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MECHANICAL AND PHYSICAL PROPERTIES OF WOOD/POLYETHYLENE COMPOSITE REINFORCED WITH TIO_2 NANOPARTICLES

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Wood/polyethylene composite
TiO_2 nanoparticles
Mechanical strengths
Thickness swelling
Contact angle

HIGHLIGHTS:

TiO_2 nanoparticles had a positive effect on the bending and tensile strength of wood plastic composites.

The effect of TiO_2 nanoparticles on the Izod impact strength of composites was not significant.

Contact angle of wood plastic composites was improved by using TiO_2 nanoparticles.

Improvement in thickness swelling of composites can be achieved by TiO_2 nanoparticles.

ABSTRACT

Wood plastic composite was fabricated using high density polyethylene and pine wood fiber. The effect of addition of TiO_2 nanoparticles at different weight fractions (0%, 1%, 3%, and 5%) on some properties of the composite was examined. The experimental composites were tested for bending strength, tensile strength, Izod impact strength, thickness swelling, and contact angle. Field emission scanning electron microscopy was also investigated to study the distribution of TiO_2 nanoparticles in the composites. The results showed that using TiO_2 nanoparticles as a reinforcing agent in wood plastic composites resulted in an increase in the tensile and bending strengths and a decrease the thickness swelling of the composites. The effect of TiO_2 nanoparticles on the Izod impact strength of composites was not significant. The results also showed that the contact angle of wood plastic composites was improved by using TiO_2 nanoparticles.

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INTRODUCTION

In recent years, the production of wood plastic composites has increased in the thermoplastic industry, and it is expected to continue to increase. The raw materials used to process wood plastic composite are mainly polyolefin thermoplastics, such as polyethylene (PE), polypropylene (PP), or polyvinylchloride (PVC), and wood flour or fibers mainly from softwood like spruce or pine (Jiang and Kamdem 2004, Selke and Wichman 2004, Kumar et al. 2011). These wood plastic composites offer several advantages, including enhancement of specific properties such as stiffness and thermal behavior, reduced price of the material, and improved recyclability compared with traditional glass fiber-reinforced plastics (Miki et al. 2014). Most wood plastic composite product applications to date, including residential deck boards, rails and balusters, window lineal, door components, boat hulls, and automotive components, have modest structural requirements. However, these composite materials do not have adequate mechanical properties for typical structural applications (Lei and Wu, 2012). The properties of wood plastic composites can be improved using nanoparticles. Polymer composites reinforced by nanoparticles have the potential to improve the physical, chemical, and mechanical properties. Improvement of the mechanical properties of nanocomposites depends on the particle size of nanomaterials, shape, volume fraction, particle size distribution, polymer matrix characteristics, and interface between the filler and the matrix. In contrast to traditional reinforcers at the microscale, the dimensions of the particles in the reinforced nanocomposites are in nanometers. An important feature of polymer nanocomposites is that the small size of the fillers results in a significant increase in the contact surface of the particles with the polymer matrix, which is very high compared to conventional composites. Even if the amount of fillers is also low, the contact level is still high. Surface-to-volume ratio of nanoparticles increases with decreasing feature size, and for feature sizes that are small enough, their properties are no longer dominated by the bulk of the material but by surface atoms (Biener et al. 2009).

Commonly, the ability of nano-sized fillers to reinforce a polymeric matrix is attributed to their large aspect ratio (the length of a particle divided by its diameter) and surface areas with abundant interfacial chemical and/or physical interactions. In addition, the percolating interphase network in composites that is induced by the surrounding interphase region of each nanoparticle might play important role in improving the properties of nanocomposites (Turku and Karki, 2014). As, nanoclay, nanosilica, *etc*. are used in wood plastic composites and they improve the properties of the composites, so that product can be used in the aerospace industries due to the widespread use of nanoparticles (Han et al. 2008).

Among the materials used to prepare nanocomposites, TiO_2 nanoparticles have the advantages of non-toxicity, chemical neutrality, corrosion resistance, high refractive index, ultraviolet filtration capacity, and high hardness (Deka and Maji, 2011). TiO_2 nanoparticles absorb ultraviolet rays (Deka and Maji 2011, Hayle and Gonfa 2014) and are antifungal and antibacterial (Filpo et al. 2013). Thus, TiO_2 nanoparticles can be used as a protective material in wood (Huang et al. 2012, Filpo et al. 2013). Wang et al. (2019) studied preparation and characterization of foamed wheat straw fiber/polypropylene composites based on modified TiO_2 nanoparticles. Mechanical testing indicated that wheat straw fiber/polypropylene treated with 4% modified nano-TiO_2 exhibited the highest flexural (29.27 MPa), tensile (14.38 MPa), and impact (4.55 KJ/m^2) strengths. Prasad et al. (2018) studied development of flax fibre reinforced epoxy composite with TiO_2 nanoparticles addition into matrix to enhance mechanical properties. They concluded that Characterization techniques such as SEM imaging can be done to know the failure details of the tensile specimen and the level of arrangement of TiO_2 nanoparticles in the matrix and fibre material for the tensile samples. Aydemir et al. (2016) also studied the influence of loading rates of TiO_2 nanoparticles on the properties of nanocomposites, and their results indicated that the thermal stability of the nanocomposites improved as the amount of TiO_2 nanoparticles increased. The results also showed that water absorption decreased and density increased as the amount of TiO_2 nanoparticles added increased. Wang et al. (2020) reported that dynamic mechanical properties, thermal conductivity, electrical conductivity and trap characteristics of epoxy resin are all adjusted after TiO_2 nanoparticles doping. All of these physical properties of epoxy/TiO_2 nanocomposite dielectric were related to the suppression of molecular motion. Rahmani et al. (2020) investigated mechanical and physical characterization of Mg-TiO_2 and Mg-ZrO_2 nanocomposites produced by hot-pressing. The results indicated that the ultimate compressive strength was increased by about 10% for 3% volume fraction of TiO_2. SEM images indicated that more agglomeration was obtained by increasing volume percentage of nanoparticles. Based on the results reported in literature, the use of TiO_2 nanoparticles as reinforcement in wood-plastic composites could be a promising approach to obtain better products. The effect of the introduction of any new reinforcement material on different aspects of the product needs to be studied. Hence, the purpose of the present work is to examine the effect of TiO_2 nanoparticles on some properties of wood/polyethylene composites. For the present work, composites containing different percentages of TiO_2 nanoparticles were prepared via a melt compounding using an internal mixer followed by hot-pressing process. Eventually, bending strength, tensile strength, Izod impact strength, thickness swelling, and contact angle of composites were evaluated. It was hypothesized that adding TiO_2 nanoparticles to wood plastic composites as reinforcement improve the some properties of composites.

MATERIAL AND METHODS

Material

The polymer used in the present study was high-density polyethylene (HDPE) with a melt flow index of 18 gram/10 min and density of 0.952 gr/cm^3 (Jam Petrochemical Company (JPC), Bushehr, Iran). TiO_2 nanoparticles were from rutile produced by US Research Nanomaterials Company (Houston, USA), and pine (*Pinus sylvestris*) wood was produced by a local factory. Maleic anhydride polyethylene (MAPE; Aria Polymer Pishgam Company, Isfahan, Iran) with a melting temperature of 190 °C was used as the coupling agent for all samples. The content of TiO_2 nanoparticles was set at four levels of 0%, 1%, 3%, and 5%.

METHODS

Preparation of wood plastic composites

For preparation of wood plastic composites, first, woods were chopped up with an industrial Flaker by a local company and subsequently ground using a laboratory mill. Wood flour was sieved, and particles were prepared with mesh size 40 to 60 to make the samples. The sieved wood flour was dried at 105 °C for 24 h. To prepare the samples with the conditions mentioned in Table 1, an internal mixer and laboratory press belonging to Iran Polymer and Petrochemical Institute were used. High-density polyethylene and maleic-anhydride-polyethylene were mixed at 180 °C and mixed at 60 rpm. TiO_2 nanoparticles were added next to the mixer. Finally, wood flour was added, and the mixing process continued until a constant torque was obtained. The mixture was pressed at 180 °C and a pressure of 20 MPa using a hot press (Toyo Seike Mini-test Press, model WCH, Japan). Finally, the samples were conditioned at a temperature of 23 °C and a relative humidity of 50% according to ASTM D618 standards prior to testing.

TABLE 1 The Formulation of Wood Plastic Composites.

Code	Wood (%)	HDPE (%)	MAPE (%)	Nano TiO_2 (%)
C	50	48	2	0
NT1	50	47	2	1
NT2	50	45	2	3
NT3	50	43	2	5

Mechanical testing of wood plastic composites

Tensile strength testing of the samples was performed with a loading rate of 5 mm/min according to ASTM D638-03 (2004). A three-point bending test was performed with the loading rate of 2 mm/min on a HOUNSFIELD machine (model H-25-KS, Redhill, England) according to ASTM D790-10 (2010). Izod impact strength tests (ASTM D256) were performed using a Zwick Testing Machine (model 5102). Notched samples were used to determine the impact strength.

Thickness swelling and contact angle testing of wood plastic composites

Thickness swelling (TS) of composites was determined after 24-h submersion in water according to ASTM D1037-99 (1999). The values of the thickness swelling in percentage were calculated using the following equation [1], where T_0 is the initial thickness of specimens, and T_t is the thickness at time t.

$$\text{Thickness swelling (\%)} = [(T_t - T_0) / T_0)] \times 100 \qquad [1]$$

To measure the contact angle of samples, a contact angle analyzer (Kruss, G10, Hamburg, Germany) was used, and distilled water solution was dripped on the surface of samples.

Field emission scanning electron microscopy

Field emission scanning electron microscopy (FESEM) was used to study the distribution of TiO_2 nanoparticles in the composite. The prepared sample was analyzed with a field emission scanning electron microscope (FESEM, Tescan Mira 3 XMU, Czech Republic), operating at an accelerating voltage of 20 kV. The specimens for FESEM observation were prepared by freeze breaking after cooling the sample in liquid nitrogen. Prior to loading the samples for FESEM analysis, the freeze broken part of the samples were sputter coated with gold with approximate thickness of 25 nm.

Variable analysis of TiO_2 nanoparticle content

The effect of the TiO_2 nanoparticles contents on the properties of the wood-plastic composites was assessed by variable analysis in factorial design, and Duncan's multiple comparison tests was used to compare the average values. SPSS software was used for statistical data analysis.

RESULTS AND DISCUSSION

Bending and tensile strengths

The variance analysis showed that the effect of TiO_2 nanoparticles on the bending and tensile strengths of composites at the 5% level was significant (Table 2). Figure 1 show the mechanical strengths of the composites with different amounts of TiO_2 nanoparticles. As shown in Figure 1, as the amount of TiO_2 nanoparticles increased from 0% to 1% and from 1% to 3%, the bending and tensile strengths of the composites increased. However, the use of 5% TiO_2 nanoparticles decreased the bending and tensile strengths of the samples compared with 3% TiO_2 nanoparticles. The highest increase of bending and tensile strengths in the composites were found to be 21.88% and 26.02% for the 3% TiO_2 nanoparticles, respectively.

TABLE 2 Statistical analysis of some properties of samples

Source	Property	df	Mean square	F	Sig.
Nano content	bending strength	3	17.366	4.772	0.034*
	Tensile strength	3	17.329	5.148	0.028*
	Izod impact strength	3	1.285	1.163	0.382^{NS}
	Thickness swelling	3	1.658	57.083	0.000*
	Contact angle	3	38.610	4.250	0.045*

*: Significant difference at the 95% level ($p \leq 0.05$), NS: Non significant difference at the 95% level

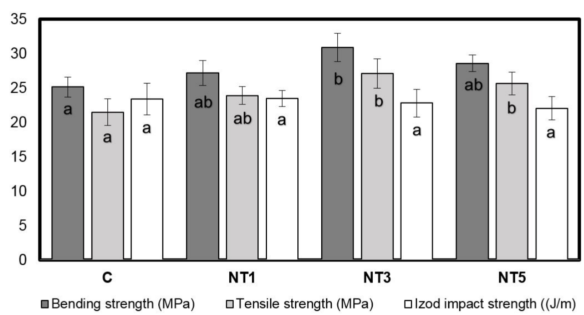

FIGURE 1 Mechanical strengths of samples: (C) Control, (NT1) 1%Nano TiO_2, (NT3) 3%Nano TiO_2, (NT5) 5%Nano TiO_2.

The increase in the bending and tensile strength of composites when using TiO_2 nanoparticles could be attributed to the high ratio of the surface to the volume of nanoparticles, which significantly increases the contact surface of the particles with the polymer matrix and improves mechanical properties. Composites made with 3% TiO_2 nanoparticles, compared with 5% TiO_2 nanoparticles, have a uniform dispersion, better interfacial interaction in composites, and more effectively transfer of stress from polymer matrix to fiber, and increase flexural and tensile strengths. Rafighi et al. (2014) investigated the mechanical properties of composite made of sawdust and high-density polyethylene, stating that the high apparent coefficient of clay nanoparticles affected the high reinforcement of the nanoparticles in the composites, increased the interface between two phases, and improved the mechanical properties. Han et al. (2008) stated that with the presence of nanoparticles in empty spaces between composite components and the formation of a more compact tissue, there is a high probability of resistance. Deka and Maji (2011) reported that the uniform distribution of TiO_2 nanoparticles and clay nanoparticles played a role in increasing the flexural and tensile properties of composites. The effect of TiO_2 nanoparticles on the some properties of nanocomposites was investigated in another study, and the results indicated that the mechanical properties of the composites, such as tensile strength, tensile modulus, flexural strength, and flexural modulus, increased as the nanoparticle loading increased (Aydemir et al. 2016). Khalid et al. (2009) observed an increase in mechanical properties of electrospun nanofibers after incorporating TiO_2 nanopowder. In still another study that was conducted to investigate the mechanical properties of nanocomposites, the mechanical properties of the composites increased when the TiO_2 nanoparticles were added (Wang et al. 2020).

At higher percentages of TiO_2 loading, the effective agglomeration of the nanoparticles resulted in a decrease in mechanical properties. As shown in Fig. 1, when nanoparticles were increased from 3% to 5%, the bending and tensile strengths of the composites were reduced. Because of the high surface energy, nanoparticles have a high tendency for agglomeration, so it is difficult to achieve uniform distribution in polyolefins (Rong et al. 2006). Nanoparticles exhibit better distribution in lower concentrations than in high concentrations, and in high concentrations, due to their higher agglomeration, they reduce the mechanical properties of composites (Kord et al. 2011). Therefore, the reduction of mechanical properties after adding 5% TiO_2 nanoparticles can be attributed to the peeling of nanoparticles of titanium dioxide, which, by decreasing the common surface in the matrix and the concentration of stress in the scraped regions, reduced the flexural and tensile strengths. Similar results were reported in previous studies (Ashori and Nourbakhsh 2011; Deka and Maji 2011). FESEM study supported the agglomeration of dioxide titanium nanoparticles at higher percentage (5%).

Izod impact strength

The variance analysis showed that the effect of TiO_2 nanoparticles on the Izod impact strength of composites at the 5% level wasn't significant (Table 2). Figure 1 show the impact strength of the composites with different amounts of TiO_2 nanoparticles. It seems that the impact strength with the addition of TiO_2 nanoparticles is decreased because of the increased brittleness of the HDPE matrix. Aydemir et al. (2016) stated that the tensile strength and elastic modulus of the nano composites increased with the addition of the TiO_2, but no improvement was observed in the impact strength due to the reduction of toughness.

Contact angle

The contact angle (CA) method is used to determine wettability of wood plastic composites (Gupta et al. 2007; Jarusombuti and Ayrilmis 2011). The variance analysis showed that the effect of TiO_2 nanoparticles on the contact angle of composites had a significant difference of 5% (Table 2). Figure 2 shows contact angle values of composites with and without TiO_2 nanoparticles. It is evident that the addition of TiO_2 nanoparticles increased the wood plastic composite's contact angle. Among the composites treated with TiO_2 nanoparticles, the sample with code NT3 had larger contact angles, while the control sample had lower values, meaning that NT3 sample surface is more hydrophobic than the control sample. The highest increase of contact angle in the composite was found to be 9.48% for the 3% TiO_2 nanoparticles. In general, the wettability of wood plastic composites was determined not only by the surface hydrophilic wood fiber loading, but also by the interfacial adhesion of the composites (Lu and Wu 2005). Therefore, samples include of TiO_2 nanoparticles showed better interfacial bonding than the control sample. The effect of increasing the interfacial adhesion between components, besides causing an increase in the tensile strength of the nanocomposites, offering more resistance to the surface contact of the composites made with TiO_2 nanoparticles.

Thickness swelling

The variance analysis showed that the effect of TiO_2 nanoparticles on thickness swelling of composites

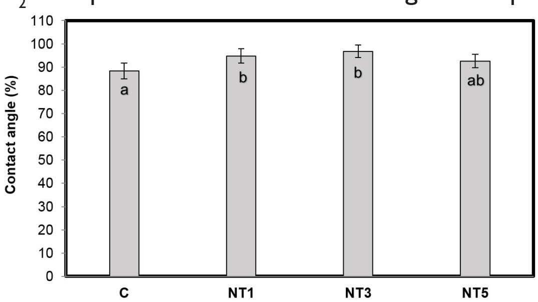

FIGURE 2 Contact angle of samples: (C) Control, (NT1) 1%Nano TiO_2, (NT3) 3%Nano TiO_2, (NT5) 5%Nano TiO_2.

had a significant difference of 5% (Table 2). According to the Figure 3, increasing the amount of TiO_2 nanoparticles from 0% to 1% and from 1% to 3% reduced the thickness swelling of the composites, but the use of 5% nanoparticles increased thickness swelling compared with the 3% nanoparticles. The highest decrease of thickness swelling in the composite was determined to be 70.83 % for the 3% TiO_2 nanoparticles. Water absorption and thickness swelling of the wood plastic composites are due to the hydrophilicity of the lignocellulosic materials, the fine pores in their structure, the pores in the interphase region, and the micro cracks when making composites (Stokke and Gardner 2003; Ghasemi and Kord 2009). The decrease in composite swelling may reflect that TiO_2 nanoparticles filled the pores in the wood-plastic composites, where they acted as a barrier in the path of water. Because the more suitable distribution of nanoparticles created a better barrier in the composites, using 3% TiO_2 nanoparticles decreased the thickness swelling of the composites. However, using 5% TiO_2 nanoparticles, the thickness swelling of the composites increased. Zhu et al. (2011) reported an increase in the barrier properties of poly lactic acid composites after adding TiO_2 nanoparticles. The results of the studies showed that high concentrations of TiO_2 nanoparticles are agglomerated in the composites and increase their water absorption and thickness swelling (Han et al. 2008). Chen et al. (2006) observed a decrease in water uptake capacity of polyacrylate coating by modified TiO_2 due to the uniform dispersion of nanoparticles. TiO_2 nanoparticles at higher concentration became agglomerated in the composite, which resulted in an increase in water uptake capacity.

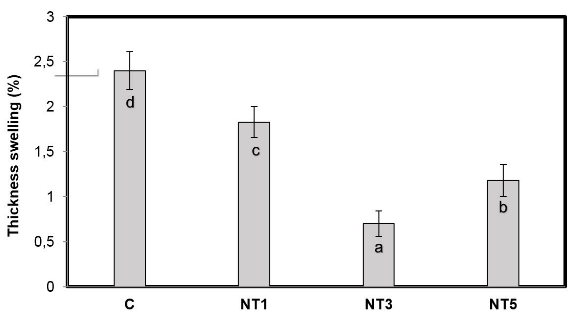

FIGURE 3 Thickness swelling of samples: (C) Control, (NT1) 1%Nano TiO_2, (NT3) 3%Nano TiO_2, (NT5) 5%Nano TiO_2.

Field emission scanning electron microscopy

In order to evaluate the nanoparticle distribution at all mixture ratios (from 0 to 5%), the ultrathin sections of the specimens were observed via FESEM as shown in Fig. 4. The obtained micrographs confirmed the results of physical and mechanical examines on the nanocomposites. It can be concluded that the distribution of 3% TiO_2 nanoparticles can be assumed

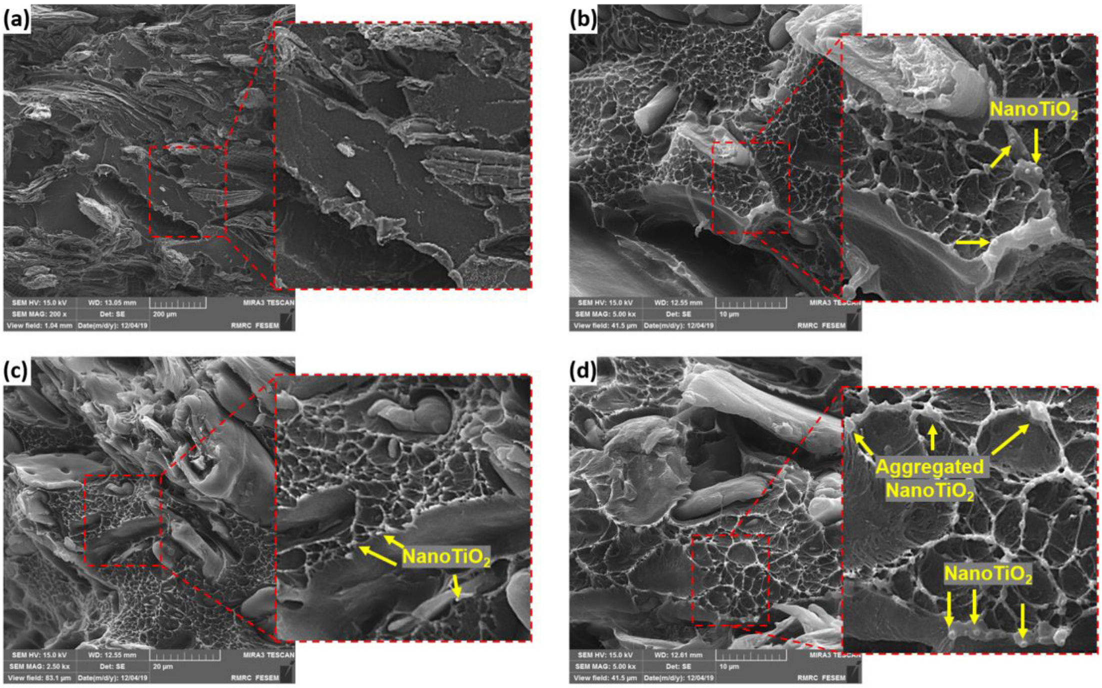

FIGURE 4 FESEM micrographs of the WPC filled with TiO_2 nanoparticles. (a) 0%, (b) 1%, (c) 3%, (d) 5%.

to be homogeneous and there is no agglomeration which results in a reduction of interaction between the material components. However, it can be seen that some of the nanoparticles formed increasing agglomeration with the increasing TiO_2 nanoparticles loading (Figure 4d). The results of observations express that the agglomeration of nanoparticles occurred in the specimens filled with high amounts of TiO_2 nanoparticles.

CONCLUSION

The work presented in this paper investigated mechanical and physical properties of wood/polyethylene composite reinforced with TiO_2 nanoparticles. Several conclusions were achieved. The use of TiO_2 nanoparticles as reinforcement in wood-plastic composites resulted in an increase in the tensile (26.02%) and bending (21.88%) strengths and a decrease the thickness swelling (70.83%) of the composites. As TiO_2 nanoparticles were added to the composites up to 3%, the mechanical properties and the thickness swelling were improved. However, as TiO_2 nanoparticles were added to the composites up to 5%, the mechanical properties decreased, and the thickness swelling was increased. The improved bending and tensile strengths and the thickness swelling of the composites when adding 3% TiO_2 nanoparticles can be attributed to the uniform dispersion of the nanoparticles in the composite and the improvement of the adhesion between phases in the composites. The decrease in the bending and tensile strengths and the increase in the thickness swelling of the composites when adding 5% TiO_2 nanoparticles compared to 3% TiO_2 nanoparticles can be attributed to the agglomeration of TiO_2 nanoparticles, which decreased the bending and tensile strengths and increased the thickness swelling of the wood plastic composites by decreasing the matrix interface and concentrating the stress in the agglomerated regions. The effect of TiO_2 nanoparticles on the impact strength of composites at the 5% level was not significant. The contact angle of wood plastic composites was improved by TiO_2 nanoparticles (9.48%). TiO_2 nanoparticles are desirable as reinforcers that improve the mechanical properties and create the dimensional stability of the wood plastic composites.

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