

# Investigation of Thermal Behavior for Natural Fibres Reinforced Epoxy using Thermogravimetric and Differential Scanning Calorimetric Analysis

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**Abstract.** This paper presented the research works on the investigation of the thermal behavior of the natural fibres; i.e. pineapple leaf fibre, kenaf fibre and mengkuang fibres reinforced epoxy. The thermogravimetric analysis and differential scanning calorimetric analysis were used to measure the thermal behavior of the treated and untreated pineapple, kenaf and mengkuang fibres reinforced epoxy. The samples for both analysis were subjected to maximum temperature 600°C at the heating rate of 10°C/min. The results showed that the treated fibres show higher maximum peak temperature as compared to the untreated fibres. Additionally, the glass transition temperature showed a lower value for all treated fibre. It can be concluded that investigation of thermal properties of these natural fibres could improve the utilization of natural fibre composites in various applications i.e. sports applications.

## 1 Introduction

Natural fibre reinforced epoxy is a relatively new material in the sports and recreation use as compared to the other synthetic materials. However, the usage of natural fibre composites are relatively high in automotive and construction applications [1]. Due to the increasing interest in green technology, the usage of natural fibres in polymer composites in the form of sports and recreation gaining its popularity [2, 3]. Researchers conducted various studies on many natural fibres such as hemp, jute, sisal, kenaf and pineapple leaf fibre (PALF) [4-6]. The differences in chemical composition make it essential to determine their thermal properties [7, 8]. One of the methods employed in studying the thermal properties of polymeric materials is thermogravimetric analysis (TGA). Additionally, differential scanning calorimetric (DSC) analysis is critical in determining the melting point and glass transition of a polymer composite.

Many researchers presented their works on the degradation of natural fibres with TGA. The thermal stability of various materials i.e. natural fibres has been identified by their degradation temperature [7, 9, 10]. Besides that, the DSC is also one of the necessary

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analysis to determine the chemical properties of the materials. According to Vilaplana et al. [11], glass transition somehow shows the transition between the glassy and the elastic state of a polymer. PALF and kenaf fibre have been widely discussed by many researchers [12-14]. However, least studies have been conducted on mengkuang fibre (*Pandanus Tectorius*) [15]. 12 different varieties of pineapple leaf fibre used in a study by Neto et al. [6] showed mechanical properties is inversely proportional to the diameter of the fibre bundle and lignin content. Yousif et al. [16] have studied the flexural properties of untreated and alkali treated kenaf fibre which results in improvement for the treated kenaf fibre.

In this work, the objective is to investigate the thermal properties of PALF, kenaf fibre, and mengkuang fibre reinforced epoxy under TGA and DSC analysis. A thorough study of the thermal properties of the untreated PALF, kenaf fibre, and mengkuang fibre reinforced epoxy are presented. Results of thermal properties of the PALF, kenaf and mengkuang composites were addressed. Finally, the study concluded in the last section.

## 2 Methodology

### 2.1 Materials selection process

The treated and untreated (UT) short PALF, kenaf and mengkuang fibres were used in this experiment. The treated fibres were soaked in 1% and 5% sodium hydroxide (NaOH) concentration for 1 hour, respectively. Then, these fibres were rinsed with tap water to remove all traces of the NaOH. The fibres were left to dry in aging oven for 24 hours at 90°C.

### 2.2 Composites preparation

The hand lay-up technique is used to prepare the composites. The epoxy resin (DER 331) and hardener (Jointmine 905-3) was mixed with the ratio of two to one (2:1) and stirred with the short fibres. The resin is then poured into the 200 mm x 200 mm x 3 mm mold (see Fig. 1). The composites were left for curing at room temperature for 24 hours. The volume fraction used in this study for UT fibres were 5% and 20% volume fraction. Treated fibres are using 10% volume fraction for both 1% and 5% NaOH concentration.



**Fig. 1.** Mould with dimension 200 mm x 200 mm x 3 mm.

### **2.3 NaOH treatment**

The PALF, kenaf, and mengkuang fibres were soaked in 1% and 5% concentration of aqueous NaOH, separately. These fibres were left in aqueous NaOH for 1 hour at room temperature. After that, the fibres were washed by using tap water, rinsed until there are no traces of any residual NaOH. Then, these fibres were left to dry in aging oven for 24 hours at 90°C.

### **2.4 Thermogravimetric analysis (TGA)**

The TGA Q500 V6.7 Build 203 machine is employed to conduct the TGA. The samples weighing 5.0 mg were subjected to a maximum temperature 600°C. The heating rate of the samples was set to 10°C/min. The final residue of the samples was recorded on the set of degradation temperature. The weight loss of the samples has been documented in response to increasing temperature.

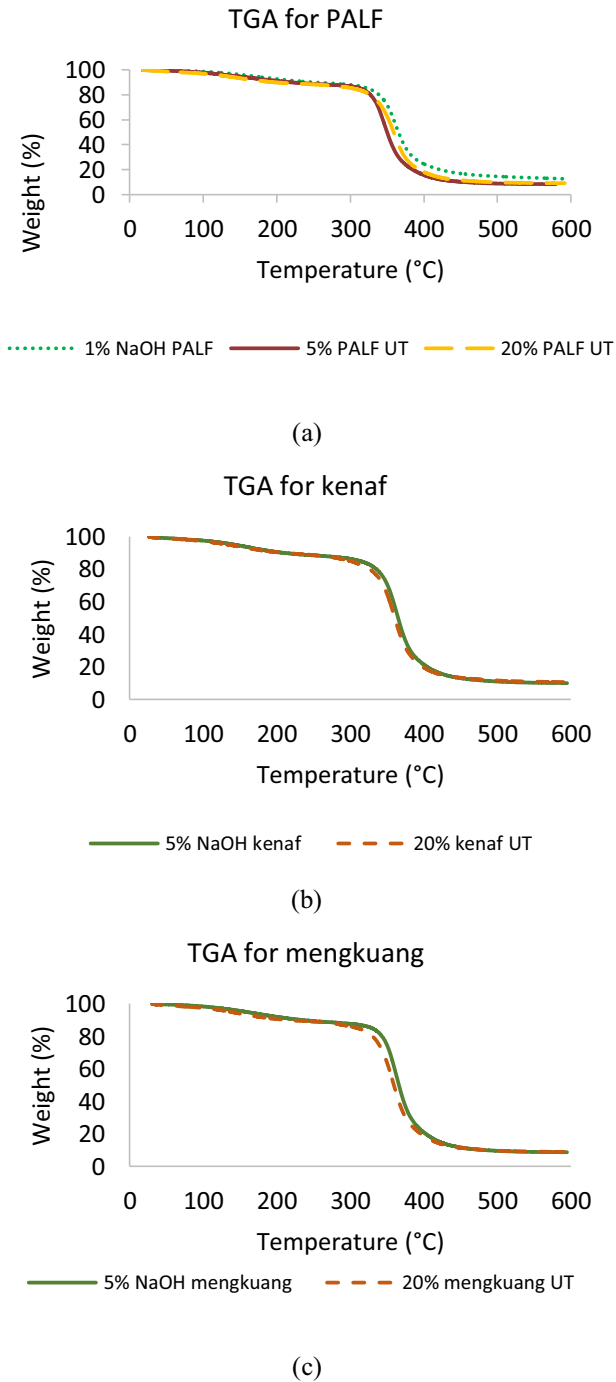
### **2.5 Differential scanning calorimetry analysis (DSC)**

Differential Scanning Calorimetry analysis was operated by using DSC Q1000 V9.9 Build 303 machine. Seven samples with the weight of 2.30 mg were used and analyzed dynamically at a heating rate of 10°C/min. The maximum temperature was set up to 600°C under a nitrogen atmosphere.

## **3 Results and discussion**

### **3.1 Thermogravimetric analysis (TGA)**

The Fig. 2 (a) up to (c) showed the results of TGA curves of PALF, kenaf and mengkuang fibres, each. In Fig. 2(a), it revealed that the TGA curve for treated and untreated (UT) PALF. The specimens show two stages of weight loss process. The first phase of transition for 1% NaOH PALF shows temperature 18°C to 175°C with weight loss 10.7% (see Table 1). De Rosa et al. [17] have studied the thermal behavior of okra fibre and revealed that the initial weight loss happened due to the vaporization from the fibre. The second stage of weight loss occurred at 265°C to 595°C with the maximum peak ( $T_m$ ) is 341°C. This phase indicated the weight loss due to decomposition of cellulose [17].  $T_m$  for 5% PALF UT was 115°C in first transition and 344°C in the second transition. Meanwhile, the 20% PALF UT produce 9.2 % of residual weight after heating from 10°C to 600°C.



**Fig. 2.** Thermogravimetric analysis curves of a) PALF b) kenaf fibre c) mengkuang fibre

The Fig. 2(b) and 2(c) showed the TGA curves of kenaf and mengkuang fibres, respectively. The decomposition of these fibres befalls in two main stages. From these figures, it clearly shows that the fibre with alkali treated has a higher value as compared to

untreated fibre. The 5% NaOH kenaf have a temperature range from 26°C - 165°C and 252°C - 595°C while the  $T_m$  for these steps were 124°C and 342°C, respectively. The 20% kenaf UT shows lower value with  $T_m$  115°C and 340°C for respectively first stage transition and second stage transition. One possible explanation is that the chemical treatment on the surface of the fibre removed a certain population of hemicelluloses and lignin constituents enhanced the thermal stability of kenaf fibre [18].

The degradation temperature for mengkuang fibre of alkali treated fibre and untreated fibre was 345°C and 339°C, respectively. However, this value is greater than reported by Sheltami et al. [15] who have compared the degradation temperature between raw, alkali-treated and bleached mengkuang fibre. The reason is that the removal of hemicellulose component, lignin, and wax in the NaOH treatment of the mengkuang fibre. This situation showed a strong agreement with Sheltami et al. [15] that stated lower degradation temperature of mengkuang fibre due to remained hemicellulose after alkali treatment.

**Table 1.** Results of TGA on treated and untreated PALF, kenaf fibre and mengkuang fibre

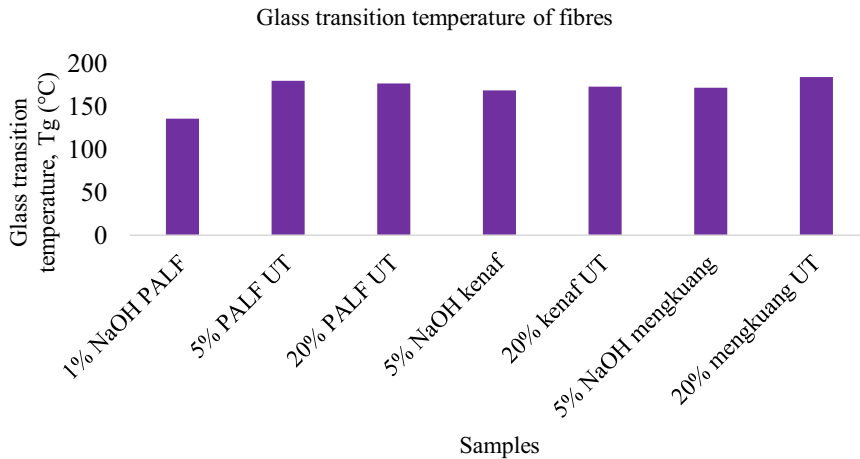
| Sample            | No. of transition | Transition temperature (°C) |       |       | Weight loss at transition (%) | Residual weight (%) at 600°C |
|-------------------|-------------------|-----------------------------|-------|-------|-------------------------------|------------------------------|
|                   |                   | $T_i$                       | $T_m$ | $T_f$ |                               |                              |
| 1% NaOH PALF      | 1                 | 18                          | 113   | 175   | 10.7                          | 13.3                         |
|                   | 2                 | 265                         | 341   | 595   | 76.1                          |                              |
| 5% PALF UT        | 1                 | 34                          | 115   | 174   | 11.7                          | 8.4                          |
|                   | 2                 | 275                         | 344   | 595   | 79.9                          |                              |
| 20% PALF UT       | 1                 | 22                          | 117   | 149   | 11.3                          | 9.2                          |
|                   | 2                 | 230                         | 338   | 595   | 79.5                          |                              |
| 5% NaOH kenaf     | 1                 | 26                          | 124   | 165   | 11.5                          | 10.2                         |
|                   | 2                 | 252                         | 342   | 595   | 78.2                          |                              |
| 20% kenaf UT      | 1                 | 30                          | 115   | 155   | 10.6                          | 10.9                         |
|                   | 2                 | 240                         | 340   | 595   | 78.5                          |                              |
| 5% NaOH mengkuang | 1                 | 30                          | 115   | 175   | 11.3                          | 8.8                          |
|                   | 2                 | 275                         | 345   | 595   | 79.9                          |                              |
| 20% mengkuang UT  | 1                 | 34                          | 117   | 150   | 10.7                          | 9.0                          |
|                   | 2                 | 230                         | 339   | 595   | 80.2                          |                              |

Table 1 shows the results of TGA for PALF, kenaf, and mengkuang fibres. The weight of the residual of 1% NaOH PALF is the highest; followed by kenaf and mengkuang fibre. The possible reason is that 1% NaOH concentration optimized the hemicellulose content in the fibre. It showed a strong agreement with the established works that the hemicellulose is responsible for the thermal degradation of fibre [13].

Additionally, the results of 5% NaOH concentration kenaf and mengkuang fibres showed that among these fibres, 5% NaOH kenaf fibre showed a greater value as compared 5% NaOH mengkuang fibre. The reason is that hemicellulose content in kenaf fibres are lower than mengkuang fibre [15, 19].

### 3.2 Differential scanning calorimetry (DSC)

Fig. 3 summarized the value of DSC analysis. The glass transition temperature ( $T_g$ ) of treated PALF, kenaf fibre and mengkuang fibre showed are lower than the untreated (UT) fibre. The  $T_g$  of 20% mengkuang fibre UT outperformed the other samples. On the other hand, the  $T_g$  of 1% NaOH PALF is the lowest value. The reason is that the NaOH treatment reduced the formation of hydrogen. It presents a strong correlation with the established studies on kenaf-filled chitosan biocomposites [20]. The result from the study shows a greater maximum peak on a sample with higher hydrogen content.



**Fig. 3.** Glass transition temperature,  $T_g$  of PALF, kenaf and mengkuang fibre.

## 4 Conclusion

The finding of this study showed that NaOH treated fibre specifically the kenaf and mengkuang produced higher maximum peak temperature than the UT fibres. However, for glass transition temperature, the treated PALF, kenaf and mengkuang fibre, the outcome showed lower value as compared to the untreated fibre. Further study needs to explore on physical properties testing such as the water absorption, melting point and density. This paper provide the DSC analysis results that can only provide glass transition temperature. The investigation of thermal properties for PALF, kenaf and mengkuang fibres could bring an improvement of using natural fibre composites for the usage of producing the sports equipment.

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