Experimental Investigation of Epoxy Matrix and Pine Sawdust Reinforced Wood-Polymer Composite Materials

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Mechanical, thermal, and water absorption properties of the composites have been studied as a function of sawdust content, using different weight percentage. The characteristics properties of the composites were studied using differential scanning calorimetry and Fourier-transform infrared spectroscopy. Field emission scanning electron microscopy was used to understand the interfacial bonding. The obtained results showed that the 15 wt% composites exhibited the highest tensile strength (7.5 MPa) and flexural strength (8.9 MPa) compared with the 5 wt%, 30 wt%, 40 wt%, and 50 wt% composites. A good interfacial combination was formed between 15 wt% of sawdust and epoxy resin. In terms of the tensile and flexural strength, the differential scanning calorimetry analysis confirmed that matrix modification could improve the mechanical properties and thermal stability of the composites compared to neat resin. The Fourier-transform infrared spectroscopy spectrum showed the presence of functional groups pertaining to composites. The absorption data of the composite showed that the water uptake increased as the amount of sawdust in the composite increased. The 5 wt%, 15 wt%, 30 wt%, and 40 wt% sawdust composites also displayed less water absorption behavior (1.534%, 1.871%, 2.492%, and 4.127%, respectively) compared to the 50 wt% composite.

DOI: 10.15376/biores.17.1.1161-1172

Keywords: Pine wood, epoxy; Green composite; Moisture effect; Mechanical properties

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INTRODUCTION

The wood waste for consumable items is getting prime attention due to stringent environmental laws and waste management issues. The recycling of wood waste can be increased by developing wood waste composite products (Khan *et al.* 2021). Furthermore, the shortage of wood materials has become a major problem facing the world, which necessitates the utilization of waste materials, such as sawdust (SD) and particleboard that can be used as valuable resources for the manufacture of many products and provide solutions to many economic and environmental problems. Natural fibers have attracted the attention of many researchers, as they are low cost, highly available, biodegradable, and eco-friendly materials (Ashori 2008). However, the main drawbacks of wood particles are their relative low degradation temperature and their hygroscopicity, which weaken their adhesion with hydrophobic polymers. However, wood fibers show very good mechanical properties (Marcovich *et al.* 1996). At the same time, the use of natural fibers and polymers from renewable resources has recently attracted increased attention, predominantly due to

environmental concerns and the depletion of petroleum resources (Ridzuan *et al.* 2016; Vijayan and Thiagarayan 2020). They have a relatively high strength and stiffness, as well as a low cost, low density, and low CO_2 emissions, and are biodegradable and renewable. Kamdem *et al.* (2004) and Deka *et al.* (2014).

It is important to investigate the specific epoxies and polymeric materials used for wood waste composites. For instance, the use of formaldehyde resin is detrimental to the environment, due to the emission of toxic gases during composite development processes Liang *et al.* (2016). The controlled usage of synthetic adhesive is decisive to eliminate the toxic and environmental impacts. Other resins, such as soy-based resin and polyurethane resin, must be considered as an alternative to toxic resins in developing wood waste composites (Salles *et al.* 2016). Among different types of resins, epoxy resins are environmentally friendly and easier to process (Khan *et al.* 2021). They have a very wide application range from dental fillings to rocket casings. The characteristics, which provide such a diversity of applications, are explained by the chemistry of the epoxide functional group as well as the curing reaction (Bilyeu *et al.* 1999). Functionally, this resin exhibits excellent properties due to its high cross-linked characteristics, adequate strength, low cure shrinkage, increased fatigue and impact resistance, excellent thermal properties, chemical resistance, and dimensional stability. These properties help in developing molds in the desired shapes. Epoxy/hardener ratios and ideal curing schedules are important to attain the desired properties of composite materials (Khan *et al.* 2021).

The research on polymer-based composites has opened new avenues for polymer formulations and developing new types of composites with specific properties of choice for specialized applications. Khan *et al.* (2021) carried out experimental and statistical analysis of sawmill wood waste composites properties for practical applications. Wood and wood waste type and wood waste content have been found to have significant effects on all mechanical properties (Marcovich *et al.* 1996). It was concluded that no improvement in mechanical properties was found when the sawdust was chemically modified. A low interfacial area is suspected to be responsible for this fact. Many research studies elaborated the mechanical behavior of natural wood fiber composites, their varying the concentration of reinforcements, for example, the use of pine cone powder in high-density polyethylene (HDPE at different weights (5%, 10%, 15%, and 20%) substantially improves the mechanical strength (Agayev and Ozdemir 2019). Considering this, the present study focuses on the effects of the proportion of pine sawdust reinforced with epoxy resin polymer to improve the mechanical strength, thermally stability, and water absorption.

EXPERIMENTAL

Materials

Pine sawdust was provided by Department of Material and Material processing Techology, VanYY Univ. of Turkey. Firstly, the pine wood sawdust was analyzed, and it was found to have an average particles size of 100 to 200 nm and an average water content of 21.6%. Then, the pine wood sawdust was dried at a temperature of 105 to 110 °C in an oven. Purpox® epoxy resin EFLR-0190 provided by Polikor Inc. (Turkey) was used as matrix material, which is a solvent free resin with a transparent coating. The density of this resin was 1.00 to 1.10 g/cm³, the while the viscosity was 300,500 mPa. The epoxy resin and hardener were mixed at a weight ratio of 100:50 to produce the composite materials.

Preparation of the Composite

In this study, the low temperature curing epoxy resin (EFLR-0190) chemically belonging to the epoxide family and the corresponding hardener were mixed in a ratio of 100:50 by weight. The sawdust, epoxy resin, hardener were blended using a three arm mixer (Dispermant (R30 VMA Getzmann GMBH verfahrenstenchni Co., Ltd. of Germany) at 30 rpm and a temperature of 23±2 °C for 10 to 15 min. The mixture slurry was poured into a metal mold, as per ASTM standard D3039 (2017). The curing of the whole mixture in the chamber was done for 24 to 48 hours at room temperature.

Tensile Strength Test

The tensile strength tests were performed at room temperature at a rate of 2 mm/min in accordance with ASTM standard D3039 (2017), using a universal testing machine (Instron 3369, Norwood, MA) with a 50 kN load cell. The dimensions of the sample were 250 mm x 25 mm x 3 mm for the length, width, and thickness, respectively. The tensile strength testing was repeated five times for each composite and the average value of results reported.

Flexural Strength Test

The flexural testing was performed according to the ASTM standard D790 (2017). The dimensions of the specimens were 3.2 mmx 12.7 mm x 125 mm) and tested at a crosshead speed of 2 mm/min. The tensile strength testing was repeated five times for each composite and the average value of results reported.

Water Absorption Test

Composite samples were cut into blocks of (2.5 cm x 0.5 cm x 2.5cm) for this study. The specimen was maintained in distilled water at room temperature (23±2), and weights were measured after 1, 2, 3, 5, 7, 11, and 14 days. Before being weighed, the water on the specimen surface was wiped off using filter paper. The calculation was based on Eq. 1 (Biplab and Maji 2012),

$$W_{water\ absorption(\%)} = \left[\frac{W_t - W_0}{W_0}\right] \times 100 \quad (1)$$

where w_t is the weight of the sample after immersion time of t, and w_o is the initial weight of the sample.

Scanning Electron Microscopy (SEM) Analysis

Field emission scanning electron microscopy (FESEM), with a Carl Zeiss Sigma 300 (Oberkochen, Germany) with an energy dispersive X-ray spectrometer, 10 kV SE detector. Following tensile and flexural testing, FESEM was used to study the fracture surfaces of the sawdust/epoxy composites after tension failure. The sawdust/matrix adhesion within the composite was examined to determine the failure mode of the samples.

Fourier-transform Infrared (FTIR) Analysis

The interactions between the epoxy polymeric chains and the sawdust components (lignin, hemicellulose, and cellulose) were investigated by Fourier-transform infrared (FTIR) spectroscopy using a Bruker Alpha model spectrophotometer (Oberkochen, Germany) within a wavenumber range of 4000 to 500 cm^{-1}. A scanning rate of 60 scans per min at a resolution of 4 cm^{-1} was maintained during the experiment.

Differential Scanning Calorimetry (DSC) Analysis

Differential scanning calorimetry analysis of the composites were performed using a Labsys Evo DSC machine (Geneva, Switzerland); the composite samples weighing 10 mg were heated in an aluminum sample pan at a temperature range of 30 to 550 °C at a heating rate of 10 °C/min. The experiment was conducted in Ar atmosphere with the pumping rate of the Ar gas maintained at 20 mL/min.

RESULTS AND DISCUSSION

Water Absorption

Figure 1 shows the water absorption of the neat epoxy and the sawdust-epoxy composites as a function of time. From the figure, it was observed that pure epoxy blend absorbed less water than the composites, as expected. This was due to the fact that the epoxy had hydrophobic properties. The water absorption content further increased as the pine sawdust content of the samples increased. The increase in water absorption increased as sawdust content increased from 40 to 50 wt%. It was also observed that all formulations of the blend gave higher water absorption values than that of pure epoxy resin. The increase in water absorption was attributed to the hydrophilic nature of sawdust. The increased affinity of the composites to water was due to the hydroxyl groups of pine wood and poor interfaces between the pine wood sawdust and epoxy resin. In addition, the hydroxyl groups interact with water through hydrogen bonding, which results in water uptake and weight gain in the composites. A similar observation was also reported by Krishna *et al.* (2018) and by Biplab and Maji (2012).

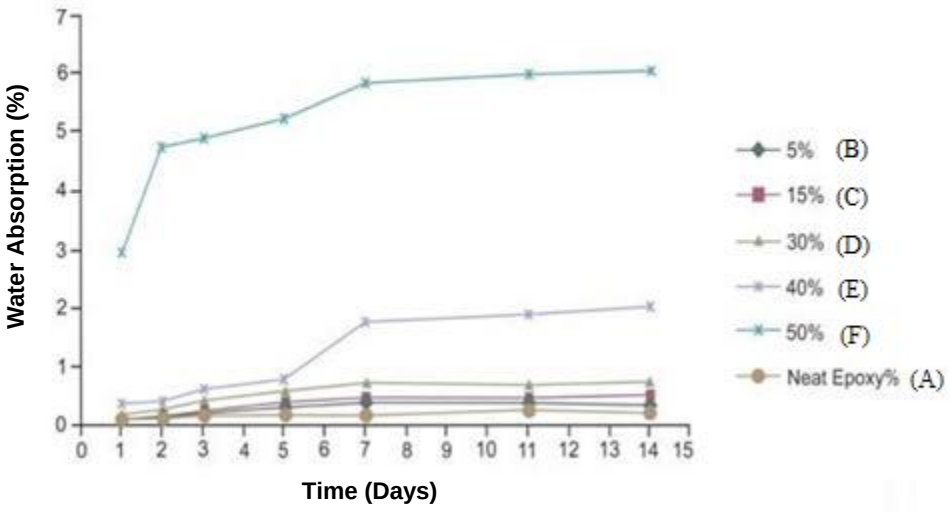

Fig. 1. Water absorption of all the composites as a function of immersion time

Tensile Strength Test

Figure 2 shows the effect of the sawdust amount on the tensile strength. Generally, the variation tendency of the specimens between the tensile strength values were close to each other. The pure epoxy sample exhibited a low tensile strength. However, the tensile strength values of the composites partially increased due to a reinforcing effect of the

sawdust. As shown in Fig. 2, it is clear that the tensile strength of the 15 wt% composite increased as the sawdust content gradually increased, up to 30 wt%, but then it decreased. This may be due to a decrease in epoxy resin percentage that binds the composite firmly The other possible reason may be the weak interfacial bonding between polymer matrix and filler contents that decreases the tensile strength of the composite. According to Huda *et al.* (2006), the accumulation of wood powder or inadequate hydrogen bonding between wood powder and epoxy resin matrix causes a decrease in tensile strength. A similar observations were also reported by Lette *et al.* (2018), Marcovich *et al.* (1996), and Khan *et al.* (2021).

Table 1. Different Epoxy to Sawdust Ratios and their Tensile and Flexural Strength

Samples	Epoxy (wt%)	Sawdust (wt%)	Tensile Strength (MPa)	Flexural Strength (MPa)
A	100	0	5.2	1.5
B	95	5	5.7	5.6
C	85	15	7.5	8.9
D	70	30	6.6	6.1
E	60	40	5.0	5.0
F	50	50	1.0	1.2

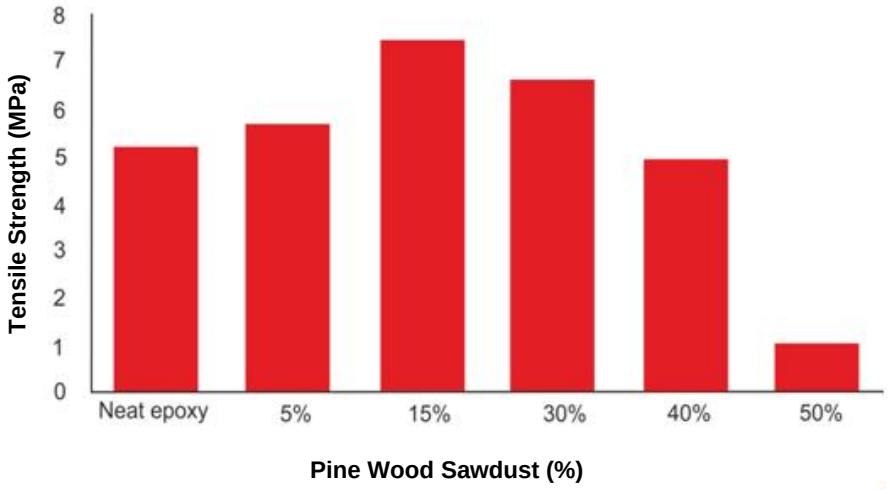

Fig. 2. The tensile strength of composites with different percentages of pine wood sawdust

Flexural Strength Test

The flexural strengths of pure epoxy and the various samples are depicted in Fig. 3. The C sample showed a higher flexural strength than the neat epoxy and the other samples. The B, and E samples had drastically improved flexural strength, but the 50 wt% sawdust composite had the lowest flexural value. It is fair to say that the flexural strengths decreased with increasing amount of sawdust. The flexural strengths of samples with 5 wt% 30 wt % and 40 wt% sawdust exhibited similar close values. Marcovich *et al.* (1996) claimed that increasing the composite fibre weight fraction would increase the void content

of the composites, which would affect the physical and mechanical properties of the composite. Ku *et al.* (2012) also found that the flexural strengths of size of sawdust epoxy composites decreased steadily with increasing great particle size. Thus, the greater the amount of the sawdust in the samples, the larger would the voids in the composites and hence the lower the physical and mechanical properties of the composites. This was found to be correct in the present study.

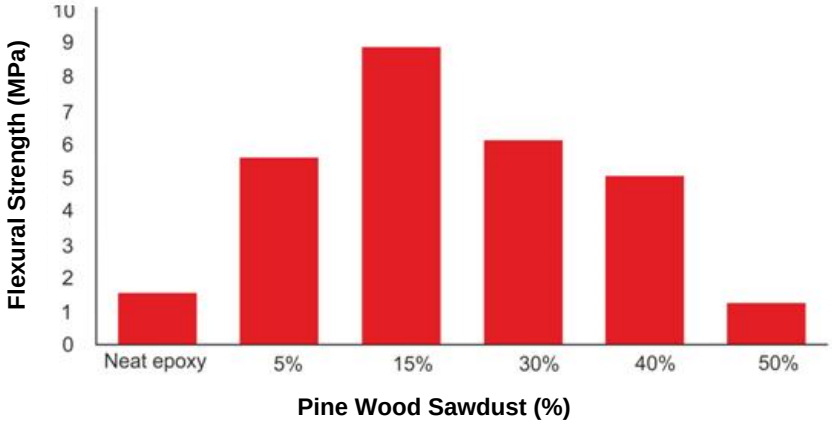

Fig. 3. Flexural strength of composites with different percentages of pine wood sawdust

Scanning Electron Microscopy (SEM) Analysis

Figures 4a through 4c shows the field emission scanning electron microscope (FESEM) examinations of the pure epoxy matrix and the 15 wt% and 50 wt% pine wood sawdust composites, respectively. In Fig. 4a, faint striations followed by a turbulent flow were found in the fractured surface of the neat resin. This shows that plastic deformation had taken place in the resin. A similar outcome was reported by Ku *et al.* (2012).

In Fig. 4b it is apparent that the sawdust had good adhesion to the epoxy resin. However, the sawdust particles agglomerated, and a considerable number of bubbles and cavities were present that could interfere in the mechanical properties of the composites. At 15 wt% sawdust there was good adhesion in epoxy, whereas at 50 wt% there apparently was not a sufficient amount of epoxy resin to fully well all of the cellulosic surfaces. From these images, we can understand why 15 wt% composites showed better mechanical properties than 50 wt% composites. The sawdust pull-out position, sawdust clumping, and in homogeneously distributed epoxy and sawdust were observed in both composites. Similar observations were found by Lette *et al.* (2018) and Kumara *et al.* (2014).

In Fig. 4c it is shown that there were more gaps between sawdust and matrix, which means a poor adhesion or interfacial bonding for 50 wt% sawdust sample. The void, matrix cavity, and small gap formed was probably caused by incomplete wettability or bonding between matrix resin and sawdust during the fabrication of composites. Additionally, this can be attributed to the breaking of the bonds between the pine wood sawdust and the epoxy, as these bonds cause the pine wood sawdust to associate and pull the matrix to a location of joining. This may be reason for composite had the lowest tensile and flexural values. These results are according to Kumar *et al.* (2015) on utilization of CF/sawdust reinforced epoxy hybrid composites on mechanical properties.

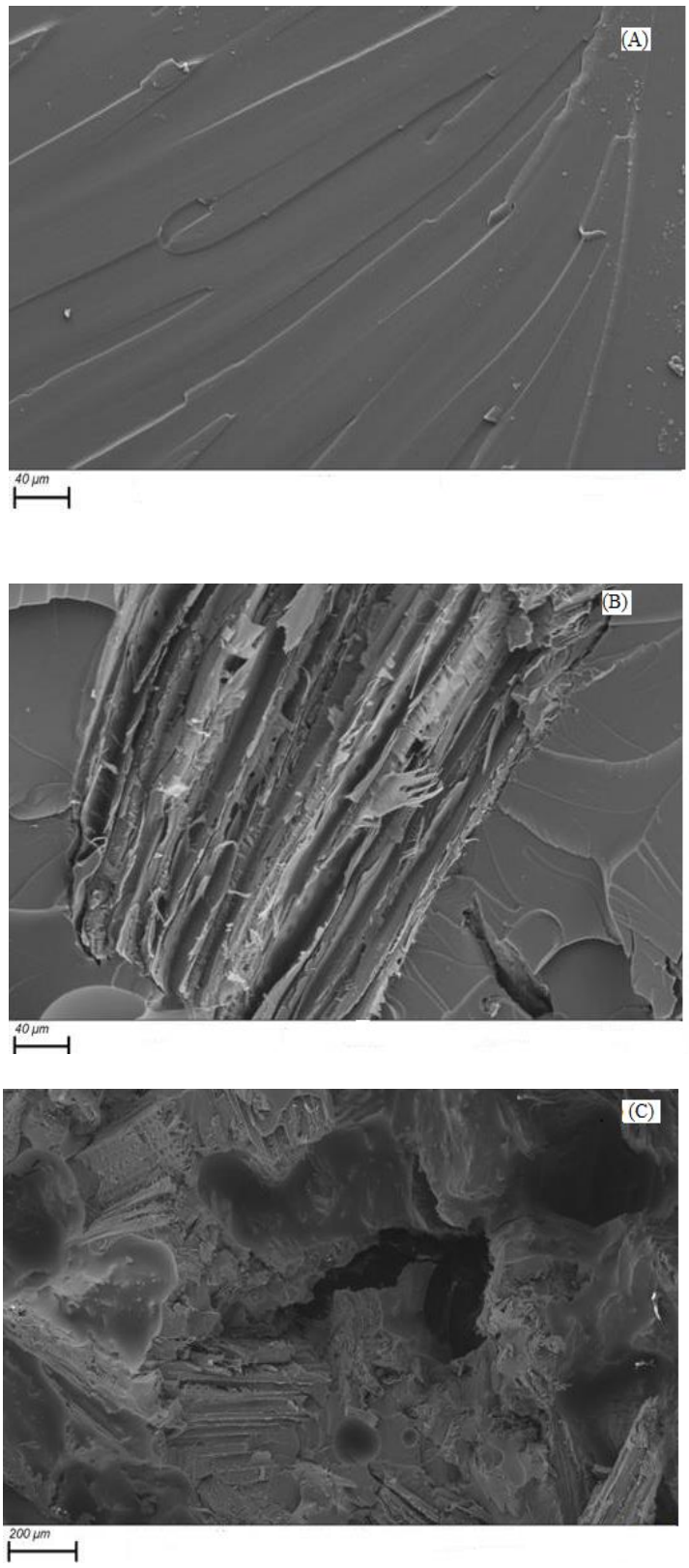

Fig. 4. SEM images of neat epoxy (a); 15 wt% sawdust (b); and 50 wt% sawdust (c) composites

Fourier-transform Infrared (FTIR) Analysis

The transmittance spectra of the FTIR for the pure epoxy, 15 wt%, and 50 wt% pine wood composites are shown in Fig. 5.

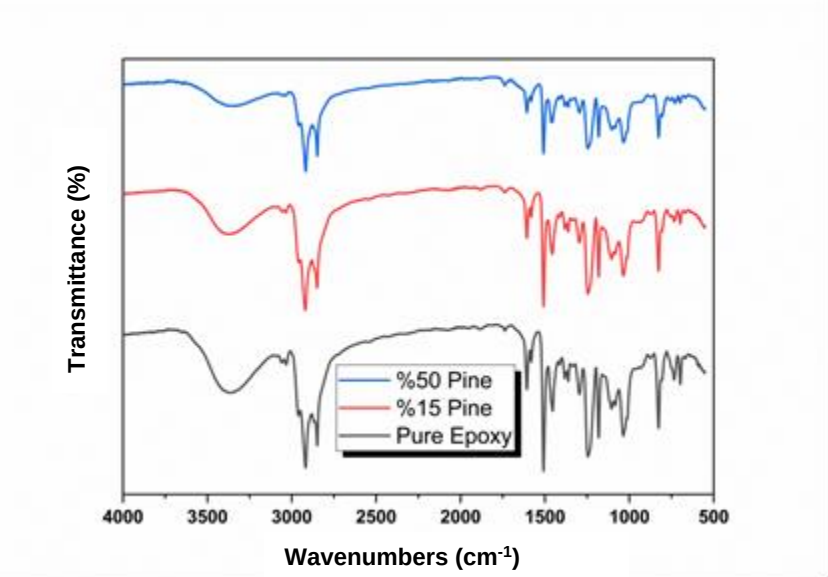

Fig. 5. FTIR spectra for the pure epoxy and 15 wt% and 50 wt% of pine wood sawdust composites

Table 1. Assignments of FTIR Wavenumbers Identified in the Pure Epoxy and Epoxy/Pine Sawdust Composite Sample (Mendes *et al.* 2021)

Wavenumber (cm^{-1})	Functional Group	Vibrational Assignment
3325	O–H	O-H Axial deformation.
3015	C-H	Axial deformation of the C–H bond of aromatic ring.
2900	CH_2 and	CH_2 group vibration stretch present in lignin.
2775	CH_2	Aromatic ring stretching.
1615		Stretching.
1500	C=C	Axial deformation.
1375		Angular deformation.
1250	CH_3	C–O vibrations: ethers and alcohols present in lignin.
1235	C-O	Symmetric axial deformation of the epoxy ring where all ring bonds expand and contract in phase.
1020		Axial deformation or stretching.
800	C–N	Asymmetric axial deformation of the epoxide ring in which the C–C bond length increases and the C–O bond length decreases.
	C–O–C	
750		
	C-O-C	Symmetric in-plane deformation.

Table 1 describes the main vibrational modes associated with epoxy, lignin, and cellulose identified in the FTIR spectra of the samples. The epoxy resin FTIR spectrum, seen in Fig. 5, is characterized by four typical bands at around 1500 cm^{-1}, 1250 cm^{-1}, 1020, and 800 to 750 cm^{-1}. The first band at 1500 cm^{-1} is related to axial deformation, the second band 1250 cm^{-1} is related to symmetric axial deformation, in which all the ring bonds expand and contract in phase, the third band at approximately 1020 cm^{-1} is attributed to the asymmetric axial deformation of the ring, in which the C–C bond increases and there is a contraction of the C–O bond, and the fourth band at approximately 800 to 750 cm^{-1} is

assigned to symmetric deformation in the plane of the C–O–C bond. The sawdust-epoxy composites FTIR spectrum, seen in Fig. 5, shows common bands at approximately 1700 cm^{-1} from carboxyl groups in hemicelluloses and 1255 cm^{-1} and approximately 1050 cm^{-1} from C–O vibrations in lignin. It is specific interaction between cellulose and/or lignin with the epoxy resin can be identified. The similar results were noticed mechanical and micro structural characterization of epoxy/sawdust composites (Mendes *et al.* 2021; Yin *et al.* 2012).

Differential Scanning Calorimetry (DSC) Analysis

Differential scanning calorimetry analysis is used to determine the amount of thermal energy released or absorbed through exothermic or endothermic reactions when a material is subjected to heating (Manivel *et al.* 2021). Figure 6 shows the DSC plot of sawdust composites and epoxy composite showing the endothermic and exothermic transition at variable temperatures. In the DSC curves of the F, C, and A samples, two endothermic peaks were observed at 50 to 100 °C and 350 to 380 °C, respectively. The early peak from 56 to 60 °C specifies the T_g of three specimens. It is observed that T_g lay in between 56 to 60 °C. Finally, the highest T_g (60 °C) was detected for pure epoxy composite. From basic research, due to the low moisture content of the pure epoxy composite, the glass transition temperature is high. The glass transition curve exhibited a similar trend for sawdust loading, both for 15 wt % and 50 wt% sawdust epoxy composite.

The second endothermic peak at a temperature between 350 to 380 °C was observed, which is corresponds to the thermal depolymerization of the hemicelluloses and cellulose. However, the 15 wt% sawdust composite exhibited an exothermic peak at 364 °C. Hence, the addition of sawdust at 15% increased the value of T_d, due to its strong adhesion between matrix and sawdust. Previous studies indicated that pure epoxy and sawmill wood sawdust epoxy composite exhibit similar T_d values (Khan *et al.* 2021).

Similar results were reported for nanocellulose and cellulose fibers (Saba *et al.* 2017; Manivel *et al.* 2021). The DSC results revealed that the 15 wt% ratio is the optimal sawdust composite thermally stable epoxy composites, as it offered suitable resistance and stability towards heat.

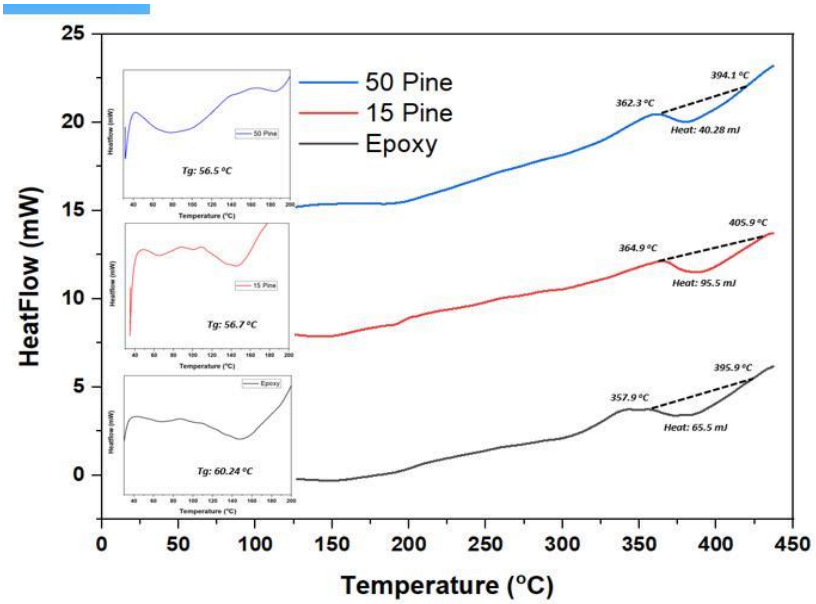

Fig. 6. Heating curves of the DSC for the epoxy/sawdust composites

CONCLUSIONS

From the research work herein, the following conclusions can be drawn:

1. The maximum tensile and flexural strength was achieved with 15 wt% sawdust and promoted better sawdust/matrix adhesion.
2. Water absorption of the composites increased with the incorporation of sawdust in the composites. Water absorption of the epoxy composite material is lower than other composites
3. The differential scanning calorimetry (DSC) analysis revealed the thermal stability (up to 365 °C) and kinetic activation energy (18.74 J/g) of the 15 wt% pine sawdust/epoxy composite, which are important prerequisites for composite applications.

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Article submitted: September 24, 2021; Peer review completed: November 14, 2021; Revised version received and accepted: December 15, 2021; Published: January 3, 2022.
DOI: 10.15376/biores.17.1.1161-1172