

# Effect of Soaking Time on the Mechanical Properties of Kenaf/Epoxy Composites

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## Abstract

Mechanical properties of Kenaf/Epoxy composites are depended on the bonding between fibers and matrix. Fiber treatment is one of the methods to improve the fibre/matrix bonding and subsequently enhances their mechanical properties. Hence an investigation was conducted on various soaking time (0, 12, 24 and 48 hours) during fiber treatment using 6% concentration of sodium hydroxide (NaOH). Two types of composite panels were then prepared which consisted of 20 wt% untreated and treated kenaf fibers with fiber size in the range of 150 – 300 micron and 350 -500 micron. The Tensile, Flexural and Fracture tests were then performed on the composite specimens according to ASTM D3039, ASTM D790 and ASTM E399 respectively. Scanning Electron Microscope (SEM) was used to observe the morphological surface of kenaf fibers and its composites. The investigation showed that 24 hours is the best soaking time for 6% NaOH concentration for both kenaf size range of 150 – 300  $\mu\text{m}$  and 350-500  $\mu\text{m}$ . The highest improvement is up to 59% and 71% of tensile and flexural strengths, achieved by 150 - 300  $\mu\text{m}$  kenaf/epoxy composite, meanwhile 95% and 60% improvement for tensile modulus and flexural modulus for the same kenaf size. The fracture toughness has also showed enhancement of 69% upon 24 hours soaking time.

**Keywords:** Alkali treatment; Flexural strength; Kenaf fibre; Tensile strength.

## 1. Introduction

In recent years, due to the increase in environmental concerns, scientists and technologists have placed so much attention on the application of natural materials. This move has encouraged industries like furniture, automotive, building construction, and packaging to search for new forms of fiber reinforced composite that can substitute the conventional composite materials which used synthetic fibers (glass fiber, carbon fiber) as reinforcement. Hence natural fibers that normally extracted from plants is suggested as a substitute reinforcement.

Taking the advantages of natural fiber composite properties compared to glass fiber composites, such as non-abrasive to equipment and freedom from health problems due to skin irritation during handling and processing, the use of natural fiber composites is preferred. Compared to inorganic reinforcing fibers, natural fibers also have advantages such as low density and bio-degradability, less abrasiveness, low cost and renewable. Natural fiber composites are environmentally superior to glass fiber composites in most cases. Natural fibers like jute, pineapple leaf, sisal, flax, hemp, kenaf, coir, and abaca have been used by researchers to replace the inorganic fibers (glass, aramid and carbon) in reinforced composites. Natural fibers will be in disadvantages condition as compared to inorganic fibers if comparison of their mechanical properties such as strength occurs as listed in Table 1.

Recently, kenaf is used as a raw material to be alternative to wood in pulp and paper industries for avoiding destruction of forests and also used as non-woven mats in the automotive industries. Kenaf (*Hibiscus cannabinus*) is a warm-season annual fiber crop growing

in temperate and tropical areas. It is a fibrous plant, consisting of an inner core fiber (75–60%), which produces low quality pulp, and an outer bast fiber (25–40%), which produces high quality pulp, in the stem. The plant grows to a height of 2.7–3.6m and is harvested for its stalks, from which the fiber is extracted [1]. Kenaf has a bast fiber which contains 75% cellulose, 15% lignin and 10% pectin and offers the advantages of being biodegradable and environmentally safe. Malaysian kenaf is composed of two distinct fibers, bast and core, with a makeup of about 35% and 65%, respectively. Each fiber has its own usage; thus, separation of the fibers produces higher monetary returns over whole-stalk kenaf. Major factors involved in separation of kenaf into its two fractions include: size and amount of each portion; type and number of separation machinery; processing rate through separation machinery; moisture content of whole-stalk kenaf; humidity of ambient air [1].

Hence, in order to produce a natural fiber composite epoxy has been chosen for this study to combine with kenaf fiber as reinforcement. Epoxy has more advantages compare to other resins as listed in Table 1, it has high mechanical and thermal properties compared to other resins. So, it is suitable to be used in this study. Epoxy resin is polymer matrix which is widely used in advanced composites since epoxy are good in stiffness, has good dimensional stability and good chemical resistance. Epoxy is also quite low in molecular weight monomers with low shrinkage during cure. Even though, it quite expensive than other resins, but its quality is more important.

However, poor adhesion between fiber and matrix is always the problem when fabricating natural fiber composites due to the hydrophobic and hydrophilic characteristics of polymer matrix and

natural fibers [2]. Poor adhesion at the interface means that the full capabilities of the composite cannot be exploited and leaves it vulnerable to environmental attacks that may weaken it, thus reducing its life span [3]. Insufficient adhesion between hydrophobic polymers and hydrophilic fibers result in poor mechanical properties of the natural fiber reinforced polymer composites.

**Table 1:** Properties of Fiber and Polymer [4]

Natural Fiber	Tensile Strength (MPa)	Elongation at break (%)	Young Modulus (GPa)
Carbon	3500 - 6000	1.5 - 2	230 - 600
Glass	3100 - 4650	4.7 - 5.3	72 - 86
Flax	300 - 1500	1.3 - 10	24 - 80
Jute	200 - 800	1.2 - 8	10 - 55
Sisal	80 - 840	2 - 25	9 - 38
Kenaf	295 - 1191	3.5	2.86
Pineapple	170 - 1627	2.4	60 - 82
Banana	529 - 914	3	27 - 32
Coir	106 - 175	14.2 - 49	4 - 6
Oil Palm	130 - 248	9.7 - 14	3.58
Ramie	348 - 938	1.2 - 8	44 - 128
Polyester	40 - 90	2	2.0 - 4.5
Vinylester	69 - 83	4.0 - 7.0	3.1 - 3.8
Epoxy	35 - 100	1.0 - 6.0	3.0 - 4.0

Several researchers employed fiber treatment to enhance the bonding between fiber and matrix and alkali treatment is one of the methods commonly used. Investigation of different percentage of the alkali solution (5%, 7%, 10% and 15% in weight) and immersion times (1, 3 and 24 h) to treat natural fibers [2] was done. In particular, some researcher used different concentrations (3%, 6% and 9%) of NaOH to pre-treat kenaf fibers for 3 h at room temperature. For the 6% NaOH concentration, two different conditions were used (immersion at room temperature and immersion in water bath at 95°C) [5,6] and found out that pre-treatment of kenaf fibers in 6% NaOH solution in water bath leads to the best results. The results based on 6% NaOH treatment is an evidence that treatment of fibers by alkalization helps in improving the mechanical interlocking and chemical bonding between the matrix and fibers resulting in better mechanical properties [2]. The alkali treatment enhances the fibers surface adhesion by removing natural and artificial impurities, as well as producing a rough surface topography [3]. One of the important parameter for alkali treatment that affects the mechanical interlocking of fiber and matrix is soaking time. The cellulose component as the main reinforcement of composite material will be damaged by excessive soaking time during alkali treatment of natural fiber thus reduce its mechanical properties [4]. Hence this study was conducted to evaluate the mechanical properties of kenaf/epoxy composites at a various soaking time (12 hr, 24 hr, 48 hr) for alkali treatment (6% NaOH concentration).

## 2. Materials and Methods

### 2.1. Fiber Preparation

As received pulverized Kenaf fibers were sieved using 500 – 350  $\mu\text{m}$  and 300 - 150  $\mu\text{m}$  sieve shaker prior to the treatment process. Kenaf fibers were then soaked in a Sodium Hydroxide solution (6% concentration of NaOH) at three different soaking times as listed in Table 2. Treated kenaf fibres were then washed with distilled water thoroughly in order to remove the excess of NaOH and dried in an oven at 80°C for 24 hours. Dried kenaf fibers were then mixed with epoxy and its hardener at 20 wt% and 80 wt% respectively. The mixture was poured into steel mould to produce a composite panel with a dimensions of 210 mm  $\times$  300 mm  $\times$  4 mm. A composite panel which consisted of untreated kenaf fibers (20 wt%) was also prepared as a control sample.

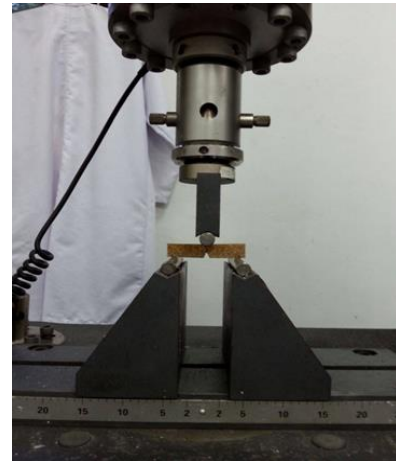
**Table 2:** Composite Composition

Kenaf/Epoxy	Kenaf Fiber Size ( $\mu\text{m}$ )	
	500 – 350	300 - 150
Soaking Time (hr)	0	
	12	
	24	
	48	

A composites prepared with kenaf fiber with size 500 – 350  $\mu\text{m}$  are named as 500  $\mu\text{m}$  kenaf/epoxy composites and for composites with kenaf fiber size of 300 – 150  $\mu\text{m}$  are named as 300  $\mu\text{m}$  kenaf/epoxy composites for easier identification and reporting.

### 2.2. Mechanical Testing

The tensile, flexural and fracture test specimens were cut from the composite panels and the tests were performed according to ASTM D3039, ASTM D790 and ASTM E399 respectively. All tests were conducted at a loading rate of 1 mm/s. The fracture test specimens were notched following the requirement of ASTM E399 prior to the test as shown in Fig. 1. Five specimens were tested for each test.



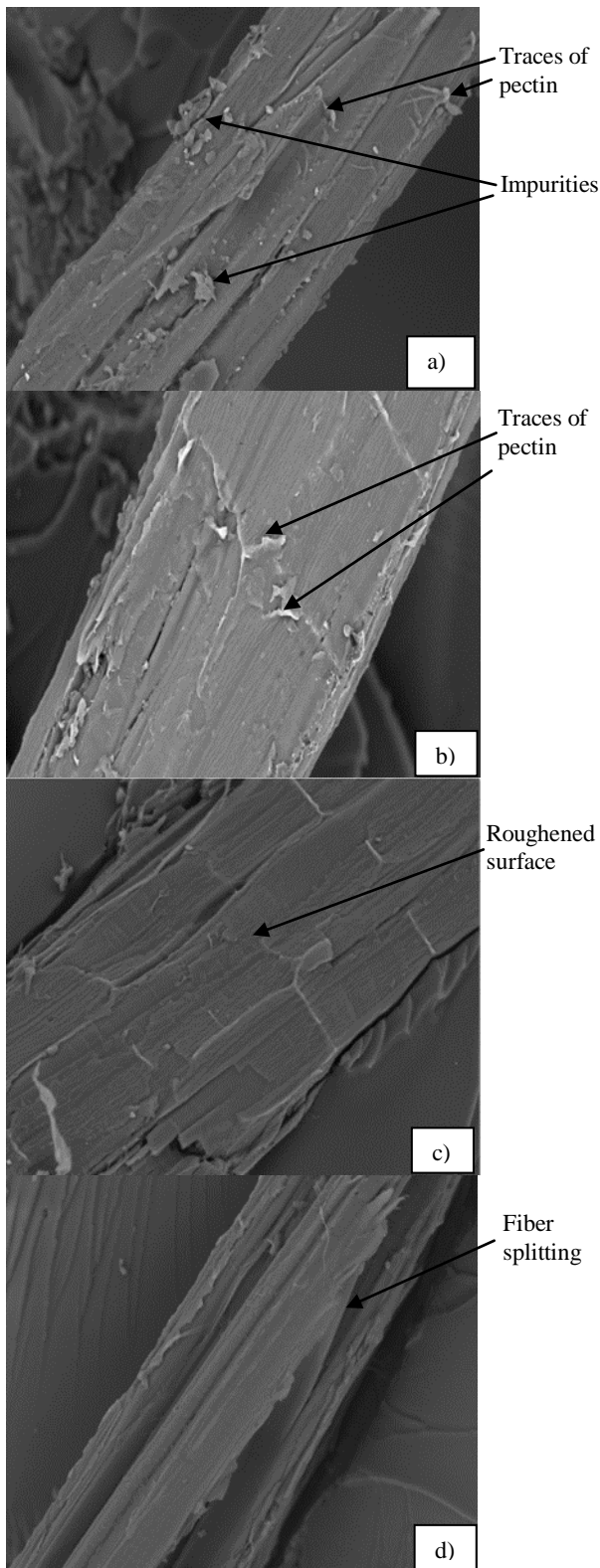
**Fig. 1:** Fracture test setup

## 3. Results and Discussion

Two groups of composite system which consists of different kenaf size as listed in Table 2 are discussed in terms of observation on the kenaf fiber surfaces before and after treatment. Their mechanical performances are also discussed based on the tensile, flexural and fracture tests.

### 3.1. Kenaf Fiber Surface Morphology

Observation of the kenaf fiber surfaces before and after treatment are shown in Fig. 2. The untreated fibers (Fig. 2 a) clearly show impurities and traces of pectin on the surface that can influence the adhesion properties with the resin in the composites manufacturing. Traces of pectin are still can be observed from Fig. 2 b) which belongs to (300  $\mu\text{m}$ ) kenaf after 12 hours soaking time during treatment. This indicates that 12 hours is not sufficient to remove all the pectin residues. Insufficient NaOH concentration also showed the similar feature [7, 8].



**Fig. 2:** Surface morphology of kenaf fibers a) Untreated kenaf, b) 12 hour, c) 24 hour and d) 48 hour soaking time.

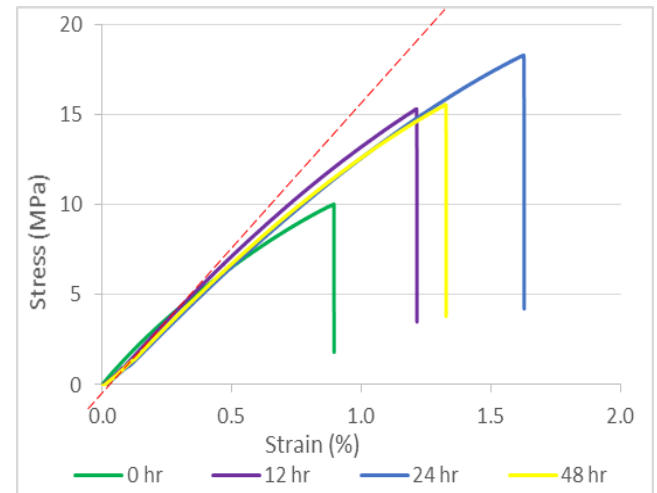
Kenaf fiber treated at 24 hours (Fig. 2 c) has a cleaner (free from pectin and impurities) and rougher surface morphology as compared to the untreated and 12 hours soaking time. Rough surface will provide good bonding between fiber and matrix hence the stress can be effectively transferred from the matrix to the fiber.

Meanwhile kenaf fiber treated in the longest time, 48 hours showed some damaged feature (Fig. 2 d) where the fiber seems splitting. This phenomenon is due to the increase of the soaking time in the NaOH solution that damages the surface of the fibers,

thus resulting in lower tensile strength [4]. Free of pectin traces means the matrix was able to come in contact with the cellulose directly lead to good bonding, meanwhile damaged or splitted fibres caused a decrease in strength to the kenaf fibers, thereby the fibers were unable to provide good reinforcement to the composites [9, 10].

### 3.2. Stress – Strain Curves

Fig. 3 shows the tensile stress-strain curves of kenaf/epoxy composites. All composites showed brittle characteristic indicated by the almost linear curve up to fracture. Only a slight deviation from tangent line (red dashed line) was observed, indicating low ductility of the composites. Stress – strain curves of flexural tests were also have the same characteristic of tensile stress-strain curves.



**Fig. 3:** Tensile stress-strain curves of composites

### 3.3. Tensile and Flexural Strengths

In general treated kenaf fibers produce higher strength of composite and improved further with the increasing of soaking time up to 24 hours as shown in Fig. 4. The increases of tensile strength and flexural strength of 500  $\mu\text{m}$  kenaf/epoxy composites are 50% and 37%, meanwhile for 300  $\mu\text{m}$  kenaf/epoxy composites the increases are 59% and 71% respectively. The increase indicates a good bonding between kenaf fiber and epoxy after treatment, where alkalization helps in improving the mechanical interlocking and chemical bonding between the matrix and fibers resulting in better mechanical properties [2]. Good bonding was also indicated by the presence of fibre fracture on the fracture surface as shown in Fig. 5.

However, beyond 24 hours soaking time or at 48 hours both tensile and flexural strengths have decreased as seen in Fig. 4. The lower strength of composite that produced by 48 hours soaking fiber treatment is believed due to damage cellulose by excessive soaking time during alkali treatment [4], the evidence of damage fiber at 48 hour treatment can be seen in Fig. 2 d).

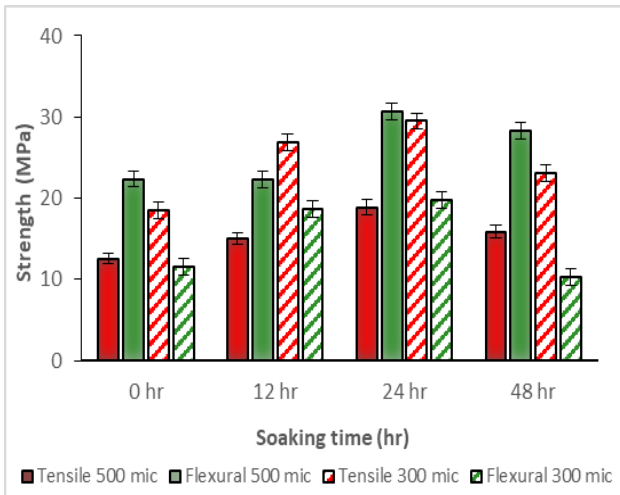


Fig. 4: Tensile and Flexural strengths of Composites

Comparing the kenaf size, (500  $\mu\text{m}$  and 300  $\mu\text{m}$ ) used to reinforce the composites, showed smaller size (300  $\mu\text{m}$ ) has more improvement of strength. This is believed due to the 6% NaOH concentration used in this study is insufficient to roughened larger (500  $\mu\text{m}$ ) kenaf fiber surface that leads to poor bonding indicated by traces of fiber pull-out on its fracture surface similar to untreated kenaf/epoxy composite as shown in Fig. 6. Hence it is suggested to increase the concentration of NaOH for further investigation.

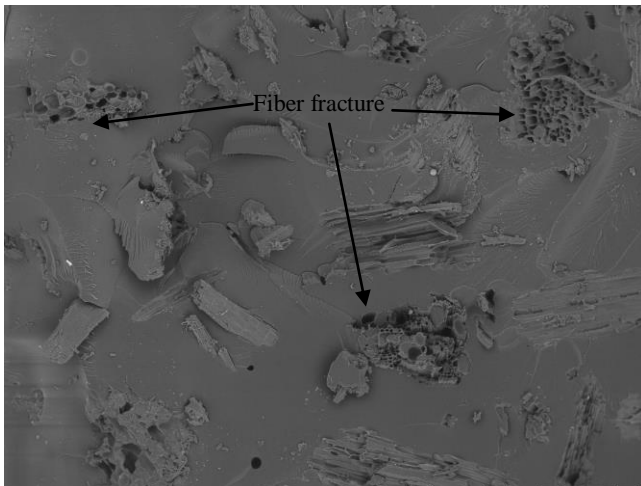


Fig. 5: Fracture surface of 24 hours soaking treatment kenaf/epoxy composite

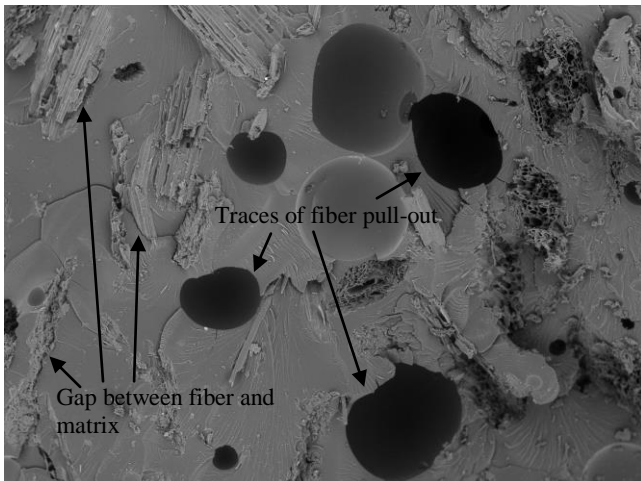


Fig. 6: Fracture surface of untreated kenaf/epoxy composite

Highest increment (71%) of flexural strength for 300  $\mu\text{m}$  kenaf/epoxy composite is believed due to the different type of loading to cause failure [11]. For tensile test the composite specimen is subjected to purely tensile load, meanwhile in flexural test the specimen was subjected to both tensile and compressive stress. Fiber is only good at taking a tensile load, hence if composite is subjected to compressive load it will be borne by the matrix only and fiber treatment has no effect on the compressive strength.

### 3.4. Tensile and Flexural Moduli

Tensile modulus of untreated and treated kenaf/epoxy at various soaking times are shown in Fig. 7. Fiber treatment showed inconsistent effect on the tensile modulus of kenaf/epoxy composite. For larger kenaf size (500  $\mu\text{m}$ ) the tensile modulus has slightly decreased to 13% - 24%. This trend is also related to the insufficient of NaOH concentration, hence unable to clean or remove all the impurities and pectin attached to the kenaf fibres as mentioned in section 3.1. Despite the decrease of tensile modulus of 500  $\mu\text{m}$  kenaf/epoxy composite, the smaller kenaf size (300  $\mu\text{m}$ ) has the highest tensile modulus improvement of 95% at 24 hour treatment. Again this is the evidence of good bonding between fibre and matrix, where both kenaf and epoxy able to elongate at the same rate up to the higher applied load.

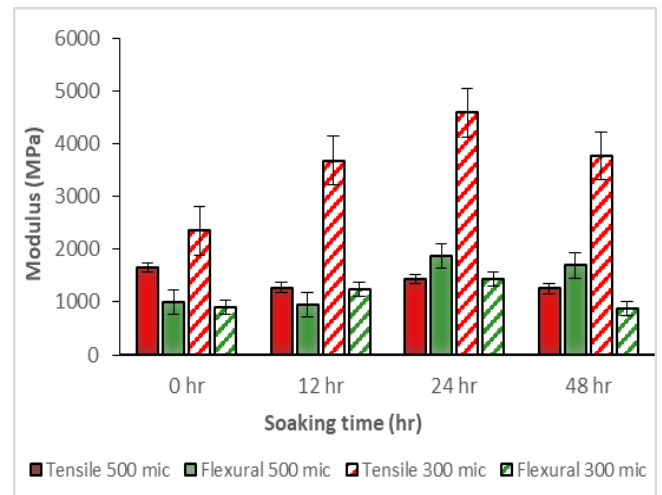


Fig. 7: Tensile and Flexural Moduli of composites

Fig. 7 has also showed the improvement of flexural modulus for both kenaf sizes. The flexural modulus of 500  $\mu\text{m}$  and 300  $\mu\text{m}$  kenaf/epoxy composite has increased by 87% and 60% respectively, which both occurred at 24 hours soaking time. These evidences has further supported that 24 hours is the best soaking time.

### 3.5. Fracture Toughness At Different Soaking Hours

Load-displacement curves shown in Fig. 8 are used to determine the maximum load to calculate the fracture toughness of composites filled with 150 – 300  $\mu\text{m}$  kenaf fiber. The curves indicate the behaviour of brittle material that is suitable to be characterised using linear elastic fracture mechanic (LEFM). The calculated fracture toughness,  $K_{IC}$  of the composites is as shown in Fig. 9. The trend of fracture toughness also followed the trend of their strength and modulus, where the treatment has successfully enhanced most of the properties, this trend was also observed by other researcher [2, 5]. The highest fracture toughness at 24 hour kenaf fiber treatment indicates its high capability to restrain the crack propagation as compared to untreated and other soaking times investigated. Again, it is related to the good bonding between fiber and matrix hence less gap between them or termed (identified) as cracks before it propagate to cause the failure.

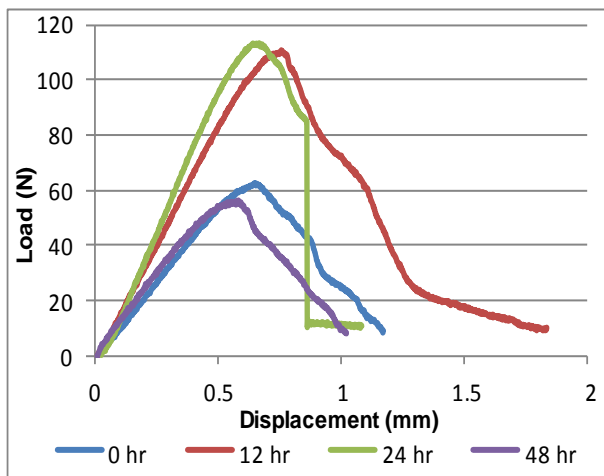


Fig. 8: Load-displacement curves of composites

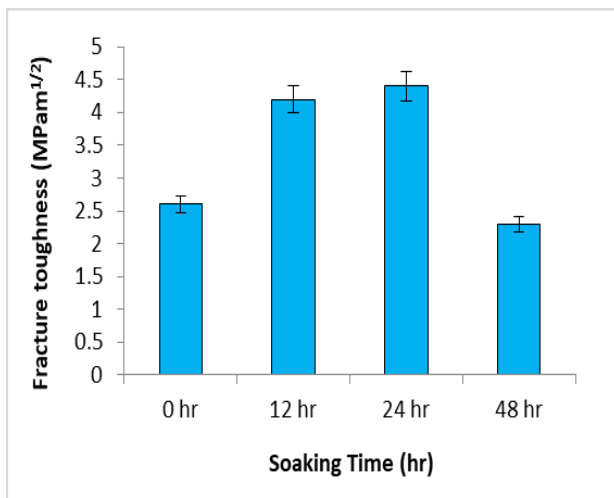


Fig. 9: Fracture Toughness and Energy absorbing capability of composites

#### 4. Conclusion

Soaking time of Sodium Hydroxide (NaOH) treatment has a significant effect towards the mechanical properties of kenaf/epoxy composite. The lower soaking time of alkali treatment does not give superior effect in order to clean the fiber surface and removing all impurities. But, the higher the soaking time caused the detrimental effect on the fiber surface and consequently on their mechanical properties. However, this investigation showed that 24 hours is the best soaking time for 6% NaOH concentration for both kenaf size range 150 – 300  $\mu\text{m}$  and 350 - 500  $\mu\text{m}$ . The highest improvement up to 59% and 71% of tensile and flexural strengths was achieved by 150 - 300  $\mu\text{m}$  kenaf/epoxy composite, meanwhile 95% and 60% improvement for tensile modulus and flexural modulus for the same kenaf size. The fracture toughness has also showed enhancement of 69% upon 24 hours soaking time. For further investigation it is suggested to use more than 6% NaOH concentration for the larger kenaf size.

#### Acknowledgement

This research was supported by a grant 600-IRMI/DANA 5/3/ARAS (0207/2016) from Universiti Teknologi MARA (UiTM) and managed by the Research Management Institute (RMI). The author would like to acknowledge the Centre of Advanced Material and Research (CAMAR), Fakulti Kejuruteraan Mekanikal UiTM for providing the facilities.

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