Mechanical, Flammability, and Morphological Properties of Nano-composite Plastic based on Hardwood Flour High-density Polyethylene Embedding by Nano-Zinc Oxide

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The influence of the nano-zinc oxide amount was evaluated relative to the flammability behavior, as well as the morphological and mechanical properties of wood plastic composites (WPCs). The polymer amount was approximately 50 wt%, and the lignocellulose material was 50 wt%. Nano-zinc oxide was applied at six weight levels including, zero (control), 1, 2, 3, 4, and 5 phc. For all treatments, the maleic anhydride polyethylene (MAPE) amount was 2 phc. The WPCs were made using a mixture of nano zinc oxide, high-density polyethylene, and mixed hardwood flour injection molding method. The morphological and mechanical properties, such as flexural strength and modulus, were measured. Various tests were conducted with a cone calorimeter, including the amount of char residue, total smoke production, time to ignition, and heat release rate, according to ASTM E1354-92 (1992). The flexural strength and modulus of composites in samples with 5 phc nano-zinc oxide were 79.9% and 27.2% greater, respectively, than in samples without nano zinc oxide. Nano-zinc oxide enhancement to 5 phc increased the ignition time and char residue 105.1% and 121.7%, respectively, and decreased the burning rate and total amount of smoke production 20.3% and 46.0%, respectively. Scanning electron microscope results indicated the presence of nano-zinc oxide agglomerates in the sample.

Keywords: Nano zinc oxide; Flammability behavior; Flexural strength; Flexural modulus

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INTRODUCTION

The mechanical properties of polymer materials are often modified using fillers. Recently, boosted polymer composites have drawn attention because of the introduction of nanotechnology in material sciences. Nanocomposites have been used to create a new type of polymer composite in which nanoparticles are used in their structures. In recent years, nano oxide particles with specific features have been used in many applications in various fields, such as in the preparation of nano polymer composites (Zan *et al*. 2004; Rong *et al*. 2005; Zhao and Li 2006).

Nano-zinc oxide is a mineral nanoparticle that has interesting chemical and physical features and has recently gained researchers' attention. Some of the specific features of nano-zinc oxide are its high chemical stability, large specific surface area, high surface energy, low dielectric constant, high catalyst activity, absorption of infrared and ultraviolet lights, and antibacterial activity against fungi (Kartal *et al*. 2009; Clausen *et al*. 2009, 2011; Weichelt *et al*. 2010; Li *et al*. 2010; Rajendra *et al*. 2010; Jafarzadeh *et al*. 2017). Therefore, nano-zinc oxide can potentially be used as insulation for UV rays (Ammala *et al*. 2002; Chae and Kim 2005; Yang *et al*. 2005; Zhao and Li 2006; Hong *et al*. 2007, 2009; Chen *et al*. 2011). The preparation of polymer nanocomposites using nano-zinc oxide can improve the optical and mechanical specifications of the polymer because there is a strong interaction between the organic polymer and mineral nanoparticles (Chae and Kim 2005; Hong *et al*. 2007, 2009). The composition, morphology, crystallinity, size, and shape of nano-zinc oxide have important effects relative to their inherent features (Lin *et al*. 2009; Jafarzadeh *et al*. 2017).

Sahraeian (2004) compared the flammability of three polymer groups (thermoplastics, thermosets, and elastomers) along with their nanocomposites containing 5% modified clay. The results showed a reduction in flammability for all polymer-clay nanocomposites, wherein the effect of nanoclay particles was significant for the reduction of flammability and improvement in the thermal stability of thermoplastic polymers. Guo *et al*. (2007) considered the effect of nanoclay particles on the flammability behavior of composites consisting of wood fibers and high-density polyethylene and found that the enhancement of nanoclay particles reduced time to ignition by 18%. Kord (2012) considered the effect of nanoclay particles on the physical and flammability characteristics of high-density polyethylene-wood flour composites and claimed that the addition of nanoclay particles caused reductions in water absorption, thickness swelling, burning rate, heat release rate, and total smoke production of the wood polymer composite. Both char residue and ignition time of nanocomposites increased. Nemati *et al*. (2016) studied the effect of nanoclay on the flammability and morphological features of nanocomposites made from recycled polystyrene and wood flour and reported that the addition of nanoclay reduced the flammability.

Although some studies have examined the effects of zinc oxide nanoparticles on the performance characteristics of WPCs, few studies have dealt with the possibility of concurrently determining the mechanical properties as well as flammability of the composites. The aim of the present study was to evaluate the role of nano-zinc oxide on the mechanical, flammability, and morphological properties of composites based on wood flour/ high-density polyethylene.

EXPERIMENTAL

Materials

Wood flour

A total of 150 µm of mixed hardwood flour, used as filler, was prepared by the Aria Cellulose Company, Tehran, Iran. It was dried for 24 h at 100 °C ± 3 °C.

Polyethylene

The H500 high-density polyethylene (HDPE) was produced (Bandar Imam Petrochemical Company, Mahshar, Iran) with a melt flow index of 23 g/10 min and a density of 0.965 g/cm^3.

Coupling agent

To ensure compatibility between the wood flour and the HDPE, 2 phc KJS111 maleic anhydride polyethylene (MAPE), produced by the Kimia Javid Sepahan Company (Isfahan, Iran), was used; it had a melt flow index of 35 g/10 min and a density of 0.93 g/cm^3.

Nano-zinc oxide

In this study, nano-zinc oxide, acquired from the Sigma-Aldrich Company, Saint Louis, USA, was used at five levels: 1 phc, 2 phc, 3 phc, 4 phc, and 5 phc. The particle sizes were between 10 nm and 30 nm. The nano-zinc oxide had 99% purity and a white color.

Methods

Material mixture

The amount of polymer (matrix) was approximately 50 wt%, and the hardwood flour was 50 wt%. Nano-zinc oxide was applied at five various weight levels including, 1 phc, 2 phc, 3 phc, 4 phc, and 5 phc and was used with control sample (without nano-particles). For all treatments, the maleic anhydride polyethylene (MAPE) amount was 2 phc (Table 1).

Table 1. Weight Percentage of Various Components in Wood-plastic Composites

Sample code	Wood Flour (wt%)	HDPE (wt%)	MAPE (phc*)	Nano-Zinc Oxide (phc)
50W50H2M	50	50	2	0
50W50H2M1N	50	50	2	1
50W50H2M2N	50	50	2	2
50W50H2M3N	50	50	2	3
50W50H2M4N	50	50	2	4
50W50H2M5N	50	50	2	5

W: wood flour; H: high density polyethylene; M: MAPE; N: Nano-Zinc Oxide.
(*) phc: parts per hundred compounds.

An in-line round twin-screw extruder (Model T20, GmbH Company, Ebersberg, Germany) was used to mix polymer matrix with wood flour, the coupling agent, and nano-zinc oxide.

After mixing, the output materials were converted into granules using a semi-industrial mill. The percentages shown in Table 1 were mixed using an antithetic round extruder. The extruder's temperatures were 165 °C, 170 °C, 175 °C, and 180 °C for regions 1 through 4, respectively. The spiral rotational speed was 60 rpm. The mixed and melted materials were transformed into granules using a granule producer instrument (WIESER, WGLS 200/200 model), made in Hamburg, Germany.

Sample production

The obtained granules were dried for 24 h at 85 °C. An injection molding machine (Imen Machine, Aslanian Company, Tehran, Iran) was applied for the preparation of obtained granule particles at 3 MPa pressure and 170 °C, according to the standard of ASTM D 3641-12 (2010), to prepare the samples for testing flexural strength. For flammability testing sample preparation, a mini test press from Toyoseiki Company

(Tokyo, Japan) was used. The samples were prepared at 200 °C for 4 min under 25 MPa. To prevent bubbles creation inside the sample, pressing was repeated several times. Using a puncher, the samples were separated, to prevent crack creation. The specimens were stored, before testing, under controlled conditions for at least 40 h (20 °C ± 2 °C and 65% ± 5% relative humidity).

The standard of ASTM D790-10 (2010) was applied to test the flexural modulus and strength at a speed of 2 mm/min using an Instron Universal Testing Machine (model 1186, Instron Corp., Canton, MA, USA). The specimens dimensions were 105 mm × 13 mm × 5 mm (length × width × thickness) for the flexural tests. For each treatment level, five replicates were measured for each property and the average values were reported.

Determination of flammability

The samples were prepared as sheets with dimensions of 100 mm × 100 mm × 6 mm. The combustion parameters, such as char residue (CR), total smoke production (TSP), time to ignition (TTI), and heat release rate (HRR) tests, were measured according to the ASTM E1354-92 (1992), using a cone calorimeter (FTT Company, Bridge House, UK). The tests were performed at an incident heat flux of 50 kW/m^2. In this study, the burning rate (BR) test was determined using a horizontal burning test instrument (Analytical Instruments Co., Jiangning County, Nanjing, China) (according to standard ASTM D635-98 (1998)). For each flammability treatment level, one replicates were measured for each property were reported.

Scanning electron microscope (SEM)

In this study, a model VEGA-II SEM (TESCAN Company, Brno, Czech Republic) was used to determine the nano-zinc oxide, and its distribution status in the wood plastic composite samples.

Statistical analysis

The analyses of mechanical tests were conducted in complete accidental factorial tests using SPSS statistical software (IBM Software, Armonk, New York, version 23), and a Duncan multi domains test was conducted to compare the means at a 95% assurance level.

RESULTS AND DISCUSSION

Mechanical Properties

In this study, nano-zinc oxide was used at six levels (zero (control), 1, 2, 3, 4, and 5 phc). The F-values and the significance level are shown in Table 2. The analysis of variance (ANOVA) indicated that the nano-zinc oxide level had significant effects on the strength and flexural modulus at the 95% level.

The flexural strength is shown in Fig. 1 for each level of nano-zinc oxide. According to this figure, the highest and lowest means of flexural strength were related to the 3 phc (56.5 MPa) and control samples (26.1 MPa), respectively.

The flexural modulus is shown in Fig. 2 for each level of nano-zinc oxide. According to this figure, the highest and lowest means of flexural modulus were related to the 3 phc (3003 MPa) and control samples (1975 MPa), respectively.

Table 2. Variance Analysis (F-Values and Significance Level) of the Effect of Nano-Zinc Oxide on Mechanical Properties

WPC Properties	Flexural Strength (MPa)	Flexural Modulus (MPa)
	44.157*	11.403*
*95% significance level		

The enhancements in flexural strength and modulus were attributed to well-dispersed fibers in the polymer matrix, and the enhancement of the interaction between lignocellulosic materials and the polymer matrix, which led to an enhancement in stress tolerance. This was in line with other researchers' findings (Herrera-Franco and Valadez-González 2005; Gao *et al*. 2012).

The addition of nano-zinc oxide increased flexural strength and modulus up to 3 phc, and then it decreased. The increase in flexural strength with an increased amount of nano-zinc oxide in nanocomposites was due to the effect of the organic chain and nanoparticles. At this time, the nano-zinc oxide boosting capability was relevant to surface high ratio and heterogeneity by the nano-zinc oxide volume with organic materials. Thus, as reinforcement materials, the nano-zinc oxide particles lead to the increase of interface surface between the two phases (Wu *et al.* 2007). Consequently, as an amplifier, nanoparticles improve the joining surface between two phases (Wu *et al*. 2007). Moreover, nanomaterials addition leads to a strong adherence with the polymer and improves the composite strength (Asif *et al*. 2007; Younesi Kordkheili *et al.* 2013).

Therefore, the results showed that the enhancement of nano-zinc oxide increased the flexural modulus and strength of wood plastic composites made of HDPE and wood flour. This was in line with the findings of Deka and Maji (2012). The decrease of flexural strength and flexural modulus with increasing levels of nano-zinc oxide was due to agglomeration of nano-zinc oxide, in accordance with result reported by Kord *et al*. (2011).

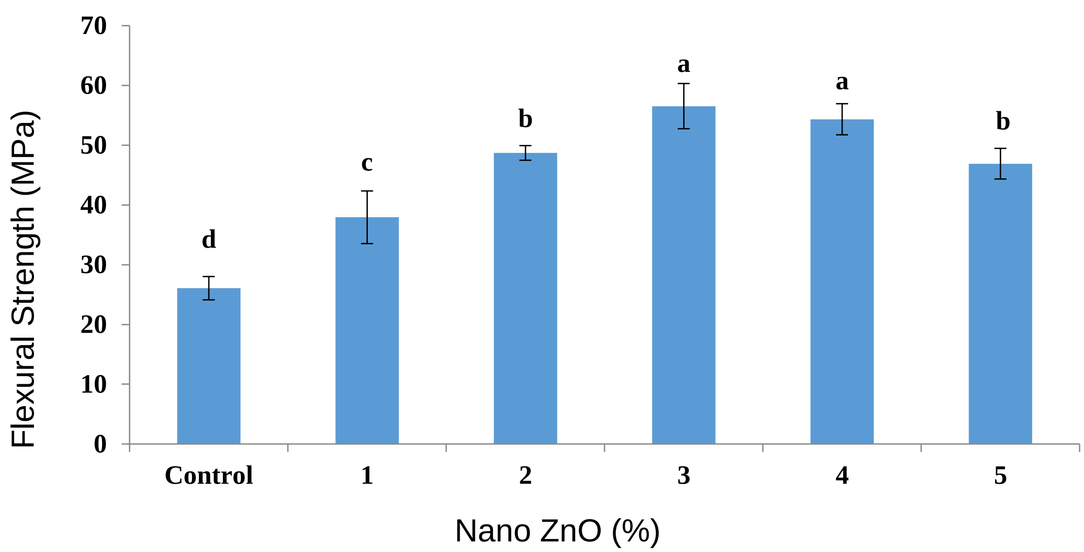


Fig. 1. The effect of nano-zinc oxide amount on flexural strength (Small letters indicate the Duncan ranking of the averages at a 95% confidence interval.)

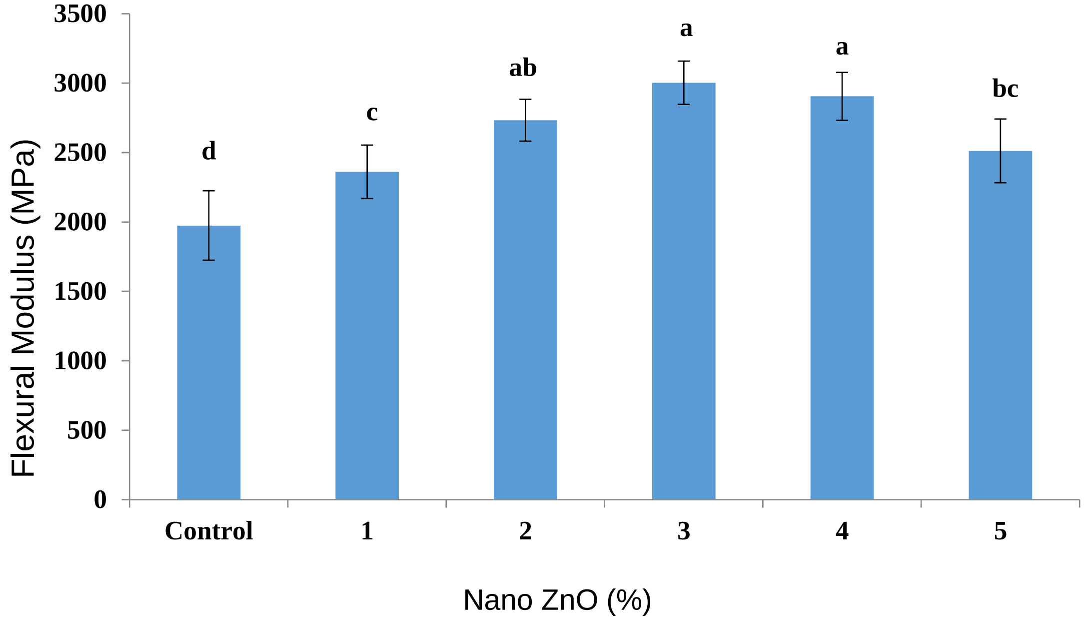


Fig. 2. The effect of nano-zinc oxide amount on flexural modulus (Small letters indicate the Duncan ranking of the averages at a 95% confidence interval.)

Flammability Behavior

The amount of char residue is shown in Table 3 for each level of nano-zinc oxide. The highest and lowest mean values of char residue were related to the 5 phc (40.8%) and control samples (18.4%), respectively. The time to ignition is shown in Table 3 for each level of nano-zinc oxide. The highest and lowest mean times to ignition were related to the 5 phc (60.1 s) and control samples (29.3 s), respectively.

The total smoke production is shown in Table 3 for each level of nano-zinc oxide. The highest and lowest mean total smoke production rates were related to the control samples (320.8 m^2/kg) and 5 phc samples (266.6 m^2/kg), respectively. The burning rate is shown in the table for each level of nano-zinc oxide. The highest and lowest mean burning rates were related to control samples (43.2 mm/min) and 5 phc samples (29.6 mm/min), respectively. The heat release rate is also shown in Table 3 for each level of nano-zinc oxide. The highest and lowest mean heat release rates were related to the control samples (83.6 kW/m^2) and 5 phc samples (51.3 kW/m^2), respectively.

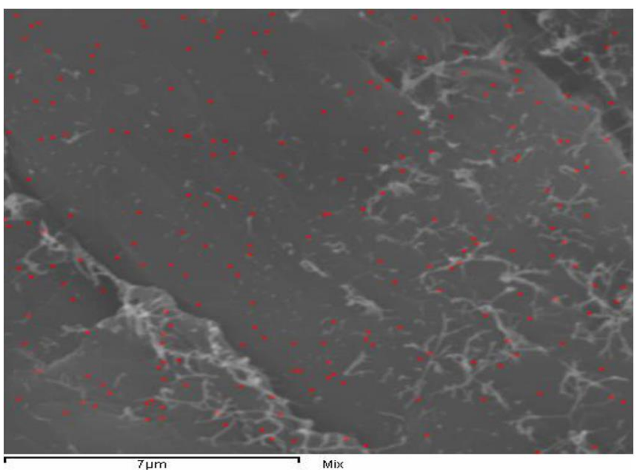
As shown in Table 3, as nano-zinc oxide increased from control samples to 5 phc in WPC nanocomposites, the amount of char residue and time to ignition increased, while the heat release rate, smoke production rate, and burning rate decreased. Seemingly, when WPC composites that contain nano-zinc oxide burned, a char layer was created on the outer layer of composites. Due to a high surface coefficient, the heat analysis temperature and heat stability increased in the composites. Enhancement of heat stability in composites postpones the entrance of oxygen into the polymer matrix (Liany *et al*. 2013). Moreover, it was expected that during the agglomeration of nanoparticles, the speed of oxygen gas emissions was delayed. However, with the enhancement of nano-zinc oxide in WPC composites, the amount of oxygen was reduced significantly (Deka and Maji 2012). These results were in line with the findings of Sahraeian (2004), Guo *et al*. (2007), and Kord (2011).

Table 3. The Effect of Nano-Zinc Oxide on Flammability Behavior

Nano-Zinc Oxide (phc)	Char Residue (%)	Time to Ignition (S)	Total Smoke Production Rate (m^2/kg)	Burning Rate (mm/min)	Heat Release Rate (Kw/m^2)
Control	18.4	29.3	320.8	43.2	83.6
1	22.7	35.4	312.5	40.3	79.1
2	26.2	39.6	300.8	37.1	71.4
3	31.3	47.2	286.4	33.8	65.6
4	37.5	53.3	273.6	31.1	58.3
5	40.8	60.1	266.6	29.6	51.3

Morphological Specifications

In order to evaluate the dispersion of ZnO nanoparticles in the WF/HDPE composites, the ultrathin sections of the samples were observed *via* SEM, as shown in Fig. 3. The dispersion of nano-zinc oxide was good in the global scale but not completely homogeneous. In general, better dispersibility occurred in the samples with lower proportion of ZnO nanoparticles (Fig. 3a).



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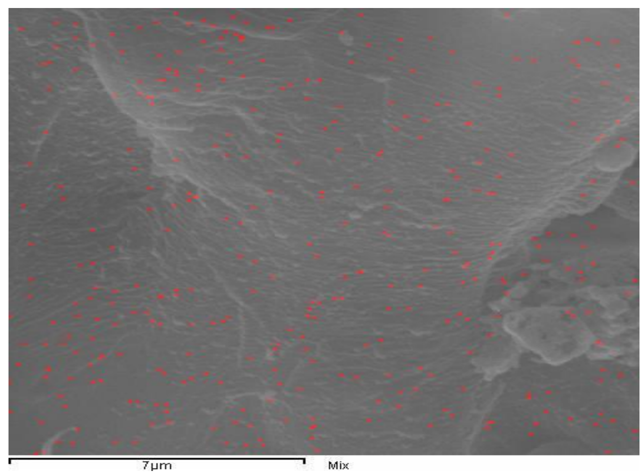

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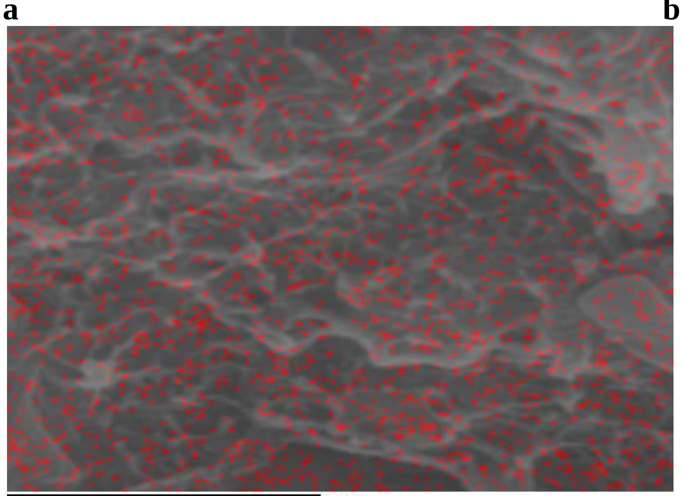

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Fig. 3. SEM of composites with (a) 1 phc, nano-zinc oxide (b) 3 phc, nano-zinc oxide and (c) 5 phc nano-zinc oxide

The good dispersion could lead to remarkable enhancements in the nanocomposite properties due to the highly interfacial adhesion. When the percentage nano-zinc oxides were increased from from 3 to 5 phc, the SEM images show that the nanoparticles had partly agglomerated (Fig. 3b and 3c).

CONCLUSIONS

The purpose of this study was to investigate the effect of nano-zinc oxide on the mechanical, flammability, and morphological properties of composites based on wood flour/ high-density polyethylene. In general, statistical results indicated that all three variable parameters had significant influence on mechanical properties of the composite samples.

1. The flexural strength and modulus of composites in samples with 5 phc nano-zinc oxide were 79.9% and 27.2% greater, respectively, than in samples without nano-zinc oxide.
2. Increasing the amount of nano-zinc oxide to 5 phc increased the char residue and time to ignition to 121.7% and 105.1%, respectively.
3. Increasing the amount of nano-zinc oxide to 5 phc decreased the heat release rate, smoke rate production, and burning rate to 63.0%, 20.3%, and 46.0%, respectively.
4. The SEM images and EDX analysis of nano-zinc oxide samples (1 phc to 5 phc) showed their improper distribution, and the presence of nano-zinc oxide accumulations that weakened the mechanical specifications of nanocomposites.

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