An Investigation of the Morphological and Tensile Properties of Vacuum Resin Impregnated Sugar Palm Fibers with Various Thermosetting Resins

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Sugar palm (*Arenga pinnata*) is a type of natural fiber that belongs to the Palmae family. It is versatile, readily available, and virtually the entire tree can be formed into many different products. This paper discusses the effect of vacuum resin impregnation on a single sugar palm fiber (SPF) using various thermosetting resins such as epoxy, vinyl ester (VE), and polyester (PE). The fibers were vacuum impregnated at a constant pressure of 600 mmHg for 5 min. The excessive resins were wiped off, and the impregnated fibers were cured in an oven for approximately 30 min at a temperature of 140 °C. Following this, the tensile properties of the single SPF impregnated with epoxy, VE, and PE were determined. The results indicated that impregnation of SPF with epoxy resin increased the tensile strength and modulus of SPF 50% and 59%, respectively. Scanning electron microscope images also illustrated that the epoxy resin offered better impregnation on the SPF compared to the other thermosetting resins. A suitable application for the impregnated SPF is as a roofing material.

Keywords: Sugar palm fiber; Single fiber; Thermosetting resin; Vacuum resin impregnation; Impregnation modification

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INTRODUCTION

Natural fibers are being used to reduce the demand for wood materials in the furniture and construction industries. Some examples of natural fibers are kenaf, pineapple leaf, oil palm, sugarcane bagasse, rice straw, banana, rice husk, and sugar palm (Bachtiar *et al.* 2010a; Ishak *et al.* 2013b). Natural fibers are widely available, low-cost, environmentally-friendly, renewable, and have low tool wear, which has made natural fibers an interesting material to explore and use (Doan *et al.* 2012; Ishak *et al.* 2013a). The *Arenga pinnata*, or commonly known as the sugar palm, belongs to the Palmae family and is a versatile crop that originated in tropical Asia. As shown in Table 1, the common name for the sugar palm varies based on where it is located. The sugar palm is known to produce sugar, similarly to sugarcane bagasse. The sap is harvested for its sugar and can be fermented to make wine and vinegar in Southeast Asia. Some additional products of the sugar palm are sugar palm juice, brown sugar, and bio-ethanol (Lasekan 2014). They most commonly grow in the wild of a primary or secondary forest. It has a maximum height of up to 20 m with a trunk covered by thick, black, and hairy fibers called ijuk (Sahari *et al.*

2012b; Rashid *et al.* 2017). The trunk is shielded by dense leaves with a white underside. There are multiple products made from sugar palm fibers such as carpets, brushes, paint brushes, clean water filters, septic tank base filters, ropes, door mats, chair or sofa cushions, and brooms (Ali *et al.* 2010).

Table 1. Common Names for *Arenga pinnata*

Native	Common Name
Malaysia	Sugar palm, *enau*, or *pokok gula kabung*
Saudi Arabia	Nakhlet es sukkar
Myanmar	Taung-ong
China	Sha tang ye zi or tang shu
Philippines	Kaong or black sugar palm
*Data taken from (Orwa *et al.* 2009)	

Polymers are classified into two categories: thermoplastic and thermosetting resins. Some examples of thermoplastics are polypropylene, polyethylene, polystyrene, polyvinyl chloride, polylactide, and polyether ether ketone. Examples of thermosetting resins are epoxy, polyester, melamine urea formaldehyde, phenol formaldehyde, melamine formaldehyde, vinyl ester, and resorcinol formaldehyde (Ali khudhur *et al.* 2013; Chalid *et al.* 2015). The polymers are reinforced with natural fibers or man-made fibers. The most commonly used thermosetting resin is epoxy because it is strong and is in a liquid state at room temperature.

Impregnation modification is a type of chemical treatment performed by infusing the cell wall of a fiber with a chemical to produce a material that has a filled cell wall and lumen. The main objective of the impregnation process is to improve the mechanical properties, dimensional stability, and to reduce vulnerability to biological attack. Ishak *et al.* (2013b) studied the impregnation modification of sugar palm fibers using unsaturated polyester and phenol formaldehyde. The impregnation process was completed under the constant pressure of 1000 mmHg at various impregnation times for up to 25 min and used phenol formaldehyde (PF) and unsaturated polyester (UP) *via* vacuum resin impregnation. Reductions were observed in the moisture content and water absorption for both PF and UP when impregnated for under 5 min, as well as an improvement in the mechanical properties (Ishak *et al.* 2013b). However, when the impregnation time was increased from 10 to 25 min, similar results were observed for both the hPF and UP. When comparing PF and UP, Ishak *et al.* (2013b) concluded that the fiber impregnated with UP offered better toughness, higher tensile strength, and higher elongation at break.

Sugar palm fibers impregnated with UP help to enhance interfacial bonding according to the results obtained from interfacial shear strength and single fiber pull-out tests (Ishak *et al.* 2013a). In the thermogravimetric (TGA) and Fourier transform infrared tests (FTIR), the impregnated sugar palm fiber showed a considerably lower reading compared with the unimpregnated fibers. The FTIR results indicated that decomposition of the fiber followed the sequence evaporation of moisture, hemicellulose, cellulose, lignin, and then ash. High interfacial bonding leads to improved tensile strength, tensile modulus, flexural strength, flexural modulus, elongation at break, and toughness. The optimum fiber content for the best aforementioned properties was found to be 30% (Ishak *et al.* 2013a).

There is a limited amount of studies conducted on the impregnation of sugar palm fibers. Therefore, the impregnation of the sugar palm fiber (SPF) will be compared with other types of natural fibers, *e.g.*, oil palm fibers. Having insight on the tensile properties of the fiber is important to foresee the properties of the composites and other fibers (Bachtiar *et al.* 2010b). In this study, the objective is to determine the tensile properties of a single sugar palm fiber impregnated with various thermosetting resins using a vacuum infusion method. This paper will discuss the tensile properties, morphology, and toughness of the studied sugar palm fibers.

EXPERIMENTAL

Materials

The sugar palm fibers were obtained from Kampung Kuala Jempol, Negeri Sembilan, Malaysia. Only mature sugar palm trees were selected to obtain the optimum properties. The fibers were taken at a height of 20 m at the flowering stage because the sugar palm trees produce large quantities of fibers during this stage. The fibres are separated by removing them from the trunk of the sugar palm tree. The material is then washed and soaked with distilled water in order to remove the impurities on the surface of the fibres. The fibres are then taken out of the container and dried for 24 hours at room temperature. Afterwards, the fibres are cut to a shorter length of approximately 15 cm. Epoxy, polyester (PE), and vinyl ester (VE) resins were supplied by Berjaya Bintang Timur Sdn. Bhd. (Cheras, Kuala Lumpur, Malaysia).

Impregnation of fiber

The fibers were treated using a vacuum impregnation process at Forest Product Division, Forest Research Institute of Malaysia (FRIM), Kuala Lumpur, Malaysia. First, the mature sugar palm fibers were cut into a shorter length of approximately 15 cm to ease the impregnation process. The cleaned fibres were placed into a 1000 mL beaker that is filled with impregnating resins, *i.e.* epoxy, VE, and PE, and then placed in a glass vessel. The glass vessel then underwent a vacuum process at for 5 minutes of impregnation time with a constant pressure of 600 mmHg. The vacuum in the vessel was then slowly released within 30 seconds. The purpose is to force resin to be absorbed inside the lumen and cell wall of the fibres. Once the fibres are impregnated with the resin, it is now a composite. The impregnated fibres are taken out from the beaker and excess resins was wiped off.

Curing of fiber

The impregnated fibers were proportionately laid on plates for the curing process. The oven temperature was set at 140 °C for approximately 30 min. After the 30 min, the cured fibers were removed from the plates and kept in an airtight vacuum bag for the mechanical testing.

Methods

Morphology Analysis

The morphology of the fiber was examined using a light microscope and scanning electron microscope (SEM) (S-3400N, Hitachi, Selangor, Malaysia) with an acceleration voltage of 10.0 kV for a clearer view of the surface of the fiber and its cross-section.

Single fiber tensile test

The single fiber was tested in accordance with the ISO 5079 (1995) standard. A total of five specimens were prepared for each impregnated fiber, including the unimpregnated specimen as the control. The fibers were labelled as control SPF, SPF/Epoxy for the fiber impregnated with epoxy, SPF/VE for the fiber impregnated with VE, and SPF/PE for the fiber impregnated with PE. The diameters of the fibers were determined using a light microscope at five different cross-sections. The tensile testing was performed at room temperature (23 °C) with a relative humidity of 50%. The test used an Instron universal testing machine (Selangor, Malaysia) with a load capacity of 5 kN and a gauge length of 20 mm.

RESULTS AND DISCUSSION

Fiber Morphology

The effects of different impregnation agents (Epoxy, PE, and VE) on the surface appearance of the SPF are shown in Fig. 1. The light microscope images of a single SPF show the structure of fibers before and after the vacuum impregnation process. Figure 1(a) is the image of the sugar palm fiber before the impregnation process (control SPF), while Figs. 1b through 1d are the images of the impregnated single SPF with epoxy, PE, and VE, respectively. As shown, the control fiber had more visible pores on the surface than the impregnated fibers. During the impregnation process, the applied pressure forced the resin to penetrate the empty spaces, *i.e.*, the lumen and cell wall of the sugar palm fibers. The resins penetrated the lumen and cell wall *via* macrofibrils that were present at both ends of the fibers. Additionally, microvoids that were available on the surface of the fiber were penetrated by the resin. The filling of voids, pores, and lumens in the fiber *via* the vacuum resin impregnation allows for a higher and more effective interfacial interaction between the surface of the fiber and the matrix, if used as reinforcements in the polymer composites (Munawar *et al.* 2018). Moreover, natural fibers tend to be hydrophilic, and the impregnation process also inhibits the moisture absorption tendencies of the fibers. This characteristic is achieved as the potential sites of the fibers for water absorption are coated with the hydrophilic resins (Ishak *et al.* 2013b).

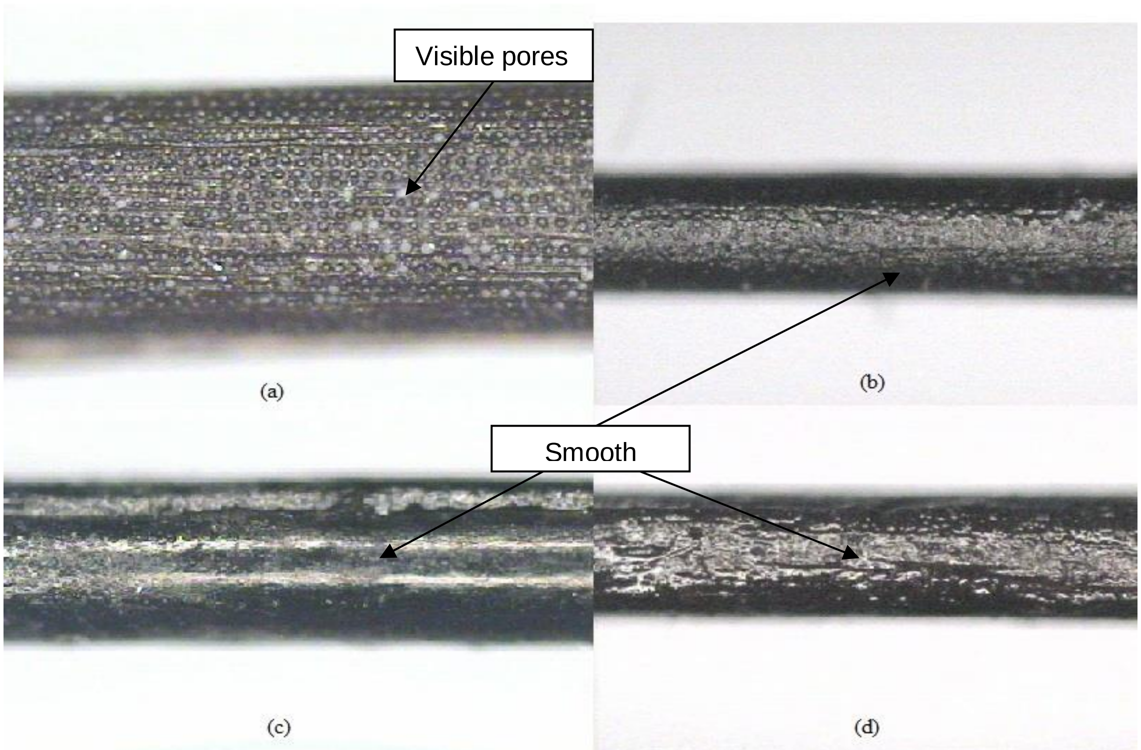

Fig. 1. Light microscope (magnification ×5) images of (a) unimpregnated fiber, (b) SPF/Epoxy, (c) SPF/PE, and (d) SPF/VE

Figure 2 shows the SEM images of the cell lumen of the impregnated SPF/PE and impregnated SPF/VE. The PE did not fill all the lumens in the fiber. Figure 2(b) shows the presence of silica and shows the surface of the fiber where pores are visible, similarly to the light microscope images in Fig. 1(a). There were some impurities covering the surface of the fiber, namely silica, peptins, and other impurities, which are shown in Fig. 2. The surface of the fiber was not smooth and contained irregular stripes and nodes. In general, the pores and holes on the surface may be beneficial for the mechanical properties, if there are effective fiber-matrix interactions, which is the site of the mechanical interlocking between the fiber and the matrix (Bachtiar *et al.* 2010b). The impregnation of resin into the fiber creates a more heterogeneous structure where fibers are being coated and infused with resin, as shown in Figs. 1 and 2 (Edhirej *et al.* 2017).

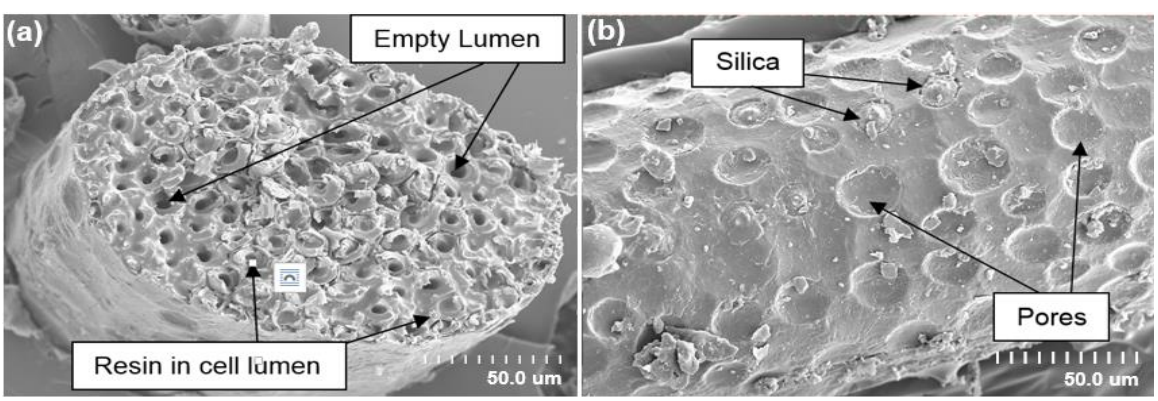

Fig. 2. SEM images of (a) SPF/PE and (b) SPF/VE

Tensile Stress-strain Relationship

The stress-strain behavior of the impregnated sugar palm fibers tested is shown in Fig. 3. The graph shows that stress and strain increased linearly up to the point where the fiber reached its maximum tensile stress and fiber breakage occurred. The point where maximum tensile stress happens is called the ultimate tensile strength. However, the strain value at that point was collected to identify the elongation at break that occurred. The fiber impregnated with epoxy exhibited the highest strength followed by SPF/VE, SPF/C, and SPF/PE. These results indicate the superiority of epoxy over PE and VE, as reported by Bennett-Huntley (2014). The author further explained that epoxy has aromatic groups that caused it to be far stronger than VE and possess higher thermostability, higher ability to bond with various substrates, and to be able to absorb larger impact. In addition, the results also indicated that VE also has the potential of enhancing the fiber properties while PE impregnation showed the least strength. PE has lower ability to bond with other material compared to VE and Epoxy (Johnson 2018).

Low stress values demonstrate poor strength of the fiber and indicate that the fiber is not suitable for high force action. A prominent change in the properties of the fibers was observed after the impregnation process using thermosetting resins was employed. Ishak *et al.* (2012) found that SPF obtained from a height of 1 m to 3 m has the lowest strength when compared to the fibers collected from a height of 9 m to 15 m, because the fibers undergo a degradation process. The maximum tensile strength is offered by the fiber from the height of 11 m. The higher strain observed in the graph indicated the ductility of the fiber. The SPF with the highest stiffness based on its location follows this sequence: sugar palm frond, sugar palm bunch, ijuk, and sugar palm trunk, with the trunk having the lowest stiffness (Sahari *et al.* 2012a).

The lignocellulosic material present in the SPF can affect its tensile properties. The main components of lignocellulosic material are lignin, cellulose, and hemicellulose. A higher cellulose content enhances the tensile properties of the fiber (Ishak *et al.* 2012). Cellulose is the main structural element and provides the mechanical properties of the fiber. Hemicellulose is like cellulose but contributes less to the strength of the fiber. These elements are present in lower amounts when collected from a lower height on the sugar palm tree. The function of hemicellulose is to bind microfibrils for use as structural reinforcements (Ishak *et al.* 2012).

Impregnating the SPF with the respected resins caused the resin molecules to settle in both the cell wall and the cell lumen, as well as coat the surface of the fiber. The process produced a larger resin-to-resin contact area. The resin embedded on the surface of the fiber acted as an interphase layer and thus improved the fiber-matrix stress transfer. This enhances the interfacial bonding and the strength of the impregnated fiber (Ishak *et al.* 2013a).

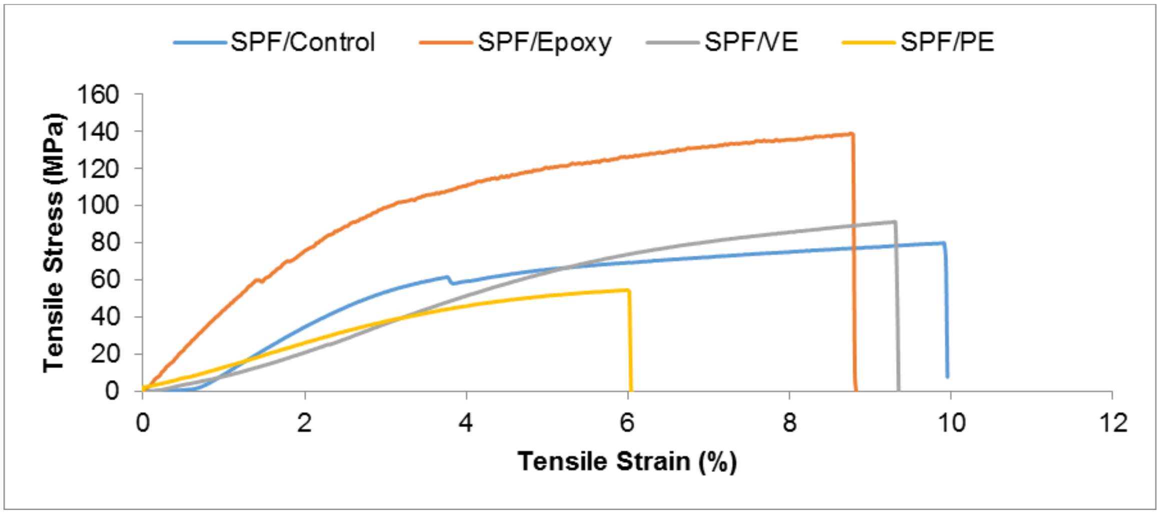

Fig. 3. Typical stress strain curves

Tensile Strength

A total of five fibers were tested for each type, and their average tensile strength reading was recorded. Figure 4 shows that the SPF/Epoxy exhibited the highest tensile strength with 112 MPa followed by SPF/VE (85 MPa), SPF/C (75 MPa), and SPF/PE (47 MPa). There was approximately a 43% increase in the tensile strength for the fiber impregnated with epoxy, while the fiber impregnated with VE increased only 13%. In addition, the fiber impregnated with PE showed a decrease in tensile strength of 37%. The improved strength is attributed to the resin embedded inside the fiber cell wall and cell lumen, which reinforces the structure of the fibers (Ishak *et al.* 2013b). In literature, the impregnation process additionally shows improvement in the tensile strength. Sahari *et al.* (2012a) suggest that the location that the sugar palm fibers are collected plays a role in their tensile strength.

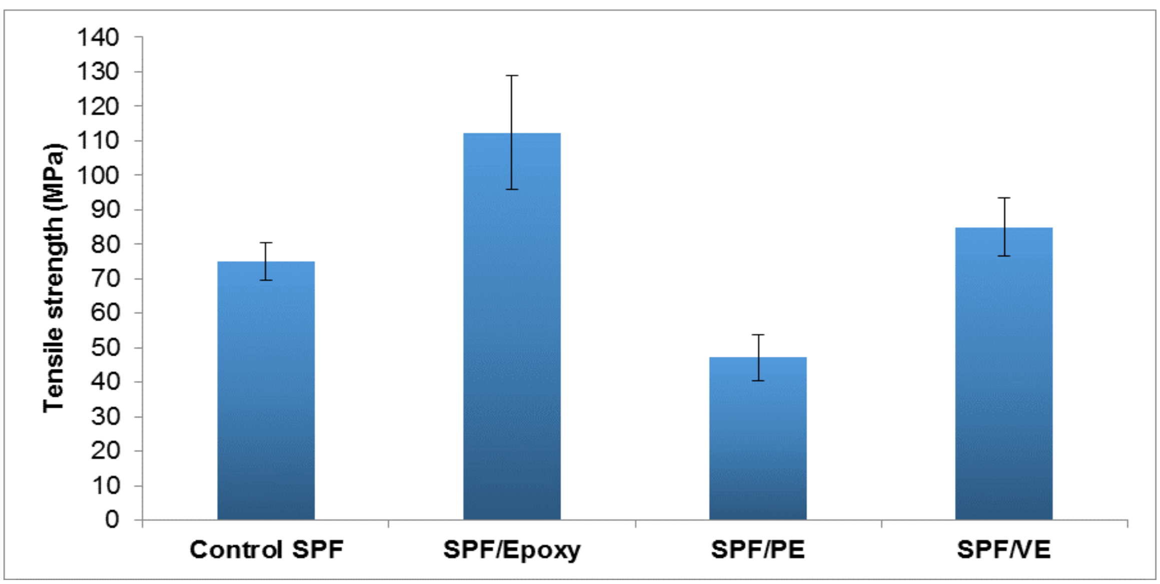

Fig. 4. The tensile strength of a single SPF with various thermosetting resins

Tensile Modulus

The tensile modulus indicates the capability of a fiber to resist deformation if stress is applied, or in other words, the stiffness of the fiber. Figure 5 shows that the SPF/Epoxy possessed the highest tensile modulus value of 31 GPa followed by SPF/C (20 GPa), SPF/VE (17 GPa), and SPF/PE (13 GPa). The tensile modulus of the fiber impregnated with epoxy increased 59%, while the tensile modulus of SPF/PE and SPF/VE decreased 34% and 11%, respectively. There was a considerable difference in the tensile strength and the tensile modulus between those three impregnation agents. The results in Fig. 5 show a similar trend as shown in Fig. 4. A fiber with poor tensile properties results in a reduction in the stiffness (Ishak *et al.* 2012). An increase in the tensile modulus showed that the resin found in both the cell lumen and cell wall resulted in a stiffer fiber. A previous study by Bachtiar *et al.* (2010b) reported that as the diameter of the fiber increases, the lumen size increases. Thus, it additionally lowers the stress at break and the tensile modulus. The SPF obtained from lower heights has a lower tensile modulus when compared with fibers obtained at heights of 7 m and above (Ishak *et al.* 2012). In addition, the study found that the amount of lignin in the fiber affects the magnitude of the tensile modulus. A higher lignin content produces fiber with a higher tensile modulus. The value of the tensile modulus obtained from different locations are reported as follows: sugar palm frond (10 GPa), sugar palm bunch (9 GPa), ijuk (6.9 GPa), and sugar palm trunk (3 GPa) (Sahari *et al.* 2012a).

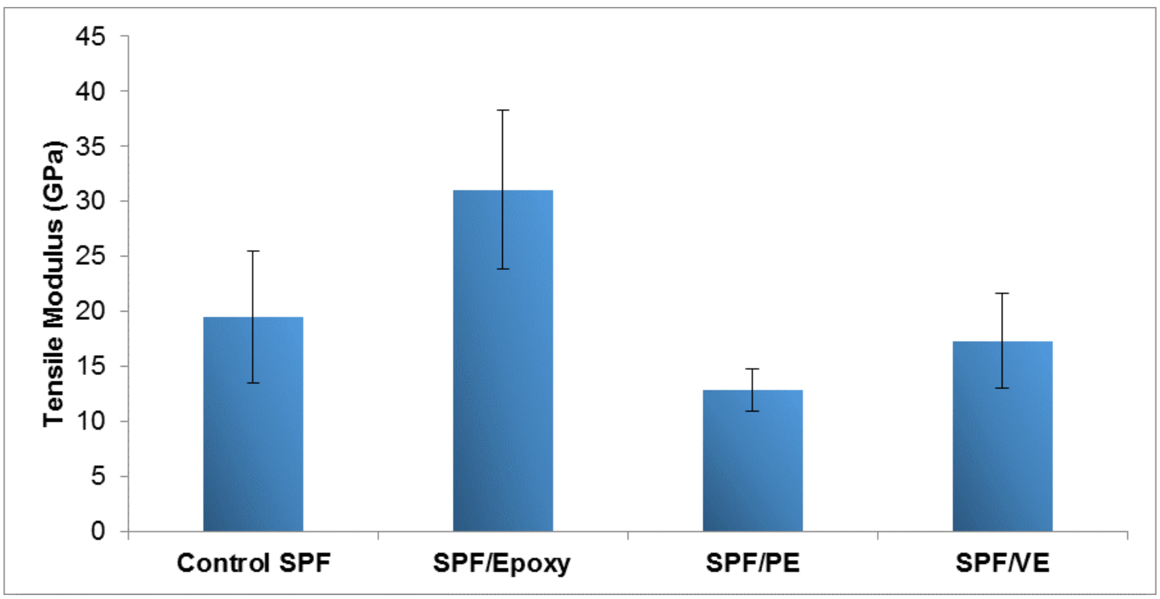

Fig. 5. The tensile modulus of a single SPF with various thermosetting resins

Elongation at Break

The elongation at break demonstrates a different type of fiber property in comparison with the tensile strength and the tensile modulus; it indicates the flexibility of a fiber. The maximum elongation of 11% was observed by the control SPF, followed by 10% for both the SPF/VE and SPF/Epoxy, and 5% for SPF/PE, as shown in Fig. 6. The properties of each resins affect the elongation at break of the impregnated fibre. Epoxy resin is superior compared to PE and VE. This explains why SPF/Epoxy constantly has better results than the other two (Bennett-Huntley 2014). Furthermore, fibers with a poor strength broke more quickly than fibers with a higher strength. This was observed in the comparison of elongation at break with tensile strength, where the SPF/PE showed the lowest tensile strength and lowest elongation at break. When a certain amount of stress was applied, the fiber did not elongate and broke off instead. The decrease in strain values was attributed to both the increased stiffness of the fibers and to the varied diameter found along the fiber. The non-homogenous nature of the SPF contributes to inconsistent diameter of the fiber diameter throughout its length, as shown in Fig. 7. Moreover, the impregnation process altered the behavior of the sugar palm fiber by reducing the ductility of the fiber. Another factor that may have contributed to the obtained results is the size of lumen. Larger lumen size in the fibers means that more resin can be taken up, resulting in the increased stiffness of the impregnated fibers. The PE impregnated fiber experienced a 58% drop in its elongation at break compared to the SPF/C. The thermosetting resins have a brittle nature that affects the elongation at break (Ishak *et al.* 2013b). A previous study found that SPF has a higher strain value of 20% (Bachtiar *et al.* 2010b). In addition, fibers obtained from sugar palm trees at lower heights (*e.g.*, 1 m) produced a minimum elongation at break (6%) compared to heights of 5 m (28%) and above. The optimum value is obtained from a height of 7 m with an elongation at break of 28% (Ishak *et al.* 2012).

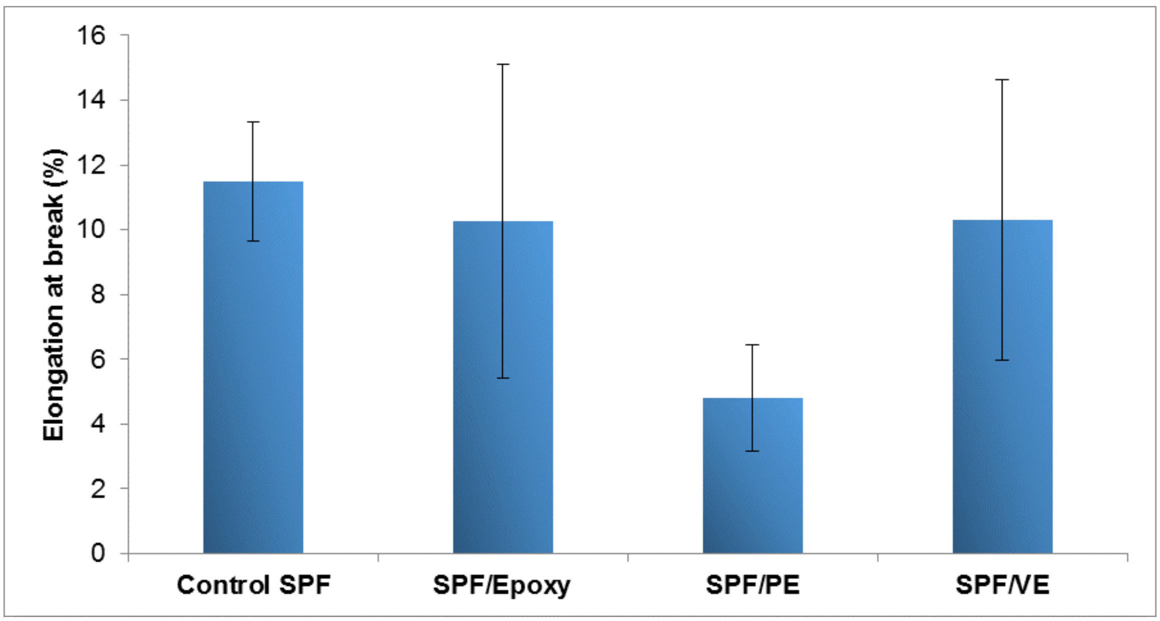

Fig. 6. The elongation at break of a single SPF

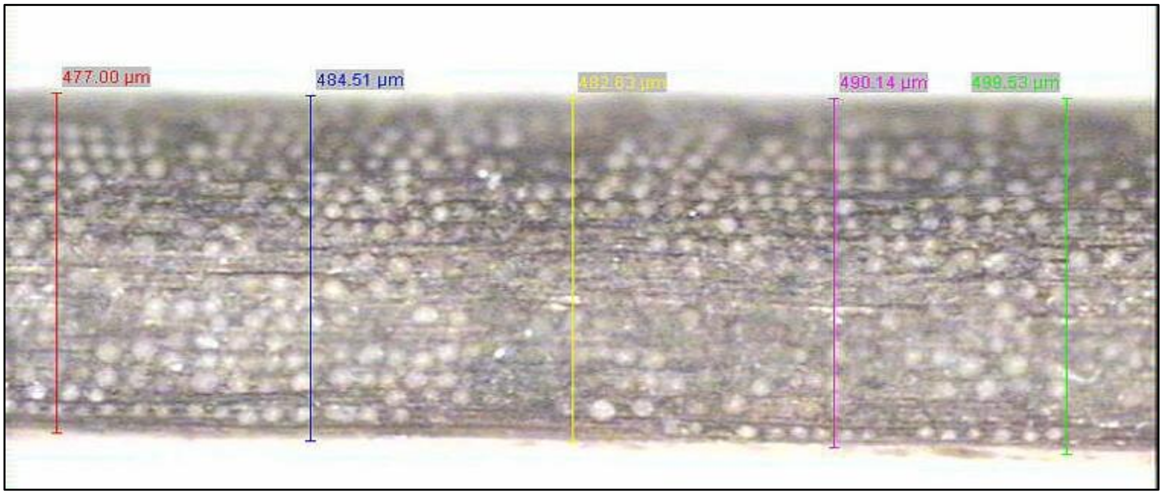

Fig. 7. The diameter calculation of a single SPF

Toughness

The toughness of the fibers was calculated based on the area under the stress-strain curve. A poor tensile strength indicated poor toughness. Toughness followed a similar trend when compared to tensile strength and tensile modulus, where SPF/Epoxy had the highest value followed by the control SPF, SPF/VE, and SPF/PE. The toughness of the fiber, which is represented by the amount of energy it can absorb before mechanical failure of the fiber occurs, increased with the infusion of epoxy in SPF. However, PE and VE impregnation reduced the toughness as compared to SPF/C, demonstrating that a lower amount of energy is absorbed by the SPF/PE and SPF/VE in comparison to SPF/C before failure occurs (Ishak *et al.* 2013b). Sugar palm fiber obtained from a height of 11 m exhibit a higher toughness compared to SPF obtained at 1 m (Adawiyah *et al.* 2013). The peak toughness of 889 MJ/m^3 was displayed by the SPF/Epoxy followed by the control SPF (553 MJ/m^3), SPF/VE (497 MJ/m^3), and SPF/PE (205 MJ/m^3). There was a considerable increase in the toughness of the SPF impregnated with the epoxy resin. This further demonstrated the effectiveness of the impregnation modification to increase the possibility of utilizing SPF in a wider range of applications.

CONCLUSIONS

In this study, the tensile properties of single SPF were investigated after impregnation with epoxy, PE, and VE. The treatment of the fibers *via* an impregnation modification gave a better result compared to the untreated fibers. The summarized findings of this study are listed as follows:

1. The impregnation process resulted in an improved morphology of SPF, making it more suitable for use in polymer composites. The filling of voids, pores, and lumens in the fiber via the vacuum resin impregnation enhances the surface morphology of the fibers and provides an optimum fiber-matrix bonding interface.
2. The SPF/Epoxy offered a greater tensile strength and tensile modulus than the SPF/VE, SPF/C, and SPF/PE. SPF/C acted as the control specimen as it did not undergo any impregnation process.

3. The elongation at break of the SPF/Epoxy and SPF/VE did not show much difference in comparison to the control SPF. However, the SPF/PE displayed a reduction in the elongation at break.
4. The epoxy impregnation also enhanced the toughness of the SPF.
5. The type of resin had a considerable influence on the properties of the sugar palm fibers, especially when impregnated with epoxy.

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