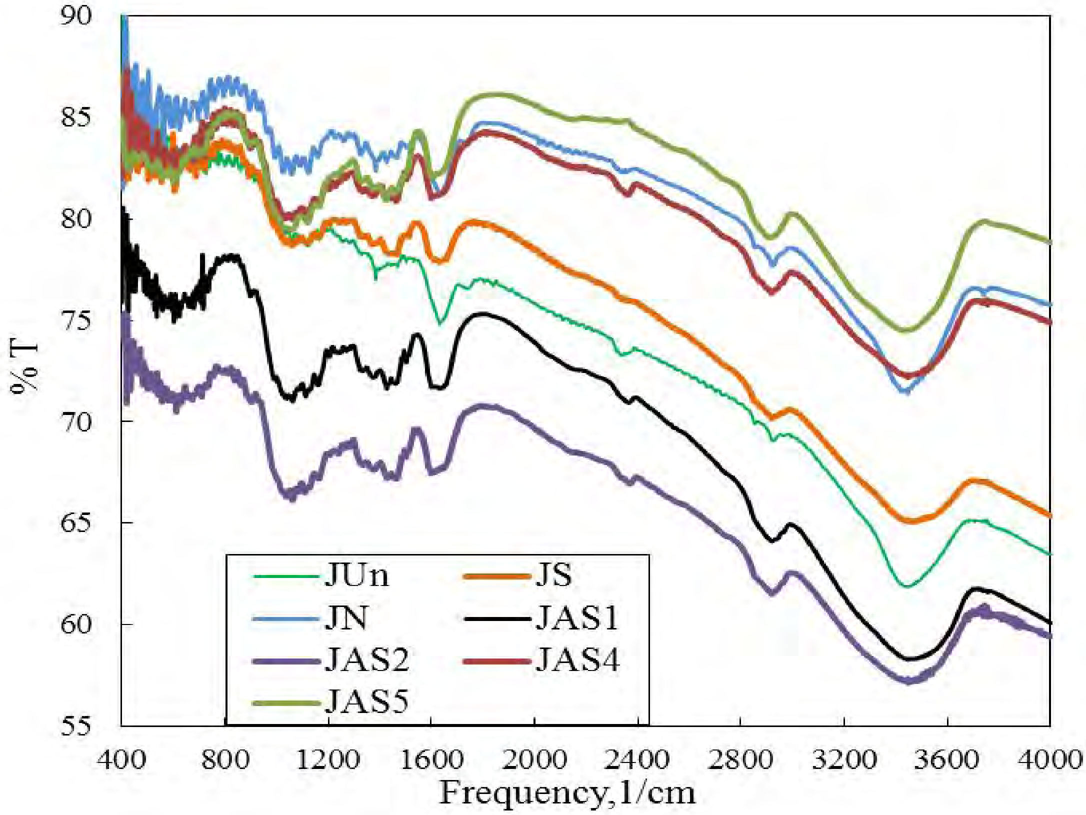


Figure 4.21: FTIR spectra of JAS and control fabrics.

the control and JA fabrics (excluding JA3 as mentioned earlier). In the FTIR spectra (Figure 4.20), there was an absorption peak at 3462 cm^{-1} for untreated jute fabrics. This peak represents the OH group in the cellulose of the jute fibers. This peak shifted to 3446 cm^{-1}for the fabrics JA1 and 3449 cm^{-1} for the fabrics JA2. In this region, it is found that the percentage transmittance (%T) of these fabrics is higher than that of untreated jute fabrics. This indicates the lower amount of OH group present in the fibers. The increase of the peak at 2924 cm^{-1} in the treated fabrics implies an increase of aliphatic link (C-H) due to the oley chloride reaction (Corrales et al. 2002).

Figure 4.21 shows the comparison of the FTIR spectrum of all the control and AS fabrics. It can be noted that there is an absorption band at 1730 cm^{-1} and 1240 cm^{-1} for the untreated jute fibers, which no longer exists for the AS fabrics. The hemicelluloses are soluble in aqueous alkaline solutions. During alkali treatment, a substantial portion of uronic acid and fatty substances might be removed resulting in disappearance of this peak at 1730 cm^{-1} and 1240 cm^{-1} (Liu and Dai 2007). In addition, the peak at 1622 cm^{-1} in the FTIR absorption spectrum of the JAS1 fabrics represents the R–NH_2 bending for the primary amines. It was found that the Poly-diallyl-dimethylammonium chloride (PDAC) homopolymer (Albafix WFF) were successfully applied on the jute fabrics. The jute treated with PDAC (Albafix WFF) shows a signal at 1503 cm^{-1}, which can be attributed to amide II (NH) modes on the basis of the structure of this chemical. This finding led us to the conclusion that in the presence of sodium hydroxide, PDAC homopolymer (Albafix WFF) can be fixed into the jute fibers through the reaction of the aldehyde groups of the hemi-cellulose with the amino groups provided by PDAC, which react with the reactive dye molecule significantly so that dye uptake of JAS4 fabrics had increased substantially (scheme 4.3 and 4.4 in the next section).

Figure 4.22 shows the FTIR spectrum of all the JG and control fabrics. Absorption peak

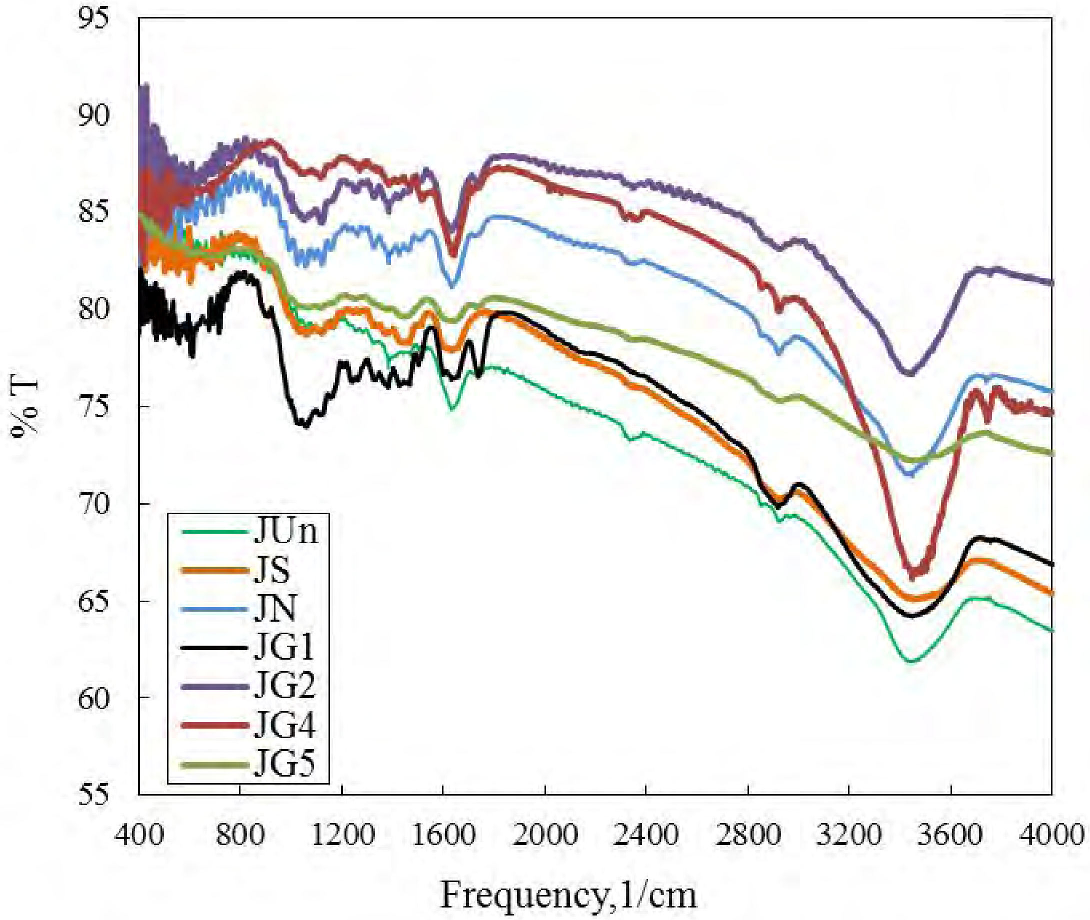


Figure 4.22: FTIR spectra of JG and control fabrics.

at 3462 cm^{-1} for untreated jute fabrics shifted to 3433 cm^{-1}, 3435 cm^{-1}, 3457 cm^{-1}, and 3448 cm^{-1} for the fabrics JG1, JG2, JG4, and JG5, respectively. A strong band at 1742 cm^{-1} in the infrared spectrum of JG1fabrics is due to C=O stretching in an ester. In case of azo compounds (-N=N-), the N=N stretching vibration occurs in the region of 1630-1570 cm^{-1} of JG2, JG4, and JG5 fabrics. This indicates the lower amount of OH group present in the fibers.

Figure 4.23 shows the FTIR spectrum of all the JGS and control fabrics. Absorption peak at 3462 cm^{-1} for untreated jute fabrics shifted to 3443 cm^{-1}, 3448 cm^{-1}, 3466 cm^{-1}, and 3467 cm^{-1} for the fabrics JGS1, JGS2, JGS4, and JGS5, respectively. The fabrics JGS5 gave the highest percentage transmittance i.e. lowest absorption value among the fabrics in the hydroxyl region. This indicates the lower amount of OH group present in

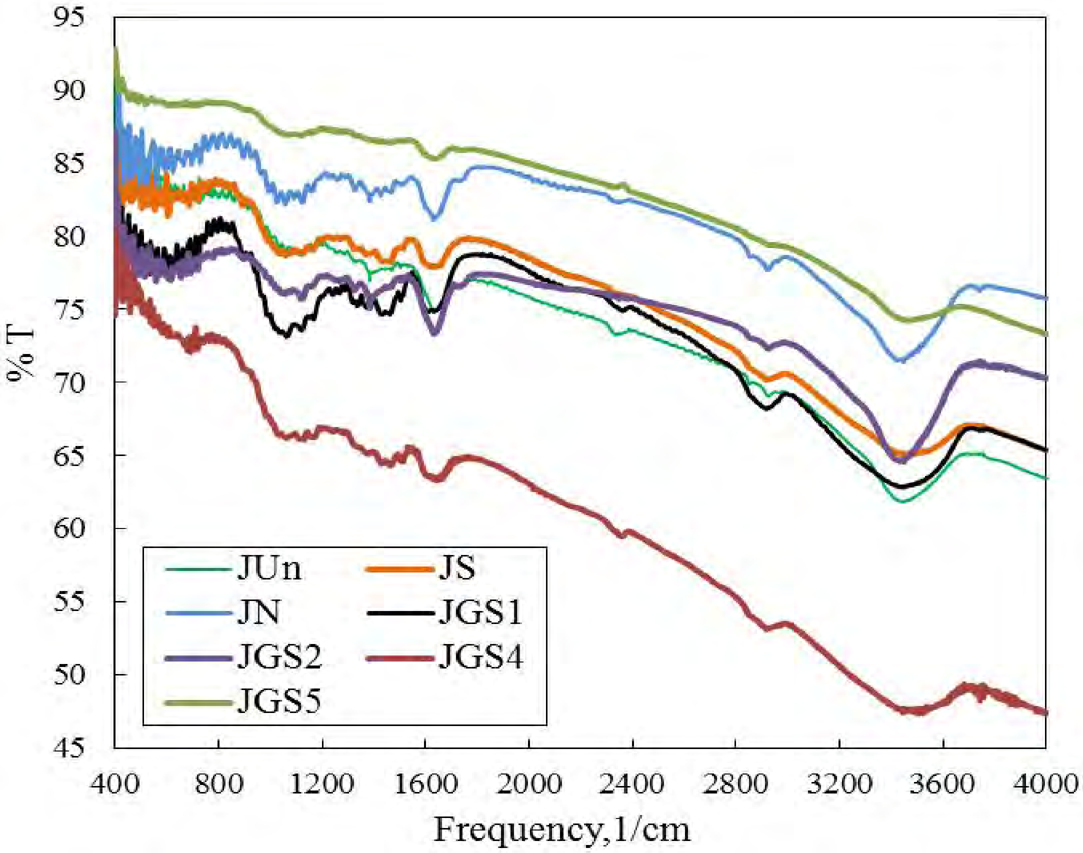


Figure 4.23: FTIR spectra of JGS and control fabrics.

aliphatic link (C-H) due to the oley chloride reaction (Corrales et al. 2002). It can be noted that there is an absorption band at around 1730 cm^{-1} and 1240 cm^{-1} for the untreated jute fibers, which no longer exists for JGS fabrics. The reason of disappearing of the peak at 1730 cm^{-1} and 1240 cm^{-1} is the same as described above for the JAS fabrics. The JGS fabrics show the signal at 1503.54 cm^{-1} that corresponds to NH modes in the chemical structure of treated fibers. Therefore, it can be said that presence of sodium hydroxide can fix Glytac into the jute fibers through the reaction of Glytac and reactive dye molecule so that dye uptake of JGS5 fabrics have increased significantly.

Figure 4.24 shows the comparison of the FTIR spectrum of JI and control fabrics. In the FTIR spectra, JI4 and JI5fabrics have the highest percentage transmittance i.e. lowest absorption value in the hydroxyl region among the fabrics including untreated jute fabrics. The increase of the peak at 2924 cm^{-1} in the JI fabrics suggests an increase of

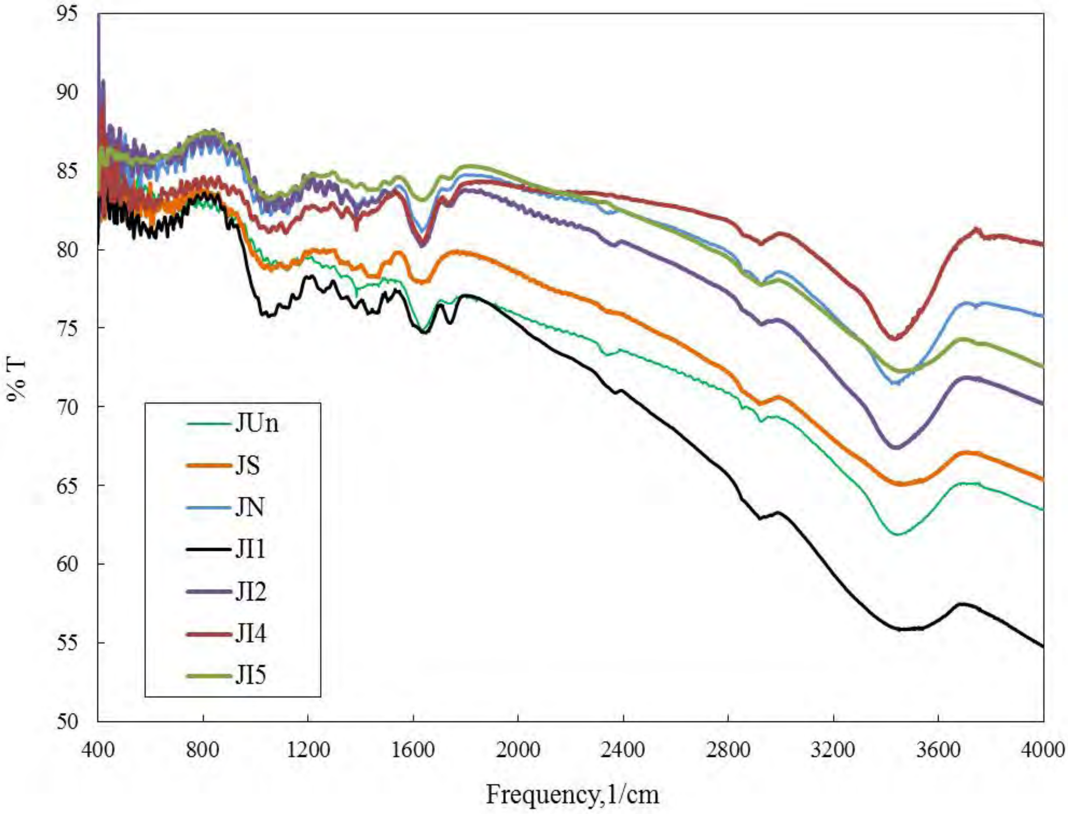


Figure 4.24: FTIR spectra of JI and control fabrics.

aliphatic link (C-H). Relatively strong peak observed in the JI1 fabrics at 1740 cm^{-1} indicates C=O vibration. The JI1 and JI5 fabrics show the signals at 1502.57cm^{-1} and 1509.32 cm^{-1}, respectively, which can be attributed to NH modes in the chemical structure of treated fibers as mentioned above.

4.2.4 Conclusions

All the treated plain jute fabrics of Table 4.4 were characterized by water absorbency, fabric strength, and FTIR tests. Application of Indosol E-50 changes the hydrophilic nature of the jute fabrics significantly. Along with Indosol E-50 treatments, few other cases of treatments as well rendered the fabrics surface more hydrophobic than those of raw jute fabrics. There were no adverse effects on tensile strength of the fabrics as a result of new treatments. FTIR test results confirm the changes the functionality of jute fibers which might improve the adhesion properties between jute fabrics and PP matrix. These results have demonstrated a new approach to modify jute fiber as a reinforcing material in polymer composites.

4.3 Most Effective Treatment Procedure for Dyeing of Jute Fabrics

All the dye treated plain jute fabrics (subcategory 2 to 5) of Table 4.4 was considered for the analysis of their dye performance in an attempt to select the most effective dye treatment procedure. As mentioned before, the fabrics of subcategory 2 to 5 of each major chemical group JA, JAS, JG, JGS, and JI correspond to treatment of fabrics in the presence of Drimarene Red K-8b (reactive dye). To observe the effects of Albafix WFF, Glytac, and Indosol E-50 on the dyeing performance, the normal dyed jute fabrics JN (jute fabrics dyed with the mixture of 1% Drimarene Red K8b, sodium chloride (40g/l), and sodium carbonate (10g/l)) were considered here as the control sample. Dyeing performance of all the dye treated plain jute fabrics are discussed in the subsequent sections.

4.3.1. Effects of treatments on exhaustion *(% E)*

The effects of AS2 to AS5 treatments (Table 3.1) at different concentration (10-30 g/l) of Albafix WFF on the dye bath exhaustion of JAS fabrics are shown in Figure 4.25. Also, shown in Figure 4.25 are the effects of normal dye treatment on the exhaustion values of JN fabrics. Dye bath solutions before and after dyeing are shown in Figure 4.26 to 4.28. As shown in Figure 4.25, the exhaustion of all the JAS fabrics (treated by

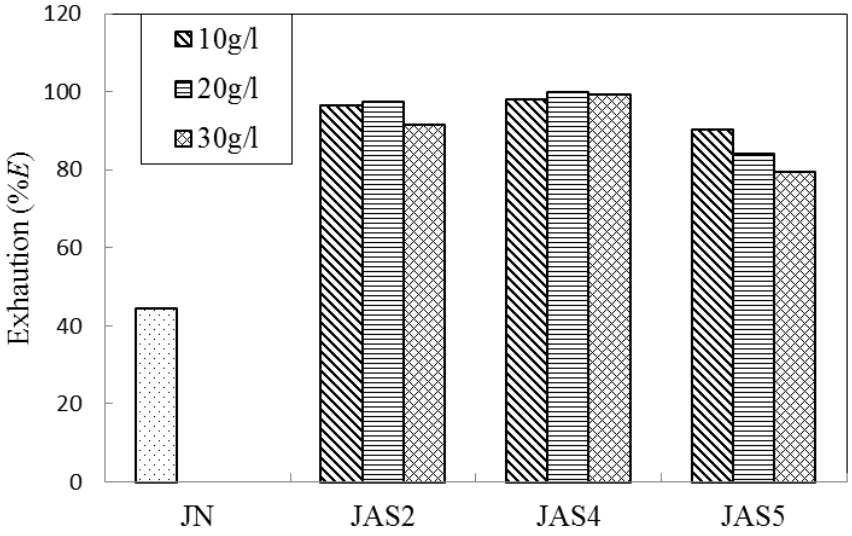


Figure 4.25: Exhaustion of JAS2 –JAS5 and JN fabrics.

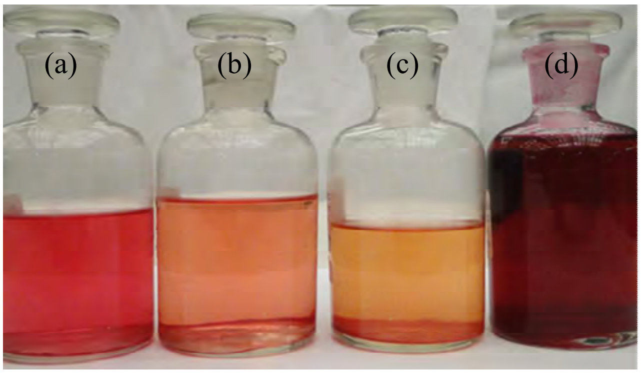


Figure 4.26: Dye bath solution of procedure AS2 after dyeing at the concentration of Albafix WFF (a) 30g/l, (b) 20g/l, (c) 10g/l, and (d) before dyeing.

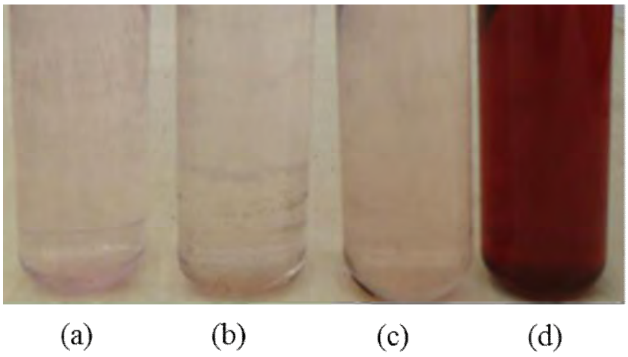


Figure 4.27: Dye bath solution of procedure AS4 after dyeing at the concentration of Albafix WFF (a) 30g/l, (b) 20g/l, (c) 10g/l, and (d) before dyeing.

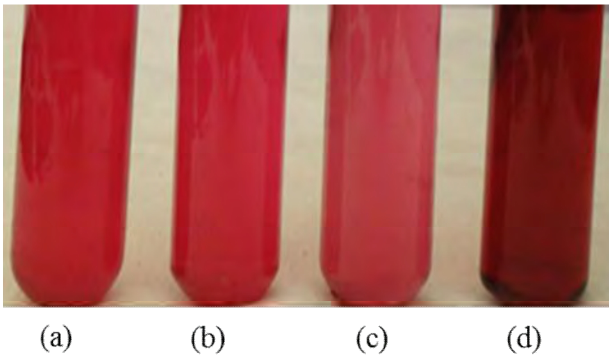
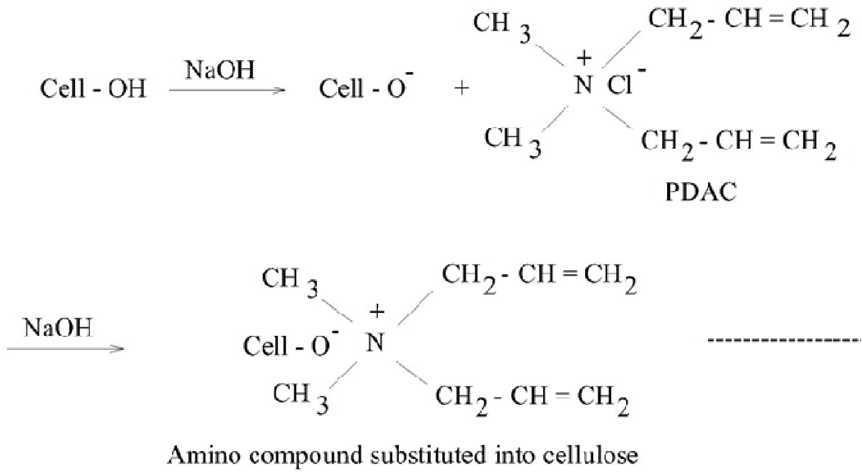
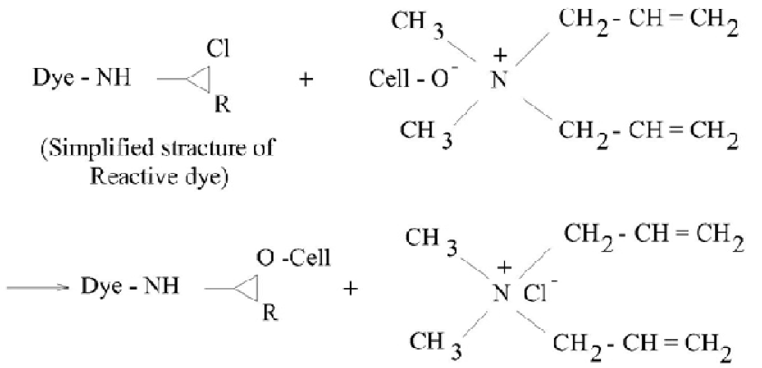


Figure 4.28: Dye bath solution of procedure AS5 after dyeing at the concentration of Albafix WFF (a) 30g/l, (b) 20g/l, (c) 10g/l, and (d) before dyeing.

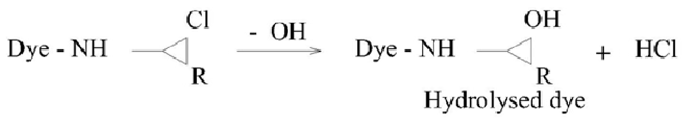
new techniques) is much higher than that of normal dye treated fabrics JN. Specifically, exhaustion of JAS fabrics is above 90%. The exhaustion of Drimarene Red K-8b corresponding to AS4 treatment procedure at 20g/l concentration is above 99% that leaves the dye bath colorless as clearly seen form Figure 4.27. This value is 125% more than that of normal dyeing procedure. The reason for increasing the exhaustion significantly that there is no chance for hydrolysis of dyes due to the absence of salt (NaCl) and alkali (Na_2CO_3) in the dye bath. This may be also explained by the fact that the increase in zeta potential values of the jute fibers diminished the electrical repulsion between jute fibers and anionic dyes (Wang et al., 2010). On the other hand, by introducing quaternary amino compound, jute fiber would be cationized giving a high substantivity for anionic dyes because of Coulombic attraction between the positive charge on fiber and the negative charge on anionic dyes. Possible reaction mechanism among cellulose (jute fiber), Poly- diallyl- dimethylammonium chloride (PDAC or Albafix WFF) and reactive dye (Drimarene Red K8b) can be explained by the scheme 4.1 to 4.4 below. Therefore, modification of jute fabrics with the mixture of Albafix WFF and sodium hydroxide can increase the usage of reactive dye significantly.



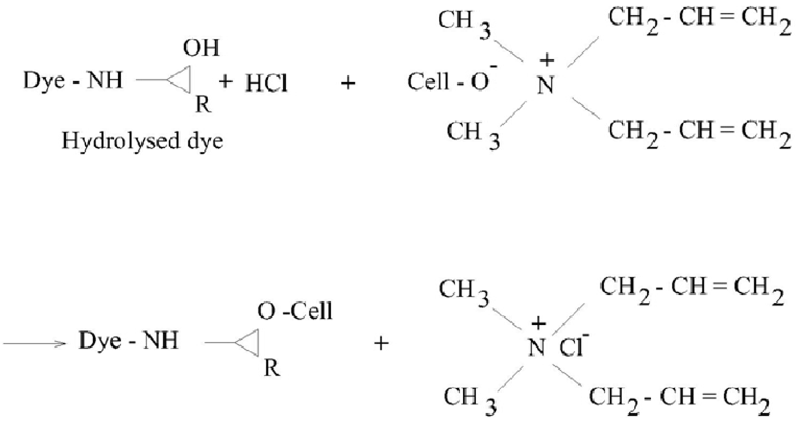
----------------- Scheme 4.1



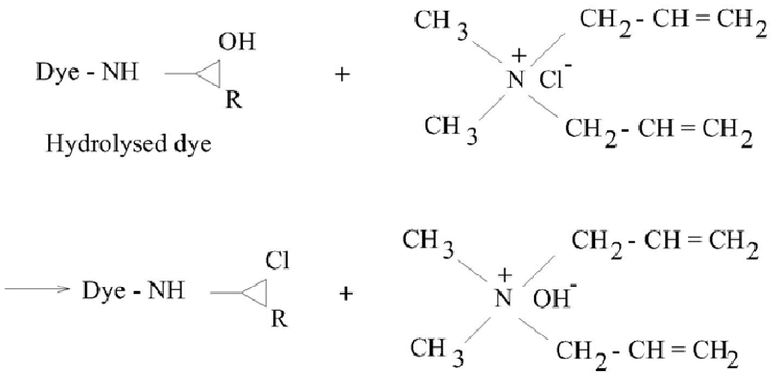
----------------------------Scheme- 4.2



Side reaction of Reactive dye



---------------------------Scheme- 4.3



---------------------------Scheme- 4.4

Table 4.6: Effects of AS treatments on dyeing performance of jute fabrics

Sample	Concentration of Albafix WFF (g/l)	*K/S* (Before soap)	*K/S* (After soap)	% *E*	% *F*	% *T*
JN	-	22.49	17.52	44.44	77.90	34.62
JAS2	10	41.10	39.90	96.58	97.08	93.76
	20	41.69	41.16	97.26	98.72	96.02
	30	40.01	38.60	92.50	96.48	89.24
JAS4	10	41.99	40.67	98.15	96.87	95.06
	20	43.94	42.66	99.88	97.09	96.97
	30	42.08	40.19	99.34	95.50	94.88
JAS5	10	38.12	36.72	90.32	96.33	87.00
	20	34.03	32.52	84.23	95.55	80.49
	30	32.56	29.71	79.30	91.25	72.36

4.3.2 Effects of treatments on total dye utilization (% *T*)

Table 4.6 shows the Exhaution (%*E*), Fixation (%*F*), and total dye utilization (%*T*) of all the AS dye treated fabrics at different Albafix WFF concentration. It shows that total dye utilization (%*T*) of these dye treated fabrics (JAS) are significantly higher than that of normal dyed jute fabrics. JAS4 fabrics at 20g/l Albafix WFF concentration give the highest %*T* value (96.97), which is 180% higher than that of the normal dyed jute fabrics (JN). On the other hand, JAS5 fabrics at 30g/l Albafix WFF concentration give the lowest %*T* value (72.36), which is still 109% higher than that of the normal dyed jute fabrics. Hence, dyeing with reactive dye in the presence of Albafix WFF and sodium hydroxide increases the dyeability of jute fibers that can save approximately 62% dye.

4.3.3 Color strength (*K/S*)

The color strength of all the dye treated jute fabrics of Table 4.4 was evaluated in terms of *K/S* values from the Kubelka- Munk function (Eq. 3.2), where the reflectance *R* was measured with a Minolta Spectrophotometer. Higher value of *K/S* indicates the higher dye uptake (depth of color) of the fabrics (Yamada et al. 2005). To compare the depth of color of the fabrics, *K/S* value for procedures 2 to 5 of Table 3.1 are plotted in Figure 4.29.

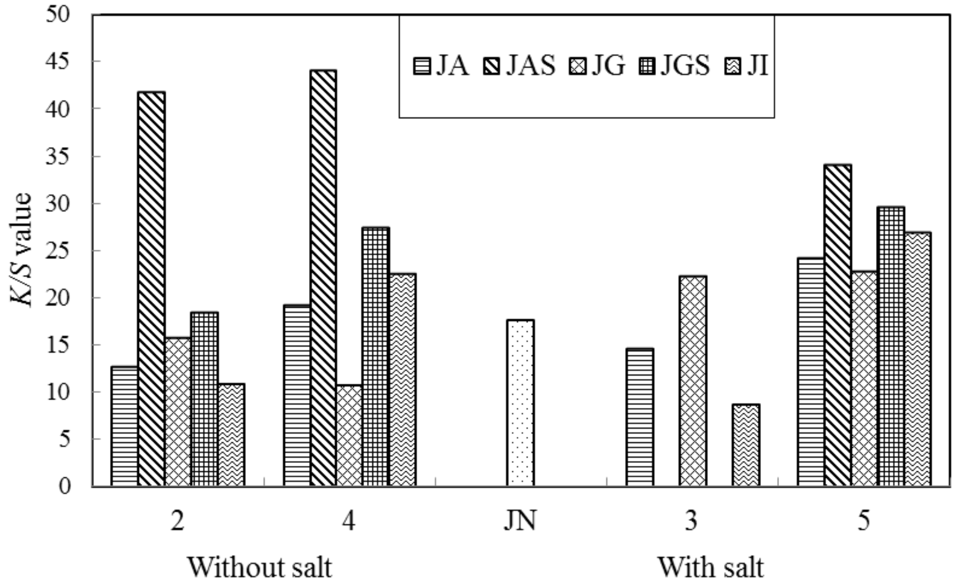
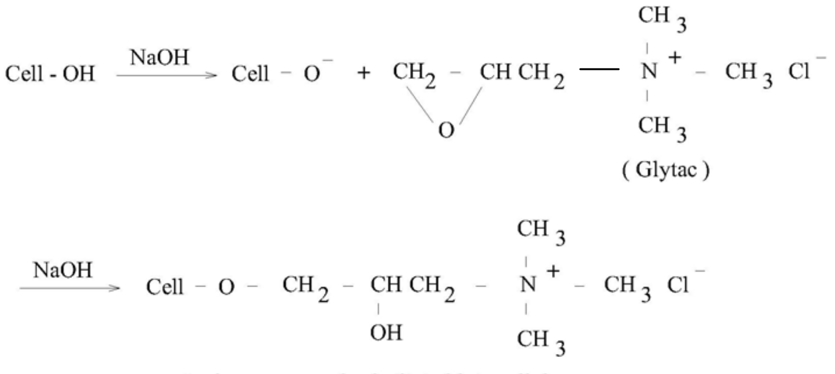


Figure 4.29: Color strength (*K/S*) of different dye treated and normal dyed jute fabrics.

From Figure 4.29, the maximum *K/S* value is found for the sample JAS4 (pretreated jute fabrics with the mixture of Albafix WFF and sodium hydroxide again dyed with Drimarene Red K8b only) among all the samples including normal dyed jute fabrics. There is a significant improvement in *K/S* value of the samples JAS4 and JAS2 which are 95% and 85% higher than that of normal dyed jute fabrics. This tells that without exhausting agent (sodium chloride) and fixing agent (sodium carbonate), jute fiber molecule reacts with dye molecule more effectively for treatment procedures AS4 and AS2 (schemes 4.1 to 4.4). This is due to the fact that the dye reactivity on pretreated fabrics was greater because of the presence of amino groups provided by Albafix WFF (Yamada et al. 2005). Apart from the JAS4, JAS2, and JGS2 fabrics, all the fabrics treated by procedure 5 have the higher *K/S* value than the *K/S* values of other samples including JN. From the comparison between JAS4 and JAS5 fabrics, it is seen that JAS5 fabrics give 22% less *K/S* value than that of JAS4 fabrics. This decrease in *K/S* value may be explained as below.

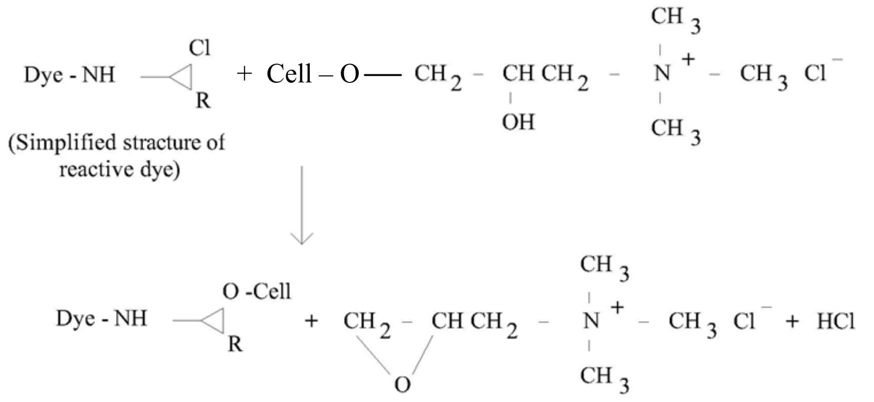
For JAS5 fabrics, (treatment with the mixture of Drimarene Red K8b, sodium chloride, and sodium carbonate of the fabrics pretreated with Albafix WFF and NaOH), strong repulsion forces may exist between fiber and dye molecule in the dye bath which would lead to hindering the absorption of dye. It is also noticed that JA5 fabrics have the maximum *K/S* value among all the JA fabrics, which is 38% higher than that of normal dyed jute fabrics. Further, there is a significant change in *K/S* value of the JA4 fabrics, which is 10% higher than that of normal dyed jute fabrics (JN). Thus, it can be said that without exhausting agent (sodium chloride) and fixing agent (sodium carbonate), Albafix WFF treated jute fiber molecule also reacts with dye molecule effectively, so dye uptake of JA4 fabrics is higher than that of normal dyed jute fabrics (JN).

It is also observed from Figure 4.29 that JGS5 fabrics (dyed with sodium chloride and sodium carbonate of the fabrics pretreated with Glytac and NaOH) have 69% higher *K/S* value than that of normal dyed jute fabrics. Also, JGS2 and JGS4 fabrics (dyed without sodium chloride and sodium carbonate) have the higher *K/S* values, which are respectively increased by 5% and 56% from that of JN fabrics. It is also noted that there are no significant changes between the *K/S* values of JGS4 and JGS5 fabrics. This is due to the fact that the presence of NaOH and Glytac provides the more amino group into the fiber causing more dye uptake by the jute fabrics. Possible reaction mechanism among cellulose, Glytac, and reactive dye in the presence of NaOH can be explained by the schemes 4.5 to 4.7 below which describe the possible reasons of increasing the depth of the color of JGS jute fabrics. Therefore, pretreatment of fabrics with the mixture of Glytac and NaOH can increase the dye uptake of reactive dye.

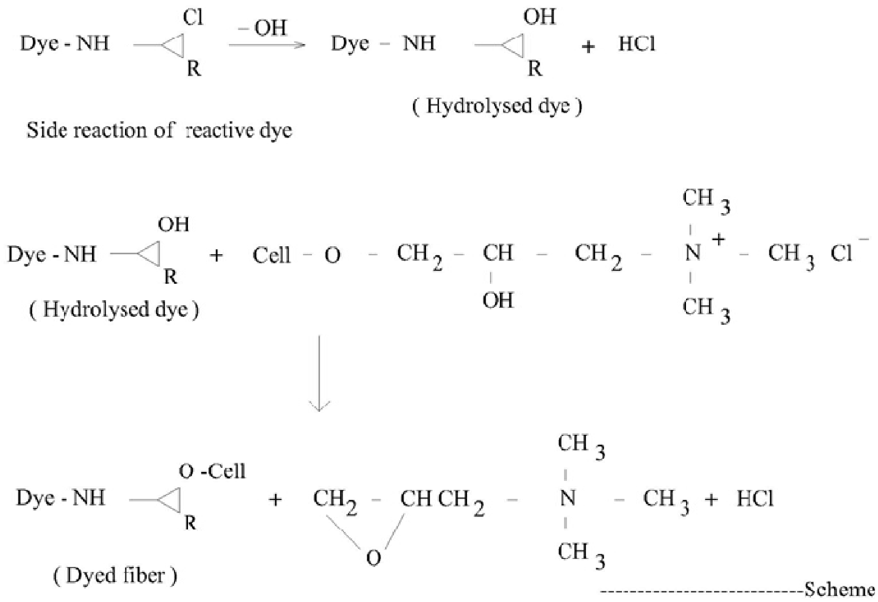


Amino compound substituted into cellulose

--------------------------Scheme 4.5



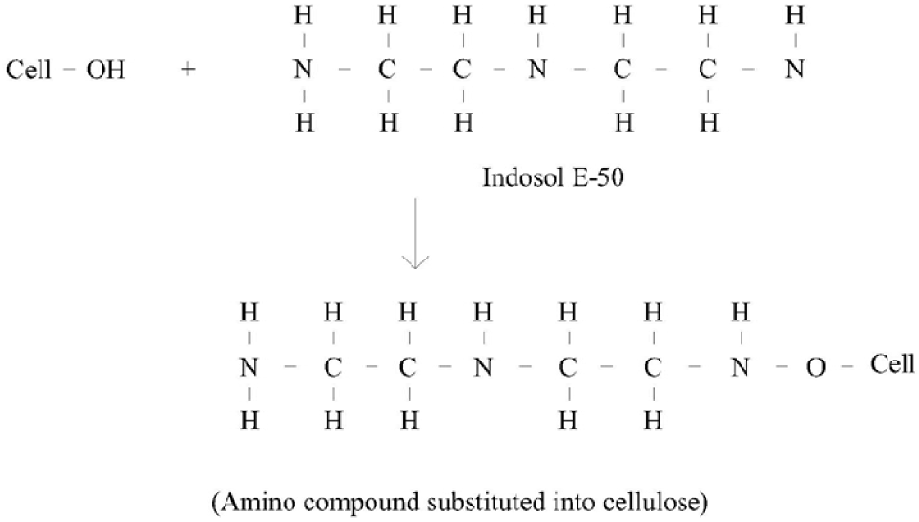
----------------------------Scheme- 4.6



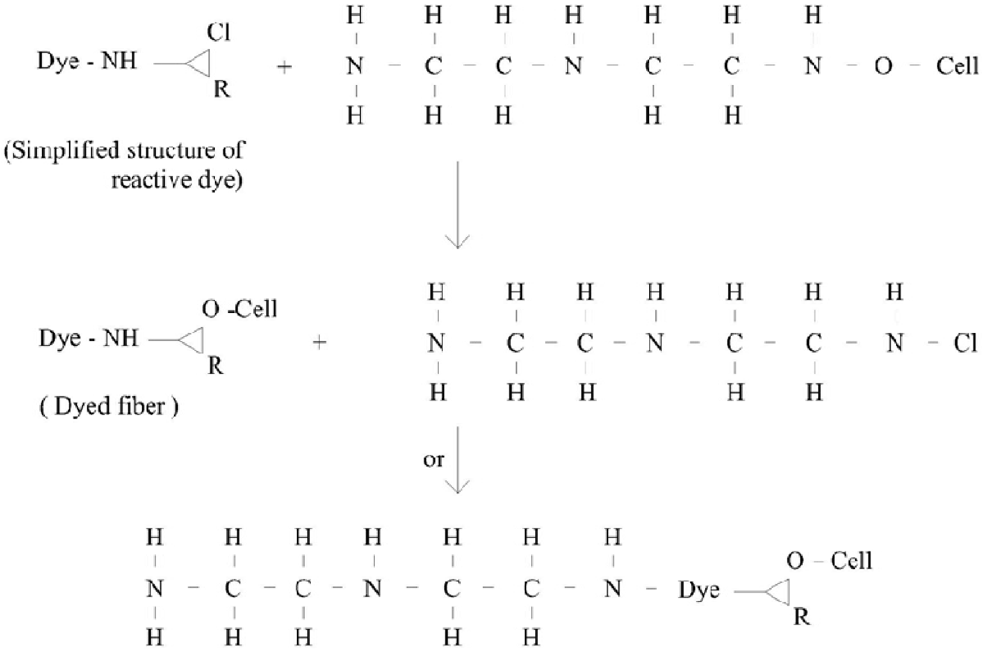
---------------------------Scheme 4.7

It is also noticed that JG5 fabrics have the highest and JG4 fabrics have the lowest *K/S* values among all the JG fabrics including normal dyed fabrics. JG5 fabrics show 30% higher *K/S* value than that of normal dyed jute fabrics. It is also observed that JG3 and JG5 fabrics (dyed along with sodium chloride and sodium carbonate) have almost equal *K/S* values.

Figure 4.29 also shows that JI5 fabrics have the highest and JI3 fabrics have the lowest *K/S* values among all the JI fabrics including normal dyed fabrics. It is seen that JI5 and JI4 fabrics exhibit, respectively, 53% and 28% higher *K/S* value than that of normal dyed jute fabrics (JN). Significantly higher *K/S* values of JI4 and JI5 fabrics can be described by the following proposed reaction schemes.

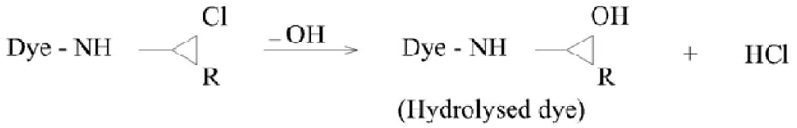


-----------------------Scheme- 4.8

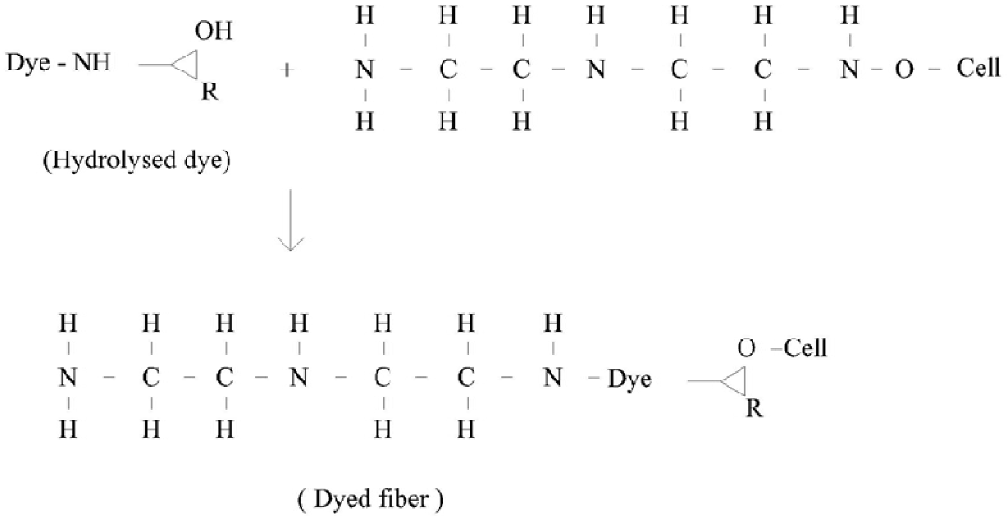


(Dyed fiber)

-----------------------Scheme- 4.9



Side reaction of Reactive dye



--------------------------Scheme- 4.10

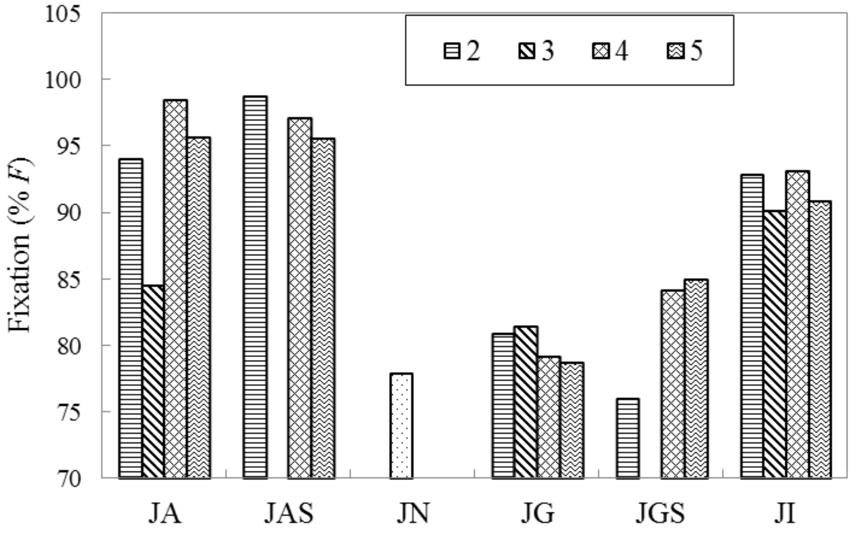


Figure 4.30: Dye fixation of fabrics treated by different procedures.

4.3.4 Fixation properties

Percent fixation of all the fabrics is portrayed in Figure 4.30. The results demonstrate that the treatment groups A, AS, and I have significant influence on the fixation properties of the dyed jute fabrics. In general, most of the treated jute fabrics (except JGS2) have the better fixation value than that of normal dyed jute fabrics. JAS2 fabrics (treated with the mixture of Albafix WFF, sodium hydroxide, and Drimarene Red K8b) have highest fixation value among all the fabrics. This value is about 27% more than that obtained by normal dyeing (JN fabrics). The fixation values of the fabrics JA4 and JA5 are about 26% and 23% more than that of normal dyed jute fabrics, respectively. JI4 fabrics (Indosol E-50 treated jute fabrics again dyed with Drimarene Red K8b) exhibit the highest fixation value (93.11%) among all the JI fabrics, which is about 20% more than that of normal dyed jute fabrics. Increase in fixation value of the fabrics dyed by the present proposed procedures could be explained based on the forces of repulsion and attraction expected to occur during the dyeing process. These forces arise due to the presence of free hydroxyl groups in jute cellulose, anionic groups present in dyes, and amino ions in Albafix WFF or Glytac or Indosol besides other factors (Gupta and Haile 2007). The presence of amino groups on treated jute fibers reduces the repulsion between the free hydroxyl groups of cellulose and the anionic groups of dyes. As a result, most of the dye treated jute fabrics have shown higher fixation.

4.3.5 Wash fastness properties

Table 4.7 shows the wash fastness rating of all the fabrics dyed by both the present proposed and normal procedures. It shows that there is not much difference in case of change in color between the normal dyed fabrics and the fabrics dyed by the proposed procedures. This confirms the effectiveness of dye fixation of the proposed dyeing procedures. JAS5 fabrics show the best wash fastness rating among all the fabrics including normal dyed fabrics. However, JAS2 and JAS4 fabrics show good to moderate rating in both the cases of change in color and staining on cotton and acetate fibers and excellent to very good staining on other fibers. So, it can be mentioned from Table 4.7 that the proposed treatment procedures have no effects on change in color and staining in comparison with normal dyed jute fabrics. It may be due to the formation of strong ionic bond between the fiber and dye as it is equally good for the covalent bond that normally links the dye and fiber. However, in case of staining, most of the fabrics show moderate to fair rating. JG3 fabrics show poor rating of staining on cotton. Further, JGS2 and JGS5 fabrics show good to moderate wash fastness rating in both the cases of change in color and change in staining. Unfix dye removed from the JGS5 fabrics during washing had higher substantivity towards cotton than the dye removed from normal dyed jute fabrics. As a result GS5 treated jute fibers show poor staining on cotton. The wash fastness depends upon the physical and chemical properties of the fibers, the class of the dyes and their forces of interaction, and their interaction with soap solution. Also, it shows that there were not much difference in wash fastness rating of normal dyed fabrics (JN) and I dye treated fabrics (JI2 to JI5). Hence, it can be said that there were no adverse effect on change in color or change in staining by the present treatments. So it can be concluded from Table 4.7 that all the dye treated jute fibers had no noteworthy effects on change in color and staining of multifibers.

Table 4.7: Wash fastness rating of different dye treated and normal dyed jute fabrics

Sample	Wash fastness rating						
	Change in color	Staining on multifibers					
		Acetate	Cotton	Nylon	Polyester	Acrylic	Wool
JN	4-5	4-5	4	4-5	4-5	4-5	5
JA2	4	4	4-5	4-5	5	5	5
JAS2	3-4	4	3-4	4-5	5	4-5	5
JG2	5	4-5	3-4	5	5	4-5	5
JGS2	4-5	3-4	3-4	3-4	4-5	4-5	4-5
JI2	3-4	4	4	4	4-5	5	5
JG3	4	4	2	3-4	3-4	3	4-5
JI3	4	4	3	3-4	4-5	3-4	4-5
JA3	3	3-4	5	4-5	3-4	4-5	3-4
JA4	4	4	4	4	4	5	5
JAS4	4	4	3-4	4-5	5	4-5	5
JG4	3	4	4	4	4	4-5	5
JGS4	4	4-5	4-5	4-5	5	5	5
JI4	3	4	4-5	4	4-5	5	4-5
JA5	4-5	3	4-5	3	4	5	4-5
JAS5	4	4-5	4-5	4-5	5	5	5
JG5	3-4	3-4	3	3-4	4	4	5
JGS5	3-4	4	3	4	4	3-4	5
JI5	4	3-4	3	3-4	4	3-4	4-5

4.3.6 Conclusions

Chemical modification of jute fabrics with Albafix WFF (A), mixture of Albafix WFF and sodium hydroxide (AS), Glytac (G), mixture of Glytac and sodium hydroxide (GS), or Indosol E-50 (I) in the presence of reactive dye (Drimarene Red K8b) with or without salt and soda ash was performed by different treatment procedures of Table 3.1. The effects of different procedures of treatment of jute fabrics on the dyeing performance was investigated and analyzed. The analysis of dyeing performance shows that the AS4 treatment is the most effective treatment procedure among all the proposed dye treatments of Table 3.1 including the normal dyeing procedure. Because JAS4 fabrics (Albafix WFF and sodium hydroxide pretreated jute fabrics dyed with 1% Drimarene Red K8b only) had shown the best dyeing performance in terms of color strength and dye fixation properties among all the proposed dye treated fabrics including normal dyed fabrics. In particular, JAS4 fabrics had 95% and 25% higher color strength and dye fixation than the corresponding values obtained by normal dyeing process. Further, the fabrics dyed by the AS4 treatment procedure exhibited good wash fastness.

4.4. Most Effective Treatment Procedure for Plain Jute Fabrics Reinforced Polypropylene Composites

In this section, experimental results of different properties of plain jute fabrics reinforced PP composites are presented and discussed in detail. To improve the compatibility between the hydrophilic jute fabrics and hydrophobic PP matrix, 23 different treated plain fabrics of Table 4.4 are considered for the reinforcement of PP composites. For convenience and simplicity, the composites reinforced with jute fabrics treated by 23 different procedures are denoted by various symbols. A complete list of symbols of composites of jute fabrics treated by proposed treatment procedures is shown in Table 4.8. Some other symbols corresponding to composites of plain jute fabrics treated by conventional treatment procedures are denoted by CS and CN, which correspond to NaOH treated fabrics and normal dyed fabrics, respectively. Composites of untreated jute fabrics are denoted by CUn. The composites CN, CS, and CUn are used as control samples in this study for the purpose of comparison. The properties of composites are discussed in the subsequent sections.

Table 4.8: Symbols of composites of fabrics treated by proposed treatment procedures

Symbols of groups of composites	Symbols of individual composite				
	1	2	3	4	5
CA	CA1	CA2	CA3	CA4	CA5
CAS	CAS1	CAS2	-	CAS4	CAS5
CG	CG1	CG2	CG3	CG4	CG5
CGS	CGS1	CGS2	-	CGS4	CGS5
CI	CI1	CI2	CI3	CI4	CI5

4.4.1 Mechanical properties

Experimental results of tensile strength of all the composites of treated and control plain jute fabrics are shown in Figure 4.31. It is observed that all the composites of treated fabrics have higher tensile strength than that of composites of untreated fabrics. It is not observed that A5, AS5, G5, GS4, and I2 dye treated jute fabrics/PP composites show the highest value of tensile strength among their respective groups of composites of fabrics treated in presence of dye. Specifically, these composites have, respectively, 43, 73, 36, 19, 27% higher tensile strength than that of composites (CS) corresponding to NaOH treated fabrics. Thus, among all the composites as shown in Figure 4.31, CAS5 composites have the highest tensile strength (44.32MPa), which is 109% higher than that of the untreated fabrics/PP composites, 73% higher than that of the composites (CS) of sodium hydroxide treated fabrics, and 60% higher strength than that of composites (CN) of normal dyed fabrics. When compared only among the composites of fabrics treated without dye, it is seen that composites of AS1 treated fabrics have the highest tensile strength, which is 83% higher than that of the untreated fabrics/PP composites and 52% higher than that of the composites of NaOH treated fabrics. CS (corresponding to treatments without dye) and CN (corresponding to treatment in presence of dye) composites are increased by 21% and 30% respectively, compared to that of the composites of untreated fabrics.

For most of the treated jute fabrics/PP composites, the Young's modulus increases and the elongation at break decreases with respect to the composites of untreated jute fabrics as shown in the Figures 4.32 and 4.33, respectively. CA5 composites exhibit the highest tensile modulus (4.03GPa), which is 93% higher than that of composites (CUn) of untreated fabrics and 56% higher than that of composites (CS) of NaOH treated fabrics as shown in Figure 4.32. CAS1 and CAS5 composites have respectively, 38% and 28% higher tensile modulus than that of composites (CS) corresponding to NaOH treated fabrics.

Since the composites CA5, CAS5, CG5, CGS4, and CI2 corresponding to fabrics treated in the presence of dye have the highest tensile strength, these composites have considered for the analysis of flexural properties. Besides, the composites CA1, CAS1, CG1, CGS1, and CI1 corresponding to fabrics treated without dye are also considered for flexural properties for the purpose of comparison. Figure 4.34 and 4.35 shows the comparison of flexural strength and modulus of these composites, respectively. CA5 and CAS5 composites have almost equal flexural strength (Figure 4.34), which is 26% higher than that of the composites of untreated jute fabrics and 14% higher than that of CS composites. The highest flexural modulus (4.73 GPa) is observed for CAS5 composites (Figure 4.35), which is 76% higher than that of composites of untreated jute

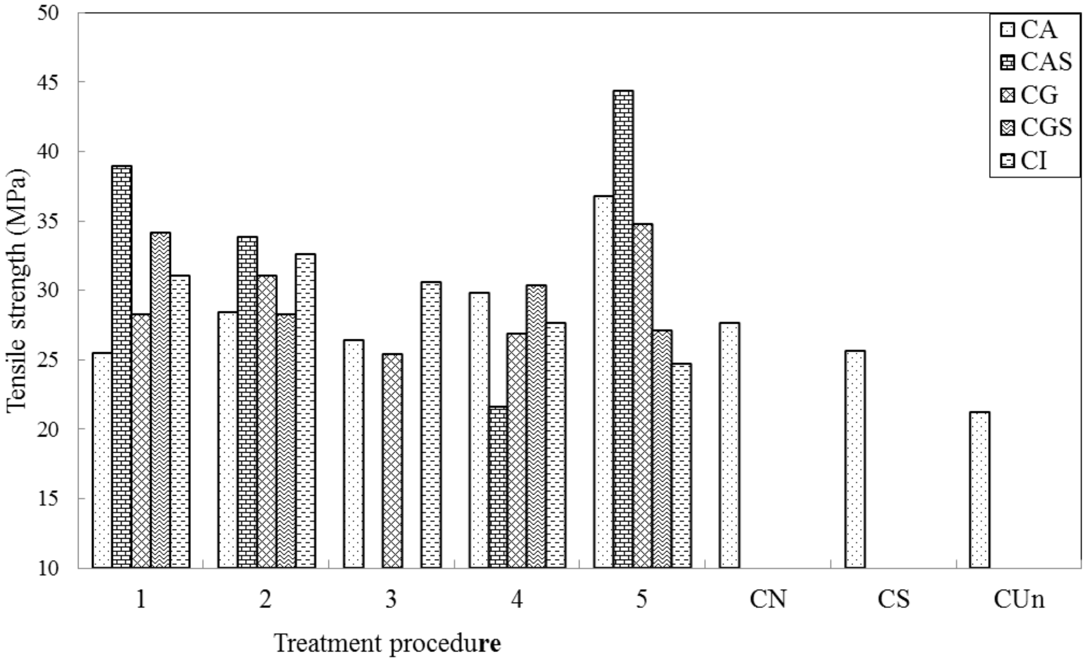


Figure 4.31: Comparison of tensile strength of all the treated and control plain jute fabrics/PP composites.

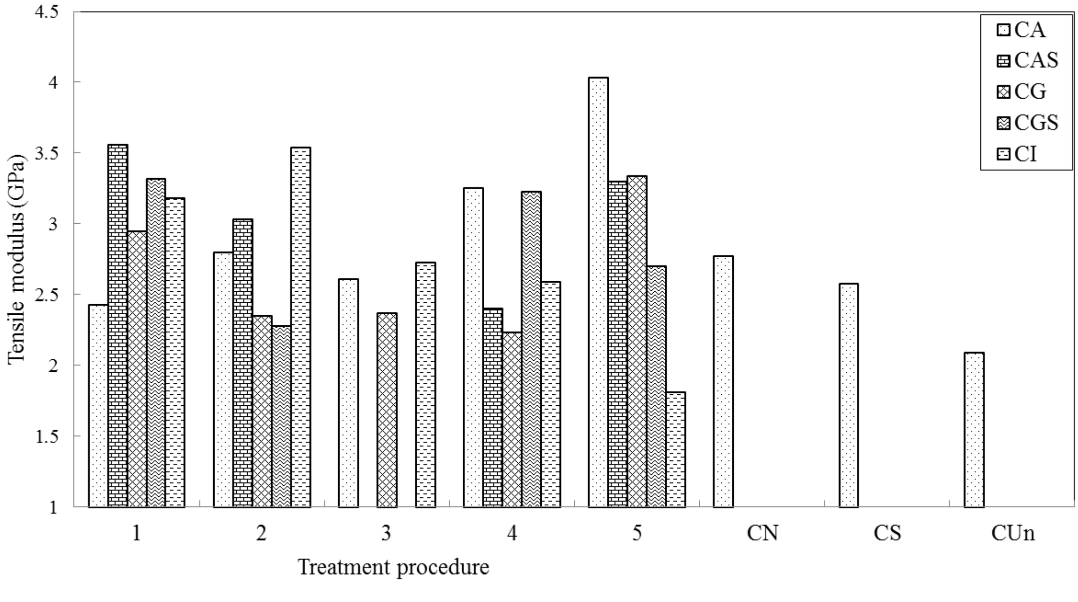


Figure 4.32: Comparison of tensile modulus of all the treated and plain jute fabrics/PP composites.

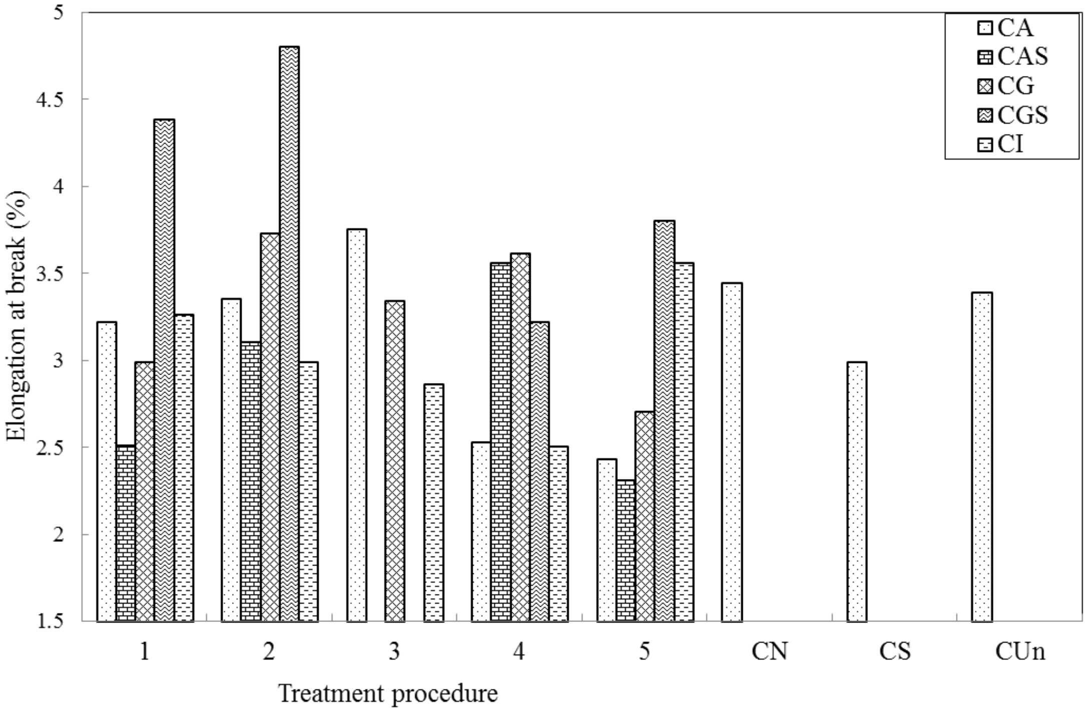


Figure 4.33: Comparison of elongation at break of all the treated and control plain jute fabrics/PP composites.

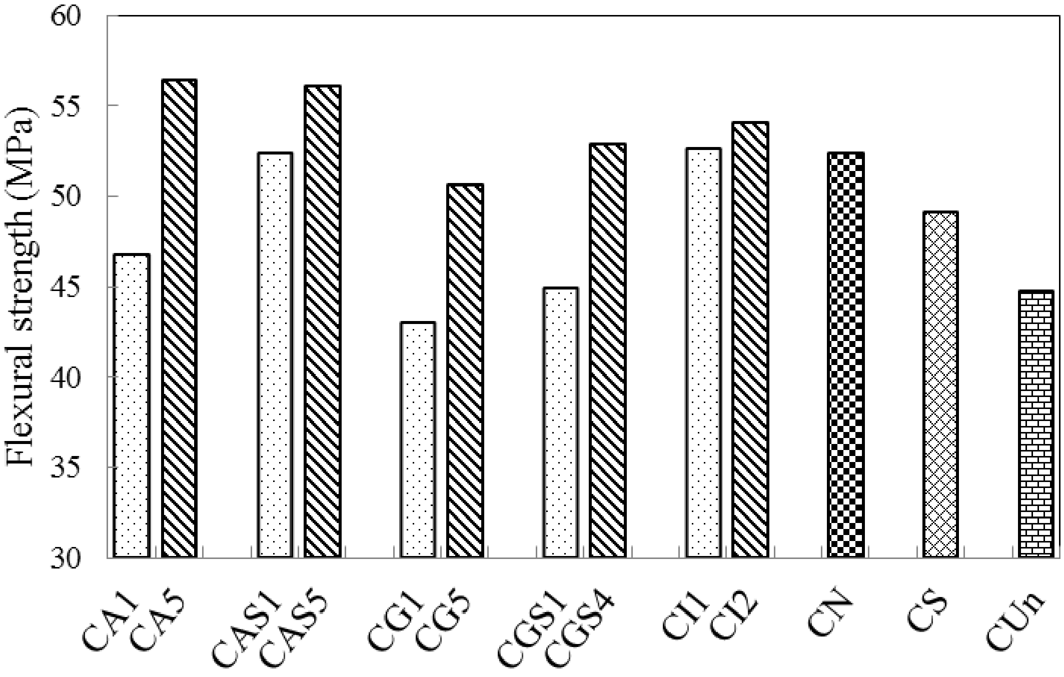


Figure 4.34: Comparison of flexural strength of treated and control plain jute fabrics/PP composites.

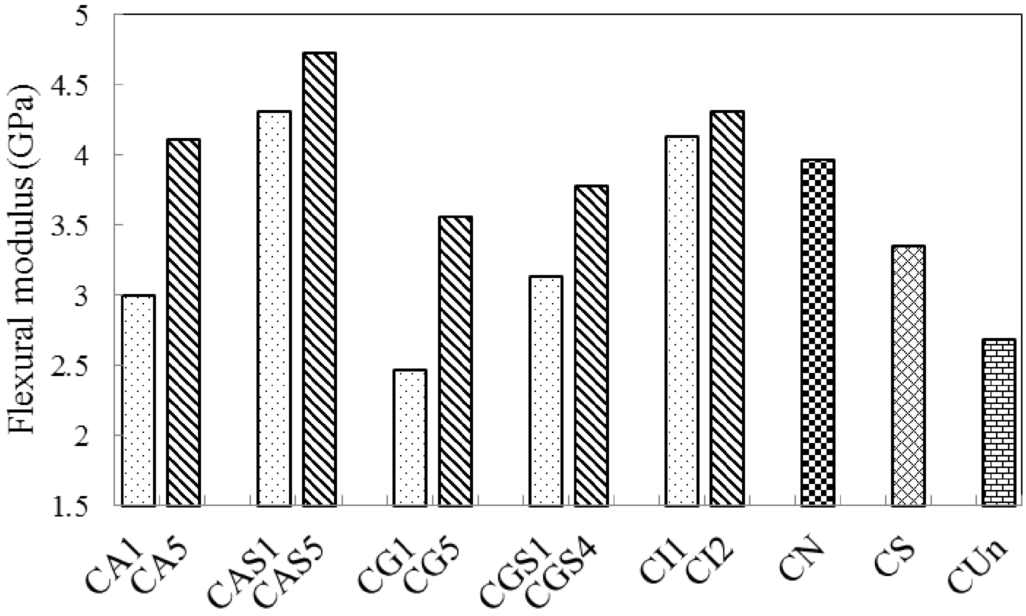


Figure 4.35: Comparison of flexural modulus of treated and control plain jute fabrics/PP composites.

fabrics and 29% higher than that of composites (CS) of NaOH treated fabrics. Also, this value is 19% higher than that of composites (CN) of normal dyed fabrics.

The effects of these treatments including normal dyeing and NaOH treatments of jute fabrics on the impact strength of composites are shown in Figure 4.36. The maximum value of impact strength was found to be 47% for the CAS5 composites and 27% for the composites (CS) of NaOH treated fabrics.

4.4.2 SEM analysis

SEM micrographs of tensile fracture surface were taken for better understanding of fiber matrix interlocking. It is well known that, with effective bonding of fiber with matrix, strong interfacial adhesion can be achieved and interfacial interactions will result a good mechanical properties for the composites. Therefore, the study of SEM images supports the tensile and impact testing results. In general, the fracture styles of untreated jute

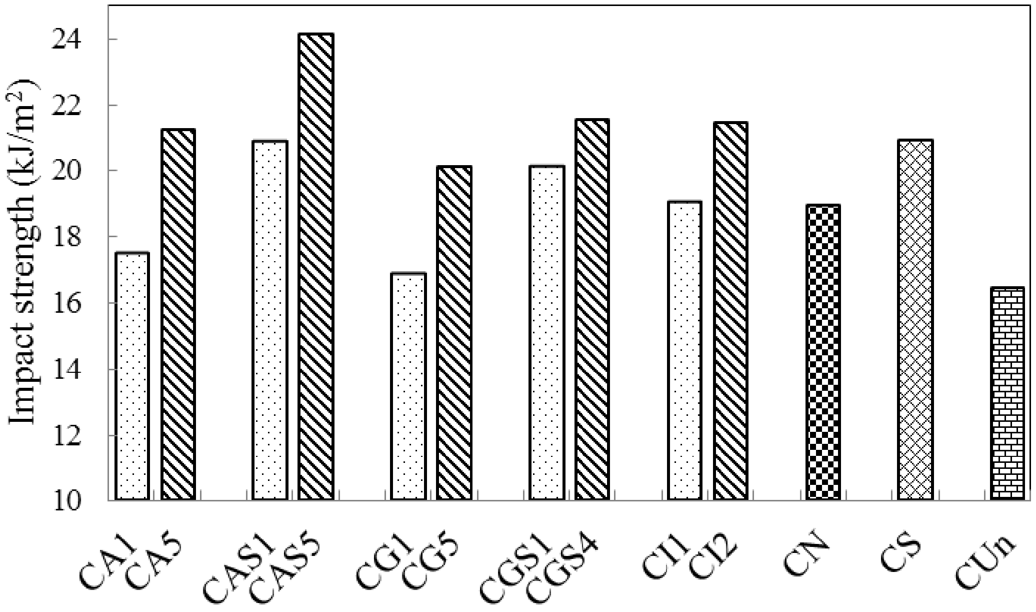
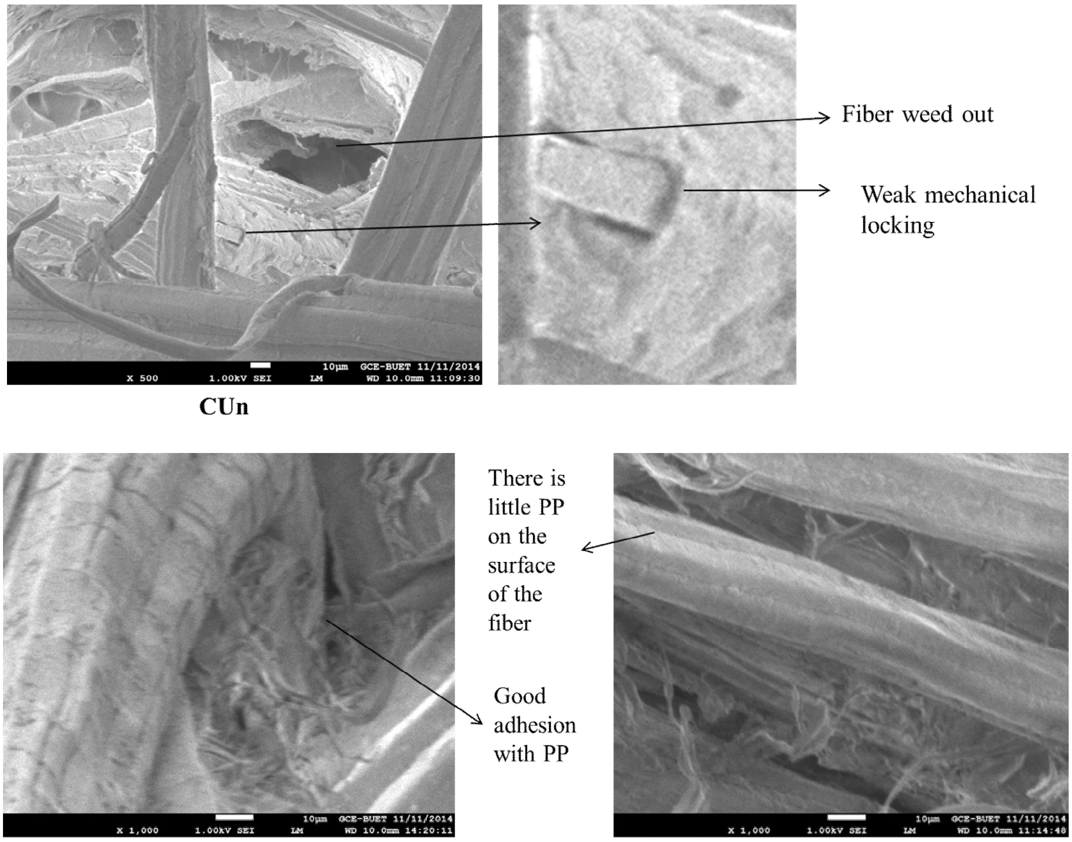


Figure 4.36: Comparison of impact strength of treated and control plain jute fabrics/PP composites.

fabrics reinforced PP composites includ fiber breakage, fiber pull-out, and interfacial debonding. It was clear from earlier discussion that the fiber-matrix compatibility of this kind of composites was poor, a situation which implied that when the stress was applied and the jute fibers would be easily pulled out from the matrix. Therefore, the interfacial structure of this kind of composites could not transfer stress effectively (Yuanyuan et al. 2010).

The SEM micrographs (1000X) of the tensile fracture surfaces of all the control composites (CS, CN, and CUn) are shown in Figure 4.37. Figure 4.38 shows the comparison of SEM micrographs of tensile fracture surfaces of the composites (CA1, CAS1, CG1, CGS1, CI1) corresponding to fabrics treated in the absence of dye and the composites (CA5, CAS5, CG5, CGS4, CI2) corresponding to fabrics treated in the presence of dye. It is clearly shown in the SEM micrographs of the composites of untreated fabrics that the jute fibers were pulled out from the PP matrix during the fracture process. It is clearly observed from the micrograph of CUn composites that the jute fibers easily pulled out



CUn

CN

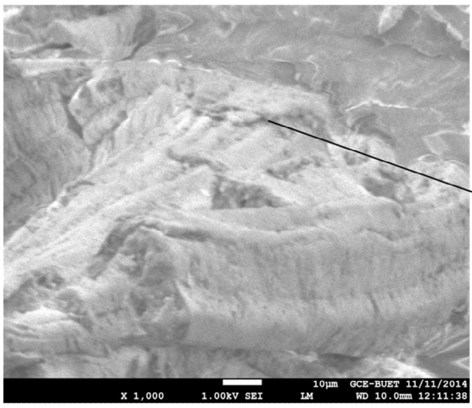
CS

Figure 4.37: SEM micrographs of tensile fracture surface of CUn, CN, and CS composites.

from the matrix and there is little trace of PP on the surface of the NaOH treated fabrics, which indicates no or poor adhesion between fibers and matrix. On the other hand, all the composites of treated jute fabrics have different fracture surfaces, considerable amount of PP matrix remained and adhered to the surface of the jute fibers. It is also observed that the jute fibers failed by tearing, but no complete interfacial failure occurred in the treated fabrics PP composites. This indicates that the adhesion between the treated jute fibers and PP matrix was strong enough as shown in the micrographs of all the treated jute fabrics/PP composites (Figure 4.38). Similar observations of SEM of tensile fracture surfaces of JRPCs were reported in the previous researches by Zhidan et al. 2007 and Yuanyuan et al. 2010). Thus, SEM analysis of tensile fracture surfaces of all the treated fabrics PP composites ensures the improvement of the interaction between the jute fiber and PP matrix in composites. A relatively large amount of PP left on the surfaces of the fiber

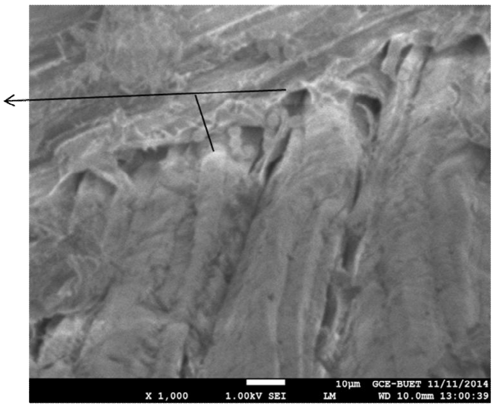
Treatments corresponding to the absence of dye

Treatments corresponding to the presence of dye



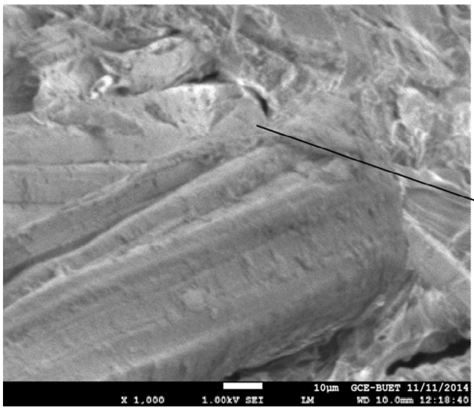
Good adhesion between PP and fiber

CA1



Strong mechanical locking

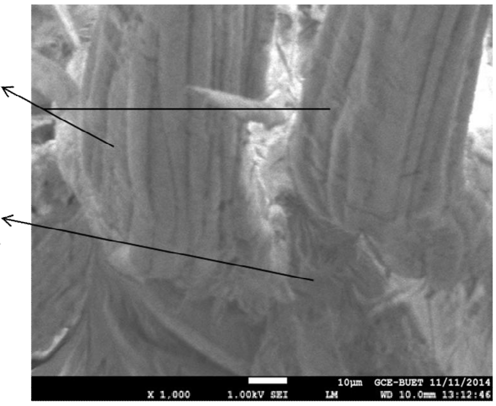
CA5



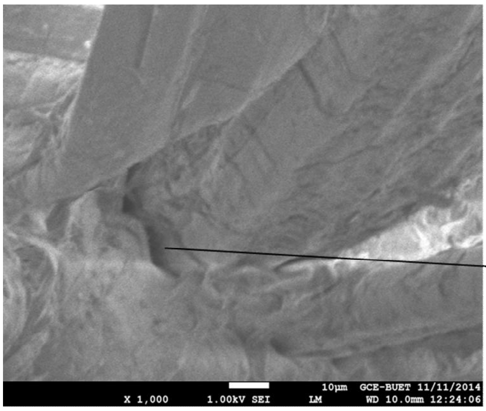
CAS1

Huge PP left over the surface of the fiber

Strong mechanical locking



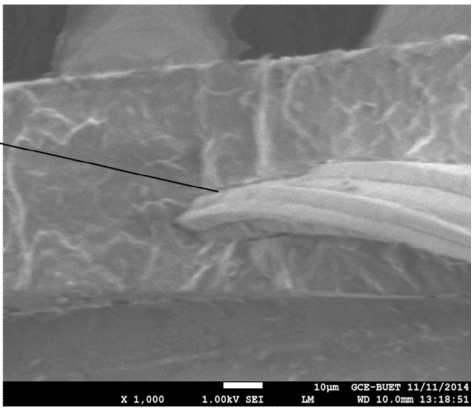
CAS5



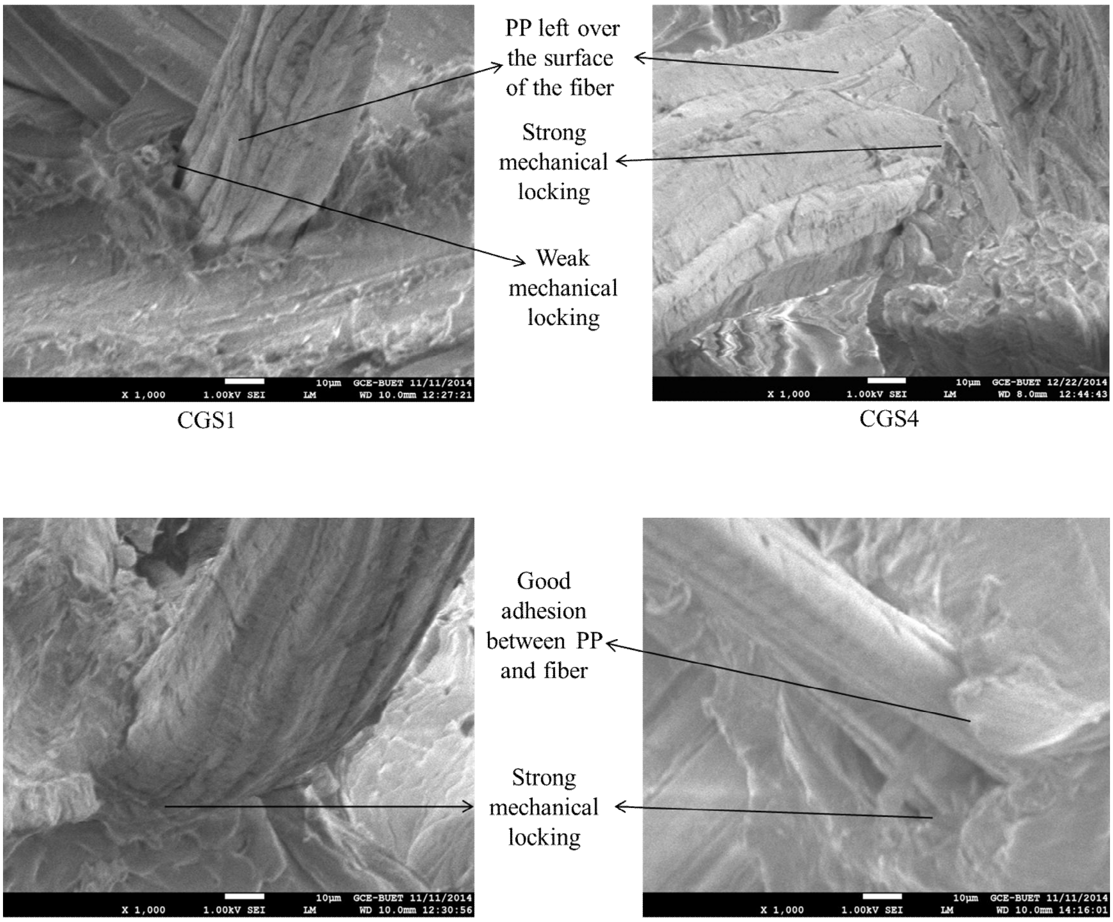
Weak mechanical locking

CG1

Strong mechanical locking



CG5



CGS1 CGS4

CI1 **CI2**

Figure 4.38: Comparison of SEM micrographs of tensile fracture surface of composites with fabrics treated in the presence and absence of dye.

corresponding to the treatment in the presence of dye, specially it is clearly seen in the AS5 treated fabrics, which indicates strong interfacial adhesion between the jute fiber and PP matrix. Therefore, it can be said that the interfacial bonding between the jute fabrics and PP matrix in the composites (CA5, CAS5, CG5, CGS4, and CI2) corresponding to the treatments in the presence of dye is significantly improved than that of the composites (CA1, CAS1, CG1, CGS1) corresponding to the treatments without dye as seen in Figure 4.38. This finding conforms with the results for tensile strength of these composites as discussed in sections 4.4.1.

4.4.3 TGA study

During the processing of jute fabrics reinforced PP composites, jute fabrics were exposed both to high temperature and trapped air, which might cause thermal degradation. If serious degradation of jute fabrics happens during high melt processing temperature, the mechanical reinforcement effects of the fabrics can be much decreased and lead to discoloration as well as unpleasant odor of the composites. Therefore, Thermogravimetric analysis (TGA) was used to determine the high temperature degradation behavior of the composites. Figure 4.39 shows the thermograms of untreated jute fabrics, PP, and untreated jute fabrics/PP composites, in which DTG curve of jute fabrics shows the main three-stage process of untreated jute fiber corresponding to three peaks. The first very small peak $<100^{o}$C corresponds to the heat of vaporization of water absorbed in the fiber. The second peak at about 299^{o}C is due to the thermal degradation of hemicellulose and the glycosidic linkages of cellulose and the very strong third peak at about 377^{o}C indicates the degradation of cellulose, leading to the formation of char. It is observed from the thermograms of PP that a broad single peak starts at around 291^{o}C (visualized at TG thermogram) with a maximum degradation at 432^{o}C at the rate of 1.3 mg/min (DTG thermogram). This peak corresponds to the rupture of C-C chain bonds along with H- bond (Joseph et al. 2003; Doan 2007). PP is completely depleted at 488^{o}C with formation of 13% char residue (TG thermogram). 50% degradation temperature of PP is observed at 417^{o}C (TG data).

The thermal analysis of all the composites reinforced with JA2, JAS4, JG4, JGS4, and JI2 fabrics and control composites were performed in nitrogen atmosphere which is shown in Figures 4.40 to 4.44. Two major peaks of jute fabrics/PP composite are clearly observed in DTG curve of each figure. The thermogram of CUn composites shows the first minor peak at 374^{o}C corresponding to the degradation of cellulose and the second major peak at 437^{o}C mainly for the degradation of dehydro cellulose and PP. However, second major peaks of CA2, CAS4, CG4, CGS4, and CI2 composites are shifted to the higher temperature region with respected to the composites of untreated jute fabrics (Figure 4.40 to 4.44). It is noticed that the TG and DTG curves of the composites of treated fibers shifted to slightly higher temperature region that indicates the higher thermal stability of composites. These are expected results for flame retardant materials since untreated cellulose undergoes decomposition vigorously to volatiles with little char formation, whereas flame retardant cellulose retards decomposition to volatiles to some extent and increase char formation (Lewis et al. 1975; Mark et al. 1975; Krevelin 1975).

Cellulose consists of long and linear homopolymeric chains of glucopyranose units linked together by 1, 4-glucosidic bonds. The cellulose molecule is not planar but has a screw axis, each glucose unit being at right angles to the previous one. After treating with Albafix and/or dye, jute fiber forms octahedral complexes in its structure reducing free rotation about the anhydro glucopyranose C-O-C link, which does not occur due to steric effects in the solid state. The naturally polymerized molecules are thus rigid. The adjacent long chains were held together in the main chain by hydrogen bonds in addition to certain dispersion intermolecular forces. The pyrolytic reaction became complicated and required high energy. It was proposed that besides removing hemicellulose, NaOH treatments can give rise to a lignin-cellulose complex, therefore the NaOH treated fibers become more stable and this is reflected in the increased amount of residual char (Ray et al. 2002). An increased in thermal resistance and residual char formation in the cases of treated fabrics in different treatment procedures and normal dyed fabrics may be caused by the reactions among the active groups in cationic agents (Albafix WFF or Glytac or Indosol E50), dye and hydroxyl groups of the fibers. These results imply the higher thermal stability of the composites of treated fabrics compared with the composites of untreated fabrics. Particularly, I2 treated jute fabrics/PP composites confirm the highest thermal stability among all the composites including control composites. This indicates that jute fibers chemically bonded with matrix require more energy to break the bond having more thermal energy.

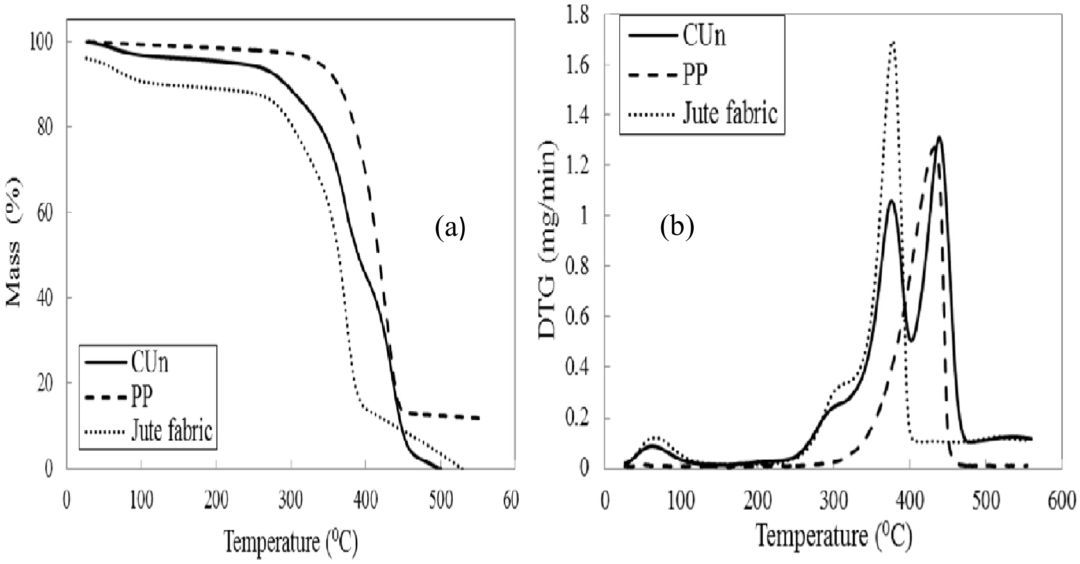


Figure 4.39: Thermograms of jute fabric, PP, and untreated jute fabrics/PP composites (a) TG and (b) DTG responses.

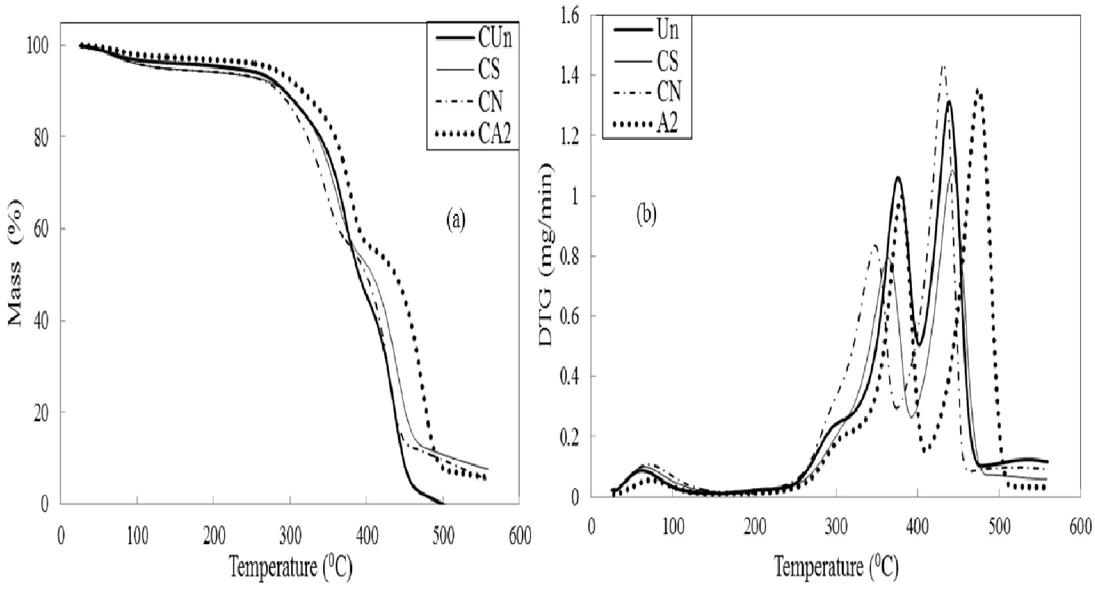


Figure 4.40: Thermograms of CA2 and control jute fabrics/PP composites (a) TG and (b) DTG.

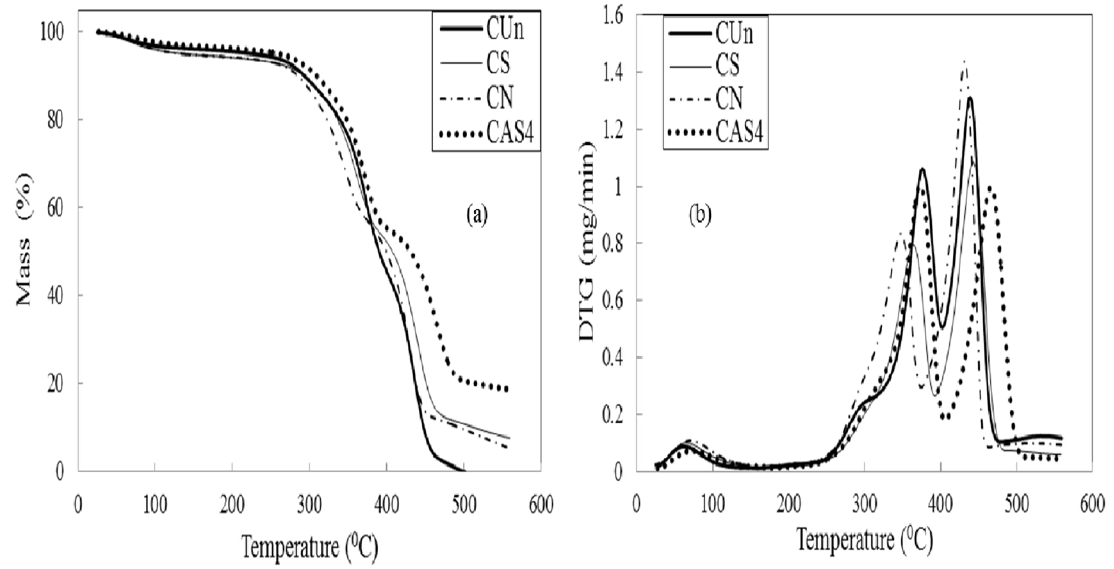


Figure 4.41: Thermograms of CAS4 and control jute fabrics/PP composites (a) TG and (b) DTG.

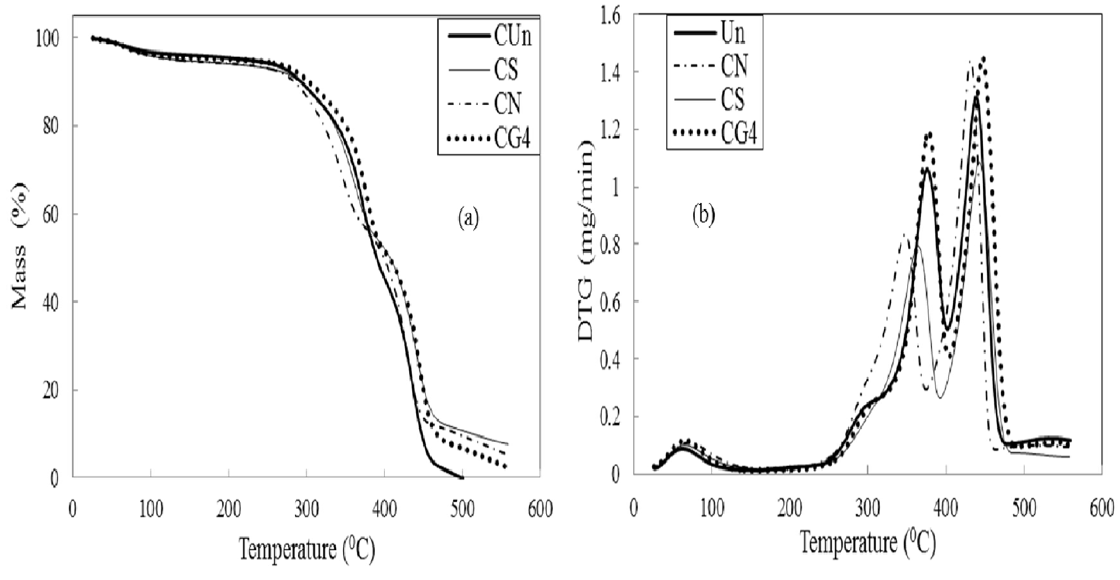


Figure 4.42: Thermograms of CG4 and control jute fabrics/PP composites (a) TG and (b) DTG.

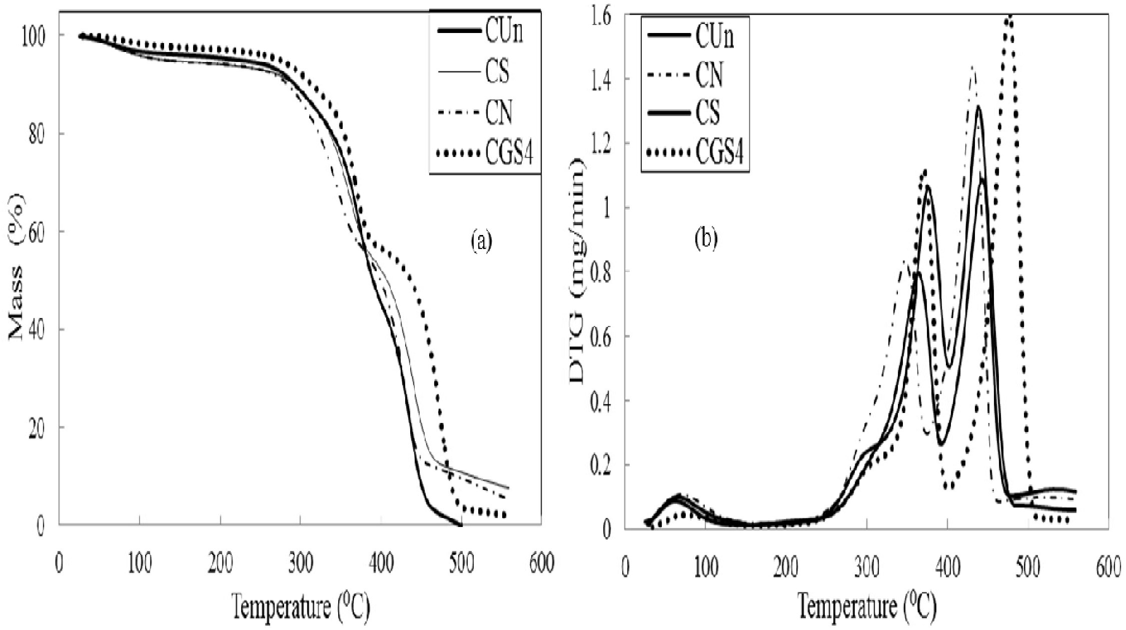


Figure 4.43: Thermograms of CGS4 and control jute fabrics/PP composites (a) TG and (b) DTG.

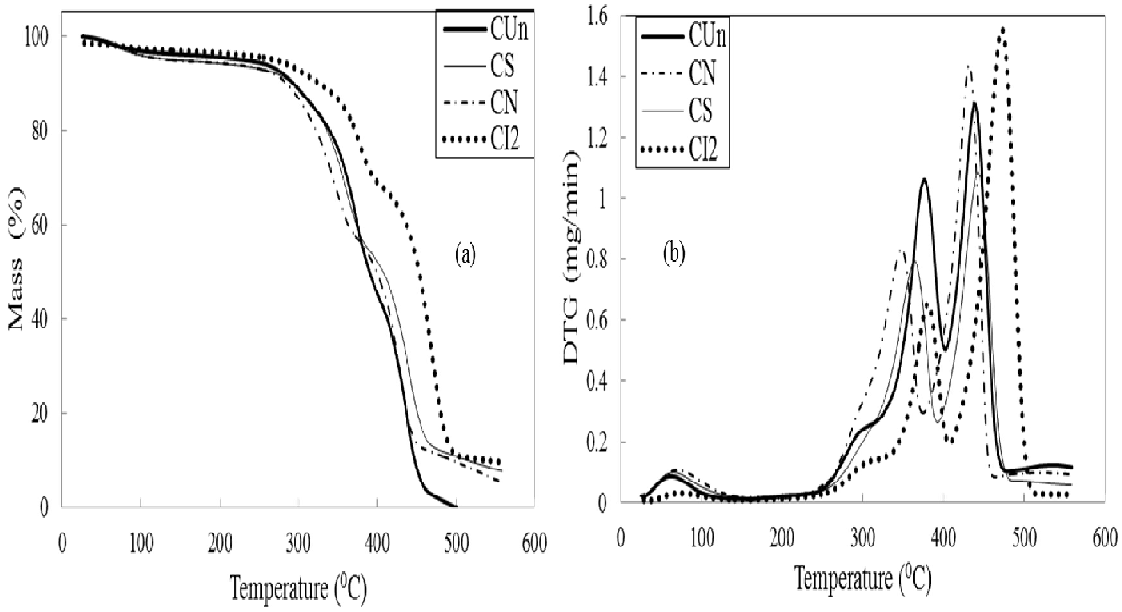


Figure 4.44: Thermograms of CI2 and control jute fabrics/PP composites (a) TG and (b) DTG.

Table 4.9: TG analysis of treated and control plain jute fabrics/PP composites

Sample	50% mass loss temp., °C	Maximum Degradation Temperature (MDT), °C	Residue% at 450°C
CUn	390	437	8
CS	407	443	21
CN	399	432	14
CA2	437	475	45
CAS4	431	466	42
CG4	406	447	20
CGS4	447	476	44
CI2	**453**	**472**	**52**

Table 4.9 describes the TGA data of control composites and those composites which have the highest thermal stability in each group of chemical treatments (A, AS, G, GS, and I). Considering the TGA data given in Table 4.9, it is understood that I2 treated jute fabric composites have the highest thermal stability among all the composites including control composites. Considering the 50% mass loss temperature, maximum degradation temperature (MDT), and % mass residue at 450°C, thermal stability of control and treated jute fabrics/PP composites can be arranged in the order of CUn < CN <CS < CG4 < CAS4 < CGS4 / CA2 < CI2.

4.4.4 Water uptake characteristics

Water uptake characteristics of all the treated and control jute fabrics/PP composites at 24 hours are shown in Figure 4.45. It is observed that the composites of JAS5 and JAS2 fabrics have the minimum water uptake among all the composites including the control ones. The lowest value is observed for CAS5 composites, which is 49% lower than that of the untreated jute fabrics/PP composites and 28% lower than that of the composites of NaOH treated fabrics. It is noted that composites corresponding to fabrics treated in the presence of dye (CAS5, CAS2, CG2, and CI2) have the lower water uptake compared to the composites corresponding to fabrics treated in the absence of dye. All the composites of subcategory 1 and 2, including two of the control fabrics (JN and JS) have lower water uptake than that of the composites (CUn) of untreated jute fabrics. Hence, it can be concluded that most of the treatments are effective for improving the water absorption properties of resulting composites.

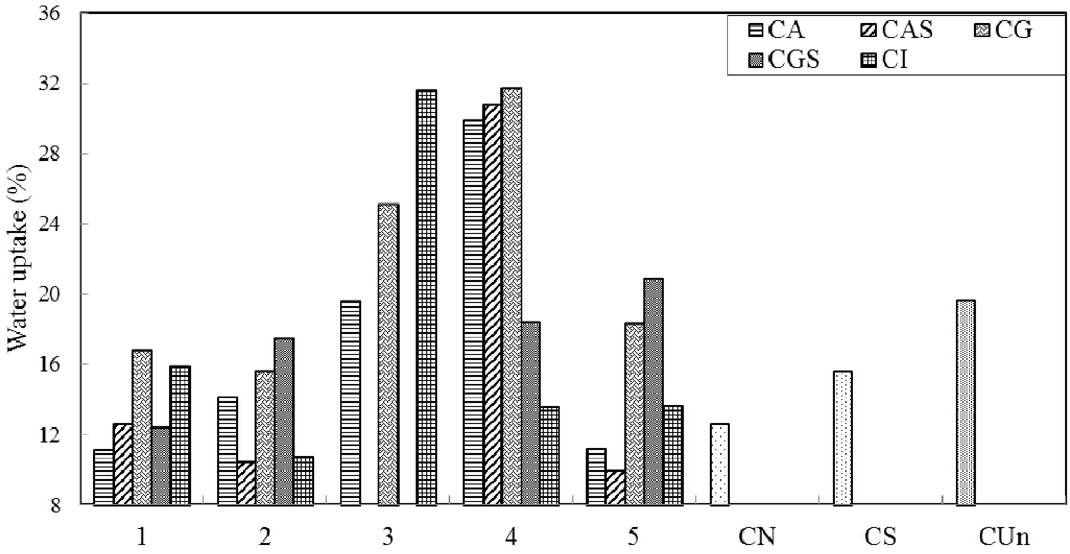


Figure 4.45: 24 hour water uptake of all the treated and control plain jute fabrics/PP composites.

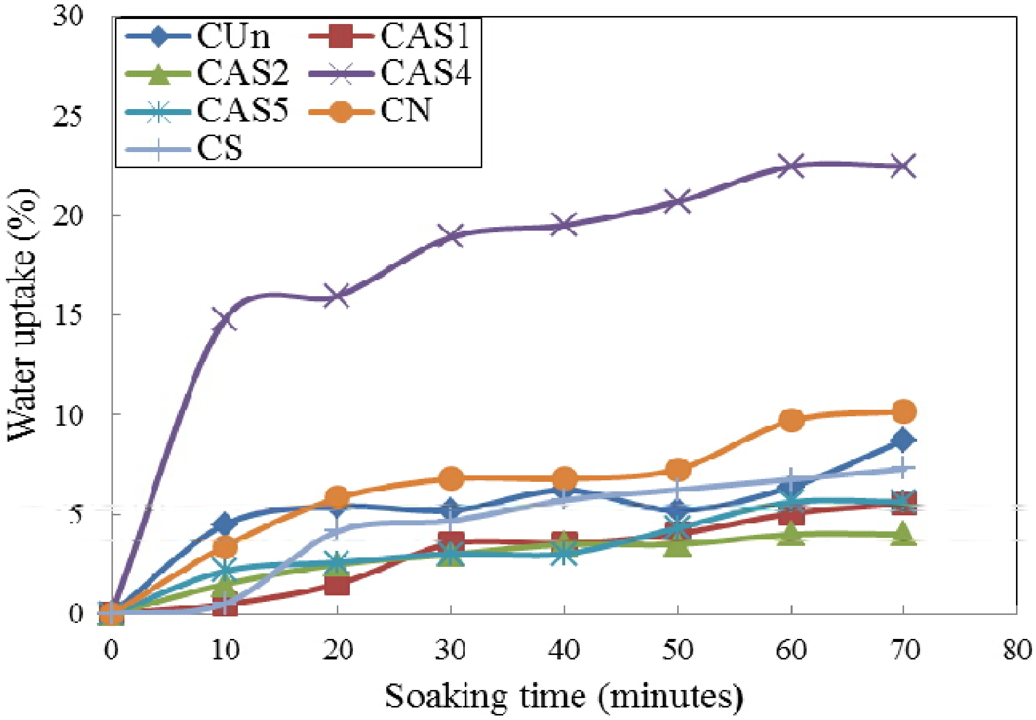


Figure 4.46: Water uptake versus soaking time (minutes) of AS treated and control jute fabrics/PP composites.

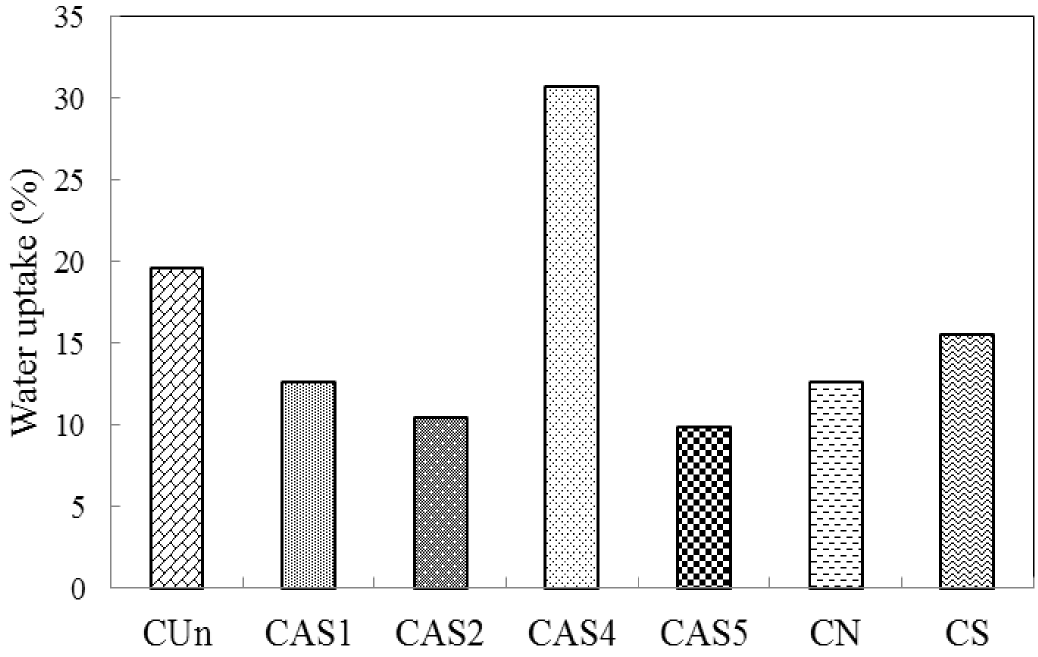


Figure 4.47: 24 hour water uptake of AS treated and control jute fabrics/PP composites.

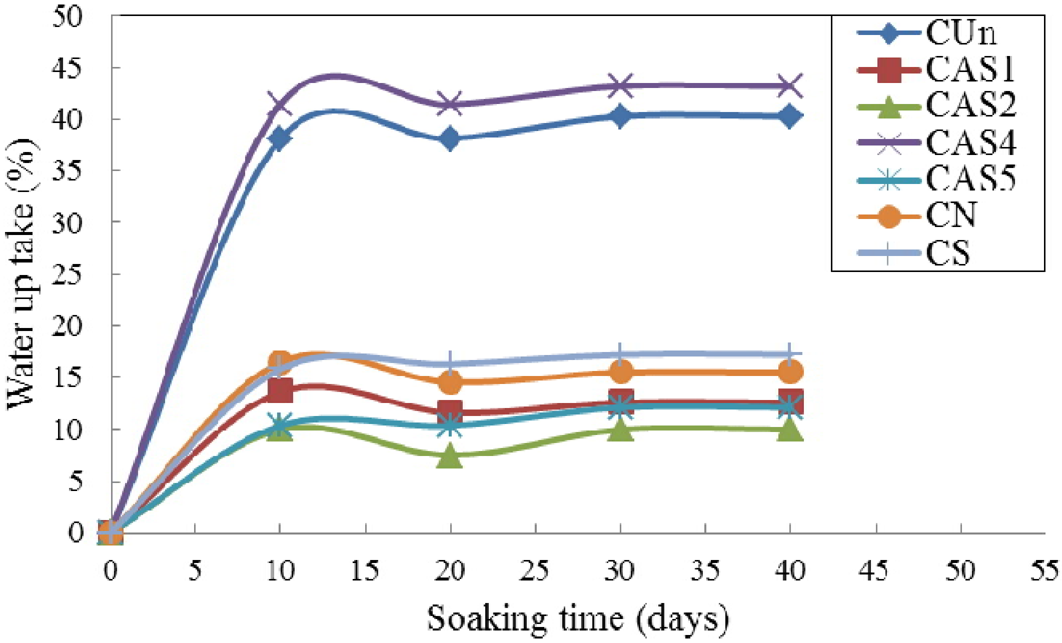


Figure 4.48: Water uptake versus soaking time (days) of AS treated and control jute fabrics/PP composites.

Since CAS5 composites (corresponding to pretreated fabrics again treated with the mixture of NaCl, Na_2CO_3, and Dye) have the lowest water uptake among all the composites as seen from Figure 4.45, composites of these group (corresponding to fabrics treated with Albafix WFF, NaOH, and/or others chemicals) are again considered for the detail analysis of their water uptake characteristics as shown in Figures 4.46 to 4.48. Figure 4.46 shows that the trend of water uptake of all the composites (except CAS4) of JAS fabrics over the short interval of time (up to 70 minutes) is very close to each other. These composites (CAS1, CAS2, and CAS5) have the lower water uptake than that of control composites. But in the long interval of time up to 40 days, water uptake of all the composites (except CAS4) of JAS fabrics differ significantly from the composites of untreated fabrics (Figure 4.47 and 4.48). It is noted that CAS4 composites have the highest and CAS5 composites have the lowest percentage of water uptake among all the composites including control composites. Twenty four water uptake characteristics (Figure 4.47) reveal that CAS5 composites have, respectively, 49% and 28% lower water uptake than that of CUn and CS composites.

4.4.5 Conclusions

Mechanical, thermal, and physical properties of all the PP composites of chemically treated plain jute fabrics are investigated and analyzed in this section. The mechanical properties such as tensile, flexural, and impact strength of the plain jute fabrics/PP composites are influenced significantly by the treatments of fabrics in which Drimarene Red K-8b (reactive dye) was specially included in the mixture of Albafix WFF and NaOH. Composites of JAS5 fabrics had the highest tensile strength, which was 109% higher than that of the untreated fabrics/PP composites (CUn), 73% higher than that of the composites (CS) of NaOH treated fabrics, and 60% higher strength than that of composites (CN) of normal dyed fabrics. CAS5 composites had the maximum flexural strength, which was 26% higher than that of the composites of untreated jute fabrics and 14% higher than that of CS composites. Also, maximum impact strength was observed for CAS5 composites, which was, respectively, 47% and 27% higher than that of CUn and CS composites. From the SEM analysis, it was clearly seen that a relatively large amount of PP left on the surfaces of the AS5 treated fibers corresponding to CAS5 composites, which indicates strong interfacial adhesion between the AS5 treated fabrics and PP matrix. Again, CAS5 composites had shown the best improvement in terms of water uptake properties. It had the lowest water uptake, which was, respectively, 49% and 28% lower than that of the composites of untreated jute fabrics and NaOH treated fabrics. Composites of JI2 fabrics confirmed the best thermal stability among all the composites of treated jute fabrics including control composites. Therefore, it can be concluded from the above discussion that the CAS5 composites corresponding to AS5 treated fabrics had shown the best performance in terms of mechanical and water uptake properties.

4.5 PP Composites of Different Jute Fabrics Treated by the Most Effective Treatment Procedure (AS5)

As mentioned in the preceding section, AS5 treatment is the most effective treatment procedure with respect to the mechanical and water absorption properties of composites of treated plain structure jute fabrics. The AS5 treatment procedure indicates the treatment with the mixture of Drimarene Red K-8b, NaCl, and Na_2CO_3 of the jute fabrics pretreated with the mixture of Albafix WFF and NaOH. Plain, twill, single jersey, and rib jute fabrics of Table 3.2 were prepared from untreated yarn as mentioned in section 2 of chapter three. These fabrics were then treated by AS5 treatment procedure. Composites of these fabrics and PP matrix were prepared by a hot press machine. Effects of AS5 treatment on the properties of jute fabrics of different structures and their composites are discussed below.

4.5.1 Effects of AS5 treatment on the properties of jute fabrics of different structures

4.5.1.1 Fabric strength

The fabric strength of AS5 treated as well as untreated jute fabrics are shown in Figure 4.49. It shows that all the treated fabrics (excluding single jersey) have higher tensile strength than that of corresponding untreated jute fabrics. It is obvious that fabric structure influences the tensile properties of the fabrics, which was described in the section 4.1.

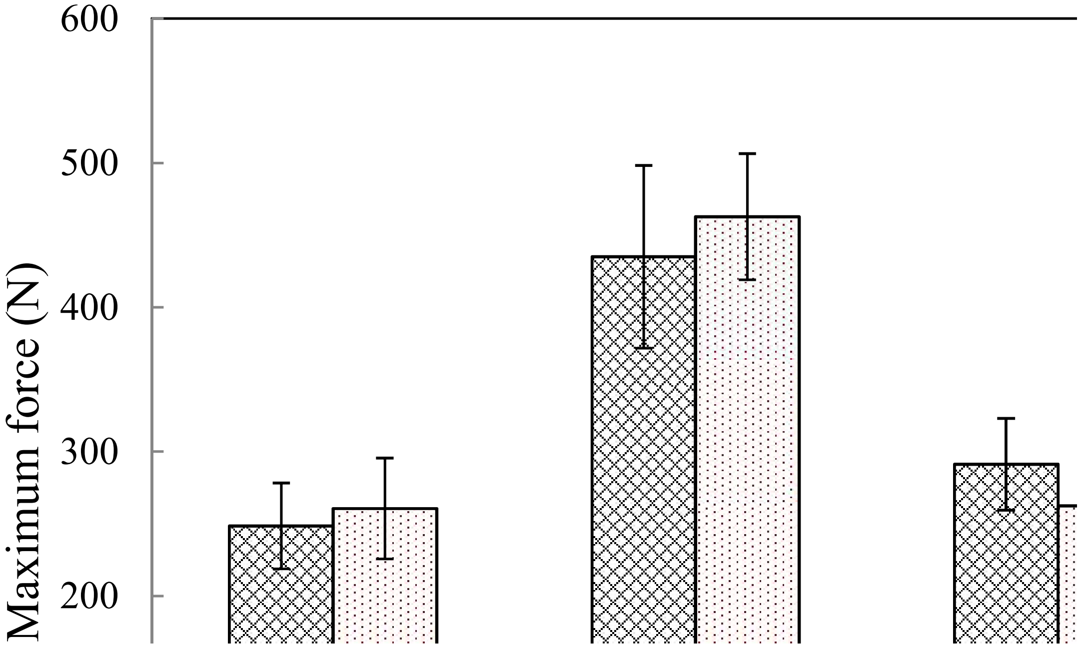


Figure 4.49: Comparison of fabric strength of untreated and AS5 treated fabrics of different structures.

4.5.1.2 Water absorption

Water absorption tests were carried out for all the AS5 treated and untreated jute fabrics having four different structures of Table 3.2. Water absorption properties of plain, twill, single jersey, and rib fabrics are shown in Figure 4.50, which indicate the time required for a drop of water to be fully absorbed by the jute fabrics. Observation of Figure 4.50 shows that single jersey and rib fabrics are influenced by this treatment significantly. On the other hand, twill fabrics are not so much influenced by this treatment procedure. Treated single jersey fabrics turned out to be the least hydrophobic in nature after treatment; because the treated single jersey fabrics exhibited the lowest value of time among all the fabrics. Time required to absorb a drop of water by the treated single jersey and rib fabrics was reduced by 79% and 80%, respectively, compared to the corresponding untreated jute fabrics.

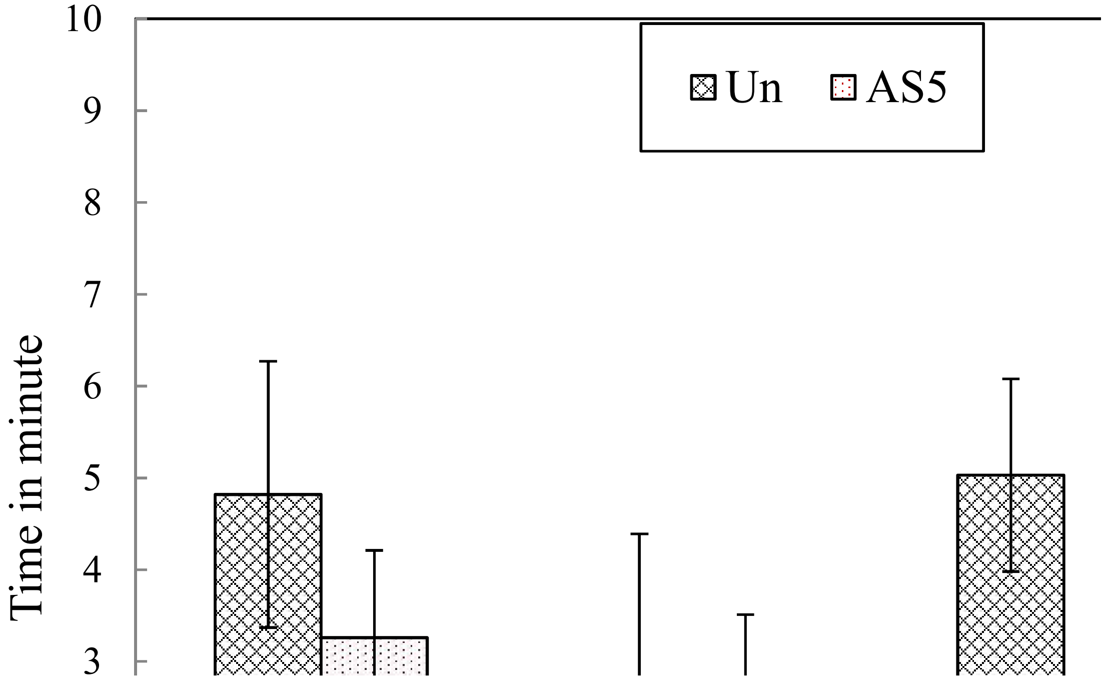


Figure 4.50: Water absorption properties of untreated and AS5 treated fabrics of different structures.

4.5.2 Effects of AS5 treatment on composites having fabrics of different structures

4.5.2.1 Mechanical properties

Tensile properties

The tensile strength of PP composites of untreated and AS5 treated jute fabrics of four different structures are shown in Figure 4.51. It is observed that all the composites of AS5 treated jute fabrics have higher tensile strength compared to the composites prepared with that of corresponding untreated jute fabrics. Treated plain and twill woven fabric composites have, respectively, 88% and 26% higher tensile strength than that of composites of untreated plain and twill woven fabrics. Composites of AS5 treated twill fabrics exhibited the highest value of tensile strength, which was 56% higher than that of treated plain fabrics PP composites.There are almost no changes of

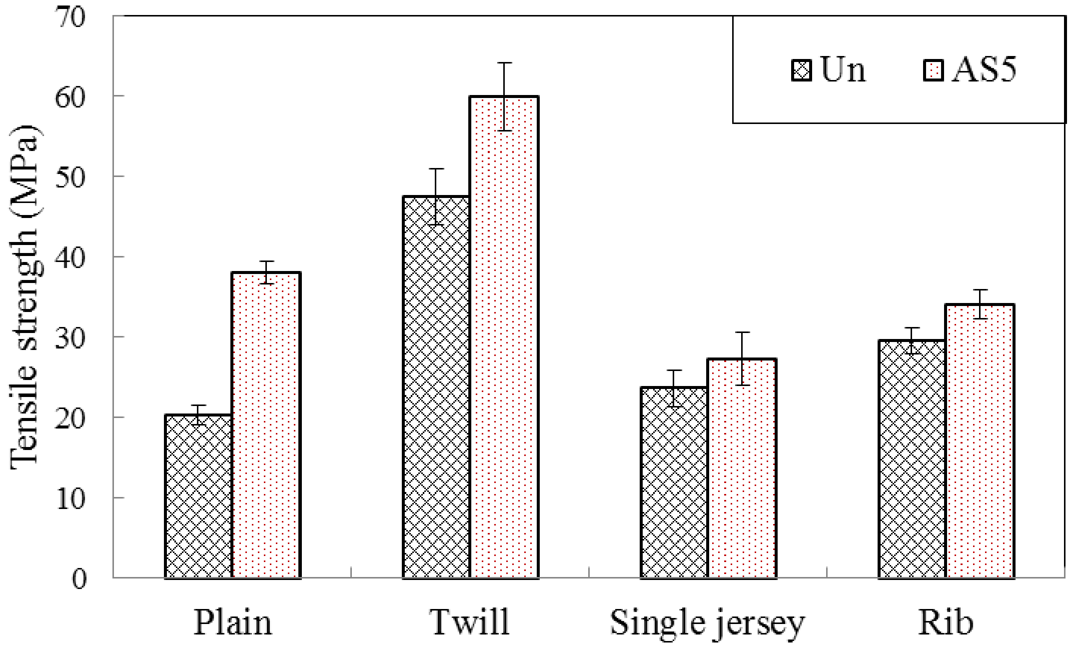


Figure 4.51: Comparison of tensile strength of composites of untreated and AS5 treated fabrics of different structures.

tensile modulus of treated single jersey and rib fabric composites compared to that of composites corresponding to untreated jute fabrics as observed in Figure 4.52. Tensile modulus of PP composites of untreated plain fabrics is found to be 1.25 GPa, which approaches to 3.13 GPa for the composites of AS5 treated plain fabrics. It is obvious that there are no significant changes between elongation at break for the composites of treated and untreated fabrics of different structures as shown in Figure 4.53.

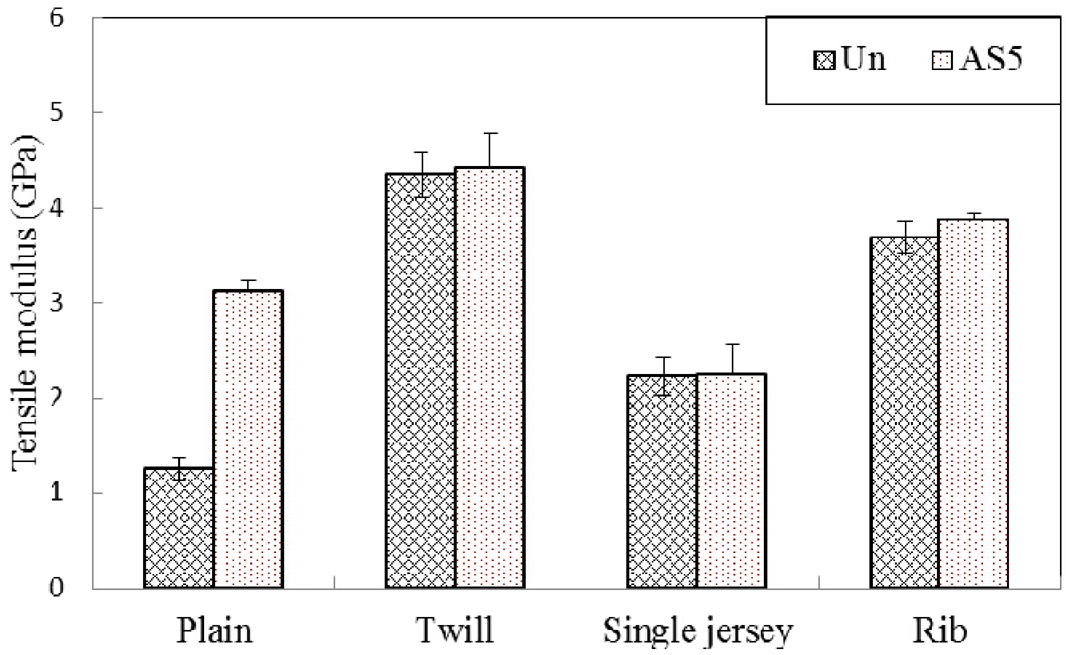


Figure 4.52: Comparison of tensile modulus of composites of untreated and AS5 treated fabrics of different structures.

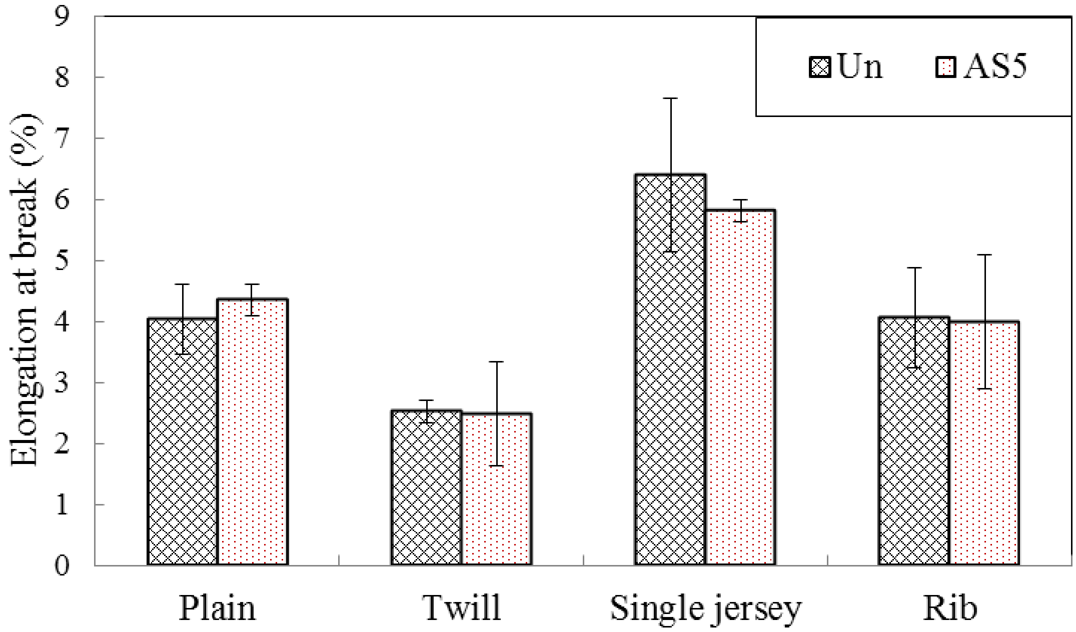


Figure 4.53: Comparison of elongation at break of composites of untreated and AS5 treated fabrics of different structures.

Flexural properties

The flexural strength and modulus of PP composites of untreated and AS5 treated fabrics of different structures are illustrated in Figures 4.54 and 4.55. It is clearly observed that composites of woven fabrics (plain and twill) exhibited better flexural strength and modulus compared to composites of knitted fabrics (single jersey and rib) in both the cases of treated and untreated fabrics. It is also observed that composites of both the treated plain and treated twill fabrics have 7% higher flexural strength than that of composites corresponding to untreated fabrics. Further, composites of treated plain and treated twill fabrics have, respectively, 17% and 27% higher flexural moduli, compared to the composites corresponding to untreated fabrics. So, it can be said that AS5 treatment plays an important role in improving the interfacial bonding between fabrics and PP matrix.

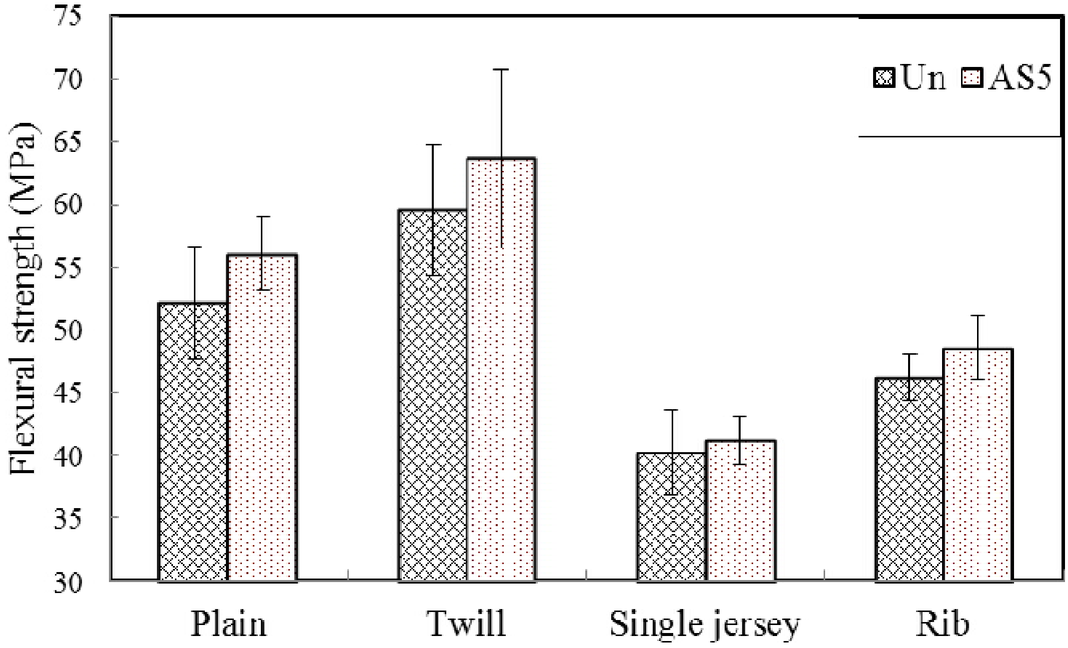


Figure 4.54: Comparison of flexural strength of composites of untreated and AS5 treated fabrics of different structures.

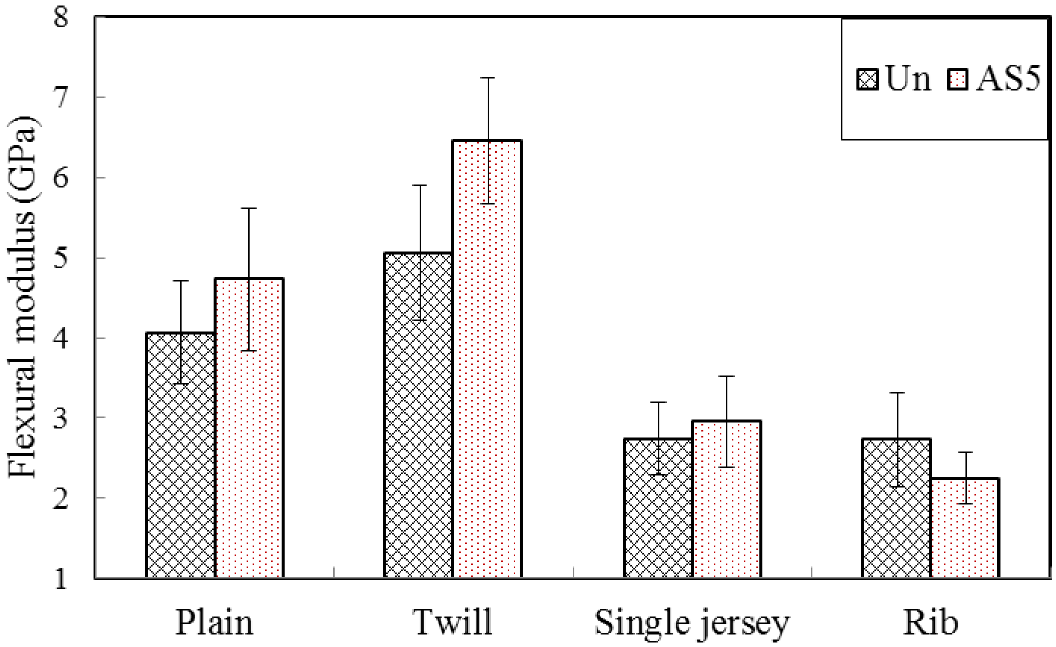


Figure 4.55: Comparison of flexural modulus of composites of untreated and AS5 treated fabrics of different structures.

Impact strength

The impact strength of all the composites of untreated and AS5 treated jute fabrics of different structures are shown in Figure 4.56. It is clearly seen that all the composites of AS5 treated jute fabrics have higher impact strength than that of the composites corresponding to untreated fabrics. Treated plain, twill, single jersey, and rib fabrics PP composites have, respectively, 24%, 17%, 11%, and 12% higher impact strength than that of composites corresponding to untreated fabrics. Therefore, AS5 treatments are effective for the twill, single jersey, and rib jute fabrics to increase the impact strength of PP composites.

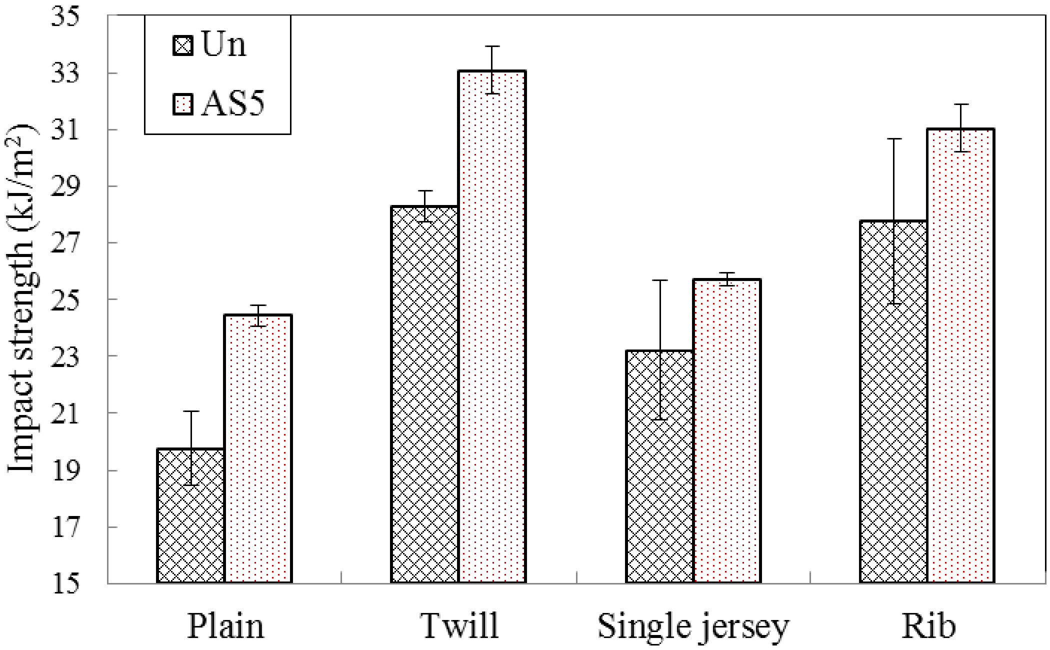
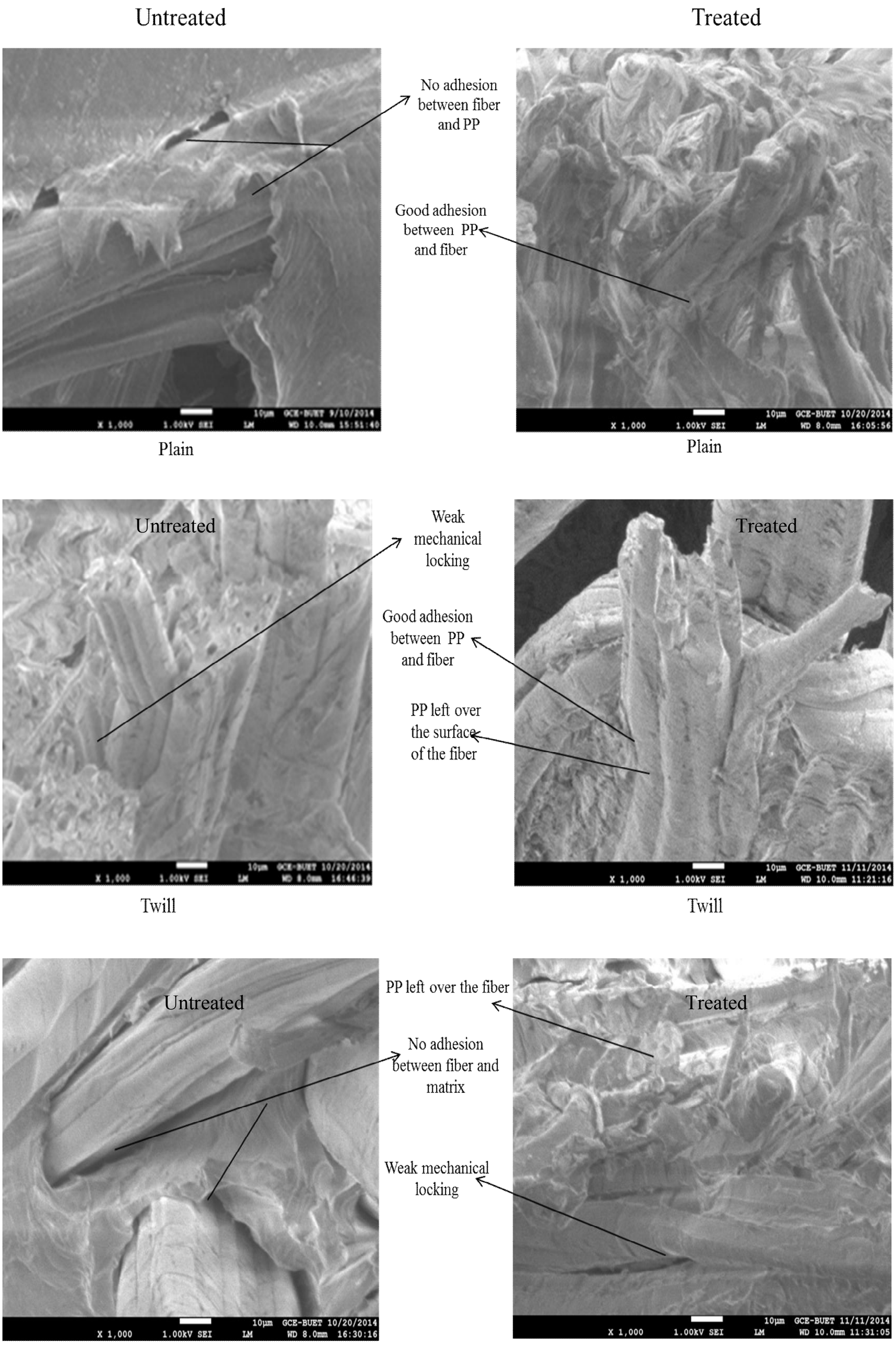


Figure 4.56: Comparison of impact strength of composites of untreated and AS5 treated fabrics of different structures.

4.5.2.2 SEM analysis

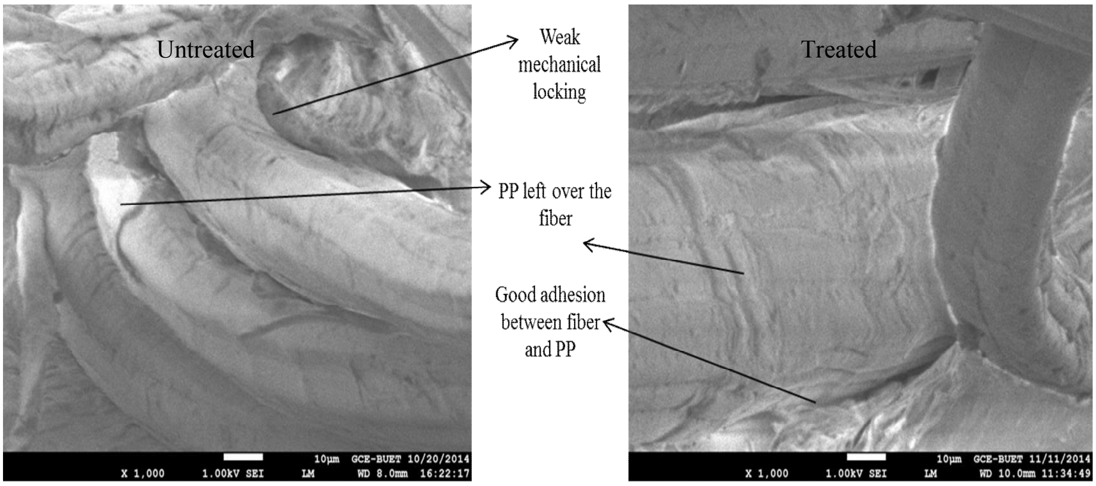
The SEM micrographs (1000X) of the tensile fracture surfaces of all the composites of untreated and AS5 treated jute fabrics of different structures are displayed in Figure 4.57. The micrographs show that there is no or only a little trace of PP matrix on the surfaces of untreated jute fibers, which indicates no adhesion between fibers and matrix in all the cases of composites reinforced with untreated jute fabrics. On the other hand, in all the cases of treated fabrics PP composites, there was an enhanced interaction between the fabrics and PP matrix as observed in Figure 4.57. All the composites of treated fabrics had a different fracture surfaces compared to the composites of untreated fabrics. A considerable amount of PP left on the fiber surfaces clearly indicated good interfacial adhesion between the jute fibers and PP matrix. Among all the composites of AS5 treated fabrics of different structures, the composites of twill fabrics demonstrated stronger interfacial interaction between jute fabrics and PP matrix. The surface of the fiber was covered with a thick layer of PP matrix, which indicated good adhesion between jute fibers and the PP matrix as shown in the SEM micrograph of composites of treated twill fabrics. Consequently, all the treated jute fabrics PP composites have



Plain
Plain

Twill
Twill

Single jersey
Single jersey



Rib **Rib**

Figure 4.57: Comparison of SEM micrographs of tensile fracture surfaces of composites of untreated and AS5 treated fabrics of different structures.

higher tensile strength than that of the composites corresponding to untreated jute fabrics as can be seen from Figure 4.51.

4.5.2.3 Water uptake characteristics

Water uptake characteristics of all the composites of untreated and AS5 treated fabrics of different structures are shown in Figures 4.58 to 4.60. These results indicate that water uptake significantly depends upon the surface chemistry of the reinforcing material. It shows that composites of AS5 treated plain and twill fabrics have lower water uptake up to time period of 40 days compared to the composites of untreated plain and untreated twill fabrics. The values of water uptake of composites of treated plain and treated twill fabrics are, respectively, 31% and 47% lower than that of composites corresponding to untreated plain and twill fabrics as shown in Figure 4.59. Further, composites of plain fabrics treated by AS5 treatment had 13% lower water uptake than that of treated twill fabrics PP composites. Treated single jersey and rib fabric composites have higher water uptake over the time interval of 24 hours than that of composites corresponding to untreated fabrics (Figures 4.58 and 4.59). However, the water uptake characteristics are reversed after a considerably long time as seen in Figure 4.60. These results demonstrate that all the composites of AS5 treated fabrics of different structures have the lower water uptake corresponding to long immersion time

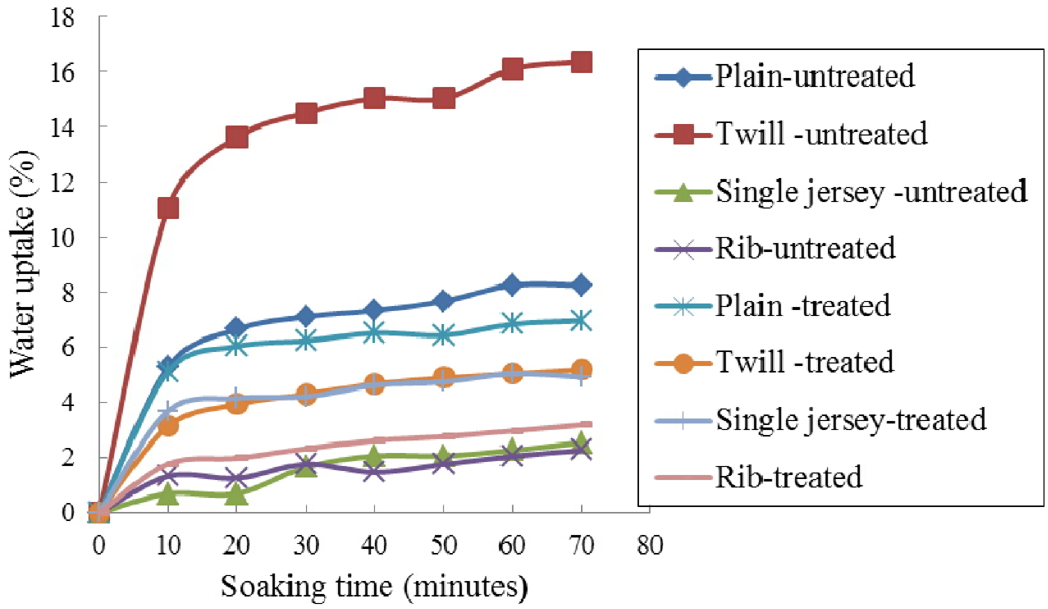


Figure 4.58: Water uptake versus soaking time (minutes) of composites of untreated and AS5 treated fabrics of different structures.

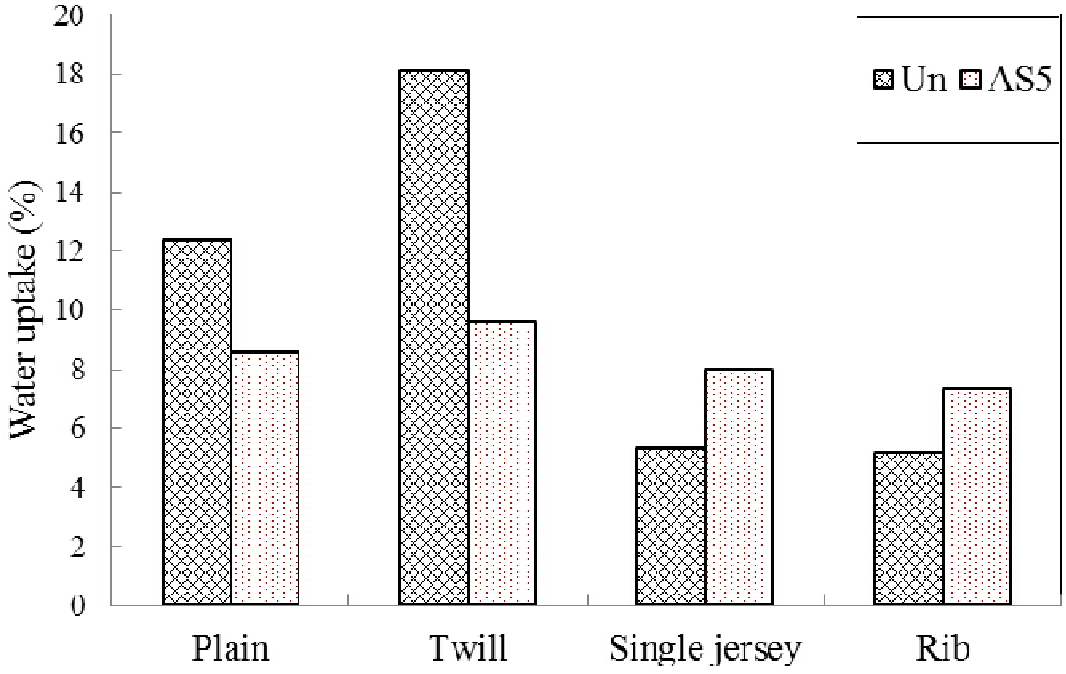


Figure 4.59: 24 hours water uptake of composites of untreated and AS5 treated fabrics of different structures.

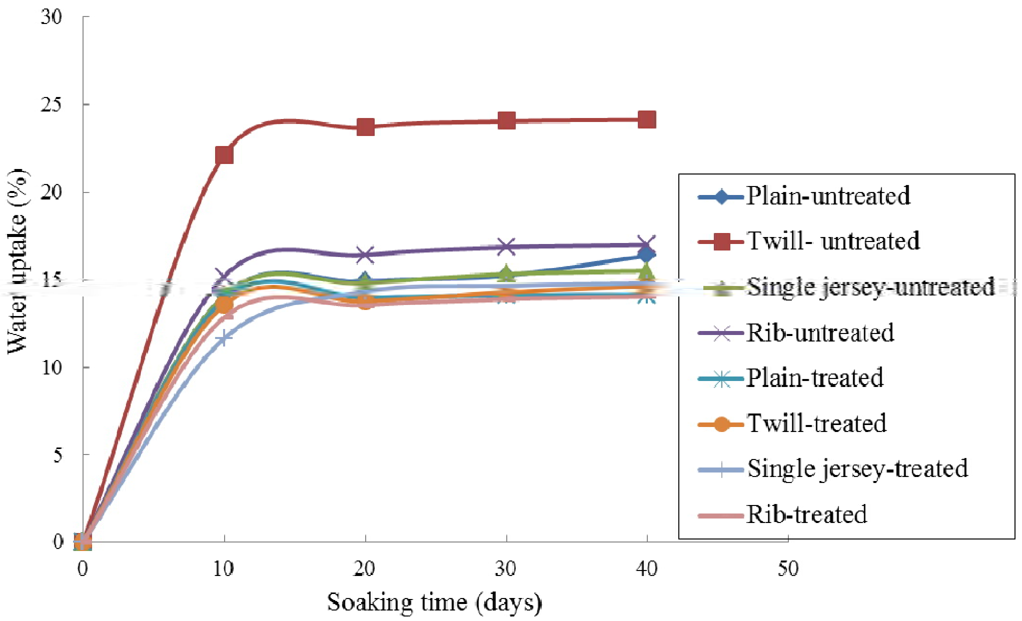


Figure 4.60: Water uptake versus soaking time (days) of composites of untreated and AS5 treated fabrics of different structures.

compared to the composites corresponding to untreated jute fabrics. This led to conclude that AS5 treatment had changed the surface chemistry of jute fabrics significantly (Scheme 4.1 to 4.4). As a result, all the composites of AS5 treated fabrics have shown the improved water uptake properties.

4.5.3 Conclusions

Properties of composites reinforced with untreated and AS5 treated jute fabrics of four different structures have been evaluated and compared. It was found that both the mechanical and water absorption properties of PP composites were influenced by the AS5 treated fabrics. Composites of AS5 treated twill fabrics exhibited the highest value of tensile strength, which was 56% higher than that of treated plain fabrics PP composites. AS5 treatment plays an important role in improving the interfacial bonding between fabrics and PP matrix. Water absorption characteristics of treated woven fabrics reinforced PP composites were much better than that of treated knitted fabrics reinforced PP composites.

CHAPTER 5

SUMMARY AND RECOMMENDATIONS

5.1 Summary

The chemical modifications of jute fabrics with various chemicals in the presence and absence of reactive dye (Drimarene Red K8b) with or without NaCl and Na_2CO_3 are made in view of their usage as reinforcing agents in PP matrix. In particular, Albafix WFF, Glytac, Indosol-E50, and conventional NaOH were included into Drimarene Red K8b for the chemical modification of as-received plain jute fabrics by 23 different treatment procedures. Treated plain jute fabrics were characterized in terms of tensile strength, color strength, exhaustion, fixation, wash fastness test, FTIR spectra, and water absorbency. Mechanical behavior, thermal behavior by TGA, surface morphology by SEM, and water uptake properties of the composites of both treated and untreated plain jute fabrics were evaluated based on which a most effective treatment procedure was selected. Later, untreated yarns were used to manufacture plain, twill, single jersey, and rib structure jute fabrics which were treated by the most effective treatment procedure and subsequently consolidated into polypropylene matrix. Performance of PP composites having both treated and untreated jute fabrics of four different structures had been evaluated and compared. Based on the experimental results of composites and fabrics, some specific conclusions may be drawn as follows:

1. Reactive dye influenced the surface chemistry of the jute fabrics effectively so that mechanical, thermal, and physical properties of jute fabrics PP composites were improved. Dye treated plain jute fabrics PP composites showed more improved mechanical, thermal, and water absorption properties than that of PP composites with plain jute fabrics treated without dye.

2. Specifically, the treatment with the mixture of Drimarene Red K-8b, NaCl, and Na_2CO_3 of the plain jute fabrics pretreated with the mixture of Albafix WFF and NaOH (AS5) is the most effective treatment procedure of jute fabrics for getting the best results of their PP composites. Composites of plain fabrics treated by AS5 treatment procedure had the highest tensile strength, which was 109% higher than

that of the untreated fabrics PP composites and 73% higher than that of the composites of NaOH treated fabrics.

3. The treatment with Drimarene Red K8b of the plain jute fabrics pretreated with the mixture of Albafix WFF and NaOH (AS4) is the most effective treatment procedure for the purpose of dyeing of jute fabrics. Plain jute fabrics treated by AS4 treatment had, respectively, 125%, 95%, and 25% higher exhaustion, color strength, and dye fixation than the corresponding values obtained by normal dyeing process.

4. The SEM investigation of the tensile fractured surfaces of composites revealed strong bonding between the PP matrix and treated fabrics which caused the improved mechanical properties of composites.

5. TGA results showed that the composites corresponding to plain fabrics treated with the mixture of Indosol E-50 and Drimarene Red K8b confirmed the best thermal stability among all the plain fabrics PP composites.

6. Mechanical properties of composites were influenced by the structure of fabrics. The composites of untreated rib, twill, and single jersey fabrics had, respectively, 40%, 46%, and 17% higher tensile strength than that of untreated plain fabrics composites.

7. Composites of twill fabrics treated by AS5 treatment procedure exhibited the highest value of tensile strength, which was 56% higher than that of AS5 treated plain fabrics PP composites.

8. Untreated weft knitted fabrics PP composites significantly improved water uptake properties compared to that of the untreated woven fabrics PP composites.

9. Composites of plain fabrics treated by AS5 treatment had 13% lower water uptake than that of AS5 treated twill fabrics PP composites

10. FTIR results showed that chemical structure of cellulose (jute fabrics) was changed so that interfacial adhesion properties between hydrophilic jute fabrics and hydrophobic PP were improved.

5.2 Recommendations for Future Work

The results presented in this thesis will be helpful for the researchers of the relevant fields and manufacturing industries of composites. To avail full advantages of composites discussed in this thesis, some other aspects should also be investigated which was not possible to carry out within the time frame of the present work. Therefore, some further investigations are recommended here for future works as follows:

1. The current research may be extended for other species of jute and other natural fibers.

2. Weathering and biodegradable behaviours of the composites may be evaluated.

3. This work was carried out for a fixed mass fraction of fabrics. The effect of mass fraction of fabrics may be analyzed and an optimum mass fraction of fabrics should be determined.

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