Mechanical Properties and Morphology of Wood Plastic Composites Produced with Thermally Treated Beech Wood

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The effect of thermal-treatment severity of wood on the mechanical and morphological properties of wood plastic composites (WPCs) was investigated. Wood chips were first heat treated at 120, 150, or 180 ºC for 30 or 120 min under saturated steam in a digester. The composites were composed of thermally treated and untreated wood flour, polypropylene, and a coupling agent, produced by melt compounding and then injection molding. The thermal-treatment of the beech wood improved some mechanical properties of the WPCs, depending on the treatment-time and temperature. The SEM micrographs of the composites showed that the outer surface of the wood fiber was coated by a section of amorphous lignin. The SEM images showed that the WPCs produced from the wood treated at 150 ºC for 30 min had considerably fewer holes and many broken fiber ends embedded in the polymer matrix, indicating better compatibility between the wood flour and the polymer matrix. Based on the results of the mechanical testing of the WPCs, the optimum thermal-treatment for WPC production was 150 °C for 30 min.

Keywords: Thermal; Heat treatment; Mechanical properties; Wood-polypropylene

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INTRODUCTION

Wood heat treatment has increased significantly in the last few years and is still growing as an industrial process to improve some properties of wood (Esteves and Pereira 2009). This method produces a new, environmentally-friendly material (Kocaefe *et al.* 2008; Gunduz *et al.* 2010; Garcia *et al.* 2012) free of the disposal hazards present in wood treated with different preservatives (Hill 2006). Many researchers have used heat treatment to improve wood properties (Seborg *et al.* 1953; Kollmann and Schnider 1963; Kollmann and Fengel 1965; Fengel 1966a, b; D'Jakonov and Konepleve 1967; Nikolov and Ensev 1967; Noack 1969; Burmester 1973; Burmester 1975; Rusche 1973a, b; Giebeler 1983; Hillis 1984). These studies reported that heat treatment at different temperatures decreased the equilibrium moisture content, increased dimensional stability (Tiemann 1920; Hu *et al.* 2013), increased mass loss and colour changes (Hakkou *et al.* 2005; Esteves and Pereira 2009), increased biological durability (Boonstra 2008; Hu *et al.* 2013), and decreased some mechanical properties, as well as the wettability of wood (Esteves and Pereira 2009), though in some studies these latter two dimensions increased (Kaboorani and Englund 2010).

Research on the thermal modification of wood-based composites has increased over the last decade (Mohebby and Ilbeighi 2007; Ayrilmis *et al.* 2011a). Many reports have been published about using thermal-treatment to transfer the heat required to induce the chemical changes in the wood cell wall components that affect the physical and mechanical properties of wood (Liu *et al.* 1994; Rowell and LeVan-Green 2005; Kaboorani *et al.* 2008; Kazemi Najafi *et al.* 2008; Ayrilmis *et al.* 2011a).

Wood fibers are primarily responsible for the dimensional stability and mechanical properties of wood plastic composites (WPCs). Usage of wood fibers or wood flour as filler in wood plastic composites has expanded in recent years. The main problem with making these composites is that wood fibers have polarity whereas thermoplastics are non-polar, and this contrast leads to weak bonds between filler and matrix (Eslam *et al.* 2011). Thermal treatment of wood-fibers can decrease their polarity and improve the compatibility between wood fibers and polymer matrix (Ayrilmis *et al.* 2011). Previous studies showed that the dimensional stability of WPCs could be improved by thermal-treatment of wood particles or fibers (Ayrilmis *et al.* 2011b; Hosseinaei *et al.* 2012; Ozdemir *et al.* 2014). However, the mechanical properties of WPCs have not been extensively investigated as a function of thermal-treatment of wood. The main objective of the present study was to determine the effects of thermal-treatment on the mechanical and morphological properties of WPCs.

EXPERIMENTAL

Thermal-treatment of Wood Chips and Preparation of Wood Flour

Before the preparation of the composites, beech lumber pieces (*Fagus orientalis* L.) were chipped by a drum-type chipper. Prior to heat treatment, the chips were conditioned in the laboratory at room temperature for 3 days. Then, the wood chips were thermally treated at different temperatures (120 °C, 150 °C, or 180 °C) for 30 min or 120 min under saturated steam in a digester. After the thermal-treatment, the chips were conditioned in the laboratory at room temperature for 5 days. The lignocellulosic material used as the reinforcing filler in the composites was Oriental beech wood flour (BWF), which was ground by a grinder. The flour size was between 40 and 60 mesh. The BWF was dried until it contained 0 to 1% moisture in an oven at 103 ± 2 °C for 24 h. The chemical properties of beech wood is given in Table 1.

Table 1. Chemical Analysis of Heartwood and Sapwood Samples from *Fagus orientalis*

Chemical components	Heartwood	Sapwood
	Control	Control
Extractives of Alcohol-Benzene soluble (%)	2.85	2.29
Extractives of Hot-Water Soluble (%)	5.34	4.70
Extractives of 1% NaOH Soluble (%)	22.85	20.46
Cellulose (%)	39.12	40.30
Lignin (%)	24.01	21.11

Thermoplastic and Coupling Agent

The polymer matrix was comprised of V30S polypropylene (PP), with a melt flow index of 16 g/10 min and a density of 0.87 g/cm^3, supplied by Marun Petrochemical Co. (Mahshahr, Iran). The maleic anhydride grafted polypropylene (MAPP, product name:

PPG101), provided by Kimia Javid Sepahan Co. (Tehran, Iran), with a melt flow index of 64 g/10 min, a density of 0.91 g/cm^3, and a grafted maleic anhydride content of 3 wt.%, was used as the coupling agent. Its grafting rate was 0.6% MA grafted with 99.4% PP.

Preparation and Testing of WPCs

The mixing of the raw materials was carried out by a counter-rotating, intermeshing twin-screw extruder (Model T20, Dr. Collin GmbH, Germany), which increased to a barrel temperature of 180 °C over six zones, from the feeding zone to the die zone, at a screw speed of 60 rpm for 14 min. The paste-like compound produced was cooled to 25 °C and then ground to produce suitable granules for further processing. Grinding was carried out in a laboratory mill (WGLS 200/200 Model, Wieser GmbH, Germany), and the granulated materials were dried at 105 °C for 4 h. Test specimens were prepared using an injection molding machine (Model EM80, Aslanian Co., Iran) set at 160 to 180 °C. From each molding operation a set of specimens was produced in order to perform different mechanical tests. The specimens were conditioned at a temperature of 23 °C and a relative humidity of 50% for at least 40 h, according to ASTM D 618-99 prior to testing. The tensile properties (ASTM D638-10), flexural properties (ASTM D790-10), and notched impact strength (ASTM D256-10) were determined according to ASTM standards. Three specimens were used for each type of test. Polypropylene, beech wood flour, and the coupling agent were then weighed and bagged according to the formulations given in Table 2.

Table 2. WPC Formulations Used for Specimen Production

Treatment Code	Beech Wood Flour (BWF) Content (wt.%)	Polypropylene (PP) Content (wt.%)	MAPP* (wt.%)
WPC-control	50	47	3
WPC-30min-120 ºC	50	47	3
WPC-30min-150 ºC	50	47	3
WPC-30min-180 ºC	50	47	3
WPC-120min-120 ºC	50	47	3
WPC-120min-150 ºC	50	47	3
WPC-120min-180 ºC	50	47	3

* MAPP = maleic anhydride grafted polypropylene.

Morphological Analysis of WPCs

The morphology of the tensile fracture surfaces of the WPCs was studied by means of scanning electron microscopy (SEM, Vega\\Tescan) at an acceleration voltage of 10 kV. The test specimens were attached to an aluminum stub and spattered with gold to eliminate the electron charging effects.

RESULTS AND DISCUSSION

The results of Duncan's multiple range test indicated that thermal-treatment of wood had a significant effect on the mechanical properties of the WPCs. The WPCs produced from wood treated at 150 ºC for 30 min and 120 ºC for 120 min had the same flexural strength values and higher than other treated WPCs (Fig. 1). All the WPCs showed higher flexural strength than the control WPC. Thermal-treatment of wood improved the flexural strength to a greater degree than it did the flexural modulus. The

flexural modulus of the WPCs produced with wood treated at 120 ºC or 150 ºC for 30 min were considerably higher than that of the other treated WPCs and control WPC. The treatments 30 min at 180 ºC, 120 min at 120 ºC, and 120 min at 150 ºC did not have significant difference in the flexural modulus as compared to the control. The WPCs produced from wood treated at 180 ºC for 30 min or 120 min had lower flexural modulus than the control WPC. This result showed that the flexural properties of the WPCs considerably decreased as the thermal-treatment duration increased from 30 min to 120 min. The significant differences ($p<0.05$) between some group averages for the mechanical properties are shown in Figs. 1 through 5. The different letter designations in the figures mean that there were significant differences ($p<0.01$) for the mechanical properties among the WPC groups according to Duncan's multiple range test.

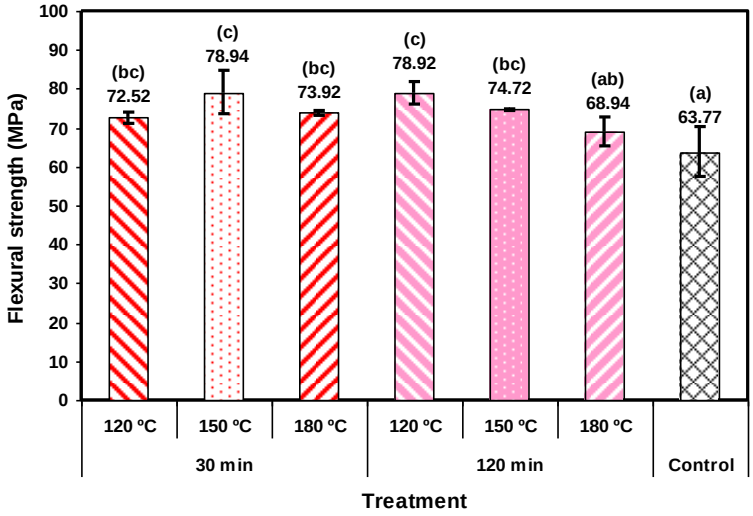

Fig. 1. The average and standard deviation of the flexural strength of the WPCs. The same letters in each bar show that there is no significant difference ($p<0.05$) between the WPC groups.

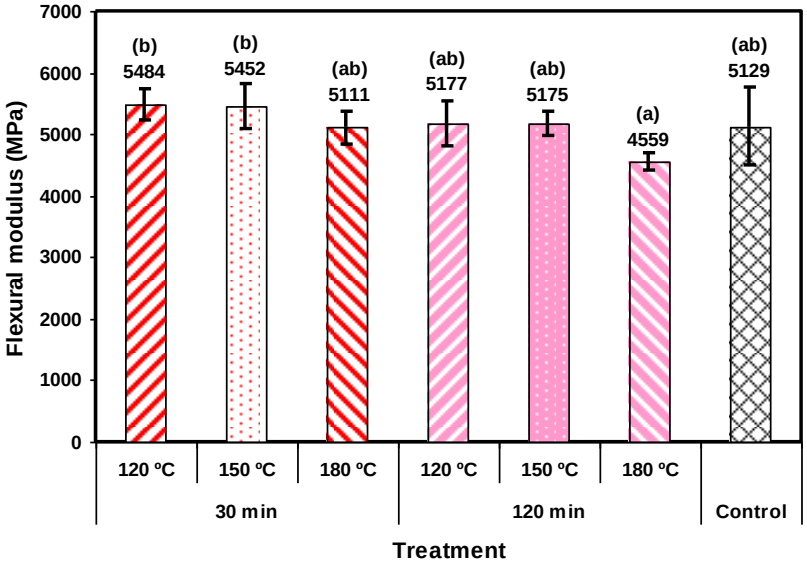

Fig. 2. The average and standard deviation of the flexural modulus measurements of the WPCs. The same letters in each bar show that there is no significant difference ($p<0.05$) between the WPC groups.

As shown in Figs. 1 and 2, the flexural strength and flexural modulus of the WPCs produced with thermally treated wood were higher than those of the control WPCs, with the exception of the flexural modulus of the WPC produced from the wood treated for 120 min at 180 ºC. This phenomenon was probably due to the increase in the amount of degraded hemicelluloses, which were hydrolyzed during the thermal-treatment, decreasing the hygroscopicity of the wood. Thermal-treatment of wood decreases the number of hydroxyl groups in the hemicelluloses, which reduces the polarity of the wood (Hill 2006; Mohebby and Ilbeighi 2007).

Wood contains large amounts of hydroxyl groups, which make them hydrophilic and therefore incompatible with hydrophobic thermoplastics (Kabir *et al.* 2012). This incompatibility results in poor interfacial adhesion between the matrix and filler, which is manifested as a reduction of mechanical properties because stress cannot be transferred properly from the matrix to the fibers (Kazayawoko *et al.* 1999). Wood flour with lower polarity is more compatible with thermoplastics, resulting in better dispersion of the wood flour in the polymer matrix and improved wettability of the fibers with the polymer (Hosseinaei *et al.* 2012). Improved interfacial adhesion through mechanical interlocking and better transfer of stress from the matrix to the fibers may be another reason for the higher flexural strength and flexural modulus of the WPCs produced from thermally treated wood. Raj *et al.* (1990) reported that thermal-treatment of wood fibers reduced the surface energy of cellulose fibers and improved the interfacial compatibility between the wood and the polymer, which increased the flexural strength and flexural modulus of the WPCs. The considerable decrement in the flexural properties of the WPCs produced from wood treated at 180 ºC for 120 min could be related to formation of soluble acidic chemicals; such as formic acid and acetic acid, from the hemicellulose degradation (Ayrilmis *et al.* 2011a).

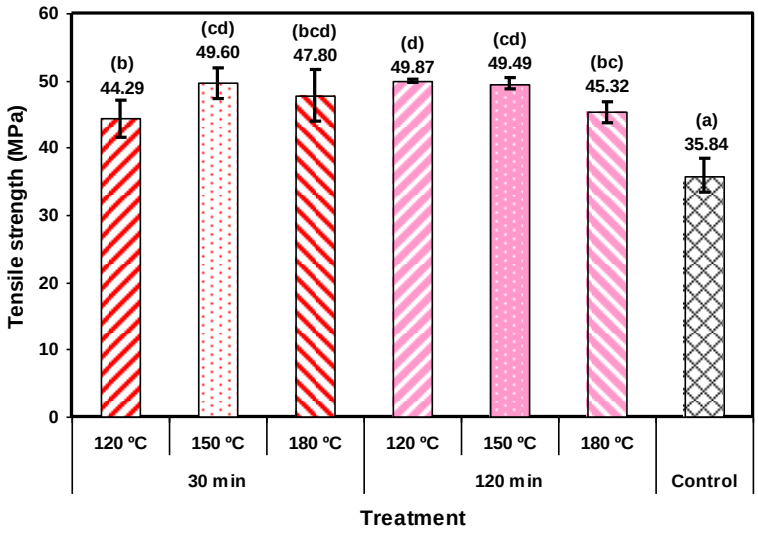

Fig. 3. The average and standard deviation of the tensile strength measurements of the WPCs. The same letters in each bar show that there is no significant difference ($p<0.05$) between the WPC groups.

Thermal-treatment of the wood significantly improved the tensile strength of the WPCs. The highest tensile strength was found in the WPCs produced from the wood treated for 120 min at 120 °C. The results showed that there was no significant difference

between the tensile strength values of the WPCs produced from wood treated at 150 °C and 180 °C for 30 min. A similar result was observed for the treatments at 150 °C and 180 °C for 120 min. In Fig. 4, it is obvious that there were no significant differences among the tensile modulus of the control specimens and those of treatments 30 min at 180 ºC, 120 min at 150 ºC, and 120 min at 180 º C. Based on Fig. 4, only specimens treated at 150 ºC for 30 min showed significantly higher tensile modulus than control ones.

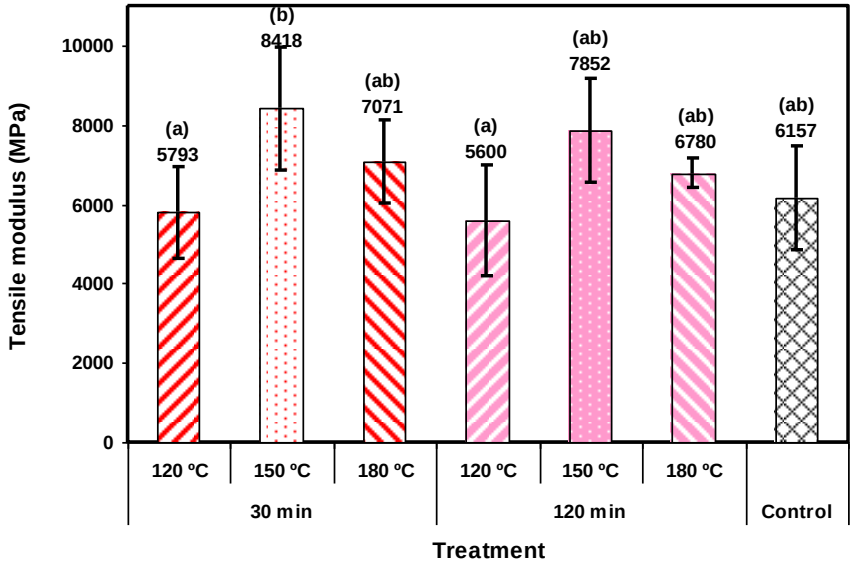

Fig. 4. The average and standard deviation of the tensile modulus measurements of the WPCs. The same letters in each bar show that there is no significant difference ($p<0.05$) between the WPC groups.

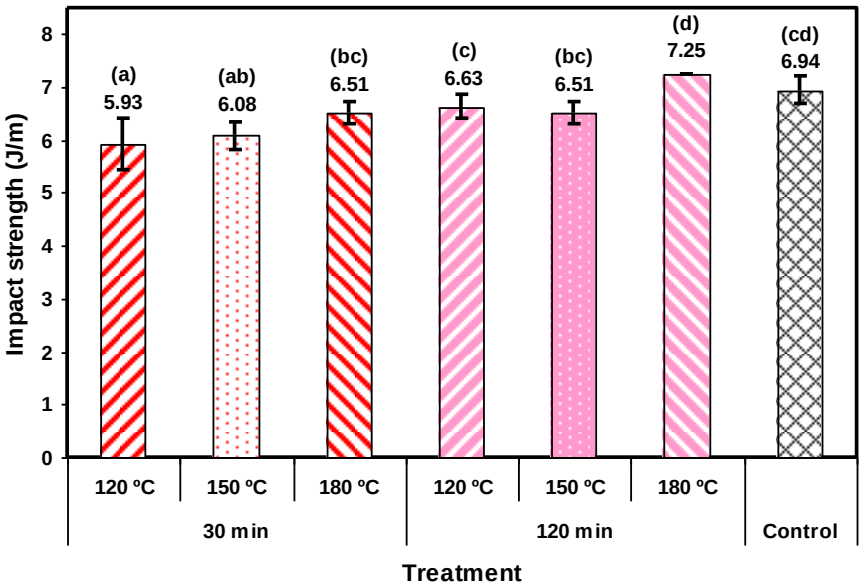

Fig. 5. The average and standard deviation of the impact strength measurements of the WPCs. The same letters in each bar show that there is no significant difference ($p<0.05$) between the WPC groups.

The results of Duncan's multiple range test indicated that the thermal-treatment had a significant effect on the impact strength of the WPCs. There were no significant differences in the impact strength values of the WPCs between the 30 min at 180 ºC and 120 min at 150 ºC treatments.

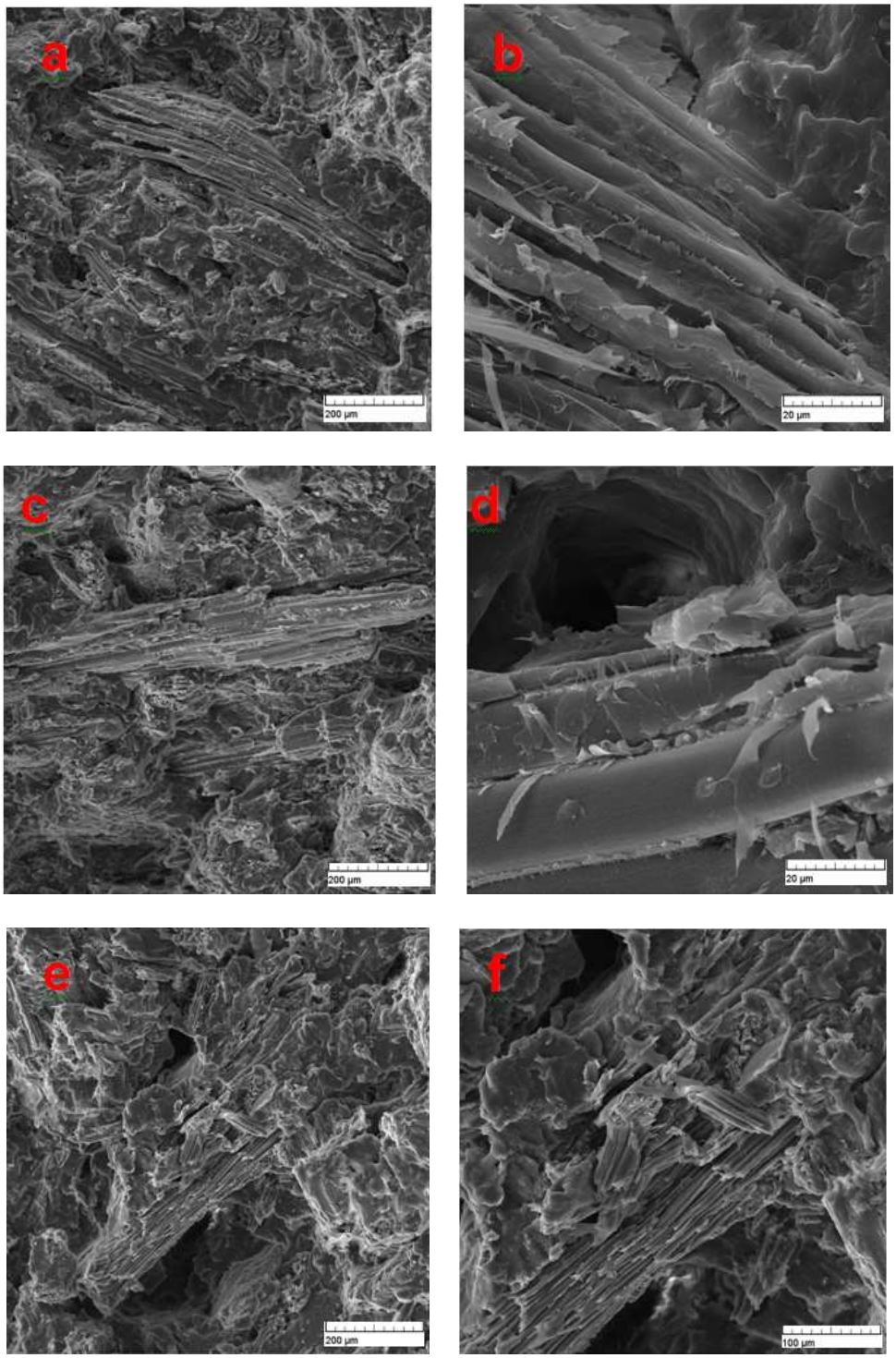

Fig. 6. SEM micrographs of the fracture surfaces in the composites under different thermal-treatments: 30 min at 150 ºC (a, b), control (c, d), and 120 min at 120 ºC (e, f)

As shown in Fig. 5, the WPCs produced from the wood treated at 120 ºC for 30 min showed lower notched impact strength than the control WPC specimens. The impact resistance of the WPCs produced from the wood treated at 180 ºC for 120 min increased by 4.5% over the control WPC specimens. This increase was mainly attributed to the high compatibility between the wood and the polymer matrix due to the softening of lignin. Furthermore, WPCs produced from the wood treated at 150 ºC for 30 min had slightly lower average impact resistance than the WPCs produced from the wood treated at 180 ºC for 120 min. Thus, it was expected that the 30 min at 150 ºC treatment caused a reduction in the adhesion between the wood filler and the polymer matrix compared to the 120 min at 180 ºC specimens.

The SEM micrographs displayed in Fig. 6 showed that the outer surfaces of thermally treated wood fibers were surrounded by some amorphous lignin, which increased the surface area of the solids within the WPCs and improved the bonding efficiency of the polymer. As known, the lignin is a thermoplastic biopolymer in wood. The softening point of lignin (spherical droplets) after steaming treatment is believed to be lower than that of the original material. This makes it possible for a plastic flow to occur *in situ* (Anglès *et al.* 2001). It is believed that the softening point of lignin after thermal-treatment decreased and a possible plastic flow in situ may occur, making the lignin more accessible to the fiber surface (Hsu *et al.* 1988; Anglès *et al.* 2001). It is also evident from Figs. 6a and 6b that WPCs produced from the wood treated at 150 ºC for 30 min had considerably fewer holes and many broken fiber ends embedded in the polymer matrix, indicating better compatibility between the wood flour and the polymer matrix. In the control WPCs, several holes were observed. These defects appeared to result from the fiber pulling out from the matrix, indicating poor bonding between the wood flour and the polymer matrix (Fig. 6). The number of such holes in the control WPCs (Figs. 6c and 6d) was larger than that of the WPCs produced from the wood treated at 120 ºC for 120 min (Figs. 6e and 6f). The difference could be due to the incompatibility between the polymer matrix and the untreated wood flour, as a result of the higher amount of hydroxyl groups in the untreated wood as compared to the treated wood.

CONCLUSIONS

Flexural strength, flexural modulus, and impact strength of the WPCs were significantly affected by the thermal-treatment of beech wood. The WPCs produced from wood treated at 150 ºC for 30 min and 120 ºC for 120 min had the same flexural strength values, which were higher than other treated WPCs. The treatments 30 min at 180 ºC, 120 min at 120 ºC, and 120 min at 150 ºC did not have significant difference in the flexural modulus as compared to the control. The WPCs produced from wood treated at 180 ºC for 30 min or 120 min had lower flexural modulus than the control WPC. This result showed that the flexural properties of the WPCs considerably decreased as the thermal-treatment duration increased from 30 min to 120 min. The SEM images of fractured specimens showed that the WPCs produced from the wood treated at 150 ºC for 30 min had considerably fewer holes and many broken fiber ends embedded in the polymer matrix, indicating better compatibility between the wood flour and the polymer matrix. Based on the results of the mechanical properties of the WPCs, the optimum thermal-treatment for the flexural properties was 150 °C for 30 min while it was found to be 180 °C for 120 min for the impact strength.

ACKNOWLEDGMENTS

The authors are grateful for the support of the Department of Wood Science and Paper Technology, Karaj Branch, Islamic Azad University, Karaj, Iran.

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Article submitted: July 20, 2015; Peer review completed: December 2, 2015; Revised version received: December 8, 2015; Accepted: December 10, 2015; Published: December 17, 2015.
DOI: 10.15376/biores.11.1.1494-1504