

Thermal Properties of Kenaf Fiber Reinforced Polyamide 6 Composites by Melt Processing

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ABSTRACT

In recent years, there has been much effort to find cost-effective ways to replace petroleum-based commodity plastics with biodegradable polymers with comparable thermal characteristics. The 5 wt.%, 10 wt.%, and 15 wt.% kenaf fiber were melted, and blended with polyamide-6 via a Brabender mixer, followed by compression molding. To evaluate the thermal properties of composites, thermogravimetric analysis (TGA), differential scanning calorimetry (DSC), and dynamic thermomechanical analysis (DMA) were conducted. According to the TGA results, increased kenaf fiber contents decreased the composite's thermal stability. Neat PA6 matrix decomposed rapidly at 425°C, which was comparatively higher than PA6 composites. From the DSC analysis, the addition of natural fibers resulted in quantified changes in the glass transition temperature (T_g), melting temperature (T_m), and crystallization temperature (T_c) of the PA6 composites. According to the DMA, the storage modulus of neat PA6 was 1177 MPa and decreased to 1076 MPa for 5 wt% of kenaf fiber in PA6 composite. The Kenaf fiber/polyamide 6 composites appeared to have lower thermal stability than neat PA6. This study demonstrated that the kenaf fiber/polyamide 6 composites were successfully prepared, and a detailed thermal analysis was conducted. Improving the KF/PA6 composites can be further studied to increase thermal stability.

Keywords: Composites, kenaf fiber, natural fiber, polyamide 6, polymer, thermal properties

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INTRODUCTION

As the world strives to become more 'green,' the importance of global involvement is desperately needed to protect the environment and make it more economical to live in (Devnani & Sinha, 2019; Sanjay et al., 2018). The need for biocomposite has become imminent because of interest in using plant fiber as an environment-friendly reinforcement and potential replacement for synthetic fiber material in the composite polymer industry (Karthi et al., 2019; Sanjay & Yogesha, 2017). The abundance of readily available natural fibers is the perfect substitute for synthetic fibers like glass and carbon, which are typically more expensive, ecologically unfriendly, and nonrenewable (Mustafa et al., 2018). The natural fibers in composites are generally lighter, less expensive, and abundantly available, all of which contribute to the viability of a circular economy by significantly reducing carbon emissions and improving the mechanical performance of biopolymers (Atiqah et al., 2020; Kamarudin et al., 2020; Mohammed et al., 2015; Sanjay et al., 2015; Srinivas et al., 2017).

There has been growing interest in thermoplastic-based natural fiber composites (NFRCS) for high-performance engineering applications (Balla et al., 2019), with successful proof of their application to various structural and non-structural applications, especially for automotive (Boland et al., 2015; Vinayagamoorthy & Manoj, 2018), packaging (Sánchez-safont et al., 2018) and the construction industry (Mochane et al., 2019; Tadimetri, 2019). Thermoplastics offered better design flexibility and relatively simple processing techniques than thermoset and elastomer polymers. Thermoplastics such as polyethylene (PE), polypropylene (PP), nylon, high-density polyethylene (HDPE), and polyvinyl chloride (PVC) could compound with natural fibers (such as wood, kenaf, flax, hemp, cotton, oil palm, sisal, jute, and straw) to produce composites with elevated temperature (Mohammed et al., 2015; Sanjay et al., 2017; Siakeng et al., 2018; Gowda et al., 2018).

Kenaf (*Hibiscus Cannabinus*, L. family *Malvaceae*) is a cellulosic source that can be cultivated in various climate conditions. It is often produced in tropical and subtropical countries like Malaysia (Akil et al., 2011; Bhambure & Rao, 2021). Kenaf fiber has surprisingly increased worldwide because it has been appraised as environmentally beneficial and has high commercial interests. Many researchers in Malaysia are exploring kenaf fibers as a low-cost, low-density, renewable, recyclable, non-abrasive behavior, and biodegradable alternative to synthetic fibers (Guillou et al., 2018; Saba et al., 2015; Salem et al., 2017). Kenaf fiber's biodegradability contributes significantly to sustainable ecology, while it is low cost and good performance resolved the manufacturer's economic concerns. Numerous studies have examined using kenaf fiber polymer composites (Bledzki et al., 2015; El-shekeil, Sapuan, Abdan, et al., 2012; Hamidon, 2019; Sood & Dwivedi, 2018; Sreenivas et al., 2020; Thiruchitrabalam et al., 2012). Bledzki et al. (2015) researched four types of natural fibers typically used in the polymer industry (softwood, abaca, jute, and kenaf), and kenaf fiber reinforcement provides a remarkable strength for biocomposites.

Recent studies emphasize increasing the properties of engineering polymers such as polyamides reinforced with natural fiber, accounting for their excellent mechanical properties, thermal properties, and chemical stability compared to PE and PP (Zhang et al., 2020). When compared to thermoset polymers (epoxy & polyester), polyamide 6 (PA6) has attracted much interest because of its resilience to high temperatures, oils, and corrosive chemicals, relative strength-to-weight ratio, and better recyclability, which is significant for life cycle analysis (LCA) (Kiziltas et al., 2016). Correspondingly, its favorable thermo-mechanical qualities have been regarded as a conventional matrix material for natural fiber-reinforced composites. Kunchimon et al. (2019) also concluded its excellent compatibility with lignocellulosic fillers due to their similar hydrophilicity (Xu et al., 2018).

This study aims to determine the effect of various kenaf fiber contents on the thermal properties of PA6 composites. A thorough study of the thermal behavior of kenaf / polyamide 6 composites in terms of TGA, DMA, and DSC shall be discussed. This paper presents the experimental setup of the composite materials and their respective thermal testing. Results of the thermal behavior on kenaf/ polyamide 6 composites will be discussed.

MATERIALS AND METHODS

Materials

In this research, kenaf fiber was supplied by National Kenaf and Tobacco Board (LKTN) Perlis, Malaysia and was dried in the oven for 24 hr at 60°C. The kenaf fiber size of 40 mesh (400 µm) was used in this study. Meanwhile, Polyamide 6 was supplied by Polycomposite Sdn. Bhd (Perlis) with a density of 1.13 g.cm⁻³.

Kenaf Fiber / Polyamide 6 Composite Fabrication

The kenaf fiber / PA6 composites were prepared in two steps: melt compounded using the Haake Polydrive R600 internal mixer followed by Vecho Vation 40 tonnes compression molding based on composite compositions of 5%, 10%, and 15% (Table 1). Before feeding the components into the mixer, all components were manually mixed. The mixing process involves processing temperatures of 230°C, 235°C, 240°C, and 245°C with a 5-min duration and a rotating speed of 50 RPM. All

composite formulations resulting from the compounding procedure were hot pressed for 5 min at 220°C with dimensions of 150 mm × 150 mm x 3 mm (width x length x thickness).

Table 1
Formulations of kenaf fiber / polyamide 6 composites

Samples	PA6 (wt.%)	Kenaf fiber (wt.%)
PA6	100	0
5% KF	95	5
10% KF	90	10
15% KF	85	15

Thermal Stability

Thermal Gravimetric Analysis (TGA). Thermal gravimetric analysis (TGA) was conducted using the TA instrument Q500 (New Castle, DE, USA) in accordance with ASTM E1131-08 (2014). These tests were performed at room temperature ranging from room temperature to 600°C at a heating rate of 10°C/min. The sample weighed 5 to 6 mg under a nitrogen atmosphere that flowed at 50 mL/min.

Differential Scanning Calorimeter (DSC). A differential scanning calorimeter (DSC) test was evaluated with a TA instrument Q20 based on an adaptation of the ASTM D3418-15 standard (2015). In DSC, the samples weighing 5 and 6 mg were heated from room temperature to 250°C at 10°C/min throughout the heating cycle. The melting and crystallization behavior of materials was studied using a DSC thermograph. Equation 1 is used to calculate the percentage of crystallinity for each curve. The peaks of fusion values were applied to calculate the crystallinity degree (X_c) by fusion enthalpy values (ΔH_m). The theoretical 100% crystalline ($\Delta H_{100\%}$) heat fusion of PA6 equals 230 J/g (Milot et al., 2015).

$$X_c = (\Delta H_m / \Delta H_{100\%}) \cdot 100 \quad [1]$$

Dynamic Thermomechanical Analysis (DMA). Kenaf fiber / polyamide 6 composites were subjected to dynamic thermomechanical analysis (DMA) to measure the glass transition temperature (T_g), storage modulus (E'), loss modulus (E''), and loss factor ($\tan \delta$) using the TA instrument Q800 following ASTM D4065-12 (2012). The test temperature ranged from room temperature to 200°C, a heating rate of 3°C/min, a frequency of 1 Hz, an amplitude of 15 m, and specimens of 60 mm x 12 mm x 3 mm. Three samples were tested, and the findings are reported as an average of tested samples.

RESULTS AND DISCUSSION

Thermogravimetric Analysis (TGA)

The weight loss (TG) and its derivative (DTG) curves of neat PA6 and KF/PA6 composites are shown in Figures 1 and 2, respectively. The neat PA6 and KF/PA6 composites had found only one stage of the degradation process above 250°C. Increments in degradation temperature were observed with the increase in kenaf fiber content loading. The TG curve shows three mass loss steps. The first mass loss step for the neat PA6 and the KF/PA6 composites was in the range of 30-130°C, which is attributed to absorbed moisture evaporation (El-shekeil, Sapuan, Abdan, et al., 2012; Şeker Hirçin et al., 2020; Xu et al., 2019) and the neat PA6 showed more mass loss up to 4% compared to less than 3% in the kenaf fiber/PA6 composites. Besides, the degradation of the three main components of kenaf fibers, lignin, hemicellulose, and cellulose, are due to the second and third mass loss phases at 200–430°C and 360–480°C, respectively.

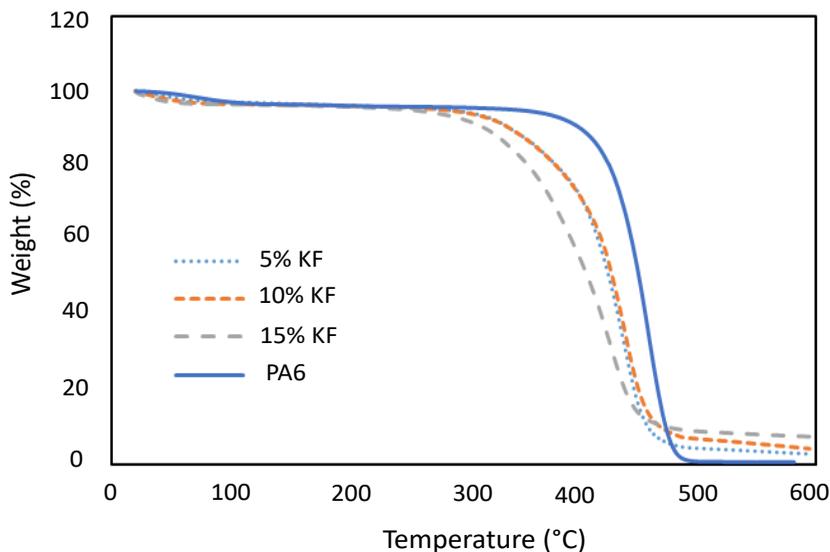


Figure 1. The weight loss (TG) curves of neat PA6 and KF / PA6 composites for various KF loadings

The thermal stability of the neat PA6 and KF/PA6 composites can be seen by their thermal decomposition temperature at the initial (T_{initial}) and final (T_{final}) as well as maximum decomposition temperature (T_{max}) (Kamarudin et al., 2020). The temperature of maximal deterioration for neat PA6 and KF/PA6 composites appears to be approximately 350°C – 450°C, similar to past researchers' findings (Kiziltas et al., 2016). Figure 2 shows thermal stability profiles for neat PA6 and KF/PA6 composites via derivative percentage weight thermograms (DTG). From the figure, the neat PA6 matrix decomposed rapidly at 425°C, which was comparatively higher than the KF/PA6 composites. The initial degradation temperature of neat PA6 was recorded at 249°C, and the degradation was completed at 457°C, with a weight loss of about 95.03%. Upon blending PA6 and kenaf fiber, the KF/PA6 composites decompose at a temperature less than neat PA6—initial and final temperatures of 15wt.% kenaf fiber showed the lowest values compared to 5 wt.% and 10 wt.% kenaf fiber. It reflected the lower thermal stability compared to 5 wt.% and 10 wt.% kenaf fiber. It also showed the lowest decomposition temperature at 361°C with a weight loss of about 88.6%.

The increase in kenaf fiber contents had recorded decreased thermal and summarized in Table 2. Lower thermal stability for all KF/PA6 composites is probably due to kenaf fiber's lower decomposition temperature behavior. It may have accelerated the disintegration of PA6's crystalline form, thereby reducing the composite's thermal stability (Kamarudin et al., 2020). Since some of PA6 has been replaced with less thermally stable kenaf fiber, the thermal stability of PA6 has been reduced. Furthermore, that may be because PA6's relative molecular mass has reduced (Zhu et al., 2020). Most researchers used chemical

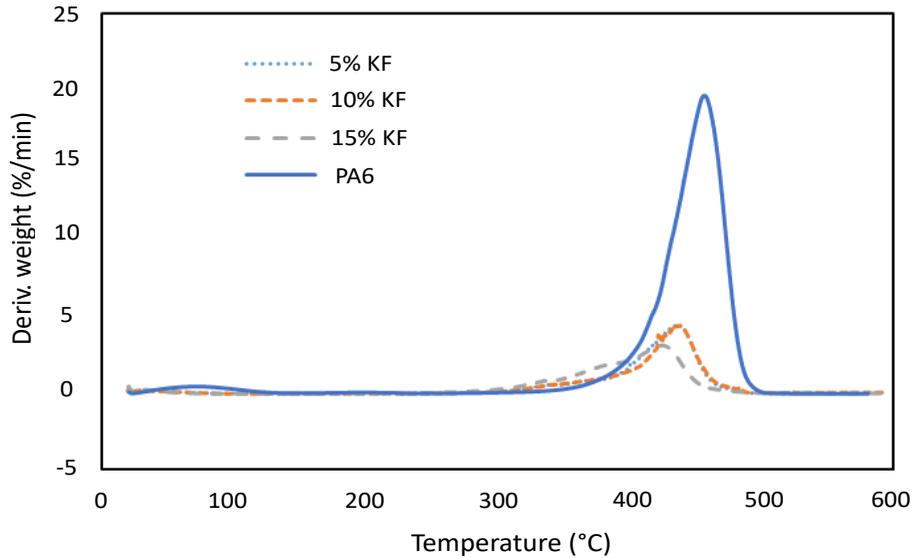


Figure 2. Derivatives percentage weight thermogram (DTG) curves of neat PA6 and KF/PA6 composites for various KF loadings

fiber modifications to overcome the composite's thermal stability (Hashim et al., 2017). One of the studies by Akhtar et al. (2016) stated that due to a higher decomposition temperature, treated composites are more thermally stable than untreated composites. The alkali treatment increases the thermal stability of natural fiber composites (El-shekeil, Sapuan, Khalina, et al., 2012). The residue weight of the composite samples at the end of the TGA measurement increased proportionally from around 0.8% (neat PA6) to 7.6% (15wt.% kenaf fiber), showing that the higher residue for higher natural fiber contents is due to the more lignin constituent in the components. The lignin forms char and maintains the structural integrity of the composite. Therefore, a higher residue was found.

Table 2

TGA results of neat PA6 and KF/PA6 composite for various kenaf fiber loadings

Sample	T _{initial} (°C)	T _{final} (°C)	T _{max} (°C)	Weight loss (%)	Residue at 600°C (%)
PA6	249.81	457.29	425.72	95.03	0.808
5% KF	163.64	437.12	394.80	94.77	2.985
10% KF	129.92	438.20	395.46	92.00	4.430
15% KF	105.13	425.32	361.69	88.76	7.657

Dynamic Mechanical Analysis (DMA)

Figure 3 shows the neat PA6 and KF/PA6 storage modulus. As the temperature increases, the storage modulus decreases due to kenaf fiber stiffness loss. The storage modulus of neat PA6 was 1177 MPa, while the KF/PA6 composite decreased to 1076 MPa with 5 wt.% kenaf fiber contents. The results showed that all KF/PA6 composites have lower storage modulus values than neat PA6. The damage to the matrix, degraded interfacial adhesion, and bonding strength between the matrix and fiber might be caused the storage modulus to reduce. The storage modulus of composites with poor adhesion has been found to produce composites with weak interfacial bonding (Akil et al., 2011). However, the 15 wt.% of kenaf fiber reinforcement was attributed to better stress transfer at the fiber interface, resulting in a higher modulus than 5 wt.% and 10 wt.%.

Figure 4 shows the loss modulus of neat PA6 and KF/PA6 composites. The loss modulus of the neat PA6 shows the highest values of 111 MPa and followed by 15 wt.%, 10 wt.%, and 5 wt.% kenaf fiber insertion composites with 89 MPa, 88 MPa, and 82 MPa, respectively. When kenaf fiber concentration is higher (15 wt.%), the polymer chain mobility's restriction becomes severe. Under external stress, the kenaf fiber particles and the PA6 matrix rubbed against one another across the interface, increasing energy consumption compared to the neat PA6 molecular chain's movement as well as the loss modulus of the composite (Zhu et al., 2020). From the overall observation, the thermal-mechanical characteristics of kenaf fiber-reinforced polyamide 6 Composites were decreased in terms of storage modulus and loss modulus.

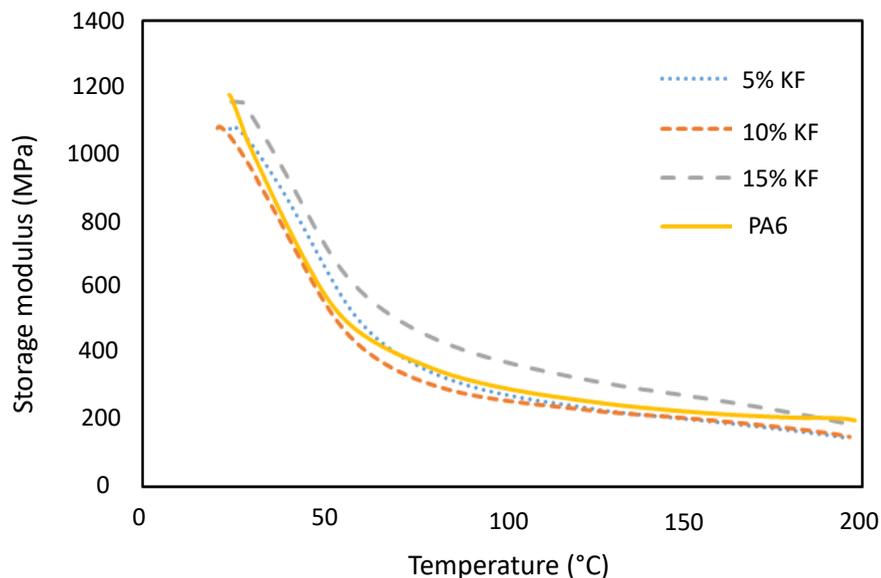


Figure 3. Storage modulus, E' of neat PA6 and KF/PA6 composites for various KF loadings

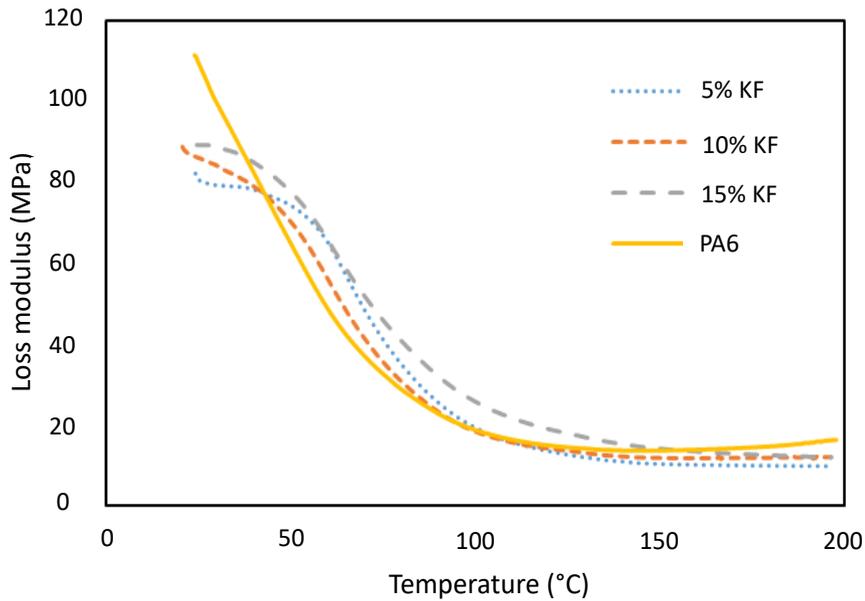


Figure 4. Loss modulus, E'' of neat PA6 and KF/PA6 composites for various KF loadings

Figure 5 shows the $\tan \delta$ (damping) of neat PA6 and KF/PA6 composites for various KF loadings. The glass transition temperature, T_g , corresponds to the $\tan \delta$ peak, and the damping is defined as the ratio of the loss modulus to the storage modulus. The KF/PA6 composite's absolute value increased when the main $\tan \delta$ peak moved to a higher temperature. The neat PA6 showed a $\tan \delta$ max peak temperature of around 52°C, whereas the KF/PA6 composites ranged from 57 to 61°C, with the presence of the 5 wt.% kenaf fiber, it is shown that there was a shift in T_g to a higher temperature. The decreased mobility of the PA6 chains due to the appearance of kenaf fiber may explain the phenomena. The highest peak of the $\tan \delta$ curve is observed on 10 wt% KF composite, indicating a greater damping property. Table 3 reports the DMA results for neat PA6 and KF/PA6 composites for various kenaf fiber loadings. The thermal properties of composites can be improved by improving fiber morphology during the treatment process (El-shekeil, Sapuan, Khalina, et al., 2012). The DMA of treated kenaf fiber improved the bondability and wettability between the polymer matrix and cellulosic reinforcement, according to Verma and Shukla (2018). It improves the load-carrying capacity of these green polymer composites.

Differential Scanning Calorimeter (DSC)

Figure 6 illustrates the DSC curves of neat PA6 and KF/PA6 composites for various KF, and Table 4 reports the DSC results for neat PA6 and KF/PA6 composites for different kenaf fiber loadings. The melting point of the neat PA6 was found to be 220°C. KF/PA6 composites ranged between 218°C and 214°C, implying that the kenaf fiber had little

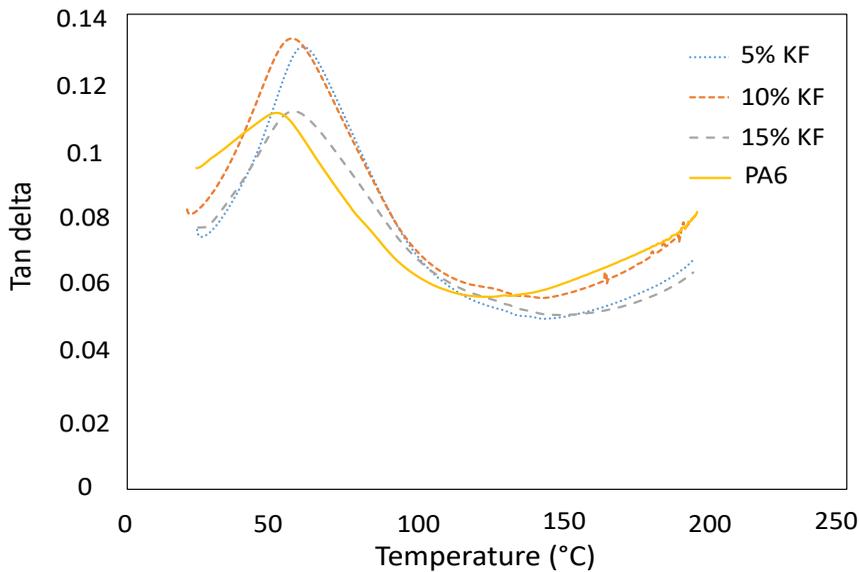


Figure 5. Tan δ (damping) of neat PA6 and KF/PA6 composites for various KF loadings

Table 3

DMA results of neat PA6 and KF/PA6 composites for various kenaf fiber loadings

Sample	Storage modulus, E' (MPa)	Loss modulus, E'' (MPa)	Tan Delta, δ
PA6	1177.49	111.49	0.1103
5% KF	1076.02	82.26	0.1303
10% KF	1077.74	88.89	0.1328
15% KF	1157.41	89.29	0.1113

influence on the T_m of the KF/PA6 composites. T_c values of neat PA6 were 208°C, and KF/PA6 composites were between 200°C and 205°C. As a result, the addition of 5 wt.%, 10 wt.%, and 15 wt.% kenaf fiber had little on the T_c of KF/PA6 composites. When the kenaf fiber was added to the PA6, the T_c value for the neat PA6 composites decreased. Adding kenaf fiber to the polymer prevents PA6 molecular chains from migrating and diffusing in the composites (Cheung et al., 2009; Huda et al., 2008). Thus, it has a negative effect on polymer crystallization, which results in a reduction in T_c . The kenaf fiber acted as a nucleating agent for the polymer to accelerate crystallization (Kamarudin et al., 2020).

The results showed that the addition of 5 wt.%, 10 wt.%, and 15 wt.% kenaf fiber in the PA6 matrix reduced the T_m . The kenaf fiber prevented the mobility of the PA6 molecular chains, resulting in partial crystals, which explains the KF/PA6 composite's decrease in

crystallinity. The fillers are good nucleating agents in polymer composites, influencing crystallization behavior significantly (Kamarudin et al., 2020). The kenaf fiber acts as a nucleating site for PA6 crystallization but can also act as a barrier to crystal development. Hence the composites have a lower crystallinity index than neat PA6.

The T_g represented the mobility of the PA6 molecular chain in the amorphous area. The T_g of neat PA6 is 52°C. After adding 5% kenaf fiber, the T_g value was increased to 160°C. T_g was detected at a low temperature since neat PA6 had no compatibility constraints, and the molecular chain motion was not hindered (Kiziltas et al., 2016). The kenaf fiber particles hindered the neat PA6 molecular chain's mobility, which required high energy and free volume to achieve the glass transition. As a result, T_g rises in KF/PA6 composites compared to neat PA6.

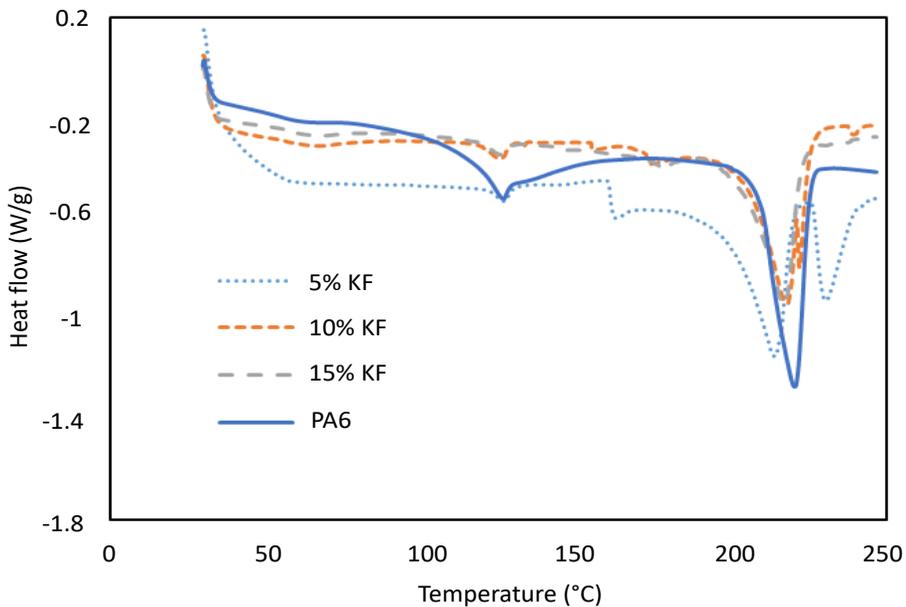


Figure 6. DSC thermograms of neat PA6 and KF/PA6 composites for various KF loadings

Table 4

DSC results of neat PA6 and KF/PA6 composites for various kenaf fiber loadings

Sample	Melting temperature, T_m (°C)	Crystallization temperature, T_c (°C)	Glass transition, T_g (°C)	Crystallinity index, X_c (%)
PA6	220.52	208.40	52.71	26.37
5% KF	214.31	200.16	160.68	22.11

Table 4 (Continue)

Sample	Melting temperature, T_m (°C)	Crystallization temperature, T_c (°C)	Glass transition, T_g (°C)	Crystallinity index, X_c (%)
10% KF	218.41	205.45	151.73	23.00
15% KF	217.13	204.15	144.03	20.28

CONCLUSION

In this study, KF/PA6 composites with different contents of KF were prepared by melt compounded, and their thermal properties were investigated in terms of TGA, DMA, and DSC. The TGA analysis proved that the kenaf fiber reinforcement had decreased the thermal stability with evidence of decreased initial and final decomposition temperature. The DMA results showed that the thermal-mechanical characteristics of kenaf fiber composites have deteriorated in terms of storage and loss modulus. However, the DSC results depicted that the glass transition temperature (T_g) of KF/PA6 composites was shifted to a higher temperature due to the affection of the crystallization behavior in KF/PA6 composites. The results showed that the addition of 5 wt.%, 10 wt.%, and 15 wt.% kenaf fiber in the PA6 matrix reduced the T_m . The composites have a lower crystallinity index than neat PA6. In conclusion, the thermal stability was decreased by increasing kenaf fiber loading. It is expected that lignocellulosic natural fibers always have higher flammability than polymer resins. Thus, improving the KF/PA6 composites can be further studied to increase thermal stability, such as through fiber treatment.

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