Synthesization and Microstructural Analysis of Arenga Pinnata Fibres and Silicone Rubber for New Silicone Biocomposite Material

Siti Humairah Kamarul Bahrain
Jamaluddin Mahmud
Faculty of Mechanical Engineering, Universiti Teknologi MARA, 40450 Shah Alam, Selangor, Malaysia

Zainudin A. Rasid
IDS i-Kohza, Malaysia-Japan International Institute of Technology, Universiti Teknologi Malaysia, 54100 Kuala Lumpur, Malaysia

June Madete
Department of Electrical and Electronic Engineering, Kenyatta University, Kenya Drive, Nairobi City, Kenya

ABSTRACT

In promoting 'green' environment, natural fibres are widely used to replace man-made fibres such as glass fibres and carbon fibres which are not environmentally friendly especially when decomposed. This study for the first time aims to introduce new biocomposite material using Arenga pinnata fibre as filler while silicone rubber as its matrix. This study seeks the best form of fibre to attain good compatibility and fibre dispersion between matrix and filler. This soft composite possesses the hyperelastic material behaviour as silicone rubber has the ability to elongate at very large deformations. To synthesize the biocomposite, the fibres were crushed using a few methods and mixed with silicone mixture. The biocomposites were then cut and these cross-sections were observed under optical microscope and evaluated using SEM. It can be observed that a finer form of filler exhibits good filler-matrix adhesion compared to coarse fillers as voids can be formed during the curing process of the composite. An additional tensile test was conducted to assess its mechanical properties and it was found that the average ultimate tensile strength and modulus of elasticity of 8wt% Arenga pinnata – silicone biocomposite were 0.75 MPa and 0.067 MPa respectively. Without Arenga pinnata (pure silicone), the average ultimate tensile strength and modulus of

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elasticity were 0.85 MPa and 0.051MPa respectively. The addition of Arenga pinnata has thus improved silicone's stiffness. In conclusion, this result proves that the proposed synthesization method has been successful.

Keywords: *Silicone Biocomposite, Arenga Pinnata Fibre, Silicone Rubber, Synthesization*

Introduction

A composite material consists of two or more materials combined on a macroscopic scale to form a new material with better properties from those of its individual material. If either the matrix, reinforcement or both is derived from a biological origin, it is classified as biocomposite materials [1]. Biocomposite materials have recently attracted many researchers to seek its benefit and make improvement on existing materials.

Due to increased awareness of environmental issues, the uses of natural fibres as filler or reinforcement in composite materials have become desirable. They promote good properties such as low density, low cost, biodegradability and most of all they can be obtained easily as they are abundant in nature [2]. Besides that, natural fibres are safe to be used compared to synthetic fibres such as glass fibres which can cause harm and environmental problems [3]. Some of the natural fibres that have already been known by researchers such as kenaf, oil palm, flax and jute are being employed to replace man-made fibres in order to promote new biocomposite material [2, 3]. Attentions to environmental issues have led to the introduction of a biotype composite material to help preserve the earth by reducing the carbon emission that can pollute our environment [4, 5].

Thus, this study for the first time selects Arenga pinnata fibre as the reinforcing material for silicone rubber. "Arenga pinnata", *Ijuk* or scientifically named as *Arenga saccharifera* is a multipurpose plant as almost all parts of the tree can be utilized. They are traditionally being used for the making of ropes for ship cordages, roofs, brooms and mats due to its high durability and high resistance to seawater [4, 6]. Meanwhile, another study found that Arenga pinnata fibres exhibit good mechanical properties (strain and tensile properties) which is comparable to other natural fibres [7]. Literature reports that Arenga pinnata fibres have been reinforced with epoxy resin [2, 8, 9], unsaturated polyester [10, 11] and few others but none so far has reinforced Arenga pinnata fibres with silicone rubber. It is crucial to obtain perfect fibre bonding so that its ultimate material's strength can be achieved. Previous study has also emphasized on the hydrophilic nature of the natural fibres. In order to overcome this, few techniques and treatments have been employed [4]. Silane treatment and alkaline treatment [6] are

among the treatments used on natural fibres to enhance interfacial adhesion between matrix and reinforcement.

As for the matrix in this study, silicone rubber known for its high elasticity has widely been used for electronic components as it exhibits good thermal stability and electrical insulation. However, silicone rubber has weakness in terms of mechanical tensile strength [12] that causes it to degrade much faster. Due to this, many researches have altered the properties of silicone rubber via filler addition to improve its mechanical properties [12]. Nano-silica, multi-walled carbon nanotubes [13] and nickel [14] are among the fillers used to improve the properties of silicone rubber. Other researches regarding silicone composite have emerged into many new developments such as in medical applications [15], thermal interface applications [16, 17] and sensor applications [18]. For the first time, an in-depth study and knowledge on synthesizing the Arenga pinnata-silicone composite process is required and it is important to obtain a reliable result as there is still no research been conducted regarding this silicone biocomposite. Therefore, the main objective of this study is to introduce a new silicone biocomposite with Arenga pinnata reinforcements using a few synthesization methods. The synthesized specimens from each method are compared to observe the interfacial adhesion and dispersion between both reinforcement and the matrix using stereozoom microscope and SEM. Finally, the best synthesizing method with new sample is tested under uniaxial tensile tests to further characterise its mechanical properties compared to pure silicone rubber. Due to the presence of Arenga pinnta combined with silicone, this new material could potentially be developed for sealing applications [19].

Materials and Methods

For the filler or reinforcement purposes, the Arenga pinnata fibres were harvested from Kuala Pilah, Negeri Sembilan, Malaysia. They can be obtained easily as the fibres were fully covered on the trunk of the tree (Figure 1). As for the matrix, silicone Ecoflex 00-30 (Platinum Cure Silicone Rubber) supplied by Smooth-On was chosen for this study.

Figure 1: Arenga pinnata tree

Fibre crushing processes

There are three methods of crushing involved in synthesizing the Arenga pinnata fibres with silicone rubber. In order to attain good interfacial bonding between the filler and the matrix, the following (Figure 2) is the flow process involved during the study where the first method involved crushing machine (Method 1). To further crush them into finer fibre form, two methods were chosen and employed to observe the best results in obtaining consistent fibre size (Method 2 or Method 3). Last but not least, sieving process was the most crucial step that must be taken in order to control the fibre size by eliminating coarser fibre (Method 4).

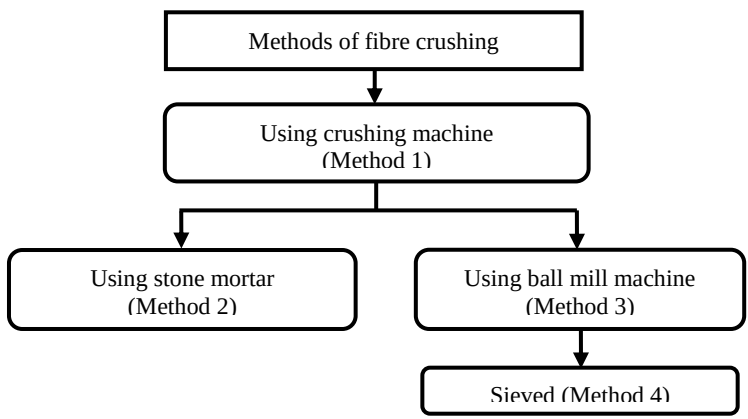


Figure 2: Crushing flow involved

Synthesizing process

The fibres were firstly cleaned thoroughly and let dried to remove any stains and contaminants before they underwent the crushing process. The crushed fibres were then weighed according to the fibre weight desired based on calculations made for one specimen. For synthesizing process, both part A and part B of silicone Ecoflex were mixed together with the ratio of 1:1. The weighted fibres were included in the silicone solution and ensured that Arenga pinnata-Silicone mixture was well mixed by manually stirring (hand-mixed) the mixture before the pouring process began. The biocomposite mixture was then let cured at room temperature for four hours.

Surface characterization

To attain good argument of the compatibility between Arenga pinnata fibre and the silicone rubber, the composite specimens were observed under stereozoom microscope and finally evaluated using Scanning Electron Microscope (SEM). The use of optical microscope was conducted in Material Science Laboratory, Faculty of Mechanical Engineering, Universiti Teknologi MARA, Shah Alam while the SEM was conducted in Makmal Penyelidikan 2, Faculty of Dentistry, Universiti Teknologi MARA, Sungai Buloh. To ensure good surface images, the specimens must be coated with a conductive metal and they were coated with gold/palladium sputter using sputter coater machine.

Investigating the tensile properties of Arenga pinnata – silicone biocomposite

The specimen of Arenga pinnata-silicone biocomposite from the best synthesizing method that had shown good bonding and fibre dispersion was prepared for uniaxial tensile test to determine its mechanical properties. Using 3382 *Universal Testing Machine 100 kN* (Instron, U.S.A), tensile test was conducted according to ASTM D412 standard with the speed rate of 500mm/min (Figure 3). The average tensile properties from nine specimens were then determined and compared to pure silicone rubber.

Figure 3: A specimen under tensile load

Results and Discussions

The main purpose to synthesize the silicone biocomposite was to investigate mechanical properties of the Arenga pinnata – silicone biocomposite through uniaxial tensile test which is further explained in another paper. Thus, the biocomposite was synthesized in dumbbell shape according to ASTM D412 testing standard [20] specifically to determine the tensile properties for rubber.

This study reported for the first time the synthesization of Arenga pinnata fibre combined with silicone rubber. It was crucial to ensure that both filler and matrix were well bonded and the fibres were well dispersed in the matrix. Furthermore, L. Gan et al. [21] and Y. Wang et al. [22] in their studies have also stated that the dispersion state of fibre in a composite plays a critical role to ensure that its capability is maximized and its mechanical properties are enhanced. Good bonding between filler and matrix will improve the mechanical properties of the composites as has been reported by N. Witt et al. in their study [23] while poor ones will limit the applications of the material [24].

Before determining the best synthesization method of this soft type biocomposite, the trial dumbbell shape specimens were cut at certain cross section with the size of 2x2 cm to observe the interfacial surface. The images captured were taken perpendicularly from the side cut (Arrow). Figure 4 shows the specimen underwent Method 1 with 8 wt% of fibres. With our naked eyes, it can be seen that the specimen exhibited rough surfaces as the fibres were coarse. Not only that, the silicone rubber itself was unable to fill up the empty spaces causing the specimen to form bubbles that eventually became voids.

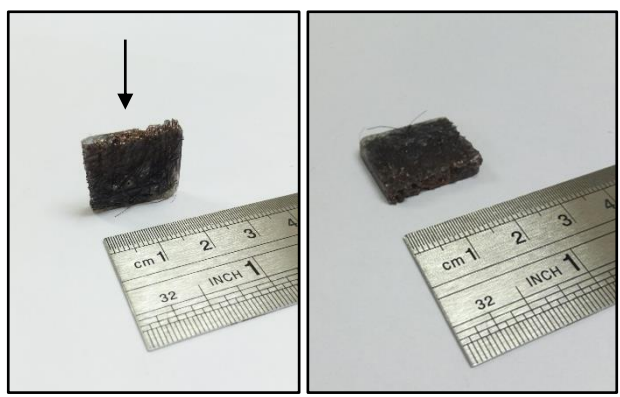
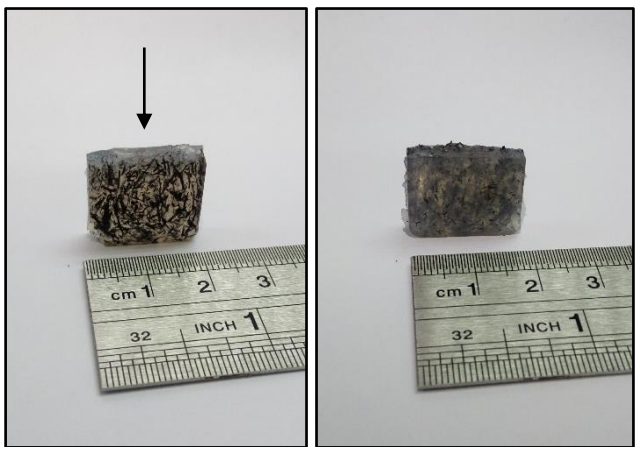


Figure 4: Synthesized specimen using Method 1

The fibres were then crushed using stone mortar to obtain finer fibres compared to fibres obtained using crushing machine as the result from Method 1 that seemed to be inappropriate for the composite as the fibres can easily be pulled off from the silicone matrix. Thus, the fibre volume was reduced to 1 wt% in order to observe the compatibility between the fibres and the silicone rubber. Result from Method 2 showed that the composite was able to synthesize well (Figure 5). However, once curing process was completed, it can be observed that coarser fibres accumulated at the bottom of the specimen (Figure 5a). This behaviour indicated that the fibres were denser than silicone rubber which caused the fibres to fall down easily and leaving the upper side specimen to be cleared (Figure 5b) although silicone rubber was known to be a viscous material.



(a) (b)

Figure 5: Synthesized specimen using Method 2 (a) lower side; (b) upper side

The following attempt was made to further seek the best method to synthesize the composites. The fibres from Method 1 were ball milled to obtain filler in powder form. Maintaining with 1 wt% of fibre content, it can be seen that a better specimen can be obtained compared to specimens synthesized using Method 1 and 2 (Figure 6). Good fibre dispersion can be observed from the above side of the specimen (Figure 7a). However, through our naked eyes, the specimen also experienced the same situation as in Method 2 where coarser fibres accumulated at the bottom of the specimen but the finer fibres can be seen to be well dispersed compared to Method 2 specimen (Figure 7b).

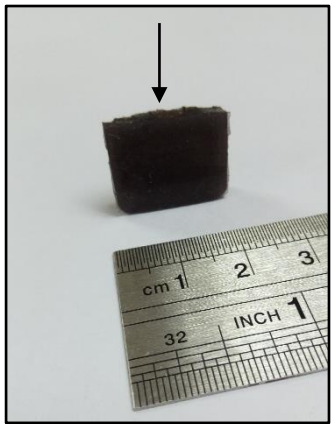
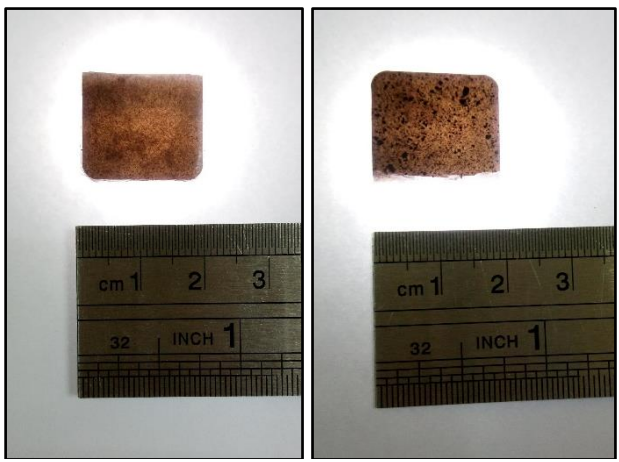


Figure 6: Synthesized specimen using Method 3



(a) (b)

Figure 7: Method 3 specimen from (a) above side; (b) bottom side view under some light

Last but not least, from the previous method (Method 3), a good compatibility between fibres and matrix was achieved. Thus, next synthesization process was taken to increase the volume of fibre to 8 wt% and to sieve the ball milled fibres in order to improve the fibres dispersion from Method 2 and 3. Compared to Method 1 specimen, the silicone rubber had the ability to fill up space and thus, a better result of composite material was achieved (Figure 8).

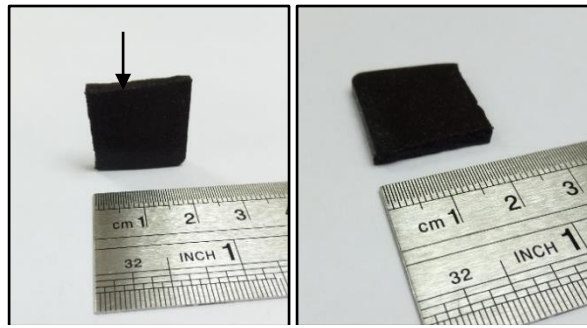
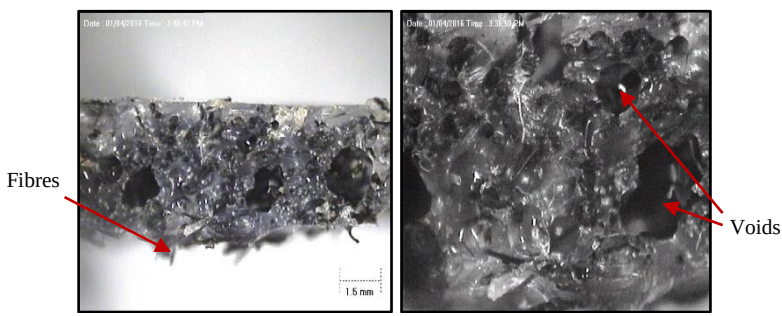


Figure 8: Synthesized specimen using Method 3 and Method 4

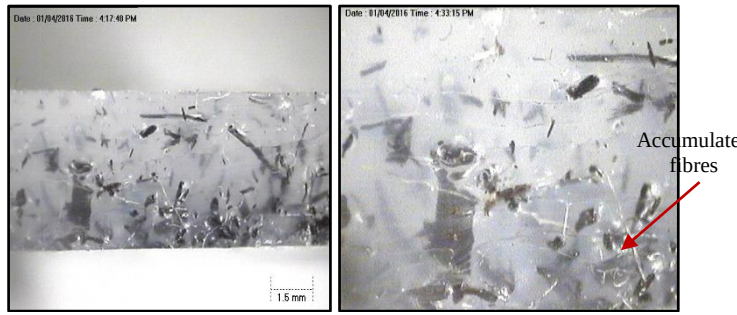
Optical microscope

Using stereozoom microscope, it was observed that specimen 1 exhibited many voids and the fibres were not well bonded with the matrix (Figure 9). As for specimen using Method 2, the images (Figure 10) showed the fibres were accumulated at the bottom of the specimen due to reason stated earlier. While for specimen 3 (Figure 11), it was observed that the fibres were well dispersed but coarser fibres were seen by our naked eyes accumulated at the bottom side of the specimen. The optical microscope was unable to capture this behaviour due to the coarser fibres that stuck to the bottom on most of the specimens. Moreover, the fibres that went through ball milled process were milled into finer forms and thus less coarse fibres were seen in specimen 3. Finally, specimen 4 indicated that the fibres were well dispersed compared to specimen 1 although they were the same in terms of volume fraction. However, with higher amount of fibre volume, it was observed that void formations occurred (Figure 12) while specimens with lower fibre volume exhibited no or less voids.



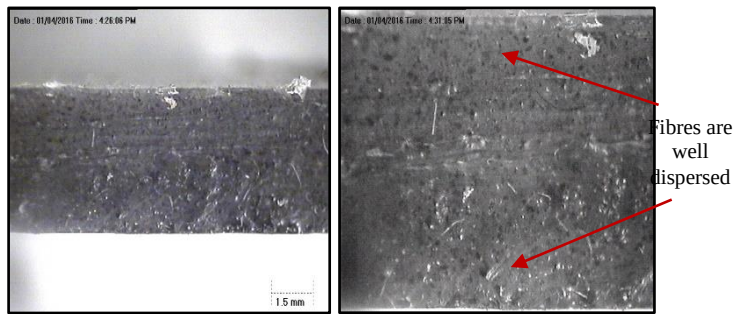
(a) (b)

Figure 9: Specimen 1 (Method 1) under optical microscope at (a) x1; (b) x4.5 magnification



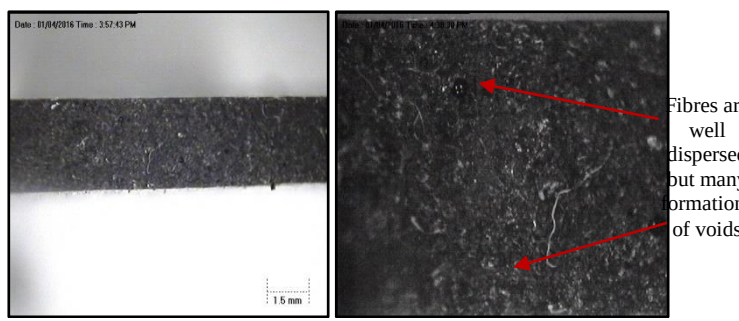
(a) (b)

Figure 10: Specimen 2 (Method 2) under optical microscope at (a) x1; (b) x4.5 magnification



(a) (b)

Figure 11: Specimen 3 (Method 3) under optical microscope at (a) x1; (b) x4.5 magnification



(a) (b)

Figure 12: Specimen 4 (Method 3 & Method 4) under optical microscope at (a) x1; (b) x4.5 magnification

Scanning Electron Microscope (SEM)

To obtain more reliable results, SEM was conducted to further investigate the interface of the composite specimens. Figure 13 shows big porosity where the silicone rubber was unable to fill up. The image also captured that the fibres were not fully coated with the silicone matrix. This condition would lead to fibre pull-out due to weak interfacial adhesion between fibre and matrix.

Based on the results shown previously (Figure 5 and 10), the SEM image (Figure 14) indicated that the coarse fibres embedded on the left side (bottom side of the specimen) while the right side of the image showed neat silicone rubber with tiny fibres. This may be due to coarser Arenga pinnata fibres that were denser than the silicone rubber as the fibres accumulated and was unable to disperse well.

Figure 15 shows powder formed fibres in silicone matrix were well dispersed and the image of the coarser fibres as in Figure 7b still cannot be captured as the powder form fibres were too fine and had been embedded at the bottom on most of the specimens (at x80 magnification). Finally, good dispersion of the filler was seen (x100 magnification) in the silicone matrix but the increased volume of filler could lead to more voids (Figure 16). This result showed better interfacial surface adhesion compared to Specimen 1 (Figure 13) that had formed bigger voids.

From both morphological analysis (stereozoom microscope and SEM), it can be observed that the fibre compatibility with silicone rubber was improved as the finer fibre form was obtained. Clearly the SEM result from Figure 13 indicated that the fibre was unable to bond well with silicone rubber due to hydrophobic behaviour that exhibited in silicone rubber. Furthermore, few studies had also made attempts to obtain good dispersion using ball milled machine [23, 25] but at different speeds and grinding durations. Last but not least, for further fabrication process, the method using stone mortar to crush the fibres can be excluded as the method using ball mill machine was more effective and efficient in obtaining finer fibre form.

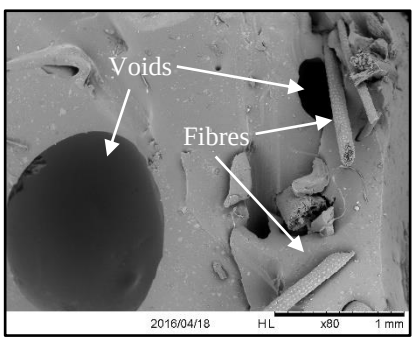


Figure 13: SEM image (Specimen 1)

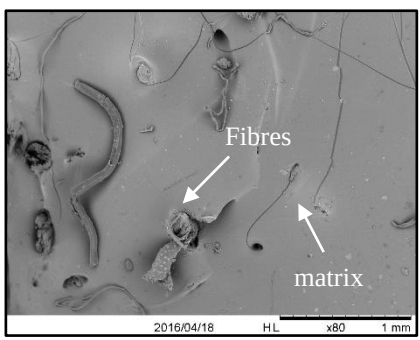


Figure 14: SEM image (Specimen 2)

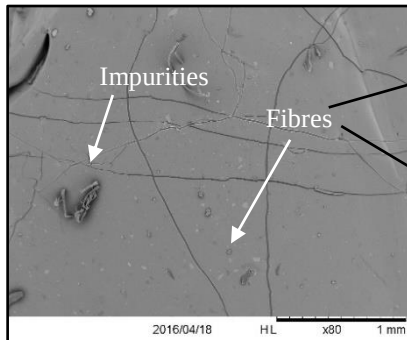


Figure 15: SEM image (Specimen 3)

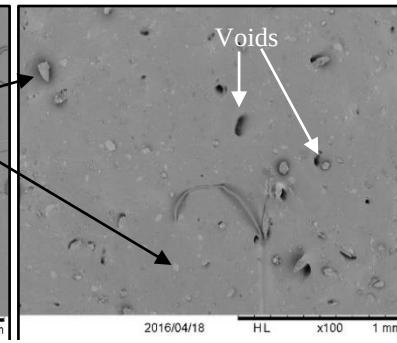


Figure 16: SEM image (Specimen 4)

Tensile properties of Arenga pinnata – silicone biocomposite

Figure 17 displays the tensile behaviour of pure silicone rubber and 8wt% Arenga pinnata – silicone biocomposite. The stress-strain curves were plotted from the average value of nine specimens. It could be observed that pure silicone rubber exhibited higher ultimate tensile strength (0.85 MPa) compared to 8 wt% Arenga pinnata – silicone biocomposite (0.75 MPa). The results also indicated that the maximum strains before failure for pure silicone rubber and 8 wt% Arenga pinnata – silicone biocomposite were 11.83 and 10.32 respectively. Obviously, this showed that pure silicone had elongated more.

It could also be observed that the stress-strain curve for pure silicone rubber displayed a highly non-linear elastic behaviour with concave upward pattern while the stress-strain curve for 8 wt% Arenga pinnata – silicone biocomposite was almost linear.

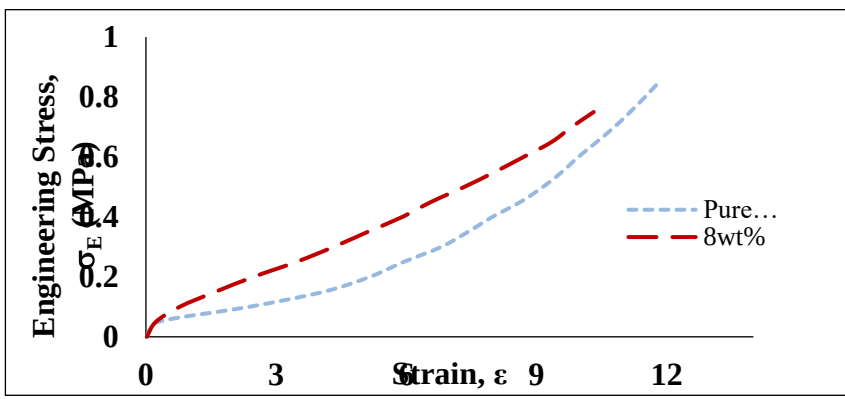


Figure 17: Stress–strain curves for pure silicone rubber and 8wt% Arenga pinnata – silicone biocomposite

From the tensile tests conducted on both nine specimens of pure silicone and 8wt% Arenga pinnata – silicone biocomposite, the determined average Modulus of Elasticity, E is presented in Table 1. Table 1 shows that the average Modulus of Elasticity for pure silicone and 8wt% Arenga pinnata silicone biocomposite were 0.051 MPa and 0.067 MPa respectively. This indicated that the reinforcement of Arenga pinnata had improved the stiffness of pure silicone.

Table 1: The average value of Modulus of Elasticity, E

	Pure silicone	**8wt% Arenga pinnata – silicone biocomposite**
Modulus of Elasticity, E (MPa)	0.051 (Average)	0.067 (Average)

Conclusion

The main objective of this study is to introduce a new silicone biocomposite with Arenga pinnata reinforcements using a few synthesization methods. For the best synthesization method, the tensile properties for selected specimen were determined. Therefore, the results showed that the main objective was achieved successfully.

The main findings deduced from this study are;

- Selection of fibre forms and synthesization method to be employed is crucial in order to ensure good bonding between the filler and the matrix
- Using ball mill machine and sieving method are found to be the best synthesization method as it produces good bonding between the filler and the matrix
- The addition of Arenga pinnata has thus improved silicone's stiffness.

Based on the results and findings, it could be concluded that this study has significantly contributed in enhancing knowledge on the synthesization of Arenga pinnata – silicone biocomposite.

Future Works

The future works could investigate the hyperelastic behaviour of Arenga pinnata – silicone biocomposite due to the variation of fibre contents (0 wt%, 4 wt%, 8 wt%, 12 wt% and 16 wt%) by adapting previous hyperelastic constitutive model [26, 27]. Apart from that, the future works can also further

explore other properties and potential applications of Arenga pinnata – silicone biocomposite. The results will be reported imminently.

Acknowledgements

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