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Effects of wood flour content and heat treatment on the dynamic mechanical and impact properties of LDPE/red balau (*Shorea Dipterocarpaceae*) composites

Ruth A. Lafia-Araga[1] · Aziz Hassan[2] · Rosiyah Yahya[2] · Normasmira Abd Rahman[2] · Fauzanie Md Salleh[3] · Ganiyat Olusola Adebayo[4]

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Abstract

Red Balau saw dust was heat-treated at 180 °C and 200 °C for one hour and compounded with low-density polyethylene, LDPE, at 9, 20 and 37 wt%. The compounded materials were injection moulded into test specimens. Charpy impact assessment of the notched samples of the composites revealed that the peak load, P, and the critical stress intensity factor, K_c, increased with wood content and treatment temperature. While the energy to failure, W , and the critical strain energy release rate, G_c, decreased with wood content, the values were highest in composites made from wood flour treated at 180 °C and reduced in 200 °C treated wood composites. This behaviour revealed that heat treatment of wood flour at the 200 °C resulted in poorer impact properties of the composites. Dynamic mechanical analysis showed an increase in storage and loss modulus of composites made from untreated wood flour relative to the heat-treated ones. Tan delta values were also found to reduce in the heat-treated wood composites as a result of decreased damping. Heat treatment of wood flour at appropriate temperature enhanced both the mechanical and dynamic mechanical properties of the composites.

Keyword Heat treatment · Wood fibre modification · Impact properties · Dynamic mechanical analysis · Injection moulding

✉ Ruth A. Lafia-Araga
ruth.araga@futminna.edu.ng

[1] Department of Chemistry, School of Physical Sciences, Federal University of Technology, Minna 920006, Niger State, Nigeria

[2] Polymer and Composite Materials Research Laboratory, Department of Chemistry, Faculty of Science, University of Malaya, 50603 Kuala Lumpur, Malaysia

[3] Chemisty Division, Centre for Foundation Studies in Science, University of Malaya, 50603 Kuala Lumpur, Malaysia

[4] Standards Organisation of Nigeria, Operational Headquarters, Lekki Peninsula Scheme 1, Lekki 101233, Lagos, Nigeria

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List of symbols

E'	Storage modulus
$E'_{25\ °C}$	Storage modulus at 25 °C
$E'_{-\ 100\ °C}$	Storage modulus at −100 °C
E''	Loss modulus
E''_{max}	Peak maximum of the loss modulus
$E''_{25\ °C}$	Loss modulus at 25 °C
$T_{\beta}^{E''}$	Temperature at maximum value of E'''
δ	Phase angle
Tanδ	Loss factor
Tan$\delta_{25\ °C}$	Loss factor at 25 °C
Tanδ_{max}	Maximum value of Tan δ peak
σ	Dynamic stress
ε	Dynamic strain
SEN	Single edge notch
a	Notch or crack length
a/D	Notch-to-depth ratio
P	Peak load
K_c	Critical stress intensity factor
Y	Geometry factor
σ_c	Critical stress for crack propagation
S	Support span
S/D	Span-to-depth ratio
W	Fracture energy
G_c	Critical stress release rate
ϕ	Geometrical correction factor

Introduction

There has been an increasing use of wood as reinforcement in wood thermoplastic composites (WTC) production. This is because wood is cheap, available, renewable, flexible, recyclable and biodegradable. It is light, with a good strength to weight ratio, good tribological and high specific properties. Wood fillers reinforcement in thermoplastic composites has been proved to result in enhanced mechanical properties [1, 2]. However, wood, being polar, will not bond easily with a non-polar matrix. Therefore, the need to modify it becomes imperative. The essence of wood fibre modifications in WTC is to enhance the compatibility between the non-polar matrix and the polar wood, which ultimately lead to improved composite properties [3].

In wood modification by heat treatment, wood is subjected to higher temperatures than drying (160–250 °C). This results in the degradation of hemicellulose which has the lowest molecular weight among the wood constituents. Hemicellulose degradation leads to reduction of the OH groups and the formation of O-acetyl groups [4]. 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 [5, 6].

It is an established fact that heat treatment could result in the loss of mechanical strength of wood [6, 7]. Consequently, using heat-treated wood as filler in WTC should, technically, worsen the properties of WTC. On the other hand, the changes imparted 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 WTC properties more than the untreated counterparts [8]. Furthermore, 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 [9].

The temperature-dependent dynamic parameters such as storage modulus, $E^{'}$, loss modulus, $E^{''}$and mechanical damping, tanδ, offer an insight into the level of interactions between the polymer matrix and fibre reinforcement [10]. Pothan et al. [11] investigated the role of fibre/matrix interactions in chemically modified banana fibre composites. The authors reported an increase in dynamic modulus and reduced damping. This behaviour was attributed to the improved interaction between the fibre and the matrix. Hashmi et al. [12] studied the effect of fibre content on the relaxation processes in LLDPE reinforced glass/Kevlar hybrid composites and found that the α-relaxation shifted to higher temperature with the addition of fibres. Mohanty et al. [13] also examined the dynamic mechanical properties of MAPE treated jute/HDPE composites and reported an increase in the storage modulus of the treated composites. Fibre content and coupling agent were also found to present a strong influence on the tan δ, α- and γ-relaxation process of HDPE.

Many applications of polyethylene (PE) exploit the excellent toughness of the many grades available. This toughness may be exhibited in a number of beneficial ways such as puncture resistance of films, drop strength of blown bottles and impact resistance of moulded items [14]. In addition, the ability of composites made from PE to withstand sudden impact is obviously of great importance for any practical application of the material. It is therefore necessary that the toughness of specifically prepared composites be tested to access the ability of fabricated items to withstand specific hazards.

Notched impact energy can be said to be a measure of crack propagation. Crack propagation occurs along the path of least resistance, requiring as little energy as possible. In composites, the crack may propagate at the matrix–fibre interface, accentuating its dependence on the nature of the interface. Heat treatment of wood flour has been reported to enhance better retention of the impact properties of water saturated LDPE/Red balau composites [15]. In a related study, Adebayo et al. [16] investigated the influence of heat treatment of mangrove fibres on the impact and thermal properties of HDPE/mangrove composites. The authors reported that heat treatment of mangrove fibres had a positive influence on the impact properties of the composites. Furthermore, it has been observed 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 [17]. 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.

Although, few studies have investigated the effect of thermally modified wood flour on the impact properties of WTC [15, 16, 18]; to the authors' knowledge, study on the effect of heat treatment of wood flour on the dynamic mechanical properties of WTC is rather rare. This report is therefore aimed at assessing the effect of wood flour content and heat treatment on the impact and dynamic mechanical properties of red balau/LDPE composites. Linear elastic fracture mechanics was applied to evaluate the effect of the content and heat treatment of wood flour on the impact properties of LDPE/red balau composites.

Materials and methods

Materials

Red balau (*Shorea Dipterocarpaceae*) sawdust, a heavy hard tropical wood, was obtained from a local saw mill in the Klang Valley, Selangor, Malaysia. It was milled to between 40 and 100 mesh (450–150 μm) sizes using a locally fabricated mill. Commercially available LDPE (Titanlene LDI300YY), with a density of 920 kg m^{-3}, molecular weight of $3.5–3.8 \times 10^5$ g mol^{-1} and MFI of 20 g/10 min with a load of 2.16 kg at 190 °C, was supplied by Titan Petchem (M) Sdn. Bhd., Malaysia, which was used as the matrix.

Processing

Wood pre-treatment

Untreated wood saw dust was dried in an oven at 60 °C for 48 h to a moisture content of less than 2% and stored in sealed plastic bags over silica gel in desiccators for not more than 24 h prior to compounding. Undried red balau wood flour was placed on a stainless-steel tray to a depth of about 5 mm and subjected to 160 °C, 180 °C and 200 °C in a vacuum oven for an effective treatment time of 1 h.

Thermal characterization of wood flour

Thermogravimetric analysis (TGA) measurements were taken using a PerkinElmer TGA 6 (USA) on 8–10 mg wood flour samples 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.

Compounding

LDPE and wood flour were pre-mixed at different compositions in 200 g portions and compounded in a twin screw co-rotating extruder (Brabender KETSE 20/40 Lab Compounder, Germany), with screw diameter and aspect ratio (*L*/*D*) of 20 mm and 40 mm, respectively. The temperatures along the barrel zones were set between 150 °C and 155 °C from the hopper to the die, and screw speed was 250 rpm. The melt pressure varied between 34 and 39 bars depending on the wood content, while the die temperature was between 164 °C and 178 °C. Vacuum venting was used to remove the volatile compounds. The samples were extruded through a circular die of 3 mm in diameter. The extruded strand was cooled in a water bath and pelletized to a length of about 3 mm for injection moulding. Extruded pellets were oven dried at 80 °C for 24 h and stored in sealed plastic bags over dried silica gel in desiccators for about 24 h before injection moulding. Composites were prepared at three different wood flour loadings of 9%, 20% and 37% by weight as shown in Table 1.

Injection moulding

Pellets were injection moulded into tensile test pieces using the BOY® 55 M (Germany), a 55 tonne clamping force injection moulding machine at a barrel temperature of between 150 °C and 155 °C, an injection pressure of between 100 and 120 bars, an injection time of 20 s and a mould temperature of 25 °C. Single gated 4 and 8 cavity tensile and impact standard bar moulds, respectively, were used in the moulding.

Dynamic mechanical analysis (DMA)

DMA tests were carried out on rectangular test strips cut from the middle section of the tensile test specimen with 60.0 mm × 13.0 mm × 3.3 mm average dimensions in the three point bending mode with a support span of 50 mm, using a TA Q-800 (Thermal Analysis Instrument, USA) dynamic mechanical analyzer. Each specimen

Table 1 Formulations of the composites

Sample code	Treatment temperature (°C)	Weight fraction of wood flour (%)	Weight fraction of LDPE (%)
$W_{UN/9}$	Untreated	9	91
$W_{180/9}$	180	9	91
$W_{200/9}$	200	9	91
$W_{UN/20}$	Untreated	20	80
$W_{180/20}$	180	20	80
$W_{200/20}$	200	20	80
$W_{UN/37}$	Untreated	37	63
$W_{180/37}$	180	37	63
$W_{200/37}$	200	37	63

was equilibrated at −100 °C for 5 min and ramped to 100 °C at a scan rate of 2 °C min^{-1} under nitrogen at a fixed frequency of 1 Hz and an amplitude of 15 µm. The storage modulus, loss modulus and tan delta peaks were obtained using the TA universal analysis software.

Impact testing

Impact test bars of average dimensions 6 mm × 12 mm × 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 (*a*/*D*) ratios 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 mid-way between the supports. For each batch, a minimum of eight specimens were tested and the results presented were taken from the average of at least six reproducible data. ASTM standard E-23 [19] was adopted as a standard in preparing the test specimens with some modifications.

Scanning electron microscopy

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

Results and discussion

Wood pretreatment

Figure 1 presents the TGA/DTG thermograms of untreated and treated wood flour at 160 °C, 180 °C and 200 °C. The TGA thermograms of untreated wood flour and that of the material treated at 160 °C overlapped, indicating that no appreciable changes were imparted to the wood flour treated at 160 °C. Treatment temperature higher than 200 °C was not achievable due to the limitations of the oven. Also, lower treatment temperature was not considered as wood flour treated at 160 °C did not impart any thermal improvement to the wood flour, relative to the untreated material (Fig. 1). Therefore, 180 °C and 200 °C were the treatment temperatures reported in this study. Previous researchers [8] had treated wood flour as fillers in WTC at 175 °C, 190 °C and 205 °C for 45 min. In addition, higher treatment time was not considered as that may mean more energy consumption resulting in higher cost. Detailed report of the thermogravimetric analysis and Fourier transform infrared spectroscopy of the wood flour was reported in our previous publications [20, 21].

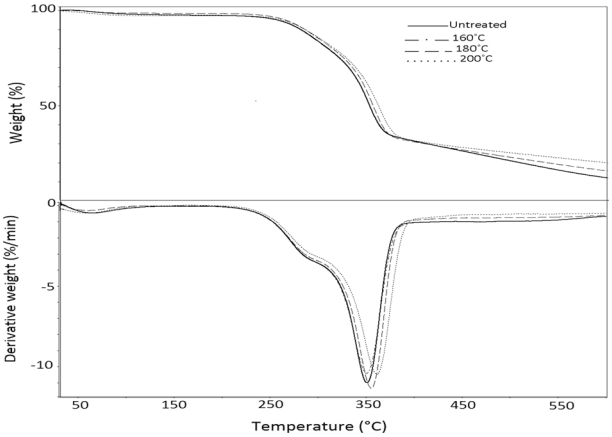

Fig. 1 TGA/DTG thermograms of untreated and heat-treated wood flour

Dynamic mechanical analysis

Storage modulus, E′

The storage modulus, E', is closely related to the load bearing capacity of a material and is linked to the flexural modulus (E). It is a measure of the energy stored in the material during a cycle and describes the elastic character or the solid-like nature of the material. In order to obtain the material's stiffness as a function of temperature, DMA experiment was performed on the neat LDPE and composites from the untreated and heat-treated wood flour.

Figure 2 presents the variation of E' with temperature for composites containing different filler loadings of untreated wood flour. The principal events in these

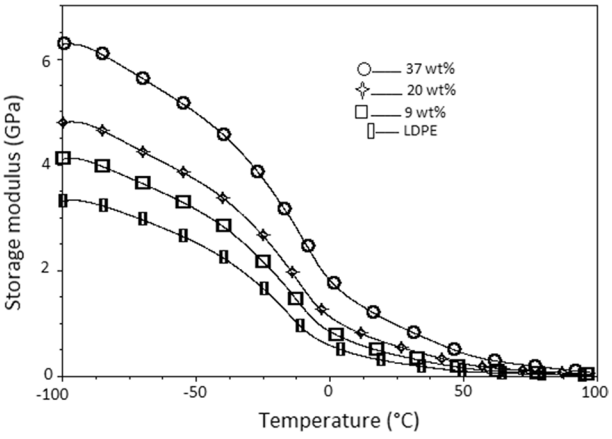

Fig. 2 Storage modulus of LDPE and composites as a function of wood content

curves are summarized in Table 2. E' values of LDPE and composites decreased steadily with increase in temperature within the temperature range employed in this experiment. This is as a result of the matrix softening due to increased segmental mobility [22]. However, E' of the composites are higher than that of the neat LDPE. This disagrees with the findings of Yang et al. [23], who observed that at lower temperatures, E' of the composites is very close to that of the matrix because the filler does not contribute meaningfully to the stiffness of the matrix. On the other hand, their finding is in agreement with this study as the E′ curves tend to converge at higher temperature. A sudden decrease is noticed between −15 °C and 10 °C which is believed to correspond to the glass transition region of the matrix. Thereafter, an appreciable decrease could not be observed (from 50 °C) as the matrix approaches its softening temperature. Fillers have a significant role in increasing the E' of polymeric matrices. As can be seen from Table 2, the values of E' at 25 °C, $E_{25\ ^\circ C}'$, increased generally with filler loading. At all filler loadings, the untreated wood flour appeared to have the highest influence on $E_{25\ ^\circ C}'$, except at 37 wt% where 180 °C treated wood flour exerted the highest influence (1.06 GPa). The reinforcing ability of the fillers could be responsible for this trend. It has been observed [22] that at lower filler loading, restriction of the matrix by the filler is also reduced due to insufficient fillers, leading to highly localized strains occurring in the matrix at low stresses. This results in the breaking of bonds between the matrix and the filler, leaving the matrix diluted by the non-reinforcing de-bonded fillers. However, at higher filler loading, the stress is more evenly distributed throughout the composite, thereby increasing E'. In addition, as the temperature increased, the presence of fillers restricted the flow of the matrix polymer. This restriction is proportional to filler loading [23] thereby enabling the material to maintain a relatively high modulus. Composites containing 200 °C treated wood flour at 37 wt% filler loading influenced $E_{25\ ^\circ C}'$ to a lesser degree (0.76 GPa) than those made from the untreated and 180 °C treated wood flour. The values of E' at $E_{-\ 100\ ^\circ C}'$, at 9 wt% and 20 wt% wood loadings were between 3.1 and 4.8 GPa. However, at 37 wt%, a 31%, 50% and 21% increments were recorded in composites loaded with untreated, 180 °C and 200 °C wood flour, respectively, relative to those containing 20 wt% wood flour. The fact that the reinforcing ability of the wood flour is more evident at higher filler levels is responsible for this behaviour.

The effect of heat treatment on the storage modulus E' of the composites is presented in Fig. 3. Heat treatment seemed to lower the values of E'. At 25 °C, E' values of 0.97, 1.07 and 0.76 GPa were observed for composites made from 37 wt% untreated, 180 °C and 200 °C treated wood flour, respectively. This implied that at 25 °C, the wood flour treated at 180 °C influenced the stiffness of the material than the untreated wood flour and that treated at 200 °C. This could be attributed to a better compatibility between heat treated wood flour and the polymer matrix. It is important to note that wood flour sample treated at 180 °C was reported to enhance the flexural modulus of LDPE matrix when compared with the untreated wood flour and that treated at 200 °C [20]. However, at −100 °C, the untreated wood flour exerted a more pronounced influence on E' than the 180 °C

Table 2 DMA data of red balau/LDPE composites containing untreated and heat treated wood flour

Wood flour content (%)	Treatment temperature (°C)	tanδ			Storage modulus, *E*'			Loss modulus, *E*"	
		$\tan\delta_{max}$	Temperature at $\tan\delta_{max}$ (°C)	$\tan\delta_{25\,°C}$	$E'_{25\,°C}$ (GPa)	$E'_{-100\,°C}$ (GPa)	$E''_{25\,°C}$ (MPa)	$T_{\beta}^{E''}$ (°C)	E''_{max}(MPa)
0	–	0.16	39	0.15	0.26	3.3	40.0	− 25	140.1
9	Untreated	0.16	43	0.15	0.40	4.1	59.2	− 19	170.3
20	Untreated	0.15	46	0.14	0.57	4.8	80.0	− 19	195.6
37	Untreated	0.14	44	0.13	0.97	6.3	134.0	− 16	248.8
9	180	0.16	43	0.15	0.31	3.1	46.6	− 18	129.8
20	180	0.16	49	0.13	0.56	3.8	67.0	− 20	150.4
37	180	0.14	48	0.12	1.07	5.7	130.7	− 17	220.5
9	200	0.16	44	0.15	0.31	3.1	46.5	− 18	130.4
20	200	0.16	50	0.13	0.51	3.8	66.1	− 18	150.3
37	200	0.15	50	0.12	0.76	4.6	93.8	− 16	181.2

$\tan\delta_{max}$, $\tan\delta_{25\,°C}$, $E_{25\,°C}'$, $E_{-100\,°C}'$, $E_{25\,°C}'$, $E_{25\,°C}''$ and $T_{\beta}^{E''}$ are the maximum value of tanδ, tan δ at 25 °C, storage modulus at 25 °C, storage modulus at − 100 °C, loss modulus at 25 °C and temperature at maximum *E*'' in *β*-transition region, respectively

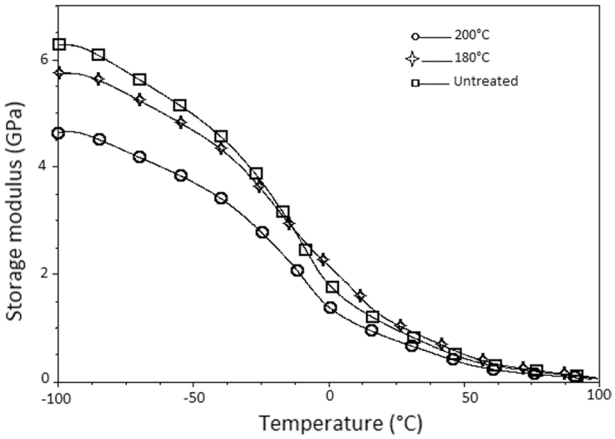

Fig. 3 Storage modulus of 37 wt% composites as a function of heat treatment

and 200 °C treated wood flour. $E_{-100\,^{\circ}C}{}'$ values of 37 wt% composites decreased from 6.3 GPa through 5.7 to 4.6 GPa when untreated, 180 °C and 200 °C treated wood flour were incorporated into LDPE, respectively. A plausible reasoning for this behaviour will be to relate the storage modulus to the flexural modulus of a material since the test was carried out in a three-point bending mode. In the mechanism of flexural deformation, a maximum tensile force is applied on one side of the test piece, translating to a compressive force on the other, such that the stress and strain that are calculated are the maximum outer fibre stresses and strains. Therefore, the parameters measured are those of the outer layer of the material rather than the bulk [23]. In addition, it has been reported that heat treatment of wood flour could lead to decreased mechanical properties [8]. Hence, it is likely that the untreated wood particles are more able to withstand the compressive force on the outer layer of the material than the heat-treated ones. This is in agreement with our earlier report on the flexural modulus of red balau/LDPE composites [20]. This result agrees with the findings of Lou et al. [18], who observed lower E' in wood flour/PP composites containing wood flour treated at 200 °C in glycerine, relative to the untreated counterparts.

Loss modulus, E''

This is also referred to as the imaginary modulus and is a measure of the viscous character or liquid-like nature of the material. It relates the ability of the material to dissipate energy and it is given by the equation;

$$E'' = \frac{\sin\left(\delta_0\right)}{\varepsilon_0} \tag{1}$$

where δ_0 is the maximum stress and ε_0 the strain at maximum stress.

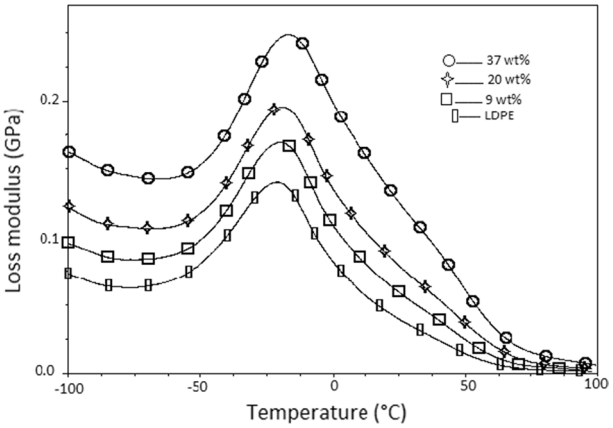

Fig. 4 Loss modulus curves of composites made from untreated wood flour at different filler loading

Loss modulus, E'', curves of LDPE and composites for untreated wood flour at different filler loadings are shown in Fig. 4. Within the experimental temperature range used in this study, only the β relaxation peak was observed for the neat LDPE and the composites. It has been suggested that in LDPE, which is a branched polymer, a clear β-relaxation peak which is associated with the relaxation of the branched points and attributed to the segmental motions in the non-crystalline phase, can be detected [24]. This peak, $T_{\beta}^{E''}$, referred to as the temperature at maximum value of E'', appeared at −25 °C in the neat matrix and shifted to −19 °C in 9 wt% and 20 wt%, then −16 °C as the wood content was increased to 37 wt% in the composites containing untreated wood flour (Table 2). This T_{β} value is associated with the motion of the long chain segments in the amorphous region of LDPE. The higher temperature transition of E'' as the wood content increased may indicate the restriction of the segmental motion of the amorphous LDPE chains, as more wood flour was added. However, low temperature, γ- and high temperature, α-transitions could not be observed within the range of temperature used in this work. In addition, the peak maximum, $E''_{\max}$, and the value of E'' at 25 °C ($E_{25\ ^{\circ}\mathrm{C}}''$) increased with filler loading (Table 2). The $E_{25\ ^{\circ}\mathrm{C}}''$ values increased from 48% through 100% to 235% in composites made from 9 wt%, 20 wt% and 37 wt% untreated wood flour, respectively, relative to the neat LDPE. In addition, an increment of 22%, 40% and 78%, respectively, was observed in $E''_{\max}$ of composites loaded with 9 wt%, 20 wt% and 37 wt% untreated wood flour, respectively, when compared to the neat matrix. The higher $E''_{\max}$ at high filler loading is due to the presence of wood fillers which reduced the flexibility of the material by introducing constraints on the segmental mobility of the polymer chains at the relaxation temperatures [25]. Mohanty et al. [13] and Joseph et al. [22] also reported a similar trend for PP reinforced with jute and sisal fibre composites, respectively. In the same way, Kalaprasad et al. [26] observed that incorporation of short sisal fibre into LDPE resulted in an increase in loss modulus.

Figure 5 presents the effect of heat treatment on the loss modulus of composites loaded with 37 wt% wood flour. As was observed earlier, $T_{\beta}^{E''}$ shifted to higher tem-

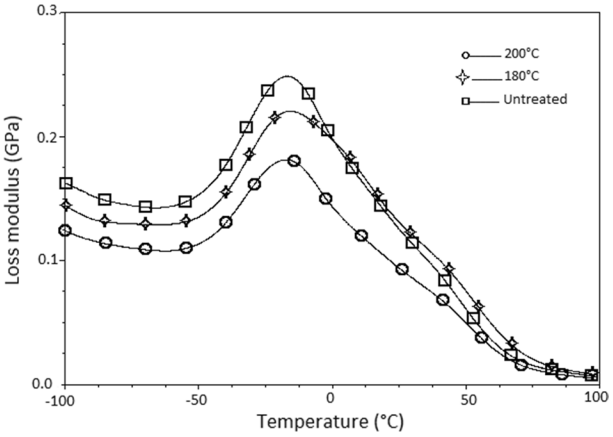

Fig. 5 Loss modulus curves of composites containing 37 wt% wood flour at different treatment temperature

perature with the incorporation of wood flour. However, loading heat-treated wood flour seems to have no effect on $T_{\beta}^{E''}$ as no appreciable change in $T_{\beta}^{E''}$ is observed with addition of wood flour treated at 180 °C and 200 °C. $T_{\beta}^{E''}$ values of −16 °C, −17 °C, and −16 °C were observed in untreated, 180 °C and 200 °C wood composites, respectively (Table 2). Furthermore, in 37 wt% wood composites, $E''_{\max}$ values were generally lower in heat-treated wood composites when compared to the untreated samples. A percentage decrease of 12% and 37% was observed in 180 °C and 200 °C treated wood composites, respectively, relative to the untreated composites. This drop in $E''_{\max}$ in the heat-treated wood composites, relative to the untreated samples, may indicate the presence of improved interfacial bonding [27] between heat-treated wood and LDPE, as a result of better compatibility arising from good wetting of the wood particles by the matrix. At 25 °C, $E''_{25\,^{\circ}\mathrm{C}}$ also reduced with heat treatment (Table 2). $E_{25\,^{\circ}\mathrm{C}}''$ values of 134.0 MPa, 130.7 MPa and 93.8 MPa were observed in composites containing untreated, 180 °C and 200 °C treated wood flour, respectively.

Tan delta (tanδ)

The ratio of the loss modulus to the storage modulus is the mechanical loss factor, also known as tanδ, given by the equation;

$$\tan\delta = \frac{E''}{E'} = \frac{\sin\delta}{\cos\delta} \tag{2}$$

where δ is the phase angle.

It represents the ratio of the energy dissipated to the energy stored per cycle of deformation and indicates the damping characteristics of the material, which typify

the capacity to reduce the transmission of vibration caused by mechanical disturbances to a structure [28].

Figure 6 shows the dependence of *tanδ* on temperature for untreated wood composites at different filler loadings. The tanδ peak maximum decreased generally with increasing wood content, in comparison with the neat matrix (Table 2). This is because as the wood content increased, less amount of LDPE matrix is available to dissipate the vibrational energy [29]. In addition, it is possible that there is an immobilisation of the LDPE matrix by the wood particles during the relaxation process. Tanδ values of 0.16 (recorded for the LDPE matrix and composites loaded with 9 wt% wood flour) dropped to 0.15 and 0.14 when 20 wt% and 37 wt% untreated wood flour were incorporated into LDPE, respectively. Although filler loading is a key parameter in determining the damping properties of composites, other factors, such as the interaction between the inclusion (filler) and the polymer matrix, will influence damping. The reduction in tanδ values denotes an improvement in the hysteresis of the system and a reduction in the internal friction [22]. In addition, the reduced free volume available for segmental motion in the matrix as filler content increased, which resulted in higher energy demands for $\tan\delta_{max}$, is responsible for this trend. As with $\tan\delta_{max}$, the value of $\tan\delta_{25\,^{\circ}C}$ reduced with filler loading. It can also be observed from Table 2 that $\tan\delta_{max}$ values are only slightly different from those of $\tan\delta_{25\,^{\circ}C}$. Nevertheless, lowest values are seen in composites containing 37 wt% wood flour irrespective of treatment temperature. This is thought to be due to the strengthening mechanism of matrix by wood flour and is in line with literature [30]

Figure 7 presents tanδ as a function of temperature for LDPE and composites made from 37 wt% untreated and heat-treated wood flour. In comparison with the neat LDPE, $\tan\delta_{max}$ values of untreated and heat-treated wood composites exhibited lower magnitudes. However, no definite trend can be seen in the composites made from untreated and heat-treated wood flour with respect to $\tan\delta_{max}$ values.

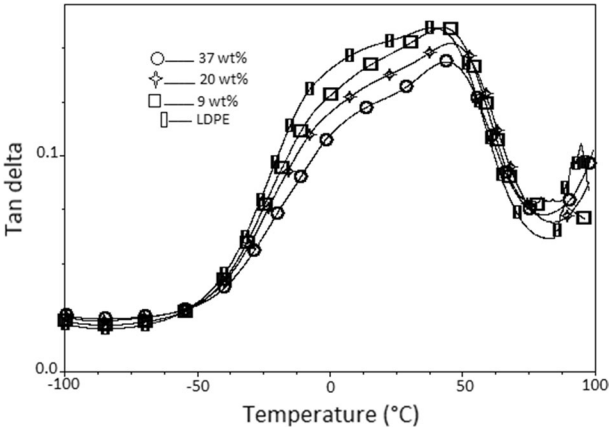

Fig. 6 Variation of tanδ with temperature in LDPE and untreated wood composites at different filler loading

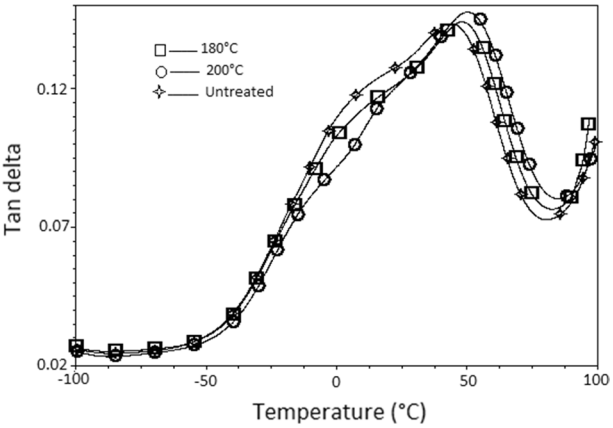

Fig. 7 Dependence of tanδ on temperature for composites made from 37 wt% untreated and heat treated wood flour

Furthermore, temperature at $\tan\delta_{max}$ increased slightly with heat treatment in the composites studied (Table 2). Values of 44 °C, 48 °C and 50 °C were recorded in composites containing 37 wt% untreated, 180 °C and 200 °C treated wood flour, respectively. It is possible that a better interaction between the heat-treated wood flour and LDPE could have resulted in reduced free volume available for segmental motion [18] which may have shifted the $\tan\delta_{max}$ temperatures to higher values, as higher energy is required for the transition to take place. Also, composites made from wood flour treated at 180 °C and 200 °C showed the same values of $\tan\delta_{25\,^{\circ}C}$, indicating that the wood flour from the two treatment temperatures has probably exerted similar influence on tanδ at ambient conditions.

As is observed in Figs. 6 and 7, all the specimens show that the transition is not equal or symmetric on both sides of the main peak. A shoulder seems to form on the left-hand side of the maximum peak, whereas, after the maximum peak (on the right-hand side), the curves declined more linearly without a shoulder. Since the matrix is LDPE, there is a possibility of the presence of some forms of short side chain branches attached to the main chain of the polymer back bone. The shoulder before the maximum peak may be due to the contributions from these short side chains. It is also necessary to state that the temperature range of this experiment will not allow for the lower temperature transitions in the sample to be captured. Therefore, the values reported are for the maximum peak.

Impact properties

Peak load, P

The peak load is the maximum force needed to cause the fracture of the sample. It depicts the maximum point on the load-deflection curve and is a function of the resistance to damage of the material. The peak load of the composites as

Table 3 Impact properties of red balau/LDPE composites containing varying contents of untreated and heat treated wood flour

Wood flour content (wt%)	Treatment temperature (°C)	Peak load per notch depth (N)				Impact energy per notch depth (mJ)			
		0.1	0.2	0.3	0.4	0.1	0.2	0.3	0.4
0	–	266.7 ± 3.2	225.3 ± 8.3	184.5 ± 3.7	137.4 ± 1.5	4616 ± 206	3483 ± 271	3174 ± 120	2117 ± 46
9	Untreated	247.6 ± 13.6	194.8 ± 5.2	155.5 ± 6.2	111.8 ± 6.9	998 ± 85	708 ± 30	586 ± 28	439 ± 31
20	Untreated	236.6 ± 9.1	183.4 ± 5.9	141.3 ± 3.3	109.2 ± 3.9	524 ± 28	399 ± 19	323 ± 17	260 ± 23
37	Untreated	244.0 ± 6.5	199.2 ± 5.7	159.2 ± 3.2	126.8 ± 3.2	258 ± 19	197 ± 07	156 ± 05	128 ± 09
9	180	247.3 ± 6.2	196.2 ± 5.3	152.6 ± 6.3	111.4 ± 4.7	948 ± 61	842 ± 26	563 ± 23	441 ± 34
20	180	241.9 ± 12.1	188.2 ± 7.3	149.6 ± 4.2	118.8 ± 2.7	529 ± 42	393 ± 16	321 ± 17	247 ± 22
37	180	254.2 ± 11.4	205.7 ± 6.4	164.7 ± 5.9	132.1 ± 5.1	256 ± 19	222 ± 70	164 ± 07	133 ± 07
9	200	246.5 ± 7.2	189.7 ± 6.9	144.8 ± 5.6	108.1 ± 1.7	1084 ± 105	771 ± 32	592 ± 49	459 ± 37
20	200	235.3 ± 5.2	178.6 ± 3.9	144.3 ± 5.4	123.1 ± 9.6	477 ± 28	357 ± 28	276 ± 13	249 ± 23
37	200	268.2 ± 14.9	216.9 ± 19.4	188.3 ± 9.6	176 ± 15.6	227 ± 07	170 ± 16	131 ± 09	105 ± 10

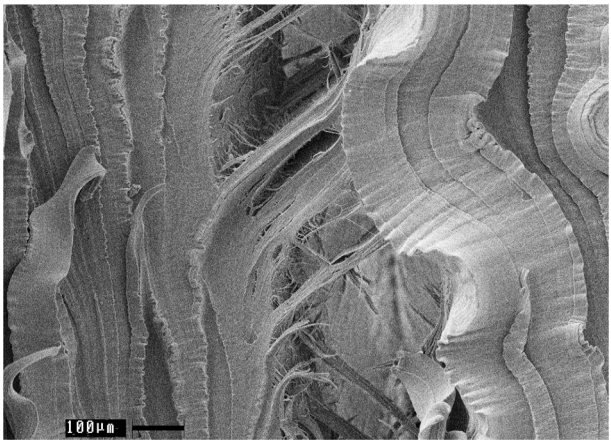

Fig. 8 Impact fractured surface of neat LDPE showing signs of ductility

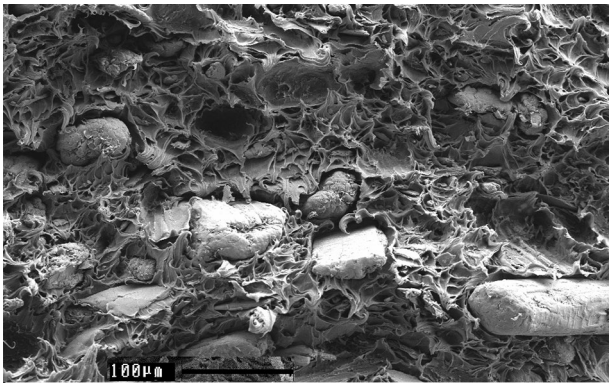

Fig. 9 Impact fractured surface of untreated wood composites at 37 wt%

a function of notch depth at different filler loadings and treatment temperature is presented in Table 3. Generally, *P* decreased with notch to depth ratio, *a*/*D*. This is because increase in *a*/*D* reduces the available length for crack propagation. This in turn decreases the maximum load required for fracture. The neat polymer exhibited the highest *P* values of 266.6 N, 225.3 N, 184.5 N and 137.4 N for *a*/*D* ratios of 0.1, 0.2, 0.3 and 0.4, respectively, because of its ductility as seen in Fig. 8. However, incorporation of wood fillers reduced the *P* of the neat matrix by introducing a measure of brittle behaviour (Fig. 9). It has been said that in WTC, the wood particles are encapsulated in the flexible polymer matrix. Therefore, the average thickness of the polymer layer between two adjacent wood particles would depend on the amount of wood flour in the mixture. Consequently, higher wood content would generally result in thinner polymer layer between wood particles. With the application of an external force, separation or cracking

along the weak polymer-wood interface results [31]. The initiation and propagation of the cracks due to debonding contributed to the weakening of the composites. At lower filler content (9 wt%), *P* values indicated that the samples could not sustain the maximum load and hence, values lower than that of the neat matrix were observed. However, as the wood content increased to 37 wt%, an increment in *P* was observed. This is in agreement with the resistance to sliding offered by the wood filler which is expected to toughen the neat matrix. The higher the wood content, the higher is the resistance. Conversely, at 20 wt% filler content, the values of *P* dropped in all the *a*/*D* ratios except for *a*/*D* ratio 0.4, where an increase in *P* values was observed. A plausible reason could not be given for this trend.

The effect of heat treatment on the *P* values of the composites is also presented in Table 3. *P* values of the neat LDPE were higher than those of the treated and untreated wood composites, except for composites made from wood treated at 200 °C at 37 wt% filler loading. Higher values of *P* in heat-treated wood composites, relative to the untreated composites, could be attributed to the improved interfacial interaction between the wood particles and the LDPE matrix due to the reduced hydrophilicity in heat-treated wood flour when compared to their untreated counterparts.

From the SEM micrograph in Fig. 9, it can be seen that the wood particles pulled out of the matrix, indicating poor bonding between the matrix and the fillers, resulting in lower *P* values of the untreated wood composites. However, Fig. 10 shows portions of the matrix still adhering to the wood particles after fracture, indicating better adhesion and consequently, higher *P* in composites made from wood treated at 200 °C.

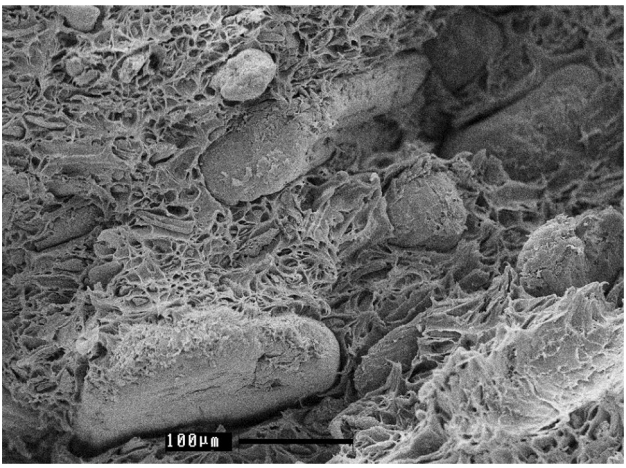

Fig. 10 Impact fractured surface of composites from wood treated at 200 °C at 37 wt% filler loading showing no sign of ductility

Critical stress intensity factor or fracture toughness, K_c

K_c represents the materials ability to withstand applied force (load). 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:

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

Rearranging Eq. 3 gives

$$\sigma Y = \frac{K_c}{\sqrt{a}} \tag{4}$$

K_c is 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 new crack surface and it characterizes 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 [32]. In a three-point bend test, σ is given by simple bending theory as,

$$\sigma = \frac{6PS}{4BD^2} \tag{5}$$

where P is the load, S is the support span, B and D are the specimen width and thickness (depth), respectively.

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

$$Y = 1.93 - 3.07\left(\frac{a}{D}\right) + 14.53\left(\frac{a}{D}\right)^2 - 25.11\left(\frac{a}{D}\right)^3 + 25.80\left(\frac{a}{D}\right)^4 \tag{6}$$

From Eq. (4), a plot of σY against $a^{-0.5}$ gives a straight line, where the slope equals the K_c of the materials.

The variation of K_c with wood content is shown in Fig. 11. As has been observed with P, K_c increased with wood content. This is consistent with literature [16, 33]. Incorporation of stiff wood particles into the flexible LDPE matrix was expected, in theory, to toughen the composites system, due to stress transfer between the wood particles and the matrix resulting in high modulus. From these results, the toughening was higher with increasing wood content. The presence of wood particles tended to reduce the resistance to crack initiation, resulting in the brittleness of the material, while at the same time, reducing crack propagation through the matrix by forcing crack lines around the particle ends [34]. As observed earlier, lower wood content did not contribute appreciably to the toughening of the composite. This is because at low wood content, wood particles may act as notches, a possible reason for the low K_c values observed at 9 and 20 wt%. However, the reinforcing effect of the wood particles is seen at 37 wt% wood flour loading, where a relative improvement in K_c values was observed.

The effect of heat treatment on the K_c values of the composites is also seen in Fig. 11. K_c improved with heat treatment, relative to the untreated wood composites

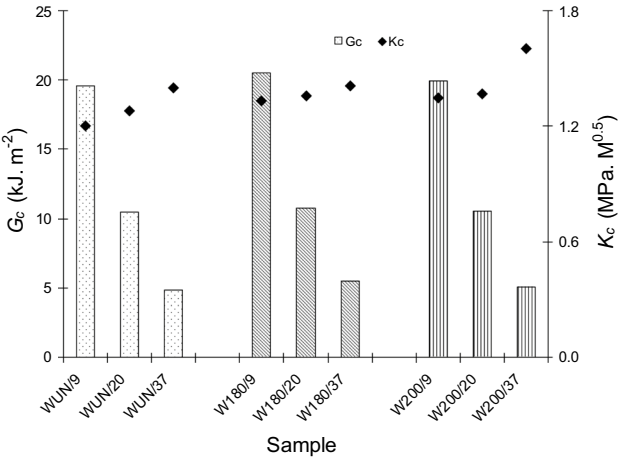

Fig. 11 Changes in K_c and G_c of composites as a function of wood content and treatment temperature

at all compositions. However, composites made from the 37 wt% untreated wood flour and those containing the same amount of wood flour treated at 180 °C had almost the same the values of K_c (1.40 MPa.m$^{0.5}$ and 1.41 MPa m$^{0.5}$, respectively), while composites filled with wood flour treated at 200 °C produced composites with the highest K_c value of 1.6 MPa.m$^{0.5}$ at 37 wt%. Improved compatibility between the wood particles and the LDPE matrix, as a result of reduced polarity of heat-treated wood flour and a better dispersion of the fillers within the matrix, must have resulted in improved toughness. Stiffness and improved adhesion have been observed to hinder crack initiation [33]

Failure energy, W

The failure energy represents the total energy the material will absorb until full penetration of the impactor tup. It is the energy under the force–deflection curve up to peak deflection where propagation of damage takes place until fracture. Table 3 summarizes the influence of filler loading at different *a/D* ratios on *W*. *W* decreased with *a/D* at the same composition. Values of 4616, 3482, 3174 and 2117 mJ were recorded for *a/D* ratios 0.1, 0.2, 0.3 and 0.4, respectively, in the neat LDPE. As mentioned earlier, this is as a result of the reduction in the fracture area as the notch depth increased. *W* of pure LDPE was significantly higher than that of the composites. The ductile nature of the neat LDPE required higher energy to break the specimen. However, as the wood content increased, a decrement in the values of *W* was observed. At high wood content, many wood particle ends exist within the composite, which could aid crack propagation and consequently, premature failure. Also, higher wood loading leads to agglomeration of the wood particles which, in addition to the already existing notches, could act as stress concentration points requiring lower failure energy. This phenomenon increased with increase in wood content, with wood flour loading at 37 wt% showing the lowest value of *W*. In addition, poor

interfacial bonding between the fillers and the matrix resulting from poor compatibility may also be responsible for the trend observed.

The effect of heat treatment on *W* of the composites is also seen in Table 3. At low wood content (9 wt%), a clear trend could not be established with respect to the effect of heat treatment on *W*. At this composition, the reinforcing effect of the wood flour was not pronounced and as such, the behaviour of the neat matrix may have predominated. However, as the wood content increased to 20 wt% and 37 wt%, the effect of heat treatment seemed evident. Composites made from wood flour treated at 180 °C appeared to influence the *W* of the composites more than those made from the untreated and wood flour treated at 200 °C. This indicated that treating wood at 180 °C had modified the wood flour to a certain degree, which enhanced better compatibility between the non-polar LDPE matrix and the polar wood particles relative to the untreated wood composites. This trend was also observed in previous reports where the tensile strength of composites from wood flour treated at 180 °C was higher than those of untreated and 200 °C treated wood composites [20]. Heat treatment of wood flour at 200 °C might have caused the deterioration of the mechanical properties of the wood particles and lowered its stress transfer efficiency, resulting in reduced *W*.

Critical strain energy release rate, G_c

G_c, is a material property referred to as the impact strength, toughness, critical strain energy release rate or crack extension force. It is effectively the energy required to increase the crack length by unit length in a piece of material of unit width. It has units of J m^{-2} [35]. The relationship between *W,* G_c and specimen geometry function, $BD\varphi$, is given by,

$$W = G_c BD\varphi \tag{7}$$

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,

$$\varphi = \frac{1}{2}\left(\frac{a}{D}\right) + \frac{1}{18\pi}\left(\frac{S}{D}\right)\left(\frac{a}{D}\right)^{-1} \tag{8}$$

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

The effect of wood content on the *G*c of the composites is presented in Fig. 11. G_c decreased with wood content, with the highest G_c value observed at 9 wt% wood flour loading and the lowest at 37 wt% filler level. As has been observed for *W*, this decrement could result from the presence of wood particle ends within the LDPE matrix which tends to aid crack propagation. Furthermore, at high wood content, the possibility of the wood particles agglomerating is high. This, as seen earlier, could act as stress concentrators, thereby enhancing crack growth. Várdai et al. [36] investigated the impact modification of polypropylene by various elastomer and polyethylene terephthalate fibre contents at a constant wood flour content of 20 wt% to produce hybrid composites. They observed that in the polypropylene homopolymer,

limited impact resistance and brittle fracture can be observed independent of wood content and the strength of interfacial adhesion. This they attributed to restricted plastic deformation in wood plastic composites. Therefore, it is expected that higher wood content should present lower impact strength.

The influence of heat treatment on the G_c of the composites is also shown in Fig. 11. From the figure, it can be seen that G_c increased marginally with heat treatment at all filler loadings, with composites made from wood flour treated at 180 °C exhibiting the highest value. A look at 37 wt% wood content revealed that a value of 4.85 kJ m^{-2}, 5.47 kJ m^{-2} and 5.08 kJ m^{-2} were obtained for composites made from untreated, 180 °C and 200 °C treated wood flour, respectively. The marginal increase in the G_c of heat-treated wood composites relative to the untreated samples indicates some level of modification by heat treatment which rendered the wood more compatible with the non-polar LDPE matrix, thereby showing some degree of resistance to crack propagation when the notched composites were subjected to impact testing.

Conclusion

The dynamic mechanical analysis revealed that composites made from the heat-treated wood flour exhibited higher storage modulus and loss modulus than those containing untreated wood flour. Furthermore, lower tan delta values were observed in heat-treated composites, relative to the untreated materials as a consequence of decreased damping, resulting from improved interaction between the heat-treated wood particle and the LDPE matrix.

Peak load and critical stress intensity factor increased with wood content and treatment temperature. While the energy to failure and the critical strain energy release rate decreased with wood content, the values were highest in composites made from wood flour treated at 180 °C and reduced in 200 °C heat-treated wood composites. This behaviour revealed that heat treatment of wood flour at the 200 °C resulted in poorer impact properties of the composites. In general, heat treatment of wood flour at appropriate treatment temperature produced composites with improved mechanical and dynamic mechanical performance.

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Compliance with ethical standards

Conflict of interest The corresponding author, on behalf of the remaining authors, wishes to declare that there is no conflict of interest.

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