Thermal Analysis of Bamboo Fibre and Its Composites

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Thermogravimetric analysis and differential scanning calorimetry were used to study the thermal degradation and thermal stability of bamboo powder and its composites (EP-BFC) in a nitrogen atmosphere. The thermal stability of EP-BFC decreased as the bamboo filler-loading increased. Compared with epoxy, bamboo powder had a lower thermal stability, which reduced the thermal stability for the higher filler-loading composites. The addition of glass fibre to the EP-BFC improved the thermal stability of the new hybrid composites. Both the hybrid and non-hybrid composites exhibited similar thermal-induced degradation profiles that had only one mass loss step. However, a noticeable difference between the percentage value of the degradation between both the hybrid and non-hybrid composites showed that the EP/G-BFC hybrids were more thermally stable than the non-hybrid EP-BFC. Different materials experienced different activities, which were clearly shown from the DSC analysis. Bamboo fibre and non-fully cured epoxy exhibit exothermic peaks, while fully cured epoxy exhibits an endothermic peak.

Keywords: Bamboo; Thermal degradation; Filler; Hybrid

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INTRODUCTION

A composite material is a combination of at least two different constituents to produce a new engineering material with improved properties. The two basic materials combined in composites are the matrix and the reinforcements. The matrix can be classified into different types, either polymer, metal, and/or ceramic. Compared with metal and ceramic materials, polymer matrices have the lowest strength and stiffness (Campbell 2010). However, polymer composites are in high demand in many fields, including structural applications (Bakis *et al.* 2002; Breuer and Sundararaj 2004; Holbery and Houston 2006; Suhaily *et al.* 2013).

Polymer composites are made of thermoplastics or thermosets as the matrix materials. Examples of commonly used thermoplastics are polypropylene (PP), polyvinyl chloride (PVC), poly lactic acid (PLA), polystyrene (PS), and high density polyethylene (HDPE). Some examples of common thermosets are epoxy, polyester, and elastomer (natural rubber) (Xanthos 2010; Huda *et al.* 2012; Davies Moulding 2014).

Epoxy is the predominant material for coatings, adhesives, corrosion protectors, and encapsulants for electronics. Epoxy-based coatings in automotive applications prevent rust and corrosion on vehicle bodies and metal parts (Bilyeu *et al.* 2000; Epoxy 2010). In fibre-reinforced composites, epoxies are the favourable choice for matrices compared with

other polymers because they possess great properties such as excellent adhesion to many substrates, high strength, chemical resistance, fatigue resistance, and simple processability (Bilyeu *et al.* 2000). There have been many previous studies on epoxy composites using synthetic man-made fibres, natural fibres, or a combination of both. These studies have demonstrated the adaptability of epoxies with many different types of reinforcement fibres. The comparable properties of epoxy-based composites have led to their use in wider applications today (Jawaid *et al.* 2010; Ku *et al.* 2011; Phong *et al.* 2013; Caprino *et al.* 2015; Saba *et al.* 2015). Glass and carbon fibre-reinforced epoxies are now the major components in the production of boats, automobiles, aircrafts, and sport equipment (Soutis 2005; Suhaily *et al.* 2013).

There are two types of reinforcements that can be added to the matrix to form composites. Long-continuous fibres, such as woven fibres, can be embedded in regular geometric arrangements in the polymer matrix and generate excellent properties in the composites compared with short-discontinuous fibres. Fibres less than 3 cm in size are called short-discontinuous fibres; they take the form of flakes and powder. These types of fibres are commonly called filler in the matrix because they do not provide noticeable reinforcement to the composites. Therefore, composites made with fillers are classified as low-performance composites (Xanthos 2010). Recently, the use of fillers in composites has been widely applied, as certain types of fillers enhance composite properties. Compared with inorganic fillers, such as carbon black and titanium dioxide, natural fillers have advantages of a lighter weight and cost reduction (Jana and Prieto 2002; Mahesha *et al.* 2014).

Different loadings of rice husk powder (RHP) filled polypropylene composites were fabricated through a compression molding technique, and their mechanical as well as impact properties were studied. The inclusion of RHP in PP matrix had developed different material's behaviour of PP from ductile to a quasi-brittle behaviour. In terms of Young's modulus and flexural modulus, both properties increased as the filler loading increased (Premalal *et al.* 2002). In a different study, the rule of mixture (ROM) model was modified to study the tensile strength of short natural fibre reinforced thermoplastics (NFRT). To justify the modified model, several types of natural fibres such as 20-mesh hardwood flour, 40-mesh hardwood flour, hemp, and rice hulls had been used as filler in thermoplastic composites. The inclusion of natural fibre as filler had increased the tensile strength of the thermoplastic materials. In terms of ROM model justification, the experimental results showed good agreement with the tensile strength prediction from the modified ROM model (Facca *et al.* 2007).

The bamboo tree, one of the fastest growing plants on earth, is found in Asia, America, Africa, and several European countries. In Malaysia, there are several famous bamboo species that grow well either in Peninsular Malaysia, Sabah, or Sarawak (FDPM 2013). Bamboo is traditionally used in household appliances and structural applications, such as long houses and bridges. An abundant source of bamboo in Malaysia gives big opportunities for its applications in daily lives. However, the lack of technology available for processing raw fresh bamboo into ready-to-use bamboo fibres slows down its development and applications in Malaysia, especially relating to the fabrication of composites (Suhaily *et al.* 2013). Bamboo can duplicate several of the processing methods of wood material, so the bamboo powder can be produced and used as fillers in polymer composites. Similar to other organic fillers made from agricultural industrial residue, bamboo powder filler has been studied for its applications in wider fields.

Different ratios of bamboo powder were compounded with polyester and fabricated using a compression molding technique. From tensile and flexural standard testing, the mechanical properties of fabricated materials were identified. Composites with 25 wt% bamboo powder were found to have the highest strength, while the impact test showed the highest value at 20 wt% of bamboo powder (Leha *et al.* 2014). Different types of conventional filler, aluminium oxide, silicon carbide, red mud, and copper slag had been used to incorporate in bamboo fibre reinforced epoxy composites to observe their effect on the mechanical properties of composites. The tensile strength of the mixed conventional filler with bamboo were found to be lower than the pure bamboo epoxy composites in most cases of different types of fillers. Compared to neat epoxy, the inclusion of bamboo fibres increased the tensile and flexural strength of the composites (Yu *et al.* 2012) Bamboo is well-known for its excellent properties and wide use as the main material in structural applications. There have been many recent studies on its development (Tanaka *et al.* 2007; Samal *et al.* 2009; Sen and Reddy 2011).

To study the thermal properties of natural fibres used as filler or reinforcement in composites, one of the commonly used analytical techniques is thermogravimetric analysis (TGA) followed by differential scanning calorimetry (DSC). In TGA, the mass of a substance is monitored as a function of temperature or time in a controlled atmosphere (Kim *et al.* 2004, 2005; Yao *et al.* 2008; Monteiro *et al.* 2012). In some situations there will be no mass change over time or changes in temperature. Thus, DSC is used to analyze the kinetics of the reactions (Haines 2002). DSC studies the difference of heat required to increase the temperature of a sample and reference as a function of temperature. The temperature range at which a material changes its state from hard, rigid or glassy state to a more pliable, compliant or rubbery state is known as glass transition temperature (T_g) (Sichina 2000; AME 2016).

Maleic anhydride polyethylene (MAPE)-treated jute/HDPE composites have better thermal properties than composites with untreated jute fibres (Mohanty *et al.* 2006). In addition, different bamboo species have different thermal stabilities, and the thermal stability varies along the bottom, middle, and top of the bamboo culm (Zakikhani *et al.* 2016). Compared to pure epoxy, the incorporation of water bamboo husk in epoxy composites obviously raised the char yield of the samples, which indicates the improvement of their thermal resistance (Shih 2007).

This study evaluated the thermal stability of bamboo powder-filled epoxy composites (EP-BFC) with different filler loading contents and determined the thermal stability of the bamboo powder (BP) itself, which was used to fabricate the BPFEC. The thermal evaluation was conducted through TGA under an inert atmosphere (nitrogen gas). The effects of BP content on the degradation temperature, mass loss rate, and residual mass were considered.

EXPERIMENTAL

Epoxy

The epoxy resin analyzed in this study was EpoxAmite 100, which was used together with the 103 slow hardener; both were purchased from Mecha Solve Engineering (Selangor, Malaysia). The epoxy and hardener were mixed with a ratio of 3:1 according to the datasheet provided by the supplier. Throughout the study, all the composites were fabricated with fixed dimensions of 300 x 300 x 5 mm^3 to easily differentiate the fibre

loading. The pure epoxy was also fabricated with the same dimensions to maintain the amount of epoxy in all samples, which was 495 g.

Bamboo Powder (BP)

The bamboo powder was manually processed. Fresh 4-year-old Buluh Aur (*Bambusa vulgaris*) bamboo culms were collected from Raub, Pahang in Peninsular Malaysia. To prevent moisture loss during transportation, Tree Wound liquid (ZKK Sdn. Bhd, Kuala Lumpur, Malaysia), a pruning sealer, was applied to the cross-section of every bamboo culm. In the laboratory, the bamboo culms were split into smaller strips approximately 1 m in length and 50 mm in width before passing through a chipper machine. Small bamboo chips from the chipper machine were oven-dried for 24 h at 80 °C. To obtain the bamboo powder, the dried bamboo chips were crushed in a flaker machine and sieved according to size. The bamboo powder selected for this study ranged from 500 µm to 1 mm in size.

Bamboo Powder Filled Epoxy Composites (EP-BFC)

Bamboo powder that ranged 500 µm to 1 mm in size was chosen as the composite filler. There were three composites fabricated with different weight percentages of bamboo filler loading (wt.%). The amount of epoxy in all three composites was similar with the previous pure epoxy plate, 495 g. The epoxy was mixed with 10% (49.5 g), 20% (99 g), or 30% (148.5 g) of bamboo powder before being poured into a square aluminium mould and cured at room temperature for 24 h. The mixture was stirred well using a wooden stick for at least 5 minutes (depends on the amount of powder) to ensure that the powder was evenly distributed in the epoxy before being poured into the mould. After demoulding the cured plates, they were placed in the oven at 80 °C for 2 h for the post-curing process.

Bamboo Powder Filled Glass/Epoxy Composites (EP/G-BFC)

The bamboo powder and epoxy used for the fabrication of the EP/G-BFC were the same types used for the fabrication of the EP-BFC. The glass fibre was the type of E600. One layer of woven glass fibre was embedded at the top and bottom part of the EP-BFC, producing a sandwich type of composite structure. One thin layer of pure epoxy was poured into the mould before laying one layer of woven glass fibre count as the second layer. The mixture of epoxy with 30% bamboo powder was then poured on top of the glass fibre, followed by another layer of glass fibre, and finished by one thin layer of pure epoxy. The layering pattern is shown in Fig. 1.

After curing at room temperature, the EP/G-BFC was placed in the oven for the post-curing process under similar conditions as the EP-BFC. Both layers of glass fibres were counted as additional reinforcement, which was embedded at the outer layer of the original EP-BFC to produce hybrid composites. As the inclusion of glass fibres did not modify the amount of epoxy and bamboo fibre loading, which was similar with the 30% EP-BFC, the weight of glass fibres was neglected.

The amount of epoxy used to wet both layers of glass fibres at the bottom and top part of the composites was 30 g each. The amount of 30 g was selected after series of calculations and through observations of several trial and error fabrications in order to produce smooth and even surface of hybrid composites with the least additional epoxy to the original EP-BFC.

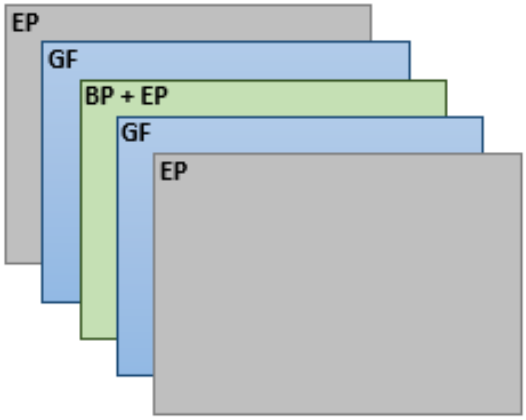

Fig. 1. Layering pattern of bamboo powder filled glass/epoxy composites

Determination of Thermal Properties

The thermal stability and thermal changes of all seven samples, which weighed 10 mg to 15 mg, were analysed using TGA and DSC. Both analyses were carried out on a Mettler-Toledo TGA/SDTA851^e device (Columbus, OH, USA) in a flowing nitrogen (N_2) atmosphere at a flow rate of 50 mL/min to avoid unwanted oxidation (Kim *et al.* 2004). The temperature range was 25 °C to 900 °C at a heating rate of 10 °C/min.

RESULTS AND DISCUSSION

Thermal Degradation by Thermogravimetric Analysis (TGA)

Figure 2 shows the TGA profiles of the epoxy composites filled with different loadings of BF at a heating rate of 10 °C/min from 0 °C to 900 °C. The thermal degradation profile of all composites exhibited the same pattern even though they had different percentages of filler loading. The profiles showed a single mass loss step, which obviously occurred at approximately 250 °C for all of the BF loadings.

Figure 3 is the image enlargement of the mass loss step from the profiles shown in Fig. 2. As the percentage of bamboo filler loading increased, the composites burned faster, as shown in Fig. 3. There was a noticeable difference between the neat epoxy resin, which started to lose mass at 300 °C, and the composites; the composites filled with the highest percentage of 30% BF loading started to lose mass at a lower temperature of 250 °C. At 440 °C, the mass loss step for all of the loadings ended, and the composites started to burn and turn into ash.

Figure 2 shows the whole temperature range after the mass loss step; a higher loading produced a higher ash content. These results confirmed that the thermal stability of the composites decreased as the BF filler loading increased. The results are in good agreement with a previous study by Kim *et al.* (2004), which studied the TGA of rice-husk flour-filled thermoplastic polymer composites. Both of the studies used natural fibres as fillers but with different types of matrices, which were the thermoplastic and thermoset. However, the difference in the types of matrices does not affect the pattern of the thermal degradation profiles of both types of composites.

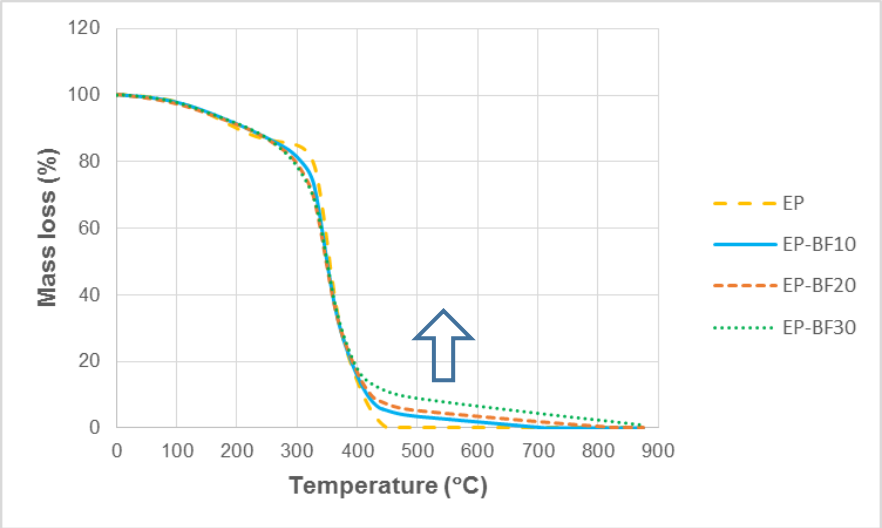

Fig. 2. Thermal analysis profiles of EP-BF composites with different filler loading. The blue arrow indicates the increasing ash content.

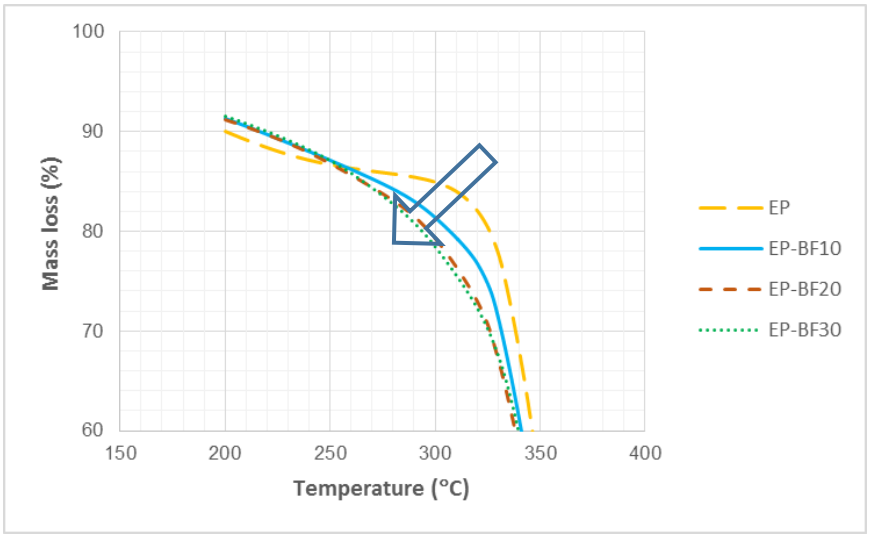

Fig. 3. Image enlargement of the mass loss step from the profiles in Fig. 2. The blue arrow shows higher loading burns at lower temperature.

To further analyze the lower stability of the higher bamboo filler loading composites, TGA evaluations of pure epoxy and pure bamboo fiber were conducted (Fig. 4). The bamboo fibre had a lower thermal stability because it degraded at a lower temperature of approximately 220 °C compared with the pure epoxy, which started to degrade at 320 °C. The degradation of the holocellulose and lignin in the bamboo fibre, and any other natural fibre, is the major source of its thermal degradation, while the degradation of epoxy occurs by random chain scission and radical chain mechanism (Kim *et al.* 2004). Therefore, the lower thermal stability of bamboo fibres reduced the thermal stability of higher bamboo filler loading composites. This is the expected consequence for the higher BF filler loading composites. A higher percentage of bamboo filler results in decreased epoxy, which is the more stable material. As shown in Fig. 4, the mass loss step for the BF and epoxy ended at approximately 300 °C and 440 °C, respectively. After the mass loss step, the ash content for BF was seen to be higher compared to the epoxy resin.

The composites containing a higher percentage of bamboo filler produced proportionally higher ash content as the materials burn out, as discussed previously in Fig. 2.

As reported in other studies (Jarukumjorn and Suppakarn 2009; Sanjay *et al*. 2015), the mechanical properties of natural fibre-reinforced polymer composites improves as the composites are additionally reinforced with other synthetic fibres, such as glass fibre mats. This process makes the composite into a hybrid system. To study the effect of hybridization with glass fibre on the thermal properties of EP-BF composites, TGA on the individual glass fibre was first conducted. The TGA profile of the neat glass fibre indicated very stable thermal degradation, as shown in Fig. 4.

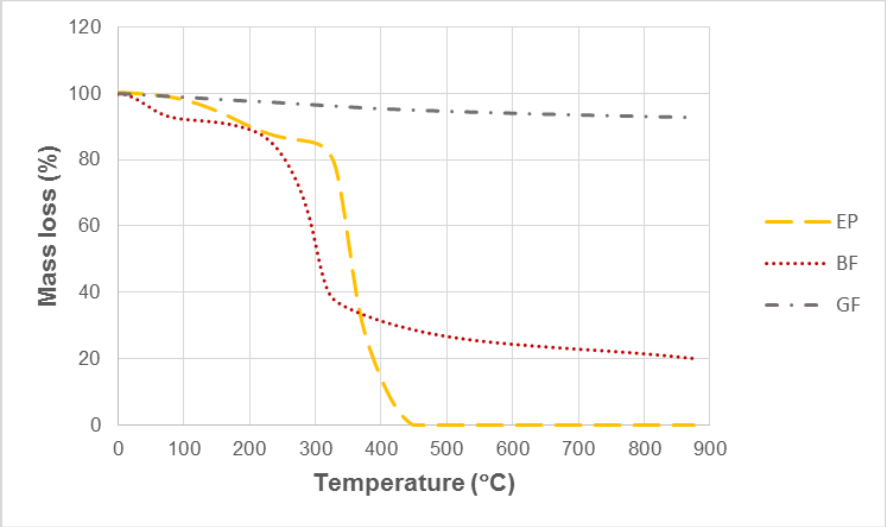

Fig. 4. Thermal analysis profiles of EP, BF, and GF

Because the glass fibre had high thermal stability, it was expected that a hybrid with the 30% bamboo powder filled epoxy composites (EP-BF30) would have enhanced thermal properties. Figure 5 shows the TGA profiles of the hybrid EP/G-BF30 and the EP-BF30 composite.

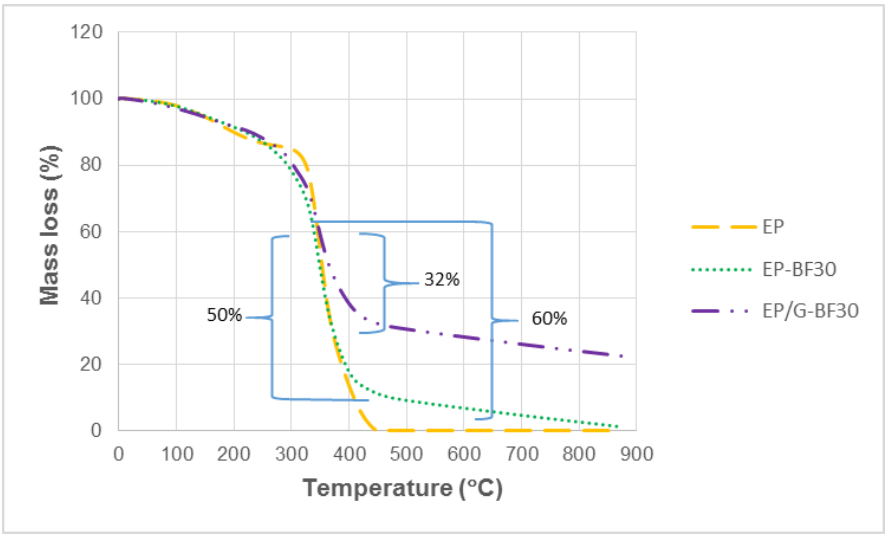

Fig. 5. Thermal analysis profiles of EP, EP-BF30, and EP/G-BF30

Both the hybrid and non-hybrid composites exhibited only one step of mass loss due to thermal degradation, but a noticeable difference in the percentage of mass loss for both composites was observed. The EP-BF30 composites were degraded by approximately 50%, which was almost twice as high the 32% mass loss from the 30% bamboo powder filled glass/epoxy composite (EP/G-BF30). The higher percentage of degradation for the EP-BF30 composites indicated their lower thermal stability compared with the hybrid composite. Compared with the neat epoxy resin, the hybrid EP/G-BF30 composites was completely stable. The neat epoxy degraded by approximately 60%, which is twice as high as the thermal-induced weight loss of the hybrid composites (32%).

Differential Scanning Calorimetry (DSC) Analysis

The comparison made through DSC analysis was slightly different compared to TGA. The data were analysed in a way to clearly observe the phase transition of different materials, which the process could be either exothermic and/or endothermic. Figure 6 shows the DSC thermograms of pure bamboo fibre and neat epoxy. From the raw data, it can be seen that the peak temperature for both samples occurred at the temperature range of 300 °C to 380 °C, which is marked in the blue circle.

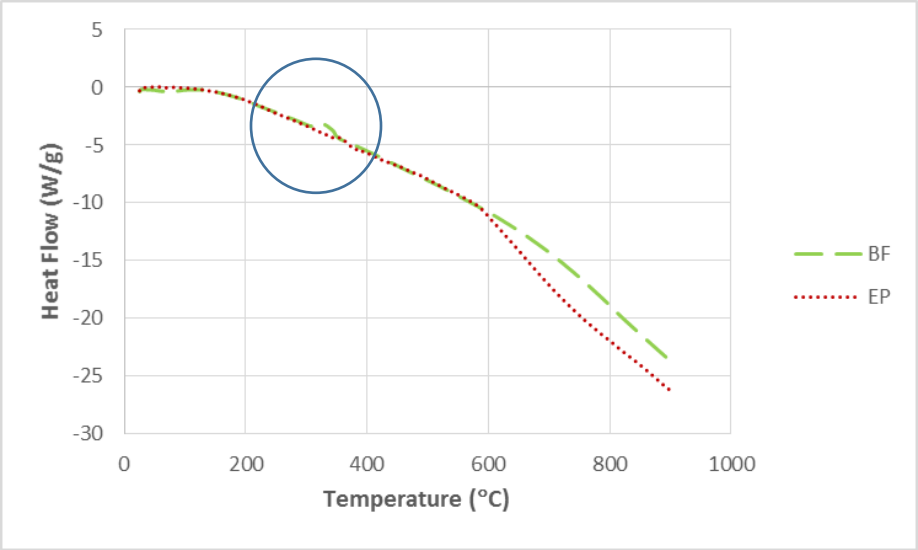

Fig. 6. DSC thermograms of Bamboo Fibre (BF) and neat epoxy (EP). The blue circle locates the peak temperature for both BF and EP.

Figure 7 illustrates the peak temperature of pure bamboo fibre from the species of *Bambusa vulgaris.* Bamboo fibres marked an exothermic peak at a temperature of 327.38 °C. From previous studies (Zakikhani *et al.* 2016), bamboo exhibits different thermal degradation temperatures among species. All bamboo species exhibited two or three continuous peaks. However, certain species such as *Gigantochloa levis* experienced exothermic peaks at a temperature higher than 234 °C, which supports the findings in the current study. Basically, peak temperature for neat epoxy varies depends on the curing stage of the epoxy. From several analysis reported previously, fully cured epoxy will not exhibit an exothermic peak (Nakamura 1991; Sichina 2000). The DSC thermograms in Fig. 7, however, shows an exothermic peak for the neat epoxy, which indicates that the neat epoxy was not fully cured.

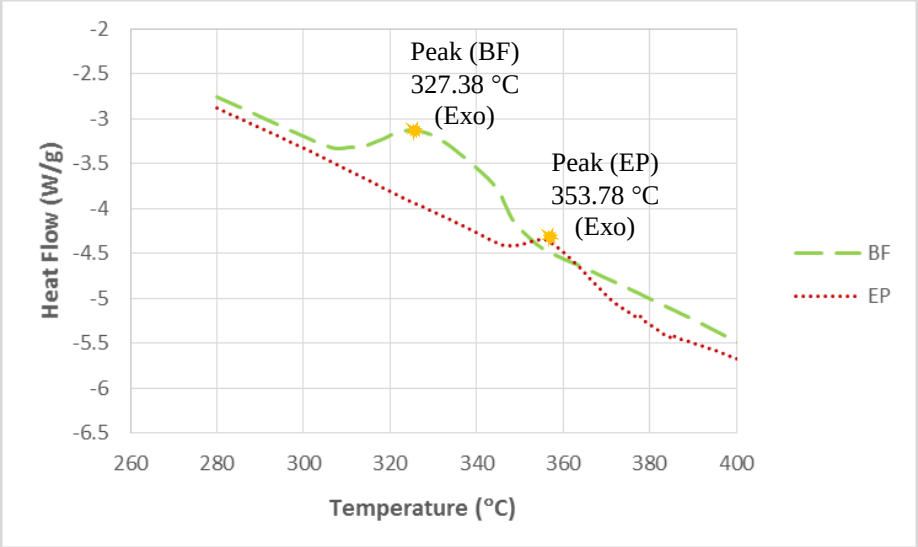

Fig. 7. Image enlargement of the peak temperature for bamboo fibre and neat epoxy from DSC thermograms in Fig. 6.

Figure 8 shows the thermograms of non-hybrid bamboo composites with different filler loading. Both samples experienced exothermic and endothermic peaks, which occurred at the temperature range of 300 °C to 400 °C marked in in the blue circle.

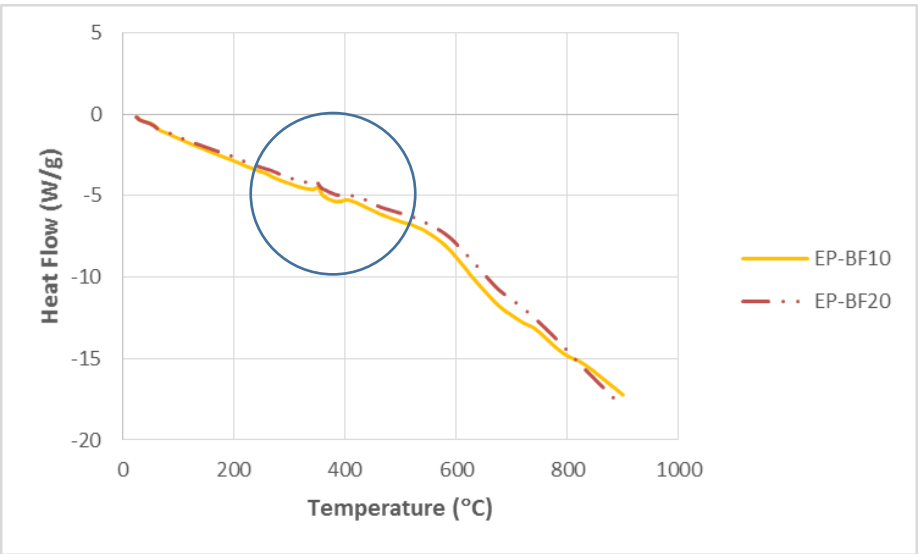

Fig. 8. DSC thermograms of bamboo composites with 10% and 20% filler loading (EP-BF10; EP-BF20). The blue circle locates the peak temperature for both EP-BF10 and EP-BF20.

Figure 9 illustrates the peak temperature of bamboo composites with 10% and 20% bamboo filler loading. Both profiles showed similar trends of thermal degradation, which exhibited an exothermic peak followed by an endothermic peak. In DSC, there was no relationship between filler loading and peak temperature, as the temperature only differs among types of material. This study was conducted for two different filler loadings to ensure both samples showed similar trends and the peak temperature lies at the same range. In Fig. 9 the endothermic peak in both profiles indicate the (T_g) of the epoxy matrix.

Compared to the neat epoxy profile in Fig. 7, which exhibit exothermic peak, the endothermic peak of epoxy matrix in bamboo composites signified a fully cured epoxy. The difference between the DSC thermograms of neat epoxy and epoxy matrix in bamboo composites was due to the post curing process during the sample preparation stage, where the neat epoxy was not placed in the oven for post curing compared with the bamboo composites.

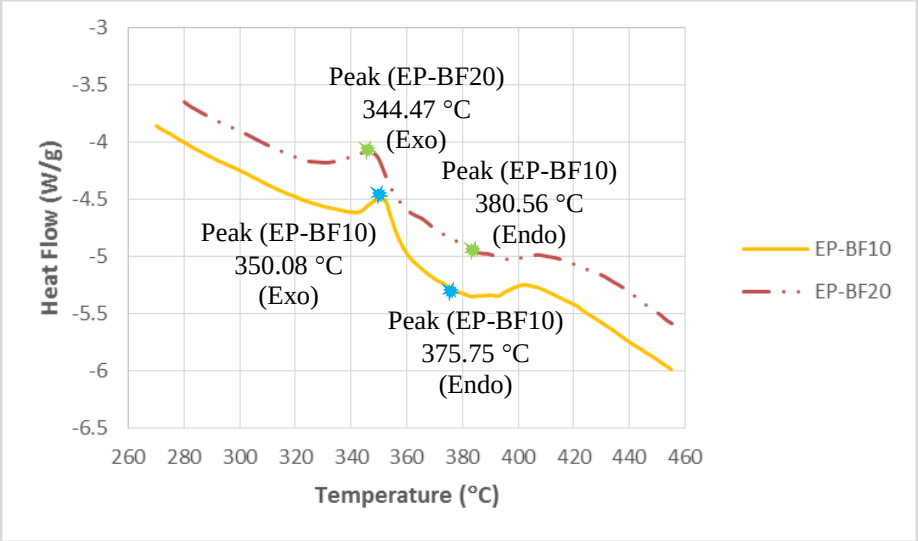

Fig. 9. Image enlargement of the peak temperature for EP-BF10 and EP-BF20 from DSC thermograms in Fig. 8.

CONCLUSIONS

1. The thermal stability of bamboo powder-filled epoxy polymer composites decreased as the BF filler-loading was increased.
2. The bamboo fibre from the species of *Bambusa vulgaris* showed low thermal stability and started to degrade at approximately 220 °C.
3. The hybrid bamboo glass composites exhibited better thermal stability than non-hybrid bamboo composites.
4. Non-hybrid bamboo composites exhibited both endothermic and exothermic transitions, which corresponded to epoxy and bamboo fibre degradation, respectively.
5. The level of curing of epoxy polymer affected the flow of heat during the thermal degradation. Fully cured epoxy experienced an endothermic transition, while non-fully cured epoxy experienced an exothermic process.

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