EFFECT OF NANOCLAY DISPERSION ON PHYSICAL AND MECHANICAL PROPERTIES OF WOOD FLOUR/ POLYPROPYLENE/GLASS FIBER HYBRID COMPOSITES

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Polypropylene/wood flour composites having different nanoclay and glass fiber contents were fabricated by melt compounding and then injection molding. The physical and mechanical properties were evaluated. The results showed that the tensile modulus and tensile strength of the PP/wood flour composites increased with increasing glass fiber content. However, the impact strength of the PP/wood flour composites progressively decreased with increasing glass fiber content. Dimensional stability of the composites could be improved by increasing the glass fiber content. Also, results indicated that the tensile modulus and tensile strength of composites increased with increase of nanoclay up to 4phc and then decreased. However the impact strength and water absorption of the composites decreased with increasing the nanoclay loading. The morphology of the nanocomposites was examined by using X-ray diffraction (XRD). Morphological findings revealed an intercalated form in the sample with 4 per hundred compounds (phc) concentration of nanoclay, which implies the formation of an intercalated morphology and better dispersion than 6phc, and the d-spacing of clay layers were improved in the composite in the presence of compatibilizer. This project has shown that the composites treated with glass fiber and nanoclay will be desirable as building materials due to their improved stability and strength properties.

Keywords: Hybrid composite; Nanoclay; Glass Fiber; Physical Properties; Mechanical properties

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INTRODUCTION

Wood plastic composites (WPCs), which are defined as composite materials containing wood (in various forms such as wood flour and fiber, kenaf fiber, hemp, sisal, etc.) and thermoplastic materials (e.g. polyethylene, polypropylene, PVC, etc.), are a relatively new family of composite materials. Compared to the traditional synthetic fillers, the natural fibers present lower density, less abrasiveness, lower cost, and they are renewable and biodegradable. WPCs are becoming more and more commonplace and are also gaining considerable popularity in the world (Bledzki and Gassan 1999; Rowell et al. 1997).

Recently, to improve of the physical and mechanical properties of composites, some approaches such as foaming (Park and Sun 2005), and chemical treatments (Kokta

et al. 1990), and hybridization with other fillers (Rozman et al. 2001; Thwe and Liao 2002), etc., have been considered by researchers.

Nanocomposites constitute a new class of composite materials which, in at least one dimension of the dispersed particles, is in the nanometer range. Polymer nanocomposites form an emerging class of mineral filled plastics that contain relatively small amounts (usually 5 to 10%) of nanometer-sized inorganic particles. While still an embryonic segment of the industry, nanocomposites comprising either nanoclay or nanocarbon fillers are expected to be a major growth segment for the plastics industry (Galgalia et al. 2005; Mani et al. 2005; Ismail and Munusam 2007). Nanocomposite technology with layered silicate nanoclays as in situ reinforcement has been intensively investigated in recent years (Ray and Okamoto 2003; Tjong 2006; Utracki 2007; Viswanathan et al. 2006). Montmorillonite (MMT) is the most commonly used layered silicate because of its natural occurrences and beneficial properties (high cationic exchange capacity, high surface area, and large aspect ratio) (Tjong 2006; Utracki 2007). Essential improvements of physical and mechanical properties including tensile modulus and strength, flexural modulus and strength, thermal stability, fire resistance, and barrier resistance have been observed for various thermoplastic and thermoset nanocomposites at low silicate content (Koo et al. 2002; Wu et al. 2007; Lei et al. 2007; Chen 2007; Samal et al. 2008).

Barrier properties, fire resistance, and mechanical properties are of great importance for the successful application of selected wood products. Many efforts have been made in the formation of wood polymer composites (WPCs), to improve such properties and to meet specific end-use requirements. Both thermoplastic and thermosetting systems have been used and have achieved certain improvements in wood properties, but both showed limitations (Hetzer and Kee 2008). Nanomodified WPCs could be a promising new approach to obtain better products. Few attempts, however, have been made with this regard.

Hybrid composites are materials made by combining two or more different types of fibers in a common matrix. Though in principle several fibers can be incorporated into the hybrid system, a combination of only two types of fiber would be the most beneficial. By hybridization it is possible to achieve a balance between performance properties and cost of the composites, which would not be obtained with a single kind of reinforcement (Rozman et al. 2001; Thwe and Liao 2002). In other words, by careful selection of reinforcements and the processing techniques, it is possible to engineer the material to better suit the various practical requirements with economic benefits.

Glass-fiber-reinforced polymers have been widely used in the automotive and aerospace industries for their high strength and low weight properties. The reinforcement of hard ceramic particles in composites improves the performance properties of composite materials (Mishra et al. 2003; Thwe and Liao 2002). Composites have wide applications in aerospace structures due to their light weight and high strength. High damping composite materials that are useful in aerospace structure have to exhibit simultaneously good mechanical properties and high damping capacity. Researchers have reported that incorporation of glass fibers with natural fibers such as wood fibers, sisal, oil palm fibers, pineapple leaf fibers, and bamboo fibers in a thermoset as well as

thermoplastic matrix resulted in improved performance (Kalaprasad et al. 2004; Panthapulakkal and Sain 2007).

The aim of this study was to investigate the effect of nanoclay dispersion on physical and mechanical properties of wood flour/glass fiber reinforced polypropylene hybrid composites.

EXPERIMENTAL

Materials

The polymer matrix, polypropylene (PP), was supplied by Arak Petrochemical Company, Iran, in the form of homopolymer pellets with a density of 0.92g/cm^3 and a MFI of 18 g/10min. The important mechanical characteristics of the PP, namely tensile strength, flexural modulus, and notched izod impact strength are 33 MPa, 1550 MPa, and 30 J/m, respectively. The cellulosic material used as reinforcing filler in the composite was wood flour (WF) from beech tree (*Fagus orientalis* Lipseky). Beech pieces were ground with a Thomas-Wiley mill to pass through a 100-mesh screen, and then they were dried again and stored in sealed plastic bags prior to compounding. Maleic anhydride grafted polypropylene (PP-g-MA) provided by Solvay with trade name of Priex 20070 (MFI=64 g/min, grafted maleic anhydride 0.1 Wt. %) was used as coupling agent. Montmorillonite modified with a quaternary ammonium salt (trimethyl ammonium chloride) of bis-2-hydroxyethyl tallow as an organic modifier, having a cationic exchange capacity (CEC) of 90 mequiv/100 g clay, a density of 1.98 g/cc, and a d-spacing of d_{001}=18.5 nm, was obtained from Southern Clay Products Co. USA, with the trade name Cloisite 30B. The E-glass fibers (GF) used in this study were supplied by Diba Glass Fiber Co (Iran). A silane coupling agent, 3-methacryloxypropyl trimethoxysilane, was coated on the glass fiber surface. Average glass fiber original lengths were 3 mm length.

Composite Preparation

Before preparation of samples, wood flour was dried in an oven at (65 ± 2) °C for 24 hours. Then polypropylene, wood flour, glass fiber, nanoclay, and PP-g-MA were weighed and bagged according to formulations given in Table 1. According to Table 1, the mass ratio of fillers (include of wood flour and glass fiber) to polypropylene was controlled at 50/50 for all formulations. The concentration was varied from 0 to 6 per hundred compounds for nanoclay and from 0 to 15% for glass fiber, individually. The amount of coupling agent was fixed at 2% for all formulations. The mixing was carried out with a HAAKE internal mixer (SYS 9000 model, USA). First the polypropylene was fed to mixing chamber. After melting of the PP, the coupling agent and nano filler were added. At the fifth minute, the wood flour and glass fiber fed and the total mixing time was 13 min. The compounded materials were then ground using a pilot scale grinder (WIESER, WGLS 200/200 Model). The resulted granules were dried at 105 °C for 4 hours. Test specimens were prepared by injection molding (Eman machine, Iran). The specimens were stored under controlled conditions (50% relative humidity and 23 °C) for at least 40 hours prior to testing.

Table 1. Composition of the Studied Formulations

Polypropylene (Wt. %)	Wood flour (Wt. %)	Glass Fiber (Wt. %)	Nanoclay (phc)*	PP-g-MA (phc)
50	50	0	0	2
50	50	0	0	2
50	50	0	0	2
50	50	0	0	2
50	45	5	2	2
50	45	5	2	2
50	45	5	2	2
50	45	5	2	2
50	40	10	4	2
50	40	10	4	2
50	40	10	4	2
50	40	10	4	2
50	35	15	6	2
50	35	15	6	2
50	35	15	6	2
50	35	15	6	2

*Per hundred compound

Measurements

The tensile tests were carried out according to the D638-03 method, using an Instron machine (Model 1186, England), The tests were performed at crosshead speeds of 5 mm/min. A Zwick impact tester (Model 5102, Germany) was used for the Izod impact test. All the samples were notched on the center of one longitudinal side according to ASTM D256. For each treatment level, five replications were tested.

Water absorption tests were carried out according to the ASTM D-7031-04 specification. Five specimens of each formulation were selected and dried in an oven for 24 hours at 102±3 °C. The weight and thickness of dried specimens were measured to a precision of 0.001 g and 0.001 mm, respectively. The specimens were then placed in distilled water and kept at room temperature. For each measurement, specimens were removed from the water, and the surface water was wiped off using blotting paper. Weight and thicknesses of the specimens were measured after 30 days. The values of the water absorption in percentage were calculated using the following equation,

$$WA(t) = \frac{W(t) - W_0}{W_0} \times 100 \quad (1)$$

where $WA(t)$ is the water absorption at time t, W_o is the oven dried weight, and $W(t)$ is the weight of specimen at a given immersion time t.

Wide angle X-ray diffraction (XRD) analysis was carried out with a Seifert-3003 PTS (Germany) with CuKα radiation (λ=1.54 nm, 50 kV, 50 mA) at room temperature. The scanning rate was 1^o /min.

RESULTS AND DISCUSSION

Figure 1 shows the variation of the tensile modulus versus glass fiber content at different levels of nanoclay in PP/wood flour composites. As can be seen, the tensile modulus was affected by glass fiber and nanoclay content. The tensile modulus of nanocomposites increased with increase of glass fiber at different levels of nanoclay. It is well established that comparatively different improvements in the wood flour/glass fiber PP composites may be attributed to the processing technique and the glass fiber form used.

These results indicate that the effect of hybridization cannot be exploited completely unless the breakage of glass fiber is minimized by modification in the processing techniques. An increase in the strength of wood flour/glass composite as a result of hybridization is expected, as the glass fiber is stronger and stiffer than natural fiber, as reported by other researchers (Mishra et al. 2003; Thwe and Liao 2002; Kalaprasad et al. 2004; Panthapulakkal and Sain 2007).

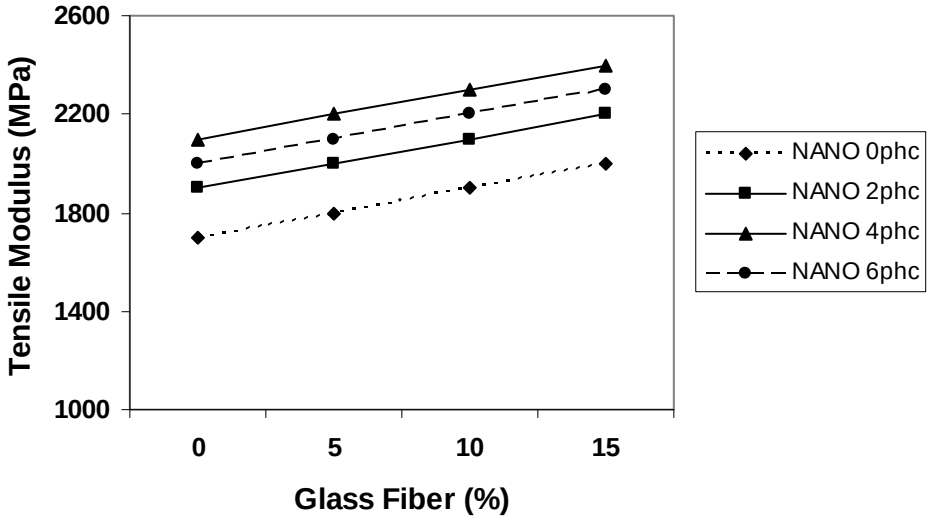

Figure 1. Tensile modulus of samples versus glass fiber content at different nanoclay levels

Also Fig. 1 shows that the tensile modulus increased with increase of nanoclay up to 4phc (per hundred compounds) at the same concentration of glass fiber and then decreased. It is well known that the nanoclay particles with very high aspect ratio can improve the tensile modulus of the polypropylene (Boukerrou et al. 2007; Chen et al. 2007; Li et al. 2007; Han et al. 2008). The increment of the modulus depends on the morphology of nanocomposites (Koo et al. 2002; Mohanty and Nayak SK 2007; Samal et al. 2008).

The reinforcing efficiency of the nanofiller is balanced by two opposite phenomena. A negative effect is attributed to migration of nanoparticles into the wood-plastic interface, causing decreased performance. At 6 phc of nanoclay, agglomeration of nanoparticles could decrease the reinforcement of clay. Dispersion of nanoclay, as a positive effect, could enhance the modulus; therefore it can be concluded that at a level of

4phc of nanoclay in the hybrid composite, the former phenomenon was dominant and the tensile modulus increased. It seems that a fully exfoliated morphology can be obtained using higher content of maleic anhydride polypropylene (MAPP). In our research in the absence of MAPP it was not possible to achieve an exfoliated morphology. It is well known that the highest tensile modulus is attributed to an exfoliated morphology in polymeric nanocomposites (Wang et al. 2001; Lei et al. 2007; Wu et al. 2007; Samal et al. 2008).

The hybrid effect of glass fiber and nanoclay on the tensile strength of wood flour/PP composite is shown in Fig. 2. The variation in tensile strength of the composite is similar to tensile modulus. A maximum tensile strength was observed at 15% glass fiber and 4phc of nanoclay content.

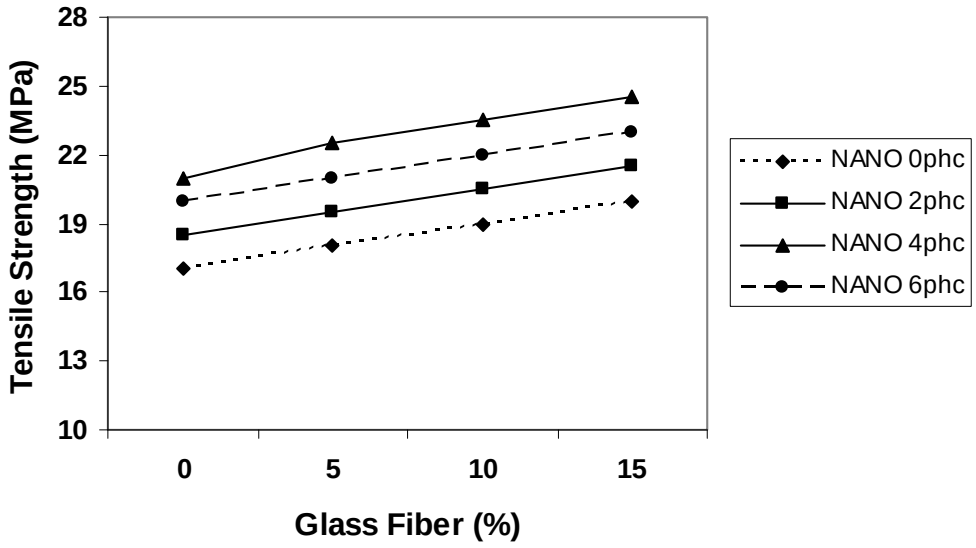

Figure 2. Tensile strength of samples versus glass fiber content at different nanoclay levels

Figure 3 shows the variation of the impact strength versus glass fiber content at different levels of nanoclay in PP/wood flour composites. As can be seen, the impact strength was affected by glass fiber and nanoclay content. The impact strength of nanocomposites decreased with increase of glass fiber at different levels of nanoclay. This could have been due to the effect of brittle glass fiber, which resulted in a lower resistance to fracturing. This shows that the glass fiber content significantly affected the impact properties.

Also Fig. 3 shows significant reduction in impact strength at 6 phc nanoclay loading. The decrease in impact strength at higher clay content levels is probably due to the formation of clay agglomerates and the presence of un-exfoliated aggregates and voids (Zhao et al. 2006; Yuan and Misra 2006). So, we found that the composites with 4 phc nancolay compared with those composites with 2 phc and 6 phc nanoclay had higher impact strength. This was attributed to a greater extent of intercalation and better dispersion of clay.

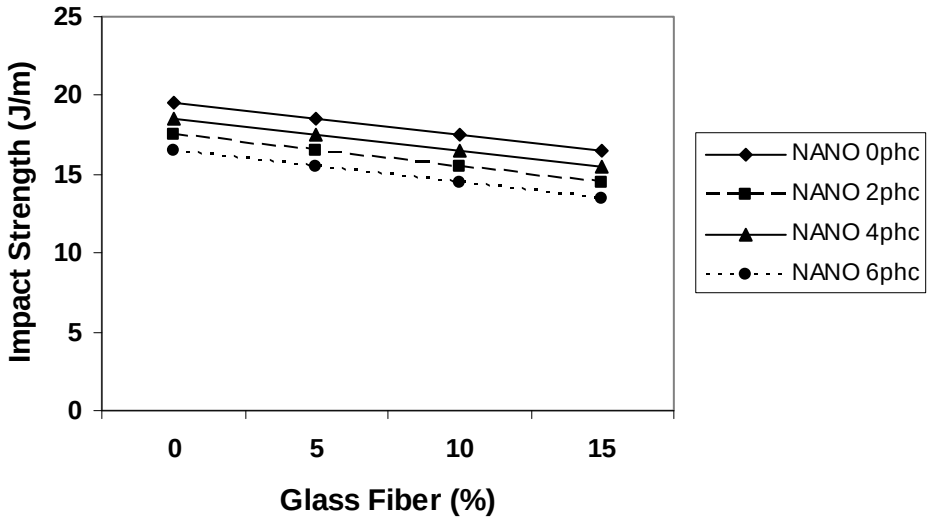

Figure 3. Impact strength of samples versus glass fiber content at different nanoclay levels

Figure 4 shows the variation of the water absorption versus glass fiber content at different levels of nanoclay in PP/wood flour composites. As can be seen, the water absorption was affected by glass fiber and nanoclay content. The water absorption of nanocomposites decreased with increase of glass fiber at different levels of nanoclay. Incorporation of glass fiber in the wood flour/PP composites decreased the water absorption significantly, which is attributed to the removal of hydrophilic natural fiber with the glass fiber in the composite. With the increase in the glass fiber content, there are less water residence sites and therefore less water is absorbed. On the other hand, the composites made from higher glass fiber content had less water absorption sites and thus lower water absorption.

Also as shown in Fig. 4, the water absorption decreased with increase of nanoclay loading at the same concentration of glass fiber. It seems that the barrier properties of nanoclay fillers inhibit the water permeation in the polymer matrix. Two mechanisms have been reported in order to account for this phenomenon.

- The first is based on the hydrophilic nature of the clay surface that tends to immobilize some of the moisture (Rana et al. 2005).
- The second involves the ability of surfactant-covered clay platelets form a tortuous path for water transport (Alexandre et al. 2006). This barrier property hinders water from going into the inner part of the nanocomposite.

It seems that both of the aforesaid mechanisms could be more efficient when the morphology is exfoliated. In other words, in an exfoliated state there is more available surface area of organoclay (with hydrophilic nature) when using a surfactant (providing a more tortuous path), so the water transport goes down under the severe conditions. Another reason for less water absorption could be the change in crystallinity of WPCs by the effect of the nanoclay as a nucleating agent (Wu et al. 2007; Hetzer and Kee 2008).

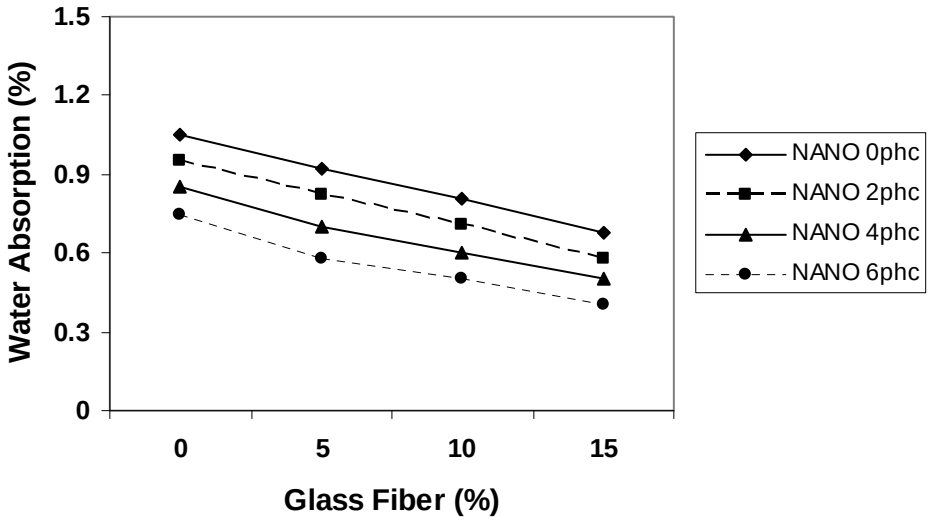

Figure 4. Water Absorption of samples versus glass fiber content at different nanoclay levels

Characterization of the morphological state of the composites was accomplished using X-ray diffraction. To verify a homogeneous dispersion of nanoclay (so-called intercalation and exfoliation) in a polymer matrix, the interlayer spacing in nanoclay (Bragg's law) and the relative intercalation (*RI*) of the polymer in nanoclay were quantified using the following equations,

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

$$RI = [(d - d_o) \div d_o] \times 100 \quad (2)$$

where n is the integer number of wavelength (n = 1), λ is the wavelength of the X-rays, d is the interlayer or d-spacing of the clay in the nanocomposite, θ is half of the angle of diffraction, and d_o is the spacing of the clay layers in the pristine clay.

The d-spacing and relative intercalation of the clay in the nanocomposites calculated from equations (1) and (2) is listed in Table 2. This table shows that the extent of intercalation of samples increased with increase of nanoclay content up to 4phc and then decreased. The peaks appearing at 4.77^0 correspond to powdered nanoclay with d_{001} = 18.5 nm. In the sample with the addition of 2 phc nanoclay, the peak was shifted to a lower angle ($2\theta = 3.98^0$, d_{001} = 22.18 nm), which implies formation of an intercalated morphology. The increase of the interlayer distance and relative intercalation might result from the stronger shear during processing when wood flour was introduced. These data show that the extent of intercalation was higher for 4 phc of nanocaly ($2\theta = 3.39^0$, d_{001}= 26.06 nm). Also, the clay was not exfoliated, since the peak still obviously existed. In other words, formation of an intercalated morphology and better dispersion was shown in 4phc of nanoclay, because the peak of that was shifted to a lower angle. It seems that this is because of the limited value of coupling agent in the nanocomposites. It is well known, through the improvement of the compatibility between neat polypropylene and

clay (using MA), the polymer chains could be well diffused into the clay layers and the basal spacing of clay layers might be increased (Koo et al. 2002; Lei et al. 2007; Samal et al. 2008). In the case of polymers containing polar functional groups, alkyl ammonium surfactant modified nanoclay is adequate to promote nanocomposite formation. However, in the case of polypropylene, it is frequently necessary to use a coupling agent, such as maleic anhydride polypropylene (MAPP) (Koo et al. 2002; Lei et al. 2007; Samal et al. 2008).

Table 2. Interlayer Spacing and Relative Intercalation in the Wood Flour Filled Polypropylene Nanocomposites

Samples	2θ (^{o})	d-spacing (nm)	Relative Intercalation (%)
Pure nanoclay	4.77	18.5	-
Composite with 2 phc nanoclay	3.98	22.18	19.89
Composite with 4 phc nanoclay	3.39	26.06	40.86
Composite with 6 phc nanoclay	3.46	25.49	37.78

CONCLUSIONS

1. The tensile modulus and tensile strength of the PP/wood flour composites increased with increasing glass fiber content. The impact strength of the PP/wood flour composites progressively decreased with increasing glass fiber content. Dimensional stability of the composites can be improved by increasing the glass fiber content.
2. The the tensile modulus and tensile strength of composites increased with increase of nanoclay up to 4phc and then decreased. However the impact strength and water absorption of the composites decreased with increasing the nanoclay loading.
3. The X-ray diffraction (XRD) patterns showed that the extent of intercalation was higher for 4phc of nanoclay than 6phc of nanoclay concentration. Also, the clay dispersion can be improved in the PP matrix in the presence of compatibilizer.
4. It seems, with increase of compatibilizer, a fully exfoliated morphology could be obtained.
5. This project has shown that the composites treated with glass fiber and nanoclay will be desirable as building materials due to their improved stability and strength properties.

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