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# Study on properties of nanocomposites based on HDPE, LDPE, PP, PVC, wood and clay

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Abstract Wood polymer nanocomposite (WPC) was prepared by solution blending of high density polyethylene, low density polyethylene, polypropylene and polyvinyl chloride (1:1:1:0.5) with wood flour and nanoclay. Xylene and tetrahydrofuran were used as solvent and the ratio was optimized at 70:30. TEM study revealed better dispersion of silicate layers in WPC loaded with 3 wt% of clay. WPC loaded with 3 wt% nanoclay exhibited higher thermal stability compared to WPC loaded with 1 and 5 wt% clay. The storage and loss modulus were found to enhance on incorporation of clay to WPC. The damping peak was found to be lowered by the addition of clay to WPC. Limiting oxygen index value increased due to incorporation of nanoclay. WPCs were subjected to exposure to cellulase producing Bacillus sp. and it showed the growth of bacteria as revealed by SEM study. Mechanical properties of WPC decreased due to degradation by bacteria. Water vapour uptake of WPC decreased due to addition of nanoclay.

Keywords Nanocomposites Wood Dolymers Thermostability . Biodegradability

## Introduction

Polymer is playing an important role in the field of composite. It is used in all class of materials from household items to different exterior products. They are widely

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used because of their versatile properties, light weight, resistance to breakage, low cost, ease of manufacture, fabrication and shaping. Moreover, they can be also used widely with other materials to improve various properties.

Amongst the different types of plastics, high density polyethylene (HDPE), low density polyethylene (LDPE), polypropylene (PP) and polyvinyl chloride (PVC) are mostly used in industries. After use, the throwing of plastic materials here and there in the form of carry bags, boxes, packaging film causes a serious threat to the environment. Recycling and reusing is one of the processes to reduce this environmental pollution problem. One of the fundamental goals of recycling is to improve different properties of the recycled plastics [[1\]](#page-15-0). The properties can be improved substantially if composites are made by combining these waste materials with cellulosic materials.

Solution blending is one of the processes to mix varieties of plastic materials. The efficiency of the solution blending process for mixing different kind of plastics can be enhanced by the use of a mixture of solvents. The percentage of individual polymer in the blend as well as their physical characteristics is needed to optimise the solvent ratio. Waste plastic materials contain substantial amount of HDPE, LDPE, PP and lesser amount of PVC, PET etc. It is difficult to segregate different kinds of plastics. The optimization of solvent ratio can be determined properly if a mixture of known percentage of virgin HDPE, LDPE, PP, PVC etc. is used as starting waste plastic materials.

Solution blending involves the use of solvents which are not eco-friendly. Melt blending suffers from some disadvantages like stress development, temperature etc. [\[2](#page-16-0)]. A high level of intercalation with partial exfoliation has been obtained for organoclays/LLDPE composites prepared by solution blending [[3\]](#page-16-0) whereas microcomposites have been formed when LLDPE is melt blended with organically modified clay [[4\]](#page-16-0). Chiu et al. [\[5](#page-16-0)] observed a well dispersion of silicate layers when they studied PP/clay composite by solution blending method.

Wood is used as a source of natural filler in wood polymer composites. It acts as a reinforcing agent in the polymer matrix. Wood flour (WF) has been treated with HDPE and nylon 6 to prepare composite [\[6](#page-16-0)]. Lei et al. [[7\]](#page-16-0) has prepared wood plastics composites based on HDPE/PET matrix and noticed a remarkable enhancement of mechanical properties. But most of reports address the use of conventional wood material as a source of cellulosic materials. The nonconventional plant materials like Nal (Phragmites karka), Kalmou (Ipomoea carniva) etc. are abundantly available in forests of Assam. They remain as bio-wastes. These non conventional plant materials can be made value added by making composites with plastic materials.

In order to make composite, the improvement in miscibility amongst plastic materials as well as with wood fillers is very important. The miscibility can be improved by the use of a suitable compatibilizer. The compatibilizer will be such that it can able to react with the hydrophilic wood fillers and hydrophobic polymers and at the same time will improve the interfacial adhesion amongst different thermoplastic materials. The use of maleated coupling agents to strengthen composites containing filler is reported in the literature [[8\]](#page-16-0). Maji et al. [\[9](#page-16-0)] has used glycidyl methacrylate as compatibilizer and found a significant improvement in properties of wood polymer composites. Luyt et al. [\[10](#page-16-0)] has studied the effect of poly(ethylene-co-glycidyl methacrylate) content on the morphology and physical properties of ethylene vinyl acetate-wood fibre composite and found an improvement of the properties.

It is well documented that the incorporation of clay as reinforcing filler in polymer matrix improves different physical and chemical properties. Organoclay has been used as nanosized filler [\[11](#page-16-0)] to improve the performance of wood/PP composite. PP/clay nanocomposites has been synthesized and found to improve the mechanical and thermal properties [[12](#page-16-0)]. The available reports provide information mostly by making composites with wood and single variety of polymer. Little information regarding the use of more than one variety of polymer for making composite is available. Thus, there is enough scope to do further study in this area.

The object of this investigation is to prepare the nanocomposites using WF,  $PE-co-GMA$ , nanoclay, polymer mixture (HDPE + LDPE + PP + PVC) by solution blending and study the various properties of the nanocomposites. Waste plastics comprise of higher amount of HDPE, LDPE, PP and lower amount of PS, PVC, PET etc. On the basis of the presence of individual polymers in waste plastics, the ratio of HDPE, LDPE, PP and PVC is chosen as 1:1:1:0.5 for our investigation.

## Experimental

## Materials

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

Preparation of wood samples

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

Preparation of wood polymer nanocomposite (WPC)

Six grams of each HDPE, LDPE and PP  $(1:1:1)$  granules were added slowly to 105 mL of xylene taken in a flask fitted with a spiral condenser at room temperature. This was followed by the addition of the PE-co-GMA (5 wt%). The temperature of the flask was then increased gradually to  $130 \degree C$  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 flask containing polymer solution was decreased from 130 to 120  $^{\circ}$ C. The PVC solution was then mixed with the polymer solution at 120 °C (approximately) under stirring condition for 1 h. A known quantity of nanomer  $(1-5 \text{ wt\%})$  was dispersed in THF solution (10 mL) by sonication. This dispersed clay–THF mixture was added gradually to the polymer solution under stirring condition. Oven-dried WF (40 wt%) was added slowly to this clay–polymer solution. The whole mixture was stirred for another 1 h. The mixture was then transferred in tray, dried and grinded. The composite sheets were obtained by using grinded WF–polymer powder and employing compression moulding press (Santec, New Delhi) at 150 C under a pressure of 80 MPa. The abbreviation used for samples are listed in Table 1.

# Bacterial media

Mineral salt medium with the following composition was prepared for bacterial growth. 4.75 g KH<sub>2</sub>PO<sub>4</sub>, 2.0 g Na<sub>2</sub>HPO<sub>4</sub>, 2.0 g (NH<sub>4</sub>)<sub>2</sub>SO<sub>4</sub>, 1.2 g MgSO<sub>4</sub>·7H<sub>2</sub>O, 100 mg  $MnSO_4 \cdot 5H_2O$ , 100 mg  $CuSO_4 \cdot 7H_2O$ , 70 mg  $ZnSO_4 \cdot 7H_2O$ , 10 mg  $H_3BO_3 \cdot 5H_2O$ , 10 mg MoO<sub>3</sub>, 1 mg FeSO<sub>4</sub> $\cdot 7H_2O$  and 0.5 mg CaCl<sub>2</sub> $\cdot 2H_2O$  were dissolved in 1000 mL of demineralised water. 3 mL of this liquid culture medium was poured into 50 mL conical test tube and were sterilized using autoclave at 121  $\degree$ C and 15 lbf pressure for 15 min. The autoclaved media were then allowed to cool down to room temperature and WPC samples were added into the media under sterile condition inside a laminar air flow hood. Media containing only polymer samples were also cultured as negative control.

Samples formulation	Code
$PB$ (HDPE + LDPE + PP + PVC)	<b>PB</b>
PB/5 wt% PE- $co$ -GMA	PB/G5
PB/5 wt% PE-co-GMA/3 wt% clay	PB/G5/N3
PB/5 wt% PE-co-GMA/40 wt% wood	PB/G5/W40
PB/5 wt% PE-co-GMA/40 wt% wood/1 wt% clay	PB/G5/W40/N1
PB/5 wt% PE-co-GMA/40 wt% wood/3 wt% clay	PB/G5/W40/N3
PB/5 wt% PE-co-GMA/40 wt% wood/5 wt% clay	PB/G5/W40/N5

Table 1 Formulation of mixture of samples and their codes

## Bacterial strains

*Bacillus* sp. Cd-3 culture was grown using nutrient broth at 37 °C for 18 h. 1 mL of bacterial cultures were centrifuged at 6000 rpm for 20 min at room temperature and the pellets were washed with 0.9% NaCl and re-suspended in 1 mL of mineral salt medium. Now 0.5 mL of the culture medium containing  $1 \times 10^8$ /mL microbes was inoculated to the test tube containing 50 mL media for each test. The test tubes were then incubated under sterile condition at 37  $^{\circ}$ C and 100 rpm for the degradation study.

# **Measurements**

Transmission electron microscopy (TEM)

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

Thermal property

Thermal properties of neat polymer and the wood/polymer/clay composites were measured in a thermogravimetric analyser (TGA) (TGA-50, shimadzu) at a heating rate of 10  $\degree$ C/min up to 600  $\degree$ C under nitrogen atmosphere.

Dynamic mechanical analysis (DMA)

The DMA was performed using an Universal 2980 DMA V1.7B TA instrument. The dimensions of specimens were taken as  $(5 \times 1.25 \times 0.35)$  cm<sup>3</sup>. Specimens were scanned over a temperature range of  $25-200$  °C. Frequency of the oscillation was fixed at 1 Hz ramped at 2  $^{\circ}$ C/min to 200  $^{\circ}$ C. Storage modulus, loss modulus and mechanical loss factor (tan  $\delta$ ) were recorded and plotted against temperature.

Limiting oxygen index (LOI)

The LOI is defined as the minimum concentration of oxygen, expressed in percent volume, in a flowing mixture of oxygen and nitrogen that will support combustion of a material initially at room temperature. It was measured by flammability tester (S.C. Dey Co., Kolkata) according to ASTM D-2863 method. The sample was placed vertically in the sample holder of the LOI apparatus. The total volume of the gas mixture  $(N_2 + O_2)$  was kept fixed at 18 cc. To begin with experiment, the volume of nitrogen gas and that of oxygen gas were kept initially at a maximum and minimum level. Now, the volume of nitrogen gas was decreased and that of oxygen gas was increased gradually. However, the total volume of gas mixture was kept fixed at 18 cc during the experiment. The ratio of nitrogen and oxygen at which the sample continued to burn for at least 30 s was recorded.

Limiting oxygen index = volume of  $O_2$ / volume of  $(O_2 + N_2) \times 100$ .

Biodegradation study

The microbial degradation was studied spectrophotometrically by using UV visible spectrophotometer (CECIL CE7400) at 600 nm against blank culture media under sterile condition.

Scanning electron microscopy (SEM)

The morphological features and bacterial growth of the WPCs were studied by using SEM (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.

Mechanical property

The tensile and flexural tests for biodegraded WPC samples 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 specimen were tested and the average values were reported.

Water vapour uptake test

The WPC samples were cut into  $(2.5 \times 0.5 \times 2.5)$  cm<sup>3</sup> for the measurement of water vapour uptake test. The samples were oven dried and conditioned at room temperature (30  $\degree$ C) and 30% relative humidity before the test. The test was carried out by placing the samples at two different relative humidities namely 95 and 65% and maintaining a temperature of 30  $^{\circ}$ C. Weights of the sample were measured after 12, 24, 36, 48, 60 and 72 h. The experiment was carried out at different time interval. It is expressed as a percentage of moisture absorbed based on oven dry weight.

Water vapour uptake  $(\%) = (W_t - W_o)/W_o \times 100$ 

where  $W_t$  is the weight of the specimen after exposure to humidity and  $W_0$  is the weight of oven dried specimen.

# Results and discussion

The HDPE, LDPE, PP and PVC were mixed at the ratio of 1:1:1:0.5 to optimize the solvent ratio. From the investigation, it was observed that xylene was a good solvent for HDPE, LDPE and PP whereas THF was a good solvent for PVC. None of these solvent alone could solubilise the mixture of HDPE, LDPE, PP and PVC (1:1:1:0.5). The solubility of the polymer mixture was checked by varying the ratio of xylene <span id="page-6-0"></span>and THF. The optimum ratio of solvent (xylene:THF) and minimum temperature at which a homogeneous solution was obtained were 70:30 and 120  $^{\circ}$ C, respectively.

### TEM results

The TEM micrographs of WPC loaded with different percentage of nanomer content are represented in Fig. 1. The dark lines observed in the figure represent the silicate layers present in the composite. At lower level of nanomer (1 wt%) loading (Fig. 1a), a well dispersion of silicate layers in the WPC was observed. The dispersion of clay layers improved up to the addition of 3 wt% of nanomer (Fig. 2b) to the composite. However, at higher level of loading (5 wt%), the silicate layers of clay became to agglomerated (Fig. 2c). Similar dispersion of nanoclay was observed by Faruk and Matuana [[13\]](#page-16-0) whilst studying the dispersion of nanoclay in HDPE/wood matrix.



Fig. 1 TEM micrograph of (a) PB/G5/W40/N1, (b) PB/G5/W40/N3 and (c) PB/G5/W40/N5



Fig. 2 Themogravimetric curves of  $(a)$  WF and  $(b)$  nanoclay

#### TGA study

Figures [2](#page-6-0) and 3 show the weight losses for WF, nanoclay, polymer blend (PB) and WPC. Table [2](#page-8-0) shows the initial decomposition temperature  $(T_i)$ , maximum pyrolysis temperature  $(T<sub>m</sub>)$ , weight losses for various samples at different temperature  $(T_D)$  and residual weight (RW%) for different samples.  $T_i$  value of nanomer was found maximum. This was followed by PB and WF.  $T_i$  value of PB improved on addition of PE-co-GMA and WF. The values were found to enhance further when nanomer was added.  $T_i$  value increased up to addition of 3 wt% clay beyond that it decreased.  $T<sub>m</sub>$  values observed for WF was due to the depolymerisation of hemicelluloses, glycidyl linkage and thermal decomposition of cellulose [\[14](#page-16-0)]. The first decomposition peak shown by the PB might be due to the dehydrochlorination of PVC [\[15](#page-16-0)], whilst the second decomposition peak was due to the decomposition of LDPE, HDPE and PP  $[16, 17]$  $[16, 17]$  $[16, 17]$  $[16, 17]$ .  $T<sub>m</sub>$  value of PB enhanced due to incorporation of PE-co-GMA and WF.  $T<sub>m</sub>$  value increased further when nanomer was added. The value increased up to addition of 3 wt% nanomer beyond that it decreased. Nanomer showed highest RW values whilst the PB showed lowest RW values. The trend of RW values of clay-treated WPC was similar to those of  $T_i$ values. The RW values observed for nanomer and WF were due to the presence of inorganic silicate layer and carbonization of wood, respectively.

Further it was observed from the Table [2](#page-8-0) that PB decomposed at higher temperature compared to WF.  $T_D$  values increased on addition of WF and PE-co-GMA to the PB. This might be due to the enhancement of interaction caused by the increase in interfacial adhesion between WF and PB by PE-co-GMA through its glycidyl group and polyethylene chain. A possible reaction mechanism of PE-co-GMA with wood and polymer is represented by Scheme [1.](#page-8-0) Awal et al. [\[18](#page-16-0)] reported



Fig. 3 Themogravimetric curves of (a) PB, (b) PB/G5/W40, (c) PB/G5/W40/N1, (d) PB/G5/W40/N5 and (e) PB/G5/W40/N3

Sample	$T_{\rm i}$	$T_{\rm m}^{\rm a}$	$T_{\rm m}^{\rm b}$	Temperature of decomposition $(TD)$ in °C at different weight loss $(\%)$				RW% at 600 $^{\circ}$ C
				20%	40%	60%	80%	
WF	230	315	$\overline{\phantom{0}}$	282	320	352		21.9
Nanomer	294	352	$\overline{\phantom{a}}$	514				76.0
<b>PB</b>	251	272	407	297	385	441	470	6.1
PB/G5/W40	257	277	455	303	390	444	472	7.2
PB/G5/W40/N1	260	281	466	309	404	453	478	8.4
PB/G5/W40/N3	270	319	496	322	452	476	493	11.7
PB/G5/W40/N5	263	309	480	313	414	456	481	10.2

<span id="page-8-0"></span>Table 2 Thermal analysis of wood, PB and WPC

 $T_i$  value for initial degradation

 $T<sub>m</sub>$  Value for 1st step

 $\frac{b}{m}$  T<sub>m</sub> Value for 2nd step

**Wood flour** 

Compatibilizer



Scheme 1 A plausible reaction scheme for the compatibilization of WF and polymer by compatibilizer

Polymer

that the incorporation of wood pulp, maleated PP into PP delayed the thermal degradation of the composite.  $T_D$  value increased initially up to addition of 3 wt% clay after that it decreased with the increase in the amount of clay (5 wt%). Therefore, it could be concluded that the inclusion of nanomer improved the thermal stability of WPC. The improvement in thermal stability may be attributed to the presence of silicate layer which acted as a barrier and delayed the diffusion of decomposed volatile products throughout the composites [[19\]](#page-16-0). At higher clay loading, the silicate layer might become agglomerated and decreased the barrier

Composite (compatibilized)

resistance. As a result, the diffusion of volatile products would be rapid and thermal stability would be less.

Dynamic mechanical study

Storage modulus and loss modulus of PB, PB/G5/W40 and PB/G5/W40/N3 are presented in the Figs. 4 and 5. PB/G5/W40 exhibited higher storage and loss



Fig. 4 Storage modulus of (a) PB, (b) PB/G5/W40 and (c) PB/G5/W40/N3



Fig. 5 Loss modulus of (a) PB, (b) PB/G5/W40 and (c) PB/G5/W40/N3

modulus compared to PB. This enhancement might be due to the development of strong interaction between WF and PB by PE-co-GMA. The glycidyl group and long polyethylene chain of PE-co-GMA interacted with the hydroxyl group of wood and PB, respectively. Storage and loss modulus were found to increase on addition of glycidyl methacrylate as compatibilizer to the WPC [\[20](#page-16-0)]. Both the storage and loss modulus were found maximum in the case of PB/G5/W40/N3. The long chain of polymer molecules intercalated into the interlayer space of the silicate layers and restricted the mobility of the polymer chains. As a result, stiffness of the WPC increased and both storage and loss modulus improved. Matuana et al. [[13\]](#page-16-0) studied dynamic mechanical properties of clay/HDPE/wood composite and reported that clay-reinforced HDPE/wood composite showed better storage and loss modulus compared to clay-free HDPE/wood composite. In both the cases, a reduction in modulus with the increase in temperature was observed. This was due to the softening of the matrix at higher temperature [[21\]](#page-16-0).

The damping factor or mechanical loss factor (tan  $\delta$ ) is the ratio of loss modulus to storage modulus and is shown in Fig. 6. PB exhibited a tan  $\delta$  peak with a peak temperature of 96.8 °C. There was no significant shifting of tan  $\delta$  peak in both PB/G5/W40 and PB/G5/W40/N3. However, a lowering of tan  $\delta$  peak height was observed with the addition of WF. This was due to the decrease in the volume fraction of the matrix by the incorporation of WF. Similar observation was reported by Thomas and co-workers [[22,](#page-16-0) [23](#page-16-0)]. The reduction of tan  $\delta$  peak height was more pronounced when clay was added to the composite. This might be due to the restricted movement of the polymer chains caused by the silicate layers of clay and rigid WF molecules.



Fig. 6 Tan  $\delta$  of (a) PB, (b) PB/G5/W40 and (c) PB/G5/W40/N3

Samples	LOI $(\%)$	Flame description	Smoke and fumes	Char
<b>PB</b>	22	Candlelike localised		Little
PB/G5	39	Small localized flame	Small and black smoke	Little
PB/G5/N3	47	Small localized flame	Small and black smoke	Higher
PB/G5/W40	44	Small localized flame	Small and black smoke	Little
PB/G5/W40/N1	55	Small localized flame	Small and black smoke	Higher
PB/G5/W40/N3	61	Small localized flame	Small and black smoke	Higher
PB/G5/W40/N5	66	Small localized flame	Small and black smoke	Higher

Table 3 Limiting oxygen indices and flaming characteristics of PB and wood/polymer/clay nanocomposites

## Limiting oxygen index (LOI)

Table 3 shows the results of LOI values of PB and WPC with varying percentage of nanoclay. It was observed that the value of LOI increased on addition of PE-co-GMA to the PB. The increase in interfacial adhesion amongst polymers by PE-co-GMA might be responsible for the observed higher value of LOI. The values were further improved on addition of nanoclay. The LOI values of the nanoclay-treated wood polymer composites were found more compared to nanoclay-untreated wood polymer composite. The higher the percentage of nanoclay, the higher was the LOI. WF contained cellulose, hemicelluloses, lignin and other low molecular weight components which required lower concentration of oxygen for the production of flammable volatiles and propagation of flame. As a result, the LOI of nanoclayuntreated wood polymer composite was found less. On the other hand, nanoclay produced silicate char on the surface of the clay-treated WPC which increased the flame resistance property of the composite [\[24](#page-16-0)]. The silicate rich surface had better barrier property to heat and oxygen transport due to which ignition of the composite delayed. Besides this, the nanoclay contains amino propyl triethoxy silane and octadecyl amine groups which could interact and promote adhesion with WF and PB. This adhesion might prevent the flame for propagation. The higher the percentage of clay, the stronger was the adhesion as well as resistance of flame propagation.

## Biodegradation study

The WPC samples were exposed for microbial degradation and after 8 weeks of study, wood polymer composites had shown high rate of degradation. Figure [7](#page-12-0) shows the bacterial growth of the WPC with respect to time. From the figure, it was observed that with increasing bacterial exposure time, the growth of bacterial strains increased. Wood composite as sole carbon source showed higher rate of bacterial growth after 3 weeks of incubation and it continued up to eighth week. This enhancement of bacterial growth might be due to powerful cellulolytic and pectinolytic activity of bacteria [\[25](#page-16-0)]. Other than cellulose and pectin, lignin was also

<span id="page-12-0"></span>

Fig. 7 Growth of Bacillus sp. on (a) PB, (b) PB/G5/W40, (c) PB/G5/W40/N1, (d) PB/G5/W40/N3 and (e) PB/G5/W40/N5

a constituent of Phragmites karka plant stem and it was reported that Bacillus sp. could degrade this [\[26](#page-16-0)]. The growth and degradation property of the wood composite by bacteria was also supported by the SEM study (Fig. [8](#page-13-0)). With the increase in the clay content, the rate of bacterial growth enhanced. The improved biodegradability of nanocomposite was due to the catalytic role played by the clay in the biodegradation mechanism [[27\]](#page-16-0). From the study, it was observed that WPC loaded with 5 wt% clay showed maximum bacterial growth. The mechanical properties of the degraded WPC samples were checked, compared with undegraded samples and are presented in Tables [4](#page-13-0) and [5](#page-13-0). The higher mechanical properties shown by both degraded and undegraded WPC samples loaded with 3 wt% clay was due to the better dispersion of silicate layers of nanoclay and enhanced restriction in the mobility of the polymer chains intercalated inside the silicate layers. The lower mechanical properties exhibited by 5 wt% clay loaded WPC was due to the reduction in reinforcement effect of clay caused by the migration of the same to the interface between WF and PB [[28\]](#page-16-0). It was found that with efficient degradation of WPCs, flexural and tensile properties of the WPCs decreased. This might be due to the loss of physical and chemical interaction in the WPC caused by the degradation effect of bacteria.

#### Water vapour uptake test

Water vapour absorption tests of PB, clay treated and untreated WPC were carried out at 30  $\degree$ C and two different relative humidities namely 95 and 65%, respectively and are shown in Figs. [9](#page-14-0) and [10](#page-14-0). As expected, all the samples absorbed less water vapour at 65% RH compared to 95% RH. In all the cases, water vapour absorption occurred initially at a faster rate and finally at a slower rate. The rate of water

<span id="page-13-0"></span>

Fig. 8 SEM micrographs of (a) PB/G5/W40, (b) PB/G5/W40/N1, (c) PB/G5/W40/N3 and (d) PB/G5/ W40/N5

Table 4 Flexural properties of WPC loaded with different percentage of nanoclay after the microbial degradation

Sample	Before degradation		After degradation		
	Strength (MPa)	Modulus (MPa)	Strength (MPa)	Modulus (MPa)	
PB/G5/W40	$18.01 \pm 1.12$	$3864.24 + 1.16$	$16.84 \pm 1.09$	$3686.49 \pm 1.05$	
PB/G5/W40/N1	$20.09 \pm 0.81$	$4193.27 \pm 0.74$	$17.45 \pm 1.53$	$3964.75 \pm 0.93$	
PB/G5/W40/N3	$25.87 \pm 0.93$	$4761.36 + 0.95$	$20.63 \pm 0.87$	$4239.72 \pm 1.07$	
PB/G5/W40/N5	$22.15 \pm 1.66$	$4487.65 \pm 1.08$	$17.74 \pm 1.04$	$3871.84 \pm 1.17$	

Table 5 Tensile properties of WPC loaded with different percentage of nanoclay after microbial degradation



<span id="page-14-0"></span>

Fig. 9 Water vapour absorption at room temperature and 95% relative humidity of (a) PB, (b) PB/G5, (c) PB/G5/N3, (d) PB/G5/W40, (e) PB/G5/W40/N1, (f) PB/G5/W40/N5 and (g) PB/G5/W40/N3



Fig. 10 Water vapour absorption at room temperature and 65% relative humidity of (a) PB, (b) PB/G5, (c) PB/G5/N3, (d) PB/G5/W40, (e) PB/G5/W40/N1, (f) PB/G5/W40/N5 and (g) PB/G5/W40/N3

vapour absorption increased with the increased in time. The water vapour absorption followed the order:  $PB/G5/W40 > PB/G5/W40/N1 > PB/G5/W40/N5 > PB/G5/$  $W40/N3 > PB > PB/G5 > PB/G5/N3$ . The water vapour absorption was found to decrease on addition of PE-co-GMA to the PB. The compatibilizer, PE-co-GMA increased the interfacial adhesion amongst polymers which led to the decrease in water vapour absorption. Maji and co-workers [[29\]](#page-17-0) reported that GMA-treated WPC <span id="page-15-0"></span>absorbed less water vapour compared to untreated WPC. The water vapour absorption was further decreased due to inclusion of clay. The silicate layers of clay acted as a barrier for the moisture which resulted in the decrease of the absorption of water vapour. The value was found to enhance when WF was added. The hydrophilic nature of the WF was responsible for this. Similar results were reported in the literature [[30\]](#page-17-0). The addition of clay to the WPC further decreased the rate of absorption of water vapour. The value decreased up to addition of 3 wt% clay after that it increased. The overall decrease in the moisture absorption was due to the tortuous path imparted by the organically modified silicate layers which prevented the water vapour from transportation and diffusion through the composite [[31\]](#page-17-0).

Moreover, there was less void space available in the wood for the absorption of water vapour due to occupation of the same by nanoclay and polymer. As a result, the water vapour absorption of clay-treated WPC would be less due to reduction in void space in wood and the presence of tortuous path imparted by the silicate layers. The higher water vapour absorption exhibited by the WPC containing  $5 \text{ wt\%}$  clay was due to the agglomeration of silicate layers which decreased the tortuous path for transportation and diffusion of water vapour. In general, it can be concluded that nanoclay-treated WPC absorbed less water vapour compared to clay-free WPC. The trend of water vapour absorption of the samples in the case of 65% RH and 30  $^{\circ}$ C was similar to those of samples tested at 95% RH and 30 °C.

#### **Conclusions**

The solvent ratio of xylene and THF for solution blending of HDPE, LDPE, PP and PVC  $(1:1:1:0.5)$  were optimised and found to be as 70:30. Polyethylene-co-glycidyl methacrylate (PE-co-GMA) acted as an effective compatibilizer for the composite system. The dispersion of silicate layers was found better in WPC loaded with 3 wt% of nanoclay. Thermal stability of the composite was improved due to the incorporation of clay. DMA results indicated an improvement in storage modulus, loss modulus and mechanical loss factor due to incorporation of clay to the WPC. LOI value increased after the incorporation of clay. Biodegradability of the WPC improved on addition of clay. SEM study revealed the presence of maximum bacterial growth in clay-treated WPC. Both flexural and tensile properties of the WPC decreased due to bacterial degradation of composites. Water vapour absorption of the WPC decreased on addition of nanoclay up to 3 wt% beyond that it increased.

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