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SHORT COMMUNICATION

Mechanical properties of abaca fiber reinforced polypropylene composites: Effect of chemical treatment by benzenediazonium chloride

Ramadevi Punyamurthy [a,b], Dhanalakshmi Sampathkumar [a,b], Raghu Patel G. Ranganagowda [c], Basavaraju Bennehalli [c,*], Chikkol V. Srinivasa [d]

[a] *Department of Chemistry, Jawaharlal Nehru Technological University, Hyderabad 500085, Telangana, India*
[b] *Department of Chemistry, KLE Society's BVB College of Engineering & Technology, Hubli 580031, Karnataka, India*
[c] *Department of Chemistry, Alva's Institute of Engineering & Technology, Mijar 574225, Karnataka, India*
[d] *Department of Mechanical Engineering, GM Institute of Technology, Davangere 577006, Karnataka, India*

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Abstract Untreated abaca fibers and benzenediazonium chloride treated abaca strands were employed as reinforcements for fabricating polypropylene composites by injection molding method. Various composites were fabricated with different fiber loadings of 30%, 35%, 40%, 45% and 50% with and without coupling agents. Abaca composites without coupling agents with 40% fiber loadings were found to have optimum properties when mechanical characterization was done and the investigation also revealed that untreated and treated composites with coupling agents were found to have improved tensile strength, flexural strength and impact strength when correlated to composites without coupling agents. Among various surface modifications performed, benzenediazonium chloride treated abaca strands reinforced polypropylene composites with coupling agents showed superior properties. For composites including coupling agents, surge in tensile strength and flexural strength was observed with hike in fiber content up to 50% whereas optimum impact properties were shown at 40% fiber loading. Between untreated composites with coupling agent and without coupling agent, composites with coupling agent showed 77.50% hike in tensile strength for 50% fiber loading. Benzenediazonium chloride treated composites with coupling agent showed 70.07% increase in tensile strength when compared to benzenediazonium chloride treated composites without coupling agent for 50% fiber loading. Untreated composites with coupling agent showed 64.91% increase in flexural strength when compared to untreated composites without coupling agent for 50% fiber loading. Benzenediazonium chloride treated composites with coupling

* Corresponding author.
E-mail address: basavaraju_b@yahoo.co.in (B. Bennehalli).
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agent showed 36.84% increase in flexural strength when compared to benzenediazonium chloride treated composites without coupling agent for 50% fiber loading. However, in case of impact strength, addition of coupling agent increased the impact strength up to 35% fiber loading and beyond 35% fiber loading addition of coupling agent decreased the impact strength. Untreated composites with coupling agent showed 3.53% decrease in impact strength when compared to untreated composites without coupling agent for 40% fiber loading. Benzenediazonium chloride treated composites with coupling agent showed 6.59% decrease in impact strength when compared to benzenediazonium chloride treated composites without coupling agent for 40% fiber loading.

1. Introduction

Population explosion, increasing environmental awareness and depletion of fossil resources at a faster rate, rejuvenated the interest in biopolymers obtained from renewable sources. Polysaccharides are the inexhaustible source of biorenewable polymers and among polysaccharides natural fibers are utilized widely to strengthen the composites for various applications. Natural cellulosic strands are biorenewable polymers and the benefits of employing these strands in composite fabrication are: they are available abundantly on a worldwide basis, renewable, light weight, have excellent mechanical, thermal and acoustic insulation properties, show CO_2 neutrality, non abrasive, no health hazards, biodegradable and ecofriendly. At the same time they have certain drawbacks. They have low thermal stability. They absorb water and undergo swelling resulting in dimensional instability restricting their use in outdoor applications. Moisture absorption in a composite is influenced by fiber content, cavities, viscosity of the matrix, dampness and temperature. Moreover the properties of natural fibers depend on the climate, location and weather from which they are grown and also depend on fiber length, age and extraction methods and hence very difficult to predict the composite properties. But advantages outnumber disadvantages and natural fibers by now were recognized as strengthening materials in many industries (Bledzki et al., 2008a,b; Christopher et al., 2015; Dhakal et al., 2007; Kang and Lian, 2015; Singha and Thakur, 2009; Thakur et al., 2014a,b,c; Thakur et al., 2013a; Thakur and Thakur, 2014a, b,c).

Natural fibers consist of cellulose fibrils embedded in lignin and hemicelluloses matrix and are regarded as composites. The efficiency with which the natural fiber can act as reinforcement mainly depends on cellulose and its crystallinity. Cellulose is a biopolymer polysaccharide having glucose monosaccharide units and finds extensive applications in biosorption, biomedical, packaging, biofiltration and biocomposites (Thakur et al., 2013a,b,c).

Abaca fiber is used as raw material for industries without incurring additional cost as it is an unwanted material obtained after abaca farming. These strands have greater tensile property, cannot be easily decomposed and their flexural strength values are on par with glass fibers (Bledzki et al., 2006; Huang, 2009; Ramadevi et al., 2012).

Abaca fibers have affinity towards water, whereas polymer matrix is water repellant in nature. Among the fiber and matrix compatibility problems arise due to their diversified nature leading to feeble bonding between the fiber and matrix making the fabrication of the polymer composites complicated. Chemical treatments stimulate hydroxyl groups of fibers and new moieties are incorporated which can efficiently intermingle with the matrix (Xue and Tabil, 2007; Bachitar et al., 2013).

Thermoplastics are water repellant and are incapable of properly getting mixed with the fiber. This miscibility can be improved by adding certain compounds called compatibilizers. These compounds bring about polymer modification and improve bonding of the matrix with the fiber. Some examples of compatibilizers are maleic anhydride, stearic acid etc. (Agung et al., 2012).

Use of compatibilizers allows in situ modification during blending of composites and avoids use of expensive and toxic organic solvents. Maleic anhydride is one such coupling agent which improves fiber matrix bonding thereby improving the properties (Fabiola et al., 2010). The two main functions of coupling agents are it combines with the 'OH' groups of the fiber cellulose and it also combines with the functional moieties of the polymer matrix. The advantages of using a thermoplastic polymer are that they are recyclable, tough, have high dimensional stability, flame resistance, transparency, remolded and easy to repair but the drawback is the polymer should be heated to a temperature greater than its melting point as the melt flow is poor. Polypropylene is used as the thermoplastic matrix material because of the above mentioned technical reasons and also it is economical (Thakur et al., 2014a,b,c; Kabir et al., 2012).

The focal point of this study is on the fabrication and characterization of untreated and benzenediazonium chloride treated (Diazo treated) abaca polypropylene composites. A set of composites were prepared along with coupling agents and the other set devoid of coupling agents.

2. Materials and methods

2.1. Composite fabrication

Maleic anhydride polypropylene copolymer (MAH-PP) is the coupling agent employed in this investigation and its acid number is 37–43 mg KOH/g. Its softening point and density were 153 °C and 0.89–0.93 g/cm^3. The weight of coupling agent consumed is 5% of the total weight of the fiber. Prior to mixing of raw and treated abaca fibers with matrix material, they were thoroughly and separately oven dried at 80 °C for one day. In a high speed cascade mixer abaca fibers were mixed with polypropylene matrix material. During the blending of the fibers into the matrix, the weight fractions 30%, 35%, 40%, 45% and 50% of fiber were carefully guarded. Then this

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fiber matrix mixture was heated up to 173 °C by placing in a hot mixer and then the hot agglomerate particles obtained were chilled to lab temperature using cold water by transferring to the cool mixer. Injection molding process was employed for fabricating the composites by using cold agglomerate particles which were previously dried up at 80 °C for 24 h. The process is carried out at a temperature range of 150–180 °C, injection pressure is maintained at 20 kN/mm^2 and the temperature of the mold is kept at 80 °C. Before carrying out mechanical tests, the specimens were post cured for 24 h.

2.2. Composite characterization

After composite fabrication, the composite slabs were cut into necessary dimensions according to ASTM standards by means of zig saw. The tests were carried out on eight duplicate samples and the outcomes were shown as a mean value. The tensile, flexural and impact tests were performed at a lab temperature of 30 ± 2 °C and 65% relative humidity. ASTM D 638-01 standard is used to carry out the tensile strength measurement. A specimen with length 150 mm and width of 9.5 mm and a uniform thickness of 4 mm is considered for the test. The test specimen was put in the computerized universal testing machine (Instron 5566) and force was exerted on the sample till the breakdown of the specimen becomes obvious under lab conditions. ASTM D 790-00 standard is employed to measure flexural strength on a universal testing machine using the 3-point bending method. The specimen dimensions were 79 mm length, 9.5 mm width and 4 mm thickness. The crosshead speed is maintained at 2 mm/min during testing of the specimens. Charpy impact test was carried out as per ASTM D 6110-97 standard to measure the impact strength. The specimens with a notch are considered for the test and are placed in Instron Pendulum Tester (9050 Manual Model). The length of the specimen was calculated using digital caliper whereas the width and depth were calculated by means of a micrometer screw gauge. For the test, specimen of length of 55 mm with square cross section of 9.5 mm side and the U notch is made at the center of the specimen for a depth of 4 mm.

3. Results and discussion

3.1. Chemical treatment of fibers

The exterior of the abaca fiber is modified and interfacial bonding is improved with diazo treatment. FTIR confirmed the chemical treatment and the important bands obtained in IR spectra are recorded in Table 1. The presence of hydroxyl groups and O–H stretching vibrations in the fiber (cellulose + hemicelluloses) can be correlated to the peak at 3430–3450 cm^{-1} in the spectrum of untreated fiber and Fig. 1 shows the IR spectra of diazonium treated abaca fiber which confirm the reaction between the fiber cellulose and benzenediazonium chloride. In the IR spectra, the characteristic absorption peaks at around 1500 and 1650 cm^{-1} are mainly linked to the existence of NO group and bands at 1400 and 1460 cm^{-1} are assigned to —N═N-group found in the fiber–cellulose. Also, the peak observed at 1310 cm^{-1} is characteristic of NO_2 symmetric deformation. Fig. 2 presents the SEM photographs of

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

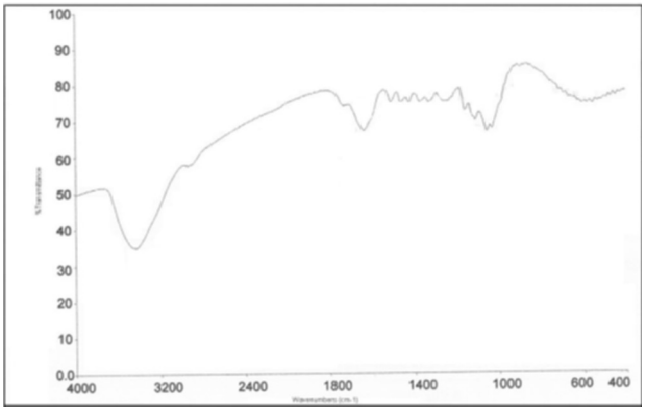

Figure 1 IR spectra of benzenediazonium chloride treated abaca fiber.

untreated abaca fiber and diazo treated abaca fiber. This revealed that the chemical reaction resulted in strong fiber matrix adhesion, which explains the observed enhancement in the properties of composites (Ramadevi et al., 2014; Islam et al., 2011). The scheme of reaction, FTIR and SEM analysis details were discussed in our previous paper (Ramadevi et al., 2014).

3.2. Tensile strength of composites

It is a must to know the optimum fiber loading to get good tensile, flexural and impact properties (Bledzki et al., 2007). So, the change of tensile, flexural and impact properties with different fiber loadings and the effects of coupling agent on these properties were studied. The tensile strength of abaca polypropylene composites using coupling agent and devoid of coupling agents is illustrated in Fig. 3.

The tensile strength values of composites in which chemically treated fibers were used as reinforcements are greater than the tensile strength of untreated composites and the use of coupling agents further increased the tensile strength which is evident from Fig. 3. For untreated and diazo treated composites without coupling agent 40% fiber loading showed

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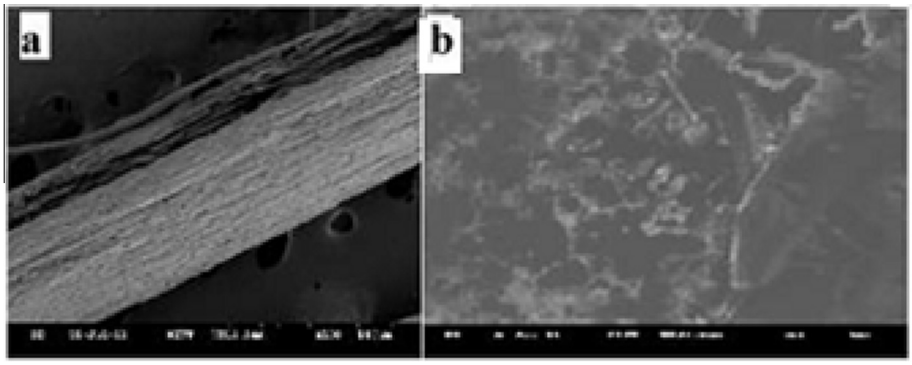

Figure 2 SEM images of (a) untreated, (b) benzenediazonium chloride treated abaca fiber.

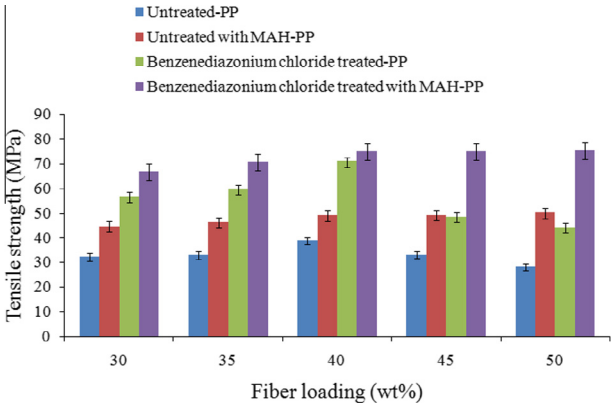

Figure 3 Tensile strength of abaca polypropylene composites.

optimum tensile properties and for composites with coupling agents tensile property increased as the fiber content increased up to 50%. For 40% fiber content, tensile strength of untreated composites with coupling agents increased by 26.05% compared to untreated composites devoid of coupling agents. And diazo treated composites containing coupling agent showed 5.67% hike in tensile strength than diazo treated composites devoid of coupling agents for the same fiber loading. For 50% fiber content, tensile strength of untreated composites containing coupling agents increased by 77.50% compared to untreated composites devoid of coupling agents. And diazo treated composites containing coupling agent showed 70.07% increment in tensile strength than diazo treated composites devoid of coupling agents for the same fiber loading. One of the major factors which determine the properties of composites is fiber volume fraction. The behavior of failure of natural fiber reinforced composites is largely influenced by variation of fiber volume fraction (Ramadevi et al., 2014).

Composites with coupling agent showed maximum tensile strength at 50% fiber loading because at 50% fiber loading same quantity of fiber and matrix led to formidable adhesion through MAH-PP by esterification (Bledzki et al., 2008a,b).

In order to enhance the strength of composites maleated coupling agents are employed. These coupling agents act on both fiber and PP matrix to accomplish superior fiber matrix adhesion. During coupling, maleic anhydride combines with OH groups of cellulose and gets rid of these groups from the fiber cells thereby reducing hydrophilic tendency. It forms carbon–carbon covalent bond with the matrix and in addition combines with hydroxyl groups of the fiber thus providing efficient interlocking. Maleic anhydride reacts with PP chain forming maleic anhydride grafted Polypropylene (MAH-PP). Then hot MAH-PP is made to react with cellulose fibers providing covalent bonds across the interface. This copolymer is first heated to 173 °C and then esterification occurs with cellulose fiber. The surface energy of fiber and the matrix becomes on par with each other and thus provides superior drenching and adhesion between fiber and matrix. Coupling agents also reduce fiber fractures and contribute to increased tensile strength (Agung et al., 2012). The reaction of anhydride of coupling agent with the hydroxyl groups of fibers reduced the compatibility problems between the fiber and matrix (Maya and Anandjiwala, 2008). Fiber matrix adhesion enhanced owing to the use of coupling agent which led to esterification between MAH-PP and hydroxyl groups of cellulose fibers (Bledzki et al., 2007). Mohanty et al. showed that sisal fiber composites with MAH-PP coupling agent had 50% higher tensile strength (Mohanty et al., 2004).

3.3. Flexural strength of composites

Flexural strength of abaca polypropylene composites containing coupling agents and devoid of coupling agents based on different fiber loadings is illustrated in Fig. 4. Polypropylene has a flexural strength value of 49.62 MPa and when fibers are reinforced in the matrix the values increased because of uniform stress distribution from polypropylene to the fibers.

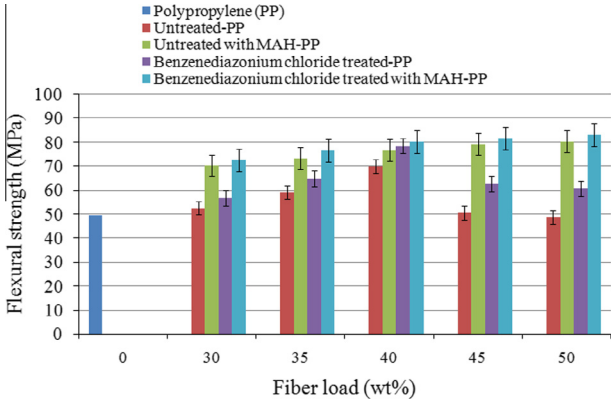

Figure 4 Flexural strength of abaca polypropylene composites.

Flexural strength of diazo treated composites is greater when compared to untreated composites and employing coupling agent further hiked the flexural strength which is evident from Fig. 4. The chemical treatment and the addition of coupling agent helped in the elimination of external fiber surface, enhanced cellulose content and fiber matrix bonding which might have led to the improvement in flexural properties (Libo et al., 2012). For untreated and diazo treated composites without coupling agents 40% fiber loading showed optimum flexural properties. For untreated and diazo treated composites with coupling agents increment in flexural strength was observed as the fiber content surged up to 50%. For 40% fiber content, flexural strength of untreated composites containing coupling agents increased by 9.5% compared to untreated composites devoid of coupling agents. And diazo treated composites containing coupling agent showed 2.21% hike in tensile strength than diazo treated composites devoid of coupling agents for the same fiber loading. For 50% fiber loading, flexural strength of untreated composites with coupling agents increased by 64.91% compared to untreated composites without coupling agents. And diazo treated composites with coupling agent showed 36.84% increase in flexural strength compared to diazo treated composites without coupling agents for the same fiber loading.

Some researchers also showed the same trend that is hike in flexural strength with surge in fiber content up to 50% for abaca reinforced MAH-PP composites (Girones et al., 2011). Addition of coupling agent increases flexural strength because of improvement in interfacial interaction.

3.4. Impact strength of composites

The impact strength of abaca polypropylene composites containing coupling agents and devoid of coupling agents based on different fiber contents is depicted in Fig. 5. The impact strength values of diazo treated composites are greater compared to untreated composites as noticed from Fig. 5. For untreated and diazo treated composites with and without coupling agents 40% fiber loading showed optimum impact strength properties. Untreated composites containing coupling agents showed a surge in the impact strength till 35% fiber content and thereafter it decreased with rise in fiber content. For diazo treated composites with coupling agent impact strength surged with hike in fiber content up to 35% and thereafter it reduced with increase in fiber loading up to 45% and then it increased for 50% fiber loading compared to diazo treated composites without coupling agents. For 40% fiber content, impact strength of untreated composites with coupling agents decreased by 3.53% compared to untreated composites without coupling agents. And diazo treated composites with coupling agent showed 6.58% decrease in impact strength compared to diazo treated composites without coupling agents for the same fiber loading.

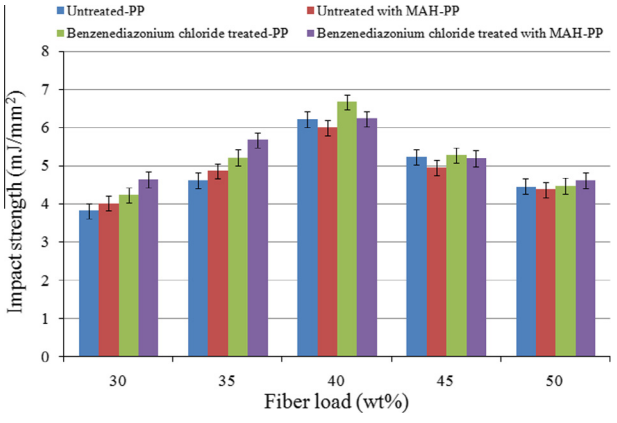

Figure 5 Impact strength of abaca polypropylene composites.

The increment in impact strength is observed for untreated and diazo treated composites containing coupling agent and devoid of coupling agents as the fiber content surged up to 40% and afterwards it dwindled with hike in fiber content. Greater force is necessary to pullout the fibers as the fiber content augmented to 40% which led to enhancement of impact strength. But beyond 40% the decrease in impact strength can be credited to micro-spaces among the fiber and matrix, which causes micro-cracks when impact takes place leading to crack propagation resulting in decrease in impact strength (Sandhya et al., 2012).

4. Conclusions

The tensile, flexural and impact strengths of diazo treated composites were higher compared to untreated composites. Composites without coupling agent and with 40% fiber loading showed optimum properties. The study clearly indicated that employing the coupling agent further improved the properties. Tensile and flexural properties showed similar trends. For composites with coupling agent, the increment in tensile and flexural properties was noticed with surge in fiber content and maximum values were shown for 50% fiber loading. In case of impact strength, there is only a small increase with the addition of coupling agent. For untreated and diazo treated composites with coupling agent, enhancement in impact strength is observed as the fiber content surged up to 40% and thereafter it declined. Untreated composites with coupling agent showed 3.53% decline in impact strength than untreated composites without coupling agent for 40% fiber loading. Diazo treated composites with coupling agent showed 6.59% decrease in impact strength when compared to diazo treated composites without coupling agent for 40% fiber loading.

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