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Effect of compatibilizer on mechanical properties of chemically treated coir/polypropylene composite

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Composites of NaOH treated coir fibre and maleic anhydride functionalized polypropylene (MAHPP) with different formulations have been made by compression molding technique and the effect of compatibiliser on mechanical strength, flexural strength, and impact properties of the composites are measured and then compared with theoretically calculated values. It is observed that the tensile strength, tensile modulus, Izod impact strength and flexural strength of surface treated and MAPP coupling composite properties are enhanced significantly than the untreated one. This is due to better adhesion between fibre and matrix. This is also confirmed by SEM, interfacial shear strength or fibre pull-out tests and trans crystalline behavior of the composites constituents in the melt form.

Keywords: Coir fibre, Composite, Compression moulding, Polypropylene

1 Introduction

The use of natural fibres, derived from renewable resources, as a reinforcing material in both thermoplastic and thermoset matrix composites provides a positive environmental benefits with respect to disposability and raw material utilization[1]. The natural fibre composites could be very cost effective material for applications in building and construction industry, furniture, storage devices, electric devices, transportation and everyday applications like lampshades, helmets, suitcases, etc[2]. There are many advantages of natural fibres over traditional reinforcing materials such as glass fibres, talc and mica[3] due to their acceptable specific strength, low cost, low density, non-abrasive, good thermal properties, enhanced energy recovery and biodegradability[4]. However, few problems related to natural fibres, such as low and variable strength, poor resistance to weather; and poor wettability with various polymeric matrices, make them less desirable than synthetic fibres as reinforcing material. It has been observed and demonstrated by researchers that the control on fibre-matrix interfacial bond strength is a critical factor to achieve the best mechanical properties of the composite materials[5]. In the natural fibre reinforced composite, high level of moisture content in fibres leads to debonding with age due to the poor exchange of moisture between fibre and matrix[6]. In literature, surface treatment of the natural fibres and use of suitable coupling agents has been tried to enhance the compatibility between fibre and matrix. Some natural fibres, like jute[7], kenaf[8], pineapple[9] and sisal[10], have been treated with alkali and plasma[11] before reinforcing with different matrices, such as vinyl ester, epoxy, polystyrene and unsaturated polyester respectively. Tensile strength of coir fibres increases by 15% when treated with 5% NaOH solution at 28±1° C for 72 h[21]. Hence, in this study, 5% of NaOH is used for coir fibres surface treatment. It has been shown an increase in adhesive ability results in significant enhancement in mechanical and flexural properties of composite as compared to untreated one.

Coir is a versatile lignocellulosic fibre having certain advantages over other natural fibres as it possesses high weather resistance due to higher amount of lignin[12]. Coir is found to be a poor reinforcement for polymers due to its large and variable diameter, high microfibrillar angle and high lignin and hemicellulose contents[13]. Bleached composite[14], alkali-treated coir fibre with PBS matrix[15] and sodium chlorite treated lignin-free coir fibre wheat gluten composite[16] show significant enhancement in micro hardness, water sensibility and mechanical properties than those of untreated coir

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fibres. The adhesion between fibre and matrix could be increased by using coupling agent into a composite. The HDPE/coir composite with stearic acid as coupling agent shows enhancement in mechanical as well as thermal stability of composite[17].

Polypropylene (PP) is a widely used polymer as a matrix in different type of composites because of its low cost, hydrophobic and inert nature, low density, excellent resistance to chemicals and biological organisms, and the wide range of applications[18]. Maleic anhydride-grafted polypropylene (MAPP) is a good coupling agent as it is widely used for coupling in natural fibre reinforced composite[19].

The present study focuses on the effect of coupling agent and alkali treatment on the properties of coir fibre reinforced maleic anhydride grafted polypropylene composites. Chemically treated coir fibre reinforced polypropylene composite with an addition of MAPP as coupling agent are expected to improve composite properties. The experimental values of composites are compared with theoretically calculated values.

2 Materials and Methods

2.1 Materials

Coir fibres having an average diameter 310 μm and length 170 mm were procured from Kerala (India) and used as reinforcing material. Polypropylene having melt flow index 38 g/10 min at 230°C/2.16 kg, supplied by Reliance Industries India, was used as matrix. Maleic anhydride grafted polypropylene (MAPP) having 0.5 % maleic anhydride content, melt flow index 40 g/10 min at 190°C was procured from Pluss Polymer, Faridabad, India and used as coupling agent. Sodium hydroxide (NaOH) and acetic acid were purchased from Merck, India for surface treatment of coir fibres.

2.2 Methods

2.2.1 Surface Treatment of Coir Fibres

The surface modification of coir fibre is carried out by using sodium hydroxide. In this process, fibres were treated with 5% (w/v) aqueous NaOH solution for 24 h at room temperature (24 ^{0}C)[6]. Then, these fibres were neutralized with acetic acid followed by washing with distilled water for complete removal of residual sodium hydroxide. Subsequently, the coir fibres were dried at 60°C for 24h. These dried fibres were chopped with cutter manually into an average length of about 15 mm.

2.2.2 Melt Spinning and Drawing of MAPP Blends as Coupling Agent

To improve adhesion between fibre and polymer matrix, coupling agent maleic anhydride grafted polypropylene (MAPP) was used. Incorporation of maleic anhydride grafted polypropylene in chips formed during composite fabrication is difficult and hence the melt spinning of maleic anhydride grafted polypropylene (MAPP) was carried out in hydraulic melt spinning unit to get its filament form. The following conditions were maintained during spinning:

Polymer throughput rate : 0.5 g/min
Melt temperature : 285^{0}C
Spinneret diameter : 0.05 cm
Ambient air temperature : 30^{0}C
Uptake velocity : 50 m/min

Maintaining above spinning conditions, filament yarn of 90 denier was produced and drawn four times for final application. Drawn filaments were chopped into mean average length of 15 mm.

2.2.3 Tensile Strength of Coir, PP and PP/MAPP Blend Fibres

The tensile strength of coir fibres (untreated and alkali treated), PP and PP/MAPP blend fibres was measured with 20 mm gauge length by using Instron tensile tester based on constant rate of extension (CRE), pressure applied on jaws 50 cN, operating at 27°C and crosshead speed of 50 mm/min. The samples were dried for 4 h in air circulated oven at 105°C to remove moisture. A minimum of 30 samples were tested to evaluate tensile properties.

2.2.4 Fabrication of Random Oriented Fibrous Composite

Three different types of the composites were fabricated using PP as matrix by compression molding machine. In order to make the random oriented fibrous composite with and without MAPP, completely dried 15 mm coir fibre and 15 mm MAPP fibres were spread over 20×20 cm^2 specimen of 100 GSM PP nonwoven uniformly with different formulations (Table 1). Number of this PP nonwoven sandwich having coir and MAPP fibres was taken in such a way that the final composite thickness is maintained as 5 mm. During composite formation in compression molding machine, initial pressure was

Table 1 — Base formulation for fabrication of composite

Coir fibre	Coir fibre vol. %	PP vol. %	PP/MAPP vol. %
Untreated	25	75	-
Alkali treated	25	75	-
Alkali treated	25	70	5

set at 0 bars and temperature was raised to 185°C. Then the pressure was raised up to 8 bars. The system was held for 15 min and then the pressure and temperature were gradually lowered to 0 bars and 24^0C respectively. The effect of alkali treatment on surface morphology of coir fibre was investigated under scanning electron microscope (Zeiss EVO 50 from Cambridge) at ×200 magnification.

The actual isothermal crystallization behavior of control PP, MAPP and PP/MAPP in the composite was investigated by hot stage platform integrated with optical light polarizing microscope at ×100 magnification. During this investigation, the control PP, MAPP and PP/MAPP were melted at 200°C and kept at constant temperature for 1 min for uniform melting. Then the temperature was lowered at the rate of 10°C/min up to 130°C and subsequently the crystallization behavior of these samples was observed.

2.3 Characterization of Composites

2.3.1 Interphase Transcrystallinity of Composites

The interphase transcrystallinity of composites was observed after 5 and 15 min at isothermal condition by a Mettler FP 900 automatic hot stage thermal controller integrated with an optical polarizing microscope (LEICA DMPL) at ×100 magnification. To observe transcrystallinity, the prepared composites were placed on hot stage microscopy. Samples were heated to 220°C to remove the polymer morphological history, and crystallization was conducted under isothermal conditions at 130°C at same heating and cooling rate of 10°C/min. As soon as the transcrystalline zones were formed, the images were captured after quenching the sample.

2.3.2 Single Fibres Pull-out Strength Test

Single fibre pull-out test of composites was performed by universal testing machine Instron 4442 with a load cell of 10 kg and a crosshead speed of 5 mm/min. A single fibre pull-out test from composite was performed as per ASTM D-638 to measure the interfacial shear strength (IFSS). The disoriented fibre composites were made by using compression molding machine from SANTEC (India). The prepared 3 mm thickness composite was quickly cooled in the air and removed from a mold. The fibre embedded length in the polymer matrix was obtained by cutting the fibre by punching a hole through the specimen. The schematic representation of single fibre pull-out the test is shown in Fig. 1; in which *d* mean diameter of coir fibre measured by an optical microscope and *L* is embedded length in the matrix. Tensile force, applied on the free end of fibre to pull it out of the matrix, was continuously monitored and recorded. IFSS value of composites was estimated from the maximum debonding force (F_d) using following equation:

$$\tau = \frac{F_d}{\pi \times d \times L} \quad \ldots (1)$$

where d is the mean diameter of the coir fibre measured by an optical microscope; and L, the embedded length in matrix. For each sample, 10 readings were taken and averaged value was recorded.

2.3.3 Surface Morphology of Fractured Composites

The surface morphology of fractured untreated and alkali-treated coir fibre, composite with incorporation of MAPP and pull-out fibre from composite was observed by scanning electron microscope (Zeiss EVO 50, Cambridge) at ×50 magnification.

2.3.4 Mechanical Properties of Composite

The tensile strength, tensile modulus and elongation % of the prepared composites made by different compositions were evaluated as per ASTM D-3039 standard method. For each sample, 15 readings were taken and their averaged value was recorded.

2.3.5 Impact Strength

To evaluate the resistance of the prepared composite material to fracture, Izod impact test was conducted using Tinius Olsen impact tester model impact 104 from the USA. The measurement was carried out as per ASTM D -256.

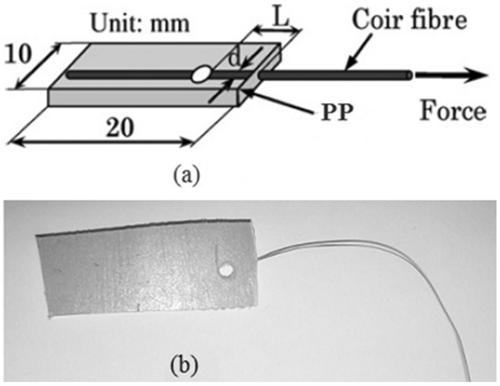

Fig. 1 — Schematic representation of single fibre pull-out test, and (b) original sample

2.3.6 Flexural Strength

Three point bending strength of composites material was determined using ZWICK/ Z010 tensile testing machine from Germany as per ASTM D 790-03 standard. For each sample, 10 readings were taken and their average value was reported. Following equation was used to determine flexural strength:

$$\text{Flexural strength } (\sigma_f) = 3PL/2bd^2 \quad \ldots(2)$$

where σ_f is the stress in the outer surface at midpoint (MPa); P, the load at a given point on the load-deflection curve (N); L, the support span (mm); b, the width of sample tested (mm); and d the depth (height) of sample tested (mm).

3 Results and Discussion

3.1 Surface Morphology of Alkali Treated and Untreated Coir Fibres

SEM image of untreated coir fibre [Fig. 2(a)] shows the typical morphology of coir fibre covered with a layer, which includes lignin, pectin and other impurities. SEM image of alkali treated coir fibre [Fig 2(b)] shows nodes and rows of pits on the surface due to the removal of pectin and lignin cover after the alkali treatment. Total weight loss of 15% in coir fibre is also observed after alkali treatment.

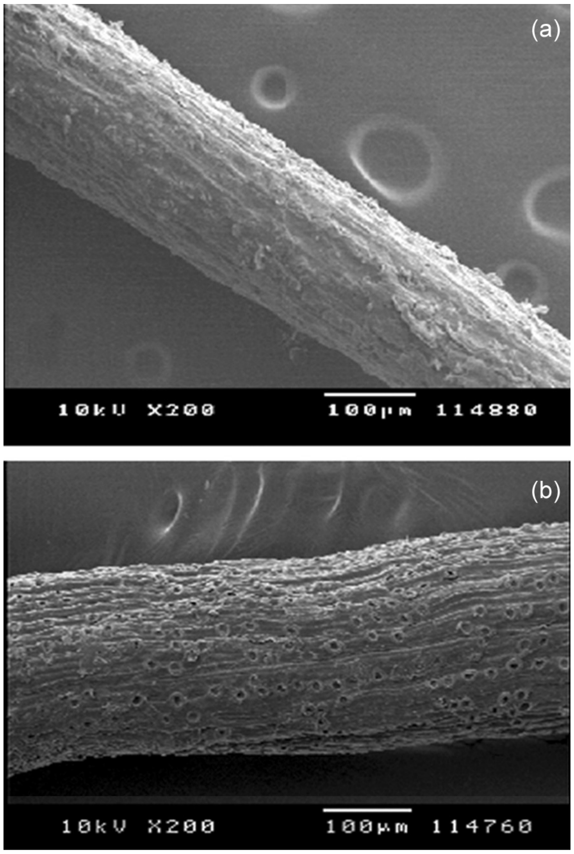

Fig. 2 — SEM images at ×200 magnification (a) untreated, and (b) alkali-treated coir fibre

3.2 Tensile Strength of Coir, PP and PP/MAPP Blend Fibres

Tensile strength values of coir, PP and PP/MAPP blend fibres are given in Table 2. After alkali treatment of coir fibres tensile strength decreases due to the removal of lignin and pectin from the fibre surface. After addition of MAPP in PP, the tensile strength and modulus increase due to more crystallization behavior of MAPP than PP in blend.

3.3 Crystallization Behavior of Control PP and its Blends

The actual crystallization behavior of PP, MAPP and PP/MAPP in the composite has been investigated using hot stage melt combined with an optical polarized microscope as per the procedure discussed above. It is found that the PP/MAPP blend and MAPP both show more spherulitic sites and a finer morphology. This indicates that MAPP might be acting as a nucleating agent. MAPP addition in PP polymer accelerates the nucleation rate via heterogeneous nucleation, while PP crystallizes via both heterogeneous and homogeneous nucleation. It is also observed that the radial growth rate of the spherulites decreases slightly with the addition of 5 % MAPP in PP polymer matrix. This may be because of the presence of a small amount of MAPP in the PP melt which influences the crystallization of PP and leads to a reduction in homogeneous nucleation and an increase in the number of effective nuclei.

3.4 Characterization of Composites

3.4.1 Interphase Transcrystallinity of Composites

The interphase characteristics of composite influence the physical and mechanical properties of the bulk composite, as it plays a very important role to decide the stress transfer efficiency of the composite. The interphase transcrystallinity of composites are seen by an optical polarizing microscope as per the procedure discussed above. From Fig. 3, it can be observed that spherulites nucleate at fibre edge and then grow outwards. It can also be seen that there is

Table 2 — Tensile properties of coir and polypropylene fibres

Fibre	Tensile strength MPa	Elastic modulus GPa
Untreated coir	137.6	2.0
Alkali treated coir	120.4	1.8
Polypropylene	25.8	1.9
Polypropylene/MAPP blend	28.9	2.1

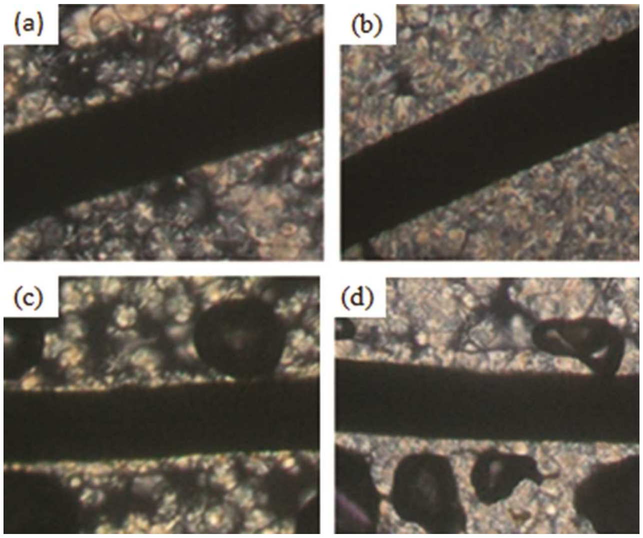

Fig. 3 — Optical polarized microscope images of trans crystalline interphase in composites with PP (All micrographs are taken under reflection mode with ×100 magnification) [(a) after 5 min, (b) 15 min with PP (c) after 5 min, and (d) 15 min with MAPP]

no interphase (trans crystallinity) around the coir fibre in coir/PP composite after 5 and 15 min. Composite with 5% MAPP shows brush like interfaces around the coir fibre after the 5 and 15 min time. This indicates that the crystallization rate has been higher for the coupled system due to the presence of MAPP. The enhanced crystallization may be one of the reasons for the formation of transcrystalline growth. This type of interaction in the composite might be helping to enhance the properties like tensile strength, impact strength and flexural strength.

3.4.2 Interfacial Shear Strength

Interfacial strength of composite in terms of resistance to single fibre pull- out from the composite system has been investigated as per the procedure discussed above. The PP/untreated coir composite shows an increase in force gradually till it reaches a maximum value, and then the force suddenly drops to a lower value. Subsequently, the fibre keeps sliding along the hole until the total embedded length of the fibre pulled out of PP matrix. This behavior is due to the smooth fibre surface and poor interface because of the incompatibility between hydrophilic fibre and hydrophobic matrix. After alkali treatment of fibres, IFSS value improves because of the rough surface and formation of pits which help in increasing mechanical bonding between the matrix and coir fibres in the composite. In addition, MAPP improves adhesion between coir fibre and polypropylene matrix and results in 185% improvement in IFSS value. The coupling agent acts as a compatibiliser for polar natural fibre and non-polar polymer matrix systems. The enhancement in IFSS value can be correlated with critical fibre length. This is the minimum fibre length which is necessary for effective strengthening and stiffening of the composite material. The critical length (l_c) can be calculated using the following equation:

$$Ic = \frac{\sigma_f \times d_f}{2\tau_f} \quad \ldots (3)$$

where d_f is the average diameter of coir fibre; σ_f, the tensile strength of coir fibre; and τ_f an interfacial shear strength of the composite.

For PP/untreated coir composite, critical fibre length is:

$$l_{c1} = \frac{137.6 \times 310}{2 \times 2} = 10.7 \text{ mm}$$

For PP/ alkali treated coir composite, critical fibre length is:

$$l_{c2} = \frac{120.4 \times 310}{2 \times 3.5} = 5.3 \text{ mm}$$

For PP/alkali treated coir/MAPP added composite, critical fibre length is:

$$l_{c3} = \frac{120.4 \times 310}{2 \times 5.7} = 3.3 \text{ mm}$$

From the above calculations, it can be seen that critical fibre length decreases with increase in interfacial shear strength due to strong mechanical and chemical bonding of fibre with matrix.

3.4.3 Fractured Surface Morphology of the Composites

Surface morphology of fractured composites and pull-out fibre from PP/alkali treated coir with MAPP composite is investigated using scanning electron microscope (SEM). Figure 4(a) shows that poor interfacial adhesion of untreated coir fibres with polymer matrix results in easy fibres pull-out from the composite. After alkali treatment of coir fibres, fibre pull-out from composite reduces, indicating better adhesion. The better adhesion can be attributed to change in surface due to removal of the lignin and formation of regular pits which helps to provide better mechanical interlocking to a polymer matrix. The composite with 5% of MAPP shows few fibres pull-out from it. This can be attributed to strong interfacial adhesion/bonding between fibre and matrix due to the better compatibility between PP/MAPP and polymer matrix.

3.4.4 Mechanical Properties of Composites

(i) Tensile Properties

- Estimation (Experimental)

Tensile properties of composites are determined in order to investigate the effect of surface treatment of coir fibres and incorporation of MAPP on the tensile strength of the composite system. Table 3 shows that in case of alkali treated coir fibre, the improvement in tensile strength and tensile modulus is observed to the tune of 58% and 48% respectively. This may be because of the roughness on fibre surface which gives mechanical interlocking to matrix after alkali treatment. Further improvement in tensile strength and modulus of about 20% and 15% respectively is observed by adding 5% MAPP into the composite system. This can be attributed to the improvement in compatibility between fibre and matrix.

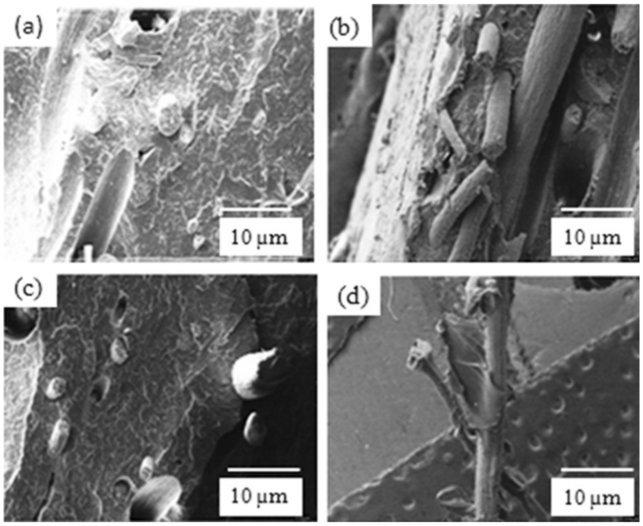

Fig. 4 — Tensile fracture images at magnification ×50 (a) PP/untreated coir, (b) PP/ alkali- treated coir (c) PP/ alkali-treated coir with MAPP and (d) pull-out fibre from PP/ alkali-treated coir with MAPP

Table 3 — Experimental and theoretically calculated values of tensile strength and elastic modulus of composites

Composite	Tensile strength, MPa		Elastic modulus, GPa	
	Experimental	Theoretical	Experimental	Theoretical
PP/untreated coir	11.6	27.6	0.62	1.54
PP/alkali treated coir	15.2	28.6	0.81	1.56
PP/alkali treated coir/MAPP	18.3	31.7	0.92	1.73

- Calculation (Theoretical)

The modified rule of mixtures for composites is used to predict the tensile strength and elastic modulus of short fibre composites by assuming a perfect interfacial bonding between matrix and fibre. The equation for calculating tensile strength and elastic modulus are given below[20, 21]:

$$\sigma_c = k_1 k_2 V_f \sigma_f + V_m \sigma_m \quad \ldots(4)$$

$$E_c = k_1 k_2 V_f E_f + V_m E_m \quad \ldots(5)$$

where σ_c, σ_m and σ_f are the tensile strength of the composite, polymer matrix and fibre respectively; k_1, a fibre reinforcement efficiency parameter, which is equal to 0.375 for randomly and uniformly distribution of fibres in the plane[21-23]; V_f and V_m, the volume fractions of fibre and matrix in composite respectively; k_2, the fibre length efficiency factor; and E_c, E_f and E_m, the elastic modulus values of composite, fibre and polymer matrix respectively. Fibre length efficiency factor is given as[20]

$$k_2 = L/(2L_c) \text{ for } L < L_c \quad \ldots(6)$$

$$k_2 = 1 - L_c/(2L) \text{ for } L \geq L_c \quad \ldots(7)$$

where L is the fibre length; and L_c, the critical fibre length.

As length L (15mm) > L_c, the tensile strength and elastic modulus of discontinuous random oriented composite (σ_c) can be estimated by the following equation[20, 21]:

$$\sigma_c = k_1[1 - L_c/(2L)] V_f \sigma_f + V_m \sigma_m \quad \ldots(8)$$

$$E_c = k_1[1 - L_c/(2L)] V_f E_f + V_m E_m \quad \ldots(9)$$

For PP/untreated coir composite, tensile strength and modulus are:

$\sigma_{c1} = 0.375\ [1- 10.7/(2\times15)]0.25\times137.6 + 0.75\times25.8 = 27.6$ Mpa

$E_{c1} = 0.375\ [1- 10.7/(2\times15)]0.25\times2.0 + 0.75\times1.9 = 1.54$ Gpa

For PP/alkali treated coir composite, tensile strength and modulus are:

$\sigma_{c2} = 0.375\ [1- 5.3/(2\times15)]0.25\times120.4 + 0.75\times25.8 = 28.6$ Mpa

$E_{c2} = 0.375\ [1- 5.3/(2\times15)]0.25\times1.8 + 0.75\times1.9 = 1.56$ GPa

For PP/alkali treated coir/MAPP treated composite, tensile strength and modulus are:

$\sigma_{c3} = 0.375\ [1- 3.3/(2\times15)]0.25\times120.4 + 0.75\times28.9 = 31.7$ MPa

$E_{c2} = 0.375\ [1- 3.3/(2\times15)]0.25\times1.8 + 0.75\times2.1 = 1.73$ GPa

Thus, this confirms the increase in tensile strength and elastic modulus because of coir fibre surface treatment and addition of coupling agent MAPP. It is found that the experimental values of composites (Table 3) are lower than the theoretically calculated values [Eqs (8) and (9)]. This is due to poor adhesion or poor bonding between matrix and fibres. Second reason of lower tensile properties may be due to void faction in composites. The equations used for theoretical calculations are based on assuming a perfect interfacial bonding between matrix and fibre. It is also observed that the tensile properties of the composites are found lower than matrix. This indicates that there is no reinforcement of fibres, may be due to poor adhesion or poor bonding between matrix and fibres and/or void fraction in composites. These composites can be said as biodegradable short fibre filler plastic composites and can be used in different areas.

(ii) Impact Strength

The behavior of composites against the suddenly applied force is investigated as per the standard procedure discussed above and the result is given in Table 4. The impact strength of composite basically depends on the nature of the fibre and its surface, polymer, and fibre matrix- interfacial bonding. In PP/untreated coir, low impact strength has been observed compared to PP/alkali treated coir because of reduced polymer and coir fibre interaction at an interface. After alkali treatment, there is the improvement in impact strength of composite due to increase in roughness on fibre surface which provides better mechanical interlocking between fibre and matrix. The impact strength of composite is increased by 21% as compared to PP/untreated coir due to the addition of MAPP. This may be due to coupling effect of MAPP in the composite which creates stronger interfacial bonding between fibre surface and matrix by chemical bonding with hydroxyl groups of fibre.

(iii) Flexural Strength

The flexural strength of composites has been studied to know the effect of unteated and surface treated coir fibres and MAPP on flexural strength of composite (Table 4). The composite prepared from alkali treated coir fibre shows improvement in flexural strength by about 29% as compared to untreated coir composite. This is due to alkali treatment of coir fibres which leads to form pits and roughness on fibre surface. A small improvement in flexural strength is observed with addition of MAPP. This can be attributed to better interfacial bonding between fibre and matrix in presence of coupling agent.

Table 4 — Different properties of composites (experimental)

Composite	Elongation at break, %	IFSS, MPa	Izod impact strength, J/m	Flexural strength, MPa
PP/untreated coir	4.8	2.0	513	19.6
PP/treated coir	4.1	3.5	585	23.9
PP/treated coir + MAPP	3.5	5.7	621	25.2

4 Conclusion

The surface treatment of coir fibre by using 5% concentration of NaOH has been done successfully. The melt spinning and drawing of MAPP is done followed by fabrication of untreated, alkali treated and alkali followed by MAPP treated composite using compression molding. The isothermal crystallization behavior of MAPP/PP shows finer spherulites and an increase in the number of effective nuclei than control PP and MAPP. From SEM image, it is found that very fewer fibres are pulled out than in alkali treated composite. This is because of strong interfacial adhesion/bonding between fibre and matrix. Interphase transcrystllinity of composites suggests that the addition of MAPP increases the fibre trans crystalline zone interface strength. The interfacial shear strength suggested that lower critical fibre lengths are required in PP/alkali treated coir, PP/alkali treated coir with MAPP than in PP/untreated coir for strengthening and stiffening of composites. There is an improvement in tensile strength and modulus of composite with MAPP by about 58% and 48% respectively when compared to composite with untreated coir. Improvement in Izod impact and flexural strength of composite with MAPP by about 21 % and 48 % respectively is observed as compared to that in composite with untreated coir. It is also observed that tensile properties of the composites are found lower than matrix. This indicates that there is no reinforcement of fibres may be due to poor adhesion or poor bonding between matrix and fibres and/or void fraction in composites. These composites can be termed as biodegradable short fibre reinforced composites and can be used in potential applications.

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