

Effect of Titanium Dioxide on Some Mechanical, Thermal, and Surface Properties of Wood-Plastic Nanocomposites

Alperen Kaymakci *

The effects of TiO₂ (titanium dioxide) loading were evaluated relative to mechanical, thermal, and surface properties of the wood-plastic nanocomposites (WPNs). Pine wood flour, mixed with maleic anhydride polypropylene (MAPP), TiO₂ nanoparticles (0 wt%, 1 wt%, 2 wt%, 3 wt%, 4 wt%, and 5 wt%), and polypropylene, was compounded in a twin screw co-rotating extruder. The mass ratio of the wood flour to polypropylene (PP) was 50/50 (w/w) in all compounds. Test specimens were produced using injection molding machine from the pellets. Flexural and tensile properties, thermogravimetric analysis, surface roughness, wettability, and morphology of the manufactured nanocomposites were evaluated. Flexural and tensile properties of the wood-plastic nanocomposites increased with the increasing content of the TiO₂. The surface roughness of the wood-plastic nanocomposites was reduced by increasing the content of TiO₂. Increasing the loading of TiO₂ increased the amount of residual ash and thermal stability.

Keywords: Wood; Nanocomposites; TiO₂; Mechanical properties; SEM

Contact information: Assistant Professor, Kastamonu University, Faculty of Forestry, Department of Forest Industry Engineering, Kastamonu, 37150, Turkey;

* *Corresponding author:* akaymakci@kastamonu.edu.tr

INTRODUCTION

Composite materials are prepared for a specific purpose, formed by the combination of two or more materials at a macroscopic level, with different morphological, physical, and mechanical properties. The advantage of composites is that they can exhibit new and different properties than the individual raw materials. Composite materials consist of a matrix and reinforcing agent components. The most frequently used reinforcing components are calcium carbonate, glass wool, talc, powder ceramics, and mica. However, these inorganic fillers cause machine wear, and they are costly (Gan 2009; Turku *et al.* 2017, 2018). The idea of utilizing lignocellulosic fibers as the reinforcing material and filler has emerged in the composite material industry. The lignocellulosic fillers used in composite production reduce the total production costs and provide significant performance improvements. Lignocellulosic materials such as wheat stalk, barley stalk, wood flour and fiber, carpenter's waste, hemp stalks, kenaf, and sunflower stalks have been utilized in the production of wood-plastic composites. These materials reduce the amount of polymer used. Wood-polymer composites (WPCs) are innovative hybrid materials and replace many traditional product applications due to their 3-D moldability, durability, and weatherability (Krause 2015). Wood-plastic composites are used in many areas such as flooring material, automobile panels, fences, garden furniture, and exterior façades (Zhang *et al.* 2010; Bajwa *et al.* 2011; Bazant *et al.* 2014; Ayrilmis *et al.* 2015; Homkhiew *et al.*

2014; Gardner *et al.* 2017; Ratanawilai and Taneerat 2018). Despite the many advantages of wood-plastic composites, some shortcomings (*i.e.*, relatively low modulus, low impact resistance, and creep performance) have led researchers to use various compatibilizers or nanoparticles that serve as interfacial adhesives to improve the performance of wood-plastic composites. Nano-sized reinforcing materials have the potential to provide significant performance improvements in wood-plastic composites due to their high specific properties. Thus, wood-plastic nanocomposites (WPNs) are expected to become high performance and value added products for end use with advantages such as high modulus value, high shock resistance, and thermal stability. Nanocomposites are materials formed by the dispersion of nanometer-sized particles within a matrix (Chen *et al.* 2007; Klyosov 2007; Lei *et al.* 2007; Ghasemi and Kord 2009; Kasraei and Azarsina 2012; Beigloo *et al.* 2017; Nikmatin *et al.* 2017; Asikuzun and Kaymakci 2018). Thus, WPNs are obtained by mixing nano material, plastic, and lignocellulosic fillers with conventional wood-plastic composite methods. Many nanomaterials such as carbon nanotube, boron nitride, clay, SiO₂, and TiO₂ are used in the production of WPNs. In particular, TiO₂ has received numerous applications due to its strong oxidizing power, chemical inertness, non-toxic, low cost, high refractive index and other advantageous surface properties. Also, the surface of TiO₂ is capable of absorbing, decomposing or reacting (oxidizing / reducing) a wide variety of inorganic and organic molecules and atoms under certain conditions. The most important among them is the H₂O dissociative or nondissociative adsorption behavior on TiO₂, which determines TiO₂ wetting and dispersing abilities in aqueous or non-aqueous medium and other surface properties (Lin 2006). However, there is a need for information on the effectiveness of these materials in the composite structure. The performance of nanoparticles with lignocellulosic materials and polymers needs further examination. This study investigated the effect of TiO₂ loading on some mechanical, thermal, and surface properties of wood-plastic nanocomposites.

EXPERIMENTAL

Materials

Yellow pine (*Pinus sylvestris*) wood flour was used as a lignocellulosic filler. The flour was purchased from a wood-plastic composite deck manufacturer (Semadeck, Tekirdag, Turkey). The wood flour (sapwood part) passing through a 40-mesh screen was retained on an 80-mesh screen. Polypropylene (PP) with a density of 0.9 g/cm³ was purchased from Borealis Incorp in Austria. It has a melting point of 170 °C and a melt flow index of 2.5 g/10 min at 230 °C. To eliminate the incompatibility between the polypropylene and the pine wood flour and to increase the bonding, maleic anhydride polypropylene (MAPP) (Optim-425; melt flow index about 120 g/10 min at 190 °C and a density 0.91 g/cm³ Pluss Polymers Pvt. Ltd., Gurugram, India) was used. As the reinforcing filler, titanium dioxide (TiO₂) (Grafen Company, Ankara, Turkey) was used. Some characteristics of the reinforcing fillers are given in Table 1.

Polypropylene, pine wood flour (WF), TiO₂, and the compatibilizer maleic acid grafted polypropylene (MAPP) were used as purchased from the manufacturer. The readymade wood flour was oven-dried at 103 °C ± 2 °C for 24 h. Drying the wood flour was important because moisture in lignocellulosic fillers causes bubbles to form during the extrusion and injection molding processes, leading to performance loss.

Table 1. Specifications of the TiO₂

Property	TiO ₂
Appearance	White powder
Average particle size	10 nm to 25 nm
Purity	> 99.5%
Surface area	> 50 m ² /g

Production of the Wood-plastic Nanocomposites

The production of WPNs was carried out in two phases: pellet production and nanocomposite production. In the first phase, small granules (pellets) were produced, while in the second phase samples were produced by injection molding. Prior to the production, the wood flour was dried until the moisture was reduced to below 1%. The dried wood flour was melted in the extruder (Aysa Machine, Istanbul, Turkey) by premixing it with the polypropylene (PP), TiO₂, and MAPP according to the production prescription (Table 2) and then pushed into the die with the screw in the double screw extruder (temperature of 185 °C to 200 °C). The molten material that exited through the die in the extruder end was cooled with cold water and left to dry. Composite samples in the shape of fine rods dried at 80 °C for 3 h were made into pellets *via* a plastic crusher (ZHL-SA, TSP Machine, Tekirdag, Turkey).

The pellets were oven-dried until reaching a content of 1% to 2% moisture before the injection molding process. The dried pellets were made into a test sample in an injection molding machine (TSPX 60; TSP Machine, Tekirdag, Turkey) operating at a screw speed of 40 rpm and a temperature of 185 °C to 200 °C. The injection pressure was set to 5 MPa to 6 MPa, the injection speed was 80 mm/s, and the cooling rate was 30 s. The specimens were stored under controlled conditions (50% relative humidity and 23 °C) for at least 24 h prior to testing.

Table 2. Composition of the Evaluated Formulations

ID	WF (wt%)	TiO ₂ (PhC)*	PP (wt%)	MAPP (wt%)
A	50	0	50	3
B	50	1	50	3
C	50	2	50	3
D	50	3	50	3
E	50	4	50	3
F	50	5	50	3

* Per hundred compounds

Methods

Mechanical properties

The flexural tests and tensile tests were conducted according to the ISO 178 (2010) and the ISO 527-1 (2012), respectively, using a Lloyd universal testing machine (Model LS 100, Bognor Regis, UK). The tests were performed at a crosshead speed of 5 mm/min. Seven replicates were tested for both the flexural and tensile strength measurements.

Thermogravimetric analysis

A Hitachi STA 7300 (Tokyo, Japan) apparatus was used for thermogravimetric analysis (TGA). Nitrogen gas was used at a flow rate of 20 mL/min; samples were in quantities of 9 mg to 10 mg. 3 samples were used for thermogravimetric analysis (TGA).

The temperature during this test was increased from room temperature to 500 °C with a heating rate of 10 °C/min. The thermal degradation temperatures and the amount of waste were noted.

Wettability and surface roughness measurements

A KSV Cam-101 (Helsinki, Finland) was used to determine the contact angle of the wood-plastic nanocomposite surface. A drop of distilled water was placed on the composite surface, and the contact angle was determined using the KSV software package. The contact angles were measured up to 180 s every 3 s. The contact angle was measured for at least five specimens of each nanocomposite group.

For the surface roughness test, a total of 48 specimens were taken as 8 samples from each group of wood-plastic nanocomposites. Surface roughness tests were performed on the Zeiss Handysurf E-35A stylus type profilometer (Oberkochen, Germany) according to ISO 4287 (1997). Two roughness parameters, average roughness (R_a) and mean peak-to-valley height (R_z), were used to evaluate the surface roughness of the wood-plastic nanocomposites.

Scanning electron microscope (SEM) analysis

The SEM analyses of wood-plastic nanocomposite materials were performed on an FEI Quanta-FEG 250 microscope (Hillsboro, OR, USA). The specimens were left in liquid nitrogen for a period and then fractured to obtain a clean fractured surface prior to the analysis. To prevent any possible reflections that may occur on the surfaces of the samples, the fractured surfaces were coated with gold dust for 120 s at 10 mA.

Statistical analysis

IBM SPSS Statistics 23 statistical software (New York, USA) was used for statistical analysis. An analysis of variance (ANOVA) was conducted ($p < 0.05$) to evaluate the effect of the TiO_2 content on the mechanical, thermal, and surface properties of WPNs. Significant differences among the average values of the wood-plastic nanocomposite types were determined using Duncan's multiple range tests.

RESULTS AND DISCUSSION

Mechanical Properties

The flexural and tensile properties of TiO_2 reinforced wood-plastic nanocomposites are shown in Table 3. With TiO_2 loading, the flexural properties and tensile properties of WPNs improved. The samples containing TiO_2 exhibited a more rigid structure than the groups without TiO_2 .

The increase in the flexural modulus of the wood-plastic nanocomposites was higher than the increase in the flexural strength. For example, the flexural strength of the nanocomposite group with 5% TiO_2 was increased by 8.9%, while the flexural modulus of the group increased by 14.7%. The specimen types presenting significant differences with further groups according to Duncan's multiple-range tests are shown by superscript letters in Table 3. The flexural strength of wood-plastic nanocomposites was significantly affected by the amount of TiO_2 .

Table 3. Mechanical Properties of the WPNs Reinforced with TiO₂

ID ¹	Flexural Strength (N/mm ²)	Flexural Modulus (N/mm ²)	Tensile Strength (N/mm ²)	Tensile modulus (N/mm ²)	Elongation at Break (%)
A	59.5 (0.9) a	4904 (336) ns ²	18.1 (2.3) a	1266 (95) a	6.1 (0.7) a
B	60.5 (1.5) a	4996 (259) ns	23.0 (2.1) b	1292 (80) a	6.1 (0.9) a
C	61.5 (3.0) a	5177 (796) ns	25.3 (1.3) c	1311 (80) a	5.8 (0.5) a
D	61.7 (2.3) a	5279 (726) ns	26.0 (0.8) cd	1411 (101) b	5.8 (0.6) a
E	64.0 (2.1) b	5486 (487) ns	26.2 (0.5) cd	1421 (65) b	4.7 (0.6) b
F	64.8 (1.2) b	5627 (570) ns	27.8 (2.2) d	1545 (80) c	3.9 (0.7) c

Groups with the same letters in a column showed no statistical difference ($p < 0.01$) between the samples according Duncan's multiple range test; values in parentheses are the standard deviations. ¹ See Table 2, ² ns: not significant

As shown in Table 3, a maximum flexural strength of 64.8 N/mm² was found in the nanocomposite groups containing 5% TiO₂. The minimum flexural strength of 59.5 N/mm² was found in control group samples without TiO₂. The flexural strength and elasticity modulus increased as the amount of TiO₂ in the nanocomposite structure increased. This can be attributed to the positive compatibility among TiO₂, WF, and PP. These results show similarities to previous studies. Hazarika and Maji (2013) investigated the synergistic effect of nano-TiO₂ and nanoclay on the ultraviolet degradation and physical properties of wood polymer nanocomposites. According to their results, the incorporation of nanoclay and nano-TiO₂ influences the mechanical properties of the wood polymer nanocomposites to a substantial amount. At a constant clay amount (3 phr), the mechanical properties of the wood polymer nanocomposites improve with an increase in TiO₂ loading. The polymer chains were fastened in the gallery layers of the nanoclay, limiting the mobility of the polymer chains and stiffening the nanocomposites. This is explained by the positive coalescence between the nanomaterials and the nanocomposite structure (Hazarika and Maji 2013).

The tensile strength, tensile modulus, and elongation at break values of the wood-plastic nanocomposites were significantly improved by the incorporation of the TiO₂. The tensile strength of wood-plastic nanocomposites increased from 18.1 N/mm² to 27.8 N/mm² as the TiO₂ content increased from 0 wt% to 5 wt%. The specimen treated with 5 wt% TiO₂ and 3 wt% MAPP showed the highest tensile strength values among the other types of wood-plastic nanocomposites. This result was consistent with the observation that the introduction of nano-sized particles into a polymer matrix increased its tensile properties. The improvement is logical because the filler in the TiO₂ form can carry more tensile load. Furthermore, the TiO₂ is much stiffer than the polymer matrix, and as a result it adds stiffness to the nanocomposites. The obtained data corresponded with other studies. Deka and Maji (2011) investigated the effect of TiO₂ and nanoclay on the properties of wood polymer nanocomposites. The study determined that the value of tensile strength increases depending on the increasing content of nanoclay and TiO₂. This increase was attributed to improved interfacial adhesion between the polymers by the compatibilizer (Deka and Maji 2011). In another study, Saeed *et al.* (2009) investigated the characterization of poly(butylene terephthalate) electrospun nanofibers containing TiO₂. They determined that the tensile strength of electrospun nanofibers increases based on an increase in the usage ratio of TiO₂. In addition, at higher percentages of TiO₂ loading (5 phr), the effective agglomeration of the nanoparticles results in decreased mechanical properties (Saeed *et al.* 2009). The tensile elongation at break values of the wood-plastic

nanocomposites were significantly improved by the incorporation of the TiO₂. The tensile elongation at break value of wood-plastic nanocomposites decreased from 6.1% to 3.9% as the TiO₂ content increased from 0 wt% to 5 wt%.

Thermogravimetric Analysis and Scanning Electron Microscopy

The results of the thermogravimetric analysis (TGA) are presented in Table 4. The thermal stability of WPNs improved with increasing TiO₂ loading.

Table 4. Thermogravimetric Analysis of the WPNs Reinforced with TiO₂

ID ¹	Onset Temperature (°C)	Peak Temperature (°C)		Total Weight Loss (%)	Residue after 500 °C
		1 st Peak	2 nd Peak		(%)
A	196.8	303.4	449.5	91.7	8.3
B	198.4	303.5	450	91.1	8.9
C	215.6	307	451.9	90.9	9.1
D	218.4	309.1	452.3	87.9	12.1
E	225.6	311.2	452.4	86.7	13.3
F	227.4	311.3	453.2	77.5	22.5

¹ See Table 2

The maximum onset temperature was 227.2 °C in the nanocomposite groups containing 5% TiO₂. The minimum onset temperature was 196.8 °C in the nanocomposite control groups. The total weight loss decreased with an increase in the amount of TiO₂ in the nanocomposites. Total weight loss, for instance, decreased from 91.7% to 77.5% when TiO₂ loading on the wood-plastic nanocomposites increased from 0 wt% to 5 wt%. The obtained data showed similarities with previous studies. Rahman *et al.* (2017) investigated the differential scanning calorimetry (DSC) and the TGA of wood polymer nanocomposites. According to their results, the presence of nanoparticles in the lignocellulose matrix results in an increase in the thermal stability of the nanocomposite. Additionally, the results show that the mass loss (%) decreases with an increase in the amount of nanoparticles in the nanocomposites (Rahman *et al.* 2017).

Wettability and Surface Roughness

The average roughness (R_a) values and mean peak-to-valley height (R_z) values of the WPNs reinforced with TiO₂ are presented in Table 5. The surface roughness values of the WPNs decreased with the addition of TiO₂ (Fig. 1). When the surface roughness values of the WPNs were examined, the highest R_a value was found in group A with 3.9 μm , and the lowest R_a value of 2.8 μm was observed in group F. The surface roughness of the WPNs was reduced by increasing the content of TiO₂.

Table 5. Surface Roughness Results of Wood-Plastic Nanocomposites

ID ¹	R_a (μm)	R_z (μm)
A	3.9 (0.21) * a	24.5 (1.98) a
B	3.6 (0.08) ab	22.7 (2.07) ab
C	3.3 (0.36) bc	21.9 (3.22) bc
D	3.01 (0.30) cd	20.7 (2.50) bcd
E	3.01 (0.26) d	19.6 (0.61) cd
F	2.8 (0.30) d	18.9 (0.38) d

* Values in parentheses are standard deviations; ¹ See Table 2

The higher surface roughness of the WPNs was attributed to the presence of gaps and micro-cracks on their surface (see Fig. 3)

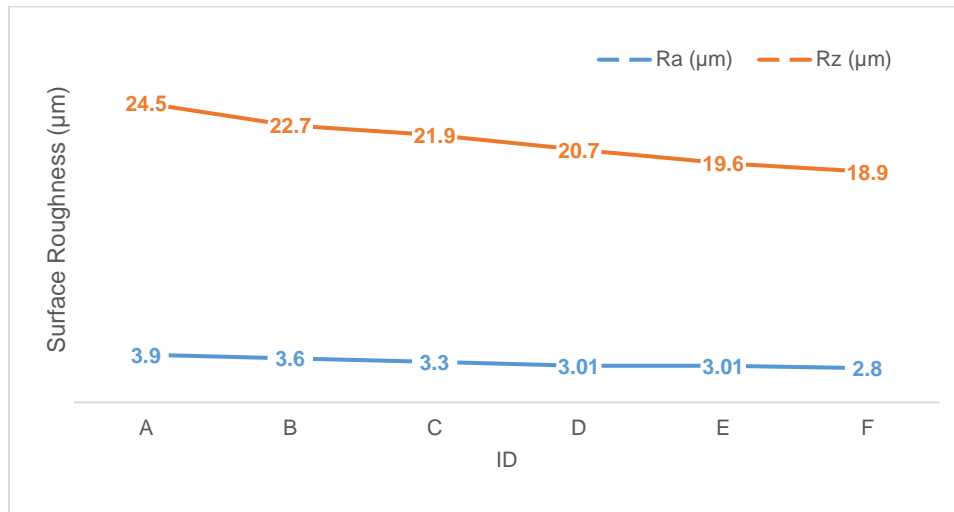


Fig. 1. Effect of TiO₂ on surface roughness values of wood plastic nanocomposites

Similar findings were observed for the R_z values. These results show similarities to other studies. Hazarika and Maji (2013) investigated the synergistic effect of nano-TiO₂ and nanoclay on the ultraviolet degradation and physical properties of wood polymer nanocomposites; the incorporation of nanoclay and nano-TiO₂ influenced the mechanical properties of the wood polymer nanocomposites to a significant amount. Nikmatin *et al.* (2017) investigated the physical, thermal, and mechanical properties of polypropylene composites filled with rattan nanoparticles. The surface morphology of polypropylene composites improves with the presence of rattan nanoparticles. In addition, the surface morphology of a composite is a key factor that affects the physical, thermal, and mechanical properties of the composites. Liang *et al.* (2014) investigated the effect of impregnated inorganic nanoparticles on the properties of the kenaf bast fibers. In this work, where the surface roughness is determined by the AFM method, the presence of nanoparticles improves the root mean square surface roughness of the kenaf bast fibers. Means and standard deviations of the WPN wettability values are given in Table 6.

Table 6. Contact Angles Measurements of Wood-Plastic Nanocomposites

ID ¹	10 s Degree (°)	30 s Degree (°)	60 s Degree (°)	90 s Degree (°)	120 s Degree (°)	150 s Degree (°)	180 s Degree (°)
A	107.9 (3.68)	105.7 (3.54)	103.8 (2.98)	100.6 (3.98)	99.6 (3.45)	98.8 (4.12)	97.2 (3.88)
B	105.8 (0.87)	104.6 (1.21)	103.8 (1.64)	101.1 (2.67)	99.8 (2.39)	97.5 (3.33)	95.7 (2.87)
C	103.0 (2.51)	102.8 (2.97)	100.6 (2.37)	99.3 (1.56)	97.7 (1.78)	95.4 (2.34)	93.7 (0.56)
D	101.7 (1.91)	100.3 (1.63)	99.7 (0.89)	97.1 (2.35)	95.6 (2.78)	93.4 (1.87)	90.8 (3.63)
E	95.7 (0.91)	94.2 (1.41)	90.3 (3.12)	89.6 (3.31)	86.5 (4.15)	84.8 (1.32)	83.9 (2.87)
F	92.1 (0.96) *	91.6 (0.85)	90.4 (1.25)	88.9 (2.75)	86.3 (0.96)	85.4 (1.12)	81.6 (1.37)

* Values in parentheses are standard deviations; ¹ See Table 2

The wettability decreased with the increasing TiO₂ content. The lowest contact angle was 92.1° in WPNs containing 5% TiO₂ (group A). The highest contact angle of 107.9° was observed in group A. The highest contact angle value of 107.9° (10 s) was found for samples containing 5% TiO₂. The contact angle of the specimens was significantly affected by increasing the TiO₂ content (Fig. 2). The obtained data show similarities with previous studies. Kasraei and Azarsina (2012) investigated the effect of silver nanoparticles on the wettability of methacrylate and silorane-based composites; the addition of silver nanoparticles causes a reduction in the water contact angle of the composites.

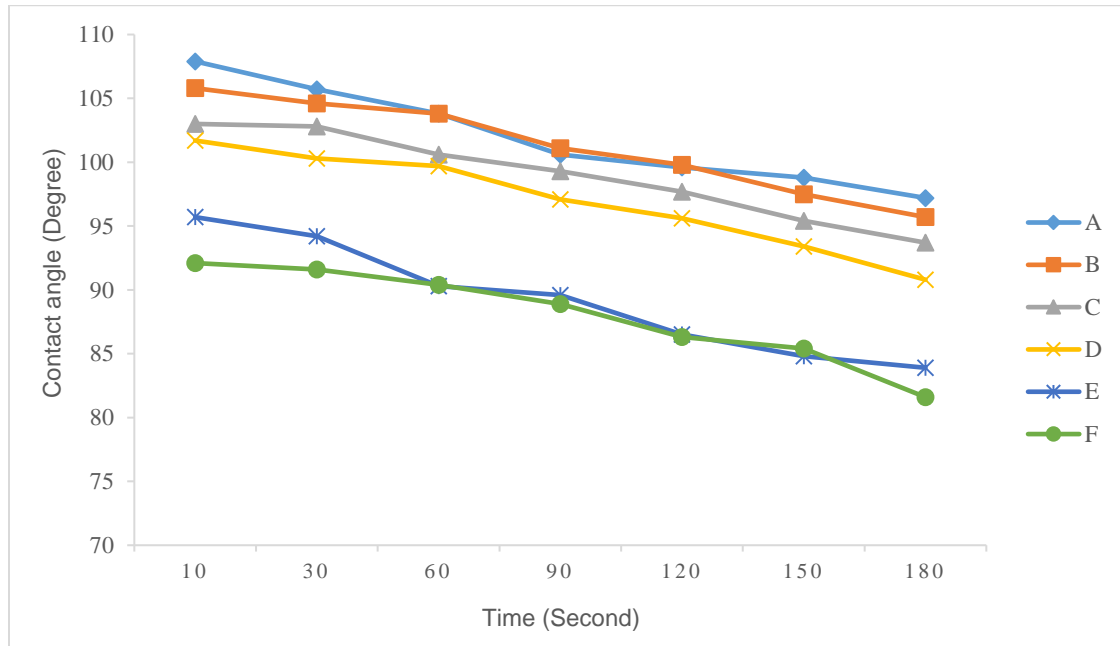


Fig. 2. Effect of TiO₂ on contact angle values of wood plastic nanocomposites

Scanning Electron Microscopy (SEM)

The morphology of the TiO₂ reinforced WPNs was studied. SEM micrographs for all groups are shown in Fig. 3. There were no obvious differences between the composites with reinforced and unreinforced groups, but the adhesion was better in the composite reinforced with TiO₂.

The improvements in flexural strength, flexural modulus, tensile strength, and tensile modulus of the WPNs with incorporation of TiO₂ was supported by SEM. No large gaps between the matrix, wood flour, and TiO₂ are shown in the images. The results confirmed the observed enhancement in the physical, mechanical, and thermal performances.

In the case of the nanocomposites filled with 0 wt% TiO₂, (Figure 3(a)) many deep holes and voids remained after the fillers were pulled out of the matrix. The presence of these holes means that the interfacial bonding between the WF, TiO₂, and the PP was weak and therefore the TiO₂ could not provide an efficient stress transfer from the matrix.

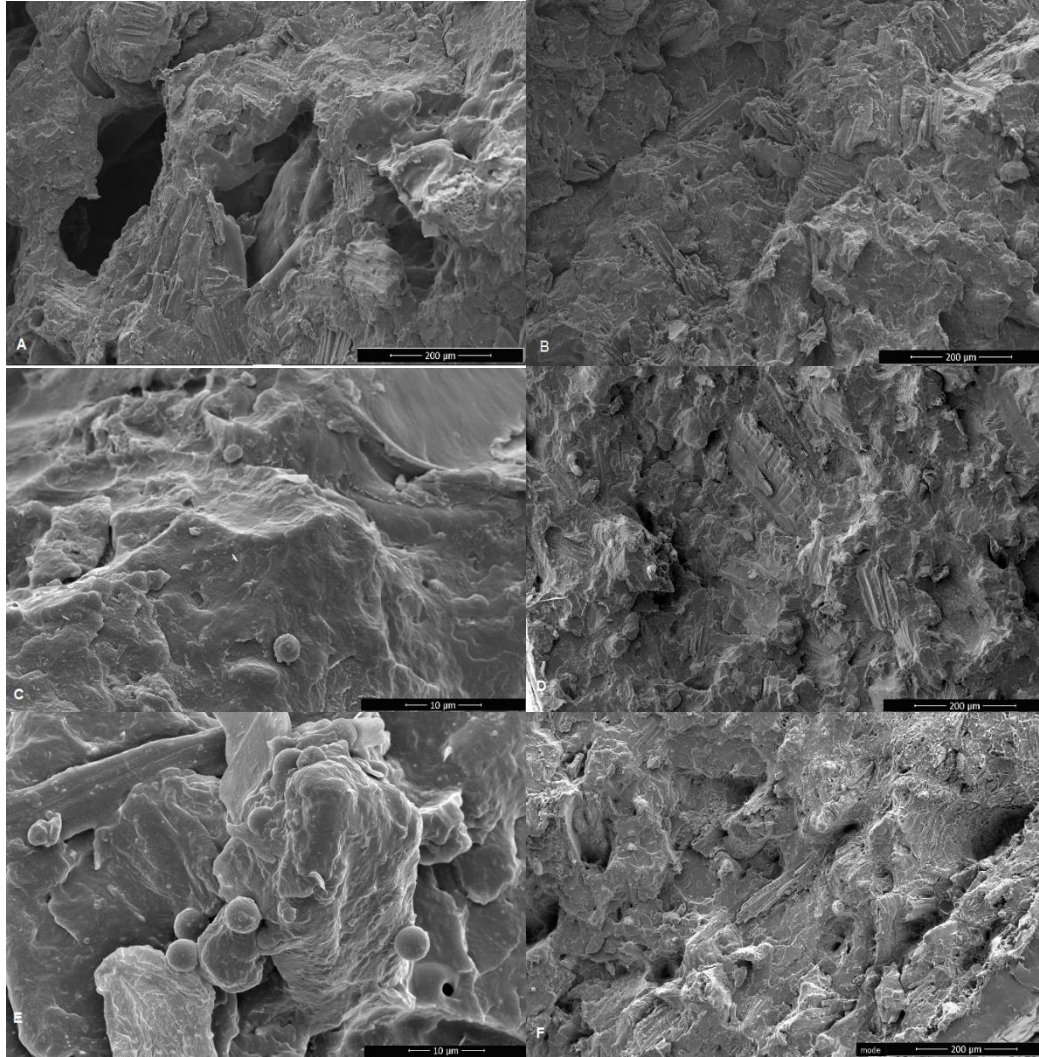


Fig. 3. SEM micrographs of the WPNs with (a) 0 wt% TiO₂, (b) 1 wt% TiO₂, (c) 2 wt% TiO₂, (d) 3 wt% TiO₂, (e) 4 wt% TiO₂, and (f) 5 wt% TiO₂

CONCLUSIONS

This study examined the effect of TiO₂ nanoparticles content on some mechanical, thermal, and surface properties of wood-plastic nanocomposites.

1. The flexural properties and tensile properties of wood polymer nanocomposites increased with increasing amounts of TiO₂.
2. Increasing the loading of TiO₂ increased the amount of residual ash and thermal stability.
3. The surface roughness of the wood-plastic nanocomposites was reduced by increasing the content of TiO₂.
4. The 50/50/5/3 formulation of PP, wood flour, TiO₂, and MAPP, respectively, for wood-plastic nanocomposites gave the best mechanical, thermal, and surface properties.

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