Effect of Heat Treatment of Wood Flour on the Water Absorption, Thickness Swelling and Impact Performance of LDPE/Red Balau Composites

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ABSTRACT: Red balau saw dust was heat-treated at 180°C and 200°C for one hour respectively, compounded with low density polyethylene (LDPE) at 20% and 37% by weight and molded in an injection molding machine. Samples were soaked in water for 147 days to test the water absorption and thickness swelling rates. The initial rate and the equilibrium water uptake values decreased with heat treatment. Furthermore, the thickness swelling of the composites were found to increase with time of immersion and wood content. A reduction in thickness swelling up to 18% and 31% was observed in composites made of wood flour heat treated at 180°C and 200°C respectively. Charpy impact tests revealed that composites made from heat treated wood flour have higher peak load (P) values when compared with the untreated samples. Similarly, the critical stress intensity factor or fracture toughness (K_c) of the composites increased in the wet samples relative to the dry-as-molded ones. This increment is proportional to the wood content, with composites loaded with 37 wt% wood flour heat treated at 180°C showing the highest value of 1.78 MPa.m$^{0.5}$. Energy to failure (W) of the moisture saturated composites were generally lower than those of the dry-as-molded ones. Critical strain energy release rate or toughness (G_c) of the composites decreased generally with water absorption, with composites made from wood flour treated at 200°C exhibiting the lowest value. Therefore, utilizing heat treated wood flour in composites production can impart properties that is able to withstand moisture related applications.

KEYWORDS: Low density polyethylene, Heat treatment, Impact properties, Injection moulding, Thickness swelling, Water absorption

1 INTRODUCTION

The hygroscopic nature of wood remains a limitation when wood thermoplastic composites (WTC) are used in outdoor or moisture related applications. In certain usage, the environmental conditions can be so harsh that the performance of these composites is undesirably affected [1]. To alleviate this shortcoming, wood flour is treated before compounding with the polymer matrix. One such treatment is the thermal treatment. The thermal modification of wood is defined as the application of heat to wood in order to bring about a desired improvement in the performance of the material [2]. High temperature treatment of wood results in several changes occurring in the wood chemistry. Hemicelluloses are degraded, producing simple sugars which may undergo reversible reactions to form highly branched polysaccharides. There is also the degradation of part of the cellulose to form furan type compounds. Thermal softening of the cell wall matrix, mainly lignin, also sets-in. This enables cross-linking to occur between carbohydrate polymers and/or between lignin and carbohydrate polymers resulting in an increase in the crystallinity of the amorphous cellulose, with consequent improvement in dimensional stability and decreased hygroscopicity of wood [3, 4, 5]. It has been reported that as wood is progressively heated to higher temperatures, production of condensable fractions occurs, with loss of water and volatile extractives at temperatures below about 140°C. Above this temperature, dehydration reactions also begin to occur, as the so-called ‘water of constitution’ is lost, leading to a decrease in O-H content. These assume greater importance as the temperature is raised further [2].

Heat-treated wood has been applied to WTC in order to improve specific properties. Okamoto *et al.* [6] used steam exploded beech, Japanese cedar and red meranti

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wood flour for the manufacture of WTC using PMMA, PVC and PS as matrices. This report showed an increase in the modulus-of-rupture and elasticity when compared to the untreated wood composites. It was also observed that the water resistance of the resultant composites was improved upon addition of heat treated wood flour, due to the hydrophobicity of the heat treated wood.

Robin and Breton [7] studied the effects of wood fibre heat-treated at 230°C on wood/recycled HDPE composites. Significant improvements in tensile and flexural modulus were observed when heat treated fibre was used, relative to untreated fibres, but no appreciable increase was observed in the elongation-at-break. It was concluded that better adhesion of the fibre to the matrix was responsible for this behaviour. This result was corroborated by Kaboorani *et al.* [8] who studied the reinforcement of HDPE with wood treated at 175°C, 190°C and 205°C and the effects on the mechanical properties of the resultant composites. Similarly, Aydemir *et al* [9] reported an improvement in the tensile and flexural properties of composites made from Nylon 6 filled with wood flour treated at 212°C. From these reports, it was inferred that the use of heat-treated wood in WTC is a good way of building up the performance of the composites. This was attributed to the increased compatibility between the polar wood and the non-polar polymer, which led to improved tensile strength and modulus of elasticity.

In another report, it was observed that beech wood became more water resistant after subjecting it to temperatures between 130°C and 160°C. It was also reported that the heat treatment resulted in the modification of the conformational arrangement of wood biopolymers due to plasticization of lignin [10]. The authors reported reduced water absorption rate of the treated wood after soaking for 24 h than the untreated counterparts. This was ascribed to the degradation of hemicellulose, the most hydrophilic polymer in wood, and the chemical modification on the wood components during heat treatment [11]. Accordingly, compounding heat treated wood with thermoplastic polymer should, technically, decrease the water absorption tendency of the composite.

The study of ways of improving the interfacial adhesion between bio-fillers and polymer matrix is very important for the use of composites in moisture-challenging applications. This is because the wide distribution, renewability and recyclability of lignocellulosics can greatly expand the applications for these low-cost materials in high performance composites. In addition, research on the effect of heat treatment of wood flour on the water absorption and impact properties of WTC is rather rare.

Therefore, this paper is aimed at modifying red balau saw dust with heat treatment, compounding it with LDPE and studying the effects of water absorption on the impact properties of the resultant composites to ascertain its usability in moisture related applications.

2 MATERIALS AND METHODS

2.1 MATERIALS

Red Balau (*Shorea dipterocarpaceae*) saw dust was obtained from a local saw mill in the Klang Valley, Selangor, Malaysia. It was milled to between 40-100 mesh (400-150 µm) sizes using a locally fabricated mill. Commercially available low density polyethylene, LDPE, (Titanlene LDI300YY), with a density of 920 kg/m^3, molecular weight of $3.5–3.8 x 10^5$ g/mol and MFI of 20 g/10 min, supplied by Titan Petchem (M) Sdn Bhd, Malaysia, were used as the matrix.

2.2 METHODS

2.2.1 Compounding

Untreated wood sawdust was dried in an oven at 60°C for 48 hours to a moisture content of less than 2% and stored in sealed plastic bags over dried silica gel in desiccators prior to compounding. Undried wood flour was subjected to 180°C and 200°C respectively in an oven for one hour effective treatment time. LDPE, untreated and the heat treated wood flours were pre-mixed in different compositions in 200 g portions at 20% and 37% weight fractions (equivalent to 40% and 60% volume fractions) as presented in Table 1 and compounded in a twin screw co-rotating extruder (Brabender KETSE 20/40 Lab Compounder, Germany) at a barrel temperature of between $150-155°C$ along the barrel zones from the hopper to the die and screw speed of 250 rpm. The melt pressure varied between $34-39$ bars depending on the wood content, while the die temperature was between $164-178°C$. The samples were extruded out through a circular die of 3 mm in diameter, cooled in a water bath and pelletized. Extruded pellets were oven dried at 80°C for 24 hours and stored in sealed plastic bags over silica gel in desiccators for injection molding.

2.2.2 Injection moulding

The pellets were injection molded into tensile and impact test pieces using the BOY 55M (Germany) injection molding machine at a barrel temperature of between 150°C and 155°C, an injection pressure of $100-120$ bars and mold temperature of 25°C.

2.2.3 Water absorption tests

Water absorption tests were performed on samples cut from the tensile test stripes using ASTM D570 [12]. Samples of 50.0 x 13.0 x 3.3 mm dimensions were dried at 90°C to a constant weight. It was then immersed in distilled water at room temperature for 147 days. At predetermined time intervals, samples were removed, wiped with a blotting paper and immediately weighed to 0.001 g accuracy then returned to the water. The measurement was taken in less than 1 minute of removal from water. Three samples were used for each measurement and the average value was recorded.

***Table 1**. Formulations of the composites*

Sample code	Weight fraction of LDPE (%)	Weight fraction of wood flour (%)	Treatment temperature (°C)
LDPE/W$_{UN/20}$	80	20	-
LDPE/W$_{UN/37}$	53	37	-
LDPE/W$_{180/20}$	80	20	180
LDPE/W$_{180/37}$	53	37	180
LDPE/W$_{200/20}$	80	20	200
LDPE/W$_{200/37}$	53	37	200

2.2.4 Thickness swelling

The thickness swelling of the water soaked specimens was taken by marking three points along the length of the samples. At pre-determined intervals, samples were removed, wiped and the dimensions on the marked spots taken using a Mitutoyo (Japan) digital calliper. Three samples were used and the average result was reported.

2.2.5 Impact testing.

Impact test bars of average dimensions of 6 mm x 12 mm x 80 mm were notched at the centre of one edge in order to produce single edge notch (SEN) impact test specimen. The notch angle was set at 45°. Each batch was notched with four different notch-to-depth ratios (a/D) of 0.1, 0.2, 0.3 and 0.4 using a *Ray-Ran* notch cutting machine. The support span-to-depth ratio (S/D) was maintained at 4 throughout the experiment. The impact test was carried out in the Charpy mode using an Instron (Dynatup 9210, USA) falling weight impact tester with a V-shaped impactor tup. The test was run at ambient temperature, with a fixed impactor load weight (m) of 6.448 kg. The impactor height was adjusted to provide an impactor velocity (v) of 2.9238 ms^{-1} and impact energy of 13.9512 J. The impactor tup struck the specimen midway between the supports. For each batch, a minimum of 10 specimens were tested and the results presented were taken from the average of at least 8 reproducible data. ASTM standard E23 [13] was used as a standard in calculating the impact properties.

2.2.6 Scanning electron microscopy (SEM)

The fractured surface of the tensile and impact test specimens was observed using the Leica S440 scanning electron microscope at an accelerating voltage of 10 kV after gold sputtering to a thickness of 0.014 µm to avoid unnecessary charges.

3 RESULTS AND DISCUSSION

3.1 WATER ABSORPTION

Water uptake, W_t, was calculated using the formula,

$$W_t = \frac{W_2 - W_1}{W_1} \times 100\ (\%) \qquad (1)$$

where W_1 and W_2 are the dry weight and weight after time t of immersion in water respectively.

A plot of moisture content against time for the different treatment temperatures and wood flour loadings is presented in Figure 1. The neat LDPE showed no water absorption as no weight increase was observed over the period of study. This is because LDPE is hydrophobic and has no tendency of absorbing water. However, the untreated and heat treated wood composites absorbed water to various extents. As the neat LDPE did not absorb water, it can be assumed that the wood flour is responsible for all the moisture absorbed by the composites. This is expected due to the hydrophilic nature of wood flour. It is sometimes believed that moisture is not an issue with WTC because wood is totally encapsulated by the matrix. If that was the case, the matrix would shield the wood from moisture because of the hydrophobic nature. Nevertheless, the composites still exhibit a degree of moisture absorption tendency [14]. This seems to indicate that wood must have been exposed on the surface of the samples which may be responsible for the various degree of water absorption exhibited by the composites. Also, it can be seen that water absorption increased with time of immersion, reaching a certain value (the equilibrium point), when the water content of the composite did not show any appreciable increase. Composites containing 20 wt% and 37 wt% untreated wood flour showed a saturation point of 3.48% and 6.02% respectively on the 119th day of immersion.

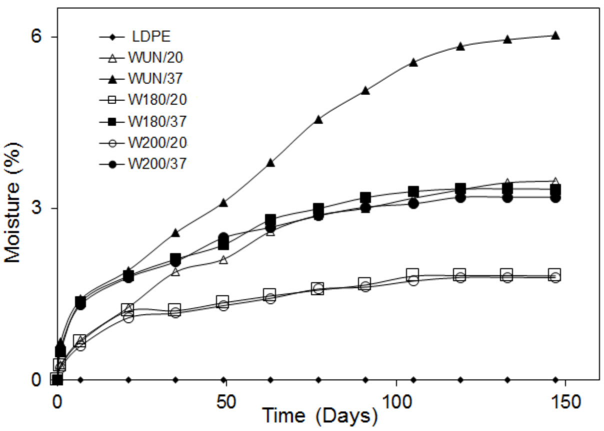

***Figure 1:** Moisture absorption of composites as a function of wood content and treatment temperature*

This is likely because of the higher number of free O-H groups on the surfaces of untreated wood composites which interact with water via hydrogen bonding, leading to more ability to absorb water [15]. The higher the wood content, the higher was the water absorption tendency (Figure 1). It is also possible that at 37 wt%, the wood flour was not continuously distributed and most of the wood particles probably contacted one another directly, in which case, complete encapsulation was not achieved as seen from the SEM micrograph in Figure 2. This could result in the higher water absorption values recorded for composites at 37 wt% wood content.

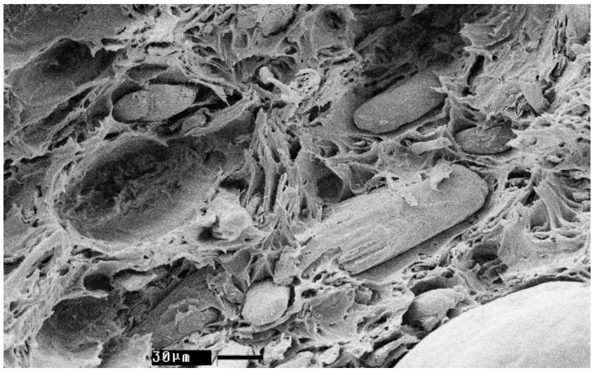

Figure 2: *SEM micrograph of tensile fractured surface of composites containing 37 wt% untreated wood*

Water absorption in WTC has been shown to not only depend on the filler particles and matrix structure, but also on the filler-matrix interface [16]. Hydrophilic fillers lead to lower filler-matrix interaction resulting in poor interface which facilitates the absorption process (Figure 2). It has also been reported that moisture can be absorbed into the composite by the dissolution of water into the polymer network, moisture sorption into the free volume, if present, in the glassy structure and by hydrogen bonding between water and the hydrophilic components of the composite. Micro-cracks can also allow moisture transportation involving flow and storage of water within the cracks [16].

Also shown in Figure 1 is the effect of heat treatment of wood flour on the water absorption of the composites. The initial rate and the equilibrium water uptake values decreased with heat treatment. Composites containing 20 wt% wood flour treated at 180°C and 200°C reached equilibrium (1.82% and 1.74% respectively) in 91 days while that containing 37 wt% attained equilibrium point (3.34% and 3.20% respectively) on the 105th day of immersion. Untreated wood composites generally show higher water absorption than the heat-treated counterparts. Composites loaded with 20 wt% untreated wood flour absorbed 92% more water when compared to that made from 180°C treated wood flour and 94% relative to those made from wood flour treated at 200°C, while composites made from 37 wt% untreated wood flour absorbed about 80% water higher than that made from wood flour treated at 180°C and 89% water more than that containing 200°C treated wood flour. This reduction in water absorption could be attributed to the removal of the water-sensitive components, hemicellulose, in wood flour by heat treatment, thereby rendering the composites more water resistant. Furthermore, free hydroxyl groups of the cellulose were reduced with heat treatment which could lead to a decrease in the water absorption of the wood flour and consequently, the composites [2]. Follrich *et al.* [17], reported that at 200°C treatment, the hydrophilicity of spruce wood decreased appreciably. This was observed as an important result for WTC compounding and suggested that it is an alternative way of increasing the adhesion between wood and hydrophobic thermoplastics without using compatibiliser. Composites compounded with 20 wt% wood flour absorbed water and reached saturation faster than that made from 37 wt% wood flour, which attained saturation more gradually. The fact that higher wood flour content means higher ability to absorb water may have resulted in delayed equilibrium moisture content attainment for these categories of composites. Increase in water absorption of untreated wood composites relative to the heat treated counterpart may be due to the hydrophilicity of wood, imparted by the presence of free O-H groups in the wood structure which has a high tendency for hydrogen bonding with water. It has also been reported that the untreated wood has higher hemicellulose content [18] which is responsible for moisture absorption. Previous reports [19] revealed that the absorption of water by non-polar polymers containing fillers depends mainly on the nature and the amount of the fillers. Furthermore, the amount of accessible O-H groups that can form hydrogen bonds with water molecules is another factor responsible for the degree of water absorption by composites. The higher the amount of the O-H groups, the higher is the initial rate as well as the level of water uptake, explaining the trend observed.

Table 2: Equilibrium moisture content of composites

Sample	Equilibrium moisture (%)
LDPE/$W_{UN/20}$	3.48
LDPE/$W_{UN/37}$	6.02
LDPE/$W_{180/20}$	1.82
LDPE/$W_{180/37}$	3.34
LDPE/$W_{200/20}$	1.79
LDPE/$W_{200/37}$	3.20

3.2 THICKNESS SWELLING BEHAVIOUR

The thickness swelling of the composites is evaluated using the following equation:

$$T_t = \frac{T_2 - T_1}{T_1} \times 100\ (\%) \tag{2}$$

where T_1 and T_2 are the thickness of the dry samples and the thickness after time t of immersion in water respectively. A plot of percentage increase in thickness against time for the samples are presented in Figure 3. The thickness swelling of the composites increased with time of immersion until a point of equilibrium is reached when the thickness remains relatively constant. From Figure 3, it can be seen that composites with higher wood content exhibited higher thickness swelling values. Wood flour is hygroscopic in nature and swells when immersed in water. The swelling of individual particle contributes to the overall swelling of the composites. Thus, it is expected that the higher the wood content, the higher is the thickness swelling. In addition, thickness swelling depends on water absorption, with the composites having the highest moisture content swelling the most.

Heat treatment temperatures also have a significant effect on the thickness swelling of the composites (Figure 3). A reduction in thickness swelling up to 18% and 31% are observed with composites made of 37 wt% wood flour heat treated at 180°C and 200°C respectively, relative to the untreated WTC. Decrease in the hygroscopic character of the wood flour and the resultant improvement in interfacial adhesion [8] could be responsible for this trend.

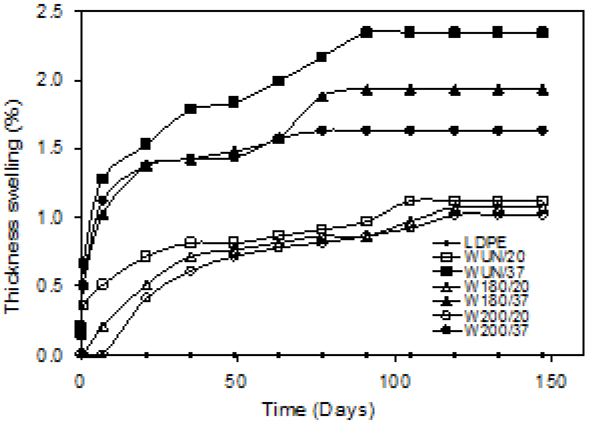

Figure 3: *Thickness swelling of composites as a function of wood content and treatment temperature*

3.3 IMPACT PROPERTIES

Impact may be defined as the sudden application of an impulsive force to a limited volume of material or part of a structure [20]. During the service life, plastic products are exposed to many impact encounters that could lead to failure. It is therefore important to know, among other things, what maximum loads can be sustained without failing. Brittle failure in materials is caused by the presence of very small, microscopic flaws or cracks that exists under normal conditions at the surface and within the body of the material. These are referred to as stress concentrators because of their ability to amplify the applied stress. Any brittle fracture process involves two steps; crack initiation and propagation in response to an applied stress. Using the principles of fracture mechanics, an expression has been developed that relates the critical stress for crack propagation, σ_c and the notch or crack length, a, as follows:

$$K_c = Y\sigma_c\sqrt{a} \tag{3}$$

where K_c is the fracture toughness or stress intensity factor, a property that is a measure of the material's resistance to brittle fracture when a crack is present. It is an indication of the energy per unit area needed to give a fresh crack surface and it characterises the severity of a crack situation as affected by crack size, stress and geometry. Y is a constant that depends on the crack length, a and the specimen sizes and geometries as well as the manner of load application [21]. In a three-point bend test, σ is given by simple bending theory as:

$$\sigma = \frac{6PS}{4BD^2} \tag{4}$$

For the three-point test specimen, where S/D is equal to 4, Y is given by:

$$Y = 1.93 - 3.07\left(\frac{a}{D}\right) + 14.53\left(\frac{a}{D}\right)^2 - 25.11\left(\frac{a}{D}\right)^3 + 25.80\left(\frac{a}{D}\right)^4 \tag{5}$$

A plot of σY against $a^{0.5}$ gives a straight line, where the slope equals the K_c of the materials.

It is assumed that composite materials fail in a linear-elastic manner. Thus, linear elastic fracture mechanics (LEFM) theory is applied. The compliance of the specimen, C, is the displacement per unit load (a reciprocal of stiffness). Therefore, when the crack has a length a, the specimen is less compliant. Generally, the concept of compliance as a ratio of deformation to applied load is given by the equation:

$$C = \frac{\delta}{P} \tag{6}$$

Since energy is stored as strain energy when the material is under stress and the material's response is considered to be entirely elastic, the energy absorbed, W is given by:

$$W = \frac{P\delta}{2} = \frac{P^2C}{2} \tag{7}$$

The total strain energy, U, for a specimen with uniform thickness, B, can be written in terms of this compliance as:

$$U = \frac{W}{B} = \frac{CP^2}{2B} \tag{8}$$

When a crack of length, a, grows into the specimen by a small amount, ∂a, the material becomes more compliant. Thus, it stores less energy at a fixed displacement, but elastic energy is stored more at a fixed load. The rate of change of elastic energy with increase in crack area is defined as the strain energy release rate, *G* [21]:

$$G = \frac{\partial U}{\partial a} = \frac{P^2}{B}\frac{\partial C}{\partial a} \tag{9}$$

The energy release rate is related to the stress intensity factor as follows:

$$G = \frac{K^2}{E} \tag{10}$$

where *E* is the Young's modulus and *K* is given by Equation 3. Substituting Equation 3 for K^2 in Equation 10, *G* becomes:

$$G = \frac{Y^2 \sigma^2 a}{E} = \frac{P^2}{2B}\frac{\partial C}{2B\partial a} \tag{11}$$

Therefore,

$$\frac{\partial C}{\partial a} = \frac{2BY^2\sigma^2 a}{P^2 E} \tag{12}$$

From Equation 4,

$$P = \frac{4BD^2\sigma}{6S} \tag{13}$$

Substituting for *P* in Equation 12 gives:

$$\frac{\partial C}{\partial a} = \frac{9aY^2S^2}{2EBD^4} \tag{14}$$

Thus, the compliance, *C*, can be written as:

$$C = \frac{9S^2}{2EBD^4}\int Y^2 a.\partial a + C_\circ \tag{15}$$

Substituting Equations 13 and 15 for *P* and *C* respectively in Equations 7 gives;

$$W = \frac{B\sigma^2}{E}\left[\int Y^2 a.\partial a + \left(\frac{SD}{18}\right)\right] \tag{16}$$

The term σ^2 in Equation 16 can be eliminated by using Equations 3 and 10 thus;

$$W = \frac{GB}{Y^2 a}\left[\int Y^2 a.\partial a + \left(\frac{SD}{18}\right)\right] \tag{17}$$

$$W = GBD\phi \tag{18}$$

where *B* and *D* refer to the width and depth of the specimen respectively.

The parameter ϕ is a geometrical correction factor determined as a function of a/D given by:

$$\phi = \frac{1}{2}\left(\frac{a}{D}\right) + \frac{1}{18\pi}\left(\frac{S}{D}\right)\frac{1}{\left(\frac{a}{D}\right)} \tag{19}$$

where *a* and *S* are the notch depth (crack length) and the specimen support span respectively. At fracture, the energy release rate, *G* assumes a critical value, G_c. Equation 18 then becomes:

$$W = G_c BD\phi \tag{20}$$

A plot of *W* against $BD\phi$ gives a straight line with the slope as the G_c of the material [22].

3.3.1 Peak load

The peak load is the maximum force needed to cause the fracture of the sample and is a function of the resistance to damage of the material.

The peak load increases with water absorption (Figure 4). The peak load of the neat LDPE remains relatively the same because it did not absorb water. Values of 273.3 N, 235.9 N, 188.9 N and 139.1 N are obtained in the wet LDPE for a/D ratios 0.1, 0.2, 0.3 and 0.4 respectively. While 266.9 N, 225.3 N, 184.5 N and 137.4 N are recorded in the dry as moulded LDPE for a/D ratios 0.1, 0.2, 0.3 and 0.4 respectively. However, the composites display an increase in *P* values with water absorption, with composites containing 37 wt% wood flour showing the highest increment above the values of the dry as moulded and wet neat LDPE. It is possible that the plasticisation of the composite system by water absorption may have rendered the composites more flexible than the dry as moulded counterparts, thereby increasing the maximum load required to break the material. Heat treated and untreated composites loaded with 37 wt% wood flour exhibit the highest values of *P*. Since this category of composites show the highest water absorption rate in their different categories, it is pertinent to think that they should also be plasticised the most. However, composites made from heat treated wood flour have lower *P* values when compared with the untreated counterparts (Figure 4). Again, reduced attraction to water by heat treatment could be responsible for this behaviour. Increase in *P* with moisture absorption agrees with earlier findings [23].

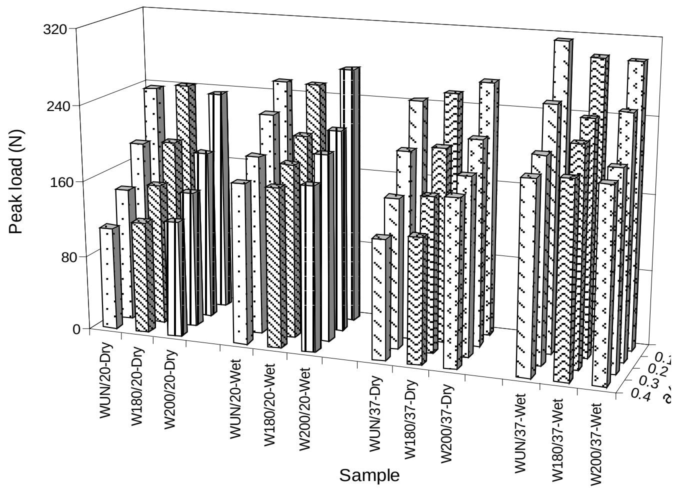

***Figure 4:** Peak load of dry as moulded and wet samples as a function of wood content and heat treatment*

3.3.2 Critical stress intensity factor (K_c)

Figure 5 presents the K_c of the dry as moulded and moisture saturated composites as a function of wood content and heat treatment. The K_c of the dry as moulded and wet LDPE is 1.70 MPa.m$^{0.5}$ and 1.63 MPa.m$^{0.5}$ respectively. These values show no appreciable change in the K_c of the neat LDPE upon moisture absorption. The hydrophobic character of LPDE could be accountable for this. As observed in the peak load, the K_c values of the composites increased in the wet samples relative to the dry as moulded ones. This increment is proportional to the wood content, with composites loaded with 37 wt% wood flour showing the highest values of 1.78 MPa.m$^{0.5}$, 1.82 MPa.m$^{0.5}$ and 1.77 MPa.m$^{0.5}$ for moisture saturated untreated, 180°C and 200°C treated WTC respectively. The dry as moulded samples exhibits values of 1.40 MPa.m$^{0.5}$, 1.41 MPa.m$^{0.5}$ and 1.60 MPa.m$^{0.5}$ for untreated, 180°C and 200°C treated WTC in that order. This is rather strange as the plasticisation effect of water should lower the stiffness of the material. On the other hand, what appears to be in display here is the fact that the material becomes pliable with water absorption, thereby increasing its resistance to brittle failure and raising its K_c. Composites loaded with 37 wt% wood treated at 180°C display a slightly higher value of K_c than the other samples. Furthermore, it has been reported that the impact behaviour of WTC is a complex phenomenon involving the nature of the fillers and matrix, the filler-matrix bond, filler distribution and orientation. In addition, possible damage modes which is also an important factor to consider have been identified as matrix cracking, interfacial bond failure, filler breakage, void growth and delamination [24]. Besides, the fact that one or more of these factors act in synergy to cause the failure of WTC could further complicate the understanding of the fracture behaviour of WTC.

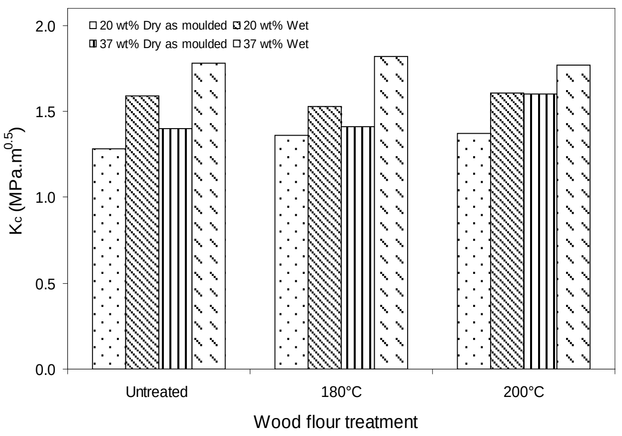

Figure 5: K_c of dry as moulded and wet samples as a function of wood content and heat treatment

3.3.3 Energy to failure (W)

The energy to failure represents the total energy the material will absorb until full penetration of the impactor tup.

The value of W for the dry as moulded and wet LDPE is relatively the same. Values of 4.62 J, 3.48 J, 3.17 J, and 2.12 J are recorded for a/D ratios 0.1, 0.2, 0.3, 0.4 respectively in the dry as moulded samples while 4.59 J, 3.51 J, 3.12 J and 2.11 J are obtained for a/D ratios of 0.1, 0.2, 0.3 and 0.4 correspondingly. This is because LDPE did not absorb water and as such, the energy required to fracture both the dry and wet specimens remain almost the same. Figure 6 presents the W values of the dry as moulded and wet composites. W of the moisture saturated composites are generally lower than those of the dry as moulded ones. This decrement is higher in composites with high wood content, with the untreated WTC showing slightly higher values than the heat treated counterparts. Again, the possibility of interfacial degradation by water might have lowered the energy to break the moisture saturated specimens relative to the dry as moulded ones, resulting in the observed trend.

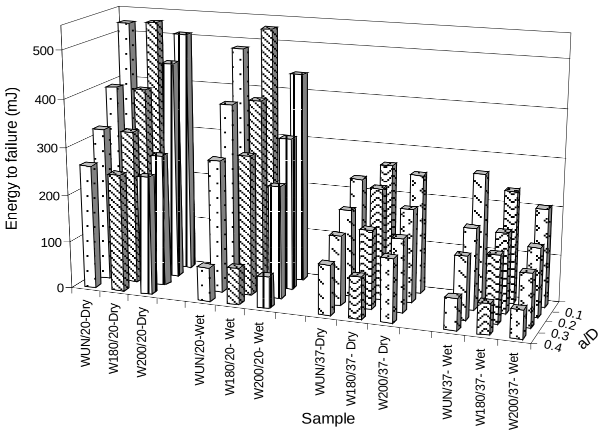

Figure 6: Energy to failure of dry as moulded and wet samples as a function of wood content and heat treatment

3.3.4 Critical strain energy release rate (G_c)

G_c, is a material property referred to as the toughness, critical strain energy release rate or crack extension force. It is the energy required to increase the crack by unit length in a piece of material of unit width [25].

The G_c values of the dry as moulded and wet LDPE are very much alike (96.6 kJ.m^{-2} and 97.2 kJ.m^{-2} respectively), an indication that moisture has no effect on the impact strength of LDPE. However, the G_c values of the composites shown in Figure 7, decrease generally with water absorption. G_c, values of 4.85 kJ.m^{-2}, 5.47 kJ.m^{-2}, 5.08 kJ.m^{-2} are obtained for dry as moulded samples loaded with 37 wt% untreated, 180°C and 200°C treated wood flour respectively while values of 4.44 kJ.m^{-2}, 4.45 kJ.m^{-2}, 3.71 kJ.m^{-2} are recorded in moisture saturated composites containing 37 wt% untreated, 180°C and 200°C treated wood flour accordingly.

The difference in G_c values between the dry as moulded and wet samples is higher in composites made from wood flour treated at 200°C. It could be that since the composite exhibit a better interfacial bonding in the dry as moulded sample (Figure 8), upon moisture absorption and the consequent degradation of the interface, crack propagation along the interfacial area is enhanced, thereby preventing dissipation of energy thus, reducing G_c.

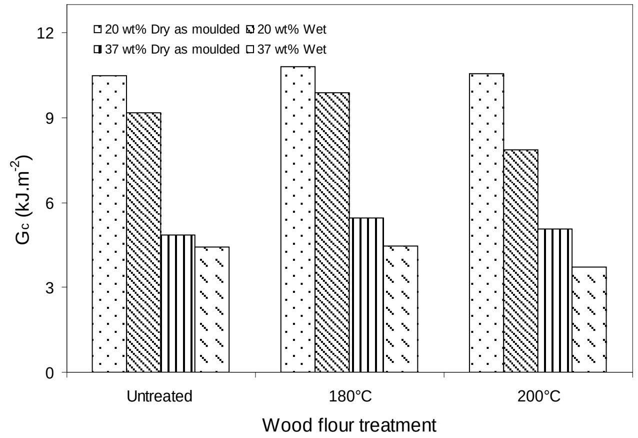

Figure 7: G_c of dry as moulded and wet samples as a function of wood content and heat treatment

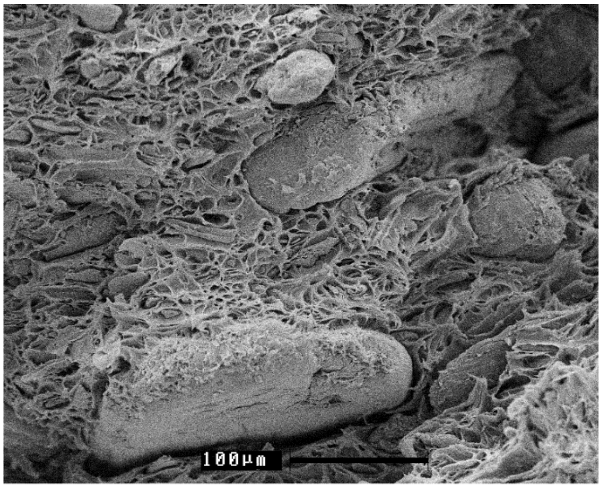

Figure 8: SEM Micrograph of Impact fractured surface of composite containing 37 wt% wood flour treated at 200°C

In the wet composites containing untreated and 180°C treated wood flour, poor interfacial adhesion already existing in the dry as moulded specimen (Figure 9) could mean further degradation of its interface with moisture absorption, leading to fibre pull-out (Figure 10), thereby showing higher energy dissipation and higher G_c.

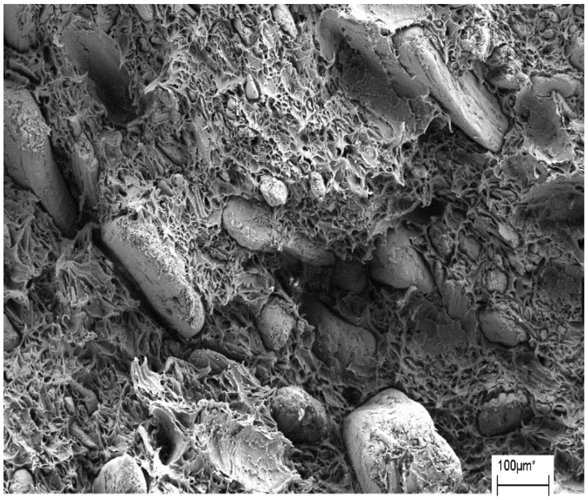

Figure 9: SEM micrograph of the impact fractured surface of composite loaded with 37 wt% wood flour treated at 180°C

Figure 10: Impact fractured surface of 37 wt% untreated wood flour

4 CONCLUSION

All the composites were found to absorb moisture to various extents due to the hydrophilic nature of wood. However, composites made from heat-treated wood flour showed a reduction in water absorption up to 90% because of the reduced hydrophilicity imparted to the wood flour by heat treatment. Accordingly, the decrease in the water absorption capacity of wood used in this study has beneficial effect on the lifetime of the product and its applications.

The impact properties deteriorated generally with water absorption in all composites studied but the extent of decline was more pronounced in the untreated wood composites when compared to the heat-treated composites. Therefore, heat treatment can reduce the moisture absorbing tendency in wood fillers leading to better water resistant composites with the ability to retain its mechanical properties after long time exposure to moisture. It is, therefore, a good method of improving wood thermoplastic composites properties in applications that pose moisture-related challenges.

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