



# Effect of kenaf fibre loading on thermal and dynamic mechanical properties of bio epoxy composites

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## Abstract

Natural fibre-based polymer composites have better improvements owing to their thermal and mechanical properties besides environmentally friendly quality. In the present work, biocomposite fabricated at kenaf fibres (KF) loading (30, 40, 50, and 60 mass%) with bio epoxy matrix by the hot compression moulding method, and their thermal and dynamic mechanical characterisation were investigated and examined. This study was conducted to investigate the thermal behaviour through thermogravimetric analysis (TGA) and the derivative of thermogravimetric analysis (DTG: the maximum thermal decomposition;  $T_{max}$ ), dynamic mechanical analysis (DMA) and thermomechanical analysis (TMA). The presented findings from TGA and DTG curves showed that the thermal stability improved with increasing the KF loadings, as evidenced by the higher residual mass % and the lower mass loss %. On the other hand, it was found that the biocomposite sample (50 mass% KF) exhibited higher thermal stability up to 558.82 °C. Furthermore, the DMA findings obtained exhibited the greatest value of the storage modulus ( $E'$ ) with the following order of KF bio composite (KF-50 > KF-60 > KF-40 > KF-30). On the other hand, the values of loss modulus ( $E''$ ) were increased as follows KF-30 > KF-40 > KF-50 > KF-60. However, the results showed that 50 mass% of KF into epoxy biocomposites recorded higher loss modulus value (323.56 MPa) among all other bio composite samples, while the values of  $\tan \delta$  were increased as follows: KF-30 > KF-40 > KF-50 > KF-60. The results obtained for the TMA revealed a better coefficient of thermal expansion (CTE) for the sample (KF-50) compared to other samples.

**Keywords** Kenaf fibres · Bio epoxy · Bio-composites · Thermal properties

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## Introduction

In the past few years, the approach to replacements of non-renewable resources has become restricted and the expected dependence on renewable resources has been simulated due to environmental and sustainability issues. Recently, natural fibre (NF) composites have been considered sustainable materials that have grown to form a significant factor in industrial applications [1]. Therefore, several efforts by researchers have altered to eco-friendly methods to decrease the costs of materials to protect the environment. The movement from synthetic fibres towards the natural fibre composite has been conducted to the innovation of biodegradable and renewable products [2–4]. The natural resources of KF exhibit great functioning compared to other natural fibres [5]. The plants of kenaf have been greatly used over the past few years among various kinds of natural resources [6]. Kenaf is considered one of the most growing interest natural fibres based on some

properties such as a higher specific strength besides a similar specific modulus strength [7]. In addition, kenaf is characterised by several advantages and is considered a kind of lignocellulosic fibre, which consists of the family of *Hibiscus cannabinus*, that has freshly risen consideration as an improving filler of biocomposites [8], therefore, it was chosen owing to its low cost, decent hardness, good comprehensive absorption enactment, suitable strength characterisations, as well as biodegradability [9]. Moreover, KF was nominated for the reason that has already been used in automotive manufacturing, while it has a great surface and creates a lightweight material with great mechanical characterisations and thus does not have to prove itself as a reliable product [9].

Nowadays, fibre-strengthened biocomposites have achieved consideration because of their enhanced potential for substituting conservative materials for several purposes [10, 11]. Recently, kenaf fibre has been broadly utilised as strengthening in biocomposites which offers the greatest attractive choice as a result of its fast development at diverse climatic conditions and warranting low cost [12]; kenaf fibre has commonly been used as a replacement for some synthetic fibres such as glass fibre to form an eco-friendly composite. Hence, much research has been investigated in recent years on the characterisation of kenaf fibre with its biocomposites [12].

Kenaf fibres are characterised by several advantages as reinforcements of biocomposites, for example, the low density with higher specific strength and toughness [13, 14]. Moreover, kenaf fibres are used as low hazard filers throughout industrial methods with low emissions of toxic fumes while exposed to heat [15]. Furthermore, they add oxygen to the natural environment and lower the cost of production costs [16]. Therefore, several researchers tend to incorporate kenaf fibres with polymers or resins because of the developing substantial alteration in mechanical performance [17].

Much literature has emphasised the advantages of Kevlar fibres on the supplied composites and hybrid composites. For example, Akil et al. [6] investigated the mechanical, thermal, and physical characterization of kenaf fibre-strengthened biocomposites. Additionally, the manufacturing procedure and its technical concerns were solved. Also, they examined the enhancement research of kenaf fibre-reinforced biocomposites.

Particularly, epoxy matrix-strengthened kenaf fibre was studied for various uses. Park et al. [18] estimated the micromechanical characterisations of resin/kenaf biocomposites with the wettability of kenaf fibres by applying non-damaging acoustic emission. Keshk et al. [19] presented the physicochemical properties of kenaf fibres to identify their chemical compounds such as cellulose, hemicellulose, and lignin and their physical characterisations, for example, viscosity, polymerisation degree,

besides crystallinity. Xue et al. [20] stated the improvements in mechanical characterisations such as Young's modulus elasticity and tensile strength for epoxy biocomposites supported with kenaf fibres.

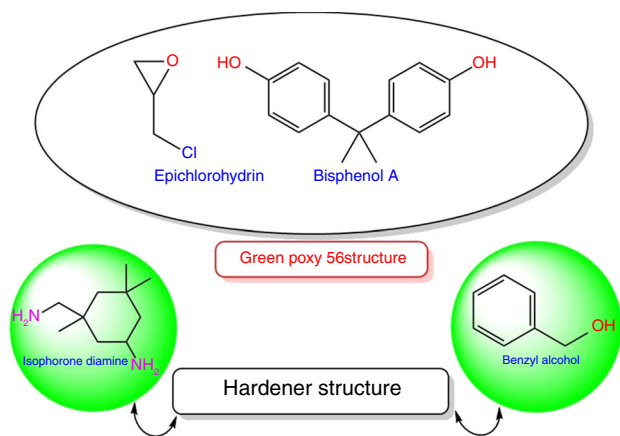
Silva et al. [21] examined the improvement of the kenaf fibre-reinforced epoxy matrix in terms of thermal and mechanical characterisations. They noted that the incorporation of kenaf with 10 and 20 vol% reduced the values of  $T_g$ , while the CTE was increased. In contrast, the 30-vol% KF biocomposite displayed a greater  $T_g$  and a lesser coefficient of thermal expansion. Abare et al. [22] estimated the physical, mechanical, and thermal characterisations of kenaf/biocomposites. They investigated different loadings of Kenaf Fibre (KF) into the bio epoxy composite. They observed that the greatest tensile strength besides Young's modulus was noted by 40.2% loading of fibre with 92.5 MPa and 9.18 GPa. In addition, the impact characterisations exhibit improvements with the enhanced loading of fibre, hence, the impact strength showed a higher value of 7280.8 J m<sup>-2</sup> with 40% fibre loading. Bakar et al. [23] examined the thermal characterisations of a KF-PVC/PVA biocomposite. They reported that the addition of kenaf fibre, developed in increasing the storage modulus.

This study aimed to evaluate the thermal and dynamic/mechanical characterisations of the KF/biocomposite. In particular, the effect of KF loading on the performance of such composite has not been explored as it is essential to identify the optimal fibre loading, specifically of the KF. In addition, this testing displayed the best mass content percentage KF, displaying the association for imperfections of manufacturing that can impact the efficiency of thermal and mechanical characterisations, in addition to examining the interfacial adhesion concerning the matrix and natural fibres. These basic findings were performed to improve a novel sustainable composite material in construction as a result of biodegradable composite materials.

## Materials and method

### Materials

In this study, kenaf fibre (KF) was gained from a local provider in Malaysia while the matrix is the commercial name of epoxy (Green Poxxy 56), which is due to the chemical name Bisphenol-A-(epichlorohydrin), which is specified as (clear liquid colour, the density at 20 °C: 1.98 g cm<sup>-3</sup>, and the viscosity at room temperature is 500 mPa.s @20 °C). On the other hand, the hardener (Isophorone diamine Benzyl Alcohol) is characterised as liquid clear, the density at 20 °C: 1.98 g cm<sup>-3</sup>, and the viscosity at room temperature is



**Fig. 1** Chemical structures of epoxy resins and hardener used in this work

30 mPa.s @20 °C. Figure 1 illustrates the chemical structure of both the used epoxy and hardener.

### Fabrication of biocomposites

The kenaf fibres were crushed and sieved to obtain a fibre size between 0.8 to 1 mm. Then the fibres are conditioned in the oven at 50 °C for 60 min before fabrication. The chopped KF were later blended thoroughly with the resin and hardener (10:3 ratio) at fibre loading as 30, 40, 50 and 60 in terms of mass %. The fabrication was done using a hot press machine in which the prepared fibre-resin mixture was placed inside the mould of dimension 300 × 300 × 3 mm<sup>3</sup> followed by curing at 100 °C for 10 min as per the recommendation from the resin manufacturer. The composites are namely as follows: KF-30, KF-40, KF-50, and KF-60 regarding their fibre mass consistent to 30, 40, and 50 besides 60 mass%, respectively.

## Characterization

### Thermogravimetric analysis (TGA)

The curve and derivative curve were obtained in the range of temperature (40–700 °C) at a heating rate of 10 °C min<sup>-1</sup> with a nitrogen purge of 50 mL min<sup>-1</sup>.

### Dynamic mechanical analysis (DMA)

The DMA parameter was performed following the ASTM D4065 using the TA (DMAQ800) instrument at the temperature range from 30 to 175 °C at the rate of 10 °C min<sup>-1</sup>.

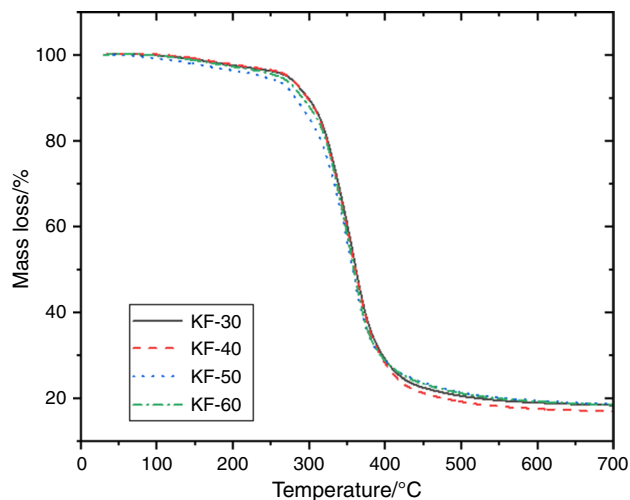
### Thermo-mechanical analysis (TMA)

The tests of TMA were monitored by using a TA instrument thermo-mechanical analyser (Model-Q400) managed in an extension mode at a rate of heating around 10 °C min<sup>-1</sup> within the range of temperature (30–180 °C). For each type, at least five specimens were examined, besides the CTE for each of those samples was estimated prior to the glass transition temperature.

## Results and discussion

### Thermogravimetric analysis

Figure 2 depicts the change of mass concerning the fibre loadings for the biocomposites. The loss in initial mass within the range of temperature (30–100 °C) was a result of the evaporation of moisture from the natural fibre [9]. After initial loss due to moisture, thermal decomposition occurred in three stages for the KF/bio-epoxy composites



**Fig. 2** Curve (TGA) curves of KF biocomposites at different loadings

**Table 1** Thermal decomposition temperature and residue from TGA curves

| Com-<br>posite<br>samples | Degradation<br>temperature/ °C |        | Maximum degradation<br>temperature from/°C DTG<br>curve<br>$T_{max}$ | Residue/% |
|---------------------------|--------------------------------|--------|--|-----------|
|                           | $T_i$                          | $T_f$  |  |           |
| KF-30                     | 232.60                         | 542.58 | 359.79   | 19.78     |
| KF-40                     | 241.42                         | 556.61 | 359.54   | 18.17     |
| KF-50                     | 215.66                         | 558.82 | 356.82   | 20.16     |
| KF-60                     | 224.44                         | 558.50 | 357.01   | 19.96     |

and the degradation temperature consistent with the maximum loss in mass can be detected in the derivative curve (DTG), as shown in Fig. 2, as well as in Table 1.

The initial temperature of decomposition ( $T_i$ ) for biocomposites displays at around 215–245 °C, in addition, the final temperature of decomposition ( $T_f$ ) ranged from 545 to 565 °C. However, the KF-50 bio composite sample exhibited the highest value of  $T_f$  (558.82 °C), while the lowest KF-40, and loading of KF (KF-30) showed a significant reduction in the  $T_f$ , which was 542.58 °C KF-60 increased with kenaf/ resin biocomposites at the loading of fibre 40%, which exhibited effective physical and mechanical, in addition, thermal characterisations. Regarding our results, we believe that our green biocomposites have the prospective to be retained in several manufacturing applications, for example, construction, automobiles, and packaging.

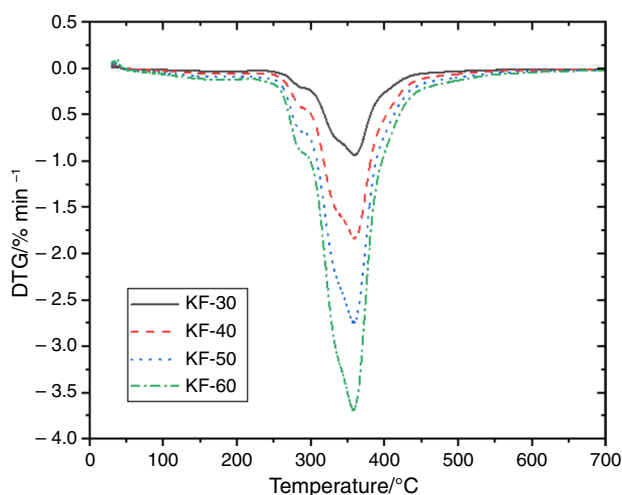
The thermal responses of the KF bio composites displayed that the KF-40 sample exhibited the highest  $T_{max}$  (359.54 °C), while the  $T_{max}$  value of KF-30 was 359.79 °C, compared to those of KF-50 and KF-60 composite samples, which were 356.82 and 357.01 °C, respectively, as shown in Fig. 3 and Table 1. A general observation was that an increase in the KF loading led to lower mass loss and higher residue %. The summarised results in Table 1, indicated that the KF loading also influenced the initial and the final temperature ( $T_i$  and  $T_f$ ), respectively. In addition, the highest fibre loading (KF-50 and KF-60) exhibited slight increases (20.16 and 19.96%), respectively, compared to other samples (KF-30 and KF-40 which were (19.78% and 18.17%), respectively. Conversely, the greatest complete residual amounts for KF-30 and KF-40 were recorded at about 19.78% and 18.17%, respectively, at 700 °C. By comparing with another related work, Abare et al. investigated the thermal characterisations of woven KF biocomposites, they

stated that the residual mass values were 15.08%, 13.81%, and 13.77% for KF-4, KF-5, and KF-6, respectively, when compared to that of pure bioepoxy of 10.41% [22].

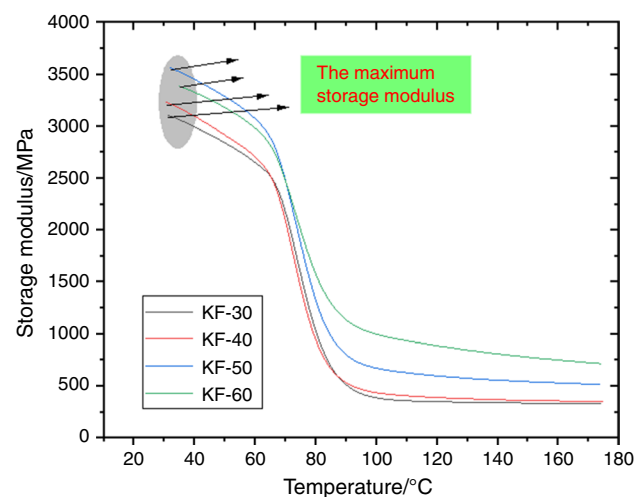
The overall findings of thermal analysis in Table 1, indicate that the incorporation of fibres enhanced the thermal stability by decreasing the  $T_{max}$  and mass loss. Related previous results investigated by Izwan et al. (2021) showed that the findings of (TGA/DTG) for KF biocomposites exhibited better thermal stability when compared to pure bioepoxy [24]. Another study was performed by Chee et al., to examine the thermal stability (TGA) for KF/biocomposites. They observed that the  $T_i$  was 282.5 °C and  $T_f$  was 408.2 °C, while the char residue was 10.4%, while the results of our study exhibited better thermal stability [25].

### Dynamic mechanical analysis (DMA)

The storage modulus ( $E'$ ) of KF/bio-epoxy composites in terms of the loading of the fibre is shown in Fig. 4, which shows three stages such as glassy and transition regions between 70 °C to 80 °C and the rubbery region. On the other hand, the glassy region is characterised by the least slope and lower molecular movement within the polymeric chains. As the material reaches the transition region, storage modulus decreases rapidly with a higher slope gradient and in this phase, the material undergoes a phase change to a rubbery state which is characterised by a greater freedom degree of molecules to transport within the chains of epoxy matrix. The temperature at which the phase transition occurs from the glassy to the rubbery region is detailed as  $T_g$  and it can be determined as the slope in the storage modulus plot or temperature corresponding to the peak of the loss modulus. The values  $E'$  vs the various temperature ranges are shown in Fig. 4. The results showed that the  $E'$  values of KF-30



**Fig. 3** Derivative curve (DTG) of KF biocomposites at different loadings



**Fig. 4** Storage modulus of KF biocomposites at different loadings

exhibited lower values (3107 MPa) than other biocomposite samples (KF-40, KF-50, and KF-60), which were (3234, 3563, and 3387 MPa), respectively. In contrast, the  $T_g$  values obtained from storage modulus vs temperature ranges in Fig. 4, exhibited slight changes. However, the bio composite sample (KF-40) showed a slight decrease in the  $T_g$  value (72.43 °C), compared to other samples, as shown in Table 2.

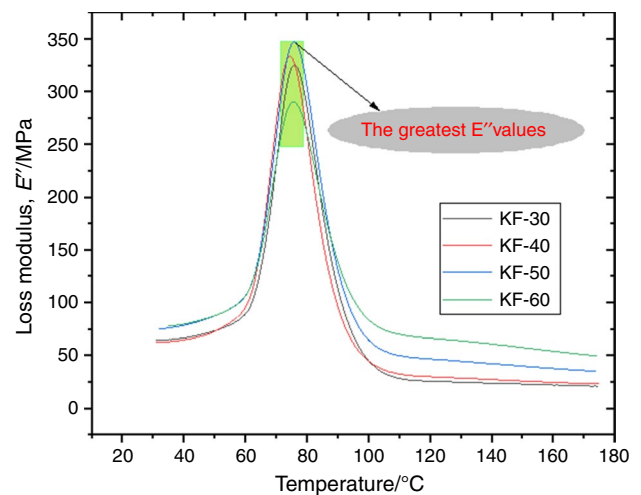
Loss Modulus ( $E''$ ) defines the energy dispersed (heat lost) through a cycle of stress in the material and can be concerned when the response of viscous of a material. A great loss modulus reveals viscous behaviour, therefore, evidenced by damping characterization [10, 21]. The  $E''$  of KF biocomposites with different loadings against different temperature ranges was shown in Fig. 5 and shown in Table 2.

The  $E''$  of the sample (KF-30) was notably higher than those of other samples at (347.94 MPa). On the other hand, the highest loading KF into bio epoxy composites (KF-60) sample exhibited a lower  $E''$  value (290.34 MPa) among all samples. However, the  $E''$  values of other samples (KF-40 and KF-50) were 331.35 and 323.56 MPa, respectively, as shown in Table 2. Thus, the results shown in Fig. 5, indicated that the increase of KF loadings into bio epoxy matrix led to an increase in the interfacial strength, which is attributed to improving the DMA properties. In addition, the increase in the loading of KF as strengthening prompted better internal resistance, causing a greater value of  $E''$ . In contrast, the values of  $T_g$  for KF-30 exhibited a slight increase (76.42 °C), compared to other samples, as shown in Table 2.

The curve of  $\tan \delta$ , additionally, termed the loss (damping) factor, is found from the ratio between the loss and storage moduli ( $E''/E'$ ), which is related to the thermal decomposition throughout both deformation cycle and elastic behaviour for the material [26–28]. Furthermore, Fig. 6 depicts the damping factor ( $\tan \delta$ ) curves of KF biocomposites. The parameter  $\tan \delta$  can be correlated with the mechanical damping factor for the material. Furthermore, the condition with a lower amount of fibre reinforcement should exhibit better structural damping as a result of the weakness's impact towards damping, particularly those associated with interfaces [29]. It was noted that the incorporation of KF with different ratios into bio epoxy matrix caused an obvious reduction in the greatest values for  $\tan \delta$ . Hence,

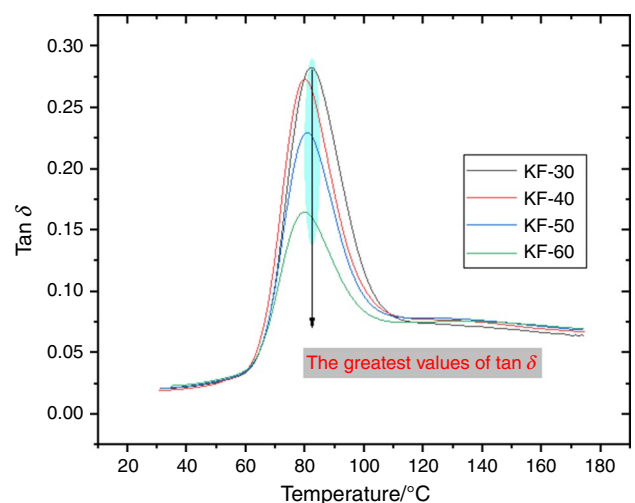
**Table 2** Parameters from DMA of KF biocomposites at different loadings

| Composite | Peak $E'$ / MPa | $T_g$ / °C | Peak $E''$ / MPa | $T_g$ / °C | Peak / $\tan \delta$ |
|-----------|-----------------|------------|------------------|------------|----------------------|
| KF-30     | 3107            | 73.84      | 347.94           | 76.42      | 0.28                 |
| KF-40     | 3234            | 72.43      | 331.35           | 74.34      | 0.27                 |
| KF-50     | 3563            | 73.71      | 323.56           | 75.65      | 0.23                 |
| KF-60     | 3387            | 73.13      | 290.34           | 75.34      | 0.16                 |



**Fig. 5** Loss modulus of the composites with respect to fibre loading of bio epoxy composites

from Fig. 6, the effect of the incorporation of KF and the variation in the temperature on damping characterisations of the biocomposites can be recorded. Regarding the lowest loading of KF (KF-30), the  $\tan \delta$  value was (0.28), which was higher damping, compared to other samples. While other biocomposite samples (KF-40, KF-50, and KF-60) exhibited the values of  $\tan \delta$  (0.27, 0.23, and 0.16), correspondingly. The lower value of  $\tan \delta$  in the damping graph shows great interfacial adhesion between hybrid natural fibre strengthening and matrix. On the other hand, the biocomposites with higher damping factors displayed lower fibre/matrix adhesion, thus resulting in a lower energy dissipation at the interface [30]. Similar findings were observed by Luz et al. [31] for the sugarcane bagasse fibre strengthened

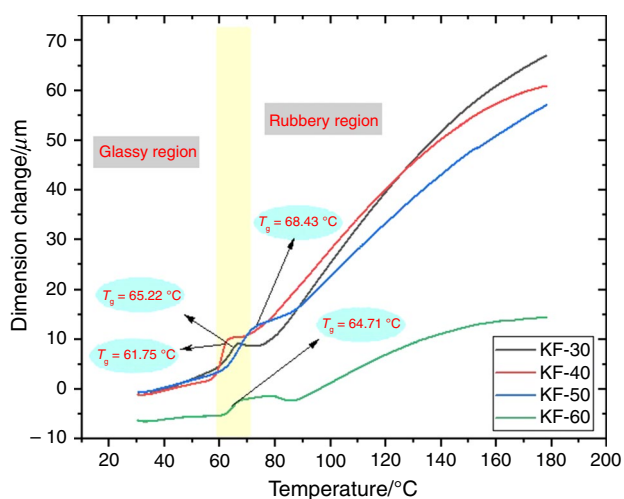


**Fig. 6**  $\tan \delta$  peaks of the composites concerning fibre loading of bio epoxy composites

with polyester composite. They found that the greatest value of the damping factor was presented by the biocomposite with higher loading of piassava fibre reinforcement. They reported that as the damping factor is associated with internal defects and interfaces of the composite, one may conclude that, in this special case, as the amount of reinforcement was increased the number of interfaces was reduced [31].

### Thermomechanical analysis (TMA)

Thermomechanical analysis of KF biocomposites was achieved to estimate the dimensional changes and the coefficient of thermal expansion (CTE), in cooperation with the regions of rubbery and glassy as a temperature function below an atmosphere of nitrogen, which are shown in Fig. 7. The CTE has proven advanced reduction by incorporating the greatest loading of KF into bio epoxy matrix. Composites displaying minimal expansion in terms of the lowest loading of KF. Furthermore, it is evident that the lowest loading of KF exhibited both contraction and expansion in extent with temperature variation. Indicated minor contraction under 100 °C relates to loss in moisture from the KF in all the cases. In addition, the plot of TMA observed that CTE for the biocomposite samples (KF-40 and KF-50) in the area of glassy and rubbery, exhibited smaller thermal expansion than the sample (KF-60). This indicated that interaction between strengthened KF besides resin matrix on the CTE for the KF biocomposites improved by the incorporation of the KF loading into the resin matrix [32]. On the other hand, the dimension changes increased with increasing temperature, as shown in Fig. 6. The temperature at which the changes in viscoelastic material from the glassy to the rubbery states is stated to that of glass transition



**Fig. 7** The dimension change vs. temperature plot of KF biocomposites at different loadings

**Table 3** CTE values and  $T_g$  values from TMA curves of bio composite samples

| Sample | CTE/ $\mu\text{m}/\text{m } ^\circ\text{C}$<br>(Below $T_g$ ) | CTE/ $\mu\text{m}/\text{m } ^\circ\text{C}$<br>(Above $T_g$ ) | $T_g$ |
|--------|---|---|-------|
| KF-30  | 104.22  | 96.33   | 65.2  |
| KF-40  | 86.31   | 207.15  | 61.75 |
| KF-50  | 59.53   | 146.25  | 68.43 |
| KF-60  | 14.32   | 13.67   | 64.71 |

temperature;  $T_g$ . It is found as the connection between the two regions, as shown in Fig. 7. Furthermore, the  $T_g$  at the glassy region is located between 60 and 65 °C for biocomposites, as shown in Fig. 7 and Table 3. Generally,  $T_g$  was found to decrease slightly with all biocomposites. However, it can be observed from Table 3 and Fig. 7 that the  $T_g$  value of KF-40, was 61.75 °C while the KF-40 sample,  $T_g$  value showed a higher value (68.43 °C). On the other hand, the  $T_g$  values for other samples KF-30 and KF-60 were 65.2 °C and 64.71 °C, respectively. Thus, this indicates that KF incorporation in the epoxy matrix is restricting the epoxy macromolecular mobility.

In previous reported work, Silva et al. [21] Comparable arguments are additionally stated in the literature on the TMA test. Beyond the assessment of TMA, it was noted that the incorporation of kenaf strengthening at 10 besides 20% increased the CTE values. On the other hand, the 30% KF biocomposite displayed a lower CTE value.

### Conclusions

In this research paper, the thermal and dynamic mechanical characterisation of KF biocomposites was examined. The findings of the study displayed that the addition of the KF as a strengthening in epoxy biocomposites extensively enhanced the thermal stability. Additionally, the sample (KF-50) exhibited higher thermal stability than those of other samples. The  $E''$  of the composites improved greatly by way of the loading of KF increased. On the other hand, the peaks depicted on the graph of the  $\tan \delta$ , which were reduced with the loading for KF rise in the biocomposite. While, the  $T_g$  values increased with the lowest loading of KF. This could be associated with an expansion in the adhesion between the fibre and the epoxy matrix. From TMA results, it was noted that the incorporation of KF with higher loading (KF-60) strengthening bio epoxy composites reduced the value of the CTE (13.6  $\mu\text{m}/\text{m } ^\circ\text{C}$ ) above the  $T_g$  region at the initial temperature. In contrast, the KF-60 biocomposite displayed a lower rate of CTE, which was 14.32  $\mu\text{m}/\text{m } ^\circ\text{C}$ , while the biocomposite sample (KF-40) displayed lower  $T_g$  values (61.75 °C) among all other samples. As a result of this

research, the incorporation of KF into the bio-epoxy matrix could be utilised in different applications, including but not limited to automotive parts, construction materials furniture. In addition, these biocomposites present a sustainable and environmentally friendly alternative to traditional materials that are often derived from non-renewable sources.

**Authors Contribution** SA and AS were involved in conceived of the presented idea, carried out the experiment, and wrote the manuscript. MH was involved in reviewing and editing: MJ involved in supervision, resources and wrote the manuscript. HF and IU were involved in verification and validation, reviewing and editing.

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**Availability of data and materials** The data that support the findings of this study are available from the corresponding author, (Jawaid, M.), upon reasonable request.

## Declarations

**Ethical approval** No ethical clearance is required.

**Human and animal rights** We declare that there are no animal studies or human participant involvement in the study.

**Conflict of interest** The authors declare no competing interests.

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