Nanoclay's Influence on Mechanical and Thermal Properties of a Polypropylene/Poplar Wood Flour Nanocomposite

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The effects of nanoclay content were investigated *vs.* the mechanical, thermal, and morphological characteristics of a nanocomposite made from poplar wood flour and polypropylene. The wood flour, polypropylene, nanoclay, and the maleic anhydride grafted polypropylene (MAPP) were mixed in an extruder, and the test specimens were made *via* injection casting. Then, the mechanical and thermal properties were examined. The results showed that the tensile strength, flexural strength, and flexural modulus were improved when the wood flour content increased from 40% to 50%. Additionally, increasing the wood flour content from 40% to 60% enhanced the tensile modulus. The addition of nanoclay at dosages up to 2 wt% enhanced the tensile strength and the tensile modulus, whereas these properties were degraded with the addition of nanoclay at up to 4 wt%. The tensile strength and flexural modulus improved when the nanoclay content increased up to 4 wt%. The crystallinity enthalpy decreased when the wood flour content increased.

Keywords: Poplar; Nanoclay; Tensile strength; Tensile modulus

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INTRODUCTION

Wood-plastic composites are a combination of polymers and cellulosic materials (Sanadi *et al*. 2001). Wood-plastic composites are environmentally friendly and are less delicate than wood. The appearance of wood-plastic composites is very similar to wood, but with higher performance characteristics. Wood-plastic composites possess highly desirable capabilities, such as low moisture absorption, resistance against oxidation, resistance against intrusion and damage by insects, light weight, high durability, great dimensional stability, favorable physical and mechanical properties, long lifetime, and the possibility to be cut, nailed, shaved, and for various covers to be applied. In recent years, the use of the fibers from both wooden and non-wooden plants as reinforcements for thermoplastics has increased extensively (Caraschi and Leao 2002). The properties of the composites can be improved by using nanomaterials to produce new products with greater added value and higher performance (Wegner and Jones 2005). Significant improvements in the mechanical properties, including strength, rigidity, and thermal properties have been achieved with no adverse effects on the density or processing of the polymer nanocomposites (Liu *et al*. 2005). Polymers reinforced with nanoclay exhibit good resistance against fire and ultraviolet (UV) radiation (Zilg *et al*. 1999).

Wang *et al*. (2006) found that after the addition of nanoclay to the polymer matrix, the nanoclay particles were distributed much better within the matrix and as a result, the tensile modulus and the tensile strength of the composite increased due to delamination and intercalation structures. The purpose of this study was to investigate the effect of the nanoclay content on the mechanical, thermal, and morphological properties of nanocomposites made from poplar wood flour and polypropylene.

EXPERIMENTAL

Materials

Reinforcement

Poplar wood flour was procured from Aria Cellulose Co. (Tehran, Iran). To ensure a uniform particle size, only particles that passed through a 50-mesh sieve and were retained by a 60-mesh sieve were used as the reinforcement material. The wood flour was oven-dried at 100 °C ± 3 °C to a moisture content of 2%.

Polymeric materials

Polypropylene with a density of 0.952 g/cm^3 and a melt flow index (MFI) of 18 g/10 min was procured from Arak Petrochemical Co. (V30S; Tehran, Iran).

Coupling agent

Maleic anhydride was coupled with polypropylene (Karangin, Karaj, Iran). The maleic anhydride had a density of 0.065 g/cm^3, an MFI of 7 g/10 min, and a 1% coupled anhydride content.

Nanoclay

Cloisite 30B nanoclay powder was procured from Southern Clay Products, Inc. (Gonzales, TX, USA) with a density 1.98 g/cm^3 and particle shape (spherical) was ordered. It was incorporated at three different levels: 0%, 2%, and 4%. Cloisite-30B is a natural montmorillonite clay modified with a quaternary ammonium salt, having a d-spacing of 18.5 Å and modifier concentration of 90 meq/100 g clay.

Methods

Mixing

The materials were mixed in a co-rotating twin-screw extruder (COLLIN Lab & Pilot Solutions GmbH, Maitenbeth, Germany). Temperatures of 165 °C, 170 °C, 175 °C, 180 °C, and 185 °C were adjusted for the thermal regions 1, 2, 3, 4, and 5 in the extruder, respectively. The rotational speed of the screw was set to 60 rpm. The mixed, molten materials were transformed into granules in a granulator machine (WGLS 200/200; Wieser, Scheffau am Tennengebirge, Salzburg, Austria) after leaving the extruder. The temperature of the dryer was fixed at 65 °C. The granule particles obtained were prepared using an injection molding machine (Imen Machine; Aslanian Company, Tehran, Iran) at 185 °C and 3 MPa, in accordance with the ASTM standard D3641-12 (2012) to create the test samples for the flexural tests, tensile tests, and differential scanning calorimetry (DSC) analyses.

Measurement of mechanical properties

The test specimens were examined according to the ASTM standard D638-10 (2010) for the tensile properties and ASTM standard D790-10 (2010) for the flexural properties. The reported results were the average of a minimum of five samples for each treatment.

Thermal analysis

The DSC test was conducted using a DSC 131 (Setaram Instrumentation SAS, Caluire-et-Cuire, France) apparatus that was available in the Sciences and Research Branch of Azad University (Tehran, Iran). Samples of 8 g were cut from the wood-plastic samples and kept in a small aluminum container before being transferred into the DSC apparatus. The samples were heated at a rate of 10 °C/min. They were initially heated from 25 °C to 200 °C, followed by cooling from 200 °C to 25 °C. The data obtained from the first cooling and the second heating were used for further analysis of the results.

Statistical analysis

The results of the mechanical tests were analyzed using a randomized statistical plan under a factorial test using SPSS software (IBM Corporation, version 11.5, Armonk, NY, USA). The *post hoc* Duncan test was used at a 95% reliability level for comparison of the averages.

RESULTS AND DISCUSSION

The poplar wood flour content was investigated at three different levels: 0%, 2%, and 4%. The F-values and significance values are listed in Table 1.

Table 1. Variance Analysis Results (F-values and Significance Values) of the Nanocomposites Made from Poplar Wood Flour/Polypropylene and Nanoclay

Variable Properties	Tensile Strength	Tensile Modulus	Flexural Strength	Flexural Modulus
Poplar Wood Flour Content	26.681*	14.522*	18.838*	160.713*
Nanoclay	27.327*	2.673 ^{ns}	3.919*	89.576*
Poplar Wood Flour Content × Nanoclay	3.666*	3.085*	4.587*	49.993*

* Significance level = 95%, ns = non-significant

Figures 1 through 4 depict the effects of the wood flour and nanoclay contents on the mechanical properties of the nanocomposites.

The effect of the wood flour content on the tensile and flexural strength properties and moduli were significant at the 5% level. The effect of the nanoclay on the tensile strength, tensile moduli, and flexural moduli were significant at the 5% level, but the effect on the flexural strength was not significant at the 5% level. The mutual effect of the flour and nanoclay contents on the tensile and flexural strengths and moduli were significant at the 5% level.

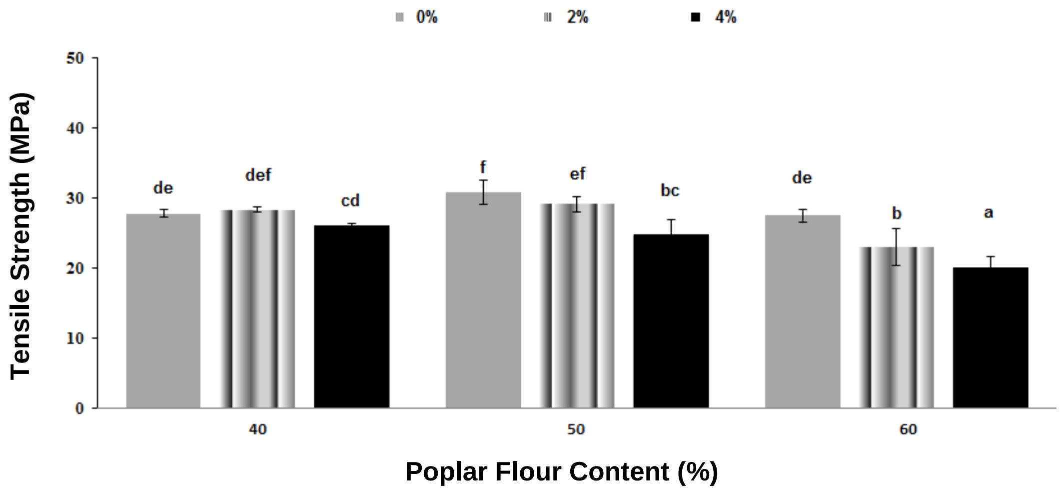

Fig. 1. Effect of the amount of nanoclay and poplar wood flour content on the tensile strength

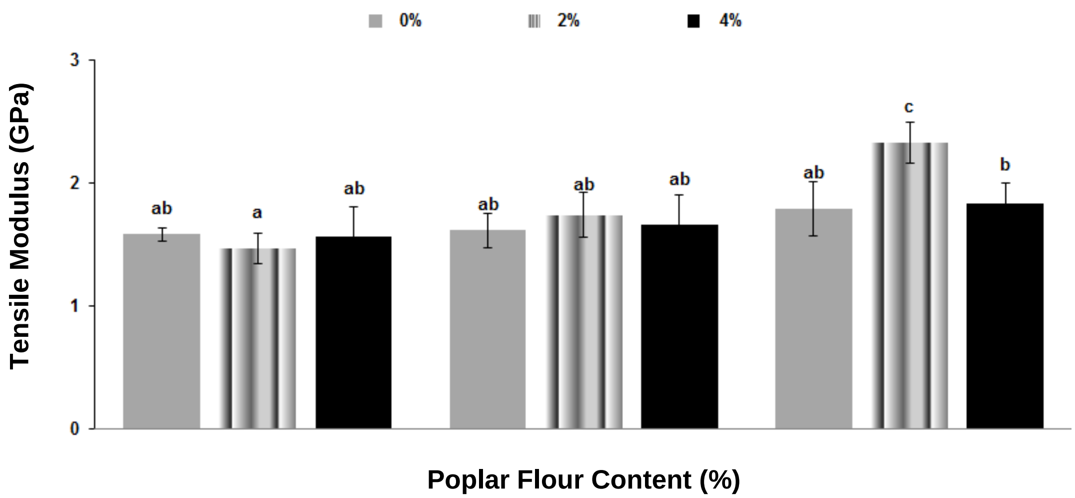

Fig. 2. Effect of the amount of nanoclay and poplar wood flour content on the tensile modulus

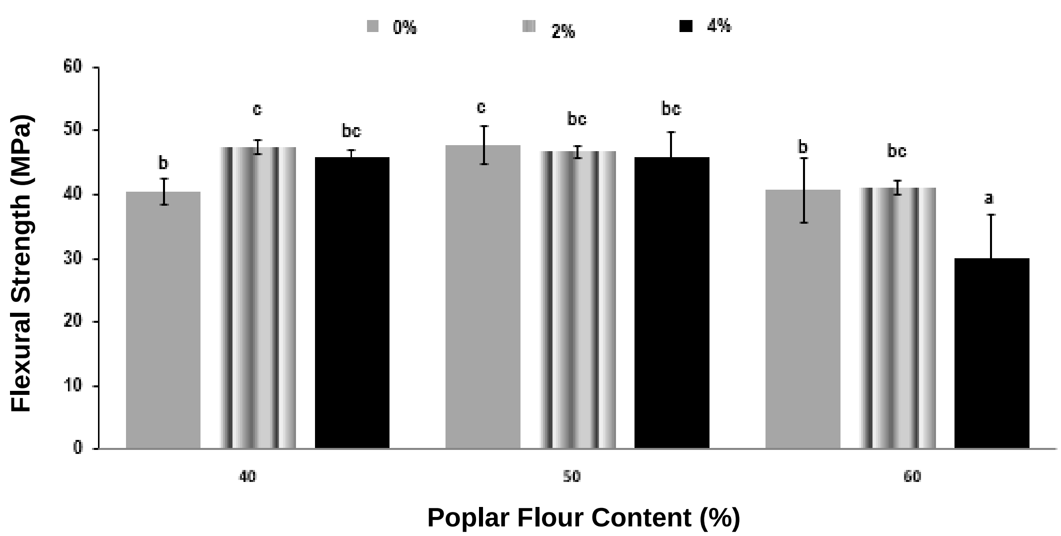

Fig. 3. Effect of the amount of nanoclay and poplar wood flour content on the flexural strength

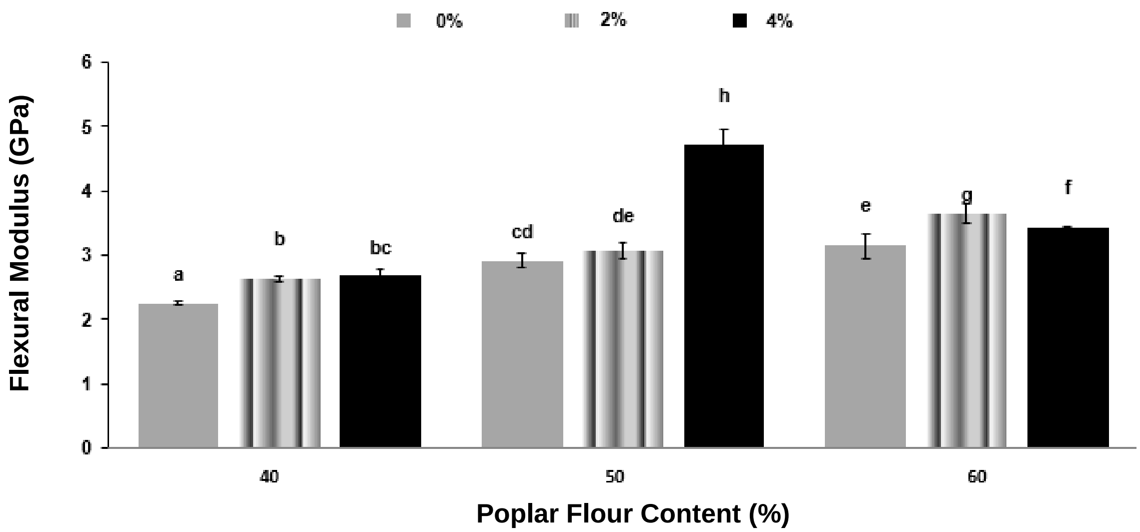

Fig. 4. Effect of the amount of nanoclay and poplar wood flour content on the flexural modulus

Effect of the Wood Flour Content on the Tensile and Flexural Strengths

The tensile and flexural strengths are mainly dependent upon the matrix characteristics and the adhesion, interface quality, and connection surface between the two phases of the composite material. The matrix phase is responsible for retaining the fibers and for load transmission to the reinforcement phase, while the principal role of the reinforcement phase is the physical and mechanical reinforcement of the matrix material (Habibi *et al*. 2017). Therefore, increasing the reinforcement content will increase the level of stress that can be tolerated by the composite material (George *et al*. 2001). Thus, increasing the wood flour content from 40% to 50% increased the tensile and flexural strengths, which was consistent with the results of Kim *et al.* (2004) and Cui *et al.* (2008). During the tensile stress test, the load transmission was developed between the matrix and filler particles in the form of shear at the interface between the matrix and the filler materials; the interface must be able to effectively perform this load transmission (Švab *et al*. 2005). Increasing the wood flour content to 60% reduced the stress transmitted from the matrix to the fibers because it decreased the compatibility between the surface properties of the polar fibers and the nonpolar polypropylene. In this case, the polymer matrix was not capable of completely covering the fibers, which decreased the tensile strength and slightly reduced the flexural strength. The same results were also reported by Nourbakhsh *et al*. (2008), Nourbakhsh and Ashori (2009), and Basiji *et al.* (2010).

Effect of the Wood Flour Content on the Tensile and Flexural Moduli

The tensile and flexural moduli of the composites are affected by the moduli of its components. Using natural fibers in the matrix increases these moduli (Clemons 2002; Febrianto *et al*. 2006; Razavi-Nouri *et al*. 2006). Because cellulosic materials have relatively large tensile moduli, they improve the tensile modulus of the composites into which they are incorporated (Oksman and Clemons 1998). Therefore, increasing the wood flour content from 40% to 60% enhanced the tensile modulus of the composites. In fact, one of the main reasons to add lignocellulosic materials to plastics is to reinforce them and increase their tensile modulus. The results of this research agreed with those reported by Kim *et al*. (2004).

Effect of the Nanoclay Content on the Tensile Strength

When the nanoclay content was increased to 4 wt%, the tensile strength of the composite was reduced. Increasing the nanoclay content likely reduced the composite strength because of the accumulation and aggregation of nanoclay particles and the formation of intercalation colonies (Golebiewski and Galeski 2007). Another reason for the cited reduction in the tensile strength with the increased nanoclay content can be associated with the absorption characteristics of the coupling agent by the nanoclay (Yeh and Gupta 2010). The coupling agent acts as a connecting bridge between the polymer and the lignocellulosic material. By improving the adhesion between them, the coupling agent improves the mechanical strength (Mustapa *et al*. 2005). When the nanoclay content is increased, more nanoclay absorbs to the coupling agent and inhibits connection of the coupling agent to the lignocellulosic particles, which negatively affects the tensile strength (Yeh and Gupta 2010).

Effect of the Nanoclay Content on the Tensile Modulus and Flexural Strength

The effect of the adding nanoparticles on the mechanical properties of the polymer nanocomposites depended on several factors, such as the size, shape, type, apparent coefficient, crystallographic structure, content, diffraction, and quality of the nanoclay particles, as well as how they are connected to the polymer (Jahromi *et al*. 2010). Therefore, the increase in the tensile modulus and the flexural strength of the composite achieved with 2 wt% nanoclay was attributed to the significant apparent coefficient of the nanoclay particles. The large coefficient of the nanoclay increased the interface between the two phases. The improved strength may have been attributable to the increased nanoclay content and the existence of the intercalation structure in the nanocomposite, which was due to an interfacial effect between the organic chains and the nanoclay particles, as well as the direction of the layered silicate particles. Moreover, the nanoclay particles increased the interface between the two phases by acting as a reinforcement (Wu *et al*. 2007). In contrast, adding more nanoclay, the swelling of the clay layers, and the creation of strong surface adhesion between the polymer and the clay also promoted flexural strength (Asif *et al*. 2007). These results were consistent with the results of Lei *et al*. (2007), Danesh *et al*. (2012), Kord (2012), and Tasooji *et al*. (2012). However, increasing the nanoclay content to 4 wt% would likely reduce the tensile modulus and the strength due to the accumulation and agglomeration of the nanoclay particles and the formation of intercalation colonies. Another reason for the reduction in the tensile modulus and flexural strength with the increased nanoclay content was attributed to the absorption characteristics of the coupling agent by the nanoclay (Yeh and Gupta 2010). When the nanoclay content increased, the nanoclay absorbed a greater amount of the coupling agent toward itself and prevented it from connecting to the lignocellulosic particles. This degrades the tensile modulus and flexural strength (Yeh and Gupta 2010). The results of this part of this study were consistent with those reported by Lei *et al*. (2007), Tabari *et al*. (2011), and Nourbakhsh (2012).

Effect of Nanoclay Content on Flexural Modulus

The flexural modulus have increased because of the relatively high apparent coefficient of the nanoclay, the development of an intercalation structure in the nanocomposite, and the establishment of strong connections of the nanoclay with the polymer matrix (Kord 2010). Increasing the nanoclay content to 4 wt% increased the flexural modulus, which agreed with the reports of Wu *et al*. (2007), Han *et al*. (2008), and Tasooji *et al*. (2012).

The DSC Test

The results of the DSC test are summarized in Table 2.

Table 2. Results of the DSC Test

Treatment No.	Treatment Code	Crystallinity Enthalpy (J/g)	Crystallinity Point (°C)	Melting Point (°C)
1	PP 100	84.01	114.81	167.68
2	40% WF 60% PP 2% M	59.35	119.76	166.32
3	40% WF 60% PP 2% M 2% NC	49.97	122.02	167.26
4	40% WF 60% PP 2% M 4% NC	40.86	121.02	168.94
5	50% WF 50% PP 2 %M	41.68	122.59	167.64
6	50% WF 50% PP 2% M 2% NC	41.16	122.60	166.36
7	50% WF 50% PP 2% M 4% NC	42.33	121.59	168.47
8	60% WF 40% PP 2% M	36.73	120.49	166.94
9	60% WF 40% PP 2% M 2% NC	31.95	121.06	166.19
10	60% WF 40% PP 2% M 4% NC	32.19	120.78	167.25

The results of the DSC analysis revealed that there was no significant difference between the melting points of the various samples.

The enthalpy of the nanoclay decreased with increased nanoclay content, except for the treatment that incorporated 50% wood flour, 50% polymer, 2% coupling agent, and 4% nanoclay. When the wood flour content was increased, the crystallinity enthalpy decreased. The polar interactions and integration of the chains due to networking tended to reduce the movability of the chains. These two groups created distance between the chains and, as a result, decreased the movability, reduced the degree of crystallization, reduced the crystallization enthalpy, and reduced the melting enthalpy. The addition of 60% wood flour reduced the crystallization enthalpy. The composite containing wood flour crystallized later than the pure polymer during cooling. Generally, the crystallization enthalpy decreased due to the negative effect of the wood flour in the composite and the inhibition of the formation of the intermolecular bindings. Increasing the amount of wood flour is believed to have increased the crystallization degree because of the role that wood particles hold as nucleation sites. As discussed before, there was no sensible change in the melting point. In their study on the thermal properties of a wood-plastic composite obtained from wood flour and heavy polypropylene, Shurabi and Golzar (2010) reported that increasing the wood enthalpy reduced the crystallinity enthalpy. Zang *et al*. (2011) declared that wood particles are like heterogeneous, crystalline cores that can cause the polymer to be connected to more fibers and melt much sooner. The crystalline shape is formed faster during the cooling period.

CONCLUSIONS

This research studied the effect of nanoclay and wood flour on the mechanical, thermal, and morphological properties of a nanocomposite made of poplar wood flour and polypropylene and the following results were obtained:

1. When the wood flour content has been increased from 40% to 50%, the tensile strength, the flexural strength, and the flexural modulus improved. These parameters decreased when the wood flour content was further increased to 60%.
2. When the wood flour content has been enhanced from 40% to 60%, the tensile modulus increased.
3. When the nanoclay content has been increased to 2 wt%, the flexural strength and the tensile modulus increased. Further addition to 4 wt% reduced these properties. However, when the nanoclay content was increased to 4 wt%, the tensile strength and the flexural modulus degraded.
4. According to the results of the present study, the optimum composition of poplar wood flour, polypropylene and nanoclay composite in terms of mechanical strength is 50% poplar wood flour and 2% nanoclay.
5. There was no significant difference between the melting points of the different samples. Increasing the wood flour content reduced the enthalpy of crystallization and increasing the amount of nanoclay had the same effect.

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