

Influence of Varying Fiber Lengths on Mechanical, Thermal, and Morphological Properties of MA-g-PP Compatibilized and Chemically Modified Short Pineapple Leaf Fiber Reinforced Polypropylene Composites

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ABSTRACT: Environmentally benign, low cost and abundantly available short pineapple leaf fibers (PALF), found mostly in the Tropical rain forest climates are ideal materials for manufacture of thermoplastic polymer-matrix composites. Here, mechanical and thermal properties of composites of maleic anhydride grafted polypropylene (MA-g-PP) and chemically modified short PALF are studied as a function of different fiber lengths at 10 vol % fibers loading with fiber orientation in the longitudinal direction. The effects of fiber lengths and fiber loading on the morphological properties are assessed via observations

by scanning electron microscopy. Fiber length of 6 mm oriented longitudinally at 10 vol % fibers loading in PP is the optimum and recommended composition, where 73% increase in impact properties, 37% increase in the flexural modulus, 33% increase in flexural strength, and 14% increase in vicat softening temperature are observed. © 2009 Wiley Periodicals, Inc. *J Appl Polym Sci* 113: 3750–3756, 2009

Key words: composites; mechanical properties; morphology; poly(propylene) (PP); thermal properties

INTRODUCTION

Natural fiber-reinforced thermoplastic composite structures have good mechanical properties with low specific mass and are widely preferred as an alternative to other conventional fibers such as glass reinforced plastics.¹ The preference in use of natural fibers over synthetic fiber reinforcement in polymer matrices in recent years is mainly due to the growth in environmental awareness in using renewable resources, ease of processing, low cost, and combination of good mechanical and thermal properties with low specific mass.

Compared with various ligno-cellulosic natural fibers, pineapple leaf fibers (PALF), exhibit excellent mechanical properties such as high-tensile strength and high degree of crystallinity, and possess very high-cellulose content (70–82%).² Pineapples are available in abundance in the northeastern part of India and in other tropical counties. But, poor composite strength has been observed due to lack of stress transfer from the polymer matrix to the load bearing natural fibers. This has been attributed to the weak adhesion as a result of poor dispersion

and incompatibility between the hydrophilic natural fibers and the hydrophobic polymer. The surface chemical modifications such as alkali treatment and treatment with maleated polypropylene (PP) of natural fibers improve fiber-matrix adhesion or physicomechanical properties of the resulting biocomposites.³

The physical properties of PALF reinforced natural rubber composites and viscoelastic properties of short PALF reinforced low-density polyethylene have already been reported⁴ but no work has been carried out on effects of maleated coupling agents such as maleic anhydride grafted polypropylene (MA-g-PP) with varying fiber lengths on surface chemically modified short PALF reinforced PP composites. We had earlier studied the effects of MA-g-PP (5%) addition and fiber (4 mm length) loading with different volume fractions (5–20%) on the mechanical and morphological properties of surface chemical modified short PALF reinforced PP composites.⁵

The combined effect of alkali treatment of the defatted fibers and the addition of MA-g-PP compatibilizer improved the interfacial properties by strengthening fiber-polymer interaction by enhancing fiber wetting and impregnation and at the same time chemically binding the two surfaces.⁵ This resulted in increasing the flexural strength (up to

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52%) and flexural modulus (up to 39%) linearly till volume fraction 10% loading of the fibers. PALF in 10 vol % loading in the PP matrix with MA-g-PP also resulted in considerably high impact strength and tensile modulus.⁵

Several other factors not studied yet such as variation in fiber length, fiber aspect ratio (average length to diameter ratio), fiber orientation, and thermal stability of the composites at elevated temperatures are investigated in this work. Mechanical properties such as impact strength, tensile strength and modulus, flexural strength and modulus, thermal properties such as vicat softening temperature (VST) at different fiber lengths of PALF in the composites, as well as their morphological properties are investigated.

EXPERIMENTAL

Materials

Polypropylene (PP) (injection grade, Repol, H110MA) with a density of 0.91 g/cc and melt flow index of 10 g/10 min, obtained from M/S Reliance Industries Limited, (Jamnagar, Gujarat, India) was used as the base polymer matrix. PALF, having diameter of 20–80 μm , density of 1440 kg/m^3 and cell length to diameter ratio of 450⁵ were obtained from Mushroom Growers Welfare Society, Agartala, Tripura (India). Further details on physical, chemical, and mechanical properties of PALF are reported in our recent publication.⁵ Maleic anhydride and benzoyl peroxide was obtained from G. S. Chemical Testing Lab and Allied Industries (Mumbai). Ethanol, benzene, sodium hydroxide, methanol, hydrochloric acid, and isopropyl alcohol were obtained from Merck (India) Ltd. (Mumbai, India). Xylene was obtained from Central Drug House (P) Ltd. (New Delhi, India).

Resin modification

PP was grafted with maleic anhydride by grafting process reported by John and coworkers.⁶ and Hujuri et al.⁵ resulting in MA-g-PP. A 50 g of PP was dissolved in 500 mL of xylene at 100°C. Four grams of maleic anhydride dissolved in a minimum quantity of isopropyl alcohol and 0.4 g of benzoyl peroxide were added to the solution of PP in xylene and continuously stirred. The resulting solution was then heated to 100°C for 7 h. Methanol was then added to obtain a white precipitate of MA-g-PP. FTIR spectrum as reported in our previous article⁵ confirmed the presence of peak characteristic of carbonyl group and PP in MA-g-PP. This was used as a compatibilizer during blending of fiber and resin.

Fiber surface modification

Before composite preparation, the fibers were dewaxed followed with alkali treatment.³ Dewaxing was carried out with 1 : 1 mixture of ethanol and benzene at 60°C for 36 h in a hot air oven, washed with cold distilled water and dried in vacuum oven at 60°C for 24 h. The defatted fibers were then treated with 2N NaOH at 23°C in a climatic chamber for 60 h and washed with acidified (dil HCl) water until the fibers showed no alkalinity reaction. The fibers were then again washed with cold distilled water and dried in vacuum oven at 60°C for 24 h.⁷

Sample preparation

The fibers chemically modified as described earlier were separated into strands and chopped into lengths of 2, 4, 6, 8, and 10 using fiber cutting machine. The required amounts of PALF and PP to be mixed for 10 vol % fraction of the fiber were calculated after determining the density of the PALF according to method described in ASTM D729 and then weighed out with an allowance for flash in the sheet mold of compression molding press.

PALF/PP composites were prepared by melt mixing in two-roll open mill fitted with rows of cartridge heaters, where front and back roll temperatures were maintained at 140°C and 160°C, respectively. The composite sample preparation was carried out in two stages. First, the untreated fibers at different lengths (2, 4, 6, 8, and 10 mm) at 10 vol % fiber loading along with PP were mixed keeping the fibers in longitudinal direction and fed into the preheated roll at 160°C without any compatibilizing agent. The mixing was carried out for 10 min with roller speed 30 rpm. In the second stage, the chemically treated fibers at different lengths (2, 4, 6, 8, and 10 mm) at 10% fiber loading along with PP and compatibilizer MA-g-PP in 5% concentration were mixed keeping the fibers in longitudinal direction at similar conditions, i.e., 160°C and 30 rpm roller speed. These premixes (treated and untreated) were scrapped out of the rollers and almost immediately compression molded to produce sheets (115 × 115 × 3.5 mm) using a 60 T (model-CIPET India) press at platen temperature of 160°C, applying 25 T load for 1 min and then cooling at a contact temperature of 120°C for 5 min. Polyester sheets were placed above and beneath the compression mold to facilitate smooth surface finish of the molded composite sheet. Test specimens were then prepared from these sheets as per ASTM D638 (tensile), ASTM D 790 (flexural), and ASTM D 256 (impact) using contour cut-copy milling machine (CEAST, Italy) with calibrated templates. Further details can be seen elsewhere.⁵

TABLE I
Impact Strength of PALF-PP Composites at 10% (Fiber Volume) Loading

Length of fiber (mm)	Raw fiber + PP (J/m)	Treated fiber + PP + MA-g-PP (J/m)
2	28.74 ± 0.4281	35.95 ± 0.7279
4	35.33 ± 0.5777	51.35 ± 1.4760
6	37.24 ± 0.5009	64.35 ± 0.5877
8	33.09 ± 0.2020	59.46 ± 0.9340
10	30.49 ± 0.8101	54.78 ± 1.5610

Mechanical properties

Tensile strength and modulus

Test samples of composites of untreated fiber + PP and chemically treated fiber + PP + MA-g-PP of dimension 165 × 13 × 3 mm were subjected to tensile test as per ASTM D 638 (Type IV) using Universal Testing Machine (UTM, model H 100 K-S supplied by Hounsfield Equipments, Salfords, Redhill Surrey, England). A load range of 10 kN, crosshead speed of 1 mm/min, and a gauge length of 50 mm were used for carrying out the test.

Flexural strength and modulus

Flexural tests under three-point bending were carried out on similar composite samples of untreated fiber + PP and chemically treated fiber + PP + MA-g-PP having dimension 80 × 12.7 × 3 mm using the same UTM as per IS 13,360 (Part 5/Sec 7): 1996, at a load range of 500 N and crosshead speed of 1.0 mm/min.

Impact strength

Notched izod impact test on specimens having dimensions 63.5 × 12.7 × 3 mm was carried out using a Junior Impactometer (CEAST, Italy) as per ASTM D 256 with a notch angle 45° and depth of 2.54 mm.

For each test, five replicate test specimens were taken and from the obtained experimental data the average value and corresponding standard deviation were calculated.

Thermal properties

Vicat softening temperature

VST was determined on composite samples of untreated fiber + PP and chemically treated fiber +

PP + MA-g-PP having dimensions 10 × 10 × 3 mm using the VST apparatus (Toyo Seiki Seisaku Sho Ltd., Japan) as per Method B 120 of IS 13360 (Part 6/Sec 1): 1999, using a force of 50 N and a heating rate 120°C/h.

For each test, two replicate test specimens were taken and from the obtained experimental data the average value and the corresponding standard deviation were calculated.

Interfacial properties

Scanning electron microscopy

The microstructures of the tensile, flexural, and impact fractured surfaces of the composites were studied with the aid of a variable pressure digital scanning electron microscope (model: LEO 1430 VP) (Cambridge, UK). The substances were mounted on special stubs and given a coating of gold by the sputter coater, which rendered them conductive to be studied under the scanning electron microscope. Coatings were applied at a thickness of about 20 nm, which was too thin to interfere with the dimensions of the surface features.

RESULTS AND DISCUSSION

Fiber surface and resin modification

The PALF fibers after dewaxing and alkali treatment showed loss in weight and became clear white in appearance due to removal of impurities after each treatment process. The chemical treatment of the fibers increased the fiber aspect ratio and matrix compatibility. Chemical treatment also partially removed lignin and hemicellulose. Scanning electron micrographs (SEM) of raw, dewaxed, and alkali-treated fibers have been reported elsewhere.⁵ The

TABLE II
Flexural Strength of PALF-PP Composite at 10% (Fiber Volume) Loading

Length of fiber (mm)	Raw fiber + PP (MPa)	Treated fiber + PP + MA-g-PP (MPa)
2	41.66 ± 1.5680	51.24 ± 0.8111
4	41.62 ± 0.3922	57.80 ± 0.5505
6	45.83 ± 0.5091	61.20 ± 0.3742
8	45.50 ± 0.7910	50.41 ± 0.3662
10	40.96 ± 0.5714	49.10 ± 0.1332

TABLE III
Flexural Modulus of PALF-PP Composite at 10% (Fiber Volume) Loading

Length of fiber (mm)	Raw fiber + PP (MPa)	Treated fiber + PP + MA-g-PP (MPa)
2	1895 ± 3.650	2177 ± 5.305
4	1760 ± 3.162	2300 ± 7.382
6	1850 ± 2.312	2537 ± 4.373
8	1864 ± 2.822	1973 ± 5.551
10	1864 ± 3.996	1945 ± 3.080

raw surface of the fiber consisted of a waxy coating called cuticle that made it to be incompatible with the polymer matrix. A swelling of the fibers was seen on dewaxing. But fibers after alkali treatment became much thinner with obvious surface roughness that led to the increased adhesion between the fiber and the matrix.⁵

Mechanical properties

The variation in impact strength, flexural strength, flexural modulus, tensile strength, and tensile modulus as a function of fiber length both in raw fiber composite and treated fiber composite with MA-g-PP compatibilizer is represented in Tables I–V.

Effect of fiber length on untreated fiber composites

It was observed that the impact strength, flexural modulus, and tensile strength of the untreated PALF-PP composites at 10 vol % fibers loading increased linearly with the increase in fiber length from 2 to 6 mm and then started decreasing. Impact strength is increased by ~30%; flexural modulus marginally by about 3%; and tensile strength by 9%. This increase is primarily attributed to reinforcement effect imparted by the fibers, which allowed stress distribution from continuous polymer matrix to dispersed fiber phase.⁸ Beyond 6 mm size, a downward trend in mechanical properties was noticed due to poor fiber-matrix adhesion resulting in nonuniform transmission of the applied stress. In the case of 6 mm length of the fiber, it was possible to control the orientation in the longitudinal direction better compared with the shorter counterparts which resulted in better properties. As the length is increased beyond 6 mm, during fabrication some

fibers were highly stressed, whereas others remained unstressed and during loaded condition prestressed fibers had to support more load resulting in less observed values when compared with 6 mm. The decrease in mechanical properties at higher fiber loading can be attributed to microcrack formation at the interface on account of poor fiber-matrix adhesion and nonuniform stress transfer due to fiber agglomeration within the matrix.⁹

Studies on mechanical properties only on treated fiber composites without MA-g-PP compatibilizer were carried out and reported at different fiber volume percent loading by us earlier⁵ and it was observed that the properties did not show any improvement but decreased with respect to virgin PP. The decrease in property after dewaxing and alkali treatment was attributed to the fact that the removal of the lignin (binding material in the fiber) and cuticle layer got in the way of binding between the fibers and the matrix and further in the absence of compatibilizer, the two surfaces could not bind chemically and thereby the stresses could not transfer from the fibers to the matrix. Therefore, further studies on mechanical properties with different fiber lengths of treated fiber composites without MA-g-PP compatibilizer were not carried out.

Effect of fiber length on chemically treated fiber composites with MA-g-PP

Incorporation of MA-g-PP reduces the surface hydrophilicity of the fibers. The maleic anhydride groups of MA-g-PP covalently links with the hydroxyl groups of the fibers forming an ester linkage, whereas the nonpolar part (PP) of MA-g-PP becomes compatible with the virgin matrix, lowers the surface energies of the fibers, and thereby

TABLE IV
Tensile Strength of PALF-PP Composite at 10% (Fiber Volume) Loading

Length of fiber (mm)	Raw fiber + PP (MPa)	Treated fiber + PP + MA-g-PP (MPa)
2	22.476 ± 0.2770	26.78 ± 0.3291
4	26.95 ± 0.2621	31.06 ± 0.4551
6	26.84 ± 0.1218	31.85 ± 0.2364
8	19.676 ± 0.1110	26.38 ± 0.1312
10	19.00 ± 0.2055	24.68 ± 0.0659

TABLE V
Tensile Modulus of PALF-PP Composite at 10% (Fiber Volume) Loading

Length of fiber (mm)	Raw fiber + PP (MPa)	Treated fiber + PP + MA-g-PP (MPa)
2	521 ± 4.548	547.40 ± 2.256
4	690 ± 4.000	707.00 ± 1.581
6	691 ± 5.788	710.00 ± 2.170
8	671 ± 4.713	724.20 ± 3.518
10	673 ± 2.434	749.00 ± 3.113

increases its wettability and dispersion within the matrix.¹⁰ This is reflected in the considerably increased values of the mechanical properties at 10 vol % fiber loading in comparison to the untreated or chemically treated composites at the same volume percent of fiber content. The composites prepared with 5% MA-g-PP concentration showed 73% increase in impact properties, 37% increase in the flexural modulus, and 37% increase in the flexural strength at an optimum fiber length of 6 mm arranged in the longitudinal direction orientation. The fiber factors that contribute to the engineering performance of a fiber-matrix composite are orientation, length, shape, and composition of the fibers. Fiber orientation in the matrix is very important and so is the length of the fibers, which can be either continuous in length or short fibers. Although continuous fibers are more efficiently oriented than short fibers, they are not necessarily better. Theoretically, continuous fibers can transmit an applied load or stress from the point of application to the reaction by a continuous load path. In practice, this is not possible because during fabrication some fibers will be highly stressed, whereas others will be unstressed and during loaded condition prestressed fibers will have to support more load. In contrast, composites made from shorter fibers if can be properly oriented could have substantial greater strength than those made from continuous fibers because the shorter fibers can be produced with few surface flaws and can almost attain their theoretical strength. Here, the 6 mm length of the fibers can be more properly oriented possibly due to their optimum size in the longitudinal direction than the shorter ones, which are more difficult to align in the longitudinal direction. Beyond 6 mm, during fabrication, fiber prestressing occurs resulting in decrease in strength. In case of

PALF fiber-reinforced low-density polyethylene composites, George et al.¹¹ also observed that considering the overall mechanical properties and processability characteristics, fiber length of 6 mm was found to be the optimum length.

Thermal properties

The variation in VSTs as a function of fiber length both in raw fiber composite and treated fiber composite with MA-g-PP compatibilizer is represented in Table VI. It is observed that the VST of the untreated PALF-PP composites at 10 vol % fiber loading increased linearly from 104 to 113°C with the increase in fiber length from 2 mm to 10 mm. On the other hand, the VST of the chemically treated fiber composites with MA-g-PP compatibilizer at 10 vol % fiber loading increased linearly from 117 to 122°C with the increase in fiber length from 2 to 6 mm and then attained a saturation point with 8 and 10 mm fiber length at around 122°C.

Although variation in thermal properties such as thermal conductivity and thermal diffusivity have been studied by Mangal et al.¹² by reinforcing varying volume fraction of PALF in PE matrix, little work has focused on the variation in thermal properties such as VST at different fiber lengths and loadings of PALF reinforced PP composites.

There is evidence that transcrystallinity occurs in natural fiber thermoplastic composites due to the nucleating properties of the cellulosic fibers and the presence of the coupling agent is further reported to increase transcrystallinity, which could enhance interfacial strength.¹³ Nielsen et al.¹⁴ suggested that incorporation of fibers or fillers into polymer matrices restricted the mobility of polymer chains and therefore increase in its thermal property of glass

TABLE VI
Vicat Softening Temperature (VST) of PALF-PP Composite at 10% (Fiber Volume) Loading

Length of fiber (mm)	Raw fiber + PP (°C)	Treated fiber + PP + MA-g-PP (°C)
2	104 ± 1.000	117 ± 0.000
4	106 ± 0.000	120 ± 0.500
6	107 ± 0.500	122 ± 0.000
8	111 ± 0.500	121 ± 1.000
10	113 ± 0.000	122 ± 0.000

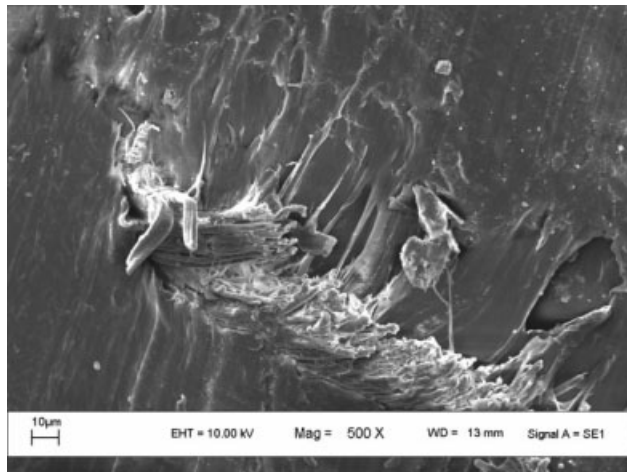


Figure 1 SEM micrograph of tensile fractured surfaces of untreated fiber composite with 6 mm fiber length and 10% fiber volume fraction.

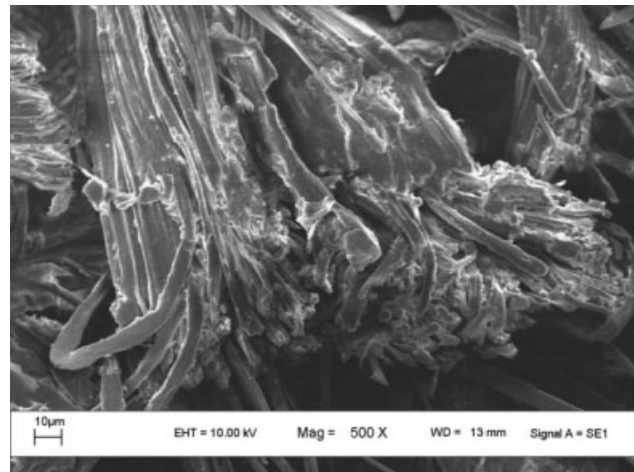


Figure 3 SEM micrograph of impact fractured surfaces of untreated fiber composite with 6 mm fiber length and 10% fiber volume fraction.

transition temperature. Similarly, Hristov et al.¹⁵ observed increase in glass transition temperature in PP–wood flour (10%) composites when compatibilizer was added due to more restricted macromolecules in the amorphous phase, likely to be near the fiber surface.

Thus, in raw fiber composites without compatibiliser there is linear increase in the VST with increase in fiber length due to increase in transcrystallinity and restricted mobility of the polymer chains in the amorphous phase. This is further manifested in presence of chemically treated fiber composites with MA-g-PP compatibilizer due to enhanced interfacial strength. The saturation point in VST with 8 and 10 mm fiber length in chemically treated 10 vol % fiber composites with MA-g-PP compatibilizer could be due to the fact that there is little scope in further

mobility of polymer chains getting restricted and increase in their transcrystallinity.

It is worth mentioning here that we have earlier reported⁵ thermogravimetric analysis of raw fibers, alkali-treated fibers, pure PP, and composites with untreated and treated fibers (10 vol %). There was a marginal improvement in thermal behavior of the fibers after alkali treatment and slight improvement was also observed in the thermal stability of the composite after alkali treatment.

Morphology of the fractured surface

The morphology of the tensile, impact, and flexural test fractured surfaces of both in raw fiber composite and treated fiber composite with MA-g-PP compatibilizer at 10 vol % fiber loading and 6 mm fiber length with orientation in the longitudinal direction

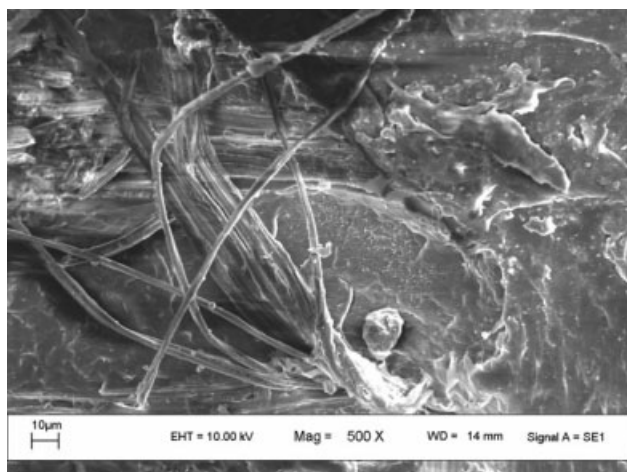


Figure 2 SEM micrograph of tensile fractured surfaces of treated fiber composite with MA-g-PP compatibilizer having 6 mm fiber length and 10% fiber volume fraction.

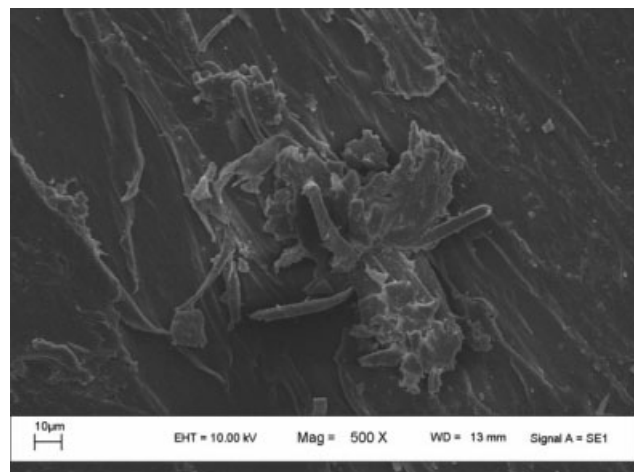


Figure 4 SEM micrograph of impact fractured surfaces of treated fiber composite with MA-g-PP compatibilizer having 6 mm fiber length and 10% fiber volume fraction.

are shown in Figs. 1–6. It is evident from the Figs. 1, 3, and 5 that in the untreated fiber composites there is poor interfacial adhesion and inadequate wetting of the untreated fibers within the PP matrix due to which the untreated fibers in the composites are bunched together tending to agglomerate. This resulted in large number of gaps after the fibers are pulled out from the matrix when stress is applied. The dewaxing followed by the alkali treatment removes the dust and other impurities from the fiber surface resulting in improvement in fiber wetting as is evident from the micrographs in Figs. 2, 4, and 6. Furthermore, because of the presence of MA-g-PP compatibilizer there is an improved fiber-matrix interfacial adhesion as seen from the micrographs, which also show considerable reduction in the gaps and the fibers remaining embedded in the matrix during pull out at the time of fracture.

CONCLUSION

A lot of improvements in the properties of the composites were observed by optimizing the fiber length, orientation, treatment procedure, and appropriate selection of coupling agents.

The mechanical, thermal, and morphological properties of the short PALF reinforced thermoplastic PP composites at 10 vol % fiber loading and varying fiber lengths from 2 to 10 mm have been investigated in this work. It was already observed in our earlier work⁵ that the alkali treatment of the defatted fibers improve the fiber-polymer interaction by enhancing fiber aspect ratio, fiber wetting, and impregnation and further improved by the action of MA-g-PP compatibilizer, which enhances the properties of the composite by chemically binding the fiber and the polymer by grafting mechanism. Here, we

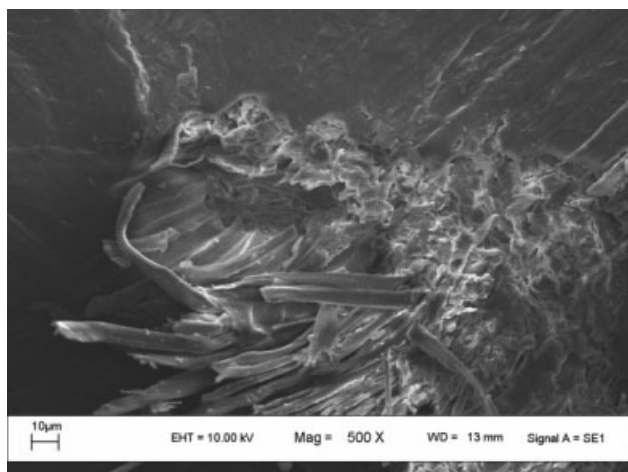


Figure 5 SEM micrograph of flexural fractured surfaces of untreated fiber composite with 6 mm fiber length and 10% fiber volume fraction.

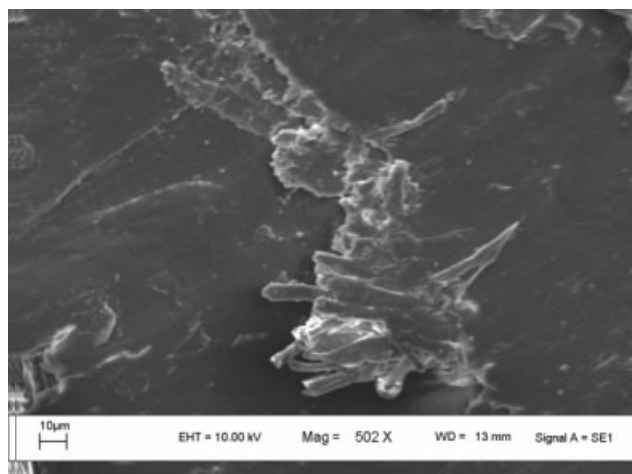


Figure 6 SEM micrograph of flexural fractured surfaces of treated fiber composite with MA-g-PP compatibilizer having 6 mm fiber length and 10% fiber volume fraction.

have observed 73% increase in impact properties, 37% increase in the flexural modulus, 33% increase in the flexural strength, and 14% increase in VST at 10 vol % PALF loading and 6 mm fiber length oriented in the longitudinal direction.

A 6-mm PALF length oriented in the longitudinal direction with 10 vol % volume fraction with MA-g-PP coupling agent is therefore the recommended length, where the impact, flexural, and vicat softening properties are found to be the maximum.

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