

Mechanical Characterization of Epoxy Filled with Seed Shells as Reinforcement



Anizah Kalam, Fauziah Md. Yusof, Zahurin Halim, Zuraida Ahmad

Abstract: *The use of natural fiber composite has been widely promoted in many industries such as construction, automotive and even aerospace. Natural fibers can be extracted from plants that are abundantly available in the form of waste such as sunflower seed shells (SSS) and groundnut shells (GNS). These fibers were chosen as the reinforcement in epoxy to form composites. The performance of composites was evaluated following the ASTM D3039 and ASTM D790 for tensile and flexural tests respectively. Eight types of composites were prepared using SSS and GNS fibers as reinforcement and epoxy as the matrix with the fiber content of 20wt %. The fibers were untreated and treated with Sodium Hydroxide (NaOH) at various concentrations (6%, 10%, 15%, and 20%) and soaking time (24, 48 and 72 hours). The treatment has successfully enhanced the mechanical properties of both composites, namely SSS/epoxy and GNS/epoxy composites. The SSS/epoxy composite has the best mechanical properties when the fibers were treated for 48 hours using 6% of NaOH that produced 22 MPa and 13 MPa of tensile and flexural strength respectively. Meanwhile, the treatment on groundnut shells with 10% sodium Hydroxide for 24 hours has increased the Flexural strength tremendously (53%), however no significant effect on the tensile strength. The same trend was also observed on the tensile and flexural modulus. The increase of 41% in flexural modulus after treatment with 10% NaOH for 24 hours was also the evidence of mechanical properties enhancement. The evidence of improved fiber and matrix bonding after fiber treatment was also observed using a scanning electron microscope (SEM). The SSS/epoxy composites performed better in tensile application, meanwhile the GNS/epoxy composites are good in flexural application.*

Keywords : Alkali treatment, Flexural test, Groundnut shell, Natural fibers, Sunflower shell, Tensile test.

I. INTRODUCTION

The use of natural fiber composite has been widely

promoted in many industries such as construction, automotive and even aerospace. Usage of natural fibers as reinforcement will reduce the dependence on synthetic fibers such as glass and carbon, which have many disadvantages even though the usage of it is commonly and widely utilized. Conventional synthetic fibers are not environmentally friendly, leading to problems of waste glass and carbon fibers, which cannot be decomposed by nature [1]. Substitute the conventional synthetic reinforcements into natural fibers (kenaf, coconut and rice) occurred due to the many advantages that are available in natural fibers such as environmentally friendly, low cost, low density, flexibility of usage and biodegradability [2], lighter weight, do not cause skin irritation, high strength-to-weight ratio, high stiffness-to-weight ratio, elimination of corrosion, available in the form of wastes and non-toxicity [3].

The development of natural fiber composites is also much convenient since the elements needed in producing the composite materials is basically a natural product that can be obtained through nature and also agriculture wastes [1]. Hence, in terms of cost, the manufacturing of natural composite materials can be a strong point in the transition from conventional synthetic fiber to natural fiber. However, with all the advantages of using natural fibers, there are drawbacks and disadvantages in every usage. Natural fibers have irregular dimensions, stiffness, susceptibility to heat, ease of water absorption and easily deteriorate [3]. Weak bindings between natural fiber and polymer due to its hydrophilic nature that attracted to the water molecule and tend to dissolve in the water molecules are always the main drawbacks of natural fiber composites. This hydrophilic character of natural fiber which leads to high water absorption, subsequently resulting in poor mechanical properties and dimensional stability of the natural fiber reinforced composites [4]. Besides that, the natural fiber structure consists of cellulose, hemicelluloses, lignin, pectin and waxy substances [5] that permit moisture absorption from fiber's surrounding that causes weak binding between polymer and fiber. Fiber treatment is one of the popular method used to eliminate lignin, pectin and waxy substances in order to improve the bonding between fiber and polymer and subsequently enhanced the mechanical properties of the composites.

Sunflower seed and groundnut shells are part of agricultural wastes that globally produced at about 14 million tonnes in 2014 and keep on increasing [6]. Hence, it is very interesting to use those seed shells as the reinforcement in the polymer matrix composite.

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In this research, sunflower seed shells, groundnut shells and epoxy were used to fabricate the composites with the aim of exploring the potential of sunflower seed shells and groundnut shells as reinforcement for epoxy matrix composite. Therefore, it is necessary to investigate the mechanical and physical properties of the composites with randomly oriented fiber and how fiber treatment can improve their mechanical properties.

II. MATERIALS AND EXPERIMENTAL PROCEDURE

A. Materials

Sunflower seed shells and Groundnut shells (Fig.1) were purchased from the local market. The shells were then crushed to reduce their sizes and sieved using 7 mesh strainer to obtain uniform sizes of short fibers in the range of 2 - 3 mm.

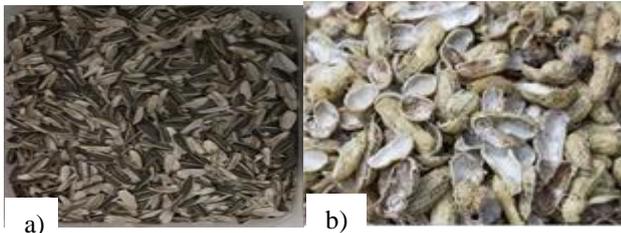


Fig. 1: a) Sunflower seed shells and b) Groundnut shells

Epoxy and hardener were purchased from Morstrong Industries Sdn. Bhd. The mixing ratio recommended by the supplier is 1:2.8 of hardener and epoxy. Sodium Hydroxide (NaOH) was manufactured by Sigma Aldrich Inc.

B. Specimen Preparation

For fiber surface treatment, Sodium Hydroxide (NaOH) was used as the alkaline in the mercerization treatment. The Sodium Hydroxide was diluted in 1000 milliliters of distilled water to form the solution that was used to soak the fibers. The details of NaOH concentration and soaking time are as tabulated in Table I.

Table I: Fiber treatment conditions

Fiber	Soaking time (Hours)	Concentration of NaOH (%)
Sunflower seed shells (SSS)	Untreated (UT)	
	24	0
	48	6
	72	6
Groundnut shells (GNS)	UT	
		10
	24	15
		20

The soaked short fibers were then washed and rinsed before drying process using an oven at 85°C for eight hours. Dried short fibers were then weighted based on the mass required to create a composite panel with 20wt % of fiber content. The weighted fibers were mixed with epoxy and hardener and poured into the steel plate mold to produce a 250 mm X 250 mm X 5 mm composite panel. Specimens for mechanical testing were cut from this panel.

C. Mechanical Testing

The Tensile tests were performed on flat composite specimens with the length of (250 mm), width (30 mm) and thickness (5 mm). The test was performed in accordance with

ASTM D3039 standard method of testing. The Flexural tests were performed on composite specimens with a length of (60 mm), width (10 mm) and thickness (5 mm). The test was performed according to ASTM D790. All the tests were conducted at room temperature and speed rate of 2 mm/min.

III. RESULTS AND DISCUSSION

A. Sunflower Seed Shell/Epoxy Composite

The effect of soaking time during treatment of SSS/epoxy composites on the tensile and flexural strength is as shown in Fig. 2. Both tensile and flexural strengths show the same trend of increasing strength upon the treatment, indicating the effectiveness of 6% of sodium hydroxide treatment on the fibers. The vast increase of tensile and flexural strengths (21% and 39%) was observed at 48 hours and beyond this soaking hours seems detrimental to the fibers indicating by the decreases of both strengths.

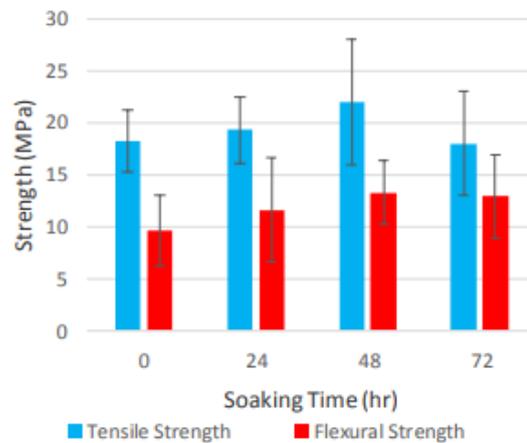


Fig. 2: Tensile and Flexural Strengths of SSS/epoxy composites

Tensile strength also showed more than 40% higher as compared to their respective flexural strength. The treatment of sunflower seed shell fibers has successfully improved the bonding between the fiber and matrix as can be seen that the gap between the fiber and matrix presence in Fig. 3 (indicated by the black arrows) is no longer be observed in Fig. 4.

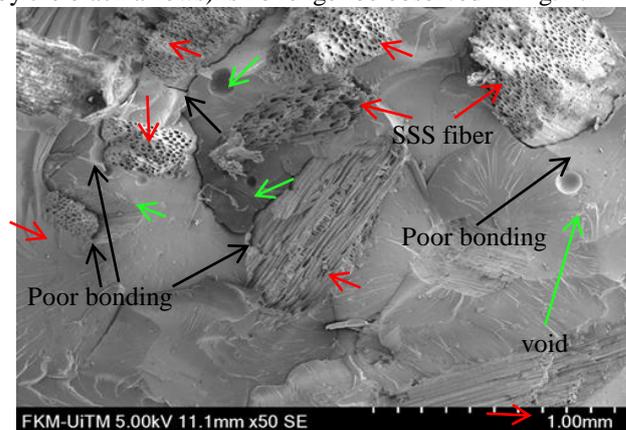


Fig. 3: Fracture surface of Untreated SSS/epoxy Composite



Poor bonding is the location of stress concentration since the stress was not able to be transferred from the matrix to the fiber (red arrows) that is supposedly the main stress carrier. Thereby, when the local stress exceeding the ultimate tensile strength of the matrix, a crack will be initiated and propagates as the load increases during loading, subsequently lead to failure. Hence the good bonding between fiber and matrix is the main factor to enhance the mechanical properties of composite materials, so that the stress can be effectively transferred to the fiber to share the load, as indicated by the increases of both tensile and flexural strengths after 24 hours treatment and further increase as the soaking time was increased to 48 hours.

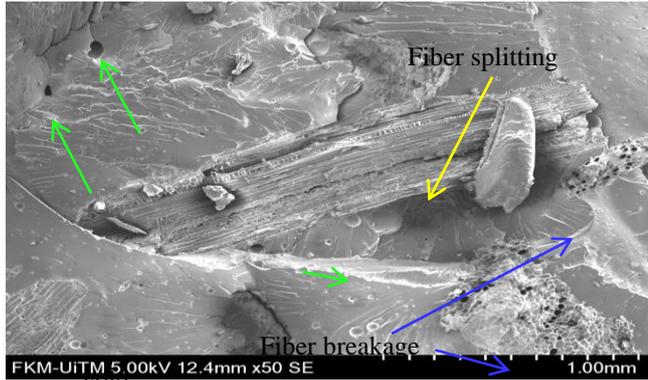


Fig. 4: Fracture surface of 72 hours treated SSS/epoxy Composite

Sodium hydroxide is supposed to remove lignin, waxy substances and impurities on the fiber surface during treatment, hence sufficient soaking time is needed to ensure the fibers are cleaned completely. As can be seen in Fig. 5 a) untreated SSS fibers has impurities on its surface and after treatment (Fig. 5 b) the fibrous structure was exposed. This led to better interfacial bonding between matrix and fibers as the impurities on the fiber surface were removed, the matrix 'glued' with the fiber better.

However, once the fibers completely cleaned (lignin, waxy substances and impurities all removed) if the soaking time is prolonged the sodium hydroxide will attack the cellulose which in turn will damage the fibers. This has explained why at 72 hours soaking time both tensile and flexural strengths start to decrease. The longer the soaking time during fiber surface treatment, will also make the fiber rigid and somewhat brittle afterward can develop crystallinity and this result has a similarity with [7] and [8]. Upon stress application, these fibers were suffered from early fiber breakage (as seen in Fig. 4) due to the brittleness even though the stresses were transferred effectively at the interface hence, lowered the tensile strength. Many traces of air bubbles (voids) were observed on the fracture surface of composite that contained treated fibers at a longer time (72 hours), these voids also served as stress concentration point similar to the gap between fiber and matrix for untreated fiber.

On the other hand, both moduli, tensile and flexural modulus were not much affected by the soaking time as the trend displayed in Fig. 6. The figure has also displayed the more consistent of tensile modulus as compared to the flexural modulus that has higher error bars. This trend indicated that as the stress increase the strain has also increased at the same rate, this might be due to the presence of

many small voids due to the fiber treatments as seen in Fig. 4 (indicated by green arrows).

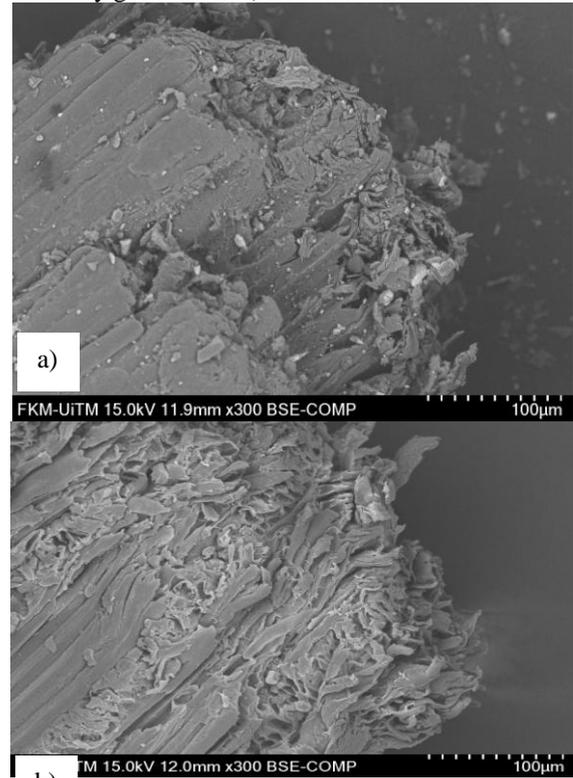


Fig. inflower seed shells a) Untreated b) treated for 45 hours

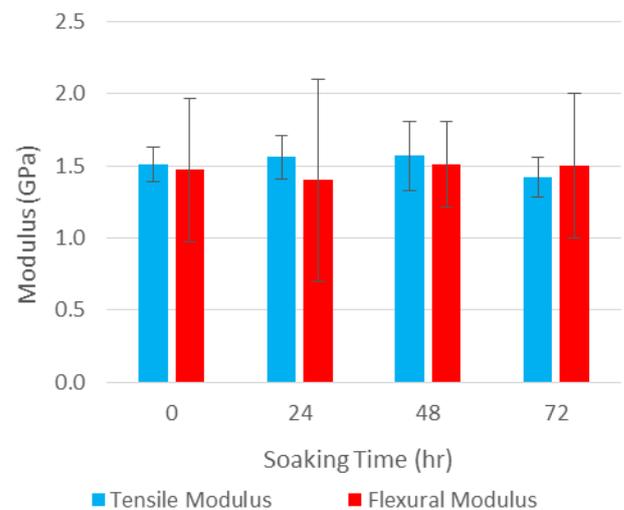


Fig. 6: Tensile and Flexural Modulus of SSS/epoxy composites

B. Groundnut Shell/Epoxy Composites

The results of the tensile and flexural strengths at various NaOH concentrations for GNS/epoxy composite is shown in Fig. 7. The treatment on groundnut shells with 10% Sodium Hydroxide in 24 hours has increased the flexural strength tremendously (53%), however no significant effect on the tensile strength.

Further increase of NaOH concentration beyond 10% seems to have a negative impact on the tensile strength. This is due to the excess of NaOH concentration tend to damage the cellulose if the lignin, waxy substances and impurities have been successfully removed.

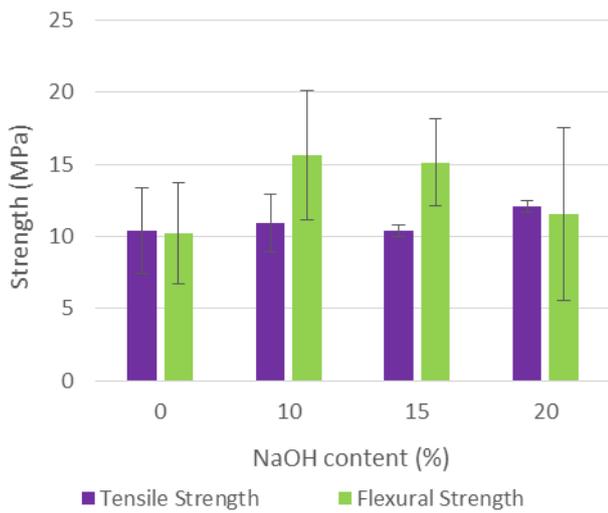


Fig. 7: Tensile and Flexural strength of GNS/Epoxy Composites

Normally, the tensile strength is expected to increase as the concentration of NaOH solution increases [8, 9], the same trend shows by flexural strength. However, the result did not agree with this due to the small aspect ratio of fiber and fiber misalignment. As the fibers become less aligned with the principal stresses, more shear stress is carried than the tensile stress. This explains why flexural strength has higher strength as compared to the tensile strength [10]. Flexural strength has also more contribution from shear stress in the compression area of the specimens.

Similarly for the tensile and flexural modulus, the effect of fiber treatment more prominent on the flexural modulus, where, at 10% concentration of NaOH solution has increased the flexural modulus by 41% from the untreated GNS fiber, as shown in Fig. 8.

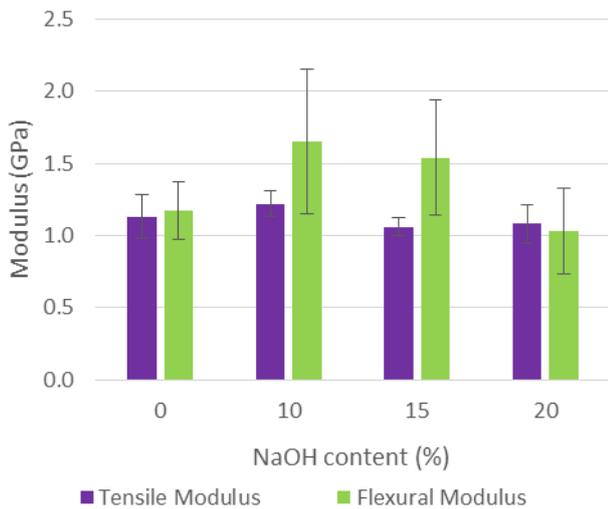


Fig.8: Tensile and flexural modulus of GNS/epoxy Composite

Beyond 10% of NaOH concentration used for fiber treatment has also decreased the flexural modulus of GNS/epoxy composites. The fracture surface of untreated GNS/epoxy composite as shown in Fig. 9 showed some traces of fiber pull out (indicated by yellow arrows), which indicates poor bonding between fiber and matrix. Besides that, some voids were also displayed where those features explained the lower strength of untreated GNS/epoxy composites similar to

what has occurred on SSS/epoxy composites as explained before.

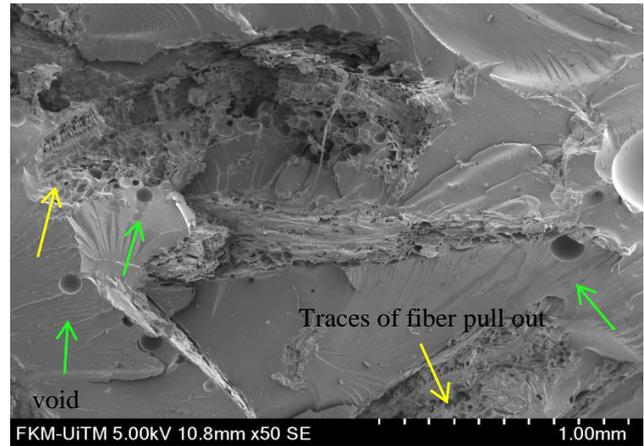


Fig. 9: Fracture surface of untreated GNS/epoxy composite.

On the other hand the fracture surface of treated GNS/epoxy composite with 20% NaOH concentration as displayed in Fig. 10 showed some small voids and fiber breakage which is similar to the characteristic of the fracture surface of SSS/epoxy composites that was being treated for 72 hours using 6% NaOH (seen in Fig. 4).

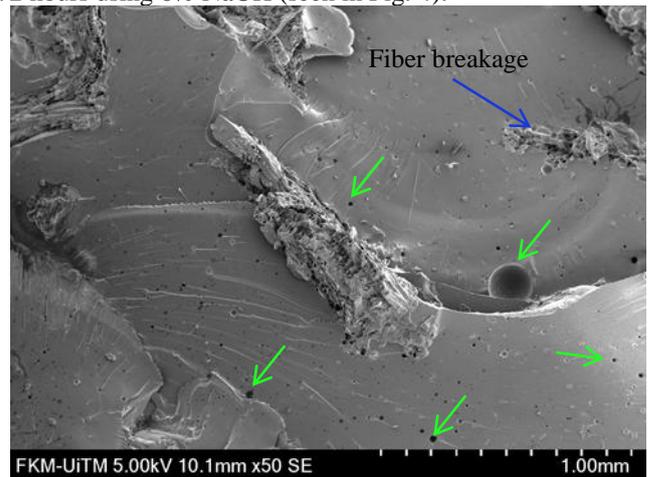


Fig. 10: Fracture surface of treated GNS/epoxy composite using 20% NaOH.

C. Comparison of SSS/Epoxy and GNS/Epoxy Composites

A Comparison of the tensile strength between SSS/epoxy and GNS/epoxy composites can be seen in Fig. 11. Generally, untreated SSS/epoxy composites have a higher tensile strength of 75% as compared to untreated GNS/epoxy composites. This is believed due to the larger aspect ratio of sunflower seed shells as compared to groundnut shells, hence increases the ability of stress transfer during loading [11]. The same behavior was also observed upon NaOH treatment with 24 hours soaking time. The fibrous shape of sunflower seed shells (SSS) fibers (Fig. 12 a)) as compared to groundnut seed shells (GNS) fibers that tend to have an irregular shape (Fig. 12 b)) also plays an important role in having higher tensile strength, as the fibrous shape has a higher aspect ratio.



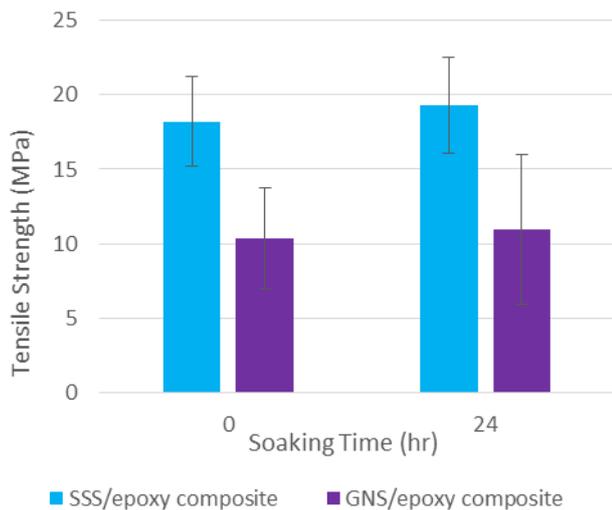


Fig. 11: Tensile strength of SSS/epoxy and GNS/epoxy composites

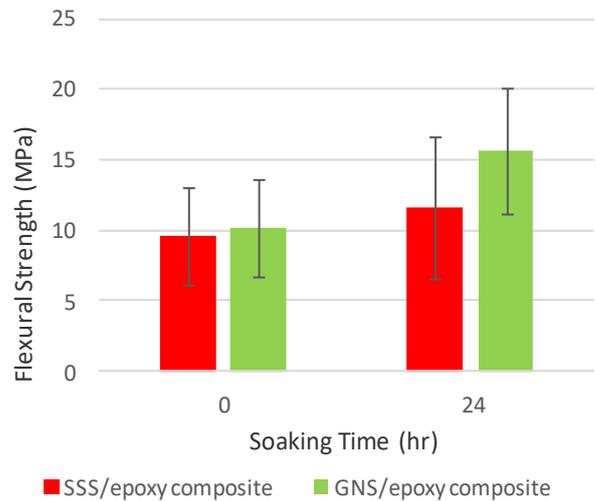


Fig. 13: Flexural strength of SSS/epoxy and GNS/epoxy composites

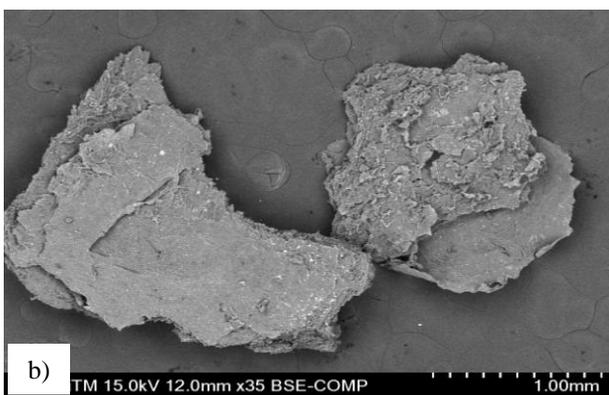
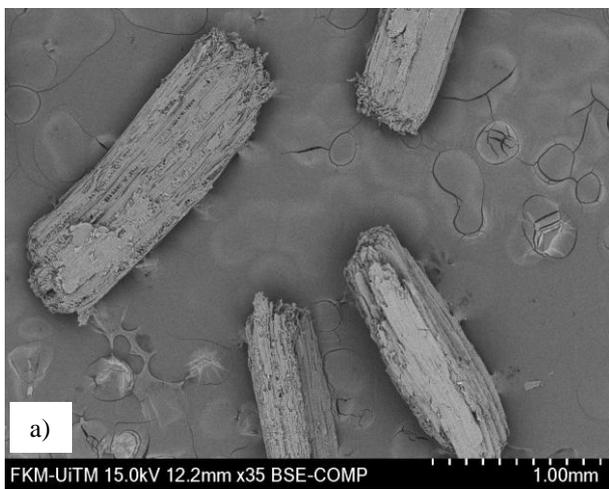


Fig. 12: Seed shell fibers a) Sunflower (SSS) and b) Groundnut (GNS)

Despite the low tensile strength, GNS/epoxy composites showed better flexural strength which is 35% higher compared to the flexural strength of SSS/epoxy composites as shown in Fig. 13. The differences are mainly contributed by the compression and shear effect during flexural loading whereas no compression effect during tensile loading. As mentioned before the irregular shape of GNS fiber tend to resist shear stress better.

Meanwhile, a comparison on the tensile modulus of both composite shows (Fig. 14) that SSS/epoxy composites have a higher tensile modulus of about 34% for both untreated and upon 24 hours treatment as well.

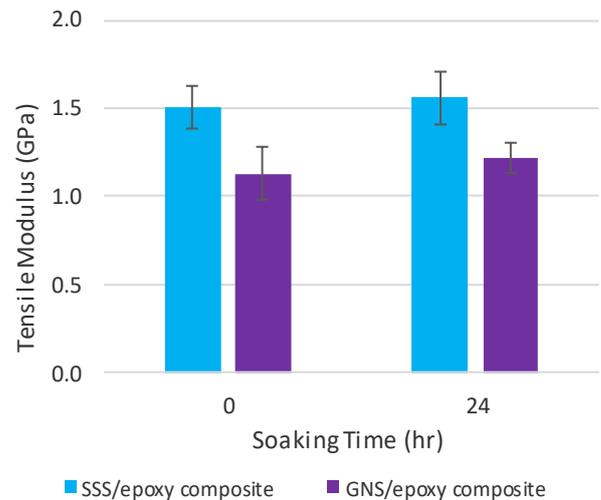


Fig. 14: Tensile Moduli of SSS/epoxy and GNS/epoxy composites

On the contrary the flexural modulus of untreated GNS/epoxy composites was 34% lower compared to the untreated SSS/epoxy composites (Fig. 15), however, upon treatment tremendous increase on flexural modulus of GNS/epoxy composites was observed surpassing (12% higher) the flexural modulus of SSS/epoxy composites. Meanwhile, no significant changes for the flexural modulus of SSS/epoxy composite.

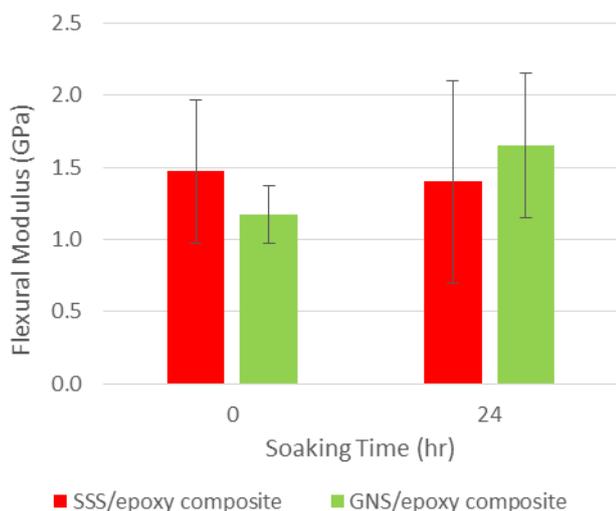


Fig. 15: Flexural Moduli of SSS/epoxy and GNS/epoxy composites

IV. CONCLUSION

Fiber treatment using Sodium Hydroxide has successfully enhanced the mechanical properties of both composites, namely SSS/epoxy and GNS/epoxy composites. The SSS/epoxy composite has the best mechanical properties when the fibers were treated for 48 hours using 6% of NaOH that produced 22 MPa and 13 MPa of tensile and flexural strength respectively the increase of about 21% and 39% as compared to the untreated SSS/epoxy composite. Meanwhile, the treatment on groundnut shells with 10% sodium Hydroxide for 24 hours has increased the Flexural strength tremendously (53%), however no significant effect on the tensile strength. The same trend was also observed on the tensile and flexural modulus of GNS/epoxy composites. The flexural modulus has increased of about 41% after treatment with 10% NaOH for 24 hours. Finally, the SSS/epoxy composites performed better in tensile application, meanwhile the GNS/epoxy composites are good in flexural application.

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