



## Investigation on the Flexural Properties and Glass Transition Temperature of Kenaf/Epoxy Composite Filled with Mesoporous Silica for Wind Turbine Applications

Chai Hua, T.<sup>1</sup> and Norkhairunnisa, M.<sup>2\*</sup>

<sup>1</sup>Department of Aerospace Engineering, Faculty of Engineering, Universiti Putra Malaysia, 43400 UPM, Serdang, Selangor, Malaysia

<sup>2</sup>Institute of Tropical Forestry and Forest Products, Universiti Putra Malaysia, 43400 UPM, Serdang, Selangor, Malaysia

### ABSTRACT

This research investigates the strength of kenaf or epoxy composite filled with mesoporous silica and studies the hybrid effects between mesoporous silica or kenaf in epoxy matrix. The volume of kenaf woven mat is maintained constantly at 7.2vol%, whereas proportion of epoxy is varied with inclusion of mesoporous silica and silicon, keeping constant the volume of the composite at 67.5cm<sup>3</sup>. The proportion of mesoporous silica is altered from 0.5vol%, 1.0vol%, 3.0vol% and 5.0vol%, while silicon is kept constant at 3.0vol%. A total of 11 specimens were produced, each with its distinctive composition and mechanical strengths. Variation of fillers composition affects the mechanical strengths of the composite. SEM analysis shows that epoxy bonds well with silicon, kenaf and mesoporous silica. Some de-bonding among the components is observed within the composite although there is also some tearing of fibres and impregnation of epoxy within fibre, proving that the components have good interaction and do not act individually. Flexural test shows that mesoporous silica improves the flexural strength of the composite, where the highest value is 35.14MPa, obtained at 5.0vol% Mesoporous Silica in Kenaf/Epoxy (SiaK/Ep). It also improves the flexural modulus, where the highest value is 1569.48MPa, obtained at 3.0vol% SiaK/Ep. DMA result reveals that adding mesoporous silica increases the T<sub>g</sub> of the composite produced. Highest T<sub>g</sub> is obtained at 0.5vol% Mesoporous Silica in Kenaf/Epoxy modified Silicon (SiaK/Ep-Si) with the value of 87.54°C.

*Keywords:* Epoxy, hybrid composite, kenaf, natural fibre reinforced polymer

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#### E-mail addresses:

peggytay\_92@yahoo.com (Chai Hua, T.),

norkhairunnisa@upm.edu.my (Norkhairunnisa, M.)

\*Corresponding Author

### INTRODUCTION

Over the years, the use of energy has increased tremendously with the increase in population.

The widely used sources of energy such as coal, coke, crude oil, natural gas, oil shale, tar sands and nuclear material are non-renewable. This has created ecological awareness and leads to increased interest in using renewable, sustainable and ecological wise energy. Wind energy is produced from one of many renewable energy sources that are continually studied and improvised in every way possible to solve global energy problem. Wind energy can be converted into electrical energy or mechanical energy with the help of wind turbine. Wind turbine basically consists of blades, nacelle, tower and base. The main part that contributes in generating electrical energy or mechanical energy is the blades. Blades with good aerodynamic shape, length and angle constitute to higher efficiency of wind turbine. It must also be made of good materials with low density, high strength and stiffness and sufficient fatigue strength to last long.

At present, Glass Fibre Reinforced Plastics (GFRP) and Carbon Fibre Reinforced Plastics (CFRP) are widely used in manufacturing wind turbine blades. These materials not only cause environmental hazards, they are also expensive. In addition, these materials depend on depleting source that almost comes to its dead end. Thus, attentions are now centred to renewable and environmental friendly source, Natural Fibre Reinforced Polymer (NFRP). Unlike GFRP and CFRP, NFRP exhibits certain excellent properties such as low density, low cost, non-abrasive properties, biodegradable and renewable. This is due to the natural fibre properties that are light and abundant in source. Varieties of natural fibre available are such as flax, sisal, cotton, linen, hemp, bamboo, bagasse, banana, jute, abaca and kenaf.

According to Debnath et al. (2013, pp. 25-40), Polymer Matrix Composite (PMC) is made up of polymer/matrix and reinforcement/fibre. Both these materials exhibit different properties, physically and chemically. The matrix benefits in determining the overall properties of the composites produced. This important role results from various functions of matrix such as acting as a bridge in binding the fibres together, transferring the loads to the fibres, providing good surface finishing and preventing the fibres from degradation due to environmental factors. The matrix must be able to deform easily under applied load, transfer load to the fibres and distribute the stress concentration evenly. The U.S. Congress, Office of Technology Assessment (1988, pp. 73-93), stated that the degradative process of a PMC includes impact damage, delamination, water absorption, chemical attack and high-temperature creep. The resistance towards these processes is determined by the properties of matrix used to produce PMC. Gonzáles et al. (2014) mentioned that since polymer matrix is easy to infiltrate in the fibre preforms and incurs lower cost, it is widely used in manufacturing composites. There are two main types of polymer matrix, namely thermosets and thermoplastics.

Gonzáles et al. (2014) stated that thermosets are produced by a chemical reaction called curing. Curing involves a combination of two polymers; resin and hardener, or resin and catalyst. At room temperature, thermosets exist as low viscosity liquids and they can easily be infiltrated into the fibre preforms. During curing, covalent bonds connect different polymeric chains which lead to three-dimensional network. Thermosets do not transform to liquid again if heated after it has been cured even though their mechanical properties decrease above Glass Transition Temperature ( $T_g$ ). What happens above  $T_g$  is that the molecular structure becomes flexible compared to below  $T_g$  which is a rigid network. This change is a reversible process. This material exhibits certain advantages such as not costly, possesses high stiffness and strength,

has good chemical attack resistance and exists as the standard matrices for PMC. However, the material is rather brittle and has limited high temperature capabilities (particularly under hot/wet conditions).

Unlike thermosets, thermoplastics transform into liquid when heated. When cooled sufficiently, it will freeze to a very glassy state. Most thermoplastics exhibit properties such as high-molecular weight polymer whose chains are connected through weak Van der Waals bonds (polyolefins), hydrogen bonds (nylon) or stacking arrangement of aromatic rings (polystyrene). These materials can have amorphous or semi-crystalline structure, changing amorphous regions with semi-crystalline regions in which the chains approximate random coils. Below  $T_g$ , thermoplastic has higher ductility and toughness than thermosets. Above  $T_g$ , both these properties increase very quickly while strength decreases. What happens above  $T_g$  is that changes occur to amorphous chains which transform from glassy state into a rubbery one. When thermoplastic is heated above the Melting Temperature ( $T_m$ ), its viscosity reduces gradually without any significant change in phase and it becomes viscous liquid that can be infiltrated into fibre preform. Advantages of thermoplastics are that it can be re-melted and remoulded. These melting/freezing cycles are repeatable. Examples of thermoplastic that are usually used in engineering aspects include polyolefins (PE, PP), polyamides (nylon) and polystyrene. These materials can be reinforced with particles or short fibres to improve stiffness, strength, barrier properties and the like. Examples of thermoplastics that are specifically for composite material production include poly (ether ester) (PEE) and polyethersulfone (PES).

The U.S. Congress, Office of Technology Assessment (1988, pp. 73-93) has a detailed explanation about the structure of thermosets and thermoplastics which determines the mechanical strength. According to the Congress, thermosets have good resistance to solvents, high dimensional stability and high temperature resistance, all due to the three-dimensional crosslink structure of thermosets. Thus, progress has been made recently to improve the toughness and maximum operating temperatures of thermosets. Thermoplastic, on the other hand, consists of long, discrete molecules that melt into viscous liquid at processing temperature, normally at 260°C to 3710°C. After forming viscous liquid and being cooled to sufficient temperature, the amorphous, semicrystalline or crystalline solid resulted determines the properties of matrix. Specifically, the degree of crystallinity has a strong effect on the resulted matrix properties.

Although polymer matrix plays a significant role in determining the overall performance of composite, the reinforcing fibres are important as well. Debnath et al. (2013, pp. 25-40) mentioned that the main function of reinforcement fibres is that they act as a load carrying member in the composite, besides providing sufficient strength and stiffness in the composite produced. In other words, inclusion of fibres improves the mechanical properties of the neat resin system. These reinforcing fibres can be divided into two main fibres, namely natural and synthetic ones.

To study the properties of natural fibres, one can look into the structure of the fibre itself. Westman et al. (2010), in their review of Natural Fibre Composites, reported that natural fibres mainly consist of cellulose, hemicelluloses, pectin and lignin. Different fibres have different ratio of these four primary elements, whereby this ratio can be affected by growing and harvesting conditions. Each of these elements has distinguished properties and functions.

Cellulose is a semicrystalline polysaccharide and it contributes to the hydrophilic properties of natural fibres. Hemicellulose is a fully amorphous polysaccharide although with lower molecular weight in comparison with cellulose. Hemicellulose is partially soluble in both water and alkaline solutions due to its complete amorphous nature. Pectin, which functions in holding the fibres together, is also a polysaccharide like cellulose and hemicellulose. Lignin is also an amorphous polysaccharide like hemicellulose, except that it is made up of mainly aromatics and has low effect on water absorption.

Weinberger (1996) stated that synthetic fibres could be tailor-made to provide significant properties that cannot be provided by natural fibres. Due to this reason, synthetic fibres will continuously be in demand. According to Beychok (2011), there are 2 types of synthetic fibre products; semisynthetics and true synthetics. There are a few significant differences between these synthetic fibres. Semisynthetics, which is also known as cellulose, are formed from natural polymeric materials like cellulose. Common semisynthetics include cellulose acetate and viscose rayon. Noncellulose, another name for true synthetics, are the products of polymerisation of smaller chemical units into long-chain molecular polymers. Common true synthetics include polyester, nylon, acrylic and modacrylic, and polyolefin. The process of forming fibres involves a spinnerette.

Fibres are produced by forcing a viscous liquid of polymer through small orifices of a spinnerette, before being solidified immediately to form filaments. The produced polymer may also be used to manufacture other non-fiber products like large amount of extruded plastics and synthetic rubber products. Typically, both synthetic fibres are produced via 2 methods; melt spinning and solvent spinning. Melt spinning, like that name, uses heat to melt the fibre polymer to suitable viscosity for the purpose of extrusion through spinnerette. Solvent spinning, on the other hand, is a process involving major operations of dry spinning and wet spinning. This process uses large amount of organic solvents, which are usually recovered for economic purposes, to dissolve fibre polymer into fluid polymer solution. Similar to melt spinning, fibre polymer is dissolved into suitable fluid polymer solution condition for extrusion through spinnerette. There is a third method, namely, reaction spinning. However, reaction spinning is not widely used. This process involves the formation of filaments from prepolymers and monomers. Once formed, they are further polymerised and cross-linked.

Debnath et al. (2013, pp. 25-40) mentioned that between the reinforcing fibre and matrix, there is a contiguous region which results from different materials in the composite. In more specific, this region is called the interphase, which has the characteristic that is not represented by any elements in the composite. It is responsible for the load bearing capacity of the composite. The wettability property of both reinforcing fibre and polymer matrix determines the adhesion efficiency of the constituents. The higher the adhesion, the more superior the mechanical properties of the resulting composite will be. This adhesion, however, can be improved by treating the natural fibres. The treatment improves the interfacial bonding strength between reinforcing fibres and polymer matrix, which leads to improved mechanical strength and dimensional stability in the composite produced. Some treatments for natural fibres include using chemical treatment such as NaOH and greener surface treatment like white rot fungus.

The U.S. Congress, Office of Technology Assessment (1988, pp. 73-93) stated in their book that interphase is the region in PMCs where loads are transmitted between the reinforcement and matrix. The limited interaction between the reinforcement and the matrix is different for every design. It may vary from strong chemical bonding to weak frictional forces. Proper coating on the reinforcing fibres can be used to control this interaction. Generally, a PMC is more rigid when its interracial bond is strong, but this will also make the PMC brittle. Comparing to weak interracial bond, the PMC stiffness reduces but toughness increases. If the interracial bond is not at least as strong as the matrix, certain loading conditions can result in de-bonding at interphase. To maximise the fracture toughness of the PMC, the most desirable coupling is usually intermediate between the strong and weak limits. The properties of the interfacial bond is also significant in determining the long-term stability of the PMC, affecting the fatigue properties, environmental behaviour and resistance to hot/wet conditions. This paper focuses on NFRP.

Salit (2014, pp. 15-38), in his book, stated that kenaf (*Hibiscus cannabinus L.*) originates from West Africa and has been cultivated since 4000 B.C. Kenaf fibres are similar to cotton and okra, they are all a member of the Hibiscus gene and family of Malvacea. It is a common wild plant in tropical and subtropical Africa and Asia. Kenaf is a fast growing tree and has a very short lifecycle. Nevertheless, it could be harvested in just 4-5 months. Kenaf stalk consists of soft inner core and fibrous outer bast surrounding the core. Comparing to soft wood fibre, kenaf fibre is longer but has a smaller diameter and three times greater tensile strength. Besides that, kenaf has superior toughness and high aspect ratio in comparison with other fibres. This makes it desirable to be used as a reinforcing fibre in thermoplastic composites. Akil et al. (2011, pp. 4107-4121) reported that the kenaf rate of photosynthesis is significantly high. This means that kenaf produces more oxygen compared to other plants and this is definitely good for the environment.

González et al. (2014) stated that there are three types of reinforcement index; particles, short fibres and long fibres. Since this paper focuses on NFRP, only short and long reinforcing fibres are explained. Short fibres are defined by their length (ranging from 100 $\mu$ m to several mm) and high aspect ratio. However, their most relevant parameters are fibre volume fraction ( $V_f$ ), average length and length distribution  $\bar{l}$ , as shown in equation 1 and fibre orientation in 2D (lamina) as shown in equation 2 or 3D (space).

There are plenty orientations of long fibre for production of NFRP, which lead to different mechanical strengths. Thus, manufacturers have a lot of choices to choose from in producing composites suitable for their desired purposes. According to González et al. (2014), unidirectional lamina is the easiest fibre arrangement. Its maximum volume fraction is determined by transverse fibre arrangement. For rectangular arrangement, the fibre fraction is 78.5vol%, while the volume fraction is 90.7vol% for hexagonal. These values are not valid in real composites, whereby the range is normally between 60 - 70vol%. This is because above this range, the matrix infiltration becomes very complicated.

Multidirectional laminates are actually unidirectional laminates stacked up with different orientations, which can be of different angles combination such as [0/90/0/0/90/0], [0/60/-

60/-60/60/0] and many others. [0/90/0/0/90/0] can also be presented as [0/90/0\_2/90/0] and [0/90/0]s while [0/60/-60/-60/60/0] can be presented as [0/60/-60<sub>2</sub>/60/0] and [0/60/-60]<sub>s</sub>. Woven fabrics are planar textile preforms produced by weaving two sets of yarn at the angles of 0° and 90°. Different weaving methods include plain, twill and satin. Weaving produces fabrics with firm construction and very low slippage. Knitting is a method of interloping yarns such as drawing loops of one yarn into the previous one. To increase stiffness, straight warp and/or filling yarns can be added. Braiding is done by interlacing yarns in the bias direction. This method is the same as rotated woven fabrics. Normally, braided fabrics are produced over mandrel. This is to manufacture flat or tubular products. Nonwoven felts are produced from a set of disordered fibres. These fibres are strengthened by bonds of different nature like simple entanglement, chemical binders or local thermal fusion.

3D fabrics are produced to exhibit higher strength and stiffness of textile preforms perpendicular to the fabric direction. Non-crimp fabrics are almost similar to multidirectional laminates which are made up of unidirectional plies with different fibre orientations, except that they are assembled not by stacking but by stitching. Stitching fabrics, on the other hand, is the method of stitching multidirectional laminates and/or 2-dimensional textile preforms to enhance out-of plane mechanical properties.

According to Hexcel (2014), a wind blade is a structural beam that is subjected to considerable lift forces throughout its operating life. In some cases, there are two strips of reinforcing materials used to provide local stiffening; one on the upwind face and the other on the downwind face. These two strips are joined structurally by shear web. This shear web is important as it allows the two strips to provide sufficient shear strength to function as wind turbine blades. There are two ways to design a shear web, either as girder-structure connected by one or two shear webs, or as a full box beam structure. In order to manufacture such structure, high stiffness materials are needed to prevent the blade from striking the tower while rotating. The longer the blade, the higher the stiffness of the material will be needed. Therefore, high quality materials are required for such applications.

Besides the shear web, the shell also needs to be made of material with high stiffness. The shell provides the aerodynamic shape of the blade and at the same time helps in stiffening and strengthening the shear web. Proper fibre orientation during construction can help to resist torsion. Blade shells are usually quite thin because the shell structure does not need to have high strength. However, its large flat shape could affect the aerodynamic shape and lead to buckling. Due to this reason, some areas of the shells are constructed as “sandwich”, laminated skins with a core. Meanwhile, low density rigid foam or balsa wood is normally used in this design. Since shell is made up the outer skin of the blade, it must have a high resistance towards harsh environmental conditions. A composite shell has streamlines aerodynamic design that provides excellent environmental resistance.

The nacelle contains the technical parts of the wind turbine. This includes the low-and-high-speed shafts, gearbox, brake, generator, blade pitch control, a hydraulic system controlling angle of the blades and yaw drive controlling position of turbine relative to wind. As the nacelle keeps all the main technical parts, it must be made of good and strong material to prevent

itself from damages and exposing its internal parts. The nacelle is usually made of fibreglass. The success of blade manufacturing depends on its effective mould design. The mould need to exhibit properties such as quick heat-up and cool-down rates and low weight so that it is easier to be lifted.

The blade root is normally circular in cross section to connect to the pitch bearing in hub. The wind blades are fixed to the hub using a bolted connection. This is to ensure that the wind blades can be removed. The girder structure of load carrying shear web must be joined to a cylindrical laminate at the root. This cylindrical laminate is usually thick and has studs or T-bolts screwed or bonded in. During curing of composite material, care must be taken so as to maintain the desired thickness in the laminate section. High thickness in laminate section could lead to a build-up of high exothermic temperatures. According to Stout (2013), wind turbines are often exposed to extreme temperature swings, ranging from -30°C to 55°C. Thus, the T<sub>g</sub> of composite material used for wind turbine blade must be higher than 55°C.

## MATERIALS AND METHODS

Mesoporous silica used is distributed by Maerotech Sdn. Bhd. Silicon used is GENIOPERL® P52 distributed by Wacker. Meanwhile, the kenaf used is kenaf woven mat from Bangladesh. The epoxy used in EpoxAmite 100 Epoxy Laminating System is distributed by Smoot-On. It exists as clear yellowish liquid form with low viscosity in room temperature. It is used together with 103 slow hardener in the ratio of 100 epoxy : 28.4 hardener. Both have specific gravity of 1100kg/m<sup>3</sup> and specific volume of 0.009m<sup>3</sup>/kg. The mixture takes around 20 – 24 hours to cure under room temperature.

3vol% of silicon and a variation of mesoporous silica percentage (0.5vol%, 1.0vol%, 3.0vol% and 5.0vol%) are mixed into epoxy using homogeniser at 3000rpm for a period of 1 minute. At this stage, if a lot of bubbles are observed, the mixture is placed in a vacuum oven to remove the bubbles. Next, hardener is poured into the mixture, followed by stirring process at the speed of 300rpm for a period of 3-5 minutes using a mechanical stirrer. Fabrication of the composite is done by using resin transfer infusion method. Initially, sealant tape is pasted on the fabrication table to forming a square shape that enough to fit a desired size of kenaf mat. The space within the square formed is sprayed with silicon spray release agent to ease the removal of composite from table after the fabrication. The woven kenaf mat is placed on the fabrication table followed by peel ply. Tube is then placed on two sides of the kenaf mat. T-pipe is fixed accordingly for the inlet and outlet of epoxy containing silicon and mesoporous silica. Sealant tape top is removed and vacuum bagging film is then placed on top of the sealant tape to cover the whole materials. Machine is switched on and the outlet translucent pipe is placed in beaker filled with epoxy containing silicon and mesoporous silica until all resin is sucked in. Finally, the specimen is left to cure in room temperature for one day and later post-cured in an oven at 80°C for two hours.

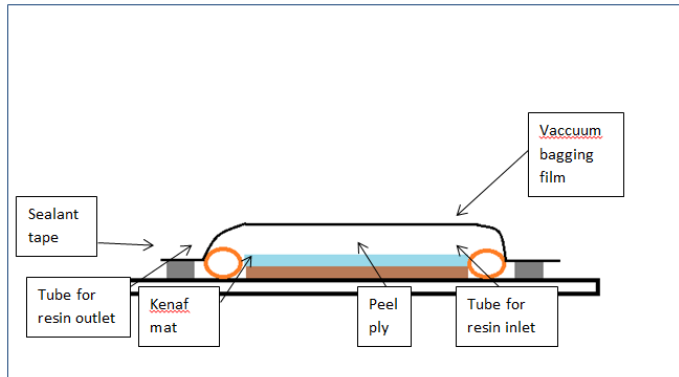


Figure 1. A schematic picture of the sample preparation using the resin transfer infusion method

## RESULTS AND DISCUSSION

Figure 2 above represents the SEM analysis of the fractured area from the flexural testing samples. Figure 2-A and Figure 2-B show some characteristics of the Epoxy modified Silicon (Ep-Si) composite. De-bonding, filler fracture and fully intact fillers are observed in both these figures. However, the de-bonding shown is not obvious and does not happen to all the interactions between the two components. Most filler are fully intact, showing good interaction. Meanwhile, the breakage of silicon indicates that the component does not act as an individual component, but as one composite. Thus, at this fracture point, no silicon is de-bonded as a whole. Silicone does not have pores within, which proves that it has excellent compressive property. This is supported by Tay (2015), who mentioned that silicon powders are hard substances with no elasticity.

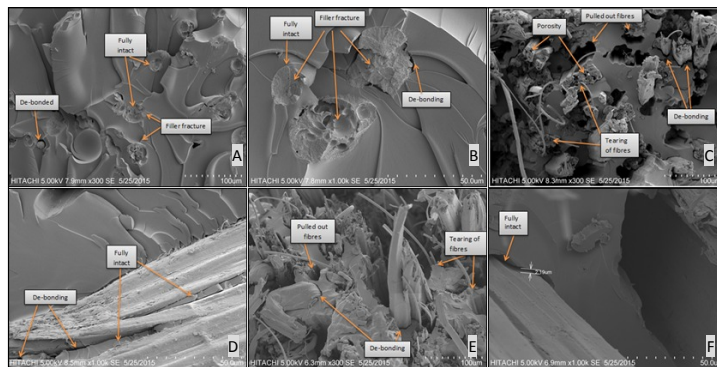


Figure 2. A and B are Ep-Si composites, C and D are 7.2vol% K/Ep, E and F is 0.5vol% SiaK/Ep-Si composite

Figure 2-C and Figure 2-D show some characteristics of 7.2vol% of Kenaf/Epoxy (7.2vol% K/Ep) composite. In Figure 2-C above, some parts are noticed to have de-bonding that shows low interaction between kenaf and epoxy. Some kenaf are observed to be porous, with no epoxy within the fibre. There are also clear pull-out and tearing of some kenaf fibres in the



same figure. Yousif et al. (2012, pp. 378-385) stated that if there were less interactions between the core of fibres and the matrix, and if the fibres had high porosity, both the phenomenon would lead low mechanical performance. According to Sergio et al. (2012, pp. 17-28), poor interfacial adhesion between the two components generates void that may have impact on the mechanical strength of the composite.

Clear pull out indicates low interfacial adhesion between them. According to Sergio et al. (2012, pp. 17-28), the tearing of kenaf could be caused by a lack of epoxy penetration inside the fibre. In Figure 2-D, epoxy is seen to impregnate well within the kenaf fibre. This contributes to the strong bonding between the two elements. According to Nishino et al. (2003, pp. 1281-1286), as resin matrix was fused into the interfibrillar region, it contributed as one of the reinforcements between the fibre and resin. There are fully intact region between the two components in Figure 2-D, indicating that the mechanical strength of this composite might not be too low after all, despite the observed de-bonding.

Figure 2-E and Figure 2-F show some characteristics of 0.5vol% SiaK/Ep-Si composite. De-bonding among the components in Figure 2-E are seen to be lesser than in the composite of 7.2vol% K/Ep. Figure 2-F shows small void spaces of  $2.19\mu\text{m}$  between reinforcing kenaf fibre and resin matrix indicating poor interfacial adhesion.

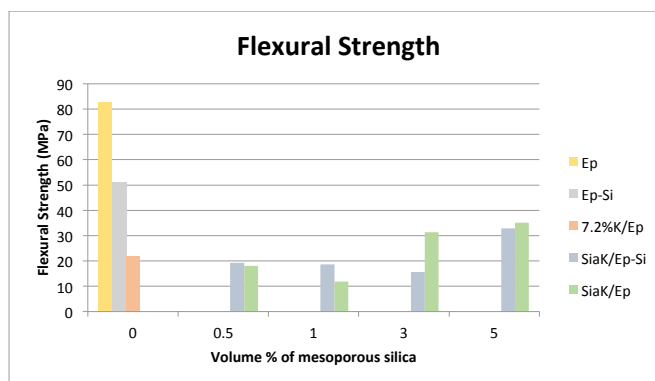


Figure 3. Flexural strength of 11 specimens produced

From Figure 3 above, blank epoxy is seen to possess the highest flexural strength. Adding silicon to blank epoxy weakens the flexural strength of the composite by 39%. Similarly, Inclusion of kenaf into blank epoxy weakens the flexural strength of the composite by 73%. From the earlier SEM analysis of Ep-Si, there is some de-bonding between epoxy and silicon, proving the reduced flexural strength of Ep-Si. The SEM analysis of 7.2vol% K/Ep reveals the presence of de-bonding and some areas of pulled out fibres. This is supported by Akil et al. (2011, pp. 4107-4121), in their journal article, who stated that adding kenaf into polymer matrix introduces a poor interfacial adhesion between the fibre and resin matrix. Polar hydroxyl groups on the surface of kenaf fibre do not easily bond with non-polar matrix.

Besides that, including kenaf into resin often results in agglomeration due to the lack of dispersion of the fibre itself, which is caused by the tendency of fibres to form hydrogen bonds

with each other. This will lead to poor properties in the final specimen produced. Meanwhile, adding mesoporous silica into 7.2vol% K/Ep decreases its flexural strength from 0.5vol% SiaK/Ep to 1.0vol% SiaK/Ep. However, there is an increment of 62% at 3.0vol% SiaK/Ep and further increment of the flexural strength at 5.0vol% SiaK/Ep. It can be concluded that mesoporous silica improves the flexural strength of 7.2vol% K/Ep composite excellently when 3.0vol% and 5.0vol% of mesoporous silica is added.

The inclusion of silicon into SiaK/Ep reduces the flexural strength gradually from 0.5vol% SiaK/Ep-Si to 3.0vol% SiaK/Ep-Si. There is a sudden increment of flexural strength at 5.0vol% mesoporous silica by 52%. Therefore, it can be said that silicon only increases the flexural strength at 5.0%vol SiaK/Ep-Si. Bajpai and Bajpai (2010, pp. 96-100) stated that higher silicon content in epoxy increases its hardness. However, in this experiment, the results reveal that the statement is only true before the optimum amount is added. By comparing the performance between SiaK/Ep and SiaK/Ep-Si, it can be seen that silicon improves the flexural strength of 0.5vol% SiaK/Ep-Si and 1.0vol% SiaK/Ep-Si but decreases the value at 3.0vol% SiaK/Ep-Si and 5.0vol% SiaK/Ep-Si.

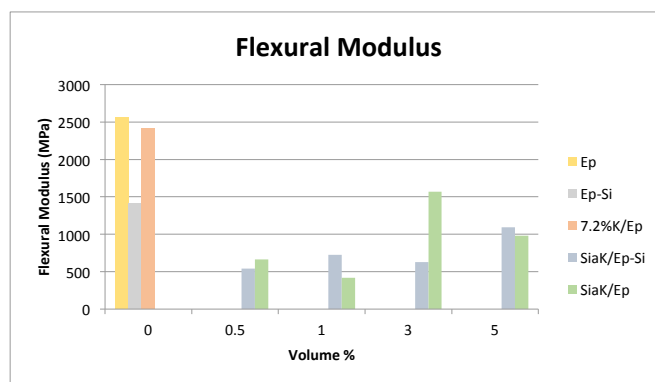


Figure 4. Flexural modulus of the 11 specimens produced

Figure 4 shows that blank epoxy exhibits the highest flexural modulus among the composites produced. When silicon is introduced into the blank epoxy, flexural modulus decreases by 45%. When kenaf is introduced into the blank epoxy, flexural modulus is reduced by only 6%. This is due to the well impregnation of epoxy into kenaf from Figure 2-D. Meanwhile, addition of mesoporous silica into 7.2vol% K/Ep shows two stages of decrement. Firstly, the flexural modulus decreases from 0.5vol% SiaK/Ep to 1.0vol% SiaK/Ep. Secondly, the flexural modulus decreases from 3.0vol% SiaK/Ep to 5.0vol% SiaK/Ep. Between 1.0vol% SiaK/Ep and 3.0vol% SiaK/Ep, there is a sudden increment of 73%. The highest value of flexural modulus is achieved by the 3.0vol% SiaK/Ep composite. The addition of silicon into SiaK/Ep composites gives an irregular trend. However, the values of all SiaK/Ep-Si do not differ much from the performance of all SiaK/Ep, except at 3.0vol% SiaK/Ep. Here, the addition of 3vol% silicon improves the flexural modulus drastically by 60%. Overall, it is shown in Figure 4 that 3.0vol% SiaK/Ep exhibits a higher flexural modulus than 7.2vol% K/Ep.

Table 1  
The parameters used in DMA

Properties	Type/Value
Clamp	Dual Cantilever
Temperature	30°C to 100°C
Ramp	5°C per minute
Oscillation Frequency	1 Hz

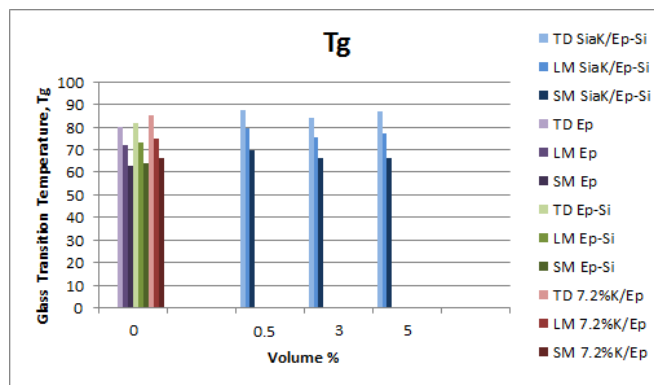


Figure 5. A summary of all the Tg values in a graph

Turi (1997) stated that the maximum values for all the three viscoelastic parameters [namely, Storage Modulus (SM), Loss Modulus (LM) and Tan Delta (TD)] can be used as the Tg of the materials. However, there are differences between these three values. Storage Modulus, which is also known as E' Onset, occurs at the lowest temperature. It is related to the mechanical failure of the composite. Loss Modulus or E Peak occurs at the middle temperature. It is related to the physical property of the composite that changes due to the glass transition. Tan Delta, which also known as Tan Delta Peak, occurs at the highest temperature. It is a good measure of Tg as it signifies the midpoint condition between the glassy and rubbery states of material. This value changes systematically with its amorphous content. Historically, Tan Delta Peak value is used as the Tg.

Figure 5 shows that the results obtained from DMA tally with the literature review conducted, where Tan Delta occurs at the highest temperature, Loss Modulus occurs at the middle temperature and Storage Modulus occurs at the minimum temperature. All the three Tg values show a similar trend, where Tg increases as silicon is introduced into the blank epoxy, whereas when kenaf is introduced into the blank epoxy, it gives a higher Tg value compared to Ep-Si. Moreover, when mesoporous silica is introduced into 7.2vol% K/Ep-Si, the Tg decreases from 0.5vol% to 3.0vol% and increases when 5.0vol% is added. This trend is also similar for all the Tg values obtained.

In order to simplify the explanation of the trend above, Figure 5 is plotted. When silicon powder is added to blank epoxy, an increase of 2.09% of Tg is obtained. Silicon, acting as a filler in the composite, is important in determining the Tg. When kenaf is introduced into blank

epoxy, Tg increases as much as 6.7%. Vinu et al. (2015, pp. 299-305) stated that including fibre and fillers into a material reduces the mobility of the polymer chains and increases the Tan Delta Peak value. Tsai and Hwang (2011) stated that mobility of polymer molecules is often reduced when fillers are added. This reduced mobility is caused by the confinement effects or the interaction between the particle and filler which then leads to increment in Tg.

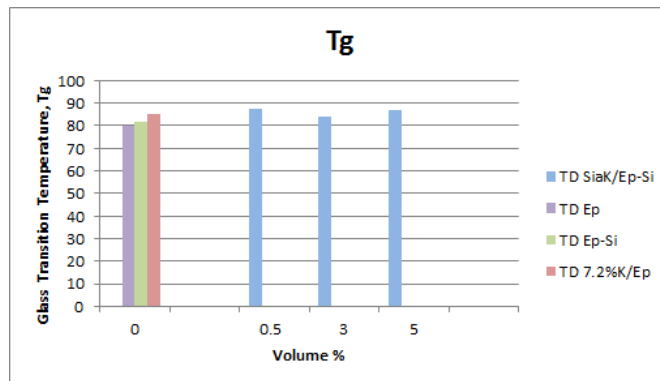


Figure 6. Tan Delta Peak values plotted

Figure 6 shows that the highest Tg obtained is 87.54°C, which is from 0.5vol% SiaK/Ep-Si composite. A reduction of 4.27% in Tg is noticed when 3vol% mesoporous silica is added to it. However, the Tg value increases by 3.63% when 5vol% mesoporous silica is added. Supposedly, as mentioned by Tsai and Hwang (2011), interaction between the particles and fillers causes a decrease in the mobility of polymer molecules, which leads to higher Tg. So as filler concentration is increased, more interaction will occur between the particles and fillers, thus higher Tg should be obtained. Similar to the experiment conducted by Sushko et al. (2014, p. 425704), as filler concentration is increased, there is a decrease of Tg value followed by continuous increment. According to them, this unexpected reduction of Tg is caused by acceleration of molecules in low filler concentration which has lesser entanglement.

Qiao et al. (2011, pp. 740-748) stated that agglomeration of fillers affects largely on the Tg of the composite produced. Higher filler concentration leads to higher chances of agglomeration. Thus, the low Tg obtained at 3.0vol% and 5.0vol% SiaK/Ep-Si is probably caused by improper dispersion of mesoporous silica in the composite. Vinu et al. (2015, pp. 299-305) mentioned that lower Tan Delta Peak values indicate better interfacial adhesion between fibre, filler and matrix. This improves the stress transfer and good load bearing capacity.

## CONCLUSION

Kenaf/epoxy composites have been fabricated using resin infusion method. The strength of kenaf/epoxy is obtained from the flexural test. The hybrid effects between kenaf/epoxy and mesoporous silica have been studied through the analysis of SEM. The specimens are produced with different vol% of fillers. 5vol% SiaK/Ep shows the highest flexural strength

obtained. The addition of mesoporous silica increases the flexural strength of 7.2vol%K./Ep from 22MPa to 35MPa. 3.0vol% SiaK/Ep shows outstanding flexural modulus value of 1569MPa compared to other compositions of SiaK./Ep and SiaK/Ep-Si. Thus, it is concluded that 3vol% of mesoporous silica is the optimum amount to be added in producing the highest flexural modulus value among SiaK/Ep and SiaK/Ep-Si.

The SEM analysis verifies that the interaction between epoxy and silicon is good in Ep-Si, revealing that the components are closely intact. 7.2vol% K/Ep shows that there are areas with pulled-out fibres. Nevertheless, there are areas where epoxy impregnates well within the kenaf, proving the existence of interfacial adhesion that leads to good mechanical properties. 0.5vol% SiaK/Ep-Si shows a better interaction between the kenaf and epoxy, with less area of pulled-out fibres. The tearing of fibres proves an excellent interfacial adhesion between the kenaf and epoxy.

The DMA result proves that mesoporous silica improves the Tg of the composite. The highest Tg obtained is from 0.5vol% SiaK/Ep-Si with the value of 87.54°C. However, Tg does not increase with the increment of mesoporous silica percentage in this experiment. Nevertheless, the composite consisting of epoxy, kenaf, mesoporous silica and silicon shows a higher Tg compared to blank epoxy and Ep-Si composites.

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