Biocomposite Silicone: Synthesis, Mechanical Testing and Analysis

Siti Noor Azizzati Mohd Noor, Mohd Arif Fikri Khairuddin, and Jamaluddin Mahmud

***Abstract*—** Silicone elastomer is categorized under hyperlastic material. Its application is relatively limited due to the rather weak mechanical properties especially room temperature vulcanized silicone rubber (RTV) such as Ecoflex® Rubbers Supersoft 0030. This paper for the first time aims to produce reinforcing silicone rubber with kenaf powder fillers formed under synthesized process to increase the stiffness properties of the silicone rubber and to determine its mechanical properties in terms of material constants value by adapting hyperelastic constitutive models (i.e. neo-Hookean and Mooney-Rivlin). Initially, a pure silicone specimen was prepared. Meanwhile, Kenaf powder was selected to be used as silicone reinforcement or a filler material for synthesizing process and silicone reinforced with kenaf under 5 to 25 PHR units (part per hundred part of silicone rubber) was synthesized. This was followed by mechanical testing (uniaxial tensile test) and theoretical analysis which adapted hyperelastic constitutive models to determine the mechanical properties in terms of material constants value. Results indicate that addition of different percentage volumetric kenaf powders filler have improved the RTV weakness which allowed for exploration of wider applications for silicone rubber Results show that the silicone composites with kenaf fillers possess high stiffness compared to pure silicone. This resulted in variation of C_1 value, where it was found that greater value of C_1 indicates stiffer material behavior. Silicone reinforced with 25 phr kenaf powder possesses the highest value, 0.053 MPa (C_1) which means this specimen is the stiffest among the specimens. Meanwhile pure silicone possesses the lowest, 0.032 MPa (C_1) value which makes it the most elastic specimen. As for the Mooney-Rivin model, a similar scenario appeared where silicone reinforced with 25 phr kenaf powder possesses the highest 0.025 MPa and 0.111 MPa (C_1 and C_2) value and pure silicone possesses the lowest 0.020 MPa and 0.072 MPa (C_1 and C_2) value. Therefore, it can be concluded that the quantity of kenaf powder does influence the production of silicones reinforced with kenaf powder in terms of behaviour.

***Keywords*—** Silicone, kenaf, synthesis, hyperelastic constitutive model.

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I. Introduction

The ability of silicone elastomer capable to stretch without experiencing in volume reduction generally depended on the bonded structure within the material [1]. This mechanical property relatively limits the applications of silicone rubber material [2]. The weaknesses can be overcome by reinforcing the silicone elastomer into biocomposite silicone material through synthesis process to improve the material stiffness [2]. This study has selected kenaf powder to be used as silicone reinforcement or a filler material. “Kenaf” or “*Hibiscus cannabinus*, L. family *Malvaceae*” is “ an herbaceous annual plant that can be grown under a wide range of weather conditions” [3]. From previous studies [3, 4], it was found that kenaf helps to improve the material strength of a composite construction with economical and availability advantages. In-depth study and knowledge on the synthesizing biocomposite silicone process is important to obtain a reliable result.

Tensile testing appeared to be one of the favourable methods to determine basic material properties of a material [5] and uniaxial tensile tests with regard to silicone elastomers have commonly been executed according to universal standards ASTM D412 [6-9] testing standards. Hence, the silicones reinforced with kenaf powder specimens were prepared according to ASTM D412 to observe the stress-stretch the behaviour of the material.

Silicone elastomers materials (rubber-like) are the most common examples of hyperelastic materials which possess hyperelasticity characteristics [8, 10-13]. Since mechanical testing is incapable of directly providing the mechanical properties of a material, hyperelastic constitutive models were adapted to theoretically quantify the material properties of the specimens (i.e. silicones reinforced with kenaf powder).

Two most popular hyperelastic constitutive models (i.e. neo-Hookean and Mooney-Rivlin) which were represented in strain energy density function (W) expressed in (1) and (2) respectively where C_1 and C_2 are the material constants, meanwhile $\bar{I}_1$ and $\bar{I}_2$ are the invariants [14, 15].

$$W = C_1(\bar{I}_1 - 3) \tag{1}$$

$$W = C_1(\bar{I}_1 - 3) + C_2(\bar{I}_2 - 3) \tag{2}$$

Hence this study focuses on synthesis a silicone rubber reinforced with kenaf powder and consequently followed by a

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mechanical testing (uniaxial tensile test) and theoretical analysis which adapted hyperelastic constitutive models (i.e. neo-Hookean and Mooney-Rivlin model) to determine their mechanical properties in terms of material constants value. This is novel as no similar syntheses related to reinforcement of silicone rubber with kenaf powder have been reported earlier.

II. Methodology

A total of three main stages (i.e. synthesis, experiment, and theoretical) are complied in this study. Figure 1 illustrates the process flow of the study which also includes the comparison between pure silicone and silicone reinforced with kenaf results.

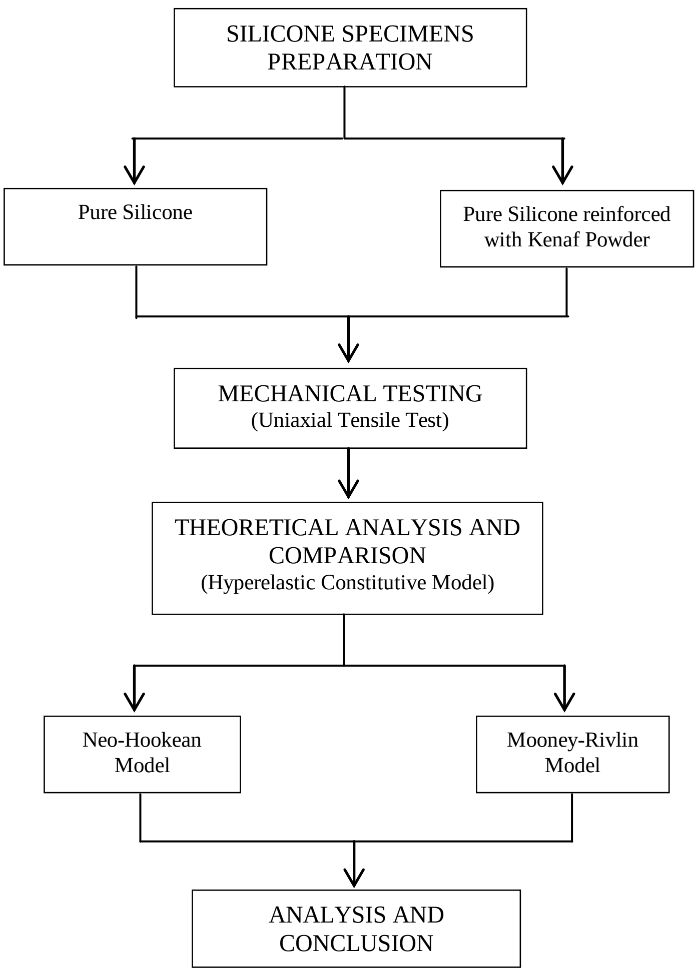


Fig. 1 Study Flow

A. *Synthesis of Biocomposite Silicone*

Initially, Pure Silicone specimens were prepared referring to ASTM D412 testing standard. Silicone matrix is quantified with parts per hundred (phr) units. To produce a single specimen of pure silicone, the ratio is 1:1 which is considered equal to 100 phr of silicone elastomer

Hence as shown in Figure 2 and 3, Pure Silicone (Ecoflex 0030) and its hardener were mixed at 50 percent of total volume for each. It is estimated that the total volume of a single dumbbell-shape ASTM D412 mold is 6cm^3 which gives 3cm^3 in volume for Ecoflex 0030 and its hardener each. It is noted that every 1cm^3 of silicone liquid is equal to 1g of its weight.

Once the mixture was ready, it was injected into ASTM D412 mould (Figure 4) properly before being placed in MCP vacuum machine for about 3 to 5 minutes. Lastly, the specimens underwent natural cure process for 72 hours at room temperature (28°C to 34°C) which completed the preparation of pure silicone specimens. One of the samples of pure silicone is shown in Figure 6(a).

Fig. 2 Silicone Ecoflex 0030 and hardener

Fig. 3 Mix Silicone Ecoflex with its harderner

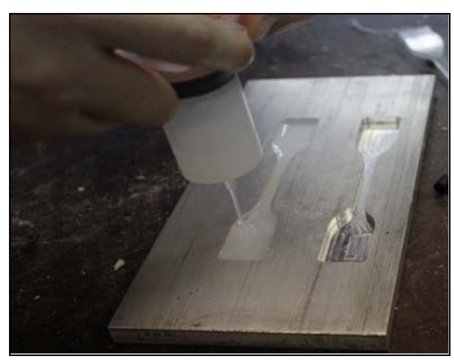


Fig. 4 Pour the mixture into ASTM D412 aluminium mould

The next process is to synthesize Kenaf Powder with Silicone. Kenaf powder (Figure 5) was extracted from bast fiber plant which was taken from the outer layer and core of the fiber (inner layer). Kenaf is widely used in many researches to determine material strength of a composite. Due to its ability to improve the tensile strength of composite, a synthesis process of kenaf powder compounded with silicone solution was done in this study.

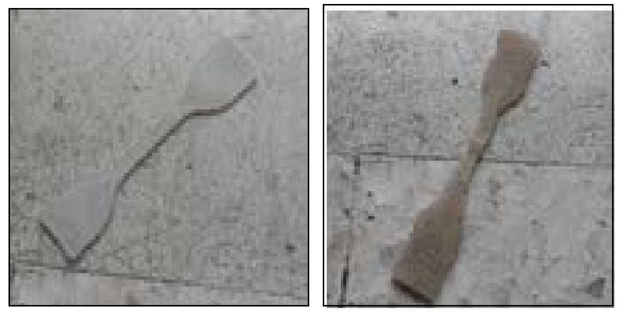
Fig. 5 Kenaf Powder

Three main steps took place in synthesizing kenaf powder with silicone which are silicone preparation (Step 1), vacuum process (Step 2) and curing process (Step 3). During silicone preparation, it is important to measure the suitable volume or weight of filler material (kenaf powder) needed for synthesis process. Five different kenaf powders in grams (0.1, 0.2, 0.3, 0.5, and 0.6) which are equal to 5, 10, 15, 20, and 25 phr being premixed with silicone matrix during silicone preparation are tabulated in Table I.

Table I Composition of Kenaf Powder

phr	Mass (g)	Density (g/cm^3)	Volume (cm^3)
5	0.1	0.5702	0.2
10	0.2	0.5702	0.4
15	0.3	0.5702	0.6
20	0.5	0.5702	0.8
25	0.6	0.5702	1.0

Later the premixed kenaf - silicone matrix was poured (injected using a syringe) also into an aluminum dumbbell-shape mold as shown in Figure 4. Once the silicone was ready, Step 2 (vacuum process) took about 3 to 5 minutes which required MCP vacuum machine to reduce entrapped air bubbles from the specimen. After that, the natural curing process (Step 3) commenced by considering room temperature (28°C to 34°C) and curing of 72 hours, then only could the prepared specimen undergo a tension test. One of the silicones reinforced with kenaf powder specimen is shown in Figure 6(b).

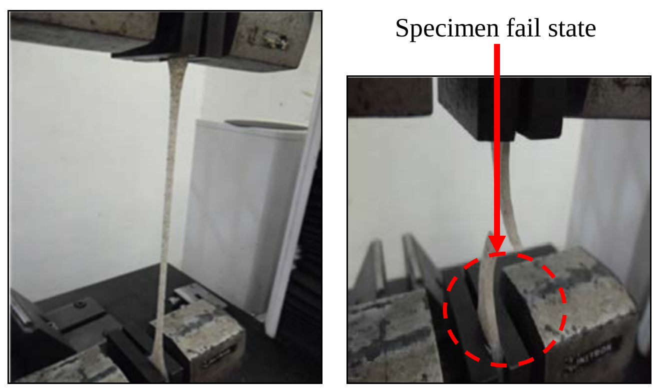


(a) (b)

Fig. 6 Specimen ready to be mechanically tested

B. Mechanical Testing

After all of the specimens had been prepared, uniaxial tensile test took place at Strength of Material Laboratory of Universiti Teknologi MARA, Malaysia. Using INSTRON 3382 Universal Testing Machine and referring to ASTM D412 testing standard, a total of five pure silicone specimens and twenty five silicones reinforced with kenaf powder specimens with different kenaf powder amount were tested till they reached their failure state. The speed was set to 500±50mm/min with the gauge width, 6mm, gauge thickness, 3mm and gauge length, 33mm. Figure 6(a) and 6(b) demonstrate the condition of the specimen while being tested and when it failed.



(a) (b)

Fig. 6 Specimen ready to be mechanically tested

C. Quantifying Material Constants

The material behaviour can be represented by the material constants ($C_1$and C_2) value which can be determined by adapting the theory of hyperelastic constitutive models (i.e. neo-Hookean and Mooney-Rivlin) in terms of stress-stretch relation formula. Referring to stress-stretch values obtained from the experiment, $C_1$and C_2 value can be determined using 3) and (4) for neo-Hookean and Mooney-Rivlin models respectively.

Stress-stretch curves were generated according to $C_1$and C_2 theoretical value determined and analysis was performed with the aid of Microsoft Excel 2010. Consequently parametric comparisons of different amounts of kenaf powder to pure silicone for all theoretical stress-stretch curves of neo-Hookean and Mooney-Rivlin were done.

$$\sigma_E = (2C_1)\left(\lambda - \frac{1}{\lambda^2}\right) \quad (3)$$

$$\sigma_E = \frac{1}{\lambda}\left[\left(2C_1 + \frac{2C_2}{\lambda}\right)\left(\lambda^2 - \frac{1}{\lambda}\right)\right] \quad (4)$$

III. Results and Discussion

Uniaxial tensile test was conducted till specimen's failure stage on pure silicone and silicone reinforced with kenaf powder. Figure 7 illustrates the experimental results which are represented by the stress-stretch curves. X-axis represents stretch values while Y-axis represents stress values in MPa.

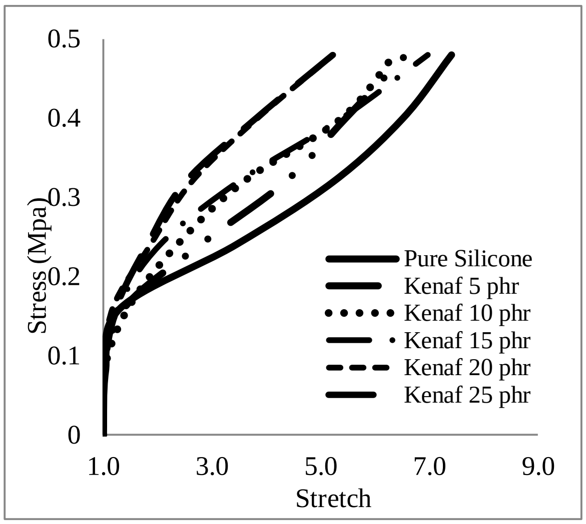


Fig. 7 Specimen ready to be mechanically tested

Each of the six curve plots shown in Figure 7 is the average of five (5) samples tested. This came from six different silicone mixtures which are pure silicone (0 phr kenaf powder), silicone reinforced with 5 phr kenaf powder, silicone reinforced with 10 phr kenaf powder, silicone reinforced with 15 phr kenaf powder, silicone reinforced with 20 phr kenaf powder and silicone reinforced with 25 phr kenaf powder.

From the results, it is noted that only silicone reinforced with 5 phr kenaf powder specimen behaves similarly to pure silicone while the remaining samples start to behave differently from pure silicone. It can be seen that amount of kenaf powder does influence the stiffness level of the specimen, where the specimen becomes stiffer along with the increment of kenaf powder amount.

Since experiment alone is incapable of quantifying the material properties, neo-Hookean and Mooney-Rivlin models were theoretically adapted to represent the properties in terms of material constants ($C_1$and C_2) value using experiment stress-stretch data previously obtained. Table II tabulates all of the material constants determined for pure silicone and each kenaf powder amount specimens.

From neo-Hookean's material constants (C_1) value was theoretically determined; Figure 8 demonstrates the differences between pure silicones with all 5 different silicone kenaf composite specimens for neo-Hookean model. This led to variation of C_1 value, where it was found that greater value of C_1 indicates stiffer material behavior. Silicone reinforced with 25 phr kenaf powder possesses the highest C_1 value which means this specimen is the stiffest among the specimens. Meanwhile pure silicone possesses the lowest C_1 value which makes it the stretchiest specimen. To summarize, the stiffness level of the specimen increases parallel to the amount of kenaf powder. Therefore the present kenaf powder in silicone matrix does influence silicone stiffness level.

As for the Mooney-Rivin model theoretical stress-stretch curve which is presented in Figure 9, a similar scenario appeared where silicone reinforced with 25 phr kenaf powder possesses the highest C_1 and C_2 values and pure silicone possesses the lowest C_1 and C_2 value. All specimens seem to be stiffer than pure silicone specimen.

Table II Material Constant theoretically determined for neo-Hookean and Mooney-Rivlin Model

	Hyperelastic Constitutive Model		
	Neo- Hookean	Mooney-Rivlin	
Kenaf Amount (phr)	C_1 (MPa)	C_1 (MPa)	C_2 (MPa)
0 (Pure Silicone)	0.032	0.020	0.072
5	0.038	0.025	0.068
10	0.040	0.025	0.075
15	0.039	0.019	0.106
20	0.052	0.024	0.111
25	0.053	0.025	0.111

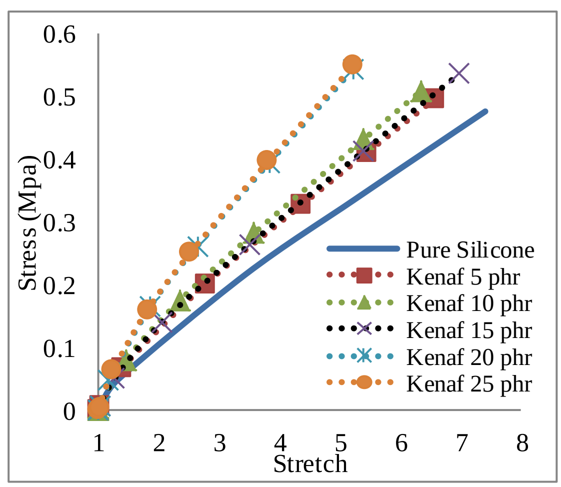


Fig. 8 Five different reinforced silicones kenaf powder with pure silicone using neo-Hookean Model

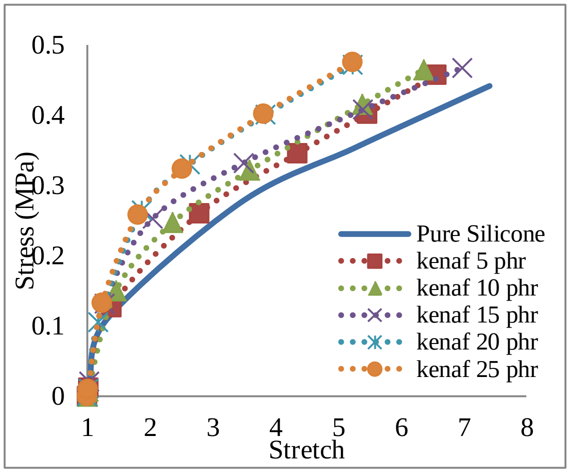


Fig. 9 Five different reinforced silicones kenaf powder with pure silicone using Mooney-Rivlin Model

IV. Conclusion

This paper has successfully presented a detailed procedure on synthesizing a biocomposite silicone mainly using a mixture of pure silicone and kenaf power. Based on experimental and theoretical results, it can be concluded that the greater value of material constant indicates high stiffness. This highlighted the influences of kenaf powder amount in the production of silicones reinforced with kenaf powder which also influence the behaviour. This study has also opened the gateway to further explore other hyperelastic constitutive models such as Ogden theory to determine material properties of hyperelastic material.

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