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Thermal and mechanical properties of treated and untreated Red Balau (*Shorea dipterocarpaceae*)/LDPE composites

Ruth A. Lafia-Araga¹, Aziz Hassan¹, R. Yahya¹, Normasmira A. Rahman¹, Peter R. Hornsby² and J. Heidarian¹

Abstract

Red Balau saw dust was heat-treated at 180°C and 200°C for 1 h. Treated and untreated wood flour were compounded with LDPE at 9%, 20%, and 37% by weight and molded in an injection molding machine. Thermal and mechanical properties of the resultant composites were investigated as a function of filler loadings and treatment temperature. Thermogravimetric analysis revealed an increase in degradation peak temperature (T_p) of the heat-treated wood and composites. DSC revealed a decreasing trend in the degree of crystallinity (X_c) of the matrix when heat-treated wood was used as filler. On the other hand, untreated wood showed an increase in X_c with increasing wood content. Tensile modulus increased with heat treatment and filler loading. Furthermore, flexural strength and modulus were found to increase with filler loading.

Keywords

wood plastic composites, injection molding, thermal properties, tensile properties, degree of crystallinity

Introduction

Wood plastic composites (WPCs) present a class of materials which combine the favorable performance and cost-effective properties of both wood and plastics. As a result of these attributes, WPCs are seen as a way to increase the value-added utilization of waste wood and wood of low-commercial value. Commonly, compression molding is used in processing WPC. This process has the limitation of producing a simple shape that may not necessarily be compact. However, the injection molding employed in this study results in a compact shape of the finished article. It has an added advantage of being applied in a wider scope especially in industrial/domestic applications. Wood, a natural cellulose composite material of botanical origin is useful for many applications because of its many excellent structural and chemical properties.¹ Incorporating wood into WPC lowers production cost, improves stiffness, and increases the profile extrusion rate of products and acts as an environmental-friendly way of reducing the use of petroleum-based plastics. This is because wood is

cheap, readily available, and renewable. Also, it has good strength-to-weight ratio and esthetic appearance.^{2,3} However, because of the moisture absorbing tendency in wood, it suffers a number of disadvantages. Poor resistance against fungal and insect attack, swelling, and shrinkage resulting from water absorption and desorption are some of these shortcomings.^{4,5} Many studies have been carried out to improve the unfavorable properties of wood. These include chemical and thermal modifications. Others are densification, surface

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treatments, corona or plasma discharge, and enzymatic modifications.^{6–8}

Thermal treatment has been reported to be an effective method to improve wood dimensional stability and/or its durability. High-temperature treatment of wood results in several changes occurring in the wood chemistry. The hemicellulose is the first structural compound to be thermally affected even at low temperatures. The degradation starts by deacetylation, so that the released acetic acid acts as a depolymerization catalyst, which further increases polysaccharide decomposition. This acid catalyzed degradation leads to the formation of formaldehyde, furfural, and other aldehydes. Thermal softening of cell wall matrix, mainly lignin, also sets in with cross-linking occurring between carbohydrate polymers and/or between lignin and carbohydrate polymers, resulting in an increase in the crystallinity of amorphous cellulose with consequent improvement in dimensional stability and decreased hygroscopicity of wood.^{5,9}

The use of thermally modified wood as a reinforcement in WPC is gradually gaining attention. The essence of thermal treatment of wood in WPC is to enhance the compatibility of the wood and the matrix by reducing hydrophilicity of wood. However, wood is thermally unstable, degrading at temperatures above 200°C. Therefore, lower processing temperatures are generally permissible because of the possibility of lignocellulosic degradation and/or the possibility of volatile emissions that could affect composite properties. Processing temperatures are, thus, limited to about 200°C, although it is possible to use higher temperatures for short periods.¹⁰

It has been reported that exposing wood to higher temperatures than normal drying to remove moisture should, in principle, result in wood with higher thermal stability because the vulnerable components (hemicelluloses) are expected to have been decomposed or at least gone through some structural changes. Also, heat treatment modifies the polar nature of wood possibly resulting in better compatibility between wood and the polymer matrix, thus leading to high quality and thermally stable composites.^{11,12} Furthermore, wood flour encounters intense heat during composite processing. Therefore, it is important to determine the extent of degradation during treatment and composites fabrication using thermal analysis. In addition, semi-crystalline thermoplastics composites have properties that are a complex function of a number of variables, such as mechanical properties, shape, size, orientation and distribution of the filler phase, and the mechanical properties of the matrix. Also, degree of crystallinity, size, and number of spherulites are factors affecting the mechanical properties of polymer matrices. Incorporation of fillers to low-density polyethylene

(LDPE) matrix can affect its crystallization and consequently, the mechanical properties which are dependent on the crystalline structure.¹³

It is an established fact that heat treatment could result in loss of mechanical strength of wood.^{4,9} Consequently, using heat-treated wood as filler in WPC should, in principle, worsen the properties of WPC. On the other hand, the changes imposed by heat treatment reduce the polarity of wood and makes it a more compatible material with non-polar thermoplastic matrix. Considering this feature, heat-treated wood should be able to improve WPC properties more than untreated ones.¹¹ Therefore, this article is aimed at modifying Red Balau saw dust with heat treatment and studying the effects on the thermal and mechanical properties of the resultant composites.

Experimental

Materials

Red Balau (*Shorea dipterocarpaceae*) saw dust was obtained from a local saw mill in the Klang Valley, Selangor, Malaysia. It was milled between 40 and 100 mesh (400–150 µm) sizes using a locally fabricated mill. Commercially available LDPE (Titanlene LDI300YY), with a density 920 kg/m³, molecular weight 350,000–380,000 g/mol, and melt flow index 20 g/10 min as measured by ASTM D 1238, supplied by Titan Petchem (M) Sdn. Bhd., Malaysia, was used as the matrix.¹⁴

Chemical composition of wood flour

Acid insoluble lignin was determined using ASTM D 1106 standard.¹⁵ Hemicellulose and cellulose were analyzed according to the method described by Rowell et al.¹⁶ Extractives of moisture free samples were assessed using ASTM D 1105 standard method with slight modifications.¹⁷ After extracting a known weight with 95% ethyl alcohol and ethanol–toluene solutions for 6 h each, the percentage mass difference was calculated. A known weight of the extracted, oven-dried moisture free wood was also taken through three rounds of hot water extractions. The combined percentage weight of the extractives was then taken. The ash content was determined according to the ASTM D 1755.¹⁸

Wood pre-treatment

Untreated wood saw dust was dried in an oven at 60°C for 48 h and stored in sealed LDPE bags over dried silica gel in a desiccator for not more than 24 h prior to compounding. Portions of untreated wood that has not been dried was subjected to 180°C and 200°C

temperatures in a vacuum oven for 1 h effective treatment time.

Compounding

LDPE, untreated, and the heat-treated wood flours were pre-mixed in different compositions in 200 g portions and compounded in a co-rotating twin screw extruder (Brabender KETSE 20/40 Lab Compounder, Germany, with screw diameter and L/D ratio of 20 mm and 40, respectively). Temperatures were set between 150°C and 155°C along the barrel zones and the screw speed used was 250 rpm. The melt pressure varied between 34 and 39 bar depending on the wood content, while the die temperature was between 164°C and 178°C. Vacuum venting was used to extract volatile compounds. The samples were extruded out through a circular die of 3 mm in diameter. The extruded strand was cooled in a water bath and pelletized. The pellets were then oven-dried at 80°C for 24 h and stored in sealed plastic bags for injection molding. Composites were prepared at three different particle loadings of 9%, 20%, and 37% by weight.

Injection molding

The pellets were injection molded into tensile (ASTM 638)¹⁹ test pieces using the BOY 55 M injection molding machine at a barrel temperature of between 150°C and 155°C, an injection pressure of 100–120 bar, and mold temperature of 25°C.

Thermogravimetric analysis

Thermogravimetric analysis (TGA) measurements were carried out using a Perkin Elmer TGA 6 on 8–10 mg samples of each of the composites in a ceramic crucible, over a temperature range from 30°C to 700°C at a heating rate of 10°C/min. The tests were conducted in a nitrogen atmosphere at a flow rate of 20 mL/min.

Differential scanning calorimetry

Differential scanning calorimetry (DSC) was undertaken using a Perkin Elmer Diamond DSC (Hyper DSC) on 5–7 mg samples crimped in aluminum pans.

Each sample was characterized between –50°C and 200°C at a scan rate of 10°C/min. Prior to this measurement, the specimen was heated to 200°C and held at this temperature for 2 min to erase previous thermal history, then cooled under a nitrogen atmosphere at a flow rate of 20 mL/min, before reheating. The melting temperature, T_m , was taken as the peak of the endothermic curve, while the crystallization temperature, T_c , was the peak of the exothermic curve.

Tensile testing

Tensile tests were carried out using a universal testing machine (Instron 5569) equipped with a load cell of 50 kN and a mechanical extensometer according to ASTM 638.¹⁹ Unconditioned specimens were tested at room temperature at a cross-head speed of 5 mm/min (10%/min). A zero span of 50 mm was chosen for the extensometer. Ten specimens were tested and the average of at least six reproducible results were presented. The tensile modulus was calculated at 0.5% strain.

Flexural testing

The same instrument used for tensile testing was used for the flexural testing but in three point bending according to ASTM D-790.²⁰ Unconditioned tensile test specimens were tested at room temperature with a span of 50 mm. Samples were tested to a maximum deflection of 30 mm at a cross-head speed of 1.28 mm/min. Ten specimens were tested and the average of at least six reproducible results was recorded.

Scanning electron microscopy

The fractured surface of the tensile test specimens was observed using the Leica S440 scanning electron microscope under an acceleration voltage of 10 kV after gold sputtering to a thickness of 0.014 µm.

Results and discussion

Chemical composition

Table 1 illustrates the percentage composition of lignin, carbohydrates, extractives, and ash in the wood flour

Table 1. Chemical composition of Red Balau saw dust

Treatment temperature (°C)	Lignin (%)	Hemicellulose (%)	Cellulose (%)	Extractives (%)	Ash (%)
Untreated	29	29	41	2	2
180	29	28	42	2	2
200	31	25	42	2	2

samples. The results suggest that carbohydrates were more susceptible to thermal degradation than lignin at the treatment temperature. Hemicellulose degraded faster than cellulose as the values decreased from 29% in untreated wood to 28%, and 25% when wood is subjected to heat treatment at 180°C and 200°C, respectively. This may possibly be because cellulose is more crystalline than hemicellulose which confers better resistance to thermal degradation. Zaman et al.²¹ have suggested that cellulose (a linear homo-polysaccharide of β -D-glucopyranose) is more thermally stable than hemicellulose. On the other hand, a steady increase in lignin content was observed as the treatment temperature increased. This clearly indicates that lignin is more thermally stable than carbohydrates and is in agreement with the literature.^{22,23} Total extractives were also determined for the untreated and heat-treated wood samples with no significant difference.

Thermogravimetric analysis

TGA and differential thermogravimetric (DTG) thermograms of untreated and heat-treated wood flour are presented in Figure 1, with principal events from these thermograms listed in Table 2. The curves exhibit two mass loss peaks. The first occurs at about 100°C and is attributed to the evaporation of moisture from the wood. The second peak, at approximately 200°C to 400°C is due to the degradation of hemicellulose, cellulose, and lignin. Lignocellulosic materials, being chemically active, decompose thermo-chemically in the range of 150°C to 500°C. Hemicellulose degrades between 150°C and 350°C, cellulose decomposes between 240°C and 350°C, and lignin between 250°C and 500°C.^{24,25} The effect of heat treatment on thermal stability of wood is also shown in Figure 1. Untreated wood has the lowest thermal stability, degrading at a lower temperature and at a faster rate than treated material. Wood treated at 200°C reveals a higher resistance to heat. Treating wood at 180°C indicates an intermediate thermal property between the untreated and the wood treated at 200°C. However, percentage mass losses of 49%, 50%, and 50% were obtained for untreated, 180°C treated and wood treated at 200°C, respectively, with corresponding degradation peak temperatures of 351.4°C, 355.9°C, and 362.4°C (Table 2). The increase in degradation peak temperature, T_p , of heat-treated wood over untreated ones is an evidence of improvement in thermal resistance of heat-treated wood. This observation may be due to the removal of hemicellulose by heat treatment thereby rendering the wood more thermally stable.²³ In addition, heat treatment of wood may improve the thermal properties by reducing its heat transfer co-efficient.²⁶ It should also be noted here that neat LDPE is more thermally stable

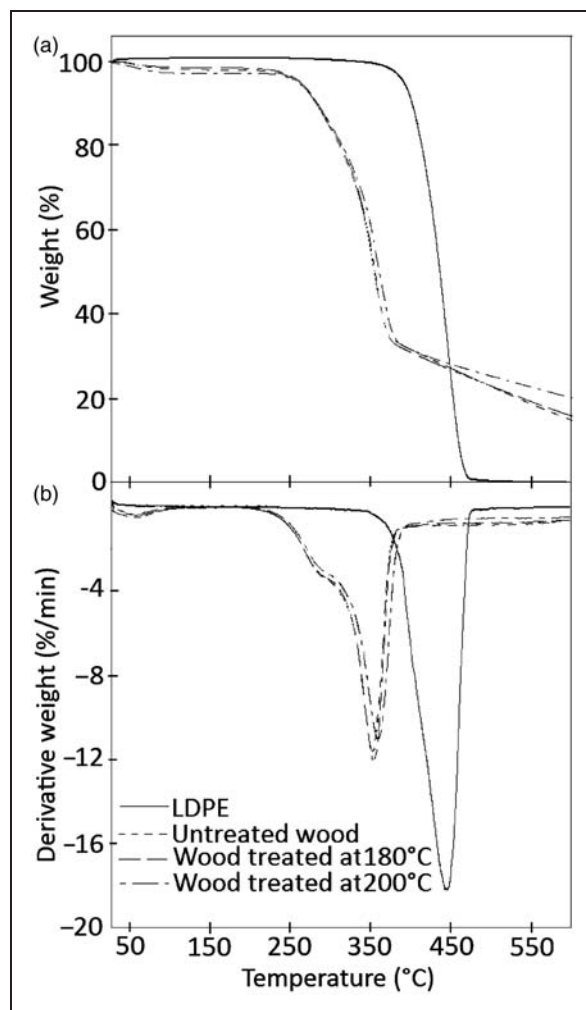
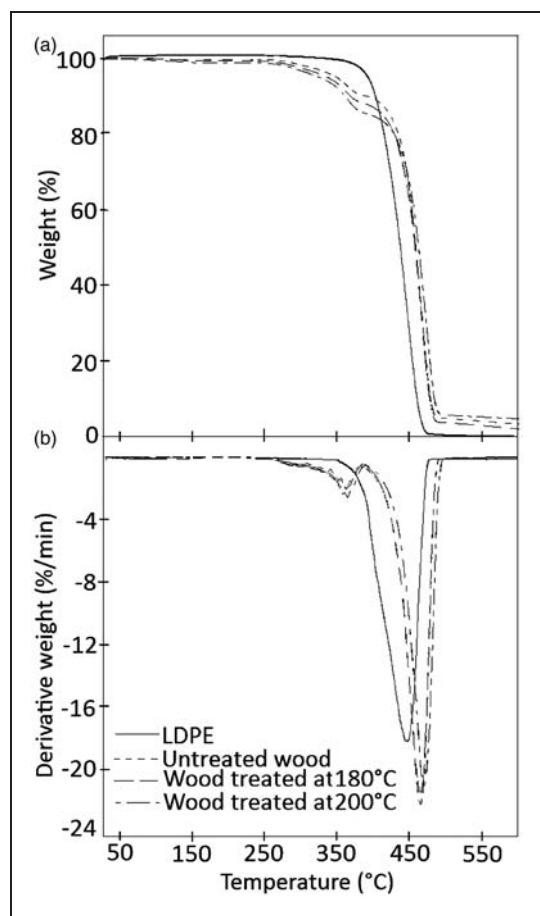


Figure 1. (a) TGA and (b) DTG thermograms of LDPE, untreated and heat-treated wood.

than the wood (Figure 1). However, when LDPE is compounded with the wood, a different thermal property profile is displayed. Figure 2 shows the TGA and DTG curves for neat LDPE and the composites from untreated and heat-treated wood at 20 wt% filler loading. The thermal degradation patterns of the LDPE and the resultant composites reveal that the neat matrix is less thermally stable than the composites. The neat LDPE degrades faster at a temperature range between 350°C and 480°C, with a lower initial mass loss. From Figure 1, it can be seen that the degradation of wood, though starting at a lower temperature than the neat matrix and composites, is more gradual with a broader range. Furthermore, the degradation profiles of the composites indicate an improvement over the wood and the neat matrix, which degrade between 250°C and 500°C, over a wider range. The T_p values of the composites were also found to have appreciably increased over the values obtained for the neat matrix and the wood (Table 2). Composites at 20 wt% from

Table 2. TGA parameters of wood, LDPE, and composites

Filler loading (wt%)	Treatment temperature (°C)	T_p (°C)	Onset temperature, T_{onset} (°C)	$T_{50\%}$ (°C)	Degradation temperature (°C)
100	–	351.2	255.7	352.5	213.5–397.8
100	180	356.6	275.6	355.7	207.1–391.7
100	200	362.4	297.0	361.6	226.5–400.7
LDPE	–	446.0	398.3	436.5	350.0–479.0
	–	466.1	424.1	458.1	305.8–492.3
9	180	448.7	361.3	434.9	250.9–478.6
	200	457.3	359.8	443.0	255.0–487.0
	–	465.3	420.2	457.8	279.4–494.8
20	180	465.5	395.6	456.5	257.2–493.6
	200	471.0	417.7	461.7	258.7–500.1
	–	462.0	397.9	447.2	245.7–488.5
37	180	458.0	366.7	440.1	232.1–488.5
	200	462.6	384.6	439.8	231.4–492.4

**Figure 2.** (a) TGA and (b) DTG thermograms of LDPE and 20 wt% composites from untreated and heat-treated wood.

wood treated at 200°C has the highest T_p value of 471.0°C, which is higher than that of wood and the neat LDPE, with maximum T_p values of 362.4°C and 446.0°C, respectively. This is indicative of an improvement in the thermal stability of the composites.²⁷

In addition, the temperature at which 50% mass of the composites degraded, $T_{50\%}$, decreases with filler content for untreated wood composites (Table 2). However, for the heat-treated wood composites, $T_{50\%}$ increases up to 20 wt%, then decreases sharply at 37 wt%. The possible reason for this trend could be the higher amount of wood present at 37 wt%. This implies that 20 wt% may be the optimum composition at which the thermal stability of heat-treated wood composites is highest. It should also be noted that this composition also gives the highest T_p for the composites from heat-treated wood. Reinforcing polymers with heat-treated wood has proved to increase the thermal resistance of the neat matrix.¹² However, as the wood content increases, T_p is found to decrease reasonably for all compositions containing untreated wood. This is as a result of the lower thermal stability of wood compared to LDPE.²⁸ Heat-treated wood composites exhibits an increase in T_p until 20 wt%, then decreases at 37 wt%. Notwithstanding this trend, composite from wood treated at 200°C seem to impact more positively on the thermal stability of the matrix than that from 180°C treated wood, which in turn shows better thermal stability than composites from untreated wood (Figure 2). This is because lignin, the most thermally stable component in wood will not degrade appreciably at 200°C. Therefore, treatment at 200°C, which could

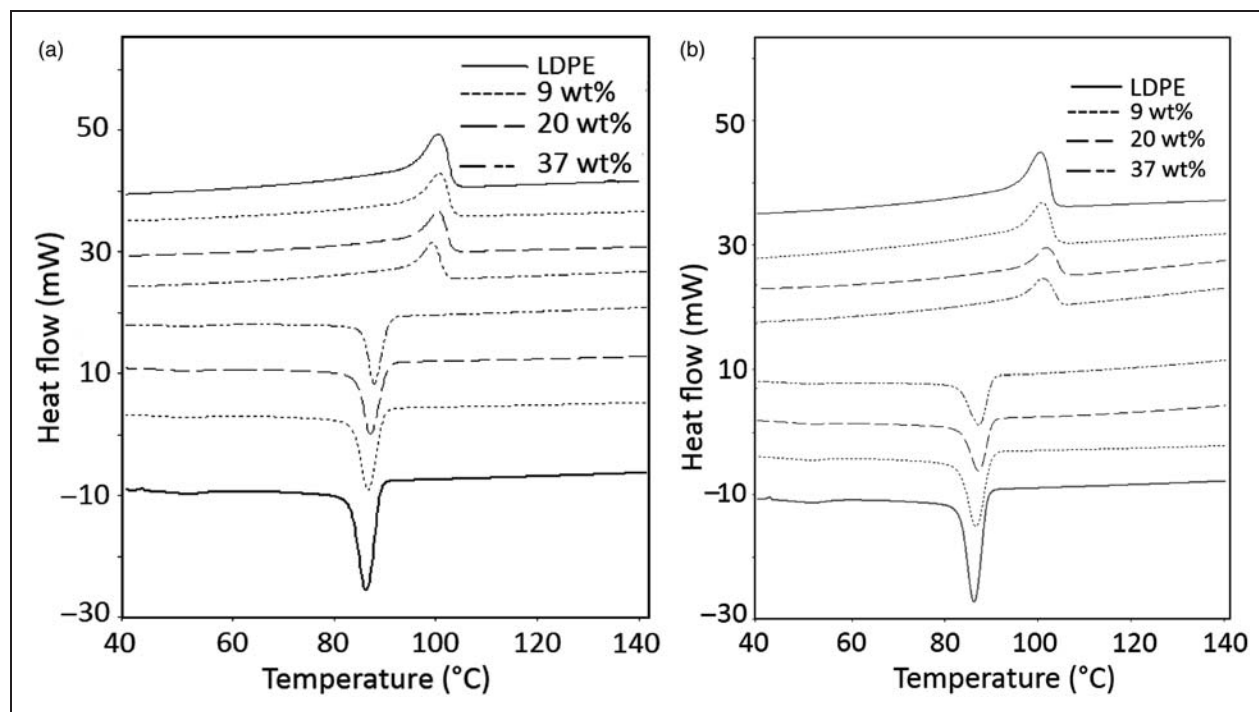


Figure 3. Heating and cooling DSC thermograms of: (a) untreated and (b) 200°C treated wood/LDPE composites.

Table 3. DSC parameters of LDPE and composites

Filler loading (wt%)	Treatment temperature (°C)	T_m (°C)	T_c (°C)	ΔH_m (J/g)	ΔH_c (J/g)	X_c (%)
0	—	100.4	86.4	97.7	77.4	33
9	—	100.7	86.7	85.9	63.5	32
	180	99.9	86.9	92.2	61.6	35
	200	100.9	86.6	78.6	57.6	30
20	—	100.4	87.2	73.9	57.1	32
	180	99.4	87.2	73.2	59.1	31
	200	101.7	87.3	44.9	57.4	19
37	—	99.3	87.8	65	48.9	35
	180	99.6	87.6	61.8	48.8	34
	200	101	87.2	26	50.4	14

significantly degrade other components of wood, will have little or no decomposition effects on lignin.²³ As a result, wood treated at 200°C has higher lignin content (Table 1) and leads to more thermally stable composites. Also, lignin has been found to exhibit effective antioxidant properties against free radical chain reactions which is the case with the thermal degradation of polyolefins.²⁹

Furthermore, it must be mentioned that at lower filler loading (9 wt%), when the amount of the matrix far outweighs the amount of wood particles present, the thermal property of untreated wood composites was significantly improved over the heat-treated ones.

At this composition, complete encapsulation of the wood particles is possible. Therefore, a better synergy is expected between the matrix and the wood particles resulting in better thermal resistance.

Differential scanning calorimetry

DSC curves corresponding to the cooling and the second heating scans of LDPE and composites from untreated wood and wood treated at 200°C are presented in Figure 3. Data extracted from these curves are given in Table 3. There is no significant change in T_m and T_c as a result of wood treatment and filler

loading. The relative percentage crystallinity of the composites was calculated according to the following equation:³⁰

$$X_c = \frac{\Delta H_m}{\Delta H_m^* W_m} \times 100(\%) \quad (1)$$

where X_c is the degree of crystallinity, ΔH_m the heat of fusion, W_m the weight fraction of LDPE in the composite, and ΔH_m^* the heat of fusion for the fully crystalline LDPE, taken as 293 J/g.³¹

Generally, there is no significant trend in X_c of wood composites, containing untreated wood and wood treated at 180°C as the wood content increases relative to the neat matrix. However, composites containing 200°C treated wood exhibits a decreasing trend in X_c with increase in filler loading. It has been reported that at higher filler loading, the particles act as diluents to the matrix and restrict crystal growth, thereby slowing down crystallization. Therefore, for the wood heat-treated at 200°C, the higher the wood fraction, the more is the resistance to formation of crystal structures,³⁰ leading to lower X_c . This may also be the reason for the increasing value of ΔH_c as the wood content increases, indicating that higher energy is released during crystallization.

Tensile properties

The tensile modulus and strength are shown in Figures 4 and 5. Tensile modulus increases with both heat treatment and filler loading (Figure 4). At 9 wt%, composites from wood treated at 180°C has a slightly lower modulus value compared to the untreated wood composites. Untreated wood composites show an increasing trend in tensile modulus at all filler loadings. Percentage increase of 69%, 100%, and 309% are obtained for 9%, 20%, and 37% by weight, respectively, compared to the pure matrix (0.23 GPa). However, the effect of the heat-treated wood is more pronounced at higher filler levels. A percentage increase in tensile modulus from 57% for 9 wt%, through 183% for 20 wt% to 400% for 37 wt% is observed for wood treated at 180°C relative to the neat LDPE. Increase in tensile modulus of composites from heat-treated wood relative to untreated ones suggests better adhesion/wetting of the filler by the matrix. The tensile strength of the composites decreases with increasing filler loading (Figure 5). This may be due to the fact that as the wood content increases, especially at 37 wt%, wetting problem becomes more prominent and an increase in wood-wood contact results. During testing, especially in tensile mode, the unwetted wood particles could become

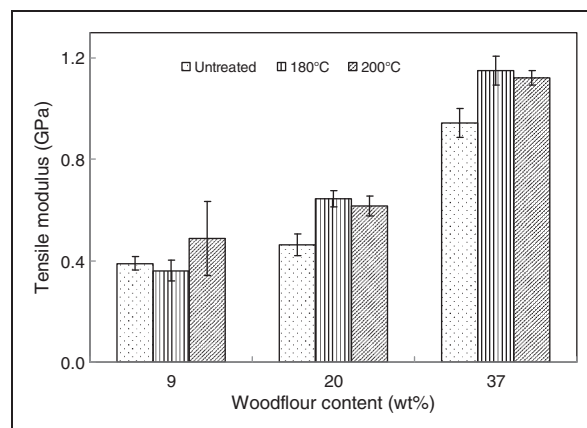


Figure 4. Tensile modulus of treated and untreated wood/LDPE composites.

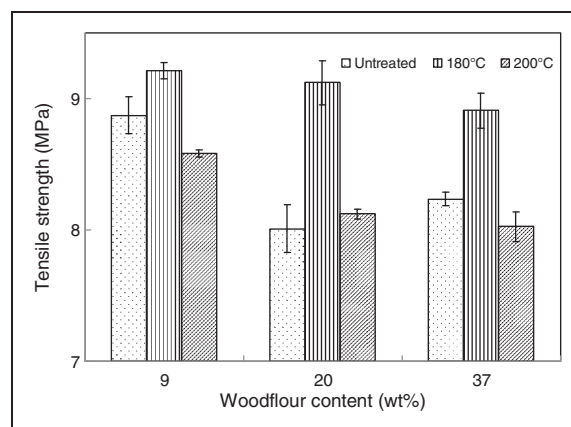


Figure 5. Tensile strength of treated and untreated wood/LDPE composites.

stress concentration areas, leading to premature failure, and consequently, low strength.

Despite the general decreasing trend with increasing filler loading, wood treated at 180°C maintained a consistently higher tensile strength for all volume fractions, indicating that heat treatment at 180°C has improved the compatibility between the hydrophilic wood flour and the hydrophobic polymer matrix to a certain degree. In addition, wood treated at 200°C seems to have the least influence on tensile strength. It is worth noting that composite from wood treated at 200°C also exhibited a trend of decreasing degree of crystallinity for the various compositions. It has been reported that in semi-crystalline thermoplastic composites, degree of crystallinity is an important parameter influencing the mechanical properties.³² Therefore, it is not surprising that it presented the least tensile strength.

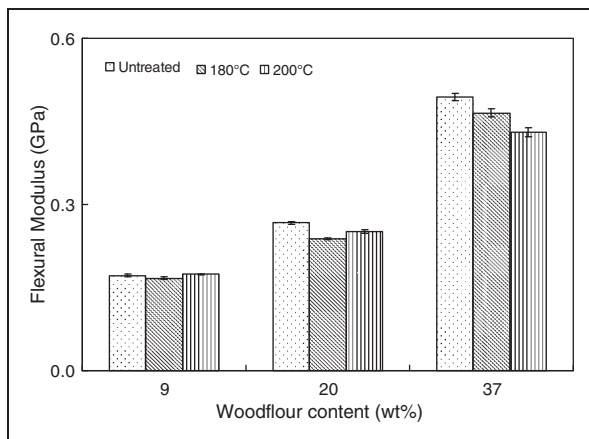


Figure 6. Flexural modulus of treated and untreated wood/LDPE composites.

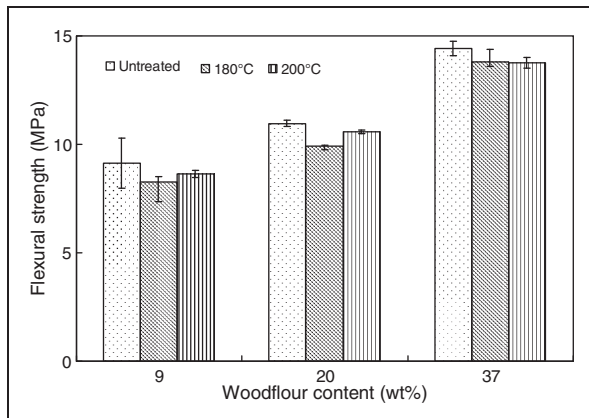


Figure 7. Flexural strength of treated and untreated wood/LDPE composites.

Flexural properties

Results from the flexural tests are presented in Figures 6 and 7. Flexural modulus and strength were found to increase with filler loading with the untreated wood composites exhibiting higher values. This is due to the reinforcing effect of the wood flour on the matrix which resulted in increased stress transfer from the fillers to the matrix. A percentage increase in modulus of 308% was observed for the untreated wood composites at 37 wt% relative to the neat matrix (0.121 GPa). This is in agreement with literature.³³ Wood treated at 180°C increased the flexural modulus to the tune of 284% at 37 wt% compared to the neat polymer.

Considering the heat-treated wood composites, the effects of wood treated at 200°C is more pronounced at lower filler loading (9 wt% and 20 wt%). Whereas, as

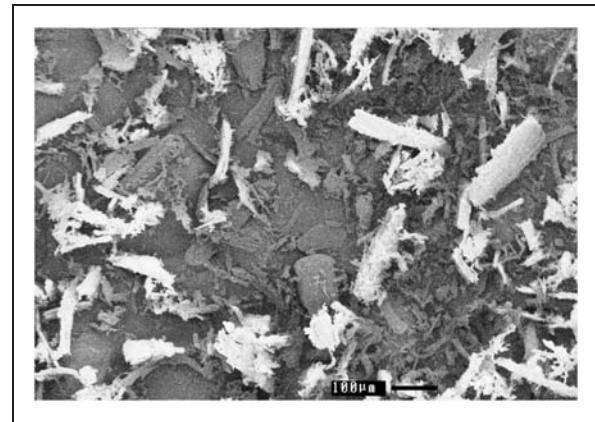


Figure 8. SEM micrograph of wood flour (dispersed on carbon tape).

the wood content increased to 37 wt%, 180°C treatment seems to enhance further the flexural modulus. This is in line with earlier findings where treatment of wood flour at 190°C produced composite with better flexural modulus than treatment at 205°C.¹¹ The same trend is observed for flexural strength.

Scanning electron microscopy

SEM micrographs of wood flour, neat LDPE, 37 wt% untreated, and treated wood composites are presented in Figures 8 and 9. From Figure 8, it can be seen that the wood flour is made up of particles. Furthermore, the neat LDPE exhibits ductile mode of failure (Figure 9(a)), while for the matrix with 37 wt% treated and untreated wood flour, the mode of failure becomes more or less brittle in nature as no sign of ductility is seen on the surfaces of all the composites studied (Figure 9(b)–(d)). In addition, the surfaces of the untreated wood composites have prominent holes due to particle pull out resulting from poor adhesion (Figure 9(b)). Under tensile stress, the particles easily pulled out from the matrix. This may mean that the interface could not effectively transfer the stress. This observation is in agreement with the lower modulus values recorded for the untreated wood composites (Figure 4). Furthermore, for the heat-treated wood composites (Figure 9(c) and (d)), the holes are not quite prominent and particles pull out appears relatively less compared to the untreated ones. The particle surface is slightly rough and nearly uniformly dispersed and embedded within the matrix. Thin films of LDPE covering the wood particles are also seen, thus, better stress transfer should be expected. This is an evidence of better wetting of the heat-treated wood particle by the matrix. This may be responsible for the higher tensile strength and modulus of heat-treated wood composites.

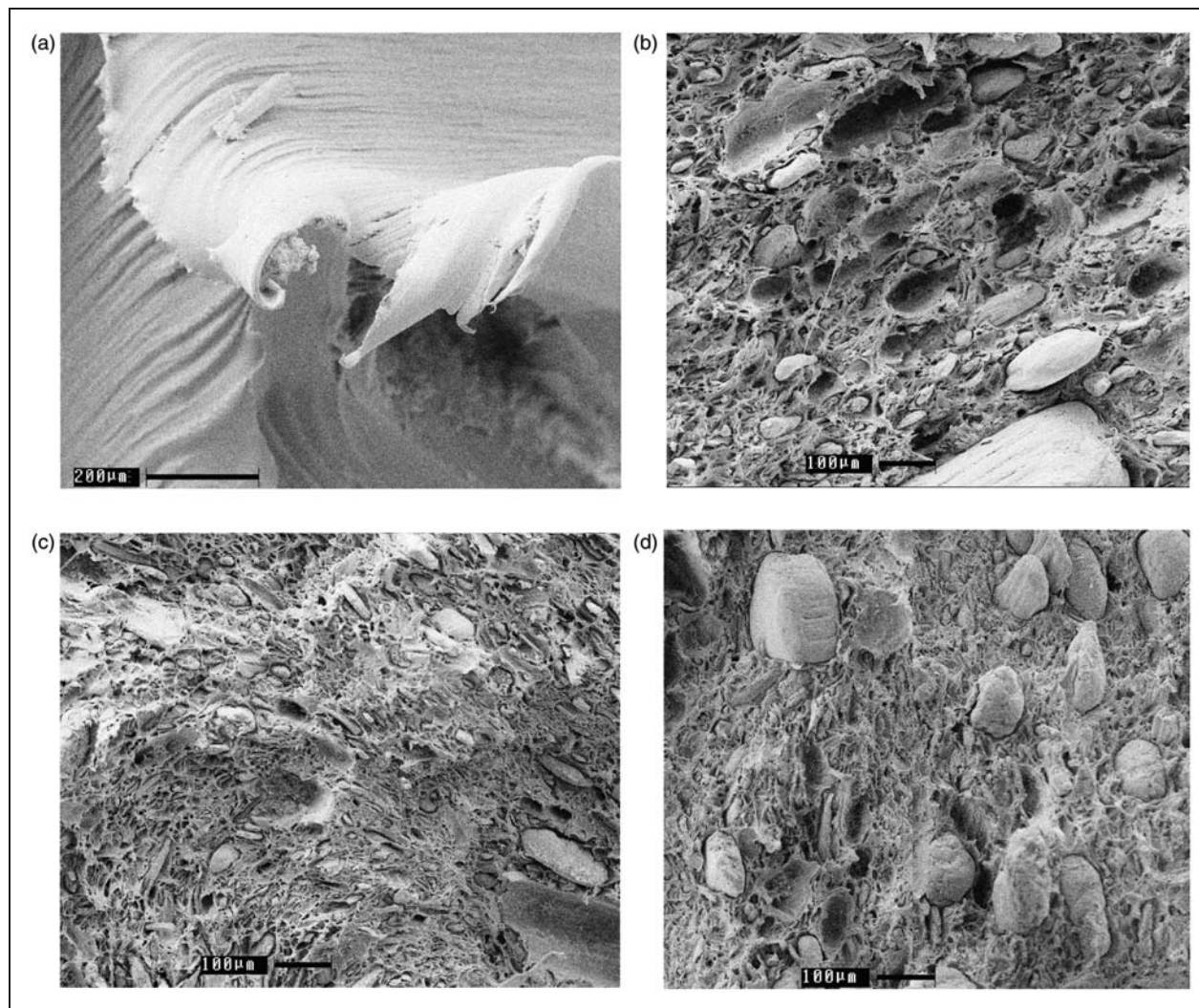


Figure 9. SEM micrograph from fractured surfaces of tensile test specimens of (a) neat LDPE and 37 wt % wood composites from (b) untreated, (c) 180°C and (d) 200°C treated wood at 100 × magnification.

Conclusions

It was observed that incorporating wood into LDPE has no significant effect on the melting and crystallization behavior of the composites containing untreated wood and wood treated at 180°C. However, composites made from wood treated at 200°C exhibits a decreasing trend in X_c as the wood content increases. TGA investigations showed that heat treatment improves the thermal properties of wood because of the degradation of hemicelluloses and modification of the lignin component in wood. Also, thermal stability of the composites was increased relative to the neat matrix, with the heat-treated wood giving higher stability compared to the untreated ones at higher filler loading.

Tensile tests revealed an increase in tensile modulus when heat-treated wood is used in comparison with untreated wood as a result of improved wetting of the wood by the matrix, leading to better interfacial adhesion. Tensile strength decreased with filler loading and treatment temperature due to the embrittlement of the matrix by the filler. However, composites containing wood treated at 180°C shows higher strength values in all the compositions studied. The influence of wood treated at 200°C on the tensile strength was related to its effects on X_c . It was found that 200°C treated wood resulted in lower crystallinity and lowest tensile strength. Improved thermal properties is advantageous in WPC processing.

Flexural modulus and strength increased with filler loading in untreated wood composites relative to composites containing heat-treated wood.

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