

Mechanical and Physical Performance of Cowdung-Based Polypropylene Biocomposites

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In this work, potential of cow dung (CD) as a reinforcing material was evaluated. CD was blended in different ratios up to 50 wt% with polypropylene (PP) using Brabender twin-screw compounder. The results show a steady decline in the biocomposite tensile and impact strength with increasing CD loading. In contrast, the storage modulus (E'), flexural modulus, and water absorption capacity of the biocomposites increased with increasing CD loading. Furthermore, it was revealed by SEM that the failure of the PP/CD biocomposites at higher filler loading was because of the weak interfacial bonding. Results established that the properties of PP/CD biocomposites are a function of CD loading. POLYM. COMPOS., 00:000-000, 2016. © 2016 Society of Plastics Engineers

INTRODUCTION

In the past few decades, various approaches have been attempted to use biomass as a natural filler for production of biodegradable composites. Effective use of biomass can open new avenues for the utilization of agricultural residues, at the same time it helps in developing the rural agri-

cultural based economy. These materials are used as inexpensive fillers in different thermoplastics such as polypropylene (PP) and polyvinyl chloride (PVC) and are often referred to as “plastic lumber.” The filler content in these products can range from 30% to 70% and are commonly used in outdoor applications, automotive panels, and furniture. The mechanical properties of plant fibers are much lower when compared to those of the most widely used competing reinforcing glass fibers. However, because of their low density, the specific properties (property-to-density ratio), strength, and stiffness of plant fibers are comparable to the values of glass fibers [1] and these products do not experience high stresses in service, their mechanical properties can be moderate to low. The market for such composites has grown strongly in the past few years and is expected to continue in a similar vein in the future [2]. Moreover, legislative pressures for greener technologies as well as customers’ demands for more environmentally friendly consumer goods are forcing materials suppliers and manufacturers to consider the environmental impact of their products at all stages of their life cycle, including materials selection, processing, recycling, and final disposal of the product. Renewable resources from agricultural or forestry products form a basis for new industrial products or alternative energy sources. Plant-based fibers are already used in a wide range of products. Plant fibers find applications as textiles and Geotextiles, twines and ropes, special pulps, insulating and padding

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TABLE 1. Approximate chemical composition of cow dung [5].

Composition	Dry wt (%)
Starch	5.2
Cellulose	31.4
Hemicellulose	15
Lignin	17
Fat	3.21
Crude Protein	18.12

materials, fleece, felts and nonwoven materials, and increasingly as reinforcement for polymers [3]. The important feature of biocomposite materials is that they can be designed and tailored to meet different requirements. Since biomass is cheap and biodegradable, the biodegradable composites from biofibers and biodegradable polymers will render a contribution in the 21st century because of serious environmental problem. The biomass filled composites reduces the use of petroleum based materials which eventually leads to a reduction in the greenhouse gas emission. Moreover, biodegradable polymers offer a possible solution to waste-disposal problems associated with traditional petroleum-derived plastics. For researchers, the real challenge lies in finding applications which would consume sufficiently large quantities of these materials to lead price reduction, allowing biodegradable polymers to compete economically in the market. In order to achieve the above mentioned goal, we have tried a relatively new kind of biofiller from cows to develop a new range of thermoplastic biocomposites [4].

Being a ruminant, cow has the advantage of digesting their food and preserve the cellulose. A ruminant digests its food in two steps, first by eating the raw material and regurgitating a semi-digested form known as cud, and then ruminating process takes place where the cud is being eaten. It has four-stomach with four chambers which are rumen, reticulum, omasum, and abomasum. In these chambers, fibers, especially cellulose and hemicellulose, are primarily broken down by bacteria and protozoa. Therefore, the CD has been used as the raw material, since it is believed to have a high percentage of cellulose which is an important structural material for the composite (Table 1). It is the amount of cellulose that served as the filler in enhancing the performance of the biocomposites.

In the present study CD has been investigated as a potential biofiller to develop the biocomposites. The resulting biocomposite was tested and characterized to evaluate the mechanical and physical properties. The possible cause of the biocomposite failure was also investigated using scanning electron microscope.

MATERIALS AND METHODS

Materials

Homopolymer polypropylene (PP) grade 600G (Melt index 11 g/10 min, density 900 kg/m³) was kindly supplied

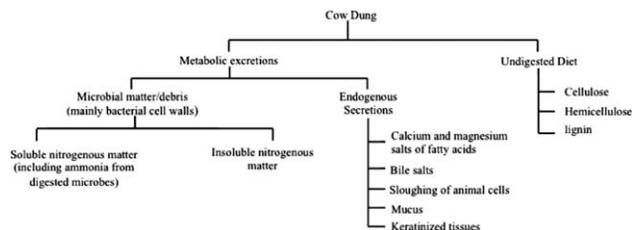


FIG. 1. Cow dung composition [6].

by Petronas Polymers Marketing and Trading Division Malaysia. No coupling agent was used in this study.

Cow Dung

The CD, which is the raw material for this project was obtained from Farm 16 of University Putra Malaysia, Serdang, Malaysia. Figure 1 shows the composition of the CD.

Formulation

The PP/CD biocomposites were prepared by blending the PP pellets with 10%, 20%, 30%, 40%, and 50% of CD (wt/wt). The PP without the CD was used as a control. The compositions of biocomposites are presented in Table 2.

Compounding Technique

The cow dung in this study was used without any treatment or modification. Prior to mixing, CD was dried for 12 h in a hot air oven at 100°C in order to remove the moisture. The fully dried cow dung was then ground using Fritsch sieve plate and shaker with a 0.25 mm sieve size to obtain the uniform size of the filler. The ground cow dung was dried again in a hot air oven (Toyoseiki Seisaku-Sho) with a temperature of 110°C until a constant weight was achieved. The blending of polypropylene and the CD was carried out in Brabender PL2000-6 twin-screw compounder at 180°C for 20 min at a roller speed of 50 RPM. The blended melt were then cut into small pieces for compression moulding.

Compression Moulding

Biocomposite sheets with 1, 2 and 3 mm thickness respectively, were compression moulded by hot pressing

TABLE 2. Composition of PP/CD biocomposite.

Biocomposite	PP (wt%)	CD (wt%)
0 (control)	100	0
10	90	10
20	80	20
30	70	30
40	60	40
50	50	50

the blended sample with a hydraulic press at 180°C. A pressure of **4 MPa** was applied for 5 minutes to preheat the sample for easy spreading between the mould plates. Further, the pressure was then increased to **15 MPa** for 3 minutes for complete pressing. This is followed by immediate cooling at 25°C for 3 min under pressure equipped with chiller facilities.

TESTING

Mechanical Testing

Three important mechanical properties, tensile strength (Ts), flexural modulus, and impact strength were tested. All test specimen dimensions were according to the respective ASTM standards and all tests were performed at room temperature.

Tensile Test. The sheet of 1 mm thickness was cut into dog-bone specimens for tensile strength testing. The tensile tests were conducted in accordance with ASTM 1882L using INSTRON (Model 4301) Universal Testing Machine with load cell of 1 kN, using a crosshead speed of 50 mm/min. The Test was performed until tensile failure occurred. Seven specimens were tested and at least five replicate specimens were presented as an average of tested specimens (the maximum and minimum data being discarded).

Flexural Modulus. The sheet of 2 mm thickness was cut into rectangular specimens for flexural modulus testing. The flexural modulus was determined using INSTRON (Model 4301) Universal Testing Machine in accordance with the ASTM D790-97 standard. The support span was 43 mm while the crosshead speed utilized was 1.3 mm/min.

Impact Test. All impact samples were cut into rectangular specimens and notched. The Izod impact tests were conducted according to ASTM D256 using CEAST (Model CE UM-636) Impact Pendulum Tester, with a 4 J hammer. Seven specimens were tested and at least five replicate specimens were presented as an average of tested specimens.

Melt Flow Index (MFI)

Melt flow rate measures the rate of extrusion of thermoplastics through an orifice at a prescribed temperature and load. Approximately, 7 gm sample was loaded into the barrel of the melt flow apparatus (Model Thermo Haake SWO MeltflickerST). The barrel was heated to a temperature of 230°C and a constant weight of 2.16 kg was applied to a plunger. A timed extrudate was collected and weighed. Melt flow rate values were calculated in g/10 min. Minimum of three samples were repeated for the accuracy of the results.

Density

The density of the biocomposites was determined using an Electronic Densimeter (Model MD-205) by the water displacement method according to ASTM 3800. Three rectangular bar shape samples (20 mm×12 mm×3 mm) were tested to determine the average value.

Water Absorption of Biocomposite

The water absorption test was carried out to observe the ability of the biocomposite in absorbing moisture. In this test, 3 mm thick samples were used. The specimens were dried in an oven at 60°C for 24 h and the weights of the specimens were immediately taken as its initial weight. The specimens were submerged in plates filled with distilled water according to their percentage, respectively. The plates were then covered using aluminium foil. The specimens were periodically taken out of the water, wiped with tissue paper to remove surface water, re-weighed and immediately put back into the plate. The results were taken for every 24 h as described in ASTM procedure D570-99 (ASTM 1999) for 17 days. The initial weight of the specimens was deducted by the weight of specimens taken daily in order to get the weight of the percentage water absorption. The averages of the three specimens for each sample were calculated.

Dynamic Mechanical Analysis

The dynamic mechanical analysis (DMA) was carried out using Q800 TA Instrument analysing machine in a single cantilever mode. The specimens were tested under the condition of static force 110 N, dynamic force 100 N, frequency 1 Hz with 17.5 mm span length was used. The scan was made from -25 to 120°C at 5°C/min rate under cryogenic environment. Specimen dimension was 12 mm width, 3 mm thickness, and 35 mm length. To establish the experimental reproducibility of DMA data, three identical samples were tested in the same mode and conditions and the result with reproducible glass transition temperature (T_g) was reported.

Interfacial Morphology Analysis

Fracture surface after tensile testing were observed with a scanning electron microscope (SEM) (Model Philips 515) under a voltage of 20 kV. Before scanning each sample was sputter coated with gold to improve its interface conductivity.

RESULTS AND DISCUSSION

Tensile Properties

Figure 2 depicts the Ts of CD-filled PP biocomposite. It is well-known fact that the filler plays an important role in determining the properties of the composite materials.

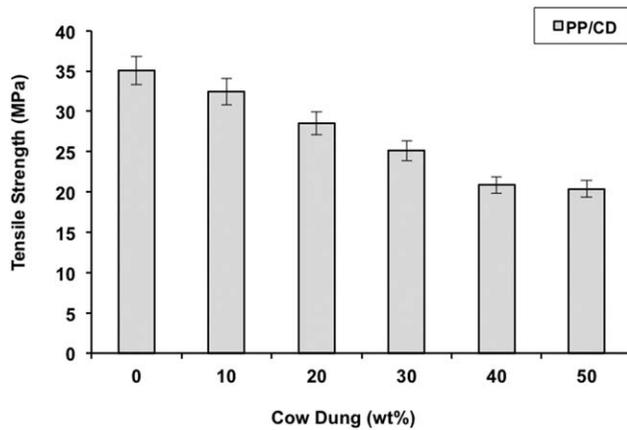


FIG. 2. Tensile strength of PP/CD biocomposite.

From the graph it is evident that, incorporation of the CD has decreased the T_s of the PP biocomposites. This decline in the T_s can be attributed to many factors. The most crucial factor that affects the mechanical properties of fiber-reinforced materials is the fiber matrix interfacial adhesion. The increase in the interfacial area with increasing CD loading, deteriorated the interfacial bonding between the hydrophilic CD and hydrophobic PP. Thus, interrupting the stress transfer in the PP matrix along the applied force. The quality of interfacial bonding is determined by several factors, such as the nature of fiber and polymer components, the fiber aspect ratio, the processing method and the treatment of the polymer of the fiber [7, 8].

Moreover, irregular shape fillers as in the case CD, are generally not effective in transferring the stress. They provide sharp corners which act as stress risers, leading to premature failure of the composites. Also, the strength of a material depends on the crystallinity of the material. Since, polypropylene is a semi-crystalline polymer, the addition of irregular shaped filler will result in the formation of defect in the crystalline structure hence, further reducing the strength of the biocomposite material. The addition of CD resulted in 7.5%, 22%, and 41% reduction in the strength at 10, 30, and 50 wt% loading respectively. Nevertheless, even with the steady decline in the T_s , the biocomposite strength remained within the acceptable limits. A similar decline in trend was observed for PP/EFB [9], PP/wood fiber [10, 11], and PP/coir fiber [12] composites. It is a generally accepted fact that the strength of a fiber-reinforced polymer matrix composite is mainly dependent on the fiber's strength. The composite will display higher strength if the matrix is ductile and the fiber has high strength/modulus ratio. A perfect example will be PP/flax fiber composites which show a superior reinforcing effect because of the higher fineness and smaller fiber diameter compared to other fibers such as hemp or kenaf. The strength of PP/flax is greater than PP/hemp or PP/kenaf composites [13].

Flexural Modulus

Modulus (stiffness) is another basic property of composites; the primary intention of filler incorporation is usually to increase the stiffness of the resultant material. Figure 3 shows the flexural modulus of the PP/CD biocomposite. As expected, the flexural modulus increases steadily with increasing filler content. An improvement of 3.3%, 10%, and 20% in flexural modulus was recorded at , 30, and 50 wt% CD loading, respectively. This is a common phenomenon, i.e. filler addition results in greater modulus following the Rule of Mixtures. Data from existing literature where the composites were fabricated using polypropylene with different natural fibers also show similar increasing trend in the modulus [1, 14–16]. There are many factors affecting the modulus of the composites. Those factors include filler content, filler properties and aspect ratio. The elastic moduli of the fibers, for instance, are expected to increase with increasing degree of molecular orientation. Well-oriented fibers such as flax will have a much higher Young's modulus than fibers with medium orientation [17]. Moreover, the fiber–matrix bonding also plays an important role in improving the modulus of the composite material. Composites with good fiber–matrix bonding displays a superior flexural modulus compared to the composites with poor fiber–matrix bonding.

Impact Strength

The effect of impact strength of the PP/CD for notched samples is depicted in Fig. 4. A decrease in the impact strength of the biocomposites was observed at 10 wt% CD loading. The formation of pores and localized defects in the biocomposites because of the addition of CD, created a high stress concentration regions which requires less energy to initiate the crack propagation. For the composite to be tough and to have a high impact strength, there should be some mechanism for dissipating the

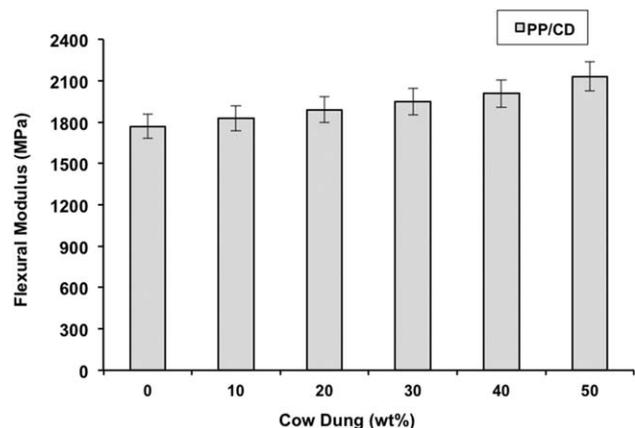


FIG. 3. Flexural modulus of PP biocomposites.

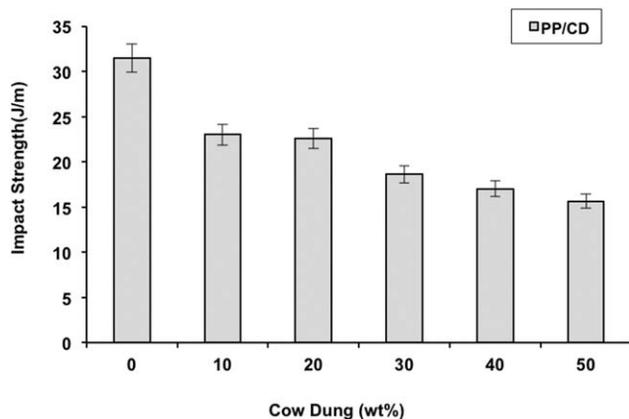


FIG. 4. Impact strength of PP biocomposites.

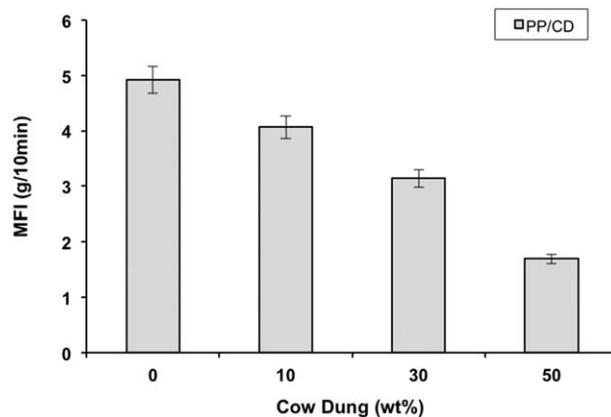


FIG. 5. MFI of PP/CD biocomposite.

absorbed energy throughout the volume of the material. If the energy is concentrated in a small volume, the material fails in a brittle manner, and the impact strength is low. Moreover, at lower CD loading, the filler volume is below a critical fraction. Therefore, the filler is not able to carry the extra load transferred to them, leading to brittle fracture of the composite.

A further increase in the CD loading gradually decreases the impact strength of the biocomposite. This is because, when a crack is generated because of an impact it propagates towards a poor interface region, causing the filler to pull out of the matrix and dissipate energy by mechanical friction. However, at the same time pulling out of the fillers prevents localization of stresses in one area along the filler. Besides, the high filler content may also reduce the crack propagation by forcing cracks to go around the filler or by bridging cracks [18]. This sort of behavior is not uncommon, as reported in many studies [19–21], the impact strength decreases as the amount of lignocellulosic filler is increased. In general, the stiffest composites exhibit the lowest impact properties as the high stress is transferred from the polymer matrix to the filler particles.

Melt Flow Index

MFI is inversely proportional to the viscosity of the polymer, higher the MFI lower the viscosity and vice versa. It provides a means of measuring flow of a melted material, or determines the extent of degradation of the plastic as a result of a moulding. Low MFI means high viscosity and high molecular weight, while a high MFI value means low viscosity and low molecular weight. Degraded materials would generally flow more as a result of reduced molecular weight, and could exhibit reduced physical properties. The MFI represents one point on the viscosity curve and it is widely used as an industrial indicator of the processability polymeric material and quality control purpose. The flow behavior of the PP biocomposites at different CD loadings is illus-

trated in Fig. 5. As the filler loading is increased the MFI starts to decrease dramatically. This is attributed to the presence of fillers in melts and their partial misalignment [22], which significantly affects the dynamics of viscoelasticity of the melts [23], hindering the mobility of molecular chains.

Density

Figure 6 presents the results obtained from the density measurement. It is clear that with increasing CD loading from 10 to 50 wt% the biocomposite density also increases from 3.6% to 22%, respectively. The highest density value of 1.1 g/cm³ was achieved for the biocomposite with 50 wt% CD loading. This increase was expected because of the higher density of the CD (0.98 g/cm³) relative to that of the PP (0.9 gm/cm³). Based on the law of mixtures, an increase in the weight fraction of the denser filler should result in higher composite density as seen in the experimental results. Moreover, the voids created in composite during the blending process tend to close up when the pressure is applied and thus, increasing the mass to volume ratio.

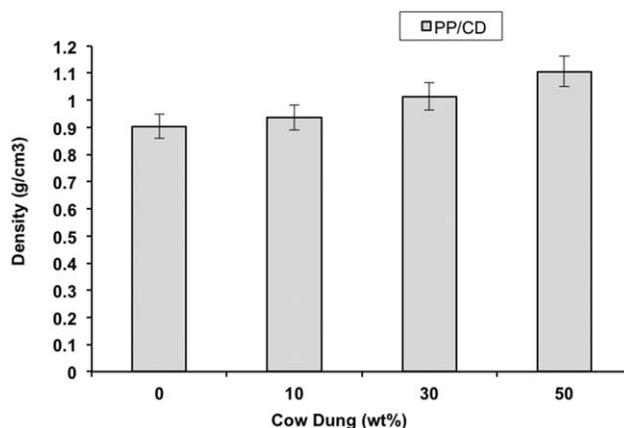


FIG. 6. Density of PP/CD biocomposite.

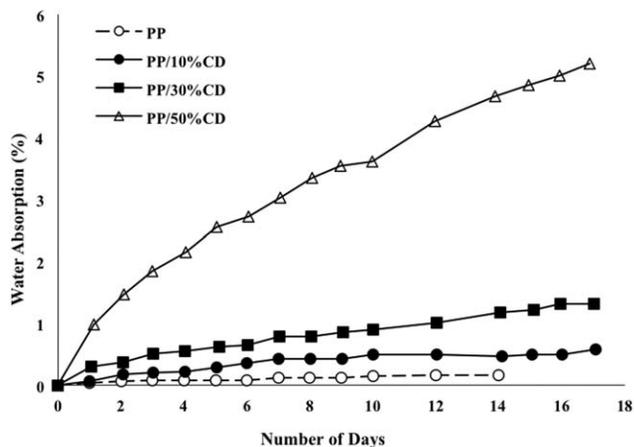


FIG. 7. Water absorption of PP/CD biocomposite at different filler loading.

Water Absorption

Figure 7 shows the plotted data obtained for the water absorption of the PP/CD biocomposite. It is observed that the pure PP barely absorb the water (0.1% on the last day) because of its hydrophobic nature. The amount of water absorbs by the cow dung in percentage varies from 0.48% to 6.14% based on the last day of the observation. The greater the amount of fillers, the higher will be the water absorption. This is because of hydrophilic nature of the CD by virtue of the presence of an abundant hydroxyl groups which are available for interaction with water molecules. In studying the treatments and properties of PP/wood flour composites, Ichazo et al. [24] observed similar phenomenon for the water absorption in the biocomposite. The water molecules can saturate the surface of the PP/CD biocomposites easily and also penetrate into the composites through voids which result in higher water uptake in a short exposure time [25]. Also, a larger surface area is expected at the lower particle size and consequently, a higher availability of OH groups coming from cellulose will enhance the rate of water absorption.

Dynamic Mechanical Analysis

The dynamic mechanical spectra of E' and $\tan \delta$ as a function of the temperature are represented in Fig. 8. Dynamic mechanical analysis has been widely used for investigating the viscoelastic behavior of composite materials. Damping factor ($\tan \delta$) measurements give practical information related to the glass transition (T_g) temperature and provide an indication of the material's ability to store and dissipate energy, while the storage modulus (E') determines the ability of a material to absorb or store energy; high storage modulus indicates the rigidity of the material. It is observed that E' for Pure PP decreases with increasing temperature. The reduction in E' with increasing temperature is related to the increase in viscosity and polymer chain mobility of the matrix at higher tempera-

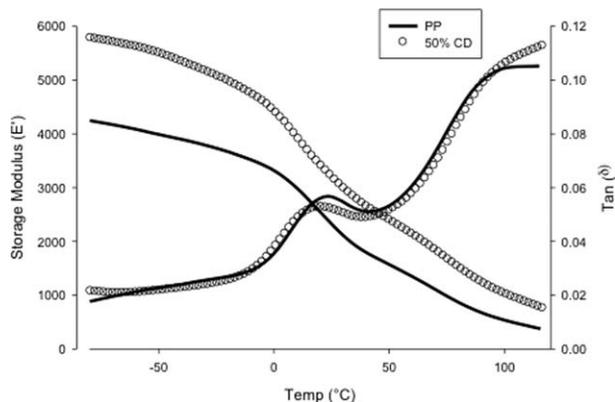


FIG. 8. Variation in storage modulus and damping factor of PP/CD biocomposite.

tures [26]. It is evident from the graph that in the entire temperature range (-80°C to 120°C) the storage modulus of the PP/CD biocomposite is higher than that of neat PP. This is because of the reinforcement imparted by the CD allows greater stress transfer at the interface from PP to the CD. Moreover, incorporation of rigid filler in a semi-rigid matrix tends to increase the stiffness of the biocomposites. However, as the temperature increases, the presences of CD restrict the flow of the polymer matrix and the restriction increases with increasing CD loading. Similar observations were recorded by Joseph et al. [27] on short sisal fiber-reinforced PP composites.

The influence of the CD into the PP matrix can be better understood by studying the relative normalized storage modulus (E^*) with respect to the temperature variation (Fig. 9). The relative normalized storage modulus is calculated according to the following equation:

$$E^* = (E'_c / E'_m) \quad (1)$$

where E'_c and E'_m are the storage modulus of the composite and the matrix, respectively, with temperatures

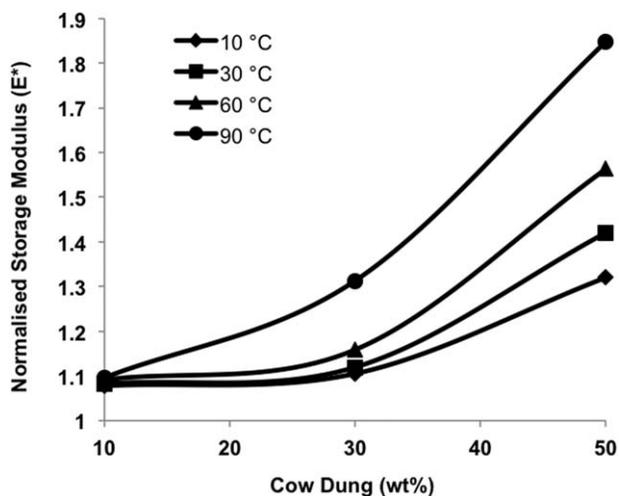


FIG. 9. Normalized storage modulus of PP/CD composites with CD loading.

TABLE 3. Variation of modulus retention with CD loading for PP/CD fiber composites.

Samples	C	Modulus retention (%)		
		E'_{30}/E'_{10}	E'_{60}/E'_{10}	E'_{90}/E'_{10}
PP	-	70.14	45.69	22.27
10	2.84	70.5	46.16	22.65
30	1.08	71.05	47.91	26.47
50	1.33	75.47	54.11	31.18

at different fiber loadings. It is clear from Fig. 9 that the storage modulus of the composites increases with CD loading. However, the relative change with CD loading is less pronounced at low temperatures.

Above T_g , the difference in the dynamic modulus becomes more pronounced because of the larger modulus ratio of the components when the polymer is in the rubbery state compared to the glassy state. Table 3 shows the variation of the modulus retention of the composites with the fiber loading. It is evident from the table that with an increase in the fiber loading, the modulus retention values increase. The effectiveness of fillers on the modulus of the composites can be represented by a coefficient C as

$$C = \frac{(E'_G/E'_R)_{\text{Composite}}}{(E'_G/E'_R)_{\text{PP}}} \quad (2)$$

where E'_G and E'_R are the storage modulus values in the glassy and rubbery region respectively. The higher the value of the constant C , the lower the effectiveness of the filler. The measured E' values at -50°C and 90°C (arbitrarily selected) were used as E'_G and E'_R respectively. The values of C obtained for different composition of biocomposites are listed in Table 3. The “ C ” value decreases as the CD loading increases, reaching a minimum at 30 wt% CD loading, and then increases for 50 wt% CD loading. This indicates that the effectiveness of the filler increases only upto 30 wt% CD loading above that difficulty in dispersing 50 wt% CD reduced the efficiency by the addition of the filler and thus the mechanical properties deteriorated.

The β -transition (glass–rubbery transition, T_g) in $\tan \delta$ spectra for pure PP was observed at 23°C . During this transition, a significant decrease of the E' is initiated, which drops even more in the melting range of PP. In contrast, the transition shifted to lower temperature (19°C) as the CD loading was increased to 50 wt%. The T_g , related to the glass–rubbery transition, since the area under the glass transition peak is proportional to the mobile amorphous phase, reduced area of the glass transition indicates a decrease in the mobile amorphous phase in the biocomposite [28]. Similar trends were observed by Khalid et al. [29] for PP/EFB and PP/cellulose composites.

An important factor to be noted is that, during blending, some of the surface material of the filler themselves is sheared off from the fibers present in the CD and blends in with the matrix. This sheared material could contain undigested materials such as lignin, hemicellulose, cellulose and other surface extracts that are inherently present in the fiber. This can be clearly seen as the dark brown tinge of the polymer matrix after blending. In other words, the matrix material is a blend of PP and several constituents that are contributed by the CD. Therefore, the shift of the β -peak to lower temperatures with the addition of filler to the matrix is probably because of the presence of sheared surface material from the CD [30].

Additionally, the α -relaxation peak shifts to higher temperatures as the filler loading increases; this shift (α -transition) can be seen between 106°C and 116°C for pure PP and biocomposite. The restricted molecular mobility because of the interactions between PP and the CD surface is attributed to the shift to higher melting temperature (T_m). This α -transition in semicrystalline polymers is related to the relaxation of restricted PP amorphous chain in the crystalline phase (defects) also known as rigid amorphous molecules [31]. The presence of crystals is necessary for this transition to occur [32, 33]. The general increase in storage and loss modulus and decrease in damping values because of the addition of the CD are in agreement with the observations of other researchers [34]. Damping also was decreased by the addition of the fillers. Kuruvilla et al. [35] also reported that by the incorporation of short sisal fibers into low-density polyethylene (LDPE), the storage and loss modulus increased, whereas the $\tan \delta$ decreased.

Interfacial Morphology Analysis

Figures 10 and 11 illustrate the SEM micrograph of the tensile and impact fracture surfaces in the PP-CD

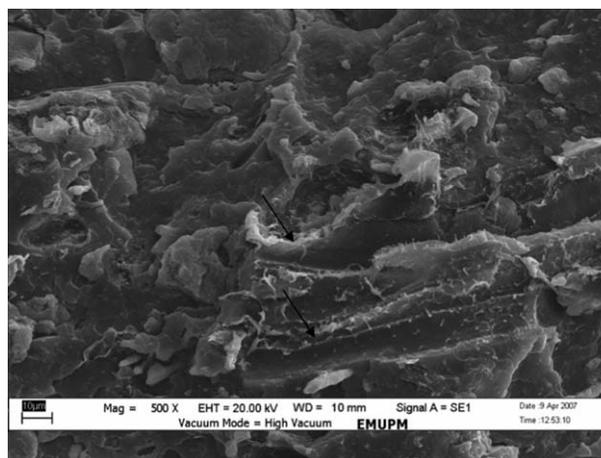


FIG. 10. SEM micrograph of tensile fracture PP biocomposite with 30% CD.

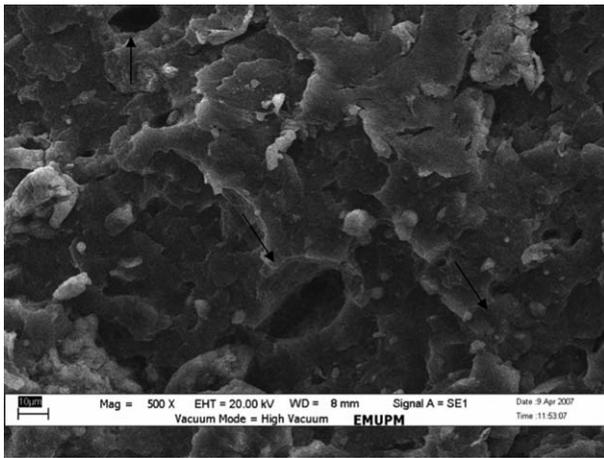


FIG. 11. SEM micrograph of impact fracture PP biocomposite with 30% CD.

biocomposites. A number of cavities on fracture surface are clearly visible, indicating the poor interfacial adhesion. When the adhesion between the fiber and matrix is poor, the biocomposite shows a tendency for pull-out leaving a smooth surface on the matrix (indicated by arrows). The holes also look smooth showing a complete separation of the CD and the polymer matrix. The failure occurs at the weakest filler–matrix interface. The micrograph of 30 wt% PP-CD biocomposites show some deep holes left after the fibers are pulled out of the matrix. The surfaces of the CD were completely devoid of matrix material. This is a clear indication of filler matrix interfacial failure followed by an extensive filler pullout from the matrix. Furthermore, the matrix also shows considerable tearing. This shows the incompatibility of untreated CD with the PP matrix. Moreover, the holes proximity indicates that the CD could not provide an efficient stress transfer from the matrix.

CONCLUSIONS

The study has shown the ability to successfully fabricate PP/CD biocomposites. A CD content of 30% by weight has proven to provide adequate reinforcement to maintain the strength of the biocomposite for Automotive and decking applications. The upward trend exhibited in flexural modulus of PP/CD biocomposite and moderated water uptake at 30 wt% indicates that the CD has the potential to make an attractive alternative to conventional fillers in preparation of biocomposites. However, the gradual drop in mechanical properties with CD loading is also observed. Results revealed that poor interfacial bonding and CD agglomeration are the main factors responsible for the observed trend. Therefore, further studies will be made in order to improve the CD dispersion and the quality of interfacial bonding between the CD and PP blend matrix.

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