

Mechanical Properties of HDPE/Bark Flour Composites

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ABSTRACT: Tensile and impact properties of Neem bark flour (BF) containing high density polyethylene (HDPE) composites were studied at 0–0.26 volume fraction of filler. Tensile modulus and strength and breaking elongation decreased with increase in BF concentration. The decrease in tensile modulus and strength was attributed to the decrease in crystallinity of the polymer compared to the imposed mechanical restraint by the BF. Analysis of tensile strength data indicated formation of stress concentration in the interphase. Because of this stress concentration and the mechanical restraint, the elongation-at-break and Izod impact strength decreased. Use of a coupling

agent, HDPE-g-MAH, brings about enhanced phase adhesion, increasing the tensile modulus and strength. Enhanced adhesion marginally lowers composite ductility at higher filler contents and aids stress transfer increasing the Izod impact strength inappreciably. Scanning electron microscopic studies indicated better dispersion of BF particles and enhanced interphase adhesion in presence of the coupling agent. © 2007 Wiley Periodicals, Inc. *J Appl Polym Sci* 105: 2598–2604, 2007

Key words: high density polyethylene; bark flour; composites; coupling agent; crystallinity

INTRODUCTION

High density polyethylene (HDPE) is a useful olefin polymer with good properties such as low density, very good chemical resistance, excellent electrical insulation, easy moldability, and the like.¹ The properties of HDPE have been modified by blending other polymers, such as butyl rubber,¹ ethylene propylene rubber,¹ polycarbonate,² etc. Use of particulate fillers, such as carbon black, calcium carbonate, talc, etc., for HDPE modification is well documented.^{3–5}

Lignocellulosic fillers such as wood flour, jute, and sisal fibers are a class of inexpensive, nonabrasive, nonhazardous organic materials, which are finding increasing use as cheapening fillers in polymers.^{6,7} In presence of these fillers, shear viscosity of the polymer increased,^{8,9} however extrudate swell decreased.¹⁰ A closely related material to wood flour is bark flour (BF), which can also be a potential material as filler in plastics. The wood bark contains lignin, hemicellulose, and other extraneous materials e.g., phenols, lignans, fatty acids, resins, which contain hydroxyl, carboxylic, ether, and phenolic functional moieties.^{10–14}

In the present study, mechanical properties of HDPE/BF composites are reported. Tensile proper-

ties such as tensile modulus, strength, and breaking elongation were analyzed following predictive models. Izod impact strength was evaluated on the basis of composite composition. Scanning electron microscopy has been used to examine the filler dispersion. Effect of a coupling agent, maleic anhydride grafted onto HDPE (HDPE-g-MAH), on the above properties has also been studied.

EXPERIMENTAL

Materials

HDPE, obtained from Gas Authority of India Ltd. (GAIL), was of injection molding grade, G-Lex I58A180 (density 0.95 g/cm³, MFI 18 g/10 min at 230°C, load 2160 g).¹⁵ Neem bark was peeled off from the trunk of a tree that is ~ 100 years old in Sikar, Rajasthan, India. The bark was washed in water, dried and ground in flour mill.¹⁶ The fine powder obtained was sieved through cotton cloth. Sieve analysis was carried out mechanically by vibratory sieve analyzer using mesh size 300–63 μm. The filler used for composite preparation was below mesh size 63 μm. Further analysis for particle size was carried out on Brookhaven Instruments Corp., 90 Plus particle Sizing Software Version 3.42. Average particle diameter was 163 nm. The coupling agent maleic anhydride-grafted high density polyethylene (HDPE-g-MAH) OPTIM TP-506/E (density 0.954 g/mL, MFI 1.24 g/10 min, maleic anhydride content (%) 0.99, acid number 11) was obtained from

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TABLE I
Values of Crystallinity (DSC) (%) of HDPE/BF and HDPE/BF/HDPE-g-MAH Composites

Φ_f	Crystallinity (DSC) (%)	
	(HDPE/BF)	(HDPE/BF/HDPE-g-MAH)
0.00	65.0	65.0
0.03	64.2	61.7
0.07	64.1	59.6
0.12	61.4	58.7
0.20	60.1	56.0
0.26	56.0	56.0

Pluss Polymers Pvt. Ltd. India.¹⁷ The coupling agent content was 5 wt % on the basis of the filler.

Compounding and molding

Vacuum-dried BF was dry-blended with HDPE and then melt-compounded in a corotating Twin Screw Extruder, model JSW J75E IV- P ($L/D = 36$; diameter $D = 30$ mm) at 174 screw rpm. The temperature from the feed zone to the die zone was 413–453 K.^{18,19} The bark flour was varied from 0 to 0.26 volume fraction, Φ_f (0–50 phr). In the HDPE/BF/HDPE-g-MAH systems, the coupling agent used was 5%, based on the weight of the BF. The extruded strands were quenched cooled by passing through water and pelletized. The pellets were air-dried for 3 h at 333 K followed by vacuum-drying at 338 K for 4 h. These granules were injection-molded into samples for tensile and Izod impact tests, on an L&T-Demag injection molding machine (model PFY 40-LNC 4P). The temperatures from the feed zone to the die zone were 423–453 K whereas the mold temperature was 303 K. The neat HDPE was also passed through the same extrusion and injection molding conditions to ensure identical processing and thermal history.

Tests and measurements

Tensile properties were measured using dumbbell-shaped samples on a Zwick Universal Tester, model Z010, at a crosshead speed of 100 mm/min and crosshead separation of 60 mm, according to the ASTM D638 test procedure.²⁰ The notched Izod impact measurements were performed on a falling hammer type Charpy Impact tester, Atsfaar model Impacts-15, following the ASTM D256 test method.²⁰ At least six samples were tested for each composite composition and the average value was reported. All the tests were performed at ambient temperature of 303 ± 2 K.

The degree of crystallinity of composite was calculated using the ratio of experimentally measured enthalpy by differential scanning calorimetry method and the enthalpy for 100% crystalline polyethylene, $\Delta H = 277$ J/g.²¹

Scanning electron microscopy (SEM) for morphological studies were performed on cryogenically fractured surfaces of the bar samples on a Stereoscan 360 to examine the dispersion of the discrete phase in the HDPE matrix. The samples were sputter-coated with silver prior to scanning.

RESULTS AND DISCUSSION

Crystallinity of HDPE

Incorporation of BF decreased the crystallinity (determined by DSC) of the continuous phase HDPE, the crystallinity decreasing with increasing volume fraction (Φ_f) of the filler (Table I). The normalized crystallinity data, i.e., ratio of the crystallinity of HDPE in the composite (C_c) to that of unfilled HDPE (C_p) followed a linear variation with the Φ_f (Fig. 1), with correlation coefficient $R^2 = 0.81$ and 0.90 for HDPE/BF and HDPE/BF/HDPE-g-MAH systems, respectively. The crystallinity of the polymer plays an important bearing on the mechanical properties of the system as will be presented in the subsequent analysis of the results.

Tensile properties

The tensile properties such as tensile modulus, breaking strength, and strain-at-break of the HDPE/BF composites were evaluated from the stress-strain curves (not shown) and are presented as the ratio of the property of the composite (subscript c) to that of the matrix polymer (subscript p) as a function of the volume fraction, Φ_f , of the BF in the following sections.

Tensile modulus

The normalized relative tensile moduli, i.e., the ratio of the normalized moduli of the composites to those of the modulus of the matrix, $(E_c/C_c)/(E_p/C_p)$, are

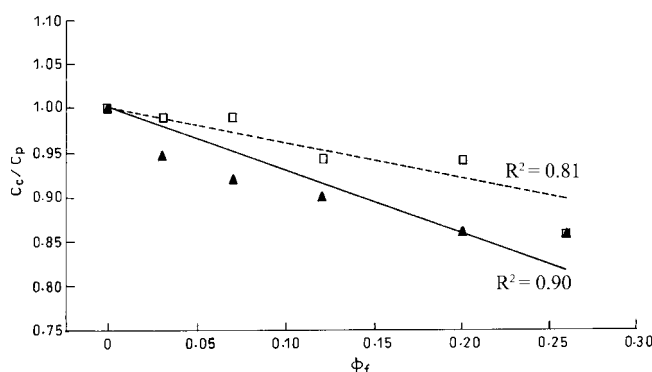


Figure 1 Normalized relative crystallinity, C_c/C_p , versus Φ_f in (\square) HDPE/BF and (\blacktriangle) HDPE/BF/HDPE-g-MAH composites.

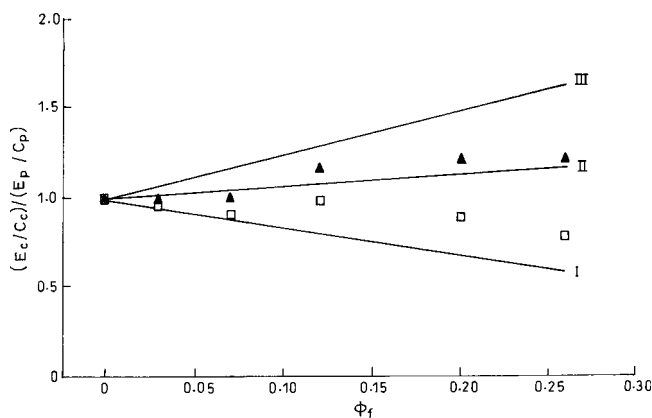


Figure 2 Normalized relative tensile modulus, $(E_c/C_c)/(E_p/C_p)$, versus Φ_f in (□) HDPE/BF and (▲) HDPE/BF/HDPE-g-MAH systems.

presented in Figure 2 as a function of Φ_f . The moduli of HDPE/BF systems were in between 0.97 and 0.78 depending on the value of Φ_f . The data were compared with a theoretical prediction known as “foam model” proposed by Cohen and Ishai²² [eq. (1)]:

$$(E_c/C_c)/(E_p/C_p) = 1 - \Phi_f^{2/3} \quad (1)$$

In the foam model, the inclusion is assumed to be a noninteracting phase equivalent to a void or pore when modulus ratio of the inclusion and the matrix becomes zero. The data showed agreement with the model up to $\Phi_f = 0.07$, however with further increase on Φ_f the data exhibited positive deviation (Fig. 2, Curve I). In these composites, the mechanical properties will depend on interphase interactions and their effect on the crystallinity of the matrix. Although chemical interaction of the BF containing hydroxyl and carboxylic polar groups¹⁰ with the hydrocarbon polymer HDPE may not be envisaged, physical type of interaction may, however, be possible. This physical interaction may arise out of differential thermal shrinkage²³ of the phases. In the molten state, the polymer expands more than the BF so that during the cooling cycle the polymer's shrinkage is higher. In the process, the polymer is anchored around the filler particles. During the tensile mode of load application, an extent of force will be expended to break these anchor points, which would enhance the modulus. The BF thus imposes mechanical restraints²⁴ that restrict the deformability of the polymer. The extent of this mechanical restraint is governed by particulate spacing and the properties of the matrix and the filler.²⁴ At the same time, this mechanical restraint impairs the crystallinity of HDPE (Table I). HDPE is a hydrocarbon polymer with very low cohesive forces, its mechanical properties accrue from its crystallinity. The composite properties will thus be a resultant of these two opposing effects.

In HDPE/BF composites, since the effect of variation of crystallinity by the BF has been eliminated by normalizing the data, the experimental results are direct indications of phase interactions, if any. Up to $\Phi_f = 0.07$, the data agreed with the foam model, which may be due to the preponderance of the matrix softening compared to the imposed mechanical restraint. However, with further increase in BF content, the extent of the latter effect takes dominance, which enhanced the data above the model.

Upon addition of the coupling agent, HDPE-g-MAH, to the HDPE/BF composites, the normalized relative moduli values increase with Φ_f (Fig. 2). The data were compared with predictive theories for two-phase systems, which account for the shape, packing fraction, and interphase adhesion between the polymer and the discrete phase (Fig. 2). Curve II demonstrates Einstein's equation without adhesion^{23,25,26} [eq. (2)], while Curve III represents Einstein's equation with adhesion^{23,25,26} [eq. (3)]:

$$(E_c/C_c)/(E_p/C_p) = 1 + \Phi_f \quad (2)$$

$$(E_c/C_c)/(E_p/C_p) = 1 + 2.5\Phi_f \quad (3)$$

The data showed good agreement with Einstein's model without adhesion. However, since the data are normalized eliminating the effect of variation of crystallinity of HDPE by the BF and the values are higher than unity, an extent of interphase adhesion may be indicative. This adhesion may be due to chemical reaction between polar hydroxyl and acidic groups in the BF and the anhydride and carboxylic groups of the HDPE-g-MAH. Similar reactions in HDPE/lignocellulosic fiber,²⁷ maleated wood fiber/HDPE,²⁸ lignocellulose/polyolefin,²⁹ and HDPE/maleated HDPE/wood flour³⁰ systems were reported where a part of the anhydride groups are hydrolyzed to carboxylic groups. However, the polar interactions in the HDPE/BF composites appear to be of limited extent in view of the very low quantity of the coupling agent: 5% on the basis of the filler. Nevertheless, the polymer matrix is stiffened by the BF particles through chemical reactions in part along with particulate interlocking by the polymer through differential thermal shrinkage,²³ as stated earlier. Increase in relative modulus was reported in wood flour-filled HDPE composites.⁷

Elongation-at-break

Normalized elongation-at-break, $(\epsilon_c/C_c)/(\epsilon_p/C_p)$, of HDPE/BF composites decreases with increase in Φ_f (Fig. 3). The elongation decreases gradually through the studied range of Φ_f . At the highest filler concentration, $\Phi_f = 0.26$, the elongation decreases to ~ 0.1 times that of the unfilled polymer. Upon use of the

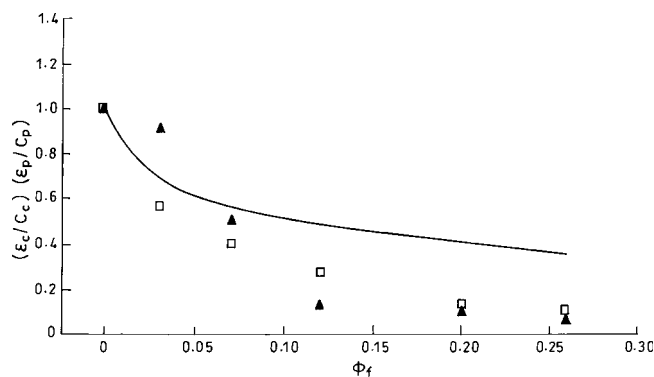


Figure 3 Normalized relative elongation-at-break, $(\epsilon_c/C_c)/(\epsilon_p/C_p)$ against Φ_f in (□) HDPE/BF and (▲) HDPE/BF/HDPE-g-MAH systems.

coupling agent, HDPE-g-MAH, the elongation maintained the decreasing trend, however the values were higher up to $\Phi_f = 0.07$, while beyond this Φ_f the data were inappreciably lower than those in the HDPE/BF composites. The elongation data were compared with Nielsen's model²³ with perfect adhesion [eq. (4)]:

$$(\epsilon_c/C_c)/(\epsilon_p/C_p) = 1 - \Phi_f^{1/3} \quad (4)$$

where ϵ_c denotes the breaking elongation of the composites and ϵ_p is the value of the polymer. The model agrees well up to $\Phi_f = 0.07$ with the data for HDPE/BF composites, the data beyond this Φ_f lie lower than the model, which may be due to vacuole formation around the BF component in the stretch direction due to nonadhesion between the members. The decrease in the elongation-at-break of HDPE in the presence of BF may be attributed to the mechanical restraint imposed by the filler on the mobility/deformability of HDPE. In the presence of the coupling agent HDPE-g-MAH, the elongation decrease was lower up to $\Phi_f > 0.07$ and marginally higher beyond $\Phi_f = 0.07$, which indicates a higher extent of mechanical restraint created through polar interaction between the BF and the coupling agent similar to other works.^{31,32}

Tensile strength

The normalized tensile strength values, $(\sigma_c/C_c)/(\sigma_p/C_p)$, of the composites were analyzed for evaluating the composite structure at large deformations using theoretical models.^{33–35} Equation (5) describes Nicolais–Narkis model, while eq. (6) represents the porosity model:

$$(\sigma_c/C_c)/(\sigma_p/C_p) = 1 - K\Phi_f^{1/3} \quad (5)$$

$$(\sigma_c/C_c)/(\sigma_p/C_p) = \exp(-\alpha\Phi_f) \quad (6)$$

These theories assume no-adhesion type structure and consider either area-fraction or volume-fraction of the discontinuous phase to determine the phase structure.^{36,37} The weight age factor K in eq. (5) denotes interphase adhesion³⁵; the lower the value, the better the adhesion. The value of $K = 1.21$ for spherical fillers with poor adhesion.³⁶ In the porosity model [eq. (6)], the discrete phase is considered equivalent to pores/voids in metals/ceramics³⁸ and polymer blends³⁶/composites.³⁹ Because of no adhesion at the interphase, the pores do not influence the composite mechanical properties. The parameter α describes stress concentration: the higher the value, the higher the stress concentration.

Table II exhibits the values of the interphase interaction parameter K and α [eqs. (5) and (6)], obtained by comparison of the experimental normalized tensile strength data of HDPE/BF composites and the theories. Variations of the data against Φ_f are shown in Figure 4. The data agreed well with the Nicolais–Narkis model [eq. (5)] with $K = 0.39$, which implies a good extent of interphase adhesion. The value of the adhesion constant is slightly higher than talc-filled i-PP/CSM rubber composites³⁴ but is quite lower than HDPE/WF,⁷ and i-PP/CaCO₃ system.²⁶ While the BF particles with polar groups may not chemically interact with the hydrocarbon polymer HDPE, this interaction appears to be due to the differential thermal shrinkage²³ of the two phases facilitated due to very fine particle size of the BF, which disperses well in the polymer matrix, as stated earlier. According to the porosity model [eq. (6)], occurrence of an extent of stress concentration was indicated with $\alpha = 0.84$, which is quite higher than the i-PP/CSM rubber/Talc system,³⁴ where $\alpha = 0.56$ but significantly lower than i-PP/Talc⁴⁰ and i-PP/CaCO₃ composites,²⁶ where the α values are 6.18 and 3.29, respectively.

The variation of the normalized relative tensile strength of HDPE/BF composites in the presence of HDPE-g-MAH is also presented in Figure 4 as functions of Φ_f . The tensile strength shows a marginal increase with Φ_f , indicating a reinforcing effect by

TABLE II
Values of Adhesion Parameter, K [eq. (5)], Stress Concentration Constant, α [eq. (6)], in HDPE/BF composites and reinforcement factor, B_a [eq. (7)], in HDPE/BF/HDPE-g-MAH composites

Φ_f	K	A	B_a
0.00	–	–	–
0.03	0.25	0.80	3.95
0.07	0.37	0.93	4.32
0.12	0.32	0.68	4.39
0.20	0.45	0.84	4.08
0.26	0.55	0.97	3.60
Mean Value	0.39	0.84	4.07

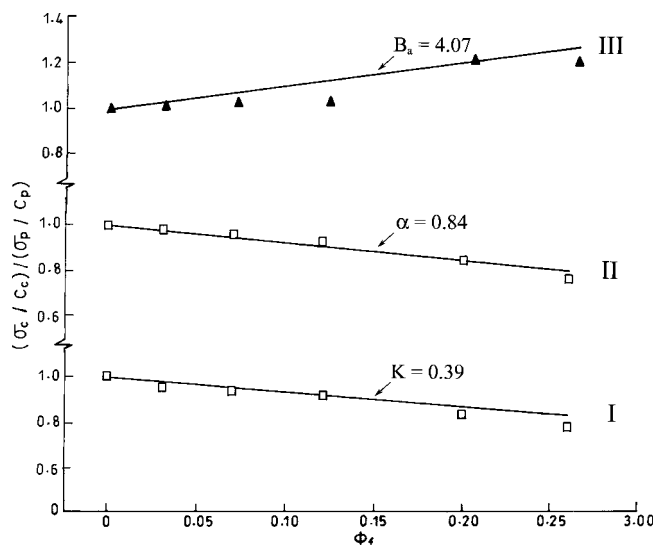


Figure 4 Plots of (I) Nicolais–Narkis model [eq. (5)], (II) porosity model [eq. (6)], with K and α values indicated in (□) HDPE/BF composites, and (III) Bela–Pukanszky model [eq. (7)], with B_a value indicated in (▲) HDPE/BF/HDPE-*g*-MAH composites, versus Φ_f .

the filler. The tensile strength data were compared with Bela–Pukanszky model^{41,42} [eq. (7)]:

$$(\sigma_c/C_c)/(\sigma_p/C_p) = (1 - \Phi_f/1 + 2.5\Phi_f) \exp(B_a\Phi_f) \quad (7)$$

where the reinforcement is denoted by B_a . From the normalized tensile strength values, B_a values were calculated from eq. (7) and presented in Table II. It may be seen that the reinforcement factor B_a value increases with Φ_f up to $\Phi_f = 0.12$, indicating a degree of reinforcement of the polymer, while at $\Phi_f > 0.2$ the value decreases to small extent. The increase in tensile strength is however quite low, which is due to the limited extent of chemical interaction between the polar groups of BF and that of HDPE-*g*-MAH, as was also observed in the modulus data. It may be pointed out that the coupling agent HDPE-*g*-MAH contains both polar and nonpolar moieties. While the nonpolar HDPE chain is completely miscible with the matrix HDPE, the anhydride group can be bonded chemically with hydroxyl groups of BF as was evidenced by FTIR and XDS studies in other works.^{19,27,28}

Impact strength

Variation of the normalized relative Izod impact strength values, $(I_c/C_c)/(I_p/C_p)$, of HDPE/BF composites are shown in Figure 5 as functions of Φ_f . The impact strength decreases significantly with increase in Φ_f ; at the highest Φ_f studied, the strength decreased to ~ 0.1 times that of the polymer. The decrease in impact strength may be due to the stiff-

ening of the polymer by the filler as was also observed in the modulus data. However, the stress concentrations created around the particles could lead to cracking and eventual failure of the structure, since the interphase was not strong enough. Formation of stress concentration was shown in the tensile strength analysis of the composites similar to other systems.^{26,34} In the presence of HDPE-*g*-MAH, the impact strength also decreased with increase in Φ_f , however the data were marginally higher than those in the absence of HDPE-*g*-MAH. This may be due to a marginally stronger interphase formed through chemical interaction between the BF and the coupling agent as was also observed in the tensile strength analysis.

Fracture surface morphology

The scanning electron microscopic (SEM) studies of HDPE, BF, HDPE/BF, and the HDPE/BF/HDPE-*g*-MAH composites are shown in Figure 6(a–l). BF particles were of different sizes and shapes with sharp faces and corners [Fig. 6(a)]. The matrix HDPE showed a reasonably smooth fracture surface with some extent of ribs and heckles with an extent of whitening around the surface of the ribs [Fig. 6(b)]. This is indicative of a reasonably tough plastic whose fracture mechanism borders brittle type of polymers. In the HDPE/BF systems, the filler appears to be well dispersed up to $\Phi_f = 0.07$ [Fig. 6(c,d)]. However, the BF particles are discernible easily almost without any adherent polymer residues beyond $\Phi_f > 0.07$ [Fig. 6(e–g)]. In these composites, the fracture surfaces also become very rough and uneven indicating no adhesion between the phases. In the presence of HDPE-*g*-MAH, there is distinct improvement in the dispersion of BF in HDPE. Up to $\Phi_f = 0.07$ [Fig. 6(h,i)], the fracture surface become quite smooth due to adherence of polymer residues with BF. Beyond $\Phi_f = 0.07$ [Fig. 6(i–l)], although

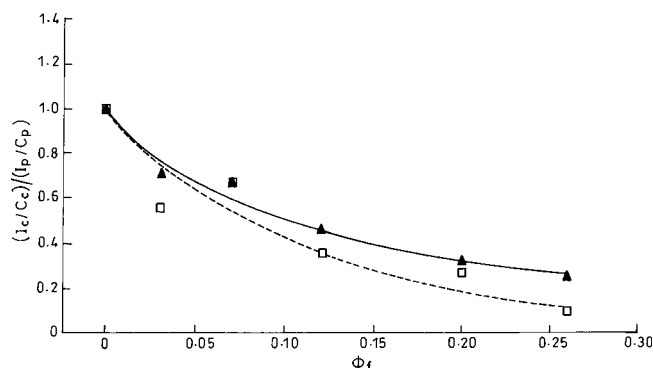


Figure 5 Normalized relative impact strength, $(I_c/C_c)/(I_p/C_p)$, versus Φ_f in (□) HDPE/BF and (▲) HDPE/BF/HDPE-*g*-MAH systems.

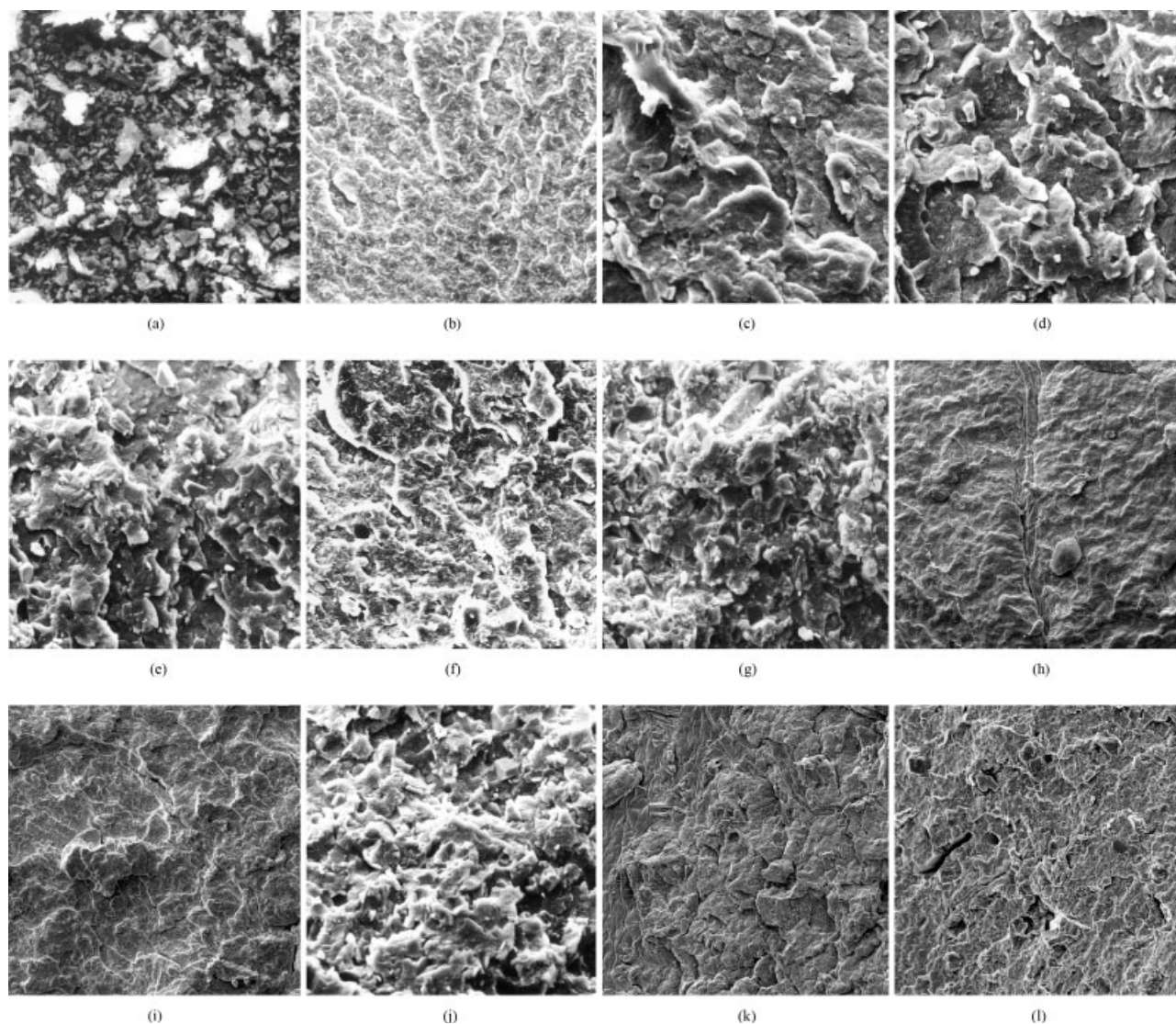


Figure 6 SEM micrographs of (a) bark flour, (b) HDPE and HDPE/BF composites with varying Φ_f values: (c) 0.03; (d) 0.07; (e) 0.12; (f) 0.20; (g) 0.26. The SEM micrographs of HDPE/BF/HDPE-g-MAH composites at corresponding Φ_f values are presented in (h), (i), (j), (k), and (l), respectively.

some bare BF particles are noticed, the fracture surface on the average is smoother due to an extent of enhanced interphase adhesion compared to the previous composites. Thus, the SEM results provide a supporting evidence to enhanced mechanical properties, i.e., increased tensile modulus, tensile strength, and Izod impact strength in the HDPE/BF/HDPE-g-MAH systems, which arise out of an extent of increased interphase adhesion.

CONCLUSIONS

Incorporation of neem BF modifies the mechanical properties of HDPE. Tensile modulus and strength as well as elongation-at-break decreased with increasing BF concentrations. The filler enhanced mechanical restraints on the molecular mobility/de-

formation of HDPE and in the process decreased its crystallinity also. As a resultant of these two opposing effects, the modulus and tensile strength decreased. Stress concentration was created at the interphase. Because of the mechanical restraints and stress concentrations, the ductility of the composites decreased leading to decrease in elongation-at-break and Izod impact strength of the composites.

In the presence of the coupling agent, HDPE-g-MAH, an extent of chemical type of phase interaction was brought about, which alongwith the mechanical restraint enhanced the tensile modulus and strength with BF contents. The elongation-at-break continued to decrease with increase in BF concentrations, in particular at $\Phi_f > 0.07$ the elongation was lower than the previous system due to this enhanced phase adhesion. The Izod impact strength values, although

decreased with increasing BF concentrations, were marginally higher than the HDPE/BF composites due to this increased interphase interaction.

SEM studies indicated that BF particles are nonadherent with HDPE, particle boundaries are discernible, in particular, beyond $\Phi_f > 0.07$. In presence of the coupling agent, the particle boundaries are not easily noticeable, as well as the fracture surfaces become smoother indicating enhanced interphase adhesion.

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