Thermal Stability and Dynamic Mechanical Properties of Kenaf/Bamboo Fibre Reinforced Epoxy Composites

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An increased awareness of environmental concerns has increased the need for innovation to produce high performance engineering materials with natural renewable resources. In this study, 3 types of natural fibre (mat form) reinforced epoxy composites were prepared by the hand lay-up method, namely, kenaf (K)/Epoxy, bamboo (B)/Epoxy, and bamboo charcoal (BC)/Epoxy. The thermal stability of the specimens was investigated by thermogravimetric analysis (TGA) and the dynamic mechanical properties. Viscous elastic behaviour of the specimens was investigated *via* a dynamic mechanical analyzer (DMA). The TGA results revealed that the BC/Epoxy composite showed the highest thermal stability compared to K/Epoxy and B/Epoxy with the highest initial and final decomposition temperature at 348 °C and 463 °C, respectively. It also showed the highest charcoal content at 11.5%. From the DMA results, the K/Epoxy composite showed better dynamic mechanical properties with the highest complex modulus (E^*) strength and the lowest damping behaviour (peak height of Tan δ). The DMA analysis also revealed that the glass transition temperature of the composites fell between 60 °C to 90 °C. This preliminary study may give a new path to develop a novel hybrid composite that offers unique properties unachievable in a single material system.

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INTRODUCTION

Natural fibre polymer composites (NFPC) are a class of composite material consisting of a polymer matrix embedded with high-strength natural fibres, such as jute, oil palm, sisal, kenaf, bamboo, or flax (Ku *et al*. 2011). The reinforcement by fibres provides strength and stiffness to the composite, while the matrix protects and transfers the load between fibres. The NFPCs are garnering much attention from the research community due to the increase in environmental consciousness and community interest, new environmental regulations, and the unsustainable consumption of petroleum. According to a recent published report in December 2015, the global natural fiber composites market is forecasted to grow at a compound annual growth rate (CAGR) of 8.2% from 2015 to 2020 (Lucintel 2015). The major driver for the growth of this market is the rise in demand for lightweight and environmentally sustainable composite materials

for various applications, such as automotive, building, construction, and others. The applications of NFPCs has increased significantly and are becoming preferred to carbon and glass fibre. This is due to natural fibres excelling in most of the parameters such as price, specific weight ratio, environmental compatibility, recyclability, and work safety. However the strength is still comparable to the synthetic fibres such as glass and carbon fibre (Faruk *et al*. 2014).

Despite the advantages possessed by a natural fibre, there are a few drawbacks that limit the applications of NFPCs in the industry, such as the hydrophilic nature of the fibre surface that leads to weak interfacial bonding between the hydrophilic fibers and hydrophobic polymer matrices, variability of properties (depending on the quality of the harvest, age and body of the plant from which they are extracted, extracted technique, and the environmental conditions), and relatively low thermal stability and mechanical properties, are comparable to synthetic fibre (Jawaid and Abdul Khalil 2011; Julkapli *et al*. 2015).

Kenaf and bamboo fibres are both commonly used reinforced natural fibres with many well-established applications. Kenaf fibre has a short plantation cycle, wide adaptation to environmental conditions, and requires a relatively low quantity of pesticides, making it more favorable when compared with other lignocelluloses fibre crops (Wang and Ramaswamy 2003). Studies have shown that the mechanical properties of neat polymers (without fibre reinforcement) were improved by the addition of kenaf fibres (Azwa and Yousif 2013).

In recent years, kenaf fibre reinforcement with synthetic fibres in several polymer matrices has been reported to enhance the mechanical properties of the fibres (Atiqah *et al*. 2014; Yahaya *et al*. 2015). Atiqah *et al*. (2014) reported the development of a hybrid composite with chopped kenaf fibre and glass fibre reinforced using an unsaturated polyester matrix. They reported that a 15% composition (15/15/v/v) shows a higher value of flexural, impact, and tensile properties compared to other formulations. Yahaya *et al*. (2015) developed a hybrid composite of woven kenaf and Kevlar fabric using an epoxy matrix. These hybrid composites with Kevlar as the skin layers have better tensile and flexural properties, while the hybrid composite using kenaf as the skin layer has higher impact properties.

Bamboo fibre is another important natural fibre that exhibits comparable mechanical properties to conventional fibres (Mahdavi *et al*. 2012). Traditionally, bamboo has been used in various living facilities and tools, such as low-cost houses, bridges, and construction platforms (Bahari and Krause 2016), which owes its high strength to its weight. This property is due to its longitudinal alignment of fibres (Abdul Khalil *et al*. 2012). Bamboo fibres are known to be strong, stiff, containing an inferior microfibrillar angle, fibre axis, and a thicker cell wall, which has led to it being considered as ''nature's glass fibre' (Li *et al*. 1995).

Many researchers have reported comparative studies based on different properties of bamboo fibre-based biocomposites compared to glass fibre-based composites. Researchers stated that bamboo fibres can replace up to 25 wt.% of glass fibres without lowering the mechanical properties of glass fibre-based composites (Mandal *et al*. 2010). Zakihani *et al.* (2017) conducted extensive studies on the morphological, mechanical, and physical properties at three portions (bottom, middle, and top) of four bamboo species, namely, *Dendracalamus pendulus* (DP), *Dendrocalamus asper* (DA), *Gigantochloa levis*

(GL), and *Gigantochloa scortechinii* (GS). They concluded that the bottom portion of every species shows the highest aspect ratio and tensile properties (Zakikhani *et al*. 2017). On the other hand, researchers have carried out experimental and numerical studies on the fracture behavior of bamboo fibre reinforced epoxy composites. They have investigated the effect of NaOH treatment on fibre surface and have concluded that fibres treated with 6% NaOH show better ultimate tensile strength and interfacial shear strength. With this they suggested bamboo fibre reinforced epoxy composite having 25 mm fibre length (prior treated with 6% NaOH) has a greater fracture toughness (K_{IC}) value than the composites having smaller fibre length such as 10 and 20 mm (Khan *et al*. 2017).

Temperature plays an influential role in the thermal stability of natural fibre composites, in which it causes direct thermal expansion or contraction and affects the rate and volume of moisture absorption that leads to fibre swelling (Wang *et al*. 2005). Researchers reported that bamboo fibres degrade around 157 °C to 357 °C (Zakikhani *et al*. 2015) and kenaf fibres degrade around 247 °C to 455 °C (Khalil and Suraya 2011). Azwa and Yousiff (2013) reported that the addition of kenaf fibres into epoxy composites improves the thermal stability of the composites as well as its charring capability (Azwa and Yousif 2013). Shah *et al*. (2017) also compared the thermal stability of bamboo powder-filled epoxy composites to bamboo powder-filled glass/epoxy composites. The hybrid bamboo glass composites exhibit better thermal stability than non-hybrid bamboo composite (Shah *et al*. 2017).

Dynamic mechanical analysis (DMA) is a versatile technique that complements the information provided by the more traditional thermal analysis techniques, such as differential scanning calorimetry (DSC), thermogravimetric analysis (TGA), and thermomechanical analysis (TMA) (Saba *et al*. 2016). Dynamic mechanical analysis yields information about the mechanical properties of viscoelastic materials as a function of stress, time, temperature, and frequency or a combination of these parameters (Romanzini *et al*. 2013). The sample is subjected to periodic (sinusoidal) mechanical stress. This causes it to undergo deformation with the same period. The oscillating stress applied to the sample produces a corresponding oscillating strain. Unless the sample is perfectly elastic, the measured strain lags behind the applied stress. This is called the phase difference or the phase angle, δ. The ratio of peak stress (stress amplitude) to peak strain (strain amplitude) gives the complex modulus, M^*, which consists of an in-phase component M' (or storage modulus) and a 90° out-of-phase component, M'' (or loss modulus). It is also possible to calculate the mechanical loss factor or tan delta (damping). These dynamic parameters provide information about interfacial bonding between the reinforced fibres and the polymer matrix of the composite material (Saba *et al*. 2016), crosslinking density (Pistor *et al*. 2012), dynamic fragility (Ornaghi *et al*. 2012), dynamic/complex viscosity, storage/loss compliance, creep compliance/stress–relaxation modulus, and the non-Arrhenius variation of relating times with temperature (Qazvini and Mohammadi 2005).

This research work was intended to investigate the thermal and dynamic mechanical properties of the single system composite of kenaf (K) mat, bamboo mat, (B) and bamboo charcoal mat (BC) reinforced with an epoxy matrix. A neat epoxy will be prepared as the controlled sample. These results will then be further utilized to develop a kenaf/bamboo-based hybrid composite.

EXPERIMENTAL

Materials

Woven kenaf fibre mats were supplied by ZKK Sdn. Bhd (Selangor, Malaysia). Bamboo fibre mats and bamboo charcoal mats were procured from Shijiangzhuang Bi Yang Technology Co. Ltd. (Hebei, China). The fibre mats are shown in Fig. 1. The unit area density of kenaf, bamboo and bamboo charcoal mat are 600 g/m^2, 800 g/m^2, and 500 g/m^2 for each respectively. The liquid epoxy resin-DER-331 (a liquid reaction product of epichlorohydrin and bisphenol A) and a modified cycloaliphatic amine hardener (JOINTMINE 905-3s) were used in this study. Both the epoxy resin and commercial curing agent were procured from Tazdiq Engineering Sdn. Bhd. (Selangor, Malaysia).
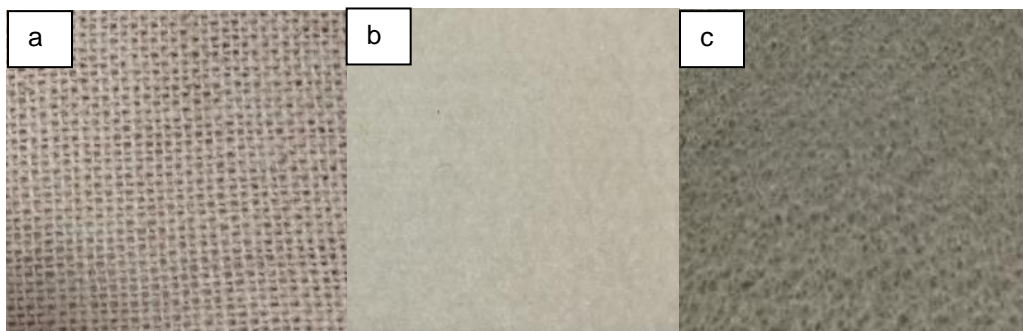

Fig. 1. (a) Kenaf mat, (b) bamboo mat, and (c) bamboo charcoal mat

Composites fabrication

In the fabrication process, the epoxy resin and hardener, using a ratio of 2:1, was uniformly mixed for 15 min. The hybrid composite was fabricated by stacking the fibre mat in a mould and impregnating it with an epoxy matrix using the hand lay-up method. The total fibre loaded into the matrix was controlled to be about 30% by weight. It was then impregnated with epoxy matrix in a mould with dimensions of 150 mm × 150 mm × 4 mm. The mould surface was treated with a release mould agent before the moulding process began, which prevents adhesion of the fibre composite to the mould and eases the removal of the fabricated part. Air bubbles were removed carefully with a roller. The mould was closed for curing at 110 °C for 10 min in a hot press under a constant pressure of 250 bar. The composites were then cooled in a cold press under a constant pressure of 250 bar for 5 min to prevent warpage of the hybrid composites. A neat epoxy matrix (unfilled) sample was also prepared. The prepared specimens are shown in Fig. 2.
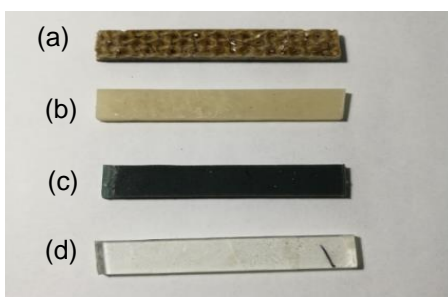

Fig. 2. (a) Kenaf/epoxy, (b) bamboo/epoxy, (c) bamboo charcoal/epoxy, and (d) neat epoxy

Methods

Thermogravimetric analysis

The thermal decomposition profile of the samples was studied using TGA according to ASTM E1131-03 (2003). A Mettler Toledo TGA/DSC 1 (Schwerzenbach, Switzerland) analyser was used for all of these measurements. Samples with 30 mg to 40 mg were placed in an alumina crucible and subjected to pyrolysis under a nitrogen atmosphere (50 mL/min). It was heated from 30 °C to 800 °C with a heating rate of 20 °C/min. The char formation during pyrolysis will be burned off on the subsequent heating segment from 800 °C to 1000 °C with a heating rate of 20 °C/min under oxygen atmosphere (50 mL/min), resulting in the char and ash content.

Dynamic mechanical analysis- Determination of the complex modulus (E*) at room temperature

The complex modulus (*E**) was studied using a Mettler Toledo DMA 1 (Schwerzenbach, Switzerland). The complex modulus of the samples was determined isothermally at 25 °C for 10 min to understand the flexural strength of the samples. The samples were measured in a 3-point bending mode (Fig. 3.) with a constant current force of 3 N applied on the specimen and displacement amplitude of 20 µm with a frequency of 1 Hz and set up for the test. The typical clamped sample dimension was 45 mm (length) × 10 mm (width) × 4 mm (thickness). The data collected on the first 4 min was discarded due to results not being stable yet as the sample adapted to the sinusoidal force. The complex modulus was determined from the average of the data points collected from 4 min onwards.
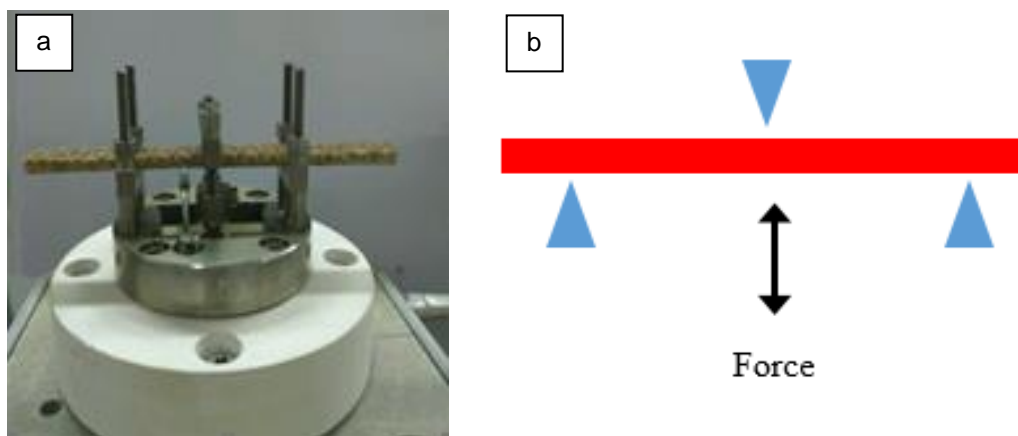

Fig. 3. (a) Specimen clamping on 3-point bending mode, (b) Illustration of 3-point bending mode in DMA

Determination of viscous elastic behaviour from 25 °C to 150 °C

The samples were measured using a single cantilever mode from 25 °C to 150 °C with heating rate of 5 °C/min (Fig. 4.). It was tested in a fixed frequency mode of 1 Hz and a displacement amplitude of 10 µm. The typical clamped sample dimensions were 10 mm (length) × 10 mm (width) × 4 mm (thickness). The glass transition temperature of the specimen was then determined according to ASTM E1640-99 (1999).
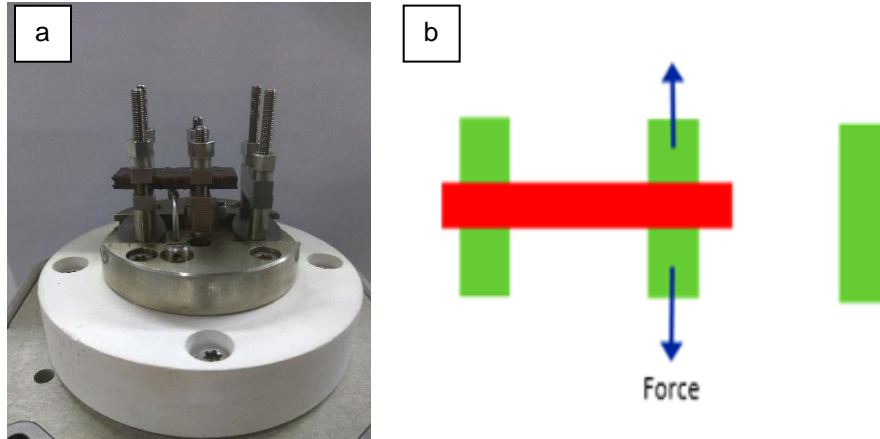
Fig. 4. (a) Specimen clamping on single cantilever mode, (b) Illustration of single cantilever mode in DMA

RESULTS AND DISCUSSION

Thermal Stability by TGA

The thermal decomposition curves of the epoxy composites as compared to the neat epoxy are shown in Fig. 5, and Table 1 presents the degradation temperature for 5 wt.%, 25 wt.%, 50 wt.%, and 75 wt.% weight loss of the specimens. All samples were subjected to pyrolysis decomposition under a nitrogen atmosphere up to 800 °C. All 3 composite specimens showed a remarkable initial weight loss with less than 10 wt.% from 30 °C up to approximately 250 °C, except for the neat epoxy sample. This can be related to the evaporation of moisture or volatile content that was contributed by the added fibre (Monteiro *et al.* 2012). The moisture contents for K/epoxy, B/epoxy, and BC/epoxy were 4.6 wt.%, 5.2 wt.%, and 3.7 wt.%, respectively. Due to the naturally hydrophilic characteristic of natural fibres, the total elimination of water was not possible even though the fibres had undergone oven-drying before the composite fabrication process. The major decomposition steps were observed at approximately 250 °C to 500 °C. The step down observed at 800 °C was due to the burn off of the char content when the atmosphere was being switched from nitrogen to oxygen, leaving the ash or residue content.

Table 1. Degradation Temperature for Epoxy Composites and Neat Epoxy

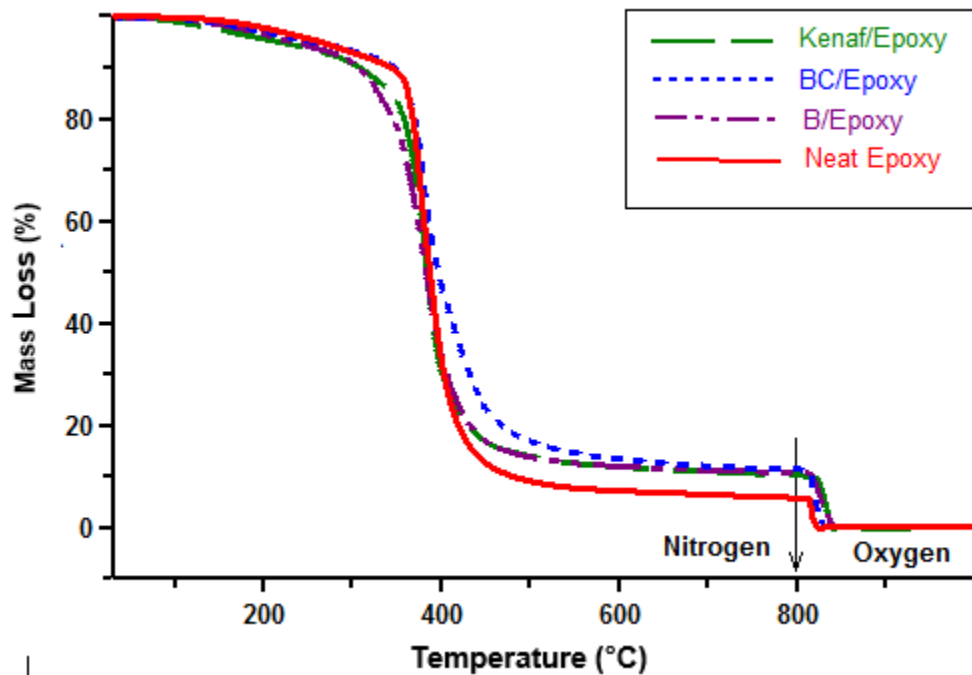| Specimens	T (°C) at 5 wt.%	T (°C) at 25 wt.%	T (°C) at 50 wt.%	T (°C) at 75 wt.%
K/Epoxy	220	366	385	413
B/Epoxy	233	356	383	417
BC/Epoxy	258	376	397	445
Neat Epoxy	266	373	387	410
Temperatures are given at 5%, 25%, 50%, and 75% of total weight loss				

Fig. 5. TGA curves of epoxy composites and neat epoxy

The major degradation event occurred in the temperature range of 250 °C to 500 °C. This was associated with the degradation of the reinforcing fibres and the polymer matrix. The DTG curves (Fig. 6), the first derivative curve of TGA, are often helpful to better define the temperature limits of a step change in mass. Mass changes occur when the sample loses materials (mass) in one of several different ways or reacts with the surrounding atmosphere. This produces steps in the TGA curve or peaks in the DTG curve. Thus, DTG curves are useful to help identify overlapping decomposition steps. The composite samples revealed 2 steps of degradation in this temperature range, except for plain epoxy, which only showed a single decomposition step. The DTG curves for K/Epoxy and B/Epoxy showed a small shoulder before the major decomposition peak, which indicated that there was weight loss. This is attributed to the decomposition of hemicellulose content, which tends to decompose at a lower temperature, 250 to 350 °C, then followed by the decomposition of the epoxy matrix and cellulosic structure that is more thermally stable compared to the hemicellulose due to its higher content of crystalline chains compared to the amorphous form (Ishak *et al*. 2012). Lignin is a complex 3-dimensional amorphous polymer with the presence of aromatic rings with various possible branches (Vanholme *et al.* 2010; Collard and Blin 2014), which makes it difficult to decompose. The decomposition mainly takes places over a wide temperature range from 160 °C to 800 °C with gradual decomposition (Yang *et al*. 2007). Finally, when the fibres are burnt under an inert atmosphere, the material will undergo pyrolysis, which forms the char residue as the leftover content. Lignin was reported to be the main constituent of residual char (Razali *et al*. 2015).
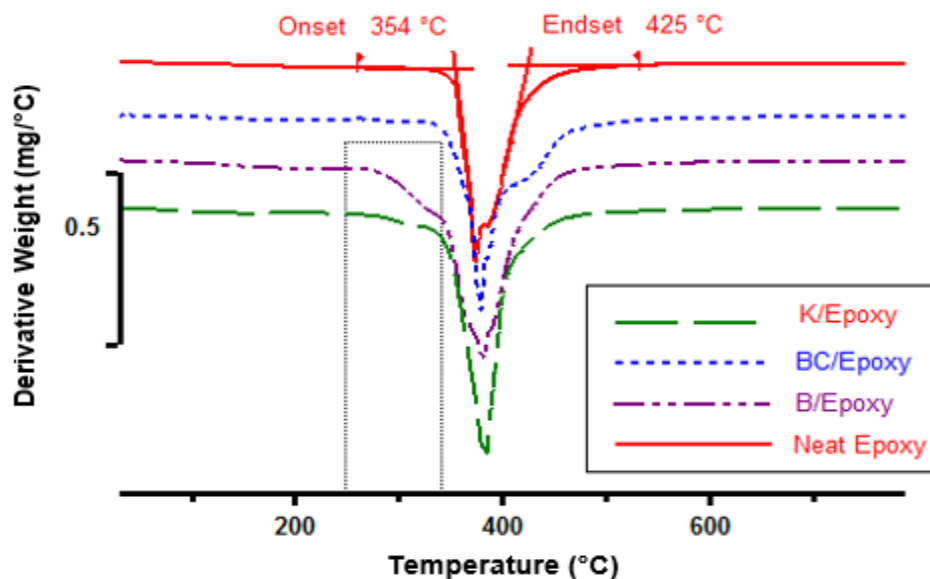
Fig. 6. DTG curves of epoxy composites and neat epoxy displaying in a relative scale

The thermal stability profile is often expressed in terms of parameters such as the initial and final decomposition temperature and char residue content. In this study, the initial decomposition temperature was investigated from the first decomposition DTG peak, either single or as a shoulder (Monteiro *et al*. 2012), by drawing the extrapolated onset point. This is the point of intersection of the baseline before a thermal effect and a tangent. The final decomposition temperature was also determined from the extrapolated endset of the DTG peak, the point of intersection of the tangent with the baseline after the effect. The char residue was determined at 800 °C from the TGA weight loss curve. An example of the curve interpretation on the onset and endset temperature with specimen neat epoxy is shown in Fig. 6, and all the results are tabulated in Table 2. The BC/Epoxy composite showed the highest thermal stability compared to K/Epoxy and B/Epoxy with the highest initial and final decomposition temperature at 348 °C and 463 °C. It also showed the highest char content at 11.5%. This could have been due to the impregnated charcoal content in the bamboo mat. Researchers have reported that the addition of bamboo charcoal in the composite enhances the thermal stability by increasing the char residue (Li *et al*. 2014). Char formation is important during a fire as it protects the core of the material and structural integrity (Azwa and Yousif 2013)
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Dynamic Mechanical Analysis

Determination of the complex modulus (E*) at room temperature

Figure 7 represents the complex modulus (*E**) obtained from the DMA by using a 3-point bending mode. The complex modulus, measured using 3-point bending, is the sum of storage modulus and loss modulus, which is approximately similar to the flexural modulus. The flexural modulus is an important mechanical property used to understand the stiffness of a material. The average complex modulus determined from 4 min onwards is shown in Table 3. The modulus of the neat epoxy was lowest among all samples. After reinforcement with the fibre mat, it greatly improved the stiffness of the

materials by increasing the modulus from approximately 2844 MPa to 3538 MPa. The K/Epoxy composite showed the highest modulus value with 3538 MPa followed by B/Epoxy and BC/Epoxy with a modulus value of 3397 MPa and 3230 MPa, respectively.

Table 2. Thermal Stability Properties of Epoxy Composites and Neat Epoxy

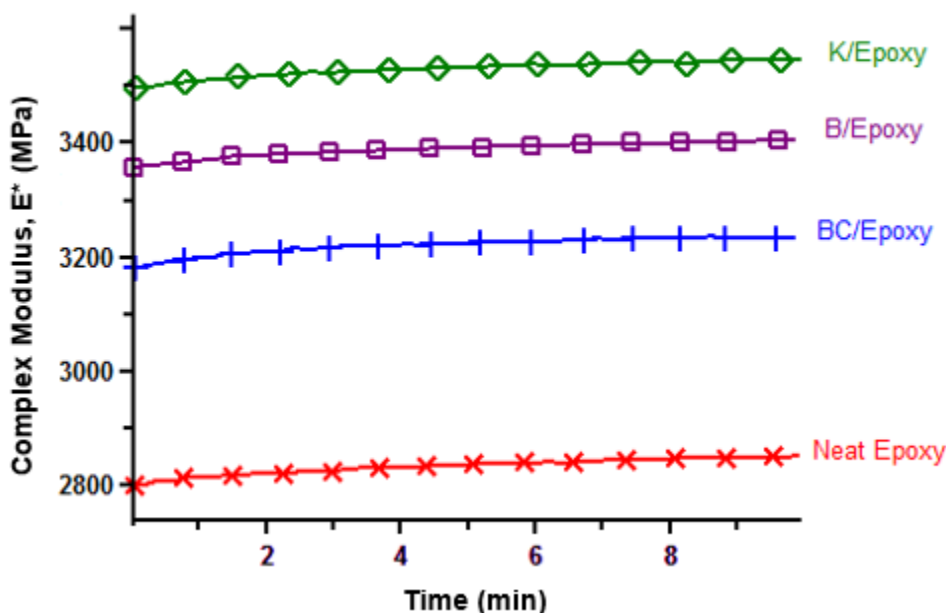| Specimens	Degradation Temperature (°C)		Char Residue (%)
	T_{IDT}[a]	T_{FDT}[b]	
K/Epoxy	282	408	10.4
B/Epoxy	282	427	10.9
BC/Epoxy	348	463	11.5
Neat Epoxy	354	425	5.98
[a] IDT = Initial decomposition temperature [b] FDT = Final decomposition temperature			

Fig. 7. Complex modulus determined at room temperature for epoxy composites and neat epoxy with 3-point bending mode

Determination of the viscous elastic behaviour from 25 °C to 150 °C

Figure 8 shows the viscous elastic behaviour of the specimens when subjected to the effect of temperatures with a fixed frequency of 1 Hz, measured using a single cantilever mode. The DMA curves were expressed in terms of storage modulus, *E'* (upper curve), loss modulus, *E''* (middle curve), and tan delta (lower curve) in Fig. 8. The storage modulus (*E'*) represents the stiffness of a material and its proportionality to the mechanical energy stored during a stress period (Saba *et al*. 2016). In other words, the higher the storage modulus, the stiffer the material. With this, the authors observed similar trends as in previous experiments in determining the complex modulus, where K/Epoxy showed the highest modulus at 25 °C followed by B/Epoxy and BC/Epoxy.

The neat epoxy showed the lowest modulus value. However, the modulus obtained from the single cantilever measurement mode was much lower compared to a 3-point bending mode (Table 3). This was due to the clamping effect in a single cantilever where the specimen was being clamped on both ends, as shown in Fig. 4 while in 3-point bending mode, the clamping effect was neglected as the specimen was not being clamped (Fig. 3).
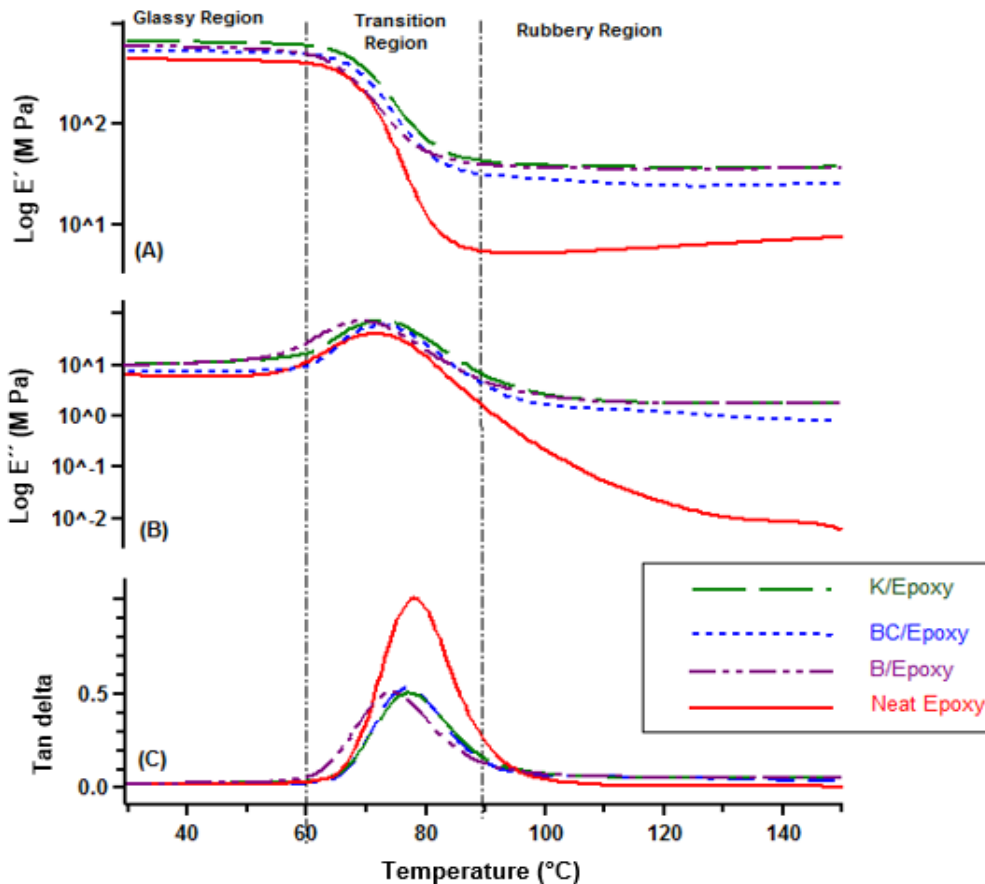

Fig. 8. Storage modulus (A), loss modulus (B), and tan delta (C) curve of epoxy composites and neat epoxy

The loss modulus, *E''*, is a viscous response of the materials. It describes the energy dissipated (lost as heat) during a deformation cycle. A high loss modulus indicates viscous behaviour and hence marked damping properties. When the timescale of molecular motion coincides with that of mechanical deformation, each oscillation is converted into the maximum-possible internal friction and non-elastic deformation. The loss modulus, which is a measure of this dissipated energy, also reaches a maximum and is denoted as the glass transition (T_g) temperature of the system (Kumar *et al.* 2011). The highest loss modulus was obtained from the K/Epoxy and the lowest loss modulus was obtained from the neat epoxy due to less heat dissipation from the epoxy. With the addition of fibre in the system, the frictional force between the molecular movement in the polymer matrix and fibres increases and thus increases the heat dissipated to the interface (Devi *et al.* 2009).

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Table 3. Complex Modulus and Storage Modulus of Epoxy Composites and Neat Epoxy Measured by 3-Point Bending and Single Cantilever Mode

Specimens	Complex Modulus, E^* at 25 °C by 3-point bending mode (MPa)	Storage Modulus, E' at 25 °C by single cantilever mode (MPa)	Storage Modulus, E' at 100 °C by single cantilever mode (MPa)
K/Epoxy	3538	666	38.9
B/Epoxy	3397	601	36.8
BC/Epoxy	3230	536	28.1
Neat Epoxy	2844	445	5.2

The loss or damping factor (Tan delta) of a system corresponds to the ratio of elastic to viscous properties. A high value therefore indicates a high degree of energy dissipation and a high degree of non-elastic deformation. As shown in the lower curve in Fig. 8, the damping factor increased as the temperature increased and reached its maximum in the transition region and gradually decreased in the rubbery region. It was observed that the damping factor was highest at the T_g temperature for all samples due to the chain segments' ability to freely move in this region. The neat epoxy showed the highest tan delta peak value (0.99); this indicated the system had a greater degree of molecular mobility (Jacob *et al.* 2006). With the incorporation of fibres, the molecular mobility decreased due to the interlocking occurring between fibres and the polymer matrix. Thus, the tan delta peak was lowered, as shown in Table 4.

Table 4. Onset of E' (T_g), E''_{max} (T_g), Tan $δ_{max}$ (T_g), and Peak Height of Tan $δ$ Curve of Epoxy Composites and Neat Epoxy

Specimens	Glass Transition (T_g) (°C)			Peak Height of Tan $δ$ Curve
	Onset of E'	Peak of E''	Peak of Tan $δ$	
K/Epoxy	67.60	73.08	77.42	0.49
B/Epoxy	64.24	69.42	74.33	0.50
BC/Epoxy	67.70	72.67	77.08	0.52
Neat Epoxy	68.86	71.83	78.00	0.99

According to ASTM E1640-99 (1999), the assignment of the glass transition temperature by dynamic mechanical analysis, the glass transition temperature can be denoted as the extrapolated onset to the sigmoidal change in the storage modulus observed going from the hard, brittle region to the soft, rubbery region of the materials under testing parameters. Alternatively, the T_g temperature can also be denoted by the peak of the loss modulus and tan delta curve. A storage or loss modulus may be displayed in a linear or logarithmic scale in which the reported T_g can differ depending upon the scale chosen. Thus, in this study the reported T_g was based on a logarithmic scale in both the storage and loss curve. All of the data are tabulated in Table 4.

The glass transition of all 4 specimens fell in the temperature range of 60 °C to 90

°C (Fig. 8, transition region). Due to the crosslinking network in the cured epoxy system, the composites behaved solid and rigid below the transition region and became soft and rubbery when the temperature passed through the transition region. Therefore, it was observed that the samples went through a drastic decrease in the storage modulus when passing through the transition region. As shown in Table 3, the addition of fibre improved the mechanical properties of the composite in the glassy region as well as in the rubbery region. The K/Epoxy still showed the highest storage modulus in the rubbery region, followed by B/Epoxy and BC/Epoxy with a storage modulus of 38.9 MPa, 36.8 MPa, and 28.1 MPa, respectively. The storage modulus of the neat epoxy decreased drastically from approximately 445 MPa to 5 MPa when the temperature passed through the transition region.

Based on the results obtained in this study, the BC/Epoxy composites showed the highest thermal stability performance while the K/Epoxy composite showed better dynamic mechanical properties. Thus, it was possible to combine both the bamboo charcoal and kenaf mat into developing a new hybrid composite system, which may enhance the final performance of the composites compared to each individual systems\.

CONCLUSIONS

1. The composites reinforced with kenaf or bamboo fibre (single system fibre reinforced composites) showed enhanced thermal stability and dynamic mechanical properties compared to the neat epoxy.

2. The following conclusion made from this work was that the bamboo charcoal/epoxy composite showed the highest thermal stability with the highest initial and final decomposition temperature but slightly lower mechanical strength, compared to the other epoxy composites in this study.

3. The kenaf/epoxy composite had the highest mechanical strength performance with the highest complex modulus strength but with a lower thermal stability performance compared to the other epoxy composites in this study.

4. Lastly, the fabricated composites were suitable for working temperatures of no more than 60 °C due to the glass transition temperature of the composites that fell in the temperature range of 60 °C to 90 °C. Below the glass transition temperature, the samples behaved in a solid and rigid manner and turned into a soft and rubbery phase as it passed through the glass transition temperature.

ACKNOWLEDGMENTS

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