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MDF dust/PP composites reinforced with nanoclay: Morphology, long-term physical properties and withdrawal strength of fasteners in dry and saturated conditions

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HIGHLIGHTS

- The nanoclay had an effect on the physical and mechanical properties of WPCs.
- A decrease in water absorption of WPC is effectively noted after adding the nanoclay.
- Withdrawal strengths of fasteners in saturated state toward unsaturated state were decreased.

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ABSTRACT

In this study, effect of nanoclay loading (0%, 2%, 4% and 6%) on long-term physical properties and withdrawal strength of fasteners before and after saturation of polypropylene/MDF dust composites was investigated. Sanding dust of medium density fiberboard (MDF) was used as lignocellulose material and polypropylene as the thermoplastic material. The results showed that long-term water absorption (WA) and thickness swelling (TS) was found to decrease as the nanoclay loading increased. Also, WA and TS both were increased with increasing MDF dust content. Maximum withdrawal strengths of fasteners (screws and nails) were obtained in the samples reinforced with 2% weight percentages nanoclay. Withdrawal strengths of fasteners in saturated state toward unsaturated state were decreased. In fact, exposure to water for long term was resulted in significant reduction in withdrawal strengths of fasteners due to the degradation of the fiber/matrix interface. The morphological analysis of wood–plastic composite (WPC) samples was carried out by field emission scanning electron microscopy (FE-SEM) to study the fiber/matrix adhesion interactions. X-ray diffraction (XRD) showed the formation of intercalated nanostructure.

1. Introduction

Wood–plastic composites (WPCs) are gaining a great attention in academic and industrial sectors due to their favorable properties, which include low density, low cost, renewability and recyclability as well as desirable mechanical properties [1–4]. Better stability and favorable mechanical properties has caused WPCs to become a preferred building material [5]. WPCs are widely used in many applications such as the building panels, decking, windowsills, flooring material for trucks, landscaping timbers and automobile [6–8]. Lignocellulosic fibers are obtained from natural resources, so it is available in various forms including wood species, wood waste and agro-based fibers in large amounts, and they have low density and are more cheaply. In other words, wood flour can be easily and cheaply obtained from sawmill wastes. Meanwhile, using sanding dusts in the manufacturing of WPCs not only reduces production costs also removes the problem of accumulating and discarding wood industry waste.

Lignocellulosic fibers are hydrophilic nature and therefore have poor resistance for wet conditions. High moisture uptake is one of the major disadvantages of lignocellulosic fiber, which limits the use of lignocellulosic fibers in WPCs in many outdoors applications [2]. Chavooshi and Madhoushi [7] investigated the MDF dust content on physico-mechanical properties of WPCs. They concluded that the withdrawal strengths of fasteners decreased and water absorption and thickness swelling increased with increasing amount of MDF dust.

One of the ways to raise water resistance as well as mechanical properties of WPCs that has gained great attention in academic and

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industrial sectors is addition of low nanoclay percentage [9]. One of the main advantages of using nanoclay particles in a polymer matrix is the substantial increase in the mechanical properties with inclusion of only a small amount of nanofiller (<10 wt.% and even less than 5%). In other words, nanoclays are not real fillers but rather additives. However, because even in such small concentrations they sometimes improve properties of materials, some call them nanofillers [10]. When compared with a traditional filler (glass, carbon black, talc, etc.), the addition of the nanoclay does not change the viscosity or the density of the system by all that much [11]. An increased number of polymer–particle and particle–particle interactions relative to conventional fillers are provided by the high specific surface area of nanoclay, which is due to its nanometer size and high aspect ratio [12]. The reason for this is the improved physical and mechanical properties of nanoclays filled composites.

Khanjanzadeh et al. [9] and Madhoushi et al. [13] showed that the water absorption and thickness swelling decreased with increasing amount of nanoclay. Also, the tensile strength of WPCs increased dramatically by adding 1–3% by weight of nanoclay, but properties gradually reduced by further increasing the amount of nanoclay [1,14]. Faruk and Matuana [8] reported that the mechanical properties of HDPE/wood-flour composites could be significantly improved with an appropriate combination of the coupling agent content and nanoclay type in the composites. Clay nanocomposites, especially composites reinforced with nanoclay, indicate dramatic increases in modulus, strength, barrier properties, flammability resistance, weathering resistance, water uptake resistance and heat resistance compared with conventional composites [15–22].

The objectives of this study were to elucidate the effect of nanoclay particles in MDF dust/PP composite on the long-term water absorption and thickness swelling and withdrawal strength of fasteners (screws and nails) in dry and saturation conditions.

2. Materials and methods

2.1. Materials

The dust from surface sanding of medium density fiberboard (MDF) boards was used in this work. The particles were sifted with a vibrating screen and particles that pass through 40-mesh (for the separation of oversize particles) were used (Fig. 1a). The sanding dusts were dried at 105 ± 5 °C for 24 h to moisture content of 2–3% (dry-base) and kept in sealed plastic bags before processing. Polypropylene (V30S trade name) supplied by Arak Petrochemical Co., Iran, which has a melt flow index (MFI) of 18 g/10 min and a density of 0.92 g/cm^3, was used as a matrix in this experiment. The maleated anhydride grafted polypropylene (MAPP), in form of powder with a density of 0.91 g/cm^3, a melt flow index of 64 g/10 min, and maleic anhydride of 2%, was supplied by Kimia Javid Sepahan Co., Iran. The nanoclay, with trade name of Cloisite®15A was used (Fig. 1b). Natural montmorillonite modified with a dimethyl, dehydrogenated tallow, 2-ethylhexyl quaternary ammonium (CEC = 125 meq/100 g clay, d_{001} = 31.5 Å) was obtained from Southern Clay Products Co., USA. The properties of nanoclay are given in Table 1. The nanoclay was then dried in a laboratory oven at 105 ± 5 °C for 24 h to moisture content of 0–2%.

2.2. Samples preparation

Formulations of the treatments used for the respective mixes prepared are given in Table 2. Compound mixing was carried out in two stages, in the first stage a mixture of polypropylene, MAPP and MDF dust was prepared by blending the four different weight percentages (0%, 2%, 4% and 6%) of nanoclay using a mechanical mixer for 15 min with a rotation speed of 20 rpm. The mixtures were then fed into a laboratory counter-rotating twin-screw extruder (WPC-4815, Borna Pars Mehr Co., Iran). The temperature profile in the extruder was 170/175/185/190/185 °C and the screw speed was set at 20 rpm.

In the second stage, the granules obtained with a laboratory grinder were powdered and then placed in hot press at 190 °C for 15 min and finally cooled to room temperature under pressure. The pressure for heating was used at 30 bar [23]. Finally, WPC samples were located at room temperature for three weeks.

2.3. Characterization

2.3.1. Mechanical properties

Samples for fasteners withdrawal strength were prepared according to the technical specifications CEN/TS15534:2007 [24]. The samples were cut to 5 cm × 5 cm × 1 cm for the withdrawal strength of fasteners. Withdrawal strengths of fasteners (screws and nails) in both cases, before and after 90 days of water immersion were measured. Six replicates were tested for every property under each formulation. To investigate the withdrawal strengths of fasteners desired were conducted using a Universal Testing Machine (Instron 4486).

2.3.2. Physical properties

Physical properties, namely water absorption (WA) and thickness swelling (TS) were tested in accordance with ASTM D 570 [25]. Before testing, samples were weighed and dimensions were measured. Conditioned samples of each WPCs type were either soaked in distilled water at 23 ± 1 °C for 90 days. At regular intervals (every 10 days), each sample was first removed from distilled water and dried with a tissue before weighting using laboratory scales. Six replications of each sample type were tested.

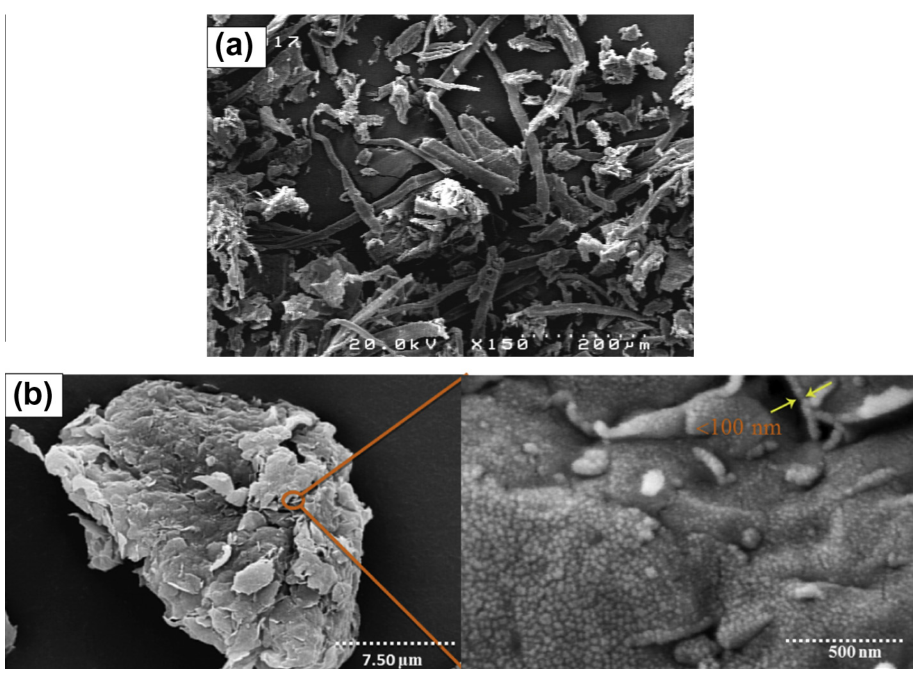

Fig. 1. FE-SEM images of MDF dust particles (a) and nanoclay (b).

Table 1
Typical physical properties of nanoclay (Cloisite®15A).

Typical properties			Typical dry particle sizes			
Color	Moisture	Weight loss on ignition	Density (g/cm^3)	10% less than	50% less than	90% less than
Off white	<2%	43%	1.66	2 µm	6 µm	13 µm

Table 2
Formulations of the studied WPCs.

Codes	MDF dust (wt.%)	Nanoclay (wt.%)	PP (wt.%)	MAPP (wt.%)
A1	40	0	56	4
A2	40	2	54	4
A3	40	4	52	4
A4	40	6	50	4
B1	50	0	46	4
B2	50	2	44	4
B3	50	4	42	4
B4	50	6	40	4
C1	60	0	36	4
C2	60	2	34	4
C3	60	4	32	4
C4	60	6	30	4

2.3.3. Fourier transform infrared spectroscopy (FT-IR)

A Nicolet™ 870 (USA) FTIR spectrometer over the wave number range of 400–4000 cm^{-1} with a resolution of 4 cm^{-1} was used to obtain the spectra. The spectrums were prepared from B2 and B4 treatments.

2.3.4. Field emission scanning electron microscopy (FE-SEM)

A field emission scanning electron microscopy (FE-SEM) (model S-4160, Hitachi, Japan) with accelerating voltage of 20 kV to investigate the microstructures and the fracture surfaces of composites. All specimens were prepared by fracture surfaces area of the composite that was then coated with a thin layer of gold.

2.3.5. X-ray diffraction

Low angle X-ray diffraction (XRD) analysis was carried out to investigate the effectiveness of the nanoclay intercalation in the WPC samples. XRD was performed in an X'Pert X-ray diffractometer (model X'Pert Pro MPD, PANalytical Co.) using Cu Kα radiation (wavelength: 1.5406 Å). The continuous scanning angle range used in this study was from 2° to 10° at 40 kV and 40 mA. The interlayer spacing (d_{001}) of clay was calculated in accordance with Bragg equation (Bragg's law):

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

where λ is the wavelength of X-ray, d is the interlayer or d-spacing of the clay in the nanocomposites and θ is the maximum diffraction angle [8].

2.3.6. Statistical analysis

SPSS programming version 18 (SPSS 18) was used for all statistical analysis. *T*-test analysis was used for comparing two independent samples (withdrawal strength of fasteners before and after saturation in water).

3. Results and discussion

3.1. Mechanical properties

The results of withdrawal strengths of fasteners (screws and nails) for WPC samples are shown in Fig. 2a and b. The fastener withdrawal strengths are reduced by increasing MDF dust content, as observed for all samples. A similar result was observed for WPCs by Chavooshi and Madhoushi [7] and Madhoushi et al. [13]. The greatest increase in the fastener withdrawal strengths was observed when 2 wt.% nanoclay was added to the WPC samples. For instance, the addition of 2 wt.% nanoclay in samples made with 40% of MDF dust led to increases of 15%, 13%, in screw and nail withdrawal strengths, respectively. This increment in fastener withdrawal strengths might be attributed to the high stiffness caused by the decreased in mobility of the polymer chains that were intercalated between the nanoclay interlayers [26]. However,

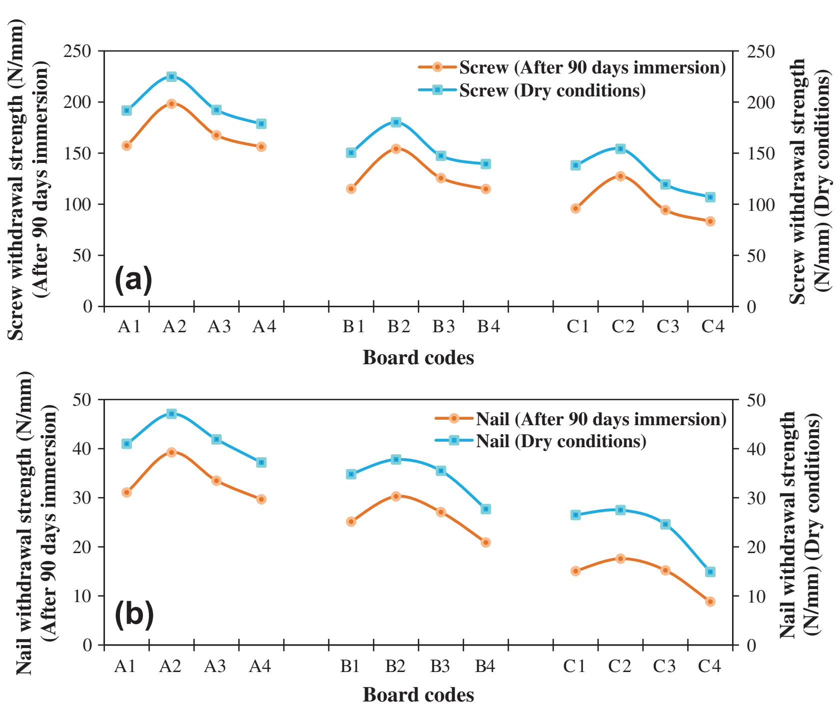

Fig. 2. Withdrawal strength of screw (a) and nail (b) in composites in dry conditions and after of immersion in water.

at higher quantity of nanoclay form interlocked aggregates which envelopment the MDF dust and hamper the formation of desirable bonding between the fiber and the polymeric phase [13].

Furthermore, screw and nail withdrawal strengths are reduced by increasing moisture content. The *T*-test statistical results revealed significant differences between nail withdrawal strengths of the composites in wet conditions compared to the dry conditions (Table 3). Average reduction in withdrawal strengths of fasteners in wet conditions compared to the dry state are shown in Table 4. Generally, for composites with higher (60 wt.%) MDF dust contents immersed in water, it is expected that the relative extent of decrease in withdrawal strengths of fasteners are greater compared to dry samples. Comparing samples after placement in water medium and dry conditions revealed that in wet conditions the nail withdrawal strengths are more affected by moisture content than the screw withdrawal strengths, which is consistent with previous findings [23]. This resistances reduction can be attributed to the degradation in fiber/matrix interfacial bonding due to the water absorption [2]. This decrease in withdrawal strengths of fasteners can be explained as, water absorption causes swelling of MDF dust, which could create micro-cracks in the composite and finally could lead to decreases energy to break.

Also, according to Fig. 2, it is clear that rate of decrease withdrawal strengths of fasteners in control samples (without the nanoclay) more than specimens filled with nanoclay. For example, nail withdrawal strengths of samples containing 40 wt.% of MDF dust (treatment of A1) decreased by 24.176% from 41 to 31.08 N/mm after immersing in water for 90 days (Table 4). The main cause behind this difference in the strength reduction of specimens is presence of nanoclay that reduces the water absorption. However, the addition of nanoclay leads to very slight enhancement in withdrawal strengths of fasteners when compared to unfilled WPC samples. Also, it could be important for external conditions where withdrawal strengths of fasteners are to be used in dry and wet conditions, like naval wharves, and even at high relative humidity [23].

Table 3
Results of *T*-test analysis for withdrawal strengths of fasteners between samples in dry and saturation conditions.

Properties	*F*	*t*	Significant
Screw withdrawal strength	4.406	0.411	0.684[ns]
Nail withdrawal strength	0.091	2.311	0.030[*]

[*] Significant difference at the 5% level ($p \leqslant 0.05\%$).
[ns] No significant.

Table 4
Rate of reduction (%) in withdrawal strengths of fasteners in wet conditions (saturation) compared to the dry conditions.

Codes	Nail withdrawal strengths	Screw withdrawal strengths
A1	24.176	17.959
A2	16.693	11.805
A3	20.101	12.895
A4	20.194	12.590
B1	27.765	23.476
B2	19.874	14.422
B3	23.697	14.788
B4	24.593	17.538
C1	43.141	30.585
C2	36.009	17.404
C3	38.140	21.020
C4	40.687	22.131

3.2. Physical properties

The values of water absorption (WA) and thickness swelling (TS) of nanoclay/MDF dust/PP composites at different periods of immersion are shown in Fig. 3. In all treatments, the both physical properties (WA and TS) were found to increase with the increase of time of immersion. WA up to 40 day of immersion occurred at a rapid rate and after 50 day of immersion at a slower rate. Finally, samples in the long run reached at saturation and did not water absorption. Also, it is observed that significant linear relationships existed between WA and TS (Fig. 3c). As expected, the WA and TS of the composites was negatively affected by the increased MDF dust content. In fact, the increase in MDF dust content from 40 wt.% (treatments series of A) to 60 wt.% (treatments series of C) increased WA and TS values. The main reason for this increase is the reduction of adhesion between filler and polymeric phase. Overall, the WA and TS curves of the WPCs increase with an increase in immersion time until equilibrium conditions are reached [27]. Also, large number of porous tubular structures present in fi-

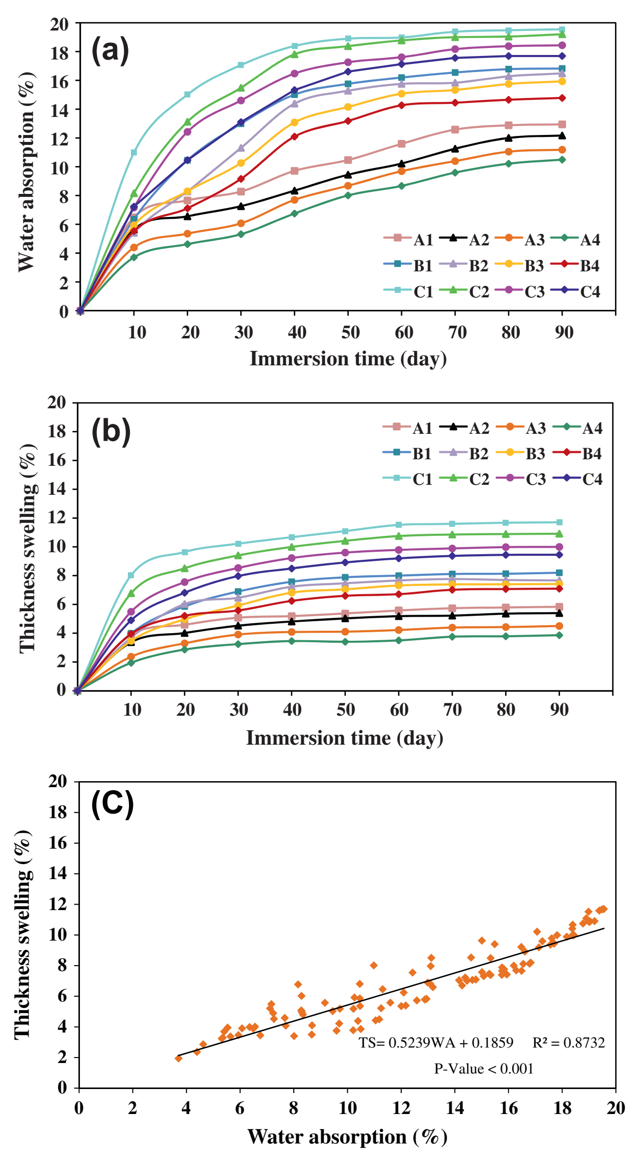

Fig. 3. Long-term water absorption (a) and thickness swelling (b) of the different treatment of nanoclay/MDF dust/PP composites with linear relationships between WA and TS (c).

ber accelerates the penetration of water by the so-called capillary action [13]. Therefore, the hydrophilic nature of cellulosic materials (high hydroxyl groups (–OH) of cellulose and hemicelluloses) is responsible for more water penetration into WPC composites [27,28]. It was said report [7,13] that the physical properties increase with increasing MDF dust content from 40% to 60%.

On the other hand, results show that the WA and TS of samples decreased with increasing nanoclay loading. Maximum value of WA and TS was obtained in treatment C1 and minimum value was observed in the case of A4 (Fig. 3). For instance, the increase of nanoclay amount from 0% (C1) to 6% (C4) in samples manufactured with 60% MDF dust the WA and TS values reduction took place about 11% and 19%, respectively. This reduction was observed in the levels of 40% and 50% MDF dust for WA about 18% and 12% and for TS about 33% and 14%, respectively (Fig. 3). In fact, a nanoclay particle in WPC samples increased the labyrinthine path for water transport and as a result water diffusivity decreased [29]. WPC samples the higher the nanoclay, the lower was the available space to hold the water [1]. Moreover, the void spaces in the WPC samples were occupied by the nanoclay. Deka and Maji [1] observed that water absorption was improved significantly with the addition of nanoclay to wood/polymer composite. This finding is also consistent with previous findings [9,13,16,30–32].

3.3. FT-IR

FTIR spectra of WPC samples (treatments of B2 and B4) are shown in Fig. 4. The main peak (a broad peak) in each of the FTIR spectra represents the hydroxyl (OH) stretching that occurs between 3200 and 3700 cm^{-1}. According to Fig. 4 it was found that the intensity of the hydroxyl peak decreased as well as shifted to lower wave number in the WPC samples. The decrease in peak intensity in the samples containing with 6% nanoclay might be attributed to the participation of hydroxyl group of nanoclay in the crosslinking reaction with MDF dust and polymer. For this reason, physical properties decrease with increasing nanoclay content from 2% to 6% (Fig. 3). The shifting of absorption peak corresponding to hydroxyl group to 3448 cm^{-1} (Fig. 4a) and 3417 cm^{-1} (Fig. 4b) confirmed the formation of hydrogen bond between wood surfaces and matrix. In addition to these peaks, there are peaks arising between 1000 and 1110 cm^{-1} due to Si–O stretching, peaks at around 841 cm^{-1} due to Al–Mg–OH deformation [33]. CH_2 bending vibration causes peaks around 1460 cm^{-1} and CH_3 bending vibration causes peaks 1350 cm^{-1}. These peaks are due to nanoclay vibrations [33,34].

3.4. XRD results

Fig. 5 showed the XRD patterns of the pure nanoclay and WPC samples with different nanoclay content. Pure nanoclay shows a sharp peak at $2\theta = 2.82°$ ($d = 31.33$ nm). WPCs loaded with nanoclay toward pure nanoclay showed a small peak. Samples containing 50 wt.% of MDF dust with 2 wt.% and 6 wt.% of nanoclay (treatment B2 and B4) exhibited a peak at $2\theta = 2.66°$ ($d = 33.133$ nm) and $2\theta = 2.96°$ ($d = 29.817$ nm), respectively. The shifting of the peak to lower angle indicated an increase in interlayer spacing of silicate layers [26]. The polymer chains were intercalated into the silicate nanolayers of the WPC samples. A shifting of the peak from higher diffraction angle to lower diffraction angle for the clay-based different composites was reported in the previous findings [1,8,9,35].

3.5. FE-SEM observation

Figs. 6 and 7 represented the FE-SEM images of nanoclay/MDF dust/PP composites. According to the FE-SEM images of Fig. 6a and b, it is clear that increase in MDF dust content (40–60%) decreased interfacial bonding between the fiber and the matrix

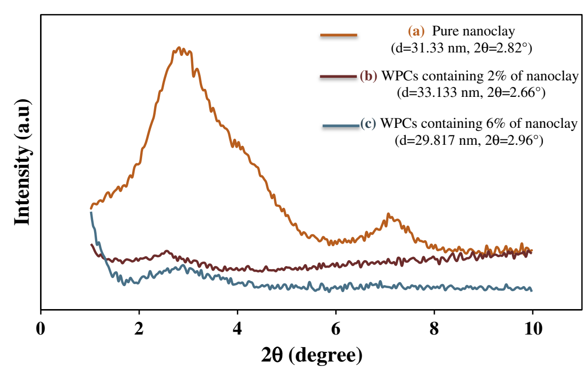

Fig. 5. XRD patterns of pure nanoclay (a) and WPC samples (treatments of B2 (b) and B4 (c)).

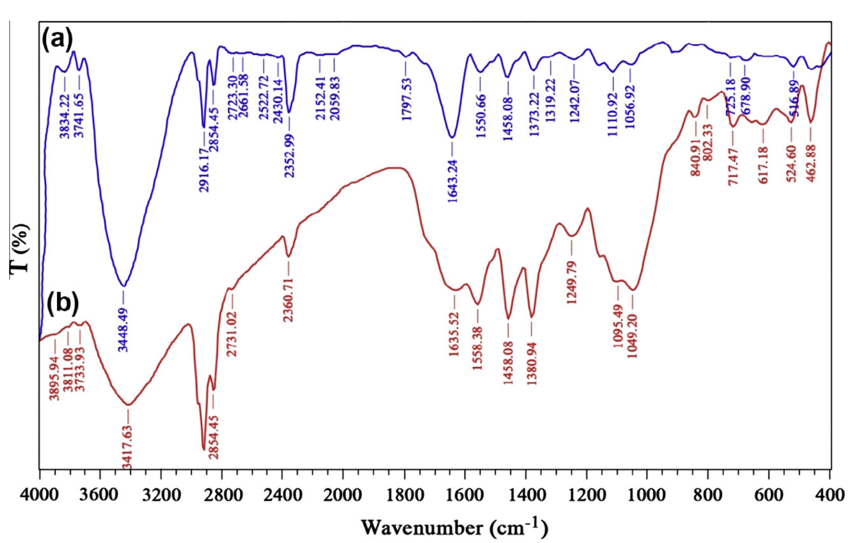

Fig. 4. FTIR analysis for B2 (2 wt.% nanoclay) (a) and B4 (6 wt.% nanoclay) (b) treatments.

polymer. Fiber pull-out can be easily observed in composites with 60 wt.% loading of MDF dust while it is relatively hard to be seen in composites with 40 wt.% loading of MDF dust. Also, in the case of the WPC samples filled with 60 wt.% MDF dust (Fig. 6b) toward filled with 40 wt.% MDF dust (Fig. 6a) many deep voids and cavities remained after the fillers were pulled out of the matrix and it proves the lack of good bonding between MDF dust and polymeric matrix. In fact, the reason that samples with 60 wt.% loading of MDF dust exhibited voids and defects located in the filler/matrix interface was the poor dispersion of the filler in the polymer matrix. The FE-SEM images in Fig. 6 supports increase of the water absorption in composites made with 60 wt.% MDF dust.

Fig. 7 displays different magnification image of samples containing 50 wt.% of MDF dust with 6 wt.% (Fig. 7a and b) and 2 wt.% (Fig. 7c and d) of nanoclay. In the case of the composite with 6% nanoclay content (B4), some agglomerated nanoclay particles were observed (Figs. 7b and 8b). Generally, at greater percentage of nanoclay, the distance between the nanoclay decreases leading to agglomerated within the matrix (Fig. 8). Also, the obvious more uniform distribution obvious in the WPC sample containing 2 wt.% nanoclay suggests that the nanoparticles and matrix were thoroughly mixed (Figs. 7d and 8a). Besides, the uniform distribution of nanoclay in the matrix improved withdrawal strengths of fasteners.

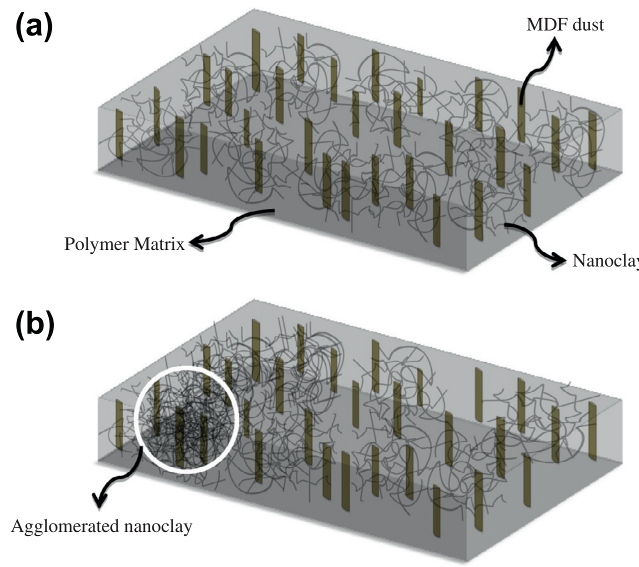

Fig. 8. Schematic representation of the morphologies of WPCs: (a) the uniform distribution of nanoclay in the matrix (sample containing 2 wt.% nanoclay) and (b) composites with 6 wt.% nanoclay content with agglomerated nanoclay particles. (Composition of WPC constituents is illusive in this schematic image).

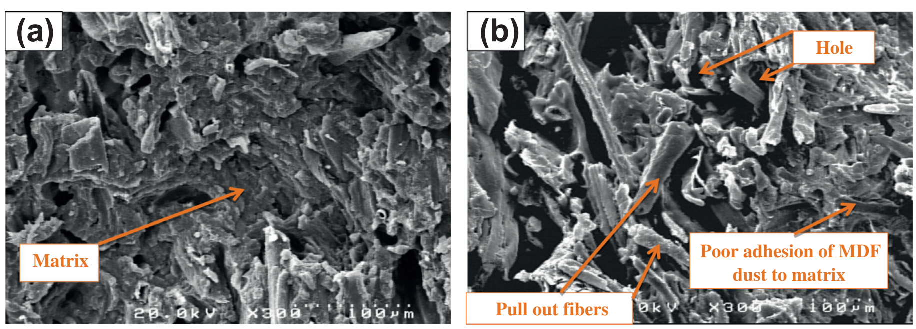

Fig. 6. FE-SEM micrographs of the composites containing 40 wt.% of MDF dust (a) and 60 wt.% of MDF dust (b) without nanoclay.

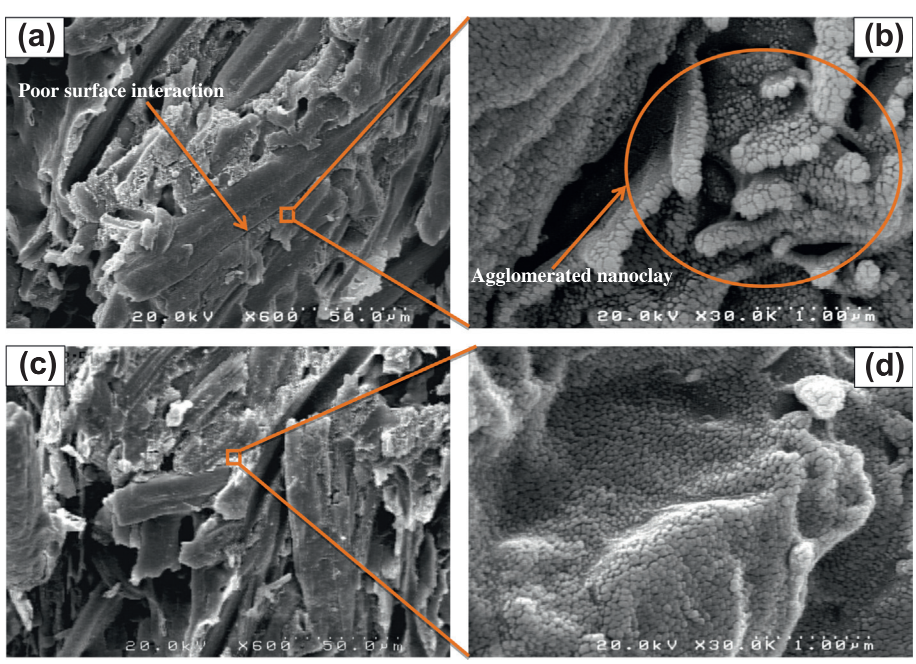

Fig. 7. SEM micrographs of the composites containing 50 wt.% of MDF dust with 6 wt.% of nanoclay (a and b) and 2 wt.% of nanoclay (c and d).

4. Conclusions

In this work, nanoclays of 2 wt.%, 4 wt.%, and 6 wt.% were filled in MDF dust/PP composites and the morphology, long-term physical properties and withdrawal strength of fasteners were evaluated before and after saturation. The results showed that the water absorption (WA) and thickness swelling (TS) can be reduced with addition of nanoclay to the MDF dust/PP composites. From the experimental results, TS has linear relationship with the WA. It was also found that withdrawal strengths of fasteners decreased and WA and TS increased with increasing amount of MDF dust. Maximum withdrawal strengths of fasteners were obtained in the samples reinforced with 2 wt.% nanoclay. The maximum decrease in withdrawal strengths of fasteners was observed in control samples (without nanoclay) in wet conditions. The addition of nanoclay slightly minimized the effect of moisture on the withdrawal strengths of fasteners.

FE-SEM observations showed that the decrement of withdrawal strength of fasteners at 4 and 6 wt.% of nanoclay was related to the agglomeration of nanoclay particles. The agglomerates of the nanoclay particles were visible in the case of composites made with 6 wt.% nanoclay. The observed FTIR indicated that in the spectrum related to composites filled with 6 wt.% nanoclay, the intensity of the hydroxyl peak was found to decrease. XRD study showed that the samples filled with 2 wt.% nanoclay toward 6 wt.% induced a greater increase in interlayer spacing and intercalated. Finally, due to the high volume production of waste sanding dusts, small dimensions and low cost, MDF dust is considered as an excellent raw material for WPC industry and can eliminate the problem of accumulating and discarding wood industries' waste.

Acknowledgments

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