

Effect of SiO₂ and nanoclay on the properties of wood polymer nanocomposite

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Received: 6 September 2011 / Revised: 18 April 2012 / Accepted: 16 June 2012 /
Published online: 23 June 2012
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Abstract Wood polymer composite (WPC) were developed by using solution blended high density polyethylene, low density polyethylene, polypropylene, poly(vinyl chloride), *Phragmites karka* wood flour and polyethylene-co-glycidyl methacrylate. The effect of addition of nanoclay and SiO₂ on the properties of the composite was examined. X-ray diffractometry and transmission electron microscopy were used to study the distribution of silicate layers and SiO₂ nanopowder in the composite. The improvement in miscibility among the polymers and WPC was studied by scanning electron microscopy. Fourier transform infrared spectroscopy study revealed the interaction between polymer, wood, clay and SiO₂. WPC treated with 3 wt% each of clay and SiO₂ showed an excellent improvement in mechanical properties, thermal and flame retarding properties. Water uptake of WPC was found to decrease on incorporation of nanoclay and SiO₂ in WPC.

Keywords Nanocomposites · Polymers · Nanoclay · Mechanical properties · Transmission electron microscopy

Introduction

During the last few decades, wood polymer composite (WPC) have emerged as an important family of green composite [1]. They are used in different outdoor, indoor applications like decking, railing, fencing, docks, landscaping timbers, and in a number of automobiles industries [2]. The composites reinforced with wood have shown a great growth due to the many advantages that they possess. Their processing

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is easy, economic, and ecological. They have relatively high strength and stiffness, low cost, low density, low CO₂ emission, biodegradability and renewable. Varieties of non conventional plant materials like Nal (*Phragmites karka*), a type of non conventional plant, is abundantly available in the forest of Assam, India. These are mostly utilized for fuel purposes or remain as bio wastes. Their uses for structural purposes are restricted due to their poor mechanical properties and dimensional stability. They can be made value added material for preparation of structural components by making composites with plastics materials. These composites will not only contribute to the economical growth but it can also reduce the amount of bio wastes. Sui et al. [3] has prepared WPC by using sunflower hull sanding dust (SHSD), a non conventional plant as reinforcing agent and PP as matrix.

Polypropylene (PP) [4], high density polyethylene (HDPE) [5], low density polyethylene (LDPE) [6], poly(vinyl chloride) (PVC) [7], are the most widely used plastics in large volume because of their versatile properties like light weight, resistance to breakage, low cost, ease of manufacture, fabrication and shaping. These materials can be used by combining with other materials, laminating or blending with other polymers to gain the benefit of their various attributes. The wastage arises from varieties of plastic articles in the form of garbage is a major environmental concern in recent days [8]. One of the ways to minimise the problem is by forming composites of waste plastics with wood. In order to improve the miscibility between hydrophobic polymers and hydrophilic wood flour (WF), compatibilizers are used. They act as a bridging agent between the two interfaces. Compatibilizers like glycidyl methacrylate, maleated PP have significantly improved the properties of the wood polymer composite [9, 10].

In the polymer composites, different types of filler are used for improving the thermal, mechanical, as well as other properties. Among them, clay is widely used as filler. The surface characteristics of nanopowders play a key role in their fundamental properties from phase transformation to reactivity. A dramatic increase in the interfacial area between fillers and polymer can significantly improve the properties of the polymer [11]. Different types of metal oxide nanoparticles such as SiO₂, TiO₂, ZnO, etc., are widely used for these purposes. These are non-toxic, stable and highly thermostable inorganic filler.

In polymer composite, SiO₂ nanopowder is one of the widely used filler. SiO₂ can enhance the mechanical as well as thermal properties of the composite. Polymer–SiO₂ composites have been explored as technological importance due to their potential applications in electrochromic windows, fuel cells, chemical separation, electrochemical sensing, and water treatment [12]. SiO₂ nanoparticles extensively increase the tensile and impact strength of epoxy nanocomposite [13]. In order to increase the hydrophobicity of the inorganic silica particles, the surface has been modified by different silane compound [14].

In this communication, we report the modification of SiO₂ by treatment with cetyl trimethyl ammonium bromide and study the effect of SiO₂ nanopowder along with nanoclay on various properties of composites based on wood, PE-*co*-GMA and polymer mixture of HDPE, LDPE, PP, and PVC.

Experimental

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), PVC (Grade: SPVC FS: 6701) were supplied by Reliance Industries Ltd. (Mumbai, India) and Finolex Industries Ltd. (Pune, India), respectively. The compatibilizer poly(ethylene-*co*-glycidyl methacrylate) (PE-*co*-GMA) (Otto chemicals, Mumbai, India), *N*-cetyl-*N,N,N*-trimethyl ammonium bromide (CTAB) (Central Drug house (P) Ltd, Delhi, India), nanomer (clay modified by 15–35 wt% octadecylamine and 0.5–5 wt% aminopropyltriethoxy silane) (Sigma-Aldrich, USA) and SiO₂ nanopowder (5–15 nm, 99.5 % trace metals basis) (Aldrich, China) were used as such received. A non-conventional wood, Nal (*P. karka*), was collected from local forest of Assam. Other reagents used were of analytical grade.

Preparation of wood samples

Nals (*P. karka*), a kind of soft wood, is available in the forest of Assam. It was collected and chopped into small strips. These 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 100 ± 5 °C till the attainment of constant weight. These dried wood strips were grinded in a mixer, sieved at 60 mesh sizes and kept for subsequent uses.

Modification of SiO₂

Ten gram of SiO₂ was taken in a round bottom flask containing 1:1 ethanol–water mixture stirred at 80 °C for 12 h. 12 g of CTAB was taken in a beaker containing ethanol–water mixture and stirred at 80 °C for 3 h. This mixture was added slowly to the flask containing SiO₂ mixture under stirring condition. The stirring was continued for 6 h. The mixture was then filtered and washed with deionised water for several times. It was collected, dried in vacuum oven at 45 °C, grinded and stored in desiccator to avoid moisture absorption. CTAB-modified SiO₂ was dispersed in a flask containing xylene and kept for checking of any settling of particles. The dispersion of SiO₂ was found to be stable. This indicated that proper modification occurred.

Preparation of wood polymer nanocomposite

The minimum ratio of xylene and THF, at which a homogenous mixture of HDPE, LDPE, PP, and PVC was obtained, was optimized as 70:30. 6 g each of HDPE, LDPE, and PP (1:1:1) were added slowly to a flask containing 105 ml of xylene at room temperature. This was followed by the addition of the PE-*co*-GMA (5 wt%). The temperature of the flask was increased from room temperature to 130 °C in order to make a homogenous solution. Now, another solution containing 3 g of PVC

in 35 ml of tetrahydrofuran (THF) was prepared. The temperature of the polymer solution containing HDPE, LDPE, and PP was brought down to 120 °C. To this, PVC solution was added gradually and stirring was continued at 120 °C (approximately) for 1 h. A known quantity of nanomer (3 wt%) and CTAB-modified SiO₂ nanopowder (1–5 wt%) was dispersed in 10 ml of THF solution by sonication. This dispersed mixture was added gradually to the polymer solution under stirring condition. Oven-dried WF (40 wt%) was added slowly to this solution. The whole mixture was stirred for another 1 h. The mixture was transferred to a 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 wt% PE-co-GMA and polymer blend/5 wt% PE-co-GMA/40 wt % wood were designated as PB, PB/G5, and PB/G5/W40. WPC filled with 3 wt% nanoclay and 1, 3, and 5 wt% SiO₂ were designated as PB/G5/W40/N3/S1, PB/G5/W40/N3/S3, and PB/G5/W40/N3/S5.

Measurements

X-ray diffraction (XRD)

The degree of nanoclay dispersion and SiO₂ distribution in the WPC were evaluated by X-ray diffraction (XRD) analysis. It was carried out in a Rigaku X-ray diffractometer (Miniflux, UK) using CuK α ($\lambda = 0.154$ nm) radiation at a scanning rate of 1°/min with an angle ranging from 2° to 70°.

Transmission electron microscopy (TEM)

The study of the dispersion of nanoclay and SiO₂ nanoparticles in WPCs was performed by using transmission electron microscope (JEM-100 CX II) at an accelerated voltage of 20–100 kV.

Scanning electron microscopy (SEM)

The compatibility among different polymers and 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.

FTIR studies

FTIR spectra of WF, nanoclay, SiO₂ nanopowder, and WPC loaded with nanoclay and SiO₂ were recorded in FTIR spectrophotometer (Impact-410, Nicolet, USA) using KBr pellet.

Mechanical property

The tensile and flexural tests for polymer blend, PE-co-GMA treated polymer blend, and WPC loaded with different percentage of nanoclay and SiO₂ were carried out using Universal Testing Machine (Zwick, model Z10) at a crosshead speed of 10 mm/min at room temperature according to ASTM D-638 and D-790, respectively. Ten samples of each category were tested and their average values were reported.

Hardness

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

Thermal property

Thermal properties of polymer blend and the 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.

Limiting oxygen index (LOI)

Limiting oxygen index (LOI) of the samples was measured by flammability tester (S.C. Dey Co., Kolkata) according to ASTM D-2863 method. The ratio of nitrogen and oxygen at which the samples continue to burn for at least 30 s were recorded.

$$\text{Limiting oxygen index (LOI)} = \text{Volume of O}_2 / \text{Volume of (O}_2 + \text{N}_2) \times 100.$$

Water uptake test

WPC samples were cut into $2.5 \times 0.5 \times 2.5 \text{ cm}^3$ for the test. 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 and 30 °C and expressed 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.

Results and discussion

XRD results

XRD results of nanoclay, polymer-blend, SiO₂ and wood polymer composite loaded with clay (3 wt%), and different percentage of SiO₂ (1–5 wt%) are represented in

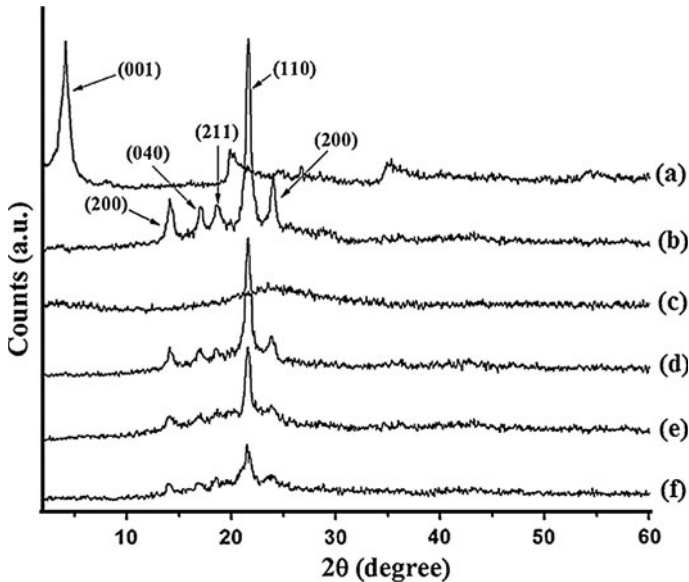


Fig. 1 X-ray diffraction of (a) nanoclay, (b) PB, (c) nano SiO₂, (d) PB/G5/W40/N3/S1, (e) PB/G5/W40/N3/S3, and (f) PB/G5/W40/N3/S5

Fig. 1. Organically modified nanoclay (curve a) shows the diffraction peak at $2\theta = 4.11^\circ$ with a basal spacing of 2.15 nm. In the diffractogram of polymer blend (curve b), the most prominent wide-angle XRD peaks appeared at $2\theta = 14.12$ (200), 17.06 (040), 18.64 (211), 21.62 (110), and 24.02 (200) were for crystalline portion of different polymers present in the blend [15–18]. Curve c shows a broad diffraction peak at $2\theta = 23.5^\circ$ for the amorphous SiO₂ nanoparticles. Curves (d–f) represent the diffractograms of WPC loaded with nanoclay (3 wt%) and different percentage of SiO₂ (1–5 wt%). The figures did not exhibit any characteristic peak of nanoclay. The exfoliation of silicate layers in the wood polymer matrix was the cause for disappearance of nanoclay diffraction peak. The crystalline peak intensity of the polymer blend appeared in the range of $2\theta = 14\text{--}25^\circ$ was found to decrease with the increase in the level of incorporation of SiO₂ (1–5 wt%). Similar decrease in peak intensities of polymer and increase in peak intensity due to incorporation of TiO₂ was observed and reported by Mina et al. [15] while studying the XRD profile of PP/titanium dioxide composite. This suggested that the nanoclay layers were exfoliated and SiO₂ particles were dispersed in the wood polymer matrix.

TEM results

TEM micrographs of polymer blend loaded with compatibilizer, WPC and WPC loaded with nanoclay and different percentage of SiO₂ (1–5 wt%) are shown in Fig. 2. The silicate layers of nanoclay are represented by dark lines and the SiO₂ nanoparticles are presented by black spots in the figures. At lower percentage of SiO₂ loading (1–3 wt%), a good dispersion of clay and SiO₂ nanoparticles was

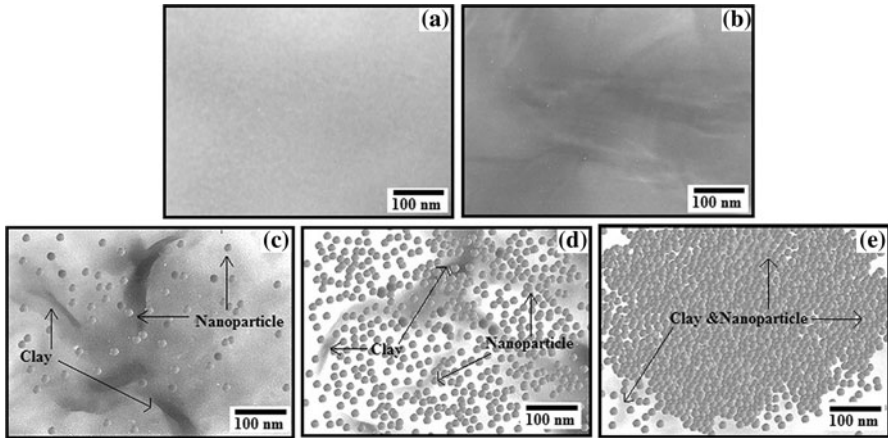


Fig. 2 TEM micrographs of (a) PB/G5, (b) PB/G5/W40, (c) PB/G5/W40/N3/S1, (d) PB/G5/W40/N3/S3, and (e) PB/G5/W40/N3/S5

observed. Na-MMT layers together with SiO₂ nanoparticles were found to disperse well in PMMA matrix as reported by Bao and Ma [19]. At higher percentage of SiO₂ (5 wt%) loading, an agglomeration of the nanoparticles was observed. The agglomeration occurred due to the surface interaction among the nanoparticles.

SEM results

Figure 3 shows the SEM micrographs of different fractured samples of PB, WPC and WPC loaded with nanoclay (3 wt%) and different percentage of SiO₂ (1–5 wt%) nanopowder. The polymers were found immiscible (Fig. 3a) at the first stage. But after the incorporation of compatibilizer, the miscibility among the

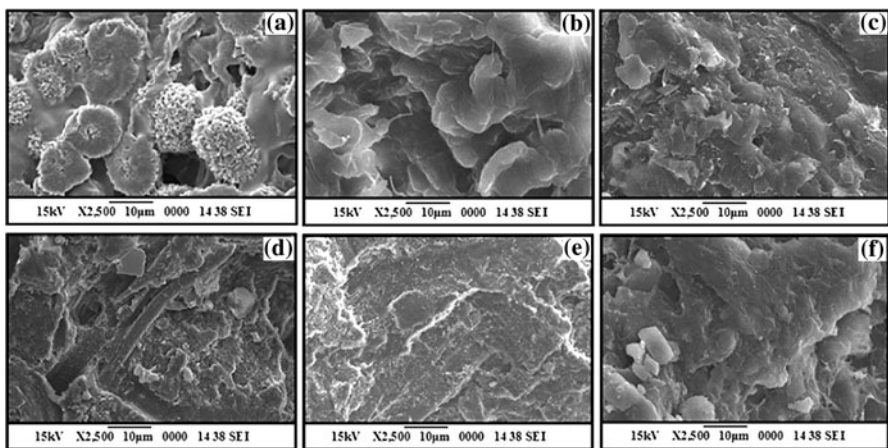


Fig. 3 SEM micrographs of (a) PB, (b) PB/G5, (c) PB/G5/W40, (d) PB/G5/W40/N3/S1, (e) PB/G5/W40/N3/S3, and (f) PB/G5/W40/N3/S5

polymers increased (Fig. 3b). The compatibilizer increased the interfacial adhesion among the polymers, and thereby improved the miscibility among them [20]. The addition of WF improved the smoothness of composite (Fig. 3c). The smoothness of surface was further improved after the incorporation of wood, clay and SiO₂. The surface smoothness was increased up to the addition of clay (3 wt%) and SiO₂ (3 wt%) (Fig. 3d–e). The surface of WPC loaded with 5 wt% SiO₂ and clay (Fig. 3f) appeared less smoother compared to WPC loaded with 3 wt% SiO₂ and clay. The increase in smoothness might be due to the improvement in interaction among PE-*co*-GMA, hydroxyl groups of wood and clay, organic surfactant present in clay and polymer. Moreover, CTAB-modified SiO₂ also has long olefinic chain which further improved the interaction among them. The agglomeration occurred at higher percentage of SiO₂ loading might be due to the surface interaction between SiO₂ nanoparticles. Similar agglomeration of SiO₂ nanoparticles at higher percent of loading was observed by Chrissafis et al. [21] during the thermal study of HDPE/fumed silica nanocomposite.

FTIR results

FTIR spectra of CTAB, SiO₂, and CTAB-modified SiO₂ are shown in Fig. 4. The absorption peaks at 2,918, 2,848, and 1,475 cm⁻¹ were assigned to asymmetric, symmetric, and scissor modes of -CH₂ stretching of the CTAB (curve a), respectively [22]. The spectrum of unmodified SiO₂ shows some absorption peaks at 3,424 for -OH stretching and 1,632 cm⁻¹ for -OH bending of hydroxyl group adsorbed on surface of the particles. The other peaks appeared in the range 1,087–465 cm⁻¹ were due to Si-O-Si group in the SiO₂ [23]. In the spectrum of CTAB-modified SiO₂,

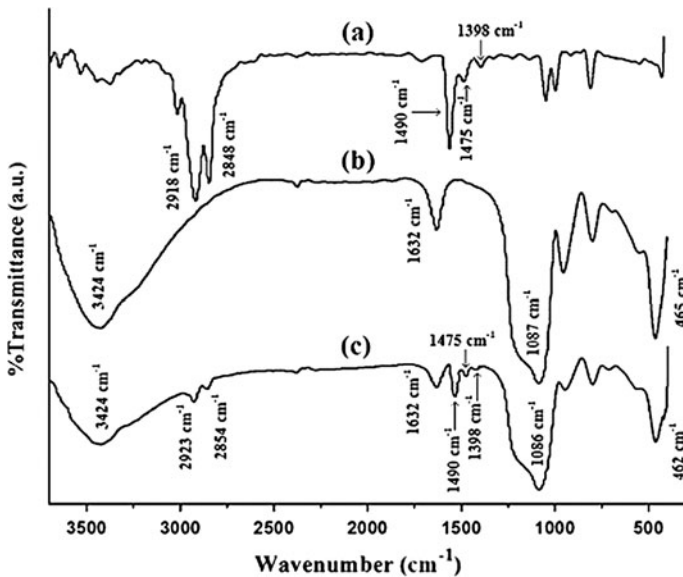


Fig. 4 FTIR spectra of (a) CTAB, (b) unmodified SiO₂, and (c) CTAB-modified SiO₂

the $-OH$ peak intensity was found to decrease. This indicated an interaction of the hydroxyl group absorbed on SiO_2 surface with CTAB. Two new peaks at $2,923$ and $2,854\text{ cm}^{-1}$ assigned for $-CH_2$ stretching of CTAB were also observed. The peaks at $1,490$ and $1,398\text{ cm}^{-1}$ were due to the asymmetric and symmetric CH_3-N^+ deformation mode of CTAB headgroup [22]. Fu and co-workers [24] modified TiO_2 with CTAB and reported that the interaction of TiO_2 occurred with Br^- of CTAB through hydrogen bond formation or electrostatic attractions. The other notable peaks appeared in the CTAB-modified SiO_2 spectrum were the characteristic peaks for CTAB and SiO_2 . These results indicated the incorporation of CTAB on the surface of SiO_2 particles.

Figure 5 shows the FTIR spectra of wood, nanoclay, PB/G5, WPC, and WPC loaded with nanoclay (3 wt%) and SiO_2 (1–5 wt%). Curve a shows the presence of absorption bands at $3,434\text{ cm}^{-1}$ for $-OH$ stretching, $2,931$ and $2,849\text{ cm}^{-1}$ for $-CH$ stretching, $1,730\text{ cm}^{-1}$ for $C=O$ stretching, $1,635\text{ cm}^{-1}$ for $-OH$ bending, $1,163$ and $1,045\text{ cm}^{-1}$ for $C-O$ stretching and $1,000$ – 645 cm^{-1} for $C-H$ bending vibration (out of plane) of the wood sample. Organically modified nanoclay (curve b) exhibited the peaks at $3,470\text{ cm}^{-1}$ ($-OH$ stretching), $2,935$ and $2,849\text{ cm}^{-1}$ ($-CH$ stretching of modified hydrocarbon), $1,620\text{ cm}^{-1}$ ($-OH$ bending) and $1,030$ – 460 cm^{-1} (oxide bands of metals like Si, Al, Mg, etc). Polymer blend loaded with compatibilizer (curve c) shows peaks at $2,919$ and $2,838\text{ cm}^{-1}$ ($-CH$ stretching), $1,473\text{ cm}^{-1}$ ($-CH$ bending), and 714 cm^{-1} ($-CH_2$ bending). PB/G5/W40 (curve d) exhibited peaks at

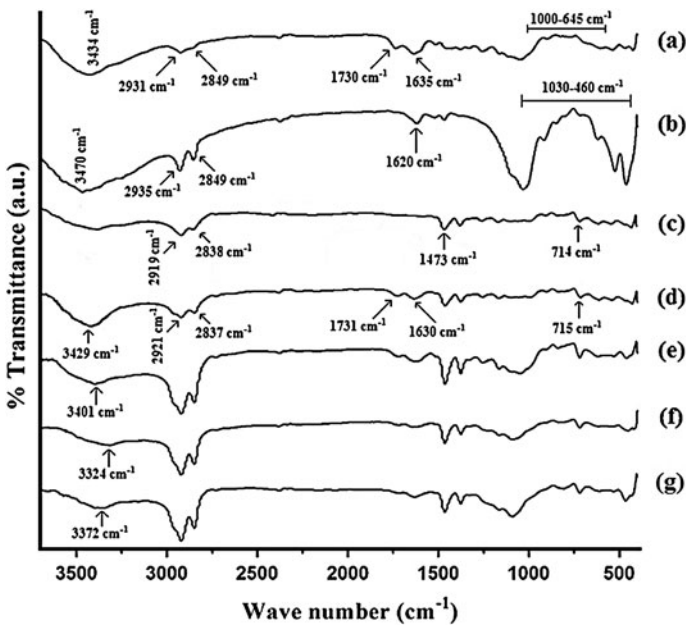


Fig. 5 FTIR spectra of (a) wood, (b) Nanoclay, (c) PB/G5, (d) PB/G5/W40, (e) PB/G5/W40/N3/S1, (f) PB/G5/W40/N3/S3, and (g) PB/G5/W40/N3/S5

3,429 cm^{-1} (–OH stretching), 2,921 and 2,837 cm^{-1} (–CH stretching), 1,731 cm^{-1} (C=O stretching), 1,630 cm^{-1} (–OH bending), and 715 cm^{-1} (–CH₂ bending).

Figure 5e–g represents the FTIR spectra of WPC loaded with 3 wt% nanoclay and 1, 3, and 5 wt% of SiO₂, respectively. It was observed that the intensity of –OH stretching decreased and shifted to 3,401 cm^{-1} (curve e), 3,324 cm^{-1} (curve f) and 3,372 cm^{-1} (curve–g) from 3,434 cm^{-1} (wood) and 3,470 cm^{-1} (nanoclay). The decreased in intensities and shifting to lower wavenumber confirmed the bond formation between the hydroxyl groups of wood, nanoclay and SiO₂. Moreover, the increase in intensities of –CH stretching at 2,921 and 2,837 cm^{-1} (curve e–g) compared to those of wood indicated an interaction between polymer, wood and compatibilizer. Similar type of decrease in intensity of –OH absorption peak, a shifting to lower wavenumber and increase in –CH peak intensities was reported by Maji and Deka [25]. In the spectra of WPC (curve e–g), the intensity of the metal oxides bond appeared in the range 1,030–460 cm^{-1} , corresponding to nanoclay and SiO₂, decreased to a desirable extent indicating the formation of bond between wood, clay, SiO₂, and polymers.

Mechanical property results

Table 1 shows the flexural and tensile properties of polymer blend, WPC and WPC loaded with nanoclay and different percentage of SiO₂ nanopowder. After incorporation of compatibilizer, both flexural and tensile properties of the polymer blend increased. This was because of the improvement in interfacial adhesion between the polymers by the compatibilizer. The values were further improved after the addition of WF. The improvements in flexural and tensile properties were due to the reinforcing action provided by WF. Moreover, the compatibilizer, PE-co-GMA, increased the interfacial adhesion between wood and polymers by its glycidyl linkage and long olefinic chain and thus improved the strength properties. Deka and Maji [26] studied the properties of polymer blend/WF composite and observed an

Table 1 Flexural, tensile and Hardness properties of polymer blend and WPC loaded with different percentage of nanoclay and SiO₂

Sample	Flexural properties		Tensile properties		Hardness (Shore D)
	Strength (MPa)	Modulus (MPa)	Strength (MPa)	Modulus (MPa)	
PB	13.47 ± 0.94	759.63 ± 1.06	5.73 ± 1.04	84.72 ± 16.37	66.6 (± 0.5)
PB/G5	16.25 ± 1.13	1031.19 ± 1.11	8.96 ± 1.26	115.51 ± 16.76	68.7 (± 0.3)
PB/G5/W40	18.14 ± 1.21	3798.72 ± 1.07	17.82 ± 1.15	263.42 ± 18.03	66.3 (± 0.7)
PB/G5/W40/N3/S1	30.52 ± 1.03	4971.60 ± 1.12	35.63 ± 1.17	636.41 ± 17.83	79.2 (± 0.1)
PB/G5/W40/N3/S3	37.26 ± 1.09	5686.91 ± 1.18	40.15 ± 1.26	692.75 ± 18.53	84.3 (± 0.6)
PB/G5/W40/N3/S5	32.53 ± 1.12	5172.50 ± 1.16	36.97 ± 1.12	647.53 ± 18.32	81.4 (± 0.2)

improvement in mechanical properties after the incorporation of compatibilizer and WF. The properties were further improved after the incorporation of clay and SiO₂. The silicate layers of nanoclay act as a reinforcing agent that binds the polymer chain inside the gallery space and thus restricts the mobility of the polymer chains. The increased in mechanical properties of wood–plastic composite after the incorporation of nanoclay was reported by Faruk and Matuana [27]. At a fixed level of nanoclay (3 wt%) loading, both flexural and tensile properties were improved up to the addition of 3 wt% SiO₂, beyond that level of SiO₂ loading, these values decreased. The surface hydroxyl groups of CTAB-modified SiO₂ nanopowder might interact with the hydroxyl groups of wood, while the long alkyl chain of CTAB could interact with the polymer blend. Hence, a well distribution of the nanoparticles occurred which increased the flexural and tensile properties of the composite. Yu et al. [28] reported that the mechanical properties of polyacrylonitrile improved due to addition of Na-MMT and SiO₂ nanopowder. At higher percentage of SiO₂ loading (5 wt%), the agglomeration of the nanoparticles resulted in a decrease in mechanical properties.

Hardness results

Hardness results of polymer blend and WPC with clay and different percentage of SiO₂ loading are also shown in Table 1. It was observed that after the incorporation of compatibilizer, hardness value of the polymer blend increased. The compatibilizer improved the interfacial adhesion between the polymers and hence increased the hardness value. After the addition of WF to the polymer blend, the value was found to decrease. But the hardness value was found to increase again after the incorporation of nanoclay and SiO₂ to the WPC. The improvement was due to the restriction in the mobility of the polymer chains provided by the silicate layers and increase in interaction between clay, SiO₂ nanopowder, wood, and polymer blend as mentioned earlier. At higher SiO₂ (5 wt%) loading, hardness value was found to decrease. The decrease in hardness value was due to the agglomeration of SiO₂ nanoparticles.

Thermal property results

Table 2 represents the initial decomposition temperature (T_i), maximum pyrolysis temperature (T_m), decomposition temperature at different weight loss (%) (T_D) and residual weight (RW, %) for the polymer blend and WPCs. The incorporation of compatibilizer and WF enhanced the T_i values of the polymer blend. The compatibilizer increased the interfacial adhesion among the polymers and with WF through its long hydrocarbon chain and glycidyl group that leads to an increase in the T_i values. Deka and Maji [26] observed an increase in thermal stability of polymer blend after incorporation of compatibilizer and WF. T_i value was further improved after the incorporation of clay and SiO₂ nanopowder. The tortuous path provided by the well distributed silicate layers of clay prevented the passage of volatile decomposed product throughout the composite. The incorporation of nanoclay improved the thermal stability of PP/WF nanocomposite [29]. The higher

Table 2 Thermal analysis and limiting oxygen index (LOI) values of polymer blend and wood polymer nanocomposite

Sample	T_i	T_m^a	T_m^b	Temperature of decomposition (T_D) in °C at different weight loss (%)				RW % at 600 °C	Limiting oxygen index (%)
				20 %	40 %	60 %	80 %		
PB	248	267	401	287	386	435	467	5.7	22
PB/G5	251	270	407	291	389	439	470	6.2	35
PB/G5/W40	256	278	448	299	402	447	477	7.1	38
PB/G5/W40/N3/S1	272	321	497	324	453	479	495	11.1	63
PB/G5/W40/N3/S3	278	329	503	335	461	484	501	14.0	68
PB/G5/W40/N3/S5	275	325	501	328	457	481	498	12.2	65

T_i value for initial degradation

^a T_m value for 1st step

^b T_m value for 2nd step

thermal diffusivity of SiO₂ nanoparticles improved the heat dispersion inside the composite. This would lead to a delay in burning of the surface and release of combustible volatile product throughout the composite. The incorporation of silica improved the thermal stability of PMMA [14]. At higher percentage (5 wt%) of SiO₂ loading, the agglomeration of oxide nanoparticles offered the passage of volatile products easily. Hence, the thermal stability decreased.

Limiting oxygen index (LOI) results

Table 2 also shows the LOI values of polymer blend, WPC and WPC loaded with nanoclay and SiO₂. It was observed that LOI value of the polymer blend increased after the addition of compatibilizer. The compatibilizer improved the interfacial adhesion among the polymers and thus increased in LOI value. LOI value was further increased after the incorporation of WF. A substantial improvement in LOI value was observed after the addition of clay and SiO₂. The value increased up to the addition of 3 wt% each of nanoclay and SiO₂, beyond that it decreased. The tortuous path provided by the silicate layers had better barrier property to the oxygen and heat which delayed the burning capacity of the composite. Guo et al. [30] observed that the flame retardancy of wood fiber–plastic composite increased after the incorporation of nanoclay. The addition of CTAB-modified SiO₂ enhanced the interaction between wood, clay, and polymer through its hydroxyl and cetyl groups. SiO₂ nanoparticles also provided some thermal barrier to the oxygen and heat leading to an improvement in flame retardant property. The improvement in flame retardancy of PP filament due to the inclusion of nano SiO₂ was reported by Erdem et al. [31]. At higher concentration of SiO₂ (5 wt%), LOI value decreased.

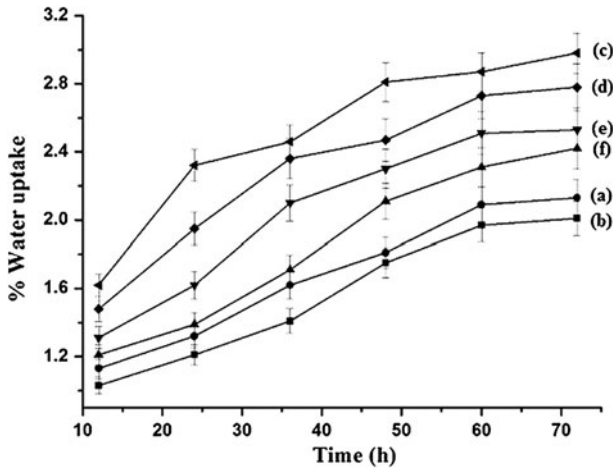


Fig. 6 Water uptake of (a) PB, (b) PB/G5, (c) PB/G5/W40, (d) PB/G5/W40/N3/S1, (e) PB/G5/W40/N3/S5, and (f) PB/G5/W40/N3/S3

The agglomeration of SiO_2 nanoparticles resulted in the decrease of interaction and hence reduction in barrier property as well as LOI value.

Water uptake results

The water uptake results of polymer blend, PE-*co*-GMA treated polymer blend and WPC loaded with clay and different percentage of SiO_2 are shown in Fig. 6. On addition of PE-*co*-GMA compatibilizer, water uptake of the polymer blend decreased. This was due to the improvement in interaction between the polymers by the compatibilizer. The value of water uptake was found to increase after the addition of WF to the blend. The hydrophilic nature of WF caused an increase in the water uptake value. The water uptake decreased after the addition of clay and SiO_2 . WPC loaded with 3 wt% each of clay and SiO_2 showed lowest water uptake followed by WPC with 3 wt% clay and 5 wt% SiO_2 , and WPC with 3 wt% clay and 1 wt% SiO_2 . The tortuous path provided by the silicate layers of clay increased the barrier property for water transport [32]. The barrier property provided by SiO_2 nanopowder might prevent the passage of water. The more the distribution of nanoparticles, the better was the barrier property. Minelli et al. [33] reported that the water uptake of polyvinyl alcohol decreased after the incorporation of SiO_2 nanopowder. At higher concentration of SiO_2 (5 wt%), the agglomeration of nanoparticles might be responsible for the increase in water uptake value.

Conclusions

Solution blending of HDPE, LDPE, PP, and PVC (1:1:1:0.5) was optimized as 70:30. Use of polyethylene-*co*-glycidyl methacrylate (PE-*co*-GMA) as

compatibilizer improved the compatibility among the polymers and WF as studied by SEM. XRD and TEM analysis showed the distribution of silicate layers of nanoclay and SiO₂ in the composite. Surface modification of SiO₂ nanoparticles by cationic surfactant CTAB and the interaction among wood, PE-co-GMA, SiO₂, and nanoclay were examined by FTIR. WPC loaded with nanoclay and SiO₂ showed a remarkable improvement in flame retardancy, mechanical properties, and thermal stability. The incorporation of nanoclay and SiO₂ nanoparticles decreased the water uptake value of WPC. WPC loaded with 3 wt% each of clay and SiO₂ exhibited maximum improvement in properties.

Acknowledgments The authors thank Council of Scientific and Industrial Research (CSIR)-New Delhi for their financial assistance (Grant number: 01(2287)/08/EMR-II).

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