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Mechanical and Thermal Properties of Short Arecanut Leaf Sheath Fiber Reinforced Polypropyline Composites: TGA, DSC and SEM Analysis

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Abstract

Short arecanut leaf sheath (ALS) fiber (2-3 mm) reinforced polypropylene (PP) composites were prepared by compression molding technique. Heat and cold press were used. Chemical composition of the fiber was analyzed and the percents of lignin, α -cellulose and hemicellulose were determined. Fiber content in the composites was optimized with the extent of mechanical properties and composites with 10% arecanut leaf sheath fiber showed higher mechanical properties. Tensile strength (TS), Bending strength (BS), elongation at break (EB%), water absorption capacity, scanning electron microscopy (SEM), thermo gravimetric analysis (TGA), differential scanning calorimetry (DSC) and biodegradation properties of arecanut leaf sheath/PP composites were investigated. ATR spectra of the polypropylene and composites were also analyzed.

Keywords: Arecanut leaf sheath fiber; α-cellulose; Thermo gravimetric analysis; Scanning electron microscopy; Differential scanning calorimetry; Composites

Introduction

Environmental policies of developed countries are increasing the pressure on manufacturers to consider the environmental impact of their products. Therefore, the interest in using natural fibers as a reinforcement of polymer-based composites is growing mainly because of its renewable origin. Polypropylene is an economical material that offers a combination of outstanding physical, chemical, mechanical, thermal and electrical properties not found in any other thermoplastic [1]. Compared to low or high density polyethylene, it has a lower impact strength, but superior working temperature and tensile strength [2].

In our country, *Areca catechu* trees are available in the coastal area which produces huge leaf-sheath. This unusable item can be used to produce composite materials [3]. Several billion pounds of fillers and reinforcements are used in the plastics industry every year. The use of these additives in plastics is likely to grow with the introduction of improved compounding technology and new coupling agents that permit the use of high filler/reinforcement content. As suggested by Katz and Milewski, fillings up to 75 parts per hundred (pph) could be common in the future: this could have a tremendous impact in lowering the usage of petroleum based plastics. It would be particularly beneficial; both in terms of the biodegradability features [4-7] and also in socio-economic terms, if a significant amount of the fillers were obtained from a renewable agricultural source. Ideally, of course, an agro-/bio-based renewable polymer reinforced with agro-based fibers [8-11] would make the most environmental sense.

Arecanut leaf sheath fiber composed of mainly α -cellulose, lignin, and hemicelluloses. In addition, it contains minor constituents such as pectic matters, Fatty and waxy matters. An excellent review by Milewski on short fiber composite technology covers a variety of reasons that result in problems associated with composite properties falling short of their true reinforcing potential. The major factors that govern the properties of short fiber composites are fiber dispersion, fiber length distribution, fiber orientation, and fiber-matrix adhesion. Mixing the polar and hydrophilic fibers with non-polar and hydrophobic matrix can result in difficulties associated with the dispersion of fibers in the matrix.

Interestingly, several types of natural fibers which are abundantly available like arecanut leaf sheath, oil palm, banana, sisal, jute, wheat, flax straw, sugarcane, cotton, silk, bamboo and coconut have proved to be good and effective reinforcement in the thermo-set and thermoplastic matrices [12-19]. Our present research focus on fabrication of agro-fiber reinforced thermoplastics compositespecially, composites made using short arecanut leaf sheath fiber. The ultimate goal of this work is to study the composite potentiality of agro-fiber towards diversified application within environmental legal framework. These composites may be used in the packaging, furniture, housing, construction industries [20], decking, window, door frames [21-24], and automobiles sectors.

Experimental

Materials

Polypropylene (PP) was purchased from Polyolefin Company, Private Ltd., Singapore (Figure 1). Polypropylene granules were grinded to get small particle (50-60 μ m) (Figure 1d) with the help of grinder for proper and homogeneous adhesion between fibers and matrix. Arecanut leaf sheath fibers (Figure 1b) were prepared from arecanut leaf sheath (Figure 1a) by soaking the leaf sheath into water for 15 days. The water loosed the fiber from the resin and waxy materials and then the fibers peeled from the resinous materials, washed with clean water and air dried properly. The arecanut leaf sheath fibers were chopped into small pieces (2-3 mm) with the help of hand scissors and cleaned with mesh and all dirt's are removed from the chopped fiber. Then the chopped fibers (Figure 1c) were cleaned with distilled water

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and exposed thoroughly to sunlight for about 24 hours. The fibers were dried at 100°C in a vacuum oven for 5 hours prior to the preparation of the composites.

Chemical composition of arecanut leaf sheath (ALS) fiber

Arecanut leaf sheath fiber contains α – cellulose, hemicellulose, lignin, aqueous extract, fatty and waxy matters, and pectic matters. In Table 1, the chemical composition of arecanut leaf sheath fiber is shown. The fiber mainly contains 66.08% of α -cellulose, 19.59% of lignin, and 7.40% of hemicellulose. Arecanut leaf sheath fiber is composed of small units of cellulose surrounded and cemented together by lignin and hemicellulose.

Composite fabrication

Composites were prepared by compounding with extrusion and hot press machine. The mould $(12\times15 \text{ cm}^2)$ was cleaned with wax as a releasing agent. The mixture of dried arecanut leaf sheath fiber and polypropylene powder was prepared according to the Table 2 that showed different weight fraction (5%, 10%, 15%, 20% and 25% fiber) and poured into the mould. The processing temperature was maintained at 190°C for 5 min under 5 bar consolidation pressure in the heat press (Carver, INC, USA Model 3856). The molds were then cooled for 1 min in a separate press under 5 bar pressure at room temperature. Figure 2 showed the final composite product.

Tensile strength and bending strength test

The tensile properties of the composites (F-F) were determined using a universal testing machine (model H50 KS-0404, Hounsfield Series S, UK). The load capacity was 5000 N; efficiency was within $\pm 1\%$. The crosshead speed was 10 mm/min and gauge length was 20 mm. For bending properties measurement, the crosshead speed was 10 mm/ min, and span distance was 40 mm. Tensile strength measurements and three point bending tests were carried out according to DIN 53455 and DIN 53452 standards methods, respectively.

Water uptake

Five composite samples $(20 \times 10 \times 2 \text{ mm}^3)$ were immersed in the beaker containing 100 ml of deionized water at room temperature (25°C) for different time periods (up to 60 h). Weight of the samples was determined initially then after certain periods of time, samples were taken out from the beaker and wiped (5 times) using tissue papers, and then weighed again. The weight gained, i.e., water uptake of the samples was determined by subtracting the initial weight from the final weight.

Soil degradation tests of the composites

Cellulose has a tendency to degrade when buried in soil whose moisture level is at least 25%. For this purpose, all composite samples were buried in garden soil for a period of 24 weeks. After certain periods of time, samples were carefully withdrawn, washed with distilled water, and dried at a temperature of 80°C for 8 h and then kept at room temperature for 24 h. The change of tensile strength was periodically noted in order to determine the degradable character of the samples in this environment.

Attenuated total reflectance (ATR) analysis

100% Polypropylene and composite were analyzed by Attenuated Total Reflectance (ATR) and spectra were recorded in the 4,000-650 cm^{-1} region on a Perkin Elmer instrument.

S. No	Name	%
1	Aqueous Extract	0.72
2	Fatty and waxy matters	5.06
3	Pectic matters	1.15
4	Lignin	19.59
5	α - cellulose	66.08
6	Hemicellulose	7.40
	Total	100

 Table 1: The chemical composition of arecanut leaf sheath fiber.

Formulations	Fiber content (wt %)	Polypropylene Powder (wt %)
F ₁	5	95
F ₂	10	90
F ₃	15	85
F ₄	20	80
F₅	25	75

Table 2: Showed different weight fraction (5%, 10%, 15%, 20% and 25% fiber) and poured into the mould.



Figure 2: Image of finished product (composite).

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Thermo gravimetric analysis and differential scanning calorimetry analysis

The thermograms and differential scanning calorimetry of the polypropylene and Fcomposite were recorded on a NETZSCH instrument (model no. STA 449 F3, Jupiter) in the temperature range of 0-900°C.

Scanning electron microscopic (SEM) investigation

The fracture surfaces of the tensile specimens of both F and F composite samples were examined using a Hitachi S-4000 field emission scanning electron microscope, operated at 5 kV. Samples were mounted with carbon tape on aluminum stubs and then sputter coated with carbon tape on aluminum stubs and then sputter coated with platinum and palladium to make them conductive prior to SEM observation.

Results and Discussion

Mechanical properties of the composites

The prepared composites were cut into desired size. Mechanical properties such as tensile strength, bending strength and elongation at break were measured. According to Figure 3, the highest tensile strength value observed for F formulation and the value is 28.7 MPa. For the formulations of F, F, F tensile strength values are gradually decreased.

Same condition is observed for bending strength. In the Figure 4, F formulation showed the maximum value (46.9MPa). From F to F the value is increased and from F to F the bending strength values are gradually declined.

From Figure 5, it is clear that the % elongation at break reduces with the increase of fiber in the composites. For F, F, F, F and Fformulations the values % elongation at break are 15.2, 13.8, 11.6, 8.9 and 6.4 respectively.

From the above results, it is observed that fiber content in the composites has the significant influence on the mechanical properties. 10% fiber with 90% PP prepared composite showed the maximum tensile strength and bending strength due to better fiber-matrix adhesion. The percentage of fiber content in the composites (above 10 wt %), TS and BS of the composites showed a decreasing trend, which may be attributed to the fact that increasing fiber content in the composite decreased the fiber-matrix adhesion.

Water uptake of the composites

Water uptake values of the F, F, F, F and F samples were calculated by immersing the samples in de-ionized water contained in a static glass beaker at room temperature. The samples were taken out of water after constant time interval and their mass gain were calculated. The results of water uptake values of the samples are shown Figure 6. F, F and F samples gained water up to 30 h whereas F and F samples gained water up to 20 h of soaking, and then the values were almost constant. The minimum amount of water was taken up by the F sample (0.15%) and the highest amount of water was counted by F sample (0.27%) at the maximum period of observation (60 h). Water was taken up by the F sample was 0.16% that was near about minimum value (0.15%).

Degradation tests of the composites

F, F, F, F and F samples were buried in soil for up to 24 weeks in order to study the effect of such an environmental condition on the

degradability of the samples. TS values are plotted against degradation time (weeks) and are shown in Figure 7. It was found that for all samples TS were decreased slowly with degradation time. After 24 weeks of soil degradation, F, F, F, F and F samples decreased almost 33, 20, 35, 37 and 42% of TS. This is clear that F sample retained its tensile properties more than other samples during soil degradation. Arecanut leaf sheath fiber is a natural biodegradable fiber and a cellulose-based fiber, which absorbs water within a couple of minutes due to strong hydrophilic character. Cellulose has a strong tendency to degrade when











Figure 7: Degradation of tensile strength of the composites during soil degradation tests.

buried in soil [25]. During soil degradation tests, water penetrates from the cutting edges of the composites in arecanut fiber based samples and degradation of cellulose occurred in the fiber and as a result, the mechanical properties of the composites decreased significantly.

ATR analysis

ATR spectra is used to measure the change of surface composition of the PP granules and optimized (wt %) fiber reinforced PP composite. Figures 8 and 9 showed the ATR record of PP granules and optimized (wt %) fiber reinforced PP composite respectively. The region from 1450 to 600 cm⁻¹ is called as fingerprint region and the absorption bands in the 4000 to 1450 cm⁻¹ region are usually because of stretching vibrations of diatomic units, and known as the group frequency region. The C-OH bending peak is observed at 650 cm⁻¹ which is absent in the pure PP granules (Figure 8). C-H vibration peak is showed at 2916 cm⁻¹ in the both figures (Figures 8 and 9).

Thermo gravimetric analysis and differential scanning calorimetry analysis

The thermograms of both PP granules and optimized (wt %) fiber reinforced PP composite were presented in Figures 10 and 11 respectively. From the Figure 10 is observed that degradation start at 380°C and 98.73% mass change is completed at 500°C whereas Figure 11 showed that mass change start at 280°C and 10.38% mass change is completed at 420°C. In case of Figure 11 degradation starts earlier which may be attributed to the fact that fiber-matrix adhesion in the composite. DSC is used to identify melting temperature and calculate the amount of energy absorbed or released by the PP granules and optimized (wt %) fiber reinforced PP composite when these are heated. From the Figures 10 and 11 it is observed that endothermic reactions are occurred in the both cases and 477.2°C and 477.6°C heat absorbed respectively.



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SEM analysis

Conclusion

Interfacial properties of arecanut leaf sheath short fibers reinforced polypropylene composites (Fand F) were investigated by SEM (Figures 12 and 13). SEM observations indicated that there is a considerable difference in the fiber-matrix interaction between F and F composites. Some gaps between fiber and matrix are clearly found for Fcomposite which are responsible for the low mechanical properties. On the other hand Fcomposite showed better fiber-matrix adhesion and gaps between fiber and matrix are not observed which are responsible for higher mechanical properties. Arecanut leaf sheath short fibers reinforced polypropylene composites were prepared by compression molding. Fiber content in the composites was optimized with the extent of mechanical properties and 10% fiber content in the composite showed higher mechanical properties. SEM supported the idea that above 10% fiber content, mechanical properties were decreased with increasing fiber content due to poor fiber-matrix adhesion. Elongation at break decreased with the increased of fiber (wt %). Water uptake behavior of optimized composite was almost same of the lowest fiber content in the composite.







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Soil degradation studies demonstrated that TS of the optimized composite (F) could be lower than that of the other composites with respect to their degradation time. These new composites would make possible to explore new applications and new markets in the packaging, furniture, housing and automotive-aviation-shipping sectors.

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