

The Effect of Water Absorption on Mechanical Properties of Wood Flour/Wheat Husk Polypropylene Hybrid Composites

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ABSTRACT

The main objectives of this research were to study the effect of water absorption on mechanical properties of hybrid fiber reinforcement for polypropylene composites. The poor resistance towards water absorption is one of the drawbacks of natural fibers. Hybrid filler-polypropylene composites are subjected to water immersion tests in order to study the effects of water absorption on the mechanical properties. Composites specimens containing 30 phr and 40 phr fiber weight were prepared by melt blending process. Water absorption tests were conducted by immersion specimens in distilled water at room temperature for different time durations (24, 48, 72, 96, 120, 144, 168, 192 hours). The tensile, flexural and impact properties were investigated before and after water absorption. The percentage of moisture uptake increased as the increasing order of the filler loading due to the high cellulose content. The phase morphology of wood flour/wheat husk polypropylene hybrid composites were investigated by SEM, the dynamic mechanical properties of the composite are analyzed by DMA & wheat, wood filler interaction are analyzed by FT-IR.

Keywords: Hybrid Polypropylene Composites; Water Absorption; Mechanical Properties

1. Introduction

Natural fibers to reinforce composite materials increased dramatically during the last few years [1]. The primary advantages of using natural fillers in thermoplastics can be listed as low densities, low cost, nonabrasive nature possibility of high filling levels, low energy consumption, high specific properties, biodegradability, availability of a wide variety of fibers throughout the world, and generation of a rural/agricultural-based economy [2]. Natural fibers are subdivided based on their origins, into four types: seed hairs (cotton, kapok), bast-fibers (flax, hemp, jute, and ramie), leaf-fibers (sisal, henequen, coir, and abaca) and wood flour (wheat husk, rice husk) [3]. Hybrid composites are materials made by combining two or more different types of fibers in a common matrix. Hybridization of two types of short fibers having different length and diameter can offer some advantages over using each of the fibers alone in a single polymer matrix. However, hybrid composites using natural fibers are less studied. In this type of studies, the hybrid composite often consists of one natural fiber and one non-natural fiber [4]. The mechanical properties of plant fibers are largely

related to the amount of cellulose, which is closely associated with the crystallinity of the fiber and the micro-fibril angle with respect to the main fiber axis. Fibers with high crystallinity and/or cellulose content have been found to possess superior mechanical properties [5]. These composites have received attention due to the thermoplastic nature of natural filler-thermoplastic composites, which allows processing of the composites using traditional processing techniques and recycling of the resultant products or wastes at the end of their useful life [6]. Fiber-reinforced composite materials offer a combination of strength and modulus that are either comparable to or better than many pure materials [7]. Natural fibers are hydrophilic in nature as they are lignocellulosic, which contain strongly polarized hydroxyl groups. But thermoplastic show hydrophobic nature [8,9]. Natural fibers show a high level of moisture absorption and insufficient adhesion between untreated fibers and the thermoplastics polymer matrix, such as polyolefin's. Due to high water absorption and less interfacial bonding characteristics, the application of these composites are restricted [10]. Improvement in the mechanical properties of such composites requires strong adhesion between the

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natural fiber and polymer matrix. The compatibility between composite components was improved using either physical or chemical modification of the polymer or filler or by using coupling agents [11]. Chemical treatment of the fiber, help to stop the moisture absorption process, chemically modify fiber surfaces and increase the surface roughness in order to increase the interfacial adhesion between the fiber and matrix, resulting in improved mechanical performance of fiber-reinforced composites [12].

The objective of this work are to compare the influence of filler content, compatibilizers and water uptake on mechanical properties of hybrid filler reinforcement polypropylene composites.

2. Experimental Procedure

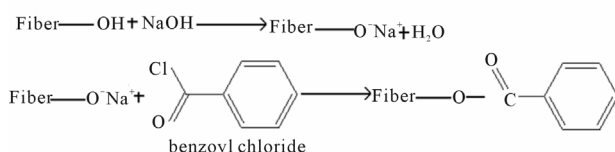
2.1. Materials

The random Polypropylene Copolymer (SRM100 NC) used in this study are a commercial product from Reliance Industries Ltd, Gujarat. The melt flow index (MFI) of polypropylene is 12 gm/10 min (at 230°C & 2.16 kg load) and density of random PP copolymer is 0.91 g/cm, respectively. The MAH-g-PP (OPTIM P-408) is supplied by the PLUSS Polymer Pvt. Ltd, Delhi. Titanate coupling agent (grade-EB 1019 A) is supplied by Industrial Product Mfg. Company, Pune. Wood fiber and wheat husk are provided from Natural Fiber Distributors Pvt. Ltd., Delhi in the form of dust with the particle size less than 20 micron.

2.2. Chemical Treatment

Benzylation

Fibers are pre-treated with 5% NaOH for about half an hour in order to activate the OH groups of the cellulose and lignin in the fiber. Fiber are then washed many times in distilled water and finally dried. An amount of pre-treated fiber are soaked in 18% NaOH solution for half an hour, filtered and washed with distilled water. The treated fibers are suspended in 10% NaOH solution and agitated with benzoyl chloride. The mixture are kept for 15 min, filtered, washed thoroughly with water and dried between filter papers. The isolated fibers are then soaked in ethanol for 1 h to remove the untreated benzoyl chloride and finally were washed with water and dried [12, 13].



2.3. Compounding and Test Specimen Preparation

Hybrid filler reinforced polypropylene composites are prepared by melt blending processes. Polypropylene and hybrid filler are dried at 80°C in an air circulating oven for 2 to 4 h before mixing. And then Polypropylene with bio-filler (10 to 50 phr) at the different loading and 4 phr compatibilizer are compounding done by twin screw extruder Rhecord hake 900(L/D ratio 24:1). During the extrusion process are screw speed was kept 40 rpm [14]. Dumbbell shaped and bar specimens are prepared by semiautomatic injection moulding machine Taxair-40.

3. Measurements

3.1. Water Absorption Test

The effect of water absorption on hybrid fiber reinforced polypropylene composites are investigated in accordance with ASTM D-570. The specimens were dried in an oven at 80°C for 2 hours and then are allowed them to cool to room temperature in desiccators before weighing. Water absorption tests were conducted by immersing the composite specimens in distilled water in beaker at room temperature for different time durations. After immersion for 24 h, the specimens were taken out from the water and all surface water was removed with a clean dry cloth or tissue paper. The specimens were weighed regularly at 24, 48, 72, 96, 120, 144, 168 and 192 hrs exposures. The moisture absorption was calculated by the weight difference. The percentage weight gain of the samples was measured at different time intervals.

3.2. Physical Property

Thickness Swelling Thickness swelling was calculated under the water absorption for 24 hours.

3.3. Mechanical Property

1) Tensile Properties

The tensile tests are carried out according to ASTM D 638 by using INSTRON (M 3382, UK) Universal Testing Machine. The capacity of the machine was 100 KN. The tensile strength and tensile modulus were measured at a crosshead speed of 50mm/min. Gauge length was measured 50 mm. The tests are performed at temperature of 23°C ± 2°C and relative humidity was 50% ± 5%.

2) Flexural Properties

The flexural strength and modulus of random PP/Bio-filler composites are measured according to ASTM D790. The test was carried out on LLOYD (model-LR 100 K, UK) Universal Testing Machine at a crosshead speed of 5 mm/min. A rectangular bar was placed on the

3-point bending configuration at 5mm/min deformation rate.

3) Impact Test

The Izod impact tests are carried out, using a impact tester (CEAST, Italy), according to ASTM D256. V-Shaped notches of 2.5 depths were produced by automatic notcher. The cutter speed and the table feed rate are about 92 and 100 mm/min.

3.4. Dynamic Mechanical Analysis (DMA)

The storage modulus (E') & loss modulus (E'') and the mechanical loss factor ($\tan \delta$), as a function of temperature (T), were determined by dynamic mechanical analyzer by using DMA Q 800, TA instruments (USA). DMA spectra are taken at a frequency of 1Hz, over a broad temperature range ($T = -50^\circ\text{C}$ to 150°C) at a programmed heating rate of $10^\circ\text{C}/\text{min}$ [2].

3.5. Scanning Electron Microscope (SEM)

The Morphology of the composites was examined by using LEO (Cambridge, UK) model 403 Scanning Electron Microscope. The samples are coated with gold prior to examination under the electron beam. An operating voltage 30 KV and magnification of $245\times$ magnification are used. The scanning electrons micrographs are then used to analyze the hybrid filler particles and particles size distribution [15].

3.6. FTIR (Fourier Transform Infra Red Spectroscopy)

A Nicolet 6000 FT-IR, Thermo Scientific, UK are used to obtain spectra for the hybrid fiber. KBr disk sample preparation methods were followed in taking infrared spectra. Fibers are ground and mixed with KBr at the ratio 1:99 then the mixer are pressed under vacuum to form pellets. FT-IR spectra are recorded in a range of $4000 - 400\text{ cm}^{-1}$ at a resolution of 4 cm^{-1} with 64 scans.

3.7. X-Ray Diffraction (XRD)

X-ray diffraction analysis are carried out on SIETRON-ICS x-ray diffractometer in order to confirm the particle size of the hybrid fiber with a diffraction angle of $20^\circ - 60^\circ$ and intensity in the range of 0 to 400 cps [16].

4. Results and Discussion

4.1. Water Absorption

The water absorption of PP/hybrid filler Composites at the different hours using coupling agent (Titanate) and matrix modifier MA-g-PP are studied and shown in **Figure 1** and **Table 1**. Polypropylene composites with the

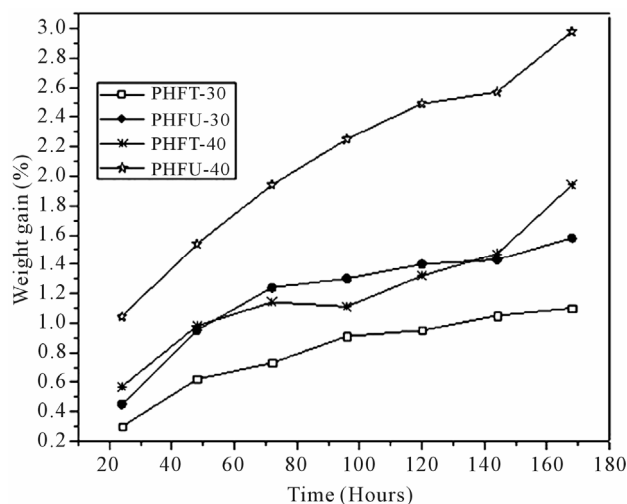


Figure 1. Comparison of water absorption of different batches of PP/hybrid fiber composites.

Table 1. Comparison of water absorption of different batches of PP/ hybrid filler Composites.

Batches		Weight gain in %						
		24 h	48 h	72 h	96 h	120 h	144 h	168 h
PHF-30	Treated	0.30	0.62	0.73	0.91	0.95	1.05	1.10
	Untreated	0.45	0.95	1.24	1.30	1.40	1.43	1.58
PHF-40	Treated	0.57	0.98	1.04	1.06	1.36	1.47	1.94
	Untreated	1.04	1.54	1.94	2.25	2.49	2.57	2.98

treated hybrid filler are shown lesser value than the polypropylene composites with untreated hybrid filler. B. R. Guduri *et al.* reported that [17] polypropylene composites with flax fiber was maximum water absorption of 2.51% at loading at 30%. But in this work maximum water absorption for treated hybrid filler is 1.15% and for untreated hybrid filler 1.78% in polypropylene composites with hybrid filler at the loading of 30 phr.

4.2. Thickness Swelling

The effect of water absorption on the thickness of PP/hybrid filler Composites sample are prepared using coupling agent (Titanate) and matrix modifier MAH-g-PP shown in **Figure 2** and **Table 2**. Polypropylene composites with the treated hybrid filler are shown lesser thickness swelling than the polypropylene composites with untreated hybrid filler due to the lesser water absorption. According the reference [18] in the benzylation chemical treatment minimum water absorption. Hence due to less water absorption less thickness swelling than the untreated hybrid filler polypropylene composites.

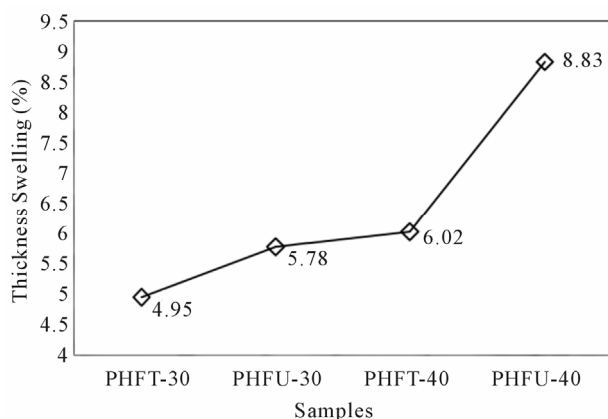


Figure 2. Comparison of thickness swelling % of different batches of PP/hybrid fiber composites.

Table 2. Comparison of thickness swelling % of different batches of PP/hybrid filler composites.

Batches	Loading of hybrid filler (phr)	Thickness swelling in %	
		Treated	Untreated
PHF-1	10	1.42	3.71
PHF-2	20	4.15	4.82
PHF-3	30	4.95	5.78
PHF-4	40	6.02	8.83
PHF-5	50	7.74	10.28

4.3. Tensile Properties

The effect of water absorption on the tensile properties of PP/hybrid filler Composites using coupling agent (Titanate) and matrix modifier MAH-g-PP are studied and shown in **Table 3**. In both case dry and water immersed Polypropylene composites with the treated hybrid filler are show higher value than the polypropylene composites with untreated hybrid filler. According to the reference [19] wood flour with polypropylene composites at the 40% loading tensile strength and modulus are decreased 14% and 29% after water absorbance. But in this work tensile strength and modulus are decreased 29% and 31% at the loading of 30 phr and at the loading of 40 phr tensile strength and modulus are decreased 23% and 27% in the hybrid fiber polypropylene composites.

4.4. Flexural Properties

The effect of water absorption on the flexural properties of PP/hybrid filler Composites using coupling agent (Titanate) and matrix modifier MAH-g-PP are studied and shown in **Table 4**. In both case dry and water immersed Polypropylene composites with the treated hybrid filler are show higher value than the polypropylene composites with untreated hybrid filler.

4.5. Impact Strength

The effect of water absorption on the impact strength of PP/hybrid filler Composites using coupling agent (Titan-

Table 3. Comparison of tensile strength and modulus of different batches of PP/hybrid filler composites.

Batches			Tensile Strength (MPa)		Tensile Modulus (MPa)	
			Treated	Untreated	Treated	Untreated
PHF-3	Dry	30	33.16	29.84	1863.08	1808.70
	Water	30	24.27	23.50	1333.12	1275.71
PHF-4	Dry	40	27.38	25.84	1653.75	1605.08
	Water	40	22.93	21.09	1244.17	1212.28

Table 4. Comparison of flexural strength and modulus of different batches of PP/hybrid filler composites.

Batches			Flexural Strength (MPa)		Flexural Modulus (MPa)	
			Treated	Untreated	Treated	Untreated
PHF-3	Dry	30	45.82	38.03	1457	1324
	Water	30	39.34	33.34	1055	845.1
PHF-4	Dry	40	42.79	36.11	1285	1259
	Water	40	32.86	29.95	787.2	710.5

ate) and matrix modifier MAH-g-PP are studied and shown in **Table 5**. In both case dry and water immersed Polypropylene composites with the treated hybrid filler are show higher value than the polypropylene composites with untreated hybrid filler.

4.6. FTIR (Fourier Transform Infrared Spectroscopy)

Treated and untreated hybrid fiber are analysed using FT-IR to know the various chemical constituents present in them. The FT-IR spectrum of those fibres is shown in **Figures 3(a) and (b)**. The peak at about 2925.5 cm^{-1} is due to the C-H asymmetric stretching from aliphatic saturated compounds in both. But in the treated hybrid fiber one another peak at 2856.2 cm^{-1} is due to C-H symmetric stretching. These two stretching peaks are corresponding to the aliphatic moieties in cellulose and hemicellulose. In the double bond region, a shoulder peak range at 1747 cm^{-1} in the both spectrums is assigned to the C=O stretching of the acetyl and uronic ester of. Groups of hybrid fiber hemicellulose or to the ester linkage of carboxylic group of the ferulic and p-coumaric acids of lignin. But in the treated spectrum another one of C=O stretching peak at 1712.4 cm^{-1} this peak is due to benzoyl chloride at treatment process. Benzene ring in the treated spectrum at the peak range 1573.5 cm^{-1} [20]. Difference between untreated and treated spectrum are explained on the basis of three peaks one symmetric stretching C-H, second two carbonyl groups C=O and last one benzene ring peak.

4.7. Dynamic Mechanical Analysis (DMA)

The dynamic mechanical properties of polypropylene composite with treated and untreated hybrid filler are studied over a wide temperature range. It is an effective means for reflecting the interaction between filler and polymer matrix in a composite by measuring the viscoelastic response of the composite. The storage modulus

Table 5. Comparison of impact strength of different batches of PP/hybrid filler composites.

Batches		Loading of hybrid filler (phr)	Impact Strength (J/m)	
			Treated	Untreated
PHFT-3	Dry	30	64.81	61.72
	Water	30	57.94	51.42
PHFT-4	Dry	40	40.12	38.58
	Water	40	34.71	30.07

Phft-polypropylene hybrid filler treated; Phfu-polypropylene hybrid filler untreated.

and tan delta for composite with treated hybrid filler and untreated hybrid filler at 30 phr loading shown in **Figures 4(a) and (b)** resp. Effect of treatment on storage modulus loss is clearly visible in graph. A relaxation peak is observed in the tan delta graph, which corresponding to the Tg of polypropylene hybrid filler composites. Tan delta for different composites shown in **Table 6**.

4.8. X-Ray Diffraction Analysis

Figures 5(a) and (b) show the XRD patterns in the range of $2\theta = 20^\circ - 60^\circ$, particle size in micron size for treated hybrid filler and treated hybrid filler in polypropylene hybrid filler composites. The micron size of hybrid filler are confirmed by the XRD graphs by using Scherer's formulae. The particle size of micron size of hybrid filler are recorded as 49 micron for treated hybrid filler and treated hybrid filler in polypropylene composites are shows 40 micron, respectively. The particle size distribution of micron hybrid filler measured with Scherer's formulae, is given below

$$\text{Particle size } d (\text{\AA}) = K \times \lambda / \Delta 2\theta \cos \theta$$

where K = order of reflection, value of K = 0.1, $\lambda = 1.542$, and θ = diffraction angle.

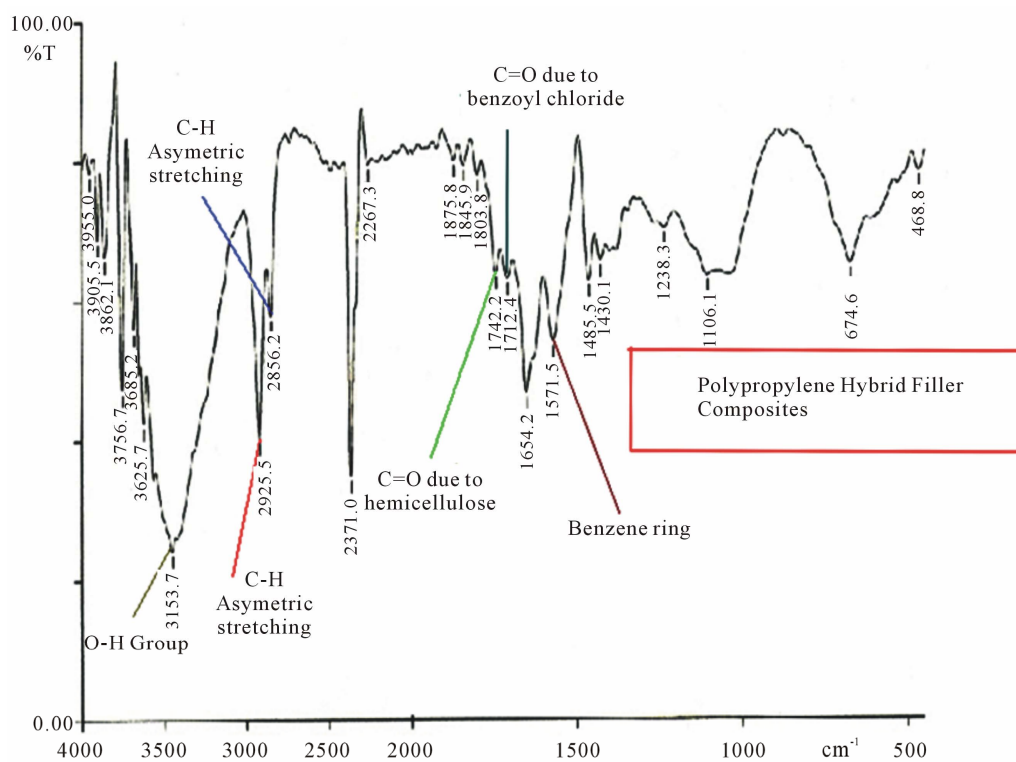
It is studied by the XRD peaks that treated hybrid filler in polypropylene hybrid filler composites show lesser particle size and higher intensity than the treated hybrid filler. Due to higher intensity it is clear that polypropylene composites with treated hybrid filler are highly stable.

4.9. Scanning Electron Microscope (SEM)

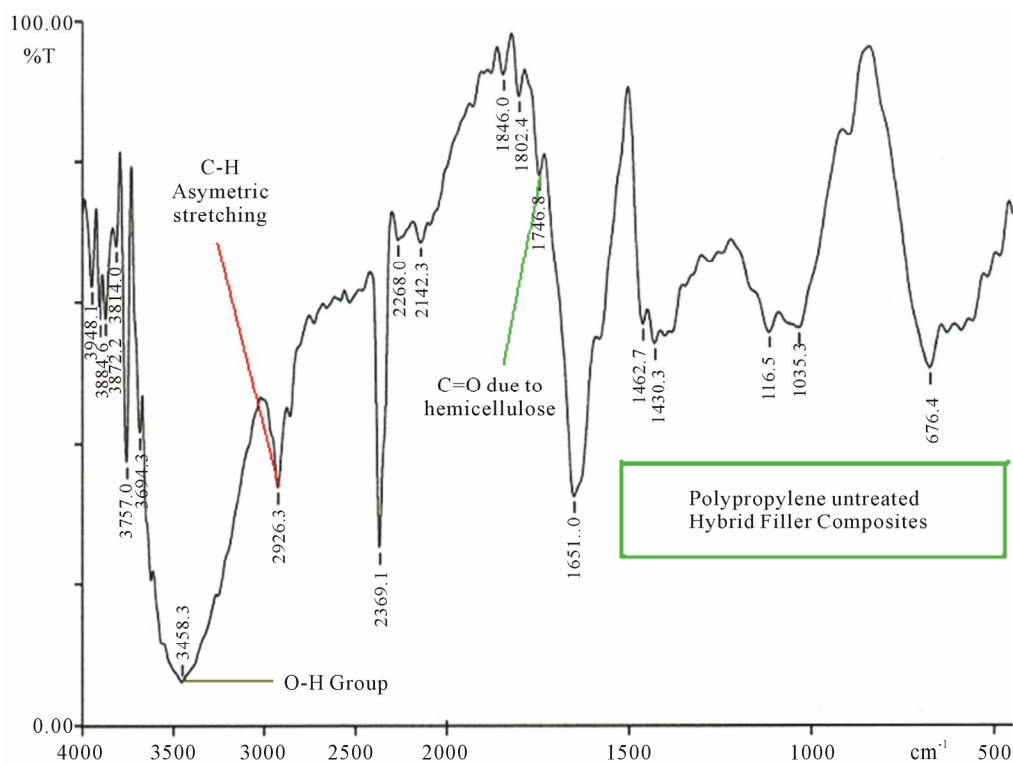
Figures 6(a) and (b) show the phase morphology of PP Composites with treated hybrid filler and untreated hybrid filler. The scanning micrographs show that the filler is homogenously dispersed with in the polymer matrix. It is observed that treated hybrid filler filled composites gives better dispersion of filler than the untreated hybrid filler.

Table 6. Comparison of tan δ of different composition of PP/hybrid filler composites.

Batches	Tan δ	Storage Modulus (E')
PHFT-30	33.66°C	3339
PHFT-40	39.86°C	3250
PHFU-30	41.09°C	1525
PHFU-40	46.14°C	1302



(a)



(b)

Figure 3. (a) FT-IR spectrum of PP/treated fiber hybrid composites; (b) FT-IR spectrum of PP/untreated hybrid fiber composites.

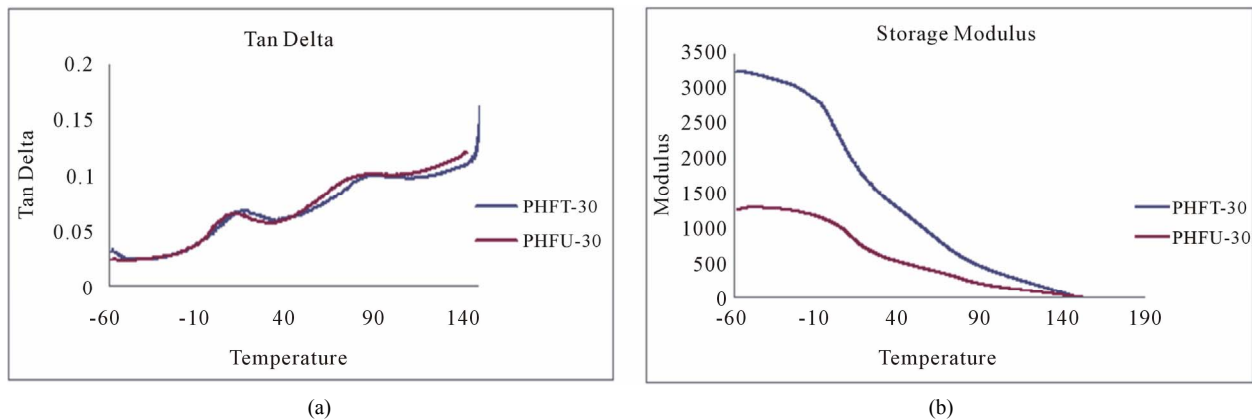


Figure 4. (a) Tan delta graph of PP/treated and untreated hybrid fiber composites; (b) Storage modulus graph of PP/treated and untreated hybrid fiber composites.

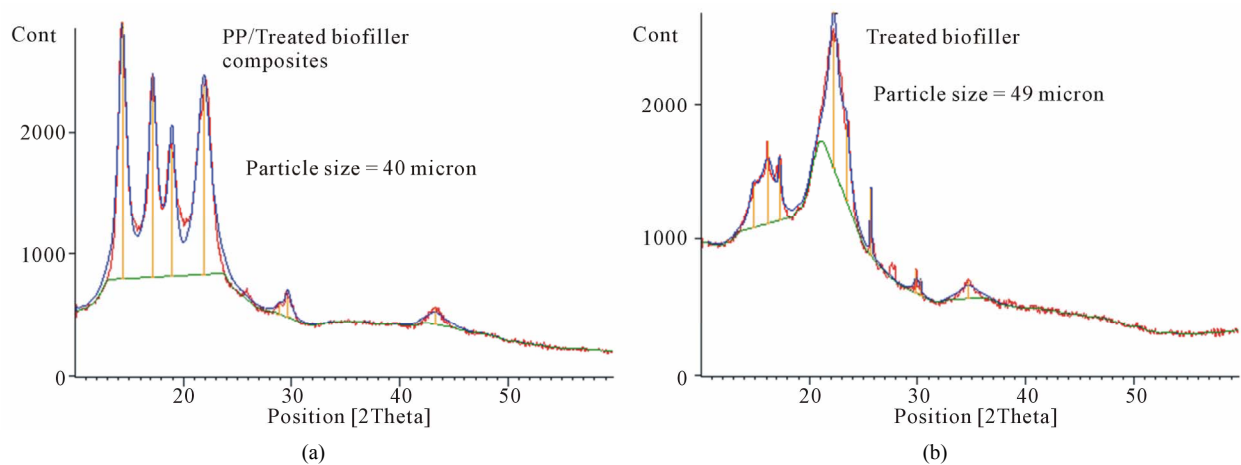


Figure 5. XRD graph of PP/treated fiber hybrid composites.

5. Conclusions

The effects of Treated hybrid filler and untreated hybrid filler loadings on the morphology, mechanical and thermal properties of PP/hybrid filler Composites are investigated and the results can be concluded as below:

- Tensile strength are decreased 29% while modulus are decreased 31% at the loading of 30 phr and at the loading of 40 phr tensile strength are decreased 23% while modulus are decreased 27% in treated hybrid filler polypropylene composites.
- Flexural strength are decreased 15% while modulus are decreased 28% at the loading of 30 phr and at the loading of 40 phr tensile strength are decreased 23% while modulus are decreased 39% in treated hybrid filler polypropylene composites.
- The impact strength are decreased 11% at the 30 phr loading and 13% at the 40 phr loading with treated hybrid filler and 17% at the 30 phr loading and 22% at the 40 phr loading with untreated hybrid filler in polypropylene hybrid filler composites.
- From the DMA graph, α relaxation peak is observed which is corresponding to T_g of the treated hybrid filler and untreated hybrid filler. It is found that PP composites with treated hybrid filler show higher value of T_g as compared to PP composites with untreated hybrid filler.
- SEM revealed that the treated hybrid filler dispersed more homogenously as compare to untreated hybrid filler. The hybrid filler is dispersed finely and uniformly in polymer matrix.
- From the X-ray diffraction, it is noticed that the average particle size of treated hybrid filler are 49 micron and treated hybrid filler in polypropylene hybrid filler composites particle size are shows 40 micron respectively.
- From the FT-IR spectra, it is noticed that the polypropylene composites stability higher with the treated hybrid filler than the untreated hybrid filler and the water absorption are lesser with the treated hybrid filler than the untreated hybrid filler.

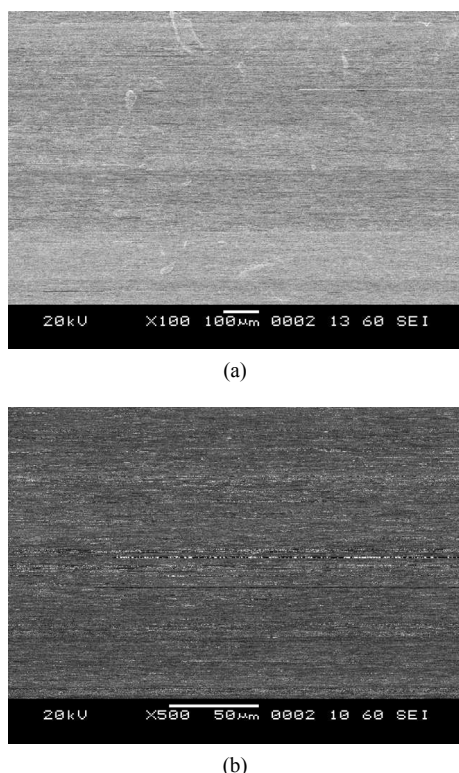


Figure 6. Morphology of PP composites with treated and untreated hybrid filler.

- Above all, the properties of PP/hybrid filler composites are found best property at the loading of 30 phr with treated hybrid filler polypropylene composites.

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