



Effect of coupling agent and nanoclay on properties of HDPE, LDPE, PP, PVC blend and *Phargamites karka* nanocomposite

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ARTICLE INFO

Article history:

Received 11 February 2010

Received in revised form 12 June 2010

Accepted 11 July 2010

Available online 16 July 2010

Keywords:

A. Nano composites

A. Wood

A. Polymers

B. Mechanical properties

D. Scanning electron microscopy

ABSTRACT

High density polyethylene (HDPE), low density polyethylene (LDPE), polypropylene (PP) and poly(vinyl chloride) (PVC) were solution blended by using a mixture of xylene and tetrahydrofuran as solvent and polyethylene-co-glycidyl methacrylate (PE-co-GMA) as compatibilizer. The minimum ratio of solvents to obtain a homogenous solution was optimised. Wood polymer composites (WPC) were prepared by using solution blended polymer, wood flour and nanoclay. X-ray diffraction studies of WPC treated with 1 and 3 phr nanoclay showed higher exfoliation compared to WPC treated with 5 phr nanoclay. TEM study also supported the above findings. FTIR studies indicated an interaction between wood, PE-co-GMA and clay. SEM study indicated an increase in miscibility among polymers due to addition of PE-co-GMA as compatibilizer. Thermal stability improved on addition of clay to the WPC. WPC treated with 3 phr clay showed highest mechanical properties. Hardness and water absorption were improved significantly with the addition of nanoclay to wood/polymer composite.

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1. Introduction

Nal (*Phargamites karka*), a type of nonconventional plant is widely available not only in the forests of Assam but also on both the banks of The Brahmaputra river. These plants are not considered for structural applications due to their poor mechanical, dimensional and other properties. They are mostly used for making temporary shades and domestic fuels. These plants can be made value added products by producing composites with different polymers.

Varieties of waste plastic materials in the form of carry bags, packaging film, container, etc. cause environmental pollution. Recycling is one of the process to reduce the pollution. But the mechanical and other properties of the recycled materials are poor due to poor compatibility among different plastics. The compatibility among different waste plastic material can be improved by the use of a compatibilizer.

Waste plastic materials consist of a higher amount of polyethylene (PE), polypropylene (PP) and a lesser amount of polyvinylchloride (PVC), polystyrene (PS), polyethylene terephthalate (PET). Solution blending is one of the process used for blending of different kind of plastics. The effectivity of the blending process can be improved by the use of a mixture of solvents. The optimum ratio of solvents can be judged properly if the percentage of individual poly-

mer along with their physical characteristics in waste plastics is known. This can be overcome if a mixture of known percentage of virgin HDPE, PP, PVC, etc. is used as starting waste plastic material.

Polymer-clay nanocomposites have evoked a great interest in recent years. Improvements in mechanical, dimensional, thermal, flame retardancy have been the reason for their widespread use [1–3].

In order to make a good composite, the improvement in adhesion among plastics materials as well as with plant material is very important. The adhesion or compatibility can be improved by using a compatibilizer. The compatibilizer should be able to react with hydrophilic plant material and hydrophobic plastic materials. Pracella et al. [4] used ethylene-glycidyl methacrylate copolymer, ethylene-propylene copolymer grafted maleic anhydride, isotactic PP grafted acrylic acid for improvement in interaction between various polyolefins and polyethylene terephthalate.

The use of maleated polypropylene compatibilizer enhances the dimensional stability and mechanical properties of the polypropylene, wood flour and nanoclay composite [5]. Glycidyl methacrylate (GMA) has been used as a compatibilizer to modify the adhesion between wood and polymer [6]. Reports on a large number of polymer nanocomposites [7–9] are available. However, far less is known regarding wood polymer nanocomposite.

The objective of the present study is to prepare the nanocomposites using wood flour, PE-co-GMA, nanoclay, polymer mixture of (HDPE + LDPE + PP + PVC) by solution blending. Efforts have also been made to study the various properties of the nanocomposite.

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2. Experimental

2.1. Materials

HDPE and LDPE (Grade: PE/20/TK/CN) were obtained by the courtesy of Plast Alloys India Ltd. (Harayana, India). PP homopolymer (Grade: H110MA, MFI 11 g/10 min) and PVC (Grade: SPVC FS: 6701) were supplied by Reliance Industries Ltd. (Mumbai, India) and Finolex Industries Ltd. (Pune, India). The compatibilizer poly(ethylene-co-glycidyl methacrylate)(PE-co-GMA) (Otto, Mumbai, India) and nanomer (clay modified by 15–35 wt.% octadecylamine and 0.5–5 wt.% aminopropyltriethoxy silane, Sigma–Aldrich, USA) were used as such received. A nonconventional wood, Nal, was collected from local forest of Assam. Other reagents used were of analytical grade.

2.2. Preparation of wood samples

Nals (*Phargmites karka*) were collected from the local forest and chopped into small strips. The chopped wood strips were initially washed with 1% soap solution, followed by washing with 1% NaOH solution and finally with cold water. The washed wood strips were oven dried at 105 ± 5 °C till attainment of constant weight. These dried wood strips were grinded in a mixer and sieved. The sieved wood flour was kept in a container for subsequent use.

2.3. Preparation of wood polymer nanocomposite

A certain amount of xylene was taken in a flask fitted with a condenser. To this, the granules of HDPE, LDPE and PP were added slowly at room temperature. The compatibilizer, PE-co-GMA was also added at the same time. The temperature of the flask was increased to 130 °C in order to make a homogenous solution. The temperature of the flask was then decreased to 120 °C. Now, another solution containing a predetermined quantity of PVC in tetrahydrofuran (THF) was prepared. Both these solutions were mixed at 120 °C (approximately) under stirring condition for 1 h. A known quantity of nanomer was dispersed in tetrahydrofuran (THF) solution by sonication. This dispersed clay-THF mixture was added gradually to the polymer solution under stirring condition. Oven dried wood flour was added slowly to this clay-polymer solution. The mixing was done for another 1 h. The mixture was transferred in tray, dried and grinded. The composite sheets were obtained by the compression molding press (Santec, New Delhi) at 150 °C under a pressure of 80 MPa.

Polymer blend (HDPE + LDPE + PP + PVC), polymer blend/5 phr PE-co-GMA, polymer blend/5 phr PE-co-GMA/3 phr nanomer and polymer blend/5 phr PE-co-GMA/40 phr wood were designated as PB, PB/G5, PB/G5/N3 and PB/G5/W40. WPCs reinforced with 1, 3 and 5 phr clay were designated as PB/G5/W40/N1, PB/G5/W40/N3 and PB/G5/W40/N5.

3. Measurements

3.1. X-ray diffraction (XRD)

The degrees of clay intercalation in WPCs were evaluated by X-ray diffraction (XRD) analysis. XRD measurements were carried out in a Rigaku X-ray diffractometer (Miniflex, UK) using Cu $K\alpha$ ($\lambda = 0.154$ nm) radiation at a scanning rate of $1^\circ/\text{min}$ with an angle ranging from 2° to 30° .

3.2. Transmission electron microscopy (TEM)

The study of the dispersion of silicate layers of nanoclay in WPCs were performed by using Transmission Electron Microscope (JEM-100 CX II) at an accelerated voltage of 20–100 kV.

3.3. FTIR studies

FTIR spectra of wood flour, nanoclay and WPCs were recorded in FTIR spectrophotometer (Impact-410, Nicolet, USA) using KBr pellet.

3.4. Scanning electron microscopy (SEM)

The compatibility among different polymers as well as morphological features of the WPC were studied by using Scanning electron microscope (JEOL JSM – 6390LV) at an accelerated voltage of 5–10 kV. Fractured surface of the samples, deposited on a brass holder and sputtered with platinum, were used for this study.

3.5. Thermal property

Thermal properties of WPCs were measured in a thermogravimetric analyser (TGA) (TGA-50, shimadzu) at a heating rate of 10 °C/min up to 600 °C under nitrogen atmosphere.

3.6. Mechanical property

The tensile and flexural tests for polymer blend and WPCs were carried out using Universal Testing Machine (Zwick, model Z010) at a crosshead speed of 10 mm/min at room temperature according to ASTM D-638 and D-790 respectively. Three samples of each category were tested and their average values were reported.

3.7. Hardness

The hardness of the samples were measured according to ASTM D-2240 using a durometer (model RR12) and expressed as shore D hardness.

3.8. Water uptake

Percentage water uptake was measured by submerging the samples in distilled water at room temperature (30 °C) for different time periods after conditioning at 65% relative humidity, according to the formulae below.

$$\text{Water uptake (\%)} = (W_s - W_1)/W_1 \times 100$$

where W_s is the weight of the water saturated specimen and W_1 is the weight of the oven dried specimen.

4. Results and discussion

In order to optimise the solvent ratio, HDPE, LDPE, PP and PVC were mixed in the ratio of 2:2:2:1. Preliminary investigations indicated that xylene was a good solvent for HDPE, LDPE and PP. Similarly, tetrahydrofuran (THF) was a good solvent for PVC. Neither xylene nor THF could solubilise the mixture of HDPE, LDPE, PP and PVC (2:2:2:1). The solubility of the polymer mixture was checked by varying the ratio of xylene and THF. The optimum ratio of solvent (xylene:THF) and minimum temperature at which a homogeneous solution was obtained, were 70:30 and 120 °C respectively.

4.1. XRD results

The XRD patterns of pure nanoclay and WPCs with different percentage of nanoclay loading are shown in Fig. 1. The interlayer spacing was calculated according to Bragg's law,

$$n\lambda = 2d\sin\theta \quad (1)$$

The organically modified nanoclay (curve a) shows a sharp peak at $2\theta = 4.1^\circ$. The peak (curve e) shifted to a lower diffraction angle

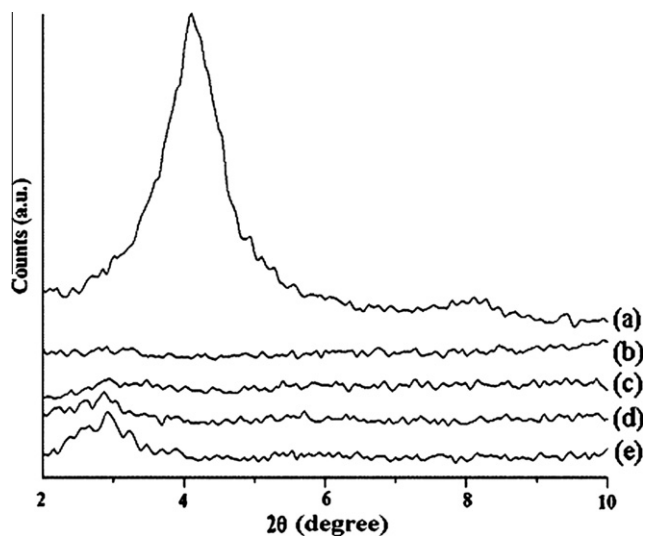


Fig. 1. X-ray diffraction of: (a) nanomer, (b) PB/G5/W40/N1, (c) PB/G5/W40/N3, (d) PB/G5/W40/N5, (e) PB/G5/N3.

($2\theta = 2.9^\circ$) in the case of nanomer and PE-co-GMA containing polymer nanocomposite. The shifting of the peak to lower angle indicated an increase in interlayer spacing of silicate layers. The polymer chains were intercalated into the silicate nanolayers of the polymer composite. A shifting of diffraction peak to the lower angle for the clay based HDPE nanocomposite was reported in the literature [10]. The peak could not be identified in the case of WPC (curves b and c) indicating an exfoliated structure. WPC loaded with 5 phr nanomer (curve d) showed a small peak at $2\theta = 2.8^\circ$. The reduction in peak intensity as well as shifting of peak to lower angle suggested an increased disorder into the nanoclay layers in the nanocomposite. The observed peak at higher loading of nano-

mer might be due to agglomeration of some clay. The silicates nanolayers might be partially exfoliated and partially intercalated. The shifting of the peak from higher diffraction angle to lower diffraction angle was reported by Lee and Kim [11] while studying the interaction of organoclay with wood/polypropylene composite.

4.2. TEM study

Fig. 2 shows the TEM micrographs of WPC with various percentage of nanomer content. The dark line represents the intersection of silicate layers in the WPC was observed even at lower level of nanomer (1 phr) loading (Fig. 2a). The nanomer exhibited better dispersion of clay layers in WPC when loading of nanomer increased to 3 phr (Fig. 2b). However, with the increase in the level of nanomer loading to 5 phr (Fig. 2c), the size of nanomer became larger or aggregated. Similar observation was reported by Zhao et al. [12] while studying the dispersion of organically modified montmorillonite into wood flour/polyvinyl chloride matrix. TEM results supported the findings of XRD studies.

4.3. FTIR study

FTIR spectra of wood, nanoclay, clay added polymer blend and WPCs are shown in Fig. 3. The FTIR spectra of wood (curve a) showed the presence of bands at 3423 cm^{-1} for $-\text{OH}$ stretching, 2923 cm^{-1} for $-\text{CH}$ stretching, 1734 cm^{-1} for $\text{C}=\text{O}$ stretching, 1636 cm^{-1} for $-\text{OH}$ bending, 1160 and 1046 cm^{-1} for $\text{C}-\text{O}$ stretching and $1000\text{--}650\text{ cm}^{-1}$ for $\text{C}-\text{H}$ bending vibration (out of plane). Organically modified nanoclay (curve b) exhibited the peaks at 3467 cm^{-1} ($-\text{OH}$ stretching) 2927 and 2852 cm^{-1} ($-\text{CH}$ stretching of modified hydrocarbon), 1619 cm^{-1} ($-\text{OH}$ bending), $1031\text{--}460\text{ cm}^{-1}$ (oxide bands of metals like Si, Al, Mg, etc.). PB/G5/N3 (curve c) exhibited characteristic peaks of $-\text{CH}$ stretching at 2927 cm^{-1} and 1460 cm^{-1} , $\text{C}-\text{CH}_3$ stretching at 2850 cm^{-1} along with $-\text{CH}_2-$ stretching at 720 cm^{-1} .

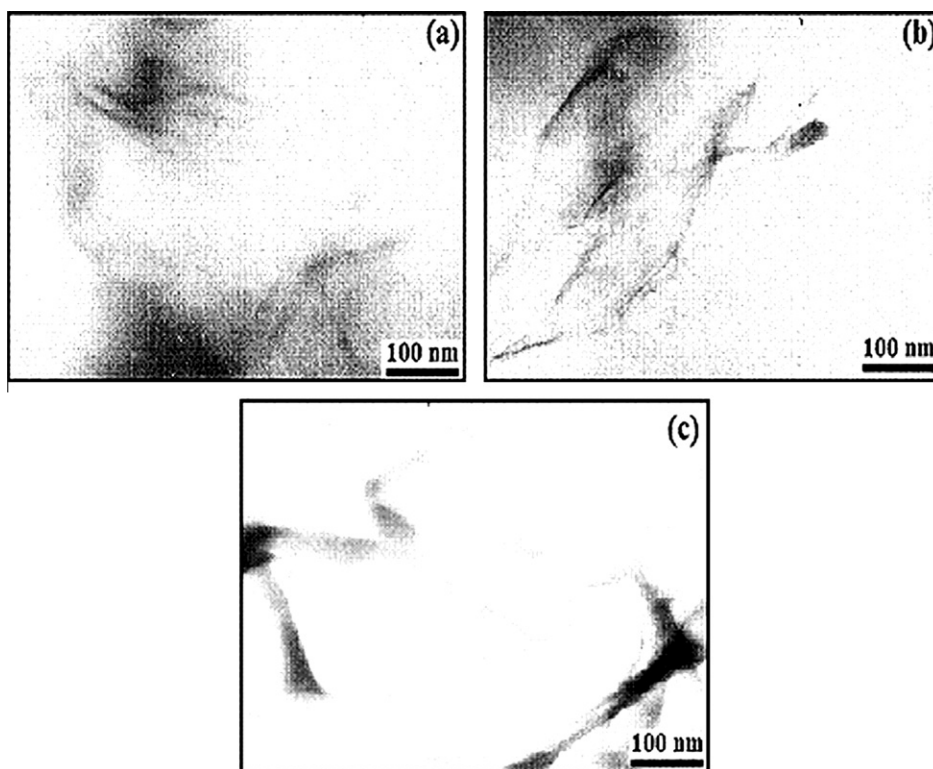


Fig. 2. TEM micrographs of: (a) PB/G5/W40/N1, (b) PB/G5/W40/N3, (c) PB/G5/W40/N5.

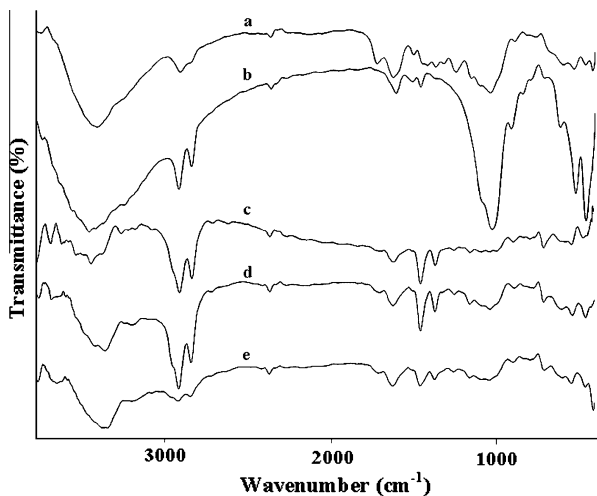


Fig. 3. FTIR spectra of: (a) wood flour, (b) nanomer, (c) PB/G5/N3, (d) PB/G5/W40/N3, (e) PB/G5/W40/N5.

Fig. 3d and e represented the FTIR spectra of wood polymer nanocomposite. It was found that the intensity of the hydroxyl peak decreased as well as shifted to lower wave number in the wood polymer composite. The decrease in peak intensity in the nanocomposite might be attributed to the participation of hydroxyl group of clay in the crosslinking reaction with wood and polymer. The shifting of absorption peak corresponding to hydroxyl group to 3415 cm^{-1} (curve d) and 3411 cm^{-1} (curve e) confirmed the formation of hydrogen bond between wood surfaces and matrix. Furthermore, the intensity of peaks at 2923 cm^{-1} corresponding to $-\text{CH}$ stretching was more in wood composites compared to pure wood which indicated the formation of bond between polymers, PE-co-GMA and wood. Similar type of shifting of hydroxyl group to lower wave number and increase in intensity of $-\text{C}-\text{H}$ stretching was reported by Awal et al. [13] while studying the FTIR analysis of PP/maleated polypropylene/wood composite. The other peaks observed in the spectra of wood polymer composites (curves d and e) at 1050 cm^{-1} and 720 cm^{-1} were due to the $\text{C}-\text{O}$ stretching of wood, oxide bands of silicon and $-\text{CH}_2-$ stretching of polymer. The peaks in the range $450\text{--}500\text{ cm}^{-1}$ were due to the oxide bands of metals like aluminium and magnesium respectively.

4.4. SEM study

SEM micrographs of different fractured samples are shown in Fig. 4. The fractured surface of untreated polymer sample is shown in Fig. 4a. The figure showed that different polymers were immiscible. The immiscibility decreased drastically when the polymer blend was treated with PE-co-GMA (Fig. 4b). The miscibility among different polymers was found to increase with the increase in the amount of PE-co-GMA (not shown in the figure). This might be due to the improvement in compatibility between HDPE, LDPE, PP and PVC in which PE-co-GMA acted as a compatibilizer among polymers. The improvement in adhesion between polyolefins and PET by using ethylene-glycidyl methacrylate copolymer as compatibilizer was reported in the literature [4]. The surface of the wood/polymer composites became smooth on addition of nanomer (Fig. 4c and d). The presence of octadecyl amine and silane groups on nanomer might have favoured the interaction between polymers and wood. This made both the polymers and wood more compatible with nanoclay. There was no significant difference in the surface characteristics on increasing the amount of nanomer from 1.0 to 3.0 phr. However, the surface appeared little bit rough on addition of 5 phr of clay (Fig. 4e). The partial agglomeration of nanoclay might be responsible for this observation.

4.5. Thermal property study

Figs. 5 and 6 show the TG and DTG thermograms of WPC and clay modified WPC. Table 1 shows the initial decomposition temperature (T_i), maximum pyrolysis temperature (T_m), decomposition temperature at different weight loss (%) (T_d) and residual weight (RW, %) for WPC and clay treated WPC. In both the cases, a decrease in weight loss below 100°C was observed which was due to the removal of moisture. WPC showed a lower T_i value compared to clay treated WPC. Similarly T_m values for WPC were observed less compared to clay treated WPC. The T_m values for the first stage in both WPC and clay treated WPC might be due to the depolymerisation of hemicellulose, glycosidic linkage of cellulose, thermal decomposition of cellulose [14] and dehydrochlorination of PVC [15]. T_m values for the second stage was due to the decomposition of HDPE and PP [16,17]. The T_m values for both the stages of pyrolysis were found to shift towards higher temperature when nanomer (1 phr) was added to the WPC. T_d values of the clay treated WPC were more compared to WPC alone. RW value for nanomer treated WPC was more compared to WPC. Therefore, it could be concluded that thermal stability of WPC improved on addition of nanomer. The increase in thermal stability in clay treated WPC may be attributed to the presence of silicate layers which acted as a barrier and delayed the diffusion of decomposed volatile products throughout the composites [18].

4.6. Mechanical property study

The flexural and tensile properties of PB, PB/G5, PB/G5/N3 and WPCs with different percentage of clay loading are shown in Table 2. The data presented were the average of three readings. It was observed that both flexural and tensile properties of polymer blends increased on addition of PE-co-GMA as compatibilizer. PE-co-GMA acted as a dispersing agent among different polymers resulting in improved interfacial adhesion due to which flexural and tensile properties increased. This was also supported by XRD and SEM study. Pracella et al. [4] studied the effect of various compatibilizers on HDPE, PP and PVC blend and found that ethylene-glycidyl methacrylate copolymer provided better interfacial adhesions among different kinds of polymer. Both flexural and tensile properties were increased on addition of wood flour. Since wood flour acted as load carrier, it reinforced the composite and enhanced the flexural and tensile properties. Moreover, PE-co-GMA improved the adhesion between wood and polymer blend through its glycidyl group and hydrocarbon backbone respectively. The incorporation of nanoclay with wood, polymer blend and PE-co-GMA further enhanced the flexural and tensile properties. Both flexural and tensile values increased with clay loading up to 3 phr, beyond that the values decreased. The observed higher values might be due to the higher dispersion of silicate layers of nanoclay in the wood polymer matrix and the restriction in the mobility of the polymer chains inside the intercalated nanolayers of clay. Besides the nanoclay contained some silane and hydrocarbon portions which facilitated the interaction between wood flour and polymer matrix. All these suggested an improvement in adhesion between polymer blend, wood and clay. The nanocomposites thus exhibited better properties over wood flour/PE-co-GMA treated polymer blend composites. The inclusion of organoclay resulted in the improvement in mechanical properties was reported by Lee and Kim [11] while studying the physical properties of wood/PP/clay nanocomposites. The decreased in mechanical properties of WPC treated with 5 phr clay might be attributed to the migration of clay to the interface between wood flour and polymer blend. The presence of high amount of fibre and its interface with polymer might have reduced the reinforcement effect of clay.

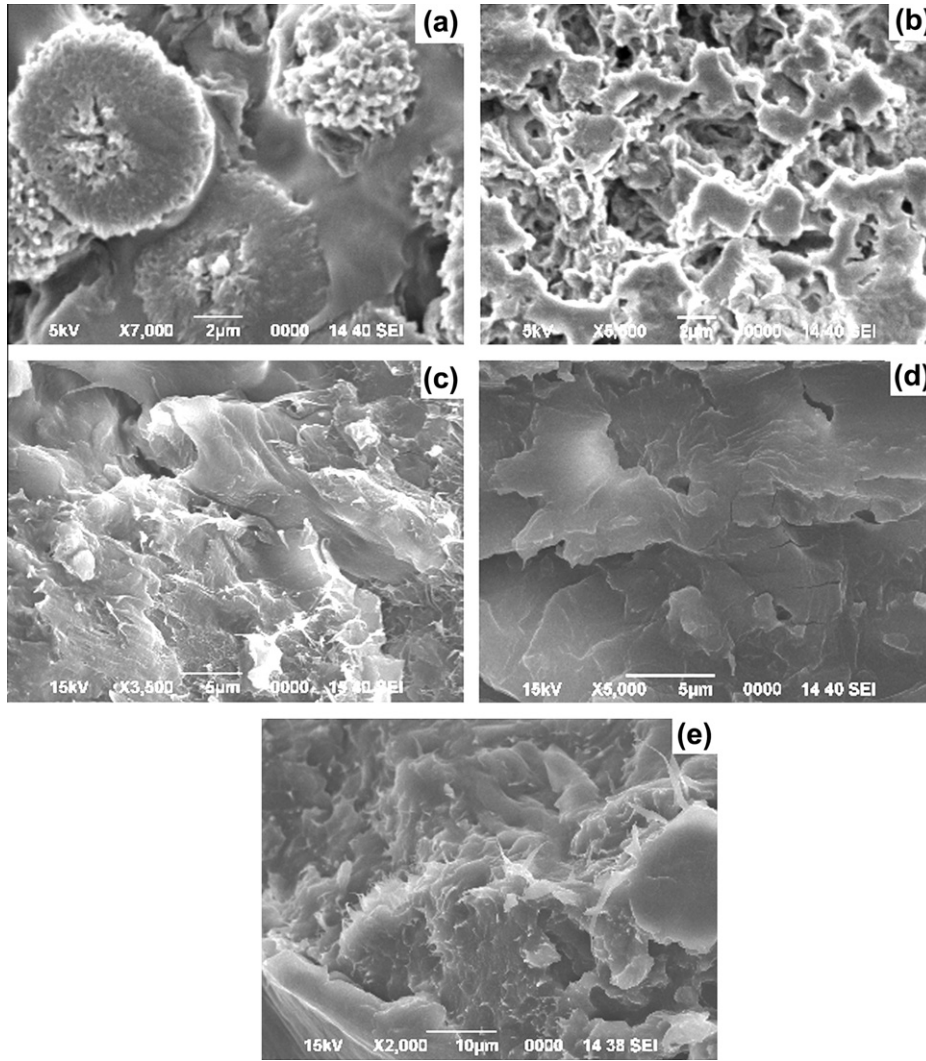


Fig. 4. SEM micrographs of: (a) PB, (b) PB/G5, (c) PB/G5/W40/N1, (d) PB/G5/W40/N3, (e) PB/G5/W40/N5.

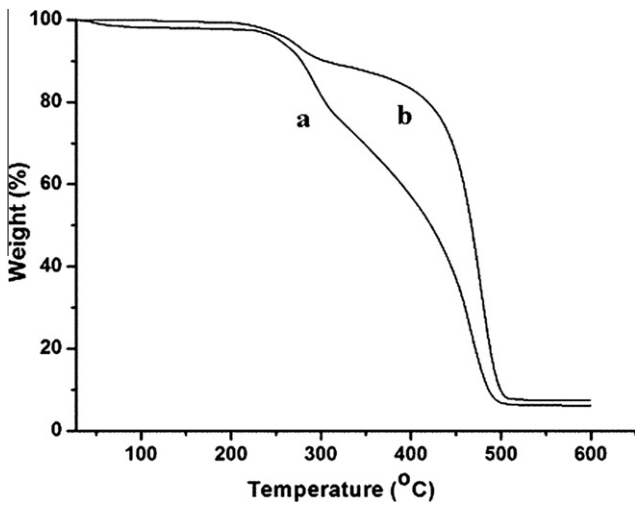


Fig. 5. Thermogravimetric curves for: (a) PB/G5/W40, (b) PB/G5/W40/N1.

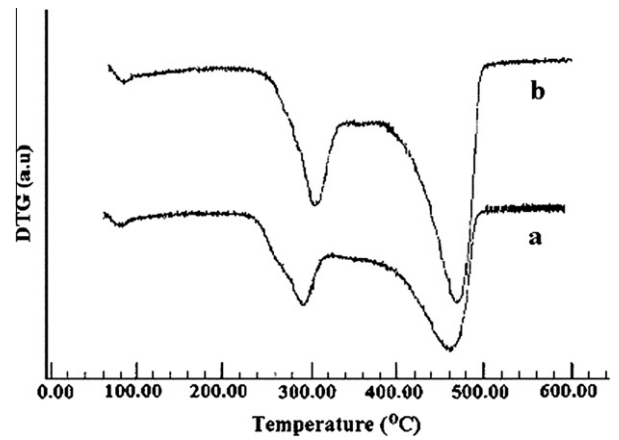


Fig. 6. DTG curves for: (a) PB/G5/W40, (b) PB/G5/W40/N1.

Similar observation was reported by Han et al. [19] during the study of nanoclay reinforced HDPE/bamboo fibre composite.

4.7. Hardness

Table 3 shows the hardness results of PB and WPCs with different percentage of clay loading. From the table, it was observed that hardness increased as PE-co-GMA was added to the polymer blend.

Table 1
Thermal properties of wood/polymer and wood/polymer/clay nanocomposites.

Sample	T_i	T_m^a	T_m^b	Temperature of decomposition (T_D) in °C at different weight loss (%)				RW% at 600 °C
				20%	40%	60%	80%	
PB/G5/W40	240	290	460	304	390	443	471	6.1
PB/G5/W40/N1	249	302	471	419	458	473	488	7.5

^a T_m value for 1st step.

^b T_m value for 2nd step.

Table 2
Flexural and tensile properties of polymer blend, wood/polymer and wood/polymer/clay nanocomposites.

Sample	Flexural properties		Tensile properties	
	Strength (MPa)	Modulus (MPa)	Strength (MPa)	Modulus (MPa)
PB	13.24 ± 1.09	763.28 ± 1.02	5.24 ± 1.13	84.38 ± 18.19
PB/G5	15.39 ± 1.31	1058.36 ± 1.26	8.46 ± 1.21	113.25 ± 16.19
PB/G5//N3	21.42 ± 0.92	4375.61 ± 1.43	24.47 ± 1.31	356.19 ± 19.07
PB/G5/W40	17.47 ± 1.06	3822.13 ± 1.03	18.11 ± 1.20	261.71 ± 17.34
PB/G5/W40/N1	20.32 ± 0.64	4215.34 ± 0.58	23.34 ± 1.03	368.14 ± 16.25
PB/G5/W40/N3	26.17 ± 0.85	4749.53 ± 0.72	31.57 ± 1.01	581.40 ± 18.60
PB/G5/W40/N5	23.44 ± 1.02	4523.82 ± 1.31	28.87 ± 1.27	547.82 ± 24.09

Table 3
Hardness value of polymer blend, wood/polymer and wood/polymer/clay nanocomposites.

Sample	Hardness (shore D)
PB	66.0 (±1.0)
PB/G5	68.5 (±0.5)
PB/G5/N3	68.0 (±1.0)
PB/G5/W40	66.0 (±0.8)
PB/G5/W40/N1	72.0 (±0.9)
PB/G5/W40/N3	77.0 (±0.6)
PB/G5/W40/N5	75.0 (±0.4)

The increased in hardness in polymer blend was due to the improvement in interfacial adhesion between various polymers by PE-co-GMA. The hardness did not improve on addition of clay to the polymer blend. However, on addition of wood flour in the PE-co-GMA modified polymer blend, the hardness value decreased. But the hardness was found to increase further as nanoclay was added. The value improved up to an incorporation of certain amount of nanoclay (3 phr) beyond that the value decreased. The improvement was due to the decrease in mobility of the intercalated polymer chains and increase in interaction between wood flour and polymer by the clay as explained earlier. At higher clay loading, migration of clay to the interface of polymer and wood flour surface might decrease the reinforcement effect of clay. As a result the hardness was found to decrease.

4.8. Water uptake test

The results of water uptake for PB, PB/G5, PB/G5/N3 and WPCs with different percentage of clay loading are shown in Fig. 7. In all the cases, the water uptake was found to increase with the increase of time of immersion. Water absorption occurred initially at a rapid rate and finally at a slower rate. The water absorption of the neat polymer decreased on addition of PE-co-GMA. It was due to the improvement in compatibility among the polymers. PE-co-GMA increased the interaction between the interfaces of the polymers and thus enhanced water resistance. Water absorption decreased further on addition of nanomer to the PE-co-GMA treated polymer blend. The water absorption was found to enhance when wood flour was added. The hydrophilic nature of wood flour was responsible for this. The water absorption of wood flour/polymer blend composite

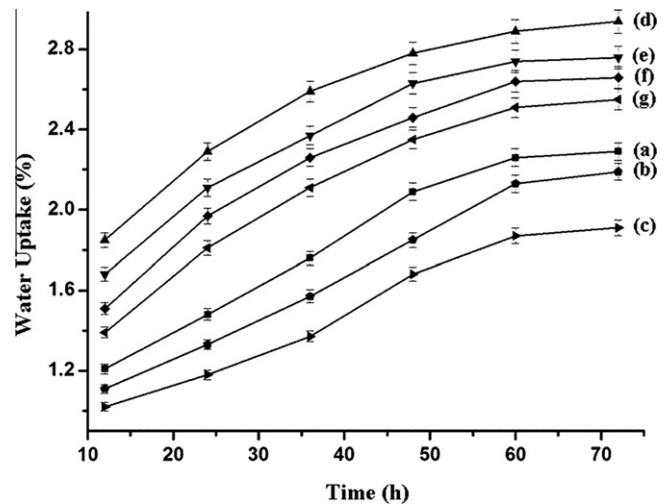


Fig. 7. Water absorption of: (a) PB, (b) PB/G5, (c) PB/G5/N3, (d) PB/G5/W40, (e) PB/G5/W40/N1, (f) PB/G5/W40/N3, (g) PB/G5/W40/N5.

decreased with the incorporation the clay. The higher the amount of clay, the lower was the water absorption. Organically modified clay increased the tortuous path for water transport and as a result water diffusivity decreased [20]. Rana et al. studied the barrier property of nanocomposite where nanoclay hindered the permeation of water through the composite [21]. Moreover, the void spaces in the wood flour were occupied by the polymer and nanoclay. This decreased the available space for water absorption. The higher the nanoclay, the lower was the available space to hold the water. Both the tortuous path and reduction in void space decreased the water absorption capacity of WPC. Hence nanoclay treated composite showed lower water absorption compared to nanoclay untreated composite.

5. Conclusions

The solvent ratio of xylene and THF for solution blending of HDPE, LDPE, PP and PVC (2:2:2:1) were optimised and found to be as 70:30. Polyethylene-co-glycidyl methacrylate (PE-co-GMA) improved the compatibility among the polymers as revealed by SEM study. XRD and TEM study indicated a better exfoliation in

WPC loaded with 1 and 3 phr clay compared to that of WPC loaded with 5 phr clay. FTIR studies showed a strong interaction among wood, PE-co-GMA treated polymer blend and nanoclay. SEM study exhibited an increase in compatibility in the polymer blend due to the addition of PE-co-GMA. The surface of WPC loaded with 5 phr clay showed more roughness compared to WPC loaded with lower amount of clay. Wood/polymer/clay nanocomposites had shown improved thermal, flexural and tensile properties over wood/polymer composites. Nanoclay treated wood/polymer composites showed lower water absorption and highest hardness over nanoclay untreated wood/polymer composites.

Acknowledgement

The authors are thankful to CSIR, New Delhi for their financial assistance.

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