

Effect of impregnation on hybrid mesoporous silica / kenaf reinforced epoxy composites in term of flexural, compressive and water absorption properties

F. Bajuri¹, N. Mazlan^{1,2*}, M.R. Ishak¹ and M.K.A. Uyup³

¹ Aerospace Engineering Department, Faculty of Engineering, Universiti Putra Malaysia, 43000 UPM Serdang, Selangor, Malaysia
Phone: +60397696403

² Institute of Advanced Technology (ITMA), Universiti Putra Malaysia, 43400, Serdang, Selangor, Malaysia

³ Forest Products Division, Forest Research Institute Malaysia (FRIM), 52109, Kepong, Kuala Lumpur, Selangor, Malaysia

ABSTRACT – In this work, mesoporous silica with designated amount was added in kenaf/epoxy composites to improve the mechanical properties of the composite and reduce the water uptake of fabricated composites. The composites were fabricated using hot press method (HP) and impregnation method (IMP). For HP specimens, silica was dispersed into epoxy resin using homogenizer before being applied to the kenaf mat and subsequently hot pressed. While for IMP specimens, the kenaf mat was placed inside the silica/epoxy solution under 600 mm Hg impregnation pressure before being hot pressed. The results for flexural properties revealed that IMP specimen with 40 vol% of kenaf fibre and 5 vol% of silica have the highest strength and modulus at 78.6 MPa and 5.11 GPa respectively. Same trend can be seen for compressive properties as the same specimen had the highest compressive strength and modulus at 69.3 MPa and 1.81 GPa respectively. Finally for water absorption properties, IMP specimens had a reduction in water uptake compared to its HP specimen counterparts with the same kenaf and silica content. IMP specimen with 60 vol% kenaf and 5 vol% silica had the lowest water uptake after 90 days of immersion in distilled water at 13.5% increase in weight.

ARTICLE HISTORY

Received: 22nd Sept 2019

Revised: 1st June 2020

Accepted: 29th June 2020

KEYWORDS

kenaf;
mesoporous silica;
hybrid composites;
natural fibre reinforced
composites

INTRODUCTION

Kenaf and other natural fibres likewise have been broadly studied as an environmentally susceptible fibre substitution of synthetic fibres to reinforce polymeric matrices [1, 2]. The attraction of natural fibres comes in many forms. Some of them are due to natural fibres' biodegradability, high toughness, reduced dermal and respiratory irritation, low costing compared to synthetic fibres, sustainability, light weight and non-abrasiveness [3–5]. The low density nature of the natural fibres contributes to its high specific strength. Non-abrasive nature of natural fibres makes them able to be handled easily while allowing high filling levels in composite [4, 5]. In the automotive industries, natural fibres have also gained attention [2, 6]. With the growing awareness of environmental issue, natural fibres are highly considered in many applications [7]. In comparison to other natural fibres, kenaf (*Hibiscus cannabinus* L.) has unique properties of being able to fixate CO₂ at a remarkably high rate [8]. It can absorb CO₂ for about 1.4 times its weight. It also has a high growth rate to in which the plant can grow up to 3 m in 3 months and able to mature in 5 to 6 months [9, 10]. The plant can be cultivated over a wide range of climatic conditions [11].

Natural fibres are hydrophilic by nature contributed by hydroxyl group of cellulose and hemicellulose from the fibre's structure [12]. Kenaf is a bast fibre with high cellulose content. High cellulose content with aligned cellulose microfibrils in the fibre's direction will contribute to better mechanical properties [13]. The problem with the usage of natural fibres to reinforce polymeric matrices is the incompatibility between the hydrophilic fibre and hydrophobic matrices. Larger gaps between fibre and matrix interface occur due to the incompatibility of the fibre and matrix leading to defective interfacial bonding [14]. Reduction of interfacial bonding between fibre and matrix will produce improper stress transfer between both materials which ultimately reduce the mechanical properties of the final product [5]. Apart from reducing mechanical properties, hydrophilic nature of the natural fibres contributes to the high moisture absorption of resulting product [15]. There are many techniques that have been applied to improve surface compatibility between fibres and matrices in the form of surface treatment such as alkalization [12], silane coupling agent treatment [13] or a combination of both [10]. Alkali treatment promotes the increase of surface roughness of the fibre which in turn will enhance mechanical bonding between the fibres and matrix while silane treatment improves the production of cross-linking in the interface area [10]. While the treatment improves the mechanical properties of the resulting composites, the overall materials are found to be costly.

Mesoporous silica can be derived from rice husk, byproduct of agricultural waste. It possesses a large specific surface area, large pore volume, low density, low dielectric constant, excellent heat insulation properties and high elastic modulus making it an interest in many researching fields [14, 16]. Mesoporous silica's high interface area of about 1500 m²/g have active function and can produce high interfacial interaction with resin by having a very small structure [14, 16]. These

properties provide mesoporous silica with the ability to enhance a composite's mechanical, physical and optical properties while providing resistance to the environmental stress caused by cracking and aging [17].

Water absorption in polymeric composites generally occurs due to capillary action. During the mixing process or fabrication procedure, micro bubbles will be produced. Micro bubbles located between polymeric chains provide spaces where water molecules can be diffused into which will increase the water uptake [16, 18, 19]. Basri et. al. reported that a high speed of mixing process with a low mixing time of silica nanoparticles into an epoxy resin system, produced specimens with the lowest water absorption rate [16]. When fibre and matrix have improper fibre wettability and poor fibre-matrix interaction, gaps will be opened in the composite system. Water molecules will fill the gaps and flaws in between the interfaces of fibres and matrix via capillary transport [16, 18, 19]. Also, when natural fibre absorbs water it will swell. Micro cracks in the matrix will be produced due to the swelling of the fibres or any other filler. This mostly happens in the case of natural fibre reinforced composites.

In this work, the objective is to improve the mechanical properties in term of flexural and compressive properties, and reduce the water absorption properties of mesoporous silica/kenaf reinforced epoxy composites. Like other natural fibres, kenaf possesses pores which aid the capillary movement of water molecule into the fibres. Thus, impregnation process was conducted to fill the pores with mesoporous silica infused epoxy to stop the movement of water molecules in the fibre's system. By removing the micro bubble interaction between matrix and fibre is promoted to improve the mechanical properties.

METHODS AND MATERIALS

Materials

The matrix used for this project is Epoxamite 100 with 103 SLOW Hardener, manufactured by Smooth-On. The resin to hardener ratio used was 100:28.4 by weight. This is as suggested by the manufacturer. The density of the epoxy is 1.10 g/cm³. The reinforcing fibre used to fabricate the composite is kenaf originated from Kelantan, Malaysia. ZKK Sdn. Bhd. provided the material in the form of randomly orientated mat. It was used as received. The density of kenaf mat used was 1.44 g/cm³. The kenaf mat was cut into 150 mm × 150 mm. The weight of the cut kenaf mat was different. Thus, during fabrication, the combination of kenaf mats with different weight were used to accommodate each type of specimen. Table 1 shows the number of kenaf mat used for each kenaf loading. Hydrophilic mesoporous silica provided by Maero Tech Sdn. Bhd. was used as a filler material. The silica was derived from rice husk. The average size of the particles was 324 nm using the particles size analyser. The bulk density of the silica used for this work is 0.21 g/cm³ per 5 g of the particles.

Table 1. Number of kenaf mat layer per each kenaf loading

Kenaf loading [vol%]	Number of kenaf mat
20	1
30	2
40	2
50	3
60	3

Composites Fabrication

For HP specimens, epoxy and silica were weighed and mixed together using Homogenizer (IKA T18 digital ULTRA-TURRAX) with mixing speed of 3000 rpm for 10 minutes. Air bubbles were produced as a result of homogenizing process. Thus, to remove the air bubbles, the mixture was put in a vacuum oven at room temperature and left there for approximately 30 min. Then the hardener is applied into the mixture and mixed manually until even while avoiding production of air bubbles. The epoxy mixture was applied to kenaf mat using hand-lay-up method, then the wet kenaf was placed inside a 150 mm × 150 mm × 3 mm waxed aluminium mould before being compressed using a 40 tonne hot press machine at 85 °C for 5 min. After removing the mould from the hot press machine it was then cold compressed for another 5 min before being removed and left at room temperature overnight. Table 2 lists the sample fabricated. Here 100 vol% means the combined volume of epoxy and kenaf. Mesoporous silica is an addition. 200HP and 201HP both contained the same amount of kenaf and epoxy (20 vol% kenaf and 80 vol% epoxy). 201HP contained an extra 1 vol% of mesoporous silica from the volume of kenaf and epoxy combined.

For IMP specimens, due to the need of submerging the fibres into the impregnating material, approximately 2.5 L of mesoporous silica particulates mixed with epoxy were prepared. The epoxy was hand mixed with silica before homogenizing at 5000 rpm for 20 min. The kenaf mats were placed inside a plastic container and the container was then placed under a bell jar. The pressure inside the bell jar was reduced to 600 mm Hg to remove the air that is trapped in the fibres for 5 min. A similar method of impregnation has been conducted using single fibre only [20]. Then the silica /epoxy mixture was infused until the fibres were fully submerged. After the fibres were fully submerged with resin, they were

left for 24 h. The impregnated fibres were compressed using a 40 tonne hot press machine under room temperature to remove excess resin, then weighed to calculate the amount of hardener needed. The hardener was applied onto the kenaf mat thoroughly, then the mat was placed in a waxed 150 mm × 150 mm × 3 mm aluminium mould before being hot pressed at 85 °C for 20 min. After removing the mould from the hot press machine it was then cold pressed for another 5 min before being removed and left at room temperature overnight. All specimens were post cured at 80 °C for 2 h in an oven. Post curing was conducted to promote cross linking between epoxy and hardener to reduce residual uncured epoxy.

Table 2. List of specimens fabricated

Specimen Name	Kenaf loading [vol%]	Mesoporous silica loading [vol%]	Fabrication Procedure
200HP	20	0	HP
300HP	30	0	HP
400HP	40	0	HP
500HP	50	0	HP
600HP	60	0	HP
201HP	20	1	HP
301HP	30	1	HP
401HP	40	1	HP
501HP	50	1	HP
601HP	60	1	HP
205HP	20	5	HP
305HP	30	5	HP
405HP	40	5	HP
505HP	50	5	HP
605HP	60	5	HP
405IMP	40	5	IMP
505IMP	50	5	IMP
605IMP	60	5	IMP

Flexural Test

Flexural test was done through a 3-point bending test under ASTM D790-03 using 5 kN INSTRON 3365 Universal Testing Machine equipped with 3-point bending crosshead. The size of the sample for the test is 125 mm × 12.7 mm [21]. The samples were cut into shape using a composite cutter. The flexural test was prepared by propping the sample on top of two support span and then adjusting the loading nose until it barely touched the midpoint of the sample. The distance of the support span was set at a ratio of 16:1 of the span-to-depth of the sample. The test begins with the loading nose moving downward perpendicular to the position of the sample at a constant rate measured using Eq. (1).

$$R = ZL^2/6d \quad (1)$$

Here, R is the loading rate in mm/min, Z is the straining rate, L is the support span and d is the depth of the specimen. A straining rate of 0.01 mm/mm/min was used for every specimen as suggested by the ASTM D790-03 [21]. For each composition, 5 specimens from two plates were tested. In total there were 10 specimens tested.

Morphological Analysis

Scanning Electron Microscopy (SEM) was conducted using Hitachi S-3400N Scanning Electron Microscope. The samples were obtained by cutting 10 mm from the fractured point of samples tested during the flexural test. Specimens were coated with gold before images were taken to avoid them from being charged under electron beam [8]. Surface morphology of the fractured surface was observed.

Compressive Test

The compressive tests were done in accordance to ASTM D695-02a using 5 kN INSTRON 3365 Universal Testing Machine [22]. The samples' length and width were 12.75 mm × 12.75 mm. A total of 5 samples per loading was tested. The loading rate used was 2 mm/min. The experiment was conducted by placing the specimen on top of the compressive jig with the thicker sections of the specimen lightly touching the jig before pressure is applied.

Water Absorption Test

The test was done in accordance to the ASTM D570-98 using a water bath [23]. Firstly, the samples were cut into the size 57 mm × 10 mm × 3 mm and dried at 40°C for 24 h before being weighed. The samples were then submerged into a glass container filled with distilled water and placed inside the water bath filled with normal tap water at the temperature of 30°C for 24 hours. For the first week, after 3, 6, 24, 48, 72 96 h the specimens were weighed. Starting from the next week, the specimens were weighed every 1-2 weeks and the distilled water replaced every 1-2 weeks until approximately 80 days. The percentage weight gain of each specimen was calculated using Eq. (2). Here, the percentage weight gain (M_i) is the percentage of weight difference between initial oven dried mass (M_d) and the mass at the interval measured (M_t).

$$M_i = \frac{M_t - M_d}{M_d} \times 100 \quad (2)$$

RESULTS

Flexural Test

Figure 1 illustrates the flexural strength of the fabricated specimens. An observation can be made where for HP composites, the increment of kenaf volume fraction resulted in the increment of flexural strength with the exceptions of 601HP and 605HP specimens where the specimens with the same silica loading and 50 vol% kenaf had a higher strength of 69 MPa for 501HP and 72 MPa for 505HP. Moreover, the inclusion of silica had a positive effect in increasing the flexural strength of the specimens. An exception can be seen for the specimens with 20 vol% kenaf. Specimen 201HP had lower strength of 43.2 MPa than the other two HP specimens with the same kenaf loading at 48.5 MPa and 48.7 MPa respectively to 200HP and 205HP. This occurrence may be caused by errors during fabrication. HP fabrication method is a manual fabrication technique which incorporates the fabricator skills. In this work, this includes, homogenizing silica into the epoxy, mixing of the hardener, spreading of resin onto kenaf fibre, compression of the specimens and sample conditioning to flexural test dimension manually using a band saw.

405IMP specimen had the highest flexural strength out of the hybrid silica/kenaf reinforced epoxy at 78.6 MPa. The production of voids is reduced significantly by soaking the fibre with silica infused epoxy under vacuum pressure resulting in composite with good adhesion to the resin. Out of the 3 IMP composites, 605IMP specimen had lower flexural strength than its counterpart HP specimens with 60 vol% kenaf at 63.7 MPa whereas other HP specimens with the same kenaf content had 66.2 MPa, 67.6 MPa and 69.0 MPa strength respective to 600HP, 601HP and 605HP. Although reduction in voids improved the strength to improper curing of the resin may contribute to a weaker composite. With impregnation process, instead of mixing the hardener with epoxy, the hardener is directly applied to the epoxy soaked mat. This procedure may cause a higher chance for the epoxy to not be fully cured due to the uneven spread of the hardener.

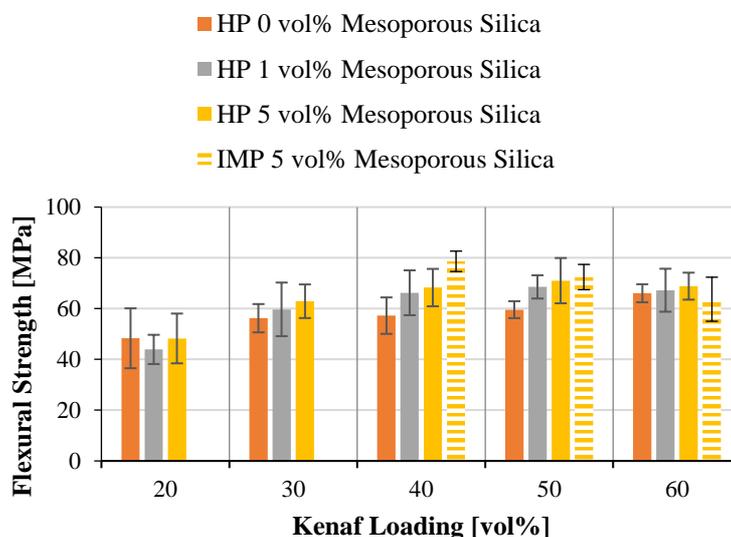


Figure 1. Flexural strength of fabricated specimens

For flexural modulus, similar trend can be seen with flexural strength as can be seen in Figure 2. However, for 505IMP and 605IMP specimen, although it had the highest flexural strength out of the hybrid silica/kenaf composites, the modulus is lower than other HP specimens with the same kenaf content. 405IMP composites retain its position with the highest value of flexural modulus at 5.11 GPa. Composites with high voids content will have lower water penetration resistance and poorer strength properties [24]. The main objective of conducting impregnation procedure is to stop the production of voids during composite fabrication. As a result IMP specimens had higher strength and modulus than other specimens. Also, as fibre content increase, the interfacial area of the fibres will increase while the reduction of matrix content will worsen the interfacial bonding between the fibres and matrix causing a decrease in the strength of the composites [25]. 405IMP being the highest in flexural strength and modulus is contributed by the combination of voids reduction due to impregnation process and, balance between fibre and matrix content that produces good interfacial bonding. For HP specimens, other than 201HP specimens, inclusion of silica improved the flexural modulus. 5 vol% generally had a better improvement in flexural modulus. Only 501HP, specimen with 1 vol% silica, at 4.23 GPa shows higher flexural modulus value compared to 5 vol% silica specimen 505HP at 4.18 GPa of the same kenaf content (50 vol%).

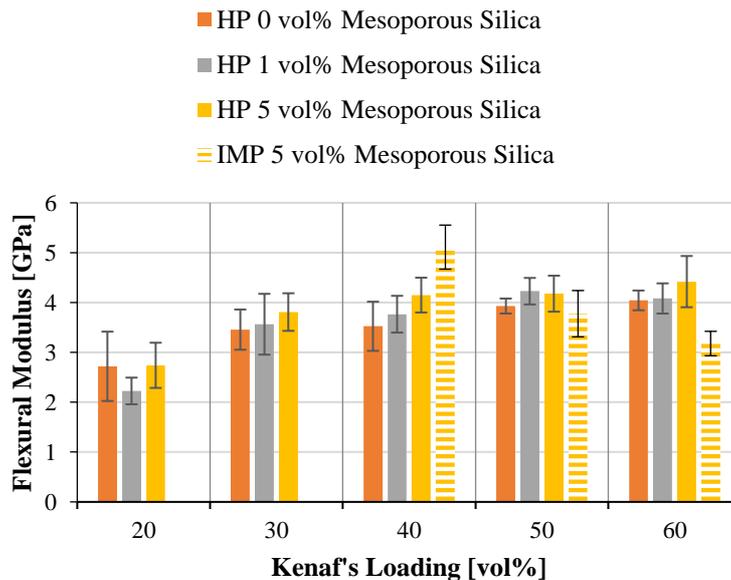
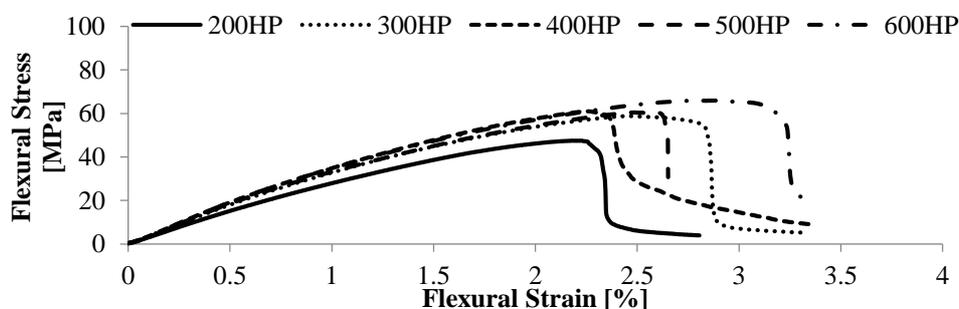


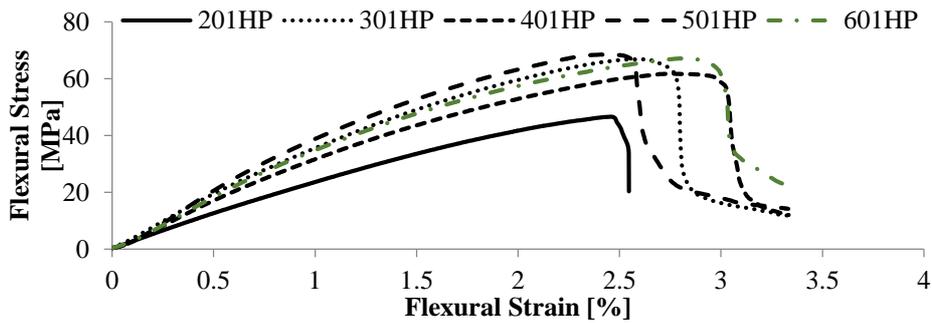
Figure 2. Flexural modulus of fabricated specimens

Flexural stress-strain curve of a plastic material consist of elastic region and plastic region. Elastic region is the region where stress increases linearly with strain. In elastic region, a material will return to its original dimension after the applied force is removed. The deformation in the plastic region is permanent meaning that a material is not yet fractured however will not recover its original shape after the sustained force is removed. For kenaf reinforced epoxy composites, all specimens only show slight elastic deformation and mostly plastic even at low strain. This statement was made due to the curvature presence of strain of as low as 0.5% in the stress-strain curve of Figure 3 and Figure 4 of the kenaf reinforced composites. Furthermore, all kenaf reinforced composites exhibited fracture at a strain value of lower than 4%.

(a)



(b)



(c)

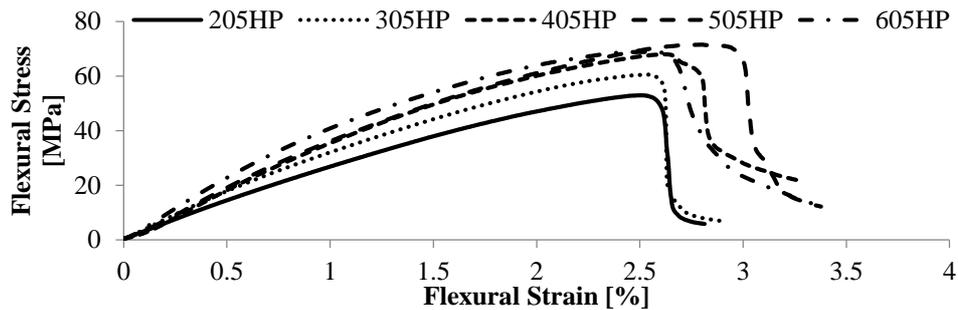


Figure 3. Flexural stress-flexural strain of HP specimens with flexural strength nearest to mean value: (a) Without silica, (b) With 1 vol% silica and (c) with 5 vol% silica

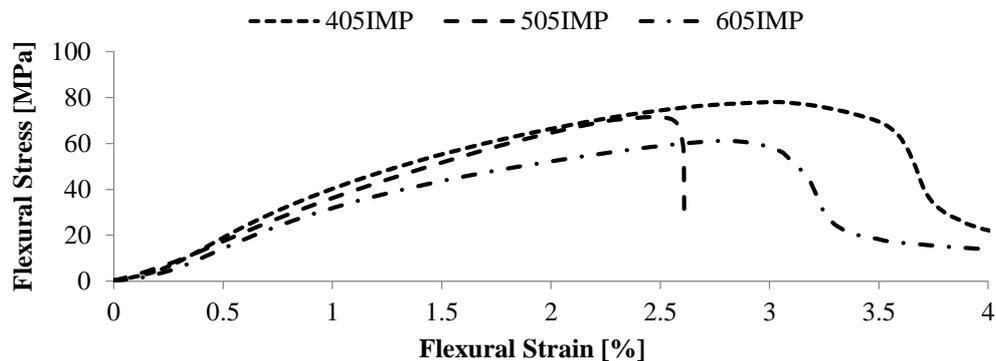


Figure 4. Flexural stress-flexural strain of IMP specimens with flexural strength nearest to mean value

Morphological Analysis

Figure 5 shows the morphological analysis of 400HP, 401HP and 405HP with 150 times magnification. The figure shows the surface of the specimens that have undergone flexural test and then split into two in the middle. The image of the surface in the split area is shown in the Figure. The images show the randomness of the kenaf used in this work as the fibres are not only protruding toward the direction of the location where the images were taken, they also faced a multitude of other directions too. From the SEM images taken, the thickness of single kenaf fibre measured ranged from $397\mu\text{m}$ to $624\mu\text{m}$. Furthermore, in Figure 5(a) and Figure 5(b) $744\mu\text{m}$ – $1487\mu\text{m}$ gaps can be seen. The gaps may either be the spaces between random kenaf mat layers that still remain even after being compressed or the spaces that were produced following flexural test due to delamination. In Figure 5(c), the fibre's lumens can be clearly seen (holes in the cross section of fibre). Other than the existence of more fibre pull out, which is the state of fibre being torn apart due to the application of forces in the opposite direction of the fibre length during flexural test, there seem to be no visible differences in the morphological structures of 400HP, 401HP and 405HP specimens. The target of doing morphological analysis is to locate the silica, whether it is in agglomerate form or single particle form. The magnification used in Figure 5 is not sufficiently large enough to make any trace of silica visible.

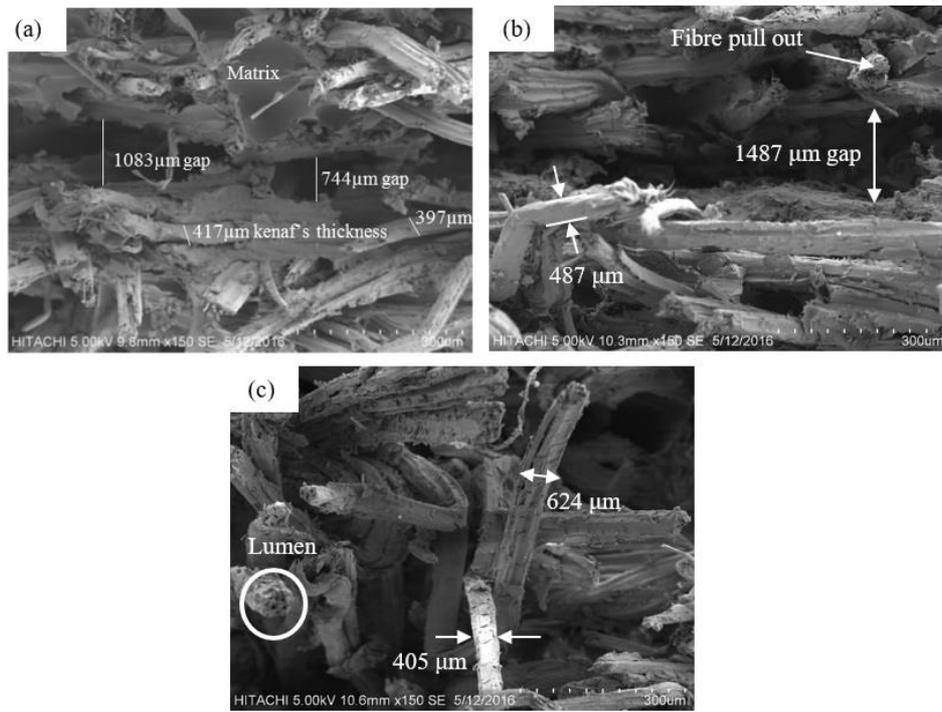


Figure 5. Surface morphology of HP specimens with 40 vol% kenaf at 150 times magnification: (a) without silica, (b) with 1 vol% silica and (c) with 5 vol% silica

Figure 6 shows the morphological analysis of 400HP, 401HP and 405HP specimens respectively with higher magnification of 600 times the original size which is the highest use in this work. From the figures, the thicknesses of single kenaf fibre measured were 735 μm, 972 μm and 487 μm respective to the order of figure appearance. From the measurements made with morphological images from Figure 5 and Figure 6, it can be noticed that the thickness of the kenaf fibres used in this work varied greatly. That is to be expected of natural based fibres as the growth of any plant is affected by multiple conditions such as the soil that is used to plant the seeds. Further analysis of Figure 6 shows that there are 27 μm to 118 μm gap in between kenaf fibres and the matrix. To promote a better stress transfer between matrix and fibre, a high level of interfacial adhesion is required. This means that the smaller the space between fibre and the matrix, is preferable. Furthermore, smaller gap means lower percentage of water can be absorbed by the composite.

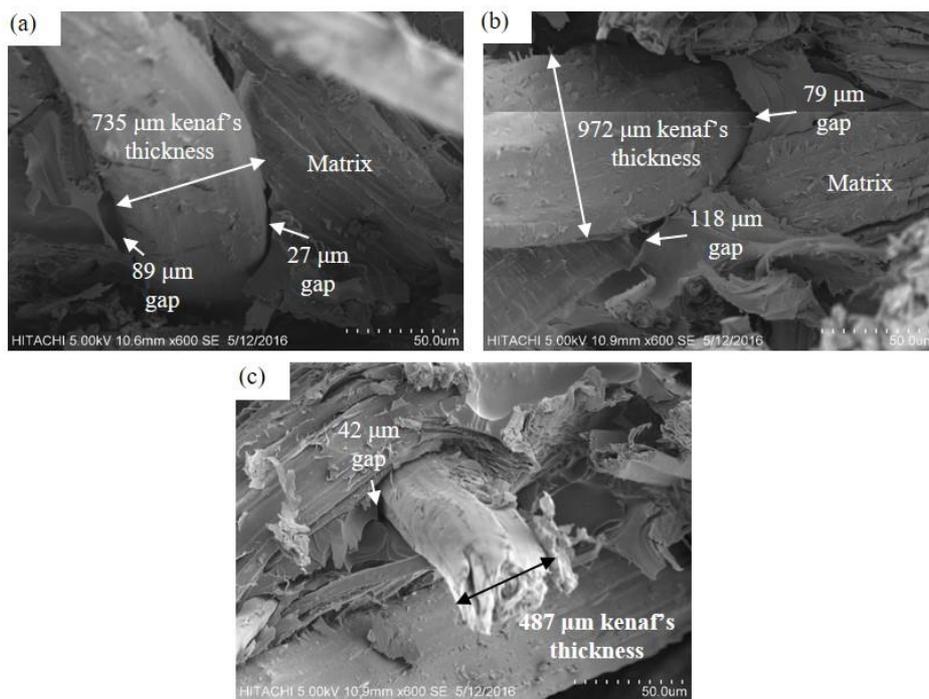


Figure 6. Surface morphology of HP specimens with 40 vol% kenaf at 600 times magnification: (a) without silica, (b) with 1 vol% silica and (c) with 5 vol% silica

Again, even with higher magnification, the existence of silica could not be verified. The average diameter of silica used in this work is 324 nm (0.324 μm) which is approximately 83 times higher than the smallest measurement made in Figure 6(a). It is inevitable that the presence of silica was undetectable unless the silica form an agglomerate large enough to be identified. In fact, it might be preferable that the agglomerate is not formed since this will indicate the homogeneity of silica dispersed into the matrix system.

Compressive Test

Figure 7 and Figure 8 respectively show the compressive strength and compressive modulus of fabricated specimens. For compressive strength and compressive modulus, like flexural properties again 405IMP specimens exhibited the highest values at 69.3 MPa and 1.80 GPa respectively. For HP specimens with 20 vol% silica content caused a reduction in compressive strength while for HP specimens with 30 and 40 vol% kenaf, 1 vol% silica inclusion reduced the compressive strength and 5 vol% silica inclusion improved the compressive strength. HP specimens with 20 vol% kenaf, 200HP, 201HP and 205HP specimens had the highest compressive strength at 58.8, 53.2 and 51.3 MPa. Apart from 305HP specimens, HP specimens with 30 vol% kenaf had the lowest compressive strength of 41.4 MPa for 300HP specimens and 39.8 MPa for 301HP specimens. Further increase in kenaf content increased the compressive strength without achieving the strength of specimens with 20 vol% kenaf. Inclusion of silica had various results upon the strength of the composites. For 20 vol% kenaf HP specimens, increase in silica content reduced the compressive strength. For 30 and 40 vol% kenaf HP specimens, 1 vol% silica inclusion reduced the composite strength when compared to specimens without silica. However 5 vol% silica increased the compressive strength. For specimens with 50 vol% kenaf, increase in silica content improved the composite strength. Lastly, for 60 vol% HP specimens, inclusion of silica improved the composite strength where composites with 1 vol% silica content exhibited higher value than composites with 5 vol% silica at 46.4 MPa for 600HP, 49.5 MPa for 601HP and 49.0 MPa for 605HP specimens. For IMP specimens, specimens with 40 vol% kenaf content exhibited the highest value over all specimens with 505IMP and 605IMP having similar compressive strength value to 200HP specimens at 57.7 and 58.9 MPa respective to 505IMP and 605IMP.

For compressive modulus, 505IMP and 605IMP both had lower compressive modulus than 405IMP specimens, but overall higher value than other composites at 1.27 and 1.20 GPa respectively. Inclusion of silica had varying impacts on the resulting composite compressive modulus. For 20 vol% kenaf HP specimens, inclusion of silica reduced the resulting composite modulus with 205HP composites showing higher value at 1.04 GPa than 201HP composites at 1.00 GPa. For 30 and 50 vol% kenaf HP specimens, while inclusion of 1 vol% silica reduced the compressive modulus, 5 vol% silica had a positive impact on the resulting composite modulus. For HP specimens with 40 vol% kenaf content, higher content of silica improved the compressive modulus even more. Finally, for HP specimens with 60 vol% kenaf, inclusion of silica had a positive impact on the resulting composite modulus with 1 vol% silica composites showing better results. The varying compressive properties results are attributed to the fibre's nature. Long fibre's such as the random kenaf mat used in this work provide support in the direction of the fibres' length. Long fibre is not strong in the opposite direction of the fibre's length. In the compressive test, the composite is compressed in the opposite direction of the fibre's length thus not providing much support. This may contribute to the lower strength of composites with higher kenaf content. The higher the kenaf content is, the lower the amount of epoxy is being used. Since, fibre does not provide much support and the support provided by epoxy has been replaced by fibre, reduction in compressive properties may occur.

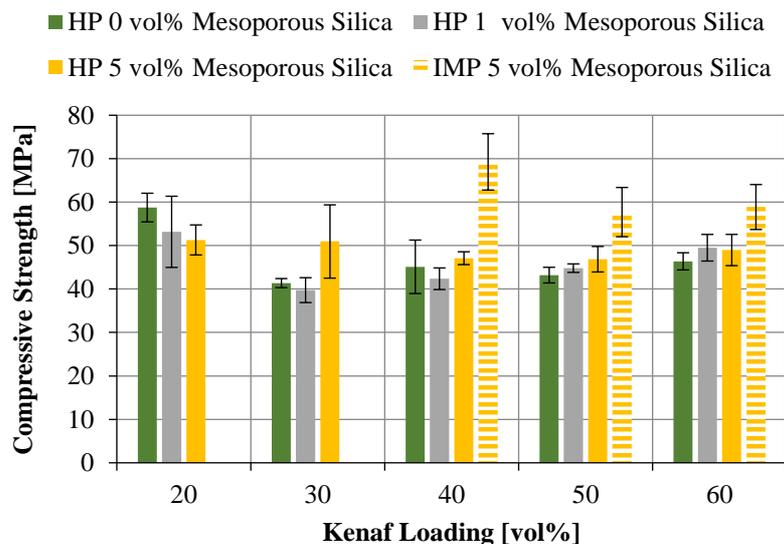


Figure 7. Compressive strength of fabricated specimens

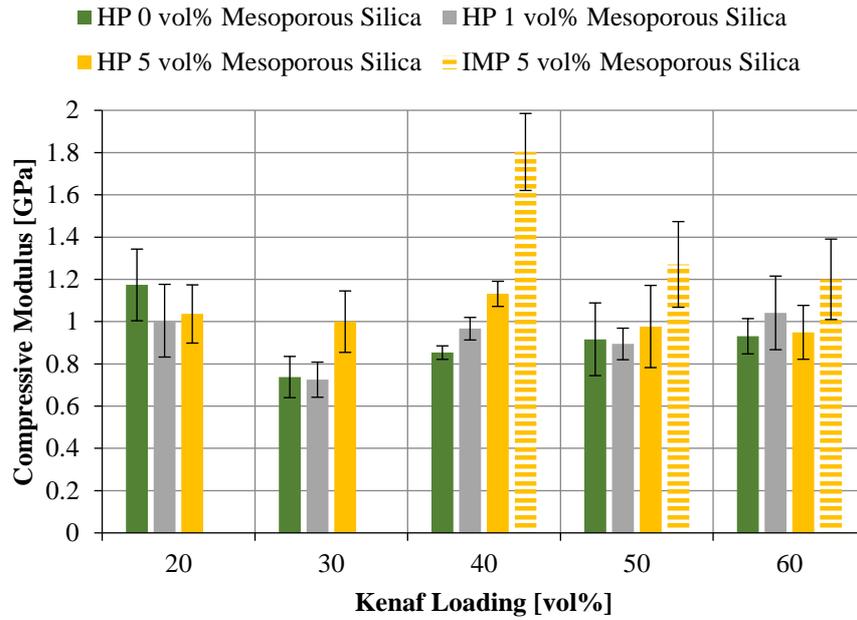
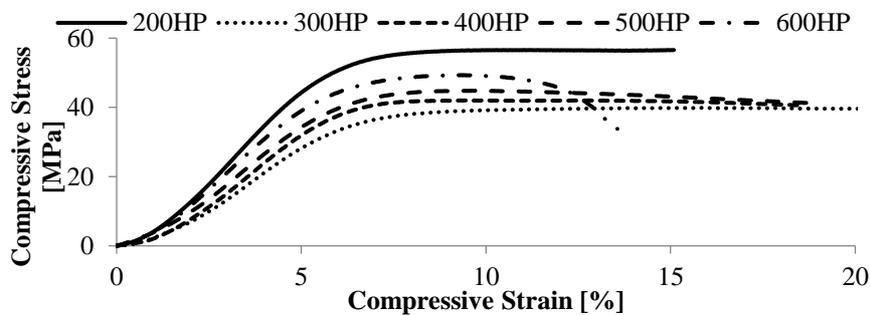


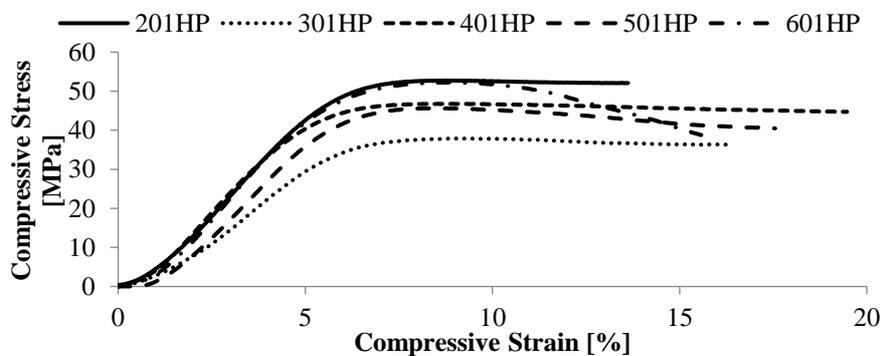
Figure 8. Compressive modulus of fabricated specimens

The compressive stress-strain curve for HP specimens and IMP specimens are shown in Figure 9 and Figure 10 respectively. For most specimens, there is a slight curvature in the beginning of the stress-strain curve that is due to the jig not touching the specimen properly. The jig is placed slightly higher than specimen to avoid accidental compression during fixture. Further than the curvature, most specimens show a linear increase in stress to strain. This shows that the kenaf reinforced composites exhibit elastic deformation prior to plastic deformation.

(a)



(b)



(c)

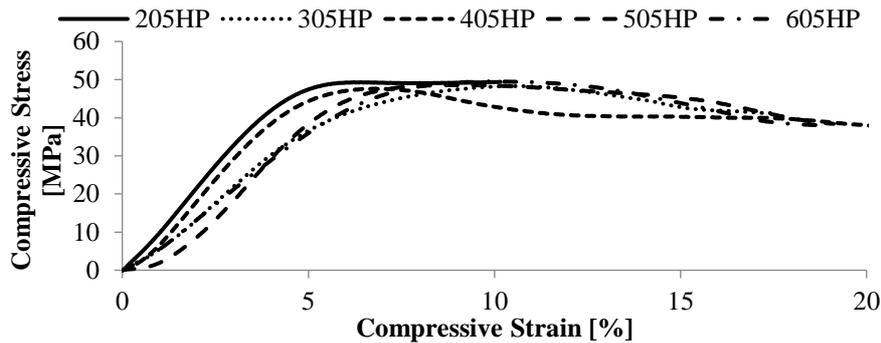


Figure 9. Compressive stress-compressive strain of HP specimens with compressive strength nearest to mean value: (a) without silica, (b) with 1 vol% silica and (c) with 5 vol% silica

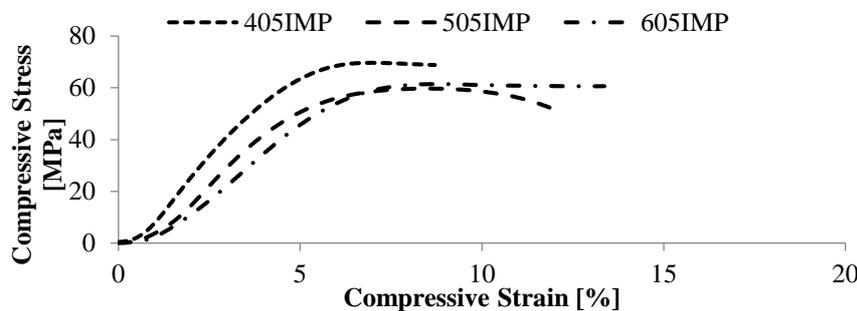


Figure 10. Compressive stress-compressive strain of IMP specimens with compressive strength nearest to mean value

Water Absorption Test

Figure 11 shows the maximum water absorption of every composites fabricated including neat epoxy after 84 days (2025 h) of immersion in distilled water at 30°C. or HP specimens increase in kenaf content increased the final water absorption. Increase in silica content also increased the water for HP specimens with 20, 30 and 40 vol% kenaf. For 50 vol% kenaf HP specimens, while there are increment in water absorption with increasing silica content, the increase was miniscule. For HP specimens with 60 vol% kenaf, specimens with both 1 and 50 vol% silica (601HP and 605HP respectively) had similar water absorption with specimen without silica (600HP). Overall, amongst the HP specimens 600HP absorbed the highest amount of water at 29.8%. Increment of water absorption with kenaf content increase is in align with the knowledge where kenaf is a hydrophilic fibres, thus by implementing the usage of higher hydrophilic material into a system, naturally the system will absorb more water. As stated previously in the introduction, silica is an extremely porous material. Especially if the silica agglomerates due to improperly disperse into the matrix, production of more space to be occupied by water molecules during immersion in water will occur. Thus, for HP specimens with 20, 30 and 40 vol% kenaf content absorbed higher percentage of water with the inclusion of silica. For HP specimens with 50 and 60 vol% kenaf, the abundant presence kenaf fibre caused the production of pores due to silica inclusion to be insignificant.

IMP specimens with 40, 50 and 60 vol% kenaf had 14.7, 15.5 and 13.5% maximum water absorption compared to HP specimens with the same kenaf and silica content at 29.8, 29.4 and 29.63% respectively. The maximum water absorption of IMP specimens were approximately half of HP specimens. This indicates that impregnation conditioning is successful at reducing the water absorption of the composites with same kenaf and silica content but different procedure (in this case HP specimens). The fibres' lumen is filled with silica/epoxy blocking the passage of water molecules. Due to immersion in negative pressure, voids are remove producing less space for water to occupy resulting in lower water uptake compared to HP specimens.

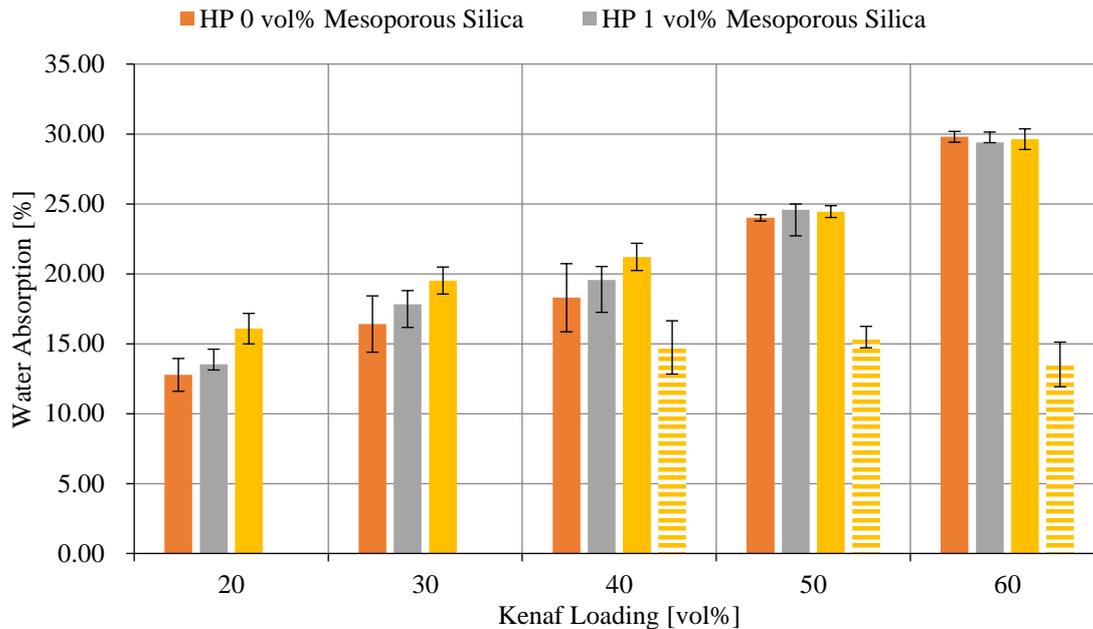


Figure 11. Maximum water absorption of fabricated composites and neat epoxy after 84 days of immersion in distilled water at 30°C

CONCLUSIONS

From the results obtained, it can be concluded that impregnation treatment was successful in improving the mechanical properties of silica/kenaf reinforced epoxy in term of flexural and compressive properties. Also, compared to HP specimens, IMP specimens had a reduction in water uptake which is the more desirable properties. Thus the impregnation process also improved the water absorption properties (by reducing the water uptake) of silica/kenaf reinforced epoxy. From the point of view of the impregnated specimens, 405IMP had the best improvement in term of flexural and compressive properties while 605IMP had the highest reduction in water uptake compared to their HP counterparts with the same content. As the impregnation process does not alter the chemical properties of natural fibres, in the future, a combination of chemical treatment and impregnation can be conducted to further improve the mechanical and water absorption properties of fabricated composites.

ACKNOWLEDGMENTS

The authors would like to thank Universiti Putra Malaysia (UPM) for providing grant (vot: 9463900) under Putra Grant (IPS) that greatly helps the progress of this work. Also, the authors would like to express their gratitude to Forest Research Institute Malaysia (FRIM) for providing the means to conduct impregnation process.

REFERENCES

- [1] S. Ochi, "Mechanical properties of kenaf fibers and kenaf/PLA composites," *Mechanics of Materials*, vol. 40, no. 4–5, pp. 446–452, 2008, doi: 10.1016/j.mechmat.2007.10.006.
- [2] Y. Xue, Y. Du, S. Elder, K. Wang, and J. Zhang, "Temperature and loading rate effects on tensile properties of kenaf bast fiber bundles and composites," *Composites Part B: Engineering*, vol. 40, no. 3, pp. 189–196, 2009, doi: 10.1016/j.compositesb.2008.11.009.
- [3] M. de F. V. Marques, R. P. Melo, R. da S. Araujo, J. do N. Lunz, and V. de O. Aguiar, "Improvement of mechanical properties of natural fiber-polypropylene composites using successive alkaline treatments," *Journal of Applied Polymer Science*, vol. 132, no. 12, pp. 1–12, 2015, doi: 10.1002/app.41710.
- [4] S. J. Kim, J. B. Moon, G. H. Kim, and C. S. Ha, "Mechanical properties of polypropylene/natural fiber composites: Comparison of wood fiber and cotton fiber," *Polymer Testing*, vol. 27, no. 7, pp. 801–806, 2008, doi: 10.1016/j.polymertesting.2008.06.002.
- [5] B. H. Lee, H. S. Kim, S. Lee, H. J. Kim, and J. R. Dorgan, "Bio-composites of kenaf fibers in polylactide: Role of improved interfacial adhesion in the carding process," *Composites Science and Technology*, vol. 69, no. 15–16, pp. 2573–2579, 2009, doi: 10.1016/j.compscitech.2009.07.015.

- [6] O. M. L. Asumani, R. G. Reid, and R. Paskaramoorthy, "The effects of alkali-silane treatment on the tensile and flexural properties of short fibre non-woven kenaf reinforced polypropylene composites," *Composites Part A: Applied Science and Manufacturing*, vol. 43, no. 9, pp. 1431–1440, 2012, doi: 10.1016/j.compositesa.2012.04.007.
- [7] A. Chafidz, M. Rizal, R. M. Faisal, M. Kaavessina, D. Hartanto, and S. M. AlZahrani, "Processing and properties of high density polyethylene/date palm fiber composites prepared by a laboratory mixing extruder," *Journal of Mechanical Engineering and Sciences*, vol. 12, no. 3, pp. 3771–3785, 2018, doi: 10.15282/jmes.12.3.2018.2.0333.
- [8] S. Serizawa, K. Inoue, and M. Iji, "Kenaf-fiber-reinforced poly(lactic acid) used for electronic products," *Journal of Applied Polymer Science*, vol. 100, no. 1, pp. 618–624, 2006, doi: 10.1002/app.23377.
- [9] T. Nishino, K. Hirao, M. Kotera, K. Nakamae, and H. Inagaki, "Kenaf reinforced biodegradable composite," *Composites Science and Technology*, vol. 63, no. 9, pp. 1281–1286, 2003, doi: 10.1016/S0266-3538(03)00099-X.
- [10] J. Summerscales, A. Virk, and W. Hall, "A review of bast fibres and their composites: Part 3 - Modelling," *Composites Part A: Applied Science and Manufacturing*, vol. 44, no. 1, pp. 132–139, 2013, doi: 10.1016/j.compositesa.2012.08.018.
- [11] N. Sallih, P. Lescher, and D. Bhattacharyya, "Factorial study of material and process parameters on the mechanical properties of extruded kenaf fibre/polypropylene composite sheets," *Composites Part A: Applied Science and Manufacturing*, vol. 61, pp. 91–107, 2014, doi: 10.1016/j.compositesa.2014.02.014.
- [12] H. M. Akil, M. F. Omar, A. A. M. Mazuki, S. Safiee, Z. A. M. Ishak, and A. Abu Bakar, "Kenaf fiber reinforced composites: A review," *Materials & Design*, vol. 32, no. 8–9, pp. 4107–4121, 2011, doi: 10.1016/j.matdes.2011.04.008.
- [13] K. L. Pickering, M. G. A. Efendy, and T. M. Le, "A review of recent developments in natural fibre composites and their mechanical performance," *Composites Part A: Applied Science and Manufacturing*, vol. 83, pp. 98–112, 2016, doi: 10.1016/j.compositesa.2015.08.038.
- [14] N. Saba, M. T. Paridah, and M. Jawaid, "Mechanical properties of kenaf fibre reinforced polymer composite: A review," *Construction and Building Materials*, vol. 76, pp. 87–96, 2015, doi: 10.1016/j.conbuildmat.2014.11.043.
- [15] M. N. A. Nordin *et al.*, "Tensile and impact properties of pulverized oil palm fiber reinforced polypropylene composites: A comparison study with wood fiber reinforced polypropylene composites," *Journal of Mechanical Engineering and Sciences*, vol. 12, no. 4, pp. 4191–4202, 2018, doi: 10.15282/jmes.12.4.2018.15.0361.
- [16] M. S. M. Basri, N. Mazlan, and F. Mustapha, "Effects of stirring speed and time on water absorption performance of silica aerogel / epoxy nanocomposite," vol. 10, no. 21, pp. 9982–9991, 2015.
- [17] M. S. Meon, M. F. Othman, H. Husain, M. F. Remeli, and M. S. M. Syawal, "Improving tensile properties of kenaf fibers treated with sodium hydroxide," *Procedia Engineering*, vol. 41, no. Iris, pp. 1587–1592, 2012, doi: 10.1016/j.proeng.2012.07.354.
- [18] P. J. Herrera-Franco and A. Valadez-Gonzalez, "A study of the mechanical properties of short natural-fiber reinforced composites," *Composites Part B: Engineering*, vol. 36, no. 8, pp. 597–608, 2005, doi: 10.1016/j.compositesb.2005.04.001.
- [19] M. H. Kothmann, R. Zeiler, A. Rios De Anda, A. Brückner, and V. Altstädt, "Fatigue crack propagation behaviour of epoxy resins modified with silica-nanoparticles," *Polymer (Guildf)*, vol. 60, pp. 157–163, 2015, doi: 10.1016/j.polymer.2015.01.036.
- [20] M. R. Ishak, Z. Leman, M. S. Salit, M. Z. A. Rahman, M. K. Anwar Uyup, and R. Akhtar, "IFSS, TG, FT-IR spectra of impregnated sugar palm (*Arenga pinnata*) fibres and mechanical properties of their composites," *Journal of Thermal Analysis and Calorimetry*, vol. 111, no. 2, pp. 1375–1383, 2013, doi: 10.1007/s10973-012-2457-5.
- [21] ASTM D790-03, "Standard Test Method for Flexural Properties of Unreinforced and Reinforced Plastics and Electrical Insulation Materials," *ASTM Stand.*, pp. 1–11, 2003.
- [22] ASTM D695-02a, "Standard Test Method for Compressive Properties of Rigid Plastics," *ASTM Stand.*, pp. 1–8, 2003.
- [23] ASTM D570-98, "Standard Test Method for Water Absorption of Plastics," *ASTM Stand.*, pp. 1–4, 1998.
- [24] V. Fiore, G. Di Bella, and A. Valenza, "The effect of alkaline treatment on mechanical properties of kenaf fibers and their epoxy composites," *Composites Part B: Engineering*, vol. 68, pp. 14–21, 2015, doi: 10.1016/j.compositesb.2014.08.025.
- [25] G. N. Farahani, I. Ahmad, and Z. Mosadeghzad, "Effect of Fiber Content, Fiber Length and Alkali Treatment on Properties of Kenaf Fiber/UPR Composites Based on Recycled PET Wastes," *Polymer-Plastics Technology and Engineering*, vol. 51, no. 6, pp. 634–639, 2012, doi: 10.1080/03602559.2012.659314.