NaOH treated Kenaf/Glass hybrid composite: The effects of nanosilica on longitudinal and transverse tensile properties

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Abstract: Nanocomposite has been one of recent study interest among researcher, scientists, and students. With public consciousness about environment, there is a change in using environmental friendly’s materials. With the incorporation of nanotechnology, the strength of the composite shouldn't be sacrificed because of the use of natural fibre. Kenaf has been studied extensively and has been one of the candidates to be used in composite and with hybridizing with glass fibre. The strength of the composite could be retained to be used widely. In this study, nanosilica was impregnated in hybrid kenaf and glass epoxy composite to and was tested for its longitudinal and transverse tensile properties. From this study, incorporation of nanosilica in hybrid kenaf and glass epoxy composite help to increase its tensile modulus up to 34.52%, and longitudinal tensile strength up to 13.64%.

Keywords: Natural fibre, hybrid composites, nanocomposites

1. Introduction

The recent plummet of public concern about environment has contributed to decreasing plastic usage. One of the ways to support this effort is by introducing alternatives to reduce or replace plastic usability. Emerging usage of natural fibre in polymer composite is one of the trends towards that objective[1].

History had shown that kenaf had been used in the manufacture of rope, sackcloth, and twine. Nowadays, the kenaf usage has transcended the traditional stereotypical application into a more specialized and commercial usage like paper production, animal feeds, manufacture of and oil spill absorbent agent, medicine, food additive, a platform for mushroom farming, environmental cleaning, oil and chemical absorbents [2]. Kenaf plants have been extensively exploited over the past years among many different types of natural resources and envisioned as one of an alternative substitute to replace conventional materials or synthetic fibres as reinforcement in composites. However, one of natural fibre concern in implementing it in polymer composite is weak adhesion between natural fibre and synthetics matrix. Modification of natural fibre surface such as mercerization, help to enhance its ability to bond with the matrix and to break the hydroxyl group in the cellulose [3][4].

As of today, many application using polymers are unable to be replaced entirely because of its mechanical strength. Therefore, hybridization between natural fibres and synthetics fibre is a need to widen the application of natural fibre to be used in polymer composite.

Composites play a significant role in engineering material, and their use has been increasing day by
day due to their specific properties such as high strength to weight ratios, high modulus to weight ratio, corrosion resistance, and wear resistance. Several attempts have been made to hybridize kenaf fibre material using synthetic (glass) [5][6]. With the advancement in nanotechnology, nanofibre has been introduced in the polymer composite system [7][8]. The concept of nanocomposites started to emerge, offering the industry with promising results and a unique level of property improvement. This method involves the incorporation of nano-sized organic and inorganic nanoparticles to the epoxy matrix [9]. In a study conducted by Jumahat A. Et al. report that nanocomposites offer higher tensile stiffness and strength when compared to the neat polymer without sacrificing the failure strain of the material [10]. Other study conducted by Sapiai N. et al. conclude that the effect of carbon nanotubes (CNTs) on flexural and tensile properties of kenaf/glass fibres hybrid reinforced epoxy composites. The flexural strength decreased with increasing in CNTs content. For tensile properties, tensile strength decreased for 0.5 weight percentage (wt%) and 0.75 wt% CNTs content but increased for 1.0 CNTs content and increasing of flexural and tensile modulus increase with increasing in CNTs content [11].

In this study, nanosilica was being introduced in different percentage (5, 13 and 21 weight percentage) in hybrid longitudinal kenaf and glass fibre epoxy composite to compare the effects of nanofiller in this composite system in terms of its longitudinal and transverse tensile properties.

2. Methodology

Materials used in this study includes epoxy polymer, kenaf fibre, sodium hydroxide pellet (NaOH), nanosilica and glass fibre. Polymer matrix used was an epoxy resin consisting of Miracast 1517 A/B, which is a multipurpose DGEBA epoxy resin cured at room temperature is shown in Figure 1(a). The hardener used for this type of adhesive is amine curing agent. The ratio of epoxy and hardener used is 100:30. Kenaf fibre used was supplied by Innovative Pultrusion Sdn. Bhd. in yarn form is illustrated in Figure 1(b). Sodium hydroxide in pellet form was used from R & M chemicals as shown in Figure 1(c). The unidirectional glass fibre was supplied by China National Building Materials (Group) Corporation; Composite Division; N196 is an E glass direct roving shown in Figure 1(d). Nanosilica F400 provided by Evonik was used in this study using 3 different weight percentage which was 5, 13 and 25 weight percent and illustrated in Figure 1(e). Nanosilica sol-gel in F400 containing 40 weight percent of nanosilica in epoxy.

Figure 1. (a) epoxy polymer ; (b) kenaf yarn ; (c) sodium hydroxide pellet ; (d) longitudinal glass fibre ; (e) nanosilica.

First, As received kenaf yarn was cut into suitable length and tied using cable ties to ease soaking process. The process begins with soaking the yarn into distilled water at temperature of 700°C for 2 hours. This process aims to clean kenaf yarn from impurities that may trap between the fibre yarn. The fibre was then oven-dried at temperature of 105°C for 12 hours before being soaked into to 7 weight concentration of sodium hydroxide (NaOH).

The solution was prepared by adding different weight percentage of sodium hydroxide pellet in
distilled water and stirred using the magnetic stirrer to expedite the dissolving process. The precaution to handle this solution should be taken into account since this process is exothermic. Kenaf bundle was then soaked in the solution for 2 hours at room temperature and washed using distilled water with drops of acetic acid several times to neutralize back the pH of the fibre. Then the fibres were left for two days to dry at room temperature before once again oven-dried at 800°C for another 6 hours to make sure the fibres are completely dry.

The untreated kenaf (kenaf pure) and Sodium Hydroxide treated kenaf fibre (kenaf 7 wt%) were then examined under Fourier Transform Infrared spectroscopy (FTIR) used to determine the organic bonding within the fibre from transmittance (%) versus wavenumber (cm⁻¹) graph. The peak of the chart is observed to determine the presence and disappearance of the bonding. The peak indicates the presence of bonding and disappearance of the peak suggest that the disappearance of the bonding. Based on this result, the best kenaf fibre was proceeded to be impregnated with epoxy to produce hybrid composite.

The selected kenaf yarns were then wounded at the aluminum frame before being wound again by unidirectional glass fibre. Then, epoxy and nanosilica gel with different weight percentage of nanosilica (0, 5, 13, 25 weight percentage) were weigh using scale before vacuumed using vacuum casting system for one hour and half to minimize the entrapped bubble in the epoxy. After that, amine hardener was added in the epoxy, and the mixture was stirred and vacuumed for 10 minutes to release the entrapped bubble caused by mixing. The epoxy mixture then being poured onto wounded kenaf fibre placed above the A3 size PTFE film.

The composites then being cut into ASTM D3039M and post cured at elevated temperature as per suggest by epoxy manufacturer.

The composite system in this study can be simplified as Table 1 below:

| Composite system                                                                                                                                 |
|--------------------------------------------------------------------------------------------------------------------------------------------------|
| 7 weight percentage of sodium hydroxide treated Kenaf hybrid glass composite (K7/G)                                                             |
| 5 weight percentage nanosilica impregnated in 7 weight percentage sodium hydroxide treated Kenaf hybrid glass composite (K7/G/5)                |
| 13 weight percentage nanosilica impregnated in 7 weight percentage sodium hydroxide treated Kenaf hybrid glass composite (K7/G/13)              |
| 25 weight percentage nanosilica impregnated in 7 weight percentage sodium hydroxide treated Kenaf hybrid glass composite (K7/G/25)              |

The tensile testing laboratory was conducted using an Instron Universal Tester machine and the BlueHill data acquisition software. It was conducted on unidirectional (UD) laminates to determine tensile properties such as tensile modulus, tensile strength, and tensile strain. Figure 2 shows a sample for transverse direction hybrid kenaf/glass fibre composite.

![Sample of transverse kenaf hybrid glass composite](image.png)
3. Result and discussion

The purpose of sodium hydroxide treatment is to eliminate lignin and hemicellulose. The removal of these components was proven to help to improve adhesion between polymer and natural fibre. The results of Fourier Transform Infrared Spectroscopy (FTIR) for NaOH treated and untreated kenaf fibres are shown in Figure 3 below. The absorbance peaks in 3,400cm⁻¹ to 3,300 cm⁻¹ region is attributed to the stretching and bending vibration, respectively, of the OH group of cellulose. The CH stretch at 2900cm⁻¹ and CH₂ symmetric bending peak at 1432cm⁻¹ were present in both treated and untreated kenaf fibre. The result shows the disappearance of peak number 1736 cm⁻¹ for treated kenaf fibre. It indicates that the fibre did not have C=O bonding. The disappearance of C=O bonding shows that the elimination of hemicellulose and lignin. It shows that alkali treatment is successful in removing lignin and hemicellulose. COOH bending peak also appears in both fibres at peak 664cm⁻¹.

Table 2 summarizes the longitudinal tensile modulus, strength, and strain at break for K7/G, K7/G/5, K7/G/13, and K7/G/25. Longitudinal tensile modulus of kenaf hybrid glass composite increases by 34.52% when added with 5 weight percentage (wt.%) of nanosilica in epoxy composite from 6.17GPa to 8.3GPa. The tensile modulus of the composite, however slightly drop by 1.08% when 13wt.% of nanosilica was added in the composite system. Nevertheless, the tensile modulus of 13wt.% nanosilica added composite was still increased by 33.06% compared without adding nanosilica in the composite system. It has the same pattern for K7/G/25, where tensile modulus was 7.21Gpa, dropped by 12.18% and 13.13% when compared to K7/G/13 and K7/G/5. Nonetheless, the longitudinal tensile modulus of K7/G/25 was still higher than K7/G by 16.86%. Therefore, from these results, it can be concluded that addition of nanosilica in kenaf glass hybrid composite helps to improve longitudinal tensile modulus.

Longitudinal Tensile strength of K7/G was 149.64Mpa. With the addition of 5% nanosilica in the hybrid composites, the tensile strength was insignificantly decreasing by 0.9%. Regardless of that, the tensile strength of K7/G/13 increase by 11.47% and 12.49% compared to K7/G and K7/G/5 respectively. The longitudinal tensile strength further increased by 13.64% compared to K7/G, which is 170.05Mpa. From the results, it can be inferred that additional of nanosilica in kenaf and glass hybrid composite assist in improving longitudinal tensile strength.

The longitudinal tensile strain at break for K7/G is 3.55%. With an addition of 5% nanosilica in the composite, the tensile strain decrease to 2.01%. In spite of that, the strain increase with the addition of 13% and 25% of nanosilica where it indicates that tensile strain at break of K7/G/13 and K7/G/25 was 2.4% and 2.68% respectively. It is shown that with addition of nanosilica, the hybrid composite
becomes more brittle.

Table 2. Longitudinal tensile modulus, strength, and strain at break for hybrid composites

| Material properties | K7/G | K7/G/5 | K7/G/13 | K7/G/25 |
|---------------------|------|--------|---------|---------|
| Tensile modulus, $E_{t,0}$ (GPa) | 6.17 | 8.3 | 8.21 | 7.21 |
| Tensile strength, $\sigma_{u,t,0}$ (Mpa) | 149.64 | 148.28 | 166.8 | 170.05 |
| Tensile strain break at, $\varepsilon_{f,t,0}$ (%) | 3.55 | 2.01 | 2.4 | 2.68 |

Table 3 summarizes the transverse tensile modulus, strength, and strain at break for K7/G, K7/G/5, K7/G/13, and K7/G/25. Tensile modulus of hybrid kenaf glass composites is slightly increasing from 3.965 GPa to 3.974Gpa for with the addition of 5 weight percentage of nanosilica where it increased by 0.23%.

Table 3. Transverse tensile modulus, strength, and strain at break for hybrid composites

| Material properties | K7/G | K7/G/5 | K7/G/13 | K7/G/25 |
|---------------------|------|--------|---------|---------|
| Tensile modulus, $E_{t,90}$ (Gpa) | 3.965 | 3.974 | 3.981 | 4.009 |
| Tensile strength, $\sigma_{u,t,90}$ (Mpa) | 12.63 | 10.30 | 10.29 | 10.22 |
| Tensile strain at break, $\varepsilon_{f,t,90}$ (%) | 0.65 | 0.48 | 0.41 | 0.37 |

The transverse tensile modulus further increases to 3.981GPa for K7/G/13 and 4.009GPa for K7/G/25 where it increased by 0.40% and 1.11% compared to K7/G respectively. It can be deduced that addition of nanosilica in hybrid kenaf glass composite, slightly increase transverse tensile modulus. Comparison between longitudinal and transverse tensile modulus of hybrid composite is shown in Figure 4.

Figure 4. Longitudinal and transverse tensile modulus K7/G, K7/G/5, K7/G/13 and K7/G/25

Figure 5. Longitudinal and transverse tensile for strength for K7/G, K7/G/5, K7/G/13 and K7/G/25

Tensile strength K7/G is 12.63MPa. The strength decreases to 10.30 MPa for K7/G/5 which is 18.43% lower than K7/G. The transverse tensile strength was further reduced with addition of 13% and 25% nanosilica where both of the K7/G/13 and K7/G/25 shown transverse tensile strength of 10.29Mpa and 10.22Mpa respectively. The transverse tensile strength was decreased by 18.52% and 19.08% respectively, compared to K7/G. It can be inferred that addition of nanosilica in hybrid composite decreased transverse tensile strength where a variety of factors will have a significant influence on the transverse strength; these factors include properties of both the fiber and matrix, the fiber-matrix bond strength, and the presence of voids. The agglomeration of the nanosilica maybe the cause of this occurrence where it hinder the further wetting of kenaf fibre and glass in epoxy matrix. Comparison between transverse tensile strength and longitudinal tensile strength of hybrid kenaf/glass...
composites are shown in Figure 5. From the graph, we can infer that the transverse tensile strength of hybrid composites are approximately 10% of its longitudinal tensile strength, which confirms that the tensile strength was mostly control by strength of the fibres.

The same trend can be seen for transverse tensile strain, where increasing weight percentage of nanosilica would decrease the tensile strain. The transverse tensile strain of K7/G is 0.65%. The transverse tensile strain drop by 26.04% for K7/G/5, which is 0.48%. The transverse tensile strain further dropped to 0.41% and 0.37% for K7/G/13 and K7/G/25 respectively. In which by percentage, 36.31% and 43.7% compared to K7/G. Therefore, it can be concluded that addition of nanosilica makes brittle hybrid composite. Figure 6 shows comparison between transverse and longitudinal tensile strain. From the results, it can be concluded that the composite is approximately five times more brittle at transverse direction of fibre compared to longitudinal direction of fibre.

From the result, it could be inferred that the addition of nanosilica improve the longitudinal tensile modulus, longitudinal tensile strength of kenaf glass hybrid composite and slightly increased its transverse tensile modulus. However, the addition of nanosilica in hybrid kenaf glass composite would decrease its transverse tensile modulus and make the composite more brittle.

4. Conclusion

Incorporation of nanosilica in kenaf/glass hybrid composite helps increasing its longitudinal tensile modulus and longitudinal tensile strength, however, for longitudinal tensile strain, impregnation of nanosilica decreased the longitudinal tensile strain which indicates that the composite is more brittle. When comparing to non-added nanosilica hybrid composite with the highest longitudinal tensile modulus and strength, the difference in longitudinal tensile modulus between K7/G and K7/G/5 which is 34.52%, while the difference longitudinal tensile strength between K7/G and K7/G/25 is 13.64%. For longitudinal tensile strain, the difference between K7/G and k7/G/5 dropped by 26.04%. It could be seen that with increasing longitudinal tensile modulus, the longitudinal tensile strain will decrease.

Impregnation of nanosilica in hybrid kenaf/glass composites help in increasing its transverse tensile modulus. However, in transverse tensile strength and strain, the incorporation has a negative impact. Therefore, it can be concluded in this study, with addition of nanosilica in kenaf hybrid glass composites, it helps to improve longitudinal and transverse tensile modulus, longitudinal tensile strength, however it decreases in terms of both longitudinal and transverse tensile strain.
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