Effect of Steamed and Non-Steamed *Populus deltoides* Fiber on the Physical, Mechanical, and Morphological Characteristics of Composites Made from Virgin Polypropylene

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The effects of steamed *Populus deltoides* fiber were studied relative to the physical, mechanical, and morphological characteristics of composites made from virgin polypropylene. Fibers of *Populus deltoides* were used during the reinforcement phase at 180 °C for 1 h. The tests were carried out with 57% of virgin polypropylene that was combined with 3% of maleic anhydride-modified polypropylene and 40% of wood fiber. The wood fiber portion consisted of either non-steamed fiber, steamed fiber, and an equal mixture of the two kinds of fiber. Mixing was done using an internal mixer at 180 °C and 60 rpm, and the standard samples were constructed by injection molding. Then, the physical and mechanical characteristics of samples were measured. To consider compatibility between the matrix and reinforcement phase, SEM pictures were taken from the break surface of composite samples. The results showed that the composites' strengths were affected by steam, as components having steamed fibers had the best mechanical strength and dimensional stability, and also the least water absorption and thickness swelling.

Keywords: Composites; Polypropylene; SEM; Steaming; Populus deltoides fiber

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INTRODUCTION

Lignocellulose material is used extensively in the construction of composites because of its low price, accessibility, and renewability. These composites share certain advantages with wood, such as low specific weight, low price, resistance to UV rays, and favorable machining features. Lignocellulose material has many other valuable features as well, such as high electronic strength, soundproofing, and thermal insulation (Arbelaiz *et al*. 2005; Mechraoui *et al.* 2007). To improve the compatibility among natural and thermoplastic fibers, the reduction of hydrophilic characteristics of the fibers can be achieved *via* reducing the accessibility of the hydroxyl groups in lignocellulose materials (Enayati *et al*. 2009). All the main wood components (cellulose, hemicellulose, and lignin) are somewhat hydrophilic; however, hemicellulose contains more polar groups, whereas lignin contains the least polar groups (Mosier *et al*. 2005). Hydroxyl groups (OH) account for the hydrophilic factor in many components. Regardless of the existence of these groups, their availability is considered as an important factor as well. Almost all of the hydroxyl groups in hemicellulose are available, while in cellulose, only the hydroxyl groups in amorphous sections of cellulose are available, which amounts to approximately 40% of the material (Mohanty *et al*. 2005).

Many methods have been used to improve the compatibility among natural fibers and thermoplastic polymers. These methods are based on chemical and physical treatments. The most important method utilizes a coupling agent such as maleic anhydride

(Kazayawoko *et al*. 1999; Albano *et al*. 2001). Processing composites with alkaline materials (such as sodium hydroxide) is another useful method for eliminating lignin and hemicellulose from the fiber surface (Ma 2007). The aforementioned method also decreases the amount of hydroxyl groups, which leads to better interaction between the fibers and polymer matrix.

Wood steaming is one of the most common methods for reducing water absorption and improving dimensional stability through the reduction of hydroxyl groups, under the effect of hemicellulose degradation (Kazayawork *et al*. 1999; Li *et al*. 2007). Wood steaming can lead to better compatibility between lignocellulose materials and the polymer matrix because of hemicellulose degradation and the reduction of woods' hydrophilic character (Enayati *et al*. 2009). Thermal treatment is widely used to improve the characteristics of lignocellulose materials. Thermal treatment can increase dimensional stability and rot resistance, as well as reduce water absorption (Enayati *et al*. 2009). Accordingly, Brugnago *et al*. (2010) found that high pressure steaming is one of the most common techniques to decompose lignocellulose materials, such as wood, to its three main components. It eventually leads to the elimination of hemicellulose and lignin of the fibers; although it causes a high level of crystallinity in the cellulose component, due to hydrolysis of its amorphous areas. Thus, steaming leads to better properties of the composites (Brugnago *et al*. 2010).

Ibrahim *et al*. (2010) examined the tensile strength and crystallinity of the composite obtained from reinforced lignocellulose of banana fibers and microfibrils under steaming treatment. The results showed that the tensile strength and crystalline structure were higher after steamed treatment compared to non-steamed treatment. In addition, these results were higher in composites composed of banana microfibrils compared to banana fibers (Ibrahim *et al*. 2010). Rowell (2006) studied the modification of consumed wooden particle in the particleboard industry and found that steaming led to the reduction of particle water absorption. The present research aimed at considering the effects of using steamed and non-steamed *Populus deltoides* fiber on the physical, mechanical, and morphological characteristics of composites made from virgin polypropylene.

EXPERIMENTAL

Materials

Reinforcement

Populus deltoides fiber was prepared from the educational forest at the University of Tehran, Iran. To ensure uniform particle size, only particles that passed through a 40-mesh sieve and were retained by a 60-mesh sieve were used as the reinforcement material. The aspect ratios of *Populus deltoides* fiber was 3.86 in the material retained on the 60-mesh sieve.

Polymer

Polypropylene (PP) with a density 0.952 g cm^{-3} and MFI 8 g/10 min was procured from Hyundai Petrochemical Co. (South Korea) under the brand name Seetec.

Coupling agent

Polypropylene modified with maleic anhydride (MAPP) with a density of 0.965 g cm^{-3} (MFI of 7 g/10 min, 1 wt% maleic anhydride) was used as the coupling agent. It was obtained Priex Resins Solvay Company (Belgium). The coupling agent was prepared at 3 wt.% content level.

Methods

Steaming the Populus deltoides fiber

In the steaming process, water and lignocellulosic materials with a ratio of 20 to 1 were heated in a laboratory cooking digester for 60 min at 180 °C and pressure. The pressure inside the cooking digester increased with increasing temperature. The maximum pressure was determined up to 700 kPa. Then the *Populus deltoides* fiber was dried at 100 ± 3 °C to a moisture content of 2%.

Sample preparation

Before sample preparation, *Populus deltoides* fiber was dried at 100 ± 3 °C for 24 h. PP, *Populus deltoides* fiber, and MAPP were then weighed and bagged according to the formulations given in Table 1. Mixing was carried out in a Haake internal mixer (Capacity 300 mL, SYSTEM 90HBI model, USA) equipped with a cam blade at 180 °C and 60 rpm. First, PP was fed into the mixing chamber. After melting, coupling agent were added. After mixing for 5 min, the *Populus deltoides* fiber was fed into the system. The total mixing time was 10 min.

Table 1. Ingredients of Different Compounds (wt.%) in the Composite Mixture

No.	Formulation	vPP%	MAPP	nsWF%	sWF%
1	vPP+nsWF	57	3%	40	-
2	vPP+sWF	57	3%	-	40
3	vPP+nsWF+sWF	57	3%	20	20

vPP = Virgin polypropylene
MAPP = Maleic anhydride polypropylene
nsWF = Non-steamed wood fiber
sWF = Steamed wood fiber

Composite preparation

The compounded materials were then ground to prepare the granules using a pilot-scale grinder (Wieser Co, WG-LS 200/200 model, Germany). The resulting granules were dried at 105 °C for 24 h. Test specimens were prepared by an injection molding machine (Imen Machine, Aslanian Co, Iran) at 175 °C and a pressure of 3 MPa according to standard ASTM D638. The specimens were stored in controlled conditions (50 % relative humidity and 23 °C) for at least 40 h prior to testing.

Measurement of water absorption and thickness swelling

Water absorption and thickness swelling tests were carried out according to ASTM D7031-10. The dimensions of the specimens for the water absorption and thickness swelling tests were 20 x 20 x 20 mm. Five specimens of each formulation were selected and dried in an oven for 24 h at 102 ± 3 °C. The weight and thicknesses of dried specimens were measured to a precision of 0.001 g and 0.001 mm. The specimens were then placed in distilled water and kept at room temperature. The weight and thickness of the specimens were measured after 7 weeks.

Measurement of mechanical properties

Tensile strength and tensile modulus were measured according to the ASTM D638-10 standard using an Instron Universal Testing Machine (model 1186) at a speed of 5 mm/min. The dimensions of the standard dumbbell (dog-bone) samples for the tensile tests were 145 x 10 x 4 mm (length x width x thickness).

Flexural strength and flexural modulus were measured according to the ASTM D747-10 standard using an Instron Universal Testing Machine (model 1186) at a speed of 2 mm/min. The dimensions of the specimens for the flexural tests were 105 x 13 x 5 mm (length x width x thickness).

Notched impact strength was measured according to the ASTM D256 standard using a Zwick Universal Testing Machine (model 5102). The dimensions of the specimens for the notched impact tests were 60 x 12 x 6 mm (length x width x thickness).

For each treatment level, five replicates were measured for each property and the average values were reported.

Scanning electron microscopy

The morphology of composites was characterized using SEM, (Leo Oxford, model 440i, UK) with 500X magnification at 25 kV accelerating voltage. Samples were first frozen in liquid nitrogen and then fractured to ensure that the microstructure remained clean and intact. The specimens were coated with a gold layer to provide electrical conductivity.

Statistical analysis

Data analysis was done by SPSS software (IBM Software, Armonk, New York; version 11.5) in randomized statistical plan under a factorial, and eventually comparison and grouping were done by the Duncan test using a 95% confidence level.

RESULTS AND DISCUSSION

Amounts of steamed and non-steamed fiber of *Populus deltoides* were investigated in this study at three different levels: vPP+nsWF, vPP+sWF, and vPP+nsWF+sWF. The F values and confidence level values are also listed in Table 2.

Table 2. Variance Analysis (F Value and confidence Level) of Steamed and Non-Steamed *Populus deltoides* Fiber in Composites

Treatment No.	Water Absorption 7W	Thickness Swelling 7W	Tensile Strength	Flexural Strength	Tensile Modulus	Flexural Modulus	Impact Strength
F-Value	90.353*	45.115*	33.973*	5.003*	15.965*	7.635*	4.667*

* 95% confidence level; ^{ns} no significance

Figures 1 through 7 indicate the effect of steamed and non-steamed *Populus deltoides* fiber content on water absorption, thickness swelling, and the mechanical properties of composites after 7 week. The effect of steamed and non-steamed *Populus deltoides* fiber was significant at a 5% level on water absorption and thickness swelling, tensile and flexural strength, tensile and flexural modulus and impact strength.

Physical Properties

Figures 1 and 2 indicate that by adding 40% steamed *Populus deltoides* fiber to composites, the water absorption and thickness swelling after treatment were reduced significantly after 7 Weeks. The percentage of reduction of water absorption and thickness swelling were 18.1% and 25.9%, respectively.

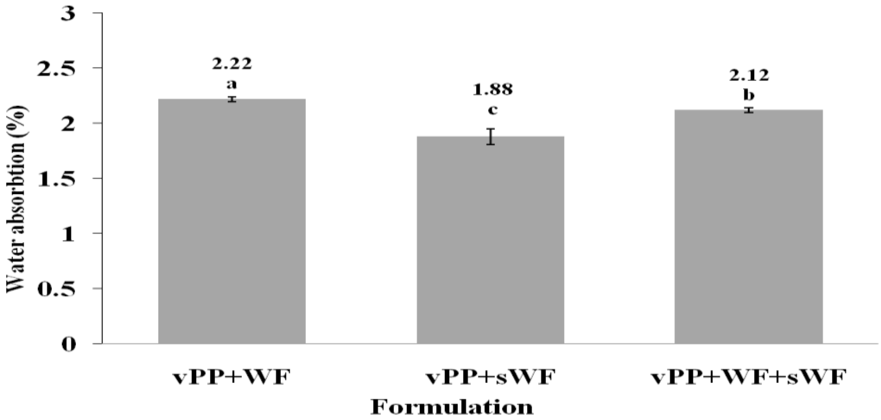

Fig. 1. The effect of steamed and non-steamed *Populus deltoides* fiber on water absorption after 7 week (Small letters indicate the Duncan of the averages at a 95% confidence level)

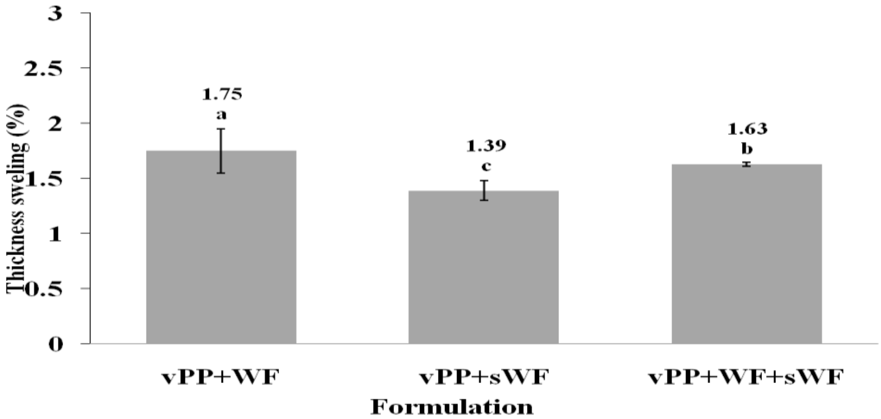

Fig. 2. The effect of steamed and non-steamed *Populus deltoides* fiber on thickness swelling after 7 week (Small letters indicate the Duncan of the averages at a 95% confidence level)

When moisture penetrated the compound materials, destruction and swelling occurred in the fibers (Espert *et al.* 2004). Furthermore, the polymer structure is influenced by some processes, such as the change of direction in polymer chains and stretch (Ghasemi and Kord 2009). In addition, water absorption causes loss of compatibility between matrix and fibers, which causes disintegration of the bonds and weak adhesion at the connection (Ghasemi and Kord 2009). The reduction of hydroxyl groups and hydrophilic characteristic of fiber in hydrothermal treatment of wood has been demonstrated (Tjeerdsma and Militz 2005). The reduction of hydroxyl groups in hemicellulose is the reason for the reduction of hydrophilic features, and reduction of hydrophilic characteristics of wood fiber leads to better compatibility of this material with non-polar polymers, such as polypropylene. As steaming eliminates hydroxyl groups in hemicellulose, the water absorption and thickness swelling levels also decrease accordingly. Renneckar *et al*. (2006) used steamed wooden fibers in the construction of composites and concluded that in making composites with steamed fibers, the water absorption content of fibers is reduced, and this phenomenon is the result of the inherent enhancement in wood's cell wall release (Renneckar *et al*. 2006). Steaming reduces the free hydroxyl groups, the results of which are properly seen in the treatments. Composites made with non-steamed *Populus deltoides* fibers had the highest level of water absorption and thickness swelling and steamed *Populus deltoides* fibers had the lowest level of water absorption and thickness swelling. This phenomenon is the result of steaming the *Populus deltoides* fibers.

Mechanical Properties

Figures 3 through 7 indicate that by adding 40% steamed *Populus deltoides* fiber to composites, the tensile and flexural strength, tensile and flexural modulus, and impact strength showed the most significant enhancements. These enhancements were 11.2%, 3.5%, 8.2%, 4.6%, and 5.4%, respectively.

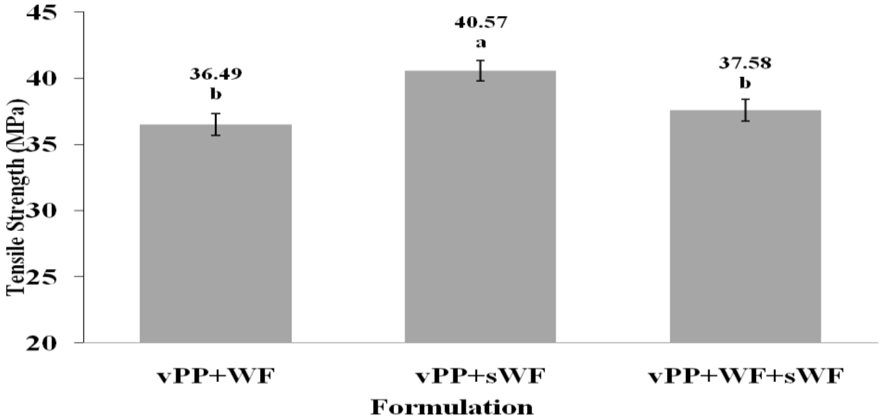

Fig. 3. The effect of steamed and non-steamed *Populus deltoides* fiber on tensile strength (Small letters indicate the Duncan of the averages at a 95% confidence level)

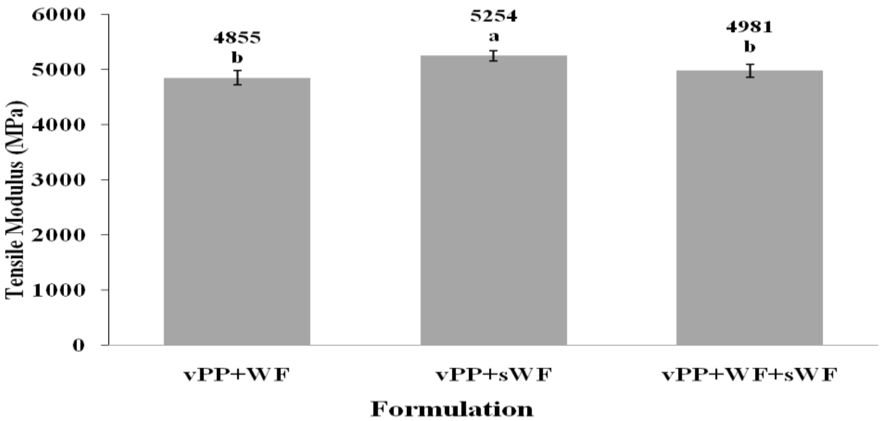

Fig. 4. The effect of steamed and non-steamed *Populus deltoides* fiber on tensile modulus (Small letters indicate the Duncan of the averages at a 95% confidence level)

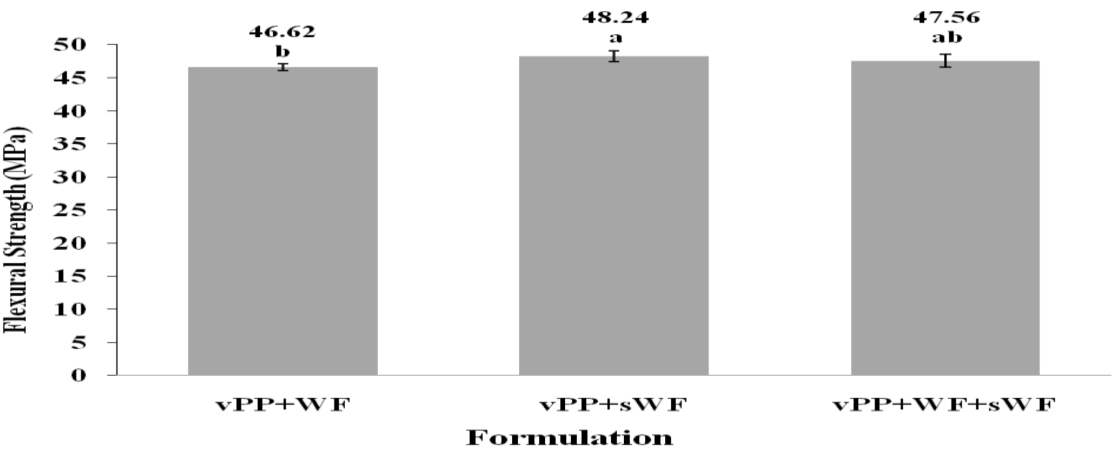

Fig. 5. The effect of steamed and non-steamed *Populus deltoides* fiber on flexural strength (Small letters indicate the Duncan of the averages at a 95% confidence level)

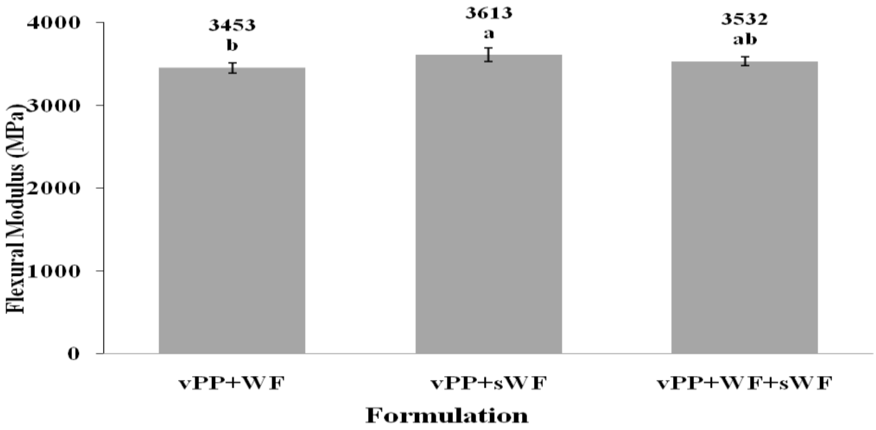

Fig. 6. The effect of steamed and non-steamed *Populus deltoides* fiber on flexural modulus (Small letters indicate the Duncan of the averages at a 95% confidence level)

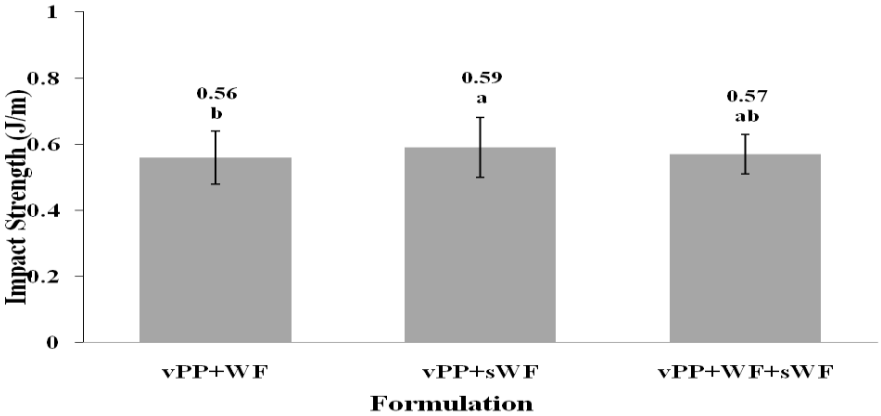

Fig. 7. The effect of steamed and non-steamed *Populus deltoides* fiber on impact strength (Small letters indicate the Duncan of the averages at a 95% confidence level)

Steaming process of *Populus deltoides* fiber caused damage to hemicellulose. Destruction of hydroxyl groups in hemicellulose will reduce its hydrophilic character. The reduction of *Populus deltoides* fiber hydrophilic characteristics improves the compatibility of this material with non-polar polymers, such as polypropylene. In fact, this phenomenon increases wood's adherence quality and mechanical properties (Hatefnia *et al.* 2012).

Another reason for the improvement of mechanical features may be the enhancement of crystallization in wooden fibers after treatment. Under the effect of thermal treatment, there is a possibility of hydrolysis of some of the amorphous areas in cellulose, as well as the elimination of hemicellulose (Yildiz and Gumuskaya 2007). The elimination of parts of the amorphous areas of cellulose enhances the fibers' degree of crystallization and mechanical resistance (Youngquist *et al*. 1994). In this research, samples containing steamed fibers had the greatest mechanical resistance, while samples containing non-steamed fibers had the least mechanical resistance. Brugnago *et al*. (2010) concluded that among all the pre-treatment methods (for lignocellulose material), the best ones are those that involve compounds of chemical and physical treatments. Steaming with high pressure is one of these methods, and it is one of the most common techniques for decomposing lignocellulose materials to their three main components. Steaming causes the elimination of hemicellulose and lignin from fibers, while the cellulose component reached high levels of crystallization because of the hydrolysis of amorphous areas. Therefore, steaming leads to the production of fibers with lower density, higher thermal resistance, and lower water absorption level (Brugnago *et al*. 2010).

Morphology

Compatibility between reinforcement fibers and polymer matrix is observable by micrographs from scanning electron microscopy (SEM). Figure 8 shows composites containing non-steamed fibers, focusing on their fiber sets. In this figure, proper overlap between matrix and reinforcement is clearly observable. Figure 9 is related to the steamed fibers. The figure clearly indicates that there is proper overlap between polymer matrix and reinforcement. Meanwhile, the steaming effect on the natural fibers which leads to defibrillation of the fibers and removal of agglomerates can be easily observed in this figure.

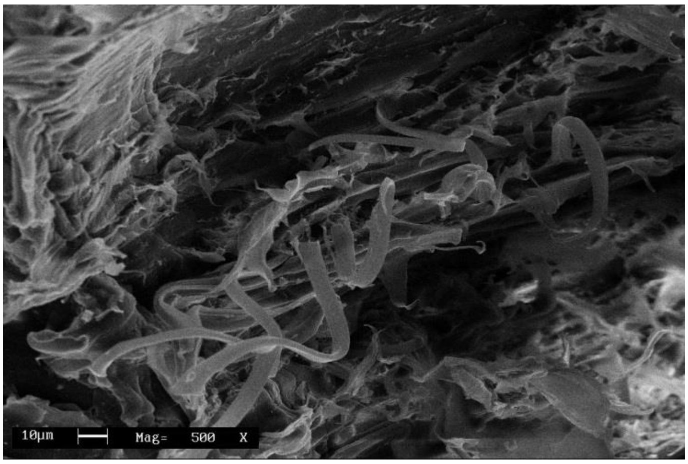

Fig. 8. SEM micrographs from vPP+nsWF composites

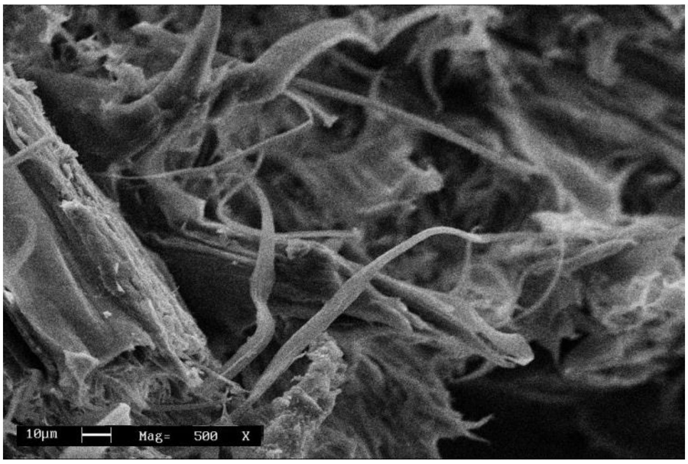

Fig. 9. SEM micrographs from vPP+sWF composites

Electron microscope figures indicate that with increasing steamed wood fiber content, the bond area between cellulose material and background matrix increased and bonds were formed between filler and matrix (Enayati *et al*. 2009). These bonds are the reason for the enhancement of mechanical resistance, reduction of water absorption, and thickness swelling of these composites. Moreover, steaming causes defibrillation of the fibers and removal of the agglomerates, and this phenomenon is clearly observable in the figures. Angles *et al*. (1999) worked on the sawdust of conifers with steaming technology. According to SEM figures, they concluded that steamed fibers had better compatibility.

CONCLUSIONS

The following results have been achieved in the present study:

1. Wood plastic composites contained steamed fiber showed the lowest water absorption and thickness swelling after 7 weeks of immersion. Steaming reduced water absorption and thickness swelling content by 18.1% and 25.9%, respectively.
2. The mechanical properties, such as tensile, flexural and impact strength, tensile and flexural moduli were increased by 11.2%, 3.5%, 5.4%, 8.2%, and 4.6% respectively.
3. The results showed steaming affected significantly on improvement of bonds between fibers and matrix. It improves the physical and mechanical qualities of composites by eliminating hemicelluloses and destroying fiber bundles.

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