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The Effect of Reinforcing Moringa Oleifera Bark Fibre on the Tensile and Deformation Behaviour of Epoxy and Silicone Rubber Composites

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ABSTRACT

The moringa oleifera bark (MOB) is well-known for its medicinal properties and various benefits, where combining it with polymers could produce a new superior composite material for medicinal applications. Because this is a novel composite material, even basic information on how the MOB fibres altered the tensile properties of epoxy and silicone rubber is still lacking. Therefore, this study investigated the tensile and deformation behaviour of two newly introduced composite materials, MOB fibre reinforced into epoxy and silicone rubber. ASTM D3039 and ASTM D412 were adapted to prepare the hard and soft composite specimens (0, 4, 8, 12 and 16wt%.), respectively. T-test was conducted to determine the significant difference. The results show that the tensile modulus of MOB-epoxy biocomposite improved from 1240 MPa to 1668 MPa (35% increment) when the fibre content was increased to 16wt%. For MOB–silicone biocomposite, a similar trend was observed where the tensile modulus also increased from 0.076 MPa to 0.12 MPa (64% increment) as the fibre concentration increased from 0 to 16wt%. In conclusion, reinforcing MOB fibre affected the stiffness of silicone rubber more than epoxy; but affected the elongation of epoxy more than silicone rubber. Based on a t-score of 17.5, a significant

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ISSN: 0128-7680 e-ISSN: 2231-8526 difference is observed in how reinforcing MOB at various wt% affected the increment of tensile modulus for both hard and soft composites. Finally, the determined tensile modulus compared to other materials could be useful for benchmarking and exploring potential applications.

Keywords: Biocomposite material, epoxy and silicone rubber, moringa oleifera barks, tensile properties

INTRODUCTION

Different composites have been produced and utilised for various purposes for decades. In 1500 B. C., the Egyptians and Mesopotamians manufactured bricks out of mud reinforced with straw to build strong, durable buildings (Keya et al., 2019). Another ancient composite which had been developed was concrete, a mix of small stones or gravel with cement. It gave a good compressive strength to the materials, which was suitable for the building sector. The revolution of composites manufacturing has developed over decades to meet specific requirements in different fields of applications. Composite materials have been widely used in many engineering applications such as in the domestic sector, automotive sector, aircraft industry and building industry (Bharath & Basavarajappa, 2016; Ahmed et al., 2021; Tataru et al., 2020). Many composite materials have considerably higher strength qualities than traditional metallic materials, allowing them to be lighter and stronger than individual materials. Because of these significant characteristics, many sectors have begun to rely on composite materials, and the development of composite materials has begun to accelerate. The research was done to improve materials by reinforcing the material with fibre, with either synthetic or natural components.

Over the past few years, the demand for synthetic fibres has risen in composite manufacturing, which has resulted in environmental sustainability issues such as climate change and pollution. Therefore, numerous studies have focused on converting synthetic to natural fibres as reinforcing materials to alleviate this problem. Natural fibres composite materials are lightweight, abundant, biodegradable and cost-effective (Maria, 2013). They may be used to replace synthetic fibres with bio-based materials that are both safer and more ecologically friendly. Among the known natural fibres are jute, hemp, flax, sisal and kenaf (Jusoh et al., 2016).

A composite material signifies combining two or more materials at a macroscopic scale to form a third useful material (Jones, 1999). A biocomposite is a material composed of two or more distinct constituent materials (one being naturally derived) combined to yield a new material with improved performance over individual constituent materials (Rudin & Choi, 2013). Since this study combines moringa oleifera bark (MOB), a natural resource, with the polymer matrix material, these new materials are introduced as biocomposites. The main idea of green materials is to address environmental concerns, which has led to the rigorous development of green composite materials among researchers. Surprisingly, green material has gained attention and is widely researched for various applications such as in technical applications, including automotive, home, building, aviation, structural and circuit boards (Bharath & Basavarajappa, 2016; Ahmed et al., 2021; Tataru et al., 2020). Within the last few decades, biocomposite materials have offered more significant sustainability, industrial ecology, eco-efficiency and green chemistry than conventional applications (Bharath & Basavarajappa, 2016). These fibres are renewable resources that can absorb CO_2 and restore oxygen to the atmosphere with little or no energy usage (Pickering et al., 2016). It is a safe material selection for composite products in the long term.

Earlier studies have documented on investigations of various barks, such as poplar bark, Dirca L. bark and black spruce bark (Safdari et al., 2011; Hudson et al., 2021; Yemele et al., 2010). This study has focused on combining MOB into the polymer. The moringa oleifera tree is well-known for its medicinal properties. It is a cultivated member of the Moringaceae monogen family, also known as the miracle tree (Rockwood et al., 2013). Its bark has been scientifically proven to have certain significant virtues, including the potential to act as a bioremediation agent, removing heavy metals from polluted water (George et al., 2016). It also has antibacterial properties that can help break down bacterial infections such as Staphylococcus aureus, Citrobacter freundii, Bacillus megaterium and Pseudomonas fluorescens (Zaffer et al., 2014). Additionally, the bark possesses anti-inflammatory properties and can treat fever, dyspepsia (discomfort digestive system) and splenomegaly (infection in the lymphatic system) (Padayachee & Baijnath, 2020). Therefore, these remarkable features have encouraged the idea of creating novel material composites by reinforcing the MOB into epoxy and silicone rubber matrices.

In composite materials, the reinforcement acts as a load bearing and contributes critically to stress transfer and strengthening the composites while the matrix material binds and protects the reinforcing material when an external load is exerted on the composite (Manu et al., 2022). The mechanical characteristics of composites are influenced by various factors, including the properties of the matrix, the properties of the fibers, and the adhesion between the fibers and the matrix. In general, achieving excellent mechanical properties in composites relies heavily on the crucial aspect of fiber-matrix adhesion (Arbelaiz et al., 2020). Matrix materials in composites are divided into two categories: thermoset polymers and thermoplastic polymers. The research of thermoset polymers as matrix materials such as silicone and epoxies has been growing due to their wide range of applications in various sectors (Bahrain et al., 2022; Alshahrani & Arun Prakash, 2022). Ali Raza et al. (2012) studied the properties of two composites; carbon black reinforced with rubbery epoxy (CB/ RE) and carbon black reinforced with silicone (CB/ silicone). The results showed that the compressive strength of CB/silicone is 1.68 times lower than that of CB/silicone, thus suggesting that CB/silicone composites is more compliant than the CB/RE composites. However the author recommended that the CB/RE composites is a promising adhesive for electronic packaging applications. In a study by Atmakuri et al. (2022), the author reported that hemp and flax fiber reinforced with epoxy composites has two times greater mechanical properties compared to the fiber reinforced with ecopoxy composites. With the improved mechanical properties of the composites, the hemp and flax fiber reinforced with epoxy matrix composites are potential to replace the synthetic fiber composites in automobile, construction, micrfluidics and biomedical industries.

This research introduces two newly developed biocomposite materials, MOB-Epoxy and MOB-silicone biocomposites, which makes it novel since there has been no reported studies on the reinforcement of Moringa Oleifera bark fibers into silicone rubber. The tensile and deformation behaviour of these two recently developed composite materials was mainly examined in this work, including addressing research questions on how MOB fibres affect the tensile properties of polymers and the potential applications for such biocomposites. The tensile properties of both biocomposites were quantified, analysed, and compared in this work to show how MOB fibre content impacts the stiffness and deformation of the biocomposites.

MATERIAL AND METHODS

Raw Material and Matrix

The stem of Moringa Oleifera was supplied by GV Medhini Consultancy & Resources Sdn. Bhd. was harvested in Kota Kemuning, Selangor, Malaysia. The epoxy resin (Mirapox MH956) and silicon matrix (Ecoflex 00-30 Platinum Cure Silicone) were purchased from Miracon Sdn. Bhd. and Castmech, respectively.

Specimen Preparation

Moringa Oleifera (MO) stems were cut into 10 cm lengths, and the bark was peeled off. The bark was rinsed with tap water to eliminate impure particles before being dried in a 100° C oven for 24 hrs. It was crushed into small chip particles using a crusher machine and milled at 270 rpm for 30 seconds 4 times using a planetary mono mill to transform it into powdery form. Lastly, it was sieved at 150 µm in size to produce a uniform fine powder.

The specimens were produced at five different fibre compositions, 0wt%, 4wt%, 8wt%, 10wt%, 12wt% and 16wt%. Each fibre composition was fabricated with five specimens. The powder weight was measured to fabricate the exact fibre-matrix content solution. The solution was mixed under continuous stirring and performed in a specified lab environment. The mixture solution was moulded into respective moulds and hardened at room temperature for 24 hrs for MOB-Epoxy and 4 hrs for MOB-Silicone (MOBSil).

Mechanical Testing: Uniaxial Tensile Test

Tensile tests followed the American Society for Testing and Materials (ASTM), D3039 for MOB-Epoxy (Batu & Lemu, 2020) and D412 for MOB-Silicone (Bahrain & Mahmud, 2019), respectively. The specimen dimensions for both materials were as shown in Figure 1. Each specimen was evaluated using a 3383 Universal Testing Machine 100kN (Instron, United States of America, 2008) at a 500 mm/min speed. Tensile modulus referred to the stiffness of the materials, and the values were generated from the machine for each specimen once it reached the failure point. Therefore, these values were recorded for analysis.



Figure 1. Specimen dimensions in millimetres (mm) for (a) MOB-Silicone (ASTM D412) and (b) MOB-Epoxy (ASTM D3039)

Statistical Analysis: Mean, SD, Var, Pair t-Test

Statistical analysis was also executed in this study to determine the mean, standard deviation and variance values. This analysis was also conducted to assess the reliability and accuracy of the data gained from the experiment. In order to compare the tensile properties of both materials (MOB-Epoxy and MOB-Silicone), a statistical analysis named t-test analysis was conducted. A t-test analysis aims to describe the significant difference between the means of the two groups concerning the tensile modulus of both materials. It required three key data values: mean values, variance, and the number of data values of each group. Equation 1 was used to calculate the t-score to define the differences between both materials.

$$t = \frac{|\bar{x}_1 - \bar{x}_2|}{\sqrt{\frac{S_1^2}{n_1} + \frac{S_2^2}{n_2}}}$$
[1]

Where, \bar{x}_1 and \bar{x}_2 are the mean value of the tensile modulus of MOB-Epoxy and MOB-Silicone, S_1 and S_2 are the variance value of the tensile modulus of MOB-Epoxy and MOB-Silicone, respectively, and n_1 and n_2 , are the number of observations for each material.

The study used the Data Analysis feature in Excel software to obtain the t-score value. It was noted that the higher values of the t-score indicated that a large difference existed between the two sample sets. The smaller t-score indicated that more similarity existed between the two samples. Therefore, this test was conducted to identify and observe the tensile properties of Moringa oleifera bark-epoxy biocomposite and Moringa oleifera bark-silicone biocomposite specimens.

RESULTS AND DISCUSSION

Moringa Oleifera Bark – Epoxy (MOB-Epoxy) Biocomposite

Figure 2 highlights the tensile behaviour of pure epoxy and MOB-Epoxy biocomposite specimens under uniaxial tension.



Figure 2. Average tensile stress-strain curves of MOB-Epoxy biocomposite

As illustrated in Figure 2, the trend observed showed that the pure epoxy specimen occupied the lowest curve (red line), thus indicating that the pure epoxy specimen was the least stiff compared to specimens with MOB fibre. By observing the curves, the slopes showed a gradually increasing trend, and the curves became more linear as the MOB fibre content increased. This finding is supported by the tensile modulus value summarised in Figure 3. The tensile modulus of the specimen gradually increased along with the further inclusion of fibre content from 1240.5 MPa to 1668.5 MPa. This increment indicated the enhancement of the properties of pure epoxy when adding the Moringa oleifera bark fibre as the reinforcement material. The low MOB content in the epoxy matrix made it strain more to reach the fracture point, where the 0wt% specimens had the highest maximum strain values followed by 4, 8, 12 and 16 of weightage (Table 1). In general, the material's properties were proven to have improved its tensile modulus. The maximum strain value was found to be 2.47 for pure epoxy, and this was reduced to 0.86 at 16wt% fibre content, in agreement with the finding in Kumar et al.'s study (Kumar et al., 2021). Both soft and hard composites became stiffer due to the reinforcement. It is due to the inclusion of filler in the composite to fill up the spaces between the chains, resulting in lower polymeric

chains mobility of the composite. Therefore, filler inclusion in the composite makes it stiffer and more resistant to deformation as it resists the movement of the polymer chain within the composite, as in agreement with previous studies (Benevides & Nunes, 2015; Ismail et al., 2015).



Figure 3. The average tensile modulus of MOB-Epoxy biocomposite

Table	1

Tensile properties (average value) of MOB-epoxy specimens

Weightage (%)	Tensile Modulus, E (MPa)	Maximum strain, ε (%)
0	1240.5	2.473
4	1312.1	1.834
8	1507.5	1.288
12	1617.3	1.197
16	1668.5	0.862

Table 2 compares the results obtained from this study with other studies. Similar to MOB-silicone biocomposite, Table 2 showed that the tensile stiffness of the current material, MOB-Epoxy biocomposite was comparable to Graphene nanoplatelets/epoxy composites and, thus, may open up ideas for new applications (Kilic et al., 2019).

Table 2

Comparison of tensile modulus, E Value between previous studies and the current study based on tensile tests

Tensile Modulus, E	Material Type	Reference
13.1 GPa	Micro-Bismuth (III) oxide (Bi2O3) epoxy composite	Muthamma et al. (2021)
3.6 GPa	Kevlar/Date palm epoxy hybrid composite	Muthalagu et al. (2020)
1.9 - 2 GPa	Kenaf epoxy biocomposite	Fauzi et al. (2016)
1.89 - 1.45 GPa	Para-aramid fibres (Kolon) epoxy composite	Obradović et al. (2021)
1.7 GPa	Graphene nanoplatelets/epoxy composites	Kilic et al. (2019)
1312 - 1668 MPa	Moringa Oleifera bark—epoxy biocomposite	Current study
577–645 MPa	Phoenix sp. fibre-reinforced epoxy composites	Rajeshkumar et al. (2017)
124 - 223 MPa	Alkali-treated bamboo fibre/epoxy	Huang and Young (2019)
113 -163 MPa	Untreated bamboo fibre epoxy composite	Huang and Young (2019)

Moringa Oleifera Bark – Silicone (MOBSil) Biocomposite

Figure 4 illustrates the average tensile stress-strain curve of MOB-Silicone specimens for every composition of fibre content (0wt%, 4wt%, 8wt%, 10wt%, 12wt% and 16wt%).



Figure 4. Tensile stress - Strain curves of MOB-silicone biocomposite

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From the stress-strain curve, pure silicone rubber (0wt%) had the highest strain as it strained more than other specimens (reinforced with MOB fibre at 4, 8, 12, and 16wt%). Additionally, the uniaxial tensile load caused the pure silicone specimen to exhibit a highly nonlinear elastic behaviour (the red line in Figure 4). It occurred due to the weak bonding of the polymer chain with silicone rubber (Sarath et al., 2020). However, when the fibre content climbed from 0 to 16wt%, the curve trend began to lessen its nonlinear behaviour, resulting in a more linear curve at 16wt%. Due to the presence of Moringa oleifera bark fibre as a filler, the curve gradually became more linear as it enhanced the tensile properties of silicone rubber, as supported by the tensile modulus value illustrated in Figure 5. In terms of strain, it could be observed that adding fibre into the silicone rubber had affected the strain behaviour as it started to decrease in elongation to the failure point from the specimen of 0 until 16wt%, as summarised in Table 3.



Figure 5. The average tensile modulus of MOB-silicone biocomposite

Weightage (%)	Tensile Modulus, E (MPa)	Maximum Strain, ε (%)
0	0.076	0.1007
4	0.080	0.1099
8	0.089	0.0993
12	0.100	0.0914
16	0.120	0.0801

 Table 3

 Tensile properties (average value) of MOB-silicone specimens

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From Table 3, the tensile modulus of the specimen increased as the fibre content increased. The lowest tensile modulus could be seen at 0wt% with a value of 0.076 MPa, and the specimen of 16wt% recorded the highest with a value of 0.12 MPa. Therefore, this study proved that adding MOB fibre caused silicone rubber to have better resistance to deformation.

Halim et al. (2022) also reported a similar trend in which adding the Silica Aerogel (SA) into the room temperature vulcanised silicone rubber (RTV-SiR) reduced the tensile strain of the biocomposite (this addition, thereby forming SA/RTV-SiR) compared to pure RTV silicone rubber. The result also showed increasing tensile modulus values from 0.74 MPa for the RTV-SiR sample to 1.34 MPa for the SA/RTV-SiR sample. The presence of fibre was found to increase the stiffness of the silicone rubber. Similar behaviour had also been reported by Koushki et al. (2020), who reinforced hemp fibre into silicone rubber was increased with further addition of fibre content, and its elongation at break was gradually decreased.

However, a study by Kumar et al. (2022) reported that the increased filler content in RTV silicone rubber had caused increased tensile strain but with an increased tensile modulus value. The pure RTV silicone rubber only strained up to 50% compared to a 15 phr nanographite/ RTV silicone rubber composite specimen which was strained up to 120%. Another study was the work of Hu et al. (2022), who investigated a composite material made of silicone rubber reinforced with boron nitride (BN). The results showed a similar pattern where the tensile modulus value increased along with the increment of fibre content. It could be concluded that adding MOB fibre has improved the tensile behaviour of silicone rubber and epoxy compared to other researched materials such as Kenaf-Silicone biocomposite (Azmi et al., 2017), Arenga Pinnata-silicone biocomposite (Bahrain & Mahmud, 2019), phoenix-epoxy biocomposite (Rajeshkumar et al., 2017) and bamboo epoxy biocomposite (Huang & Young, 2019). Table 4 shows the measured tensile modulus of MOB-Silicone biocomposite (current study) compared to other materials which other researchers had developed. It could be highlighted that pure silicone is very weak (low tensile stiffness) and, thus, not suitable for many applications. MOB-Silicone Biocomposite is a newly introduced material with the potential for an organ substitute. Therefore, this study has reinforced various weight-percentage of MOB fibre into silicone rubber to increase the tensile stiffness. As shown in Table 4, this newly introduced tensile stiffness, E, is within the range for a few potential applications, such as connective tissue.

MOB-Silicone (Soft composite) Versus MOB-Epoxy (Hard Composite) Biocomposites

For the first time, this study introduces two types of biocomposite materials, MOB-Silicone biocomposites and MOB-Epoxy biocomposites, intended for soft and hard composite

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Tensile Modulus, E	Material Type	Reference
0.4 to 8.2 MPa	RTV-Silicone Rubber/ graphene composites	Kumar et al. (2022)
0.4 to 0.7 MPa	RTV-Silicone Rubber/ graphite composites	Kumar et al. (2022)
3 to 8 MPa	Silicone/Hemp fibre biocomposite	Koushki et al. (2020)
0.3 to 0.85 MPa	Silicone-Organically modified montmorillonite composite	Bae and Chang (2013)
76 to 120 kPa	Moringa Oleifera bark- silicone (MOBSil) biocomposite	Current study
41 to 44.4 kPa	Porcine Orbital Fat & Connective Tissue in eye (OFCT)	Chen and Weiland (2011)
16.5 to 16.6 kPa	Human Orbital Fat & Connective Tissue in the Eye (OFCT)	Chen and Weiland (2011)
8.2 kPa	Hydrogel Matrix	Castilho et al. (2018)

Table 4 Tensile modulus, E of current study compared to other studies

applications, respectively. Comparing the MOB-Epoxy (hard composite) and MOB-Silicone (soft composite) biocomposite, MOB fibre interestingly affected silicone rubber and epoxy. Both the soft and hard composites became stiffer due to the reinforcement. By computation at 16wt% fibre content, the stiffness for silicone biocomposite had increased by 64%, while the increase was only 35% for epoxy biocomposite. In contrast, the stretch ratio had reduced by 27% for silicone, and the strain had reduced by 65% for epoxy biocomposite. By comparison, it can be concluded that reinforcing MOB fibre affected the stiffness of silicone rubber more than epoxy; but affected the elongation of epoxy more than silicone rubber. Table 5 shows the t-test results to highlight which Tensile Modulus of both matrix materials were affected more by the reinforcement of the MOB filler. Observing a significant difference in how the MOB filler affected the tensile modulus of silicone rubber and epoxy is interesting.

The t-score in Table 5 indicated that a large difference existed between the MOB-Epoxy and MOB-Silicone samples, where a high value of t-score was recorded at 17.55. It is well known that if the value of the t-score is small, a similarity between two samples exists. However, a large t-score indicates a difference between the groups. In this case, the characteristics of the two materials were demonstrated to be distinct, as shown by a

W_{-1}	Average Tensile	Modulus, E (MPa)
weightage (wt%)	MOB-Epoxy	MOB-Silicone
0	1240.5	0.076
4	1312.1	0.080
8	1507.5	0.089
12	1617.3	0.100
16	1668.5	0.120
Mean	1469.18	0.091
Variance	35026.272	0.000398
Observations	5	5
t-score	17.5523855	

Table 5 Comparison test between MOB-Epoxy and MOB-Silicone via t-test

significant t-score in Table 5. Their behaviour under tensile stress also behaved differently under uniaxial tensile load. The MOB-Epoxy was seen to absorb a small load to reach the breaking point. However, this was in contrast to the MOB-Silicone sample. The MOB-Silicone sample was seen to require a high amount of load as it elongated to the breaking point. It was also seen to take longer elongation as it absorbed the load to elongate compared to the MOB-Epoxy sample. Finally, it could be observed that these two materials were very distinct in terms of their behaviour toward the tensile load, properties and elongation rates.

CONCLUSION

Based on supporting the idea of promoting green materials and addressing environmental issues, this research has successfully introduced two new types of biocomposites made of silicone and epoxy matrix reinforced with natural fibre: Moringa Oleifera bark (MOB). This study has successfully reported the effect of reinforcing MOB fibre on silicone and epoxy biocomposites' tensile and deformation behaviour. In general, soft and hard composites became stiffer due to the reinforcement. Nevertheless, interestingly, the main finding deduced that the reinforced MOB fibre had affected the stiffness of silicone rubber more than epoxy; but affected the elongation of epoxy more than silicone rubber. Finally, the quantified properties were compared to other materials for benchmarking and exploring potential applications. MOB-Silicone biocomposites but were still in the range of Elastin and connective tissues, thus opening the possibility of producing synthetic tissues made of MOB-silicone biocomposites, or at least to bio-mimic the deformation of synthetic tissues.

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