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STUDY OF THE EFFECT OF CHEMICAL TREATMENTS ON THE TENSILE BEHAVIOUR OF ABACA FIBER REINFORCED POLYPROPYLENE COMPOSITES

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ABSTRACT

Abaca fibers were subjected to different chemical treatments like alkali treatment, permanganate treatment, acrylation & benzene diazonium chloride treatment and these chemically treated fibers were used as reinforcements in the preparation of polypropylene composites by hot compression moulding method. Various composites were fabricated with different fiber loadings of 20%, 30%, 40%, 50%, 60%, and 70%. Abaca composites with 40% fiber loadings were found to have optimum properties when tensile tests were carried out and the study also revealed that treated composites were found to have improved tensile properties when compared to untreated composites. Among all the treatments carried out benzene diazonium chloride treated abaca fiber reinforced polypropylene composites showed higher tensile strength. These composites showed 82.38% increase in tensile strength when compared to untreated composites for 40% fiber loading.

Indexing terms/Keywords

Abaca fibers; Chemical treatment; Surface modification; Polypropylene; Tensile strength.

Academic Discipline And Sub-Disciplines

Chemistry; Mechanical Engineering; Polymer Composites; Natural Fibers.

SUBJECT CLASSIFICATION

Natural fiber reinforced polymer composites

TYPE (METHOD/APPROACH)

ASTM Standards

INTRODUCTION

Because of population explosion, the appetite for petroleum based resources is continuously increasing and as a result they are getting depleted at a faster rate. These resources will get exhausted within a span of 50-60 years, if they are consumed at the current rate. A sense of environmental consciousness in public lead to the extensive use of renewable natural fiber composites as a substitute for non renewable petroleum based materials. Industrialists are also showing interest in investments on environment friendly, sustainable materials like natural fiber reinforced composites which are having good mechanical performance (1).

The use of natural fiber reinforced composites improves the cultivation of these plants which in turn boosts the economy of the country. Synthetic fibers like glass, aramid fibers, carbon etc. are costly and are not biodegradable and pollute the environment. On the other hand natural fibers like abaca, areca, jute, coir, flax, sisal etc. are abundantly available renewable reinforcements which are less costly and biodegradable and hence reduce the problem of polluting the environment. Hence natural fiber reinforced composites are gaining lot of importance now a days (2).

The chief components of natural fibers are cellulose, hemicelluloses, lignin and pectin. Cellulose is the major constituent which is a linear polymer of anhydro D glucose molecules which contains 3 hydroxyl groups. These hydroxyl groups are capable of forming hydrogen bonding with water thus making the natural fibers hydrophilic in nature. But they absorb less moisture after composite fabrication as they are partially enclosed in a polymer matrix. Cellulose is crystalline and plays a key role in providing strength, stiffness and structural stability. So, higher the cellulose content higher is the fiber strength. The fiber stiffness is determined by microfibrillar angle. The angle between the fiber axis and the micro fibrils is called microfibrillar angle. Cellulose is resistant to oxidising agents and alkali. In different plant based natural fibers, the chemical structure of cellulose is similar but what differs is the degree of polymerization and cell geometry. Because of this different properties are observed for different fibers.

Hemicelluloses are amorphous, branched polymers whose molecular weight is less than that of cellulose. Hemicelluloses swell in water, dissolve in alkali and easily hydrolysed in acids. Lignin is amorphous cross linked polymer consisting of phenyl propane units. Pectin is a complex polysaccharide and the branches are cross linked with arabinose sugars and calcium ions. These give flexibility to the plants. Pectin along with lignin and hemicelluloses acts as a cementing agent binding cellulosic micro fibrils together (3). Superior mechanical properties of natural fibers are mainly due to high cellulose content i.e. crystallinity, higher aspect ratio i.e. length to diameter ratio and lower microfibrillar angle (4).

The scientific name of abaca is Musa Textilis which is a species of banana grown extensively in Philippines. It is also called Manila hemp. Abaca plant belongs to the banana family Musaceae, Genus Musa and species Musa Textilis. Abaca fibers are obtained from pseudo stem of musa textilis and are bast fibers.

It is the strongest of all natural fibers and it is valued much for its long fiber length, flexibility, buoyancy and resistance to damage in salt water (5). Abaca fiber composites are used extensively in automotive industry. Mercedes Benz used abaca fiber reinforced polypropylene composites in automobile body parts. Daimler Chrysler used these abaca fiber reinforced composites in under floor protection of passenger cars. The use of abaca fiber instead of glass fiber reduces the weight of automotive parts, bringing about 60% savings in energy and reduces CO_2 emissions making it environment friendly. These composites are also used in construction as well as packaging industries (6).

Ease of availability, sustainability, high tensile strength, resistance to rotting and specific flexural strength nearer to that of glass fibers makes abaca fibers superior (7). Despite these advantages, the major drawback which limits the use of these fibers in the preparation of composites is water absorption.

Fibers are hydrophilic and matrix is hydrophobic. As a result compounding difficulties arise leading to non uniform dispersion of fibers within the matrix. Moreover natural fibers absorb water resulting in swelling and presence of voids at the interface. These drawbacks results in weakening the bond strength at the interface, matrix cracking, poor mechanical properties and reduced dimensional stability of the composites (8-9). These drawbacks can be overcome by chemical treatments like alkali treatment, permanganate treatment, acrylation & benzene diazonium chloride treatments by eliminating hydrophilic hydroxyl groups.

The binding material is the polymer matrix which holds the fibers together and transfers the load to the fibers. The advantages of using a thermoplastic polymer are that they are recyclable, tough, remolded and easy to repair but the drawback is because of the poor melt flow, they need to be heated above the melting point for processing. We have used polypropylene as the thermoplastic material because of the above mentioned technical reasons and also it is economical (4).

In the manufacture of composite materials and structures, measurement of mechanical properties play significant role in quality control. Tensile strength, the force required to pull something to the point till it breaks, is one of the basic important mechanical property of a composite that is required for analysis and design of composite materials and structures. From tensile test results we can select a material for an application; we can predict how a material will react to different types of forces. With the advancement in composite technology, the composite test methods and test equipment has become sophisticated. Tensile strength is an intensive property and hence its value does not depend on the size of the test specimen but depends on the methods of preparation of the specimen, the presence of surface defects and the temperature of the test environment and material.

This study mainly focuses on the preparation of untreated, alkali treated, permanganate treated, acrylated and benzene diazonium chloride treated abaca fiber reinforced polypropylene composites and the effect of these treatments on the tensile properties of the composites.

MATERIALS AND METHODS

Materials

Abaca fibers were collected from the Maruthi Peach Finishing Company, Tirupur, Chennai, India and Analytical grade reagents were purchased from Qualigens Company and used as received.

Alkali treatment of abaca fiber

The fibers are first dewaxed by soaking in 1:2 mixtures of ethanol and benzene for 72 h, followed by washing with deionised water and then air dried. The dewaxed fibers were immersed in 6% NaOH solution at room temperature (30-32 ^{o}C) for 1h, where the total volume of the solution was 15 times the weight of the fibers. The fibers were kept in alkaline solution for 36 h at a temperature of 30 ^{o}C; they were thoroughly washed in running water and then neutralized with 2% acetic acid solution. Lastly, the fibers were washed again in running water to remove the last traces of acid sticking to it, so that the pH of the fibers was approximately 7. Then, they were dried at room temperature for 48 h to obtain alkali-treated fibers (10).

Acrylic acid treatment of abaca fiber

Alkali treated abaca fiber was soaked in 1% acrylic acid (AA) solution at 50 ^{o}C for 1 h, then washed with distilled water thoroughly and dried in an oven for 24 h at the temperature of 70 $^{\circ}$C.

Preparation of benzenediazonium chloride

8 cm^3 of concentrated hydrochloric acid was added to a boiling tube containing 3 cm^3 of phenyl amine (aniline) and 10 cm^3 of water, the mixture was shaken until the amine has dissolved, and then the solution was cooled to 5 ^{o}C by cooling it in an ice bath. After that a solution of sodium nitrite (3 g in 8 cm^3 of water), previously cooled to 5 ^{o}C was added. The temperature of the mixture was maintained below 10 ^{o}C during the addition (11).

Benzenediazonium treatment of abaca fiber

The abaca fibers were chopped into a length of 10 mm, washed with distilled water, and was then dehydrated in an oven at 70 ^{o}C for 24 h. The dried fibers were immersed in a 6% NaOH solution taken in a 2.0 L glass beaker for 10 min at about 5 ^{o}C. A freshly prepared diazo solution was then poured slowly into the above mixture with constant stirring. Fibers were then taken out, washed with soap solution followed by distilled water, and finally dried in an open air for 48 h (12).

Permanganate treatment of abaca fiber

Abaca fibers pretreated with 6% NaOH were immersed in 0.5% $KMnO_4$ in acetone solution for 30 min. The fibers were then decanted and dried in air (13).

Composite fabrication

Abaca fibers and polypropylene filament were chopped into a length of 10 mm; they were mixed and carded in a carding frame. The weight fractions 20, 30, 40, 50, and 60% of fiber was carefully controlled during the mixing of two ingredients. The resulting material was compression moulded to the dimensions of 300 x 300 x 2.0 mm. The composite preparation process was performed in the following order. First, the heat press was pre-heated to 60 ^{o}C. Then the pressure was set as 0 MPa and the temperature raised to 100 ^{o}C. After that pressure and temperature raised to 5 MPa and 230 ^{o}C, respectively. Further, raised the pressure to 15 MPa, maintained the pressure and temperature for 30 min. Finally, lowered the pressure to 0 MPa, lowered the temperature to 30 ^{o}C and composite plate was removed from the heat press. The specimens were post cured for 24 h before the test (14).

Tensile strength of composites

Specimens prepared for the tensile strength test were cut into five specimens and the measurement was carried out according to ASTM D3039 standards. Each result is an average of five measurements. A rectangular shape specimen with the total length of 250 mm, a gauge length of 150 mm, width of 25 mm and a uniform thickness of 2.0 mm is considered for the test. The specimen was loaded in the computerised universal testing machine (Mecmesin 5 *xt*) until the failure of the specimen occurs at laboratory conditions (temperature, 30±2 ^{o}C; RH 65%)

RESULTS AND DISCUSSION

Chemical treatment of fibers

The surface of the abaca fiber is modified and interfacial bonding is improved with different chemical treatments (7-10). Alkali treatment helps in the removal of hydrogen bonding in the network structure of fibers which substantially reduces moisture absorption. This treatment increases the amount of amorphous cellulose at the expense of crystalline cellulose. Abaca fiber morphology significantly changed due to alkali treatment.

Fiber-OH + NaOH → Fiber-ONa + H_2O

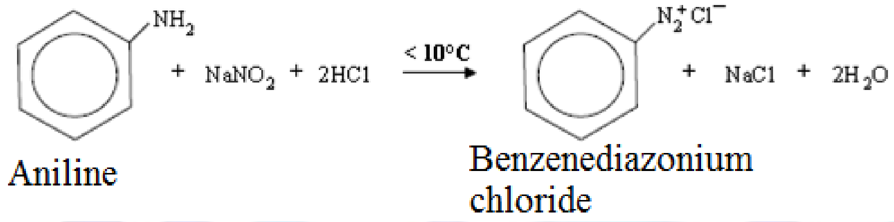
During acrylation treatment, the hydroxyl group of fiber cellulose reacts with carboxylic group of acrylic acid yielding an ester making the fibers more hydrophobic in nature. Acrylation decreased the dissemination of water into the fiber making it more hydrophobic. Acrylic acid treated abaca fiber showed lower moisture absorption. This is due to the replacement of hydroxyl group by hydrophobic ester groups.

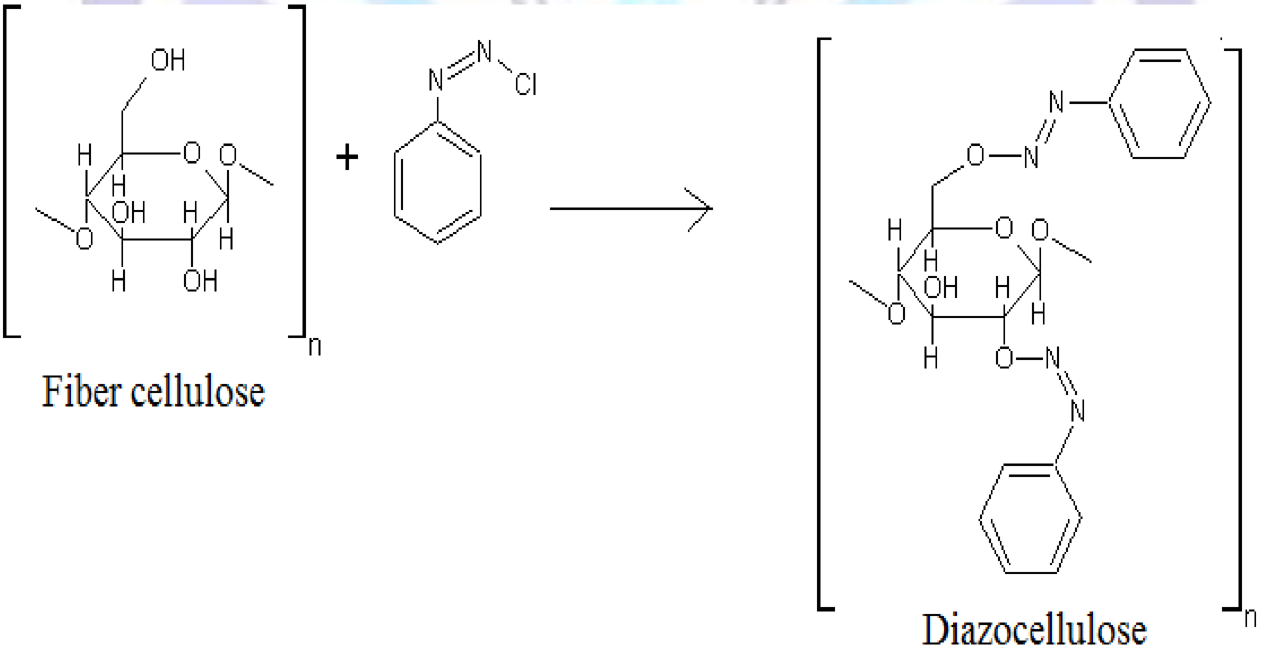
Fiber-OH + $H_2C{=}CHCOOH$ → Fiber-$OCOCH_3$ + H_2O

Permanganate treatment is carried out using $KMnO_4$ in acetone solution. In permanganate treatment, highly reactive permanganate ions react with cellulose hydroxyl groups forming cellulose manganate. This reduces hydrophilic nature of the fiber and also improves chemical interlocking at the interface and provides better adhesion with the matrix.

Fiber-OH + $KMnO_4$ → Fiber- O-H-O-Mn(=O)$_2$-OK$^+$

Benzene diazonium chloride treatment is also used in decreasing the hydrophilic nature of fibers by increasing fiber matrix adhesion. It undergoes coupling reaction with OH groups of the fiber forming diazo cellulose compound.





IR Spectra

In FTIR a Fourier transform is required to turn the raw data into the actual spectrum. It is an effective analytical instrument for detecting functional groups. This is more accurate and faster. Infrared spectra of abaca fibers showed prominent differences after chemical treatments. The important bands obtained in IR spectra are listed in Table 1.

Table 1. FTIR Peaks Position of Abaca Fibers

Wave number, cm-1	FTIR peak origin
3250-3500	Hydroxyl group and bonded OH stretching
2900	CH aliphatic and aromatic
2825	CH aliphatic and aromatic
1740	C=O stretching vibrations (carboxylic group and ester groups)
1650	NO group
1560	Lignin components
1500	NO group
1460	–N=N- group
1310	NO_2 symmetric deformation
1310	Alcohol group of cellulose
1250	Hemicelluloses and pectin
1100	C-O-C symmetric glycosidic stretch
780	Lignin Components

The IR spectra of untreated, alkali treated and acrylic acid treated abaca fiber is presented in Figure 1.

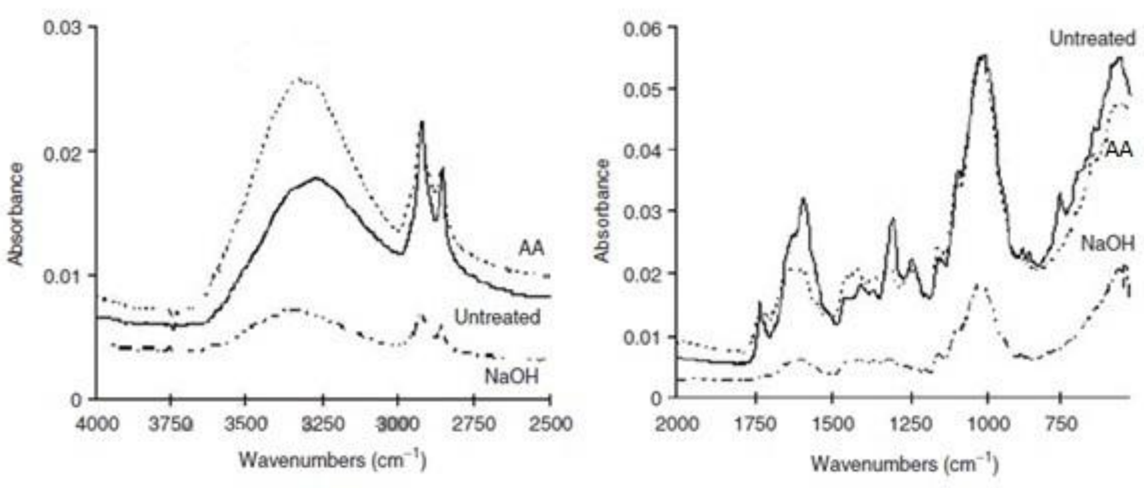


Fig 1: IR spectra of untreated, alkali treated (NaOH) and acrylic acid (AA) treated abaca fiber

A broad and intense band at 3375 cm^{-1} corresponds to the hydroxyl groups and the O-H stretching vibrations present in carbohydrate (cellulose + hemicelluloses) (15). A band at 3350 cm^{-1} which is due to the presence of alcohol group has been reduced in alkali treated fiber. This indicates the probable elimination of hemicelluloses by alkali treatment. In case of IR spectrum of acrylic acid treated fiber the peak at 3350 cm^{-1} is increased due to the hydroxyl group of the acrylic unit. A strong band at 1310 cm^{-1} exhibited by the alcohol group of cellulose OH deformation was also reduced by both alkali and acrylic acid treatments. The spectrum also shows peaks at 2900 and 2825 cm^{-1} characteristic of CH group. Because acrylic acid treatment leads to $–CH_2–$ and –CH– group formation (16), the peak is less pronounced in alkali treated fiber spectra and more in the acrylic acid treated fiber spectra.

In case of both untreated and acrylic acid treated abaca fiber spectra, a band at 1740 cm^{-1} assigned to carbonyl stretching, corresponding to hemicelluloses was observed. But this band has not been depicted in the spectrum of alkali treated abaca fiber. So, the band at 1740 cm^{-1} is due to the ester group in acrylic acid treated fiber (17). The large bands appearing at 1560 and 780 cm^{-1} indicates the presence of lignin in the untreated fiber. Further the absence of these peaks in the chemically treated fiber spectrum indicates the removal of lignin by alkali and acrylic acid treatments. The disappearance of the peak at 1250 cm^{-1} for alkali treated fibers shows the removal of the waxy epidermal tissue, adhesive pectin and hemicelluloses that unite fiber bundles together. The C-O-C symmetric glycosidic stretch at 1100 cm^{-1} is due to the polysaccharide component that was largely cellulose and appeared for both untreated and treated abaca fibers. It can be summarized that treatment with acrylic acid and alkali had removed most of the lignin and hemicelluloses components. Further, the treatment had changed the hydrophilic nature of the fibers into more hydrophobic nature.

Infrared spectra of benzene diazonium chloride treated abaca fiber (Figure 2) confirm the reaction between the fiber cellulose and benzene diazonium chloride forming diazocellulose compound. In the IR spectra, the characteristic absorption peaks at 1500 and 1650 cm^{-1} are due to the presence of NO group and peaks at 1400 and 1460 cm^{-1} are appearing for –N=N– group found in fiber-cellulose compound. Also, the absorption band observed at 1310 cm^{-1} is attributed to NO_2 symmetric deformation.

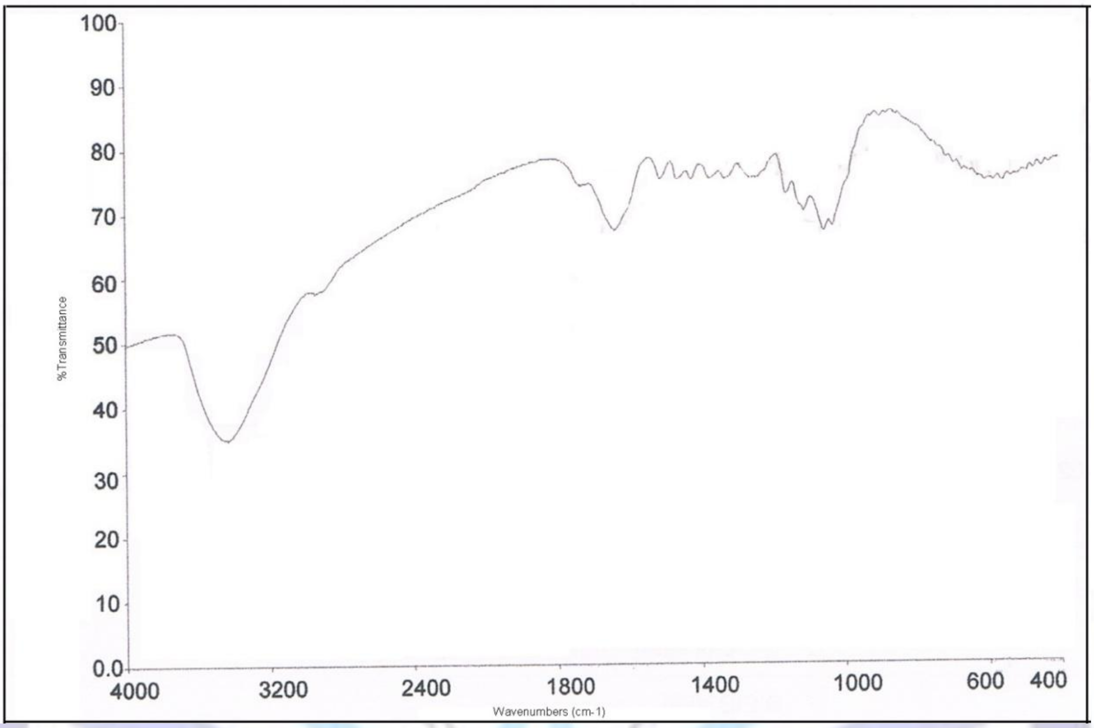


Fig 2: IR spectra of benzene diazonium chloride treated abaca fiber

The peak appeared at 1740 cm^{-1} in the IR spectra of untreated abaca fiber is found to be absent in the spectra of permanganate treated fiber (Figure 3). The absence of peak infers that after alkali and permanganate treatments, hydrolysis occurs which breaks down the ester bond or ether bond. The peaks appeared in the region 1100-1600 cm^{-1} in untreated fiber indicates the presence of hemicelluloses in untreated abaca fiber. The decrease in the intensity of these peaks in treated fiber indicates slight removal of hemicelluloses from the fiber surface (13).

It can be summarized that the chemical treatments had removed most of the lignin and hemicelluloses components from the fiber surface. Further, the treatments had changed the hydrophilic nature of the natural abaca fibers to hydrophobic nature.

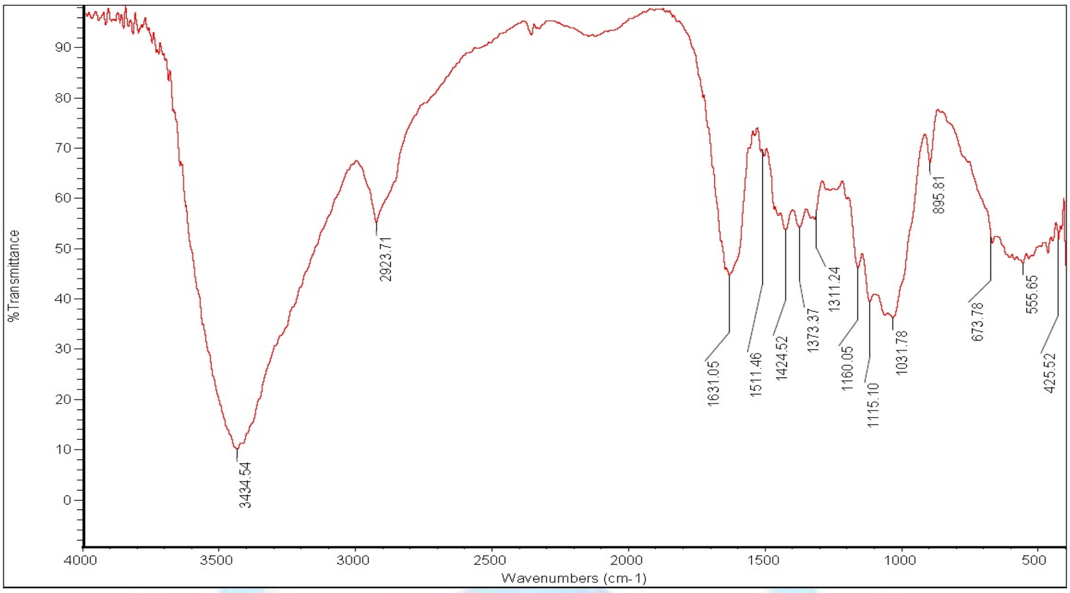


Fig 3: IR spectra of permanganate treated abaca fiber

Tensile strength of composites

In this work, abaca fiber reinforced polypropylene composites were fabricated using hot compression moulding technique. From the prepared composites, test specimens were cut to size according to ASTM standards and were evaluated for tensile properties. It is a must to know optimum fiber loading to get good tensile properties (18). So, the variation of tensile strength with different fiber loadings and the effects of different chemical treatments on tensile strength were studied. The tensile strength of abaca fiber reinforced polypropylene composites based on different fiber loading and different chemical treatments are illustrated in Figure 4.

Generally, chemically treated fiber reinforced composites have higher tensile strength than the untreated fiber reinforced composites. The observed tensile strength values of composites can be understood in terms of chemical constituents of fiber. It is observed from the Figure 4 that the tensile strength of untreated abaca fiber-polypropylene composites were slightly higher than that of the matrix. This is attributed to the low compatibility between the hydrophilic untreated fiber and hydrophobic polypropylene matrix.

Alkali treatment removes lignin and hemicelluloses and exposes more hydroxyl groups on the fiber surface making them available for bonding with matrix improving the adhesion between the fiber and matrix. Not only that, it also cleans the surface, reduces fiber diameter and increases the aspect ratio, increases stress transfer capacity thereby increasing the tensile strength (9). In case of alkali treated abaca-polypropylene composites the tensile strengths of 20%, 40% and 45% fiber loadings are 35.24 MPa, 50.24 MPa and 39.54 MPa respectively. For 40% fiber loading tensile strength increased by 24.96% compared to 20% fiber loading and for 45% fiber loading it decreased by14.83% compared to 40% fiber loading.

Permanganate treatment provides better interlocking at the interface by making the surface rough and improves fiber matrix adhesion. In case of permanganate treated abaca-polypropylene composites the tensile strengths of 20%, 40% and 45% fiber loadings are 38.8 MPa, 55.26 MPa and 42.08 MPa respectively. For 40% fiber loading tensile strength increased by 42.42% compared to 20% fiber loading and for 45% fiber loading it decreased by 23.85% compared to 40% fiber loading.

The reaction between fiber and acrylic acid enhances stress transfer capacity at the interface and improves the tensile strength. In case of acrylated abaca-polypropylene composites the tensile strengths of 20%, 40% and 45% fiber loadings are 45.84 MPa, 59.28 MPa and 45.54 MPa respectively. For 40% fiber loading tensile strength increased by 29.31% compared to 20% fiber loading and for 45% fiber loading it decreased by 23.17% compared to 40% fiber loading.

The coupling reaction between fiber and benzene diazonium chloride facilitates fiber matrix adhesion and improves the tensile strength. In case of benzene diazonium chloride treated abaca-polypropylene composites the tensile strengths of 20%, 40% and 45% fiber loadings are 52.64 MPa, 70.84 MPa and 48.56 MPa respectively. For 40% fiber loading tensile strength increased by 34.57% compared to 20% fiber loading and for 45% fiber loading it decreased by 31.45% compared to 40% fiber loading.

In case of all chemically treated abaca-polypropylene composites the tensile strength increased from 20% to 40% fiber loading, and afterwards for higher fiber loading tensile strength decreased. At 40% fiber loading it showed maximum value. This is because at 40% fiber loading, there is better fiber distribution in matrix, less fiber fractures and effective transfer of load from matrix to fibers. As fiber loading increased beyond 40%, tensile strength decreased due to poor interfacial adhesion and inefficient stress transfer from matrix to fibers (19-20).

The pure polypropylene is having tensile strength of 28 MPa. In comparison to untreated fiber composites of 40% fiber loading, alkali, permanganate, acrylated and benzene diazonium chloride treated fiber composites showed 29.35%, 42.27%, 52.62% and 82.33% increase in tensile strength respectively. From these results it is very clear that benzene diazonium chloride treated fiber composites of 40% fiber loading showed maximum tensile strength. So, these composites are best for applications where high tensile strength is required.

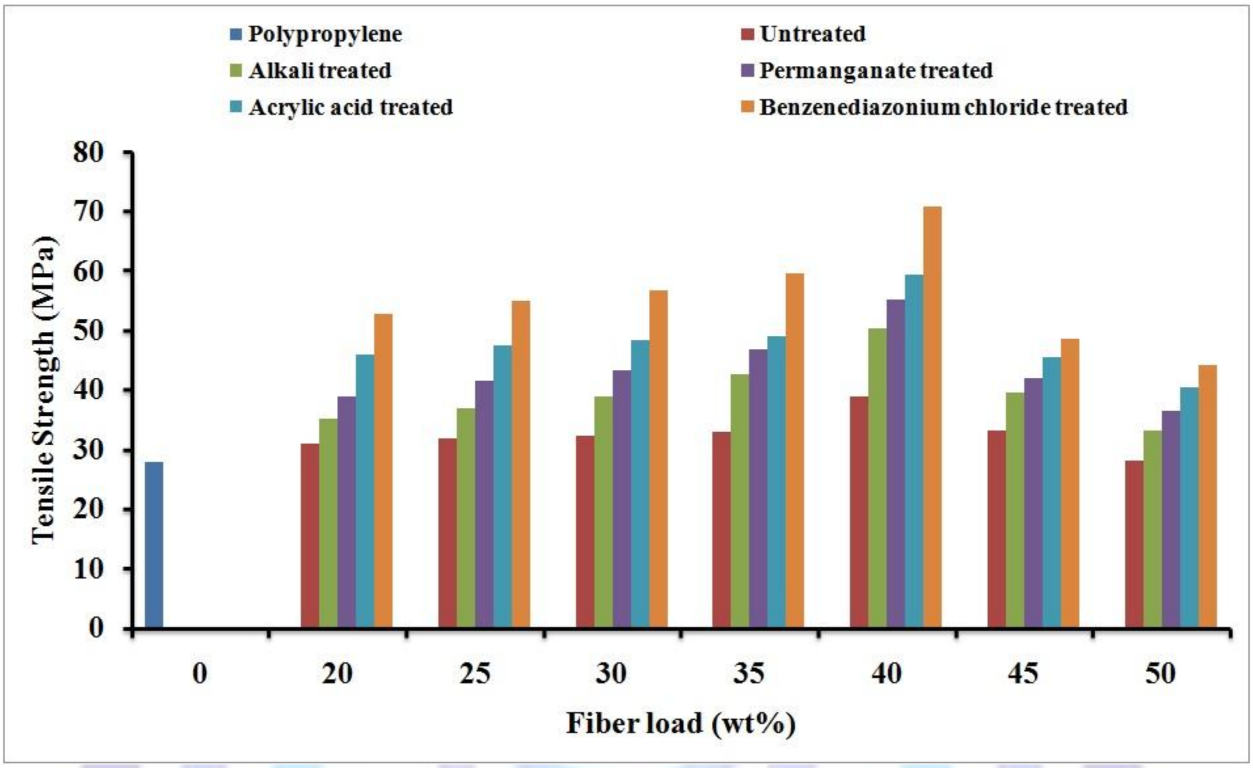


Fig 4: Tensile strengths of abaca polypropylene composites

CONCLUSIONS

Chemical treatments resulted in surface modification improving fiber matrix adhesion, reducing hydrophilic nature and facilitating better mechanical performance. The tensile strength of the composites increased with increase in fiber loading upto 40% beyond 40% it decreased. For all treatments maximum tensile strength is shown at 40%. Amongst all the chemical treatments benzene diazonium chloride treated abaca fiber reinforced polypropylene composites showed highest tensile strength for 40% fiber loading.

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