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Silicone Dielectric Elastomers with Incorporation of Different Types of Filler as Smart Materials for Wave Energy Harvester

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ABSTRACT

The combustion of fossil fuel is one of major human activities which produces greenhouse gases i.e. CO_2 and CO that leads to a climate change. As solution to fossil fuel energy, renewable energy e.g. wave energy, solar power, wind energy and hydropower are seek as alternative resources. Among of these renewable energies, wave energy is extensively studied due to the abundance of energy and easy access to countries surrounded by ocean. When utilising wave energy harvester, silicone dielectric elastomer (DE) with high tensile strength, high strain, enhanced permittivity and high electrical breakdown is sought. In this study, silicone DE films were prepared by incorporating different fillers e.g. chemical synthesized silica, chitosan and cellulose to utilise for wave energy harvester. These fillers are incorporated to enhance the network of silicone DE and hence these fillers act as reinforcing agents. Reinforcing agent fillers may cause high tensile strength without compromising inherent softness of elastomer. Utilising sustainable fillers i.e. chitosan and cellulose, which are obtained from seafood animal shells and plants, respectively, may minimises waste generated from food industries and restaurants. Herein, silicone DE films containing fillers i.e. chemical synthesized silica, cellulose, chitosan were successfully prepared by varying filler loadings at 1wt %, 3wt% and 5wt %. Heptane was used as solvent in silicone mixture for the desired viscosity at concentration of 55%, 60% and 65%. Surface morphology, stress at break (σ), strain at break (%) and Young's Modulus (Y) of prepared silicone DE films were investigated. Tensile testing verifies that film with 1wt % cellulose and 65% solvent is the optimum, which possesses strain at break of 693 % and stress at break of 3.26 MPa. High stress and strain at break are desired for silicone DE generator due to elastic but at the same time maintaining the strength of elastomer.

Key words: Cellulose, chitosan, silicone dielectric elastomer, sustainability

1. INTRODUCTION

The continuous use of non-renewable fossil fuel will deplete in future, which endangers energy security in global. Finding renewable resources is important and extensively searched in many countries for reducing the dependence on non-renewable fossil fuel energy and protecting energy security. Energy from non-renewable fossil fuel is widely used in many countries as the main resource of power electricity. The demand of energy is in line with emerging of world economy and other human activities. However, energy from fossil fuel affects the ecology and global climate change due to mining activity which releases of greenhouse gases (GHG) effect i.e. carbon dioxide (CO_2), which results in global warming [1, 2].

To overcome the depletion and harmful environmental impacts resulted from generating energy via fossil fuel, alternatives from wind, solar, hydro, wave and biomass energies are sought. These renewable energies are well-known for their environment friendly. Wave is more abundant than other renewable energy resources e.g. wind, solar and hydro, where wave is available up to 90 % of the time, while the availability of solar and wind is about 20–30 % of the time [3]. Nowadays, the sector of the electricity in Malaysia is mostly dependent on fossil fuels where almost 94.5% of electricity is generated from petroleum, natural gas and coal [4].

Few locations in Malaysia has been identified to harvest energy from ocean waves. Geographically, Malaysia is divided into West Malaysia and East Malaysia. The east coast of Peninsular Malaysia, which has a direct exposure from the

South China Sea, may be the desired location site to build wave energy harvester. East coast of Peninsular Malaysia receives sea wave that travels from the far north which consequently generates a massive amount of wave energy [5]. Furthermore, wave in Malaysia is influenced by the southwest monsoon and the northeast monsoon wind [12]. For West Malaysia i.e. Borneo island, Sabah has been identified to possess high wave energy [13]. Therefore, a study on wave energy output by simulation can be carried out at identified sea shore location around East and West Malaysia before investing millions of Ringgit for wave energy harvester.

Wave energy harvester utilise dielectric elastomer such that it transforms mechanical work to electrical energy. Silicone dielectric elastomer (DEs) possesses low stiffness e.g. high strain, and high permittivity. These characteristics are desired for generator application that requires the silicone dielectric elastomer to stretch and relax converting from mechanical energy to electrical energy by changing their shape and size under pre-charging and hence generate a voltage [16].

Cross-linked polydimethylsiloxane (PDMS) i.e. silicone elastomer with low-functional or high-functional crosslinkers has poor tensile strength without any fillers. As solution to poor tensile strength, silicone elastomer is incorporated with silica filler for network reinforcement [17]. Adding filler such as Titanium Oxide (TiO_2) may result in stiff elastomer[18]. Hence, adding different types of filler and its loading is important in order to obtain the desired properties such that high tensile strength but without compromising the softness of elastomer. Besides chemical synthesised filler e.g. commercial silica, bio-type filler such as chitosan and cellulose may possess desired mechanical properties. Most of processed chitosan and cellulose particles are made from sea shell waste and plant residue, respectively. Furthermore, the advantages of chitosan and cellulose are non-toxic, biodegradable and biocompatible [20]. Hence by incorporating silicone elastomer with bio-type fillers may produce a sustainable silicone DE. In this study, incorporating different types of fillers (chemical synthesised silica, chitosan and cellulose) in silicone DE is investigated with respect to tensile strength e.g. tensile and strain at break, and Young's modulus.

2. METHODOLOGY

Samples were generated from design of experiment (DOE) via Design Expert software with total of 24 samples. In designing DOE, three factors and three levels were selected, where loading of fillers (%), solvent (%) and type of fillers were the factors and weight percent of fillers (1wt %, 3wt %, 5wt %), ratio of solvent (55%, 60%, 65%) and types of fillers (cellulose, chitosan, chemical synthesized silica) were the levels, as shown in Table 1.

Table 1: Design of Experiment (DOE) of Prepared Silicone DE Films Containing Silica, Cellulose and Chitosan as Network Reinforcing Agents.

Solvent (%)	Sample	Silica (wt %)	Chitosan (wt %)	Cellulose (wt %)
55	S1	1	1	1
	S2	3	3	3
	S3	5	5	5
60	S4	1	1	1
	S5	5	5	5
65	S6	1	1	1
	S7	3	3	3
	S8	5	5	5

2.1. Chemical and Materials.

Silicone elastomer premix (LR3003/60A) and (LR3003/60B) with viscosity of $1.0 \times 10^6 – 3.0 \times 10^6$ mPa·s from Immortal Green Industrial Sdn. Bhd. (IGISB), 100 g of laboratory grade chitosan powder from Sigma Aldrich Corporation, 50 g of titanium dioxide powdered, 100 g of engineering grade silicone dioxide (silica) powder 99.5 % from Sigma Aldrich Corporation and 2.5 L of heptane from Johchem Scientific & Instruments Sdn. Bhd.

2.2. Apparatus and Equipment.

Speedmixer (model: ZB500S) with speed of 1350 rpm from Wenzhou Zhengbang Electronic Equipment Co. Ltd., film applicator with glass plates for casting, 75 µm mesh-sized sieve from Syarikat Saintifik Jaya, specimen container PP screw cap 60ml (reference: PPA-JWP0060P) with size 53mm x 47mm, Universal Oven, Universal testing machine (model: LR 30K),with 500 N load cell from Llyod Instruments Ltd. Scanning electron microscope (SU1510) from Hi-Tech instruments.

2.3. Preparation of film

Prior to film preparation, all fillers i.e. chemical synthesised silica, chitosan and cellulose were characterised. The filler size was verified using 75 µm mesh-sized sieve and hence all fillers possessed average particle size of 75 µm. Filler size below 100 µm is important for homogeneous silicone mixture. Filler above 100 µm will cause inhomogeneous mixture which causing uneven film due to desired film thickness in a range of 100 – 150 µm. Fillers were added into silicone mixture containing premix A and B in container. Mixtures were prepared based on the generated samples from DOE. The silicone mixture was speedmixed at 1350 rpm for 30 - 60 minutes. During mixing process, the mixture was checked for existence of chunks or undissolved particles. To overcome the chunks in the mixture, the mixture was manually stirred by a glass rod and was further speedmixed

until all chunks disappeared. Then the silicone mixture was casted on a glass plate using a film applicator. Prior to curing in the oven, the silicone film was exposed to room temperature at 24 hours for air bubble removal. After that, the silicone film was fully cured at 40 ^{o}C for 2 hours and followed by curing at 110 ^{o}C for 15 minutes to obtain a nice film formation.

2.4. Characterisation of silicone DE film

The characterisation of film was prepared for mechanical properties testing by using the universal testing machine model: LR 30K) with 500 N load cell from Llyod Instruments Ltd. The tensile testing test was done to determine the tear strength of the samples prepared. Tensile strength is the force of the tensile is required to rupture the film. The tensile strength of the prepared films was measured via Universal Testing Machine (model: LR 30K). The film must be prepared according to standard ASTM D412 at 500 mm/min cross-head speed. Tensile testing used standard ASTM D412 which possesses dog-bone strip, as details of measurement is shown in Figure 1. The dog-bone strip was placed between two clamps and initially separated by a distance of 25 mm and then was stretched with 500N load until it ruptured. The tensile strength is determined from stress-strain relationship for stress (σ) and strain (%) at break and Young's modulus (Y).

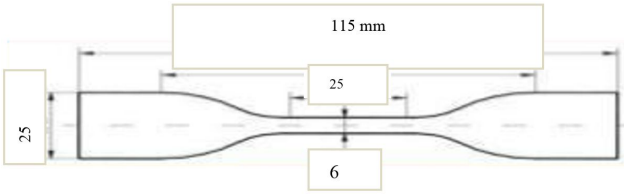

Figure 1: ASTM D412 dog-boned standard for rubbers [21]

Surface morphology of sample is investigated for analysing filler dispersion within silicone matrix by means of scanning electron microscope (SEM) (Model SU1510, Hi-Tech instruments). Prior to analysing the sample under SEM, silicone films were coated with approximately 6 µm layer of gold to increase the conductance of film for better image by means of sputtering machine from Fisons instrument.

3. RESULT AND DISCUSSION

There were 24 samples tested for characterization of film according to the third objectives. The samples with fillers of chitosan, synthesised silica and cellulose were tested for stresses and strains at break and Young's moduli, as shown in Table 2, 3 and 4, respectively, by means of Universal testing machine (model: LR 30K, Llyod instruments) with load of 500N.

3.1. Stress and strain relationship

The samples were cut into dog-boned shape for mechanical test. In order to further analyse the results from mechanical properties test, the samples had to be ensured to tear exactly at the 'neck' region (centre region), the part which is 6 mm width. The proper tear can infer that the samples are actually set symmetrically on the tensile machine, or in other words, the force applied from the machine is well distributed form two different sides. In order to understand the behaviour of inherent softness of silicone dielectric elastomer, stress versus strain curve is plotted from the results obtained from the universal tensile machine software. The curve displays the amount of deformation (strain) respect to the distinct intervals of tensile (stress). The y-axis is represented by the stress amount that the sample can possess before it tears.

The higher value in y-axis possessed by the sample, meaning the higher the before it breaks. The x-axis represents the strain, in other words how much the sample can elongate with respect to the stress applied to it. Increased strain determines the more ductile the sample, which increase the elongation with respect to high strain.

Table 2: Tensile results for samples with chitosan

	Loading of filler (%)	Solvent (%)	Stress at Break (MPa)	Strain at Break (%)	Young's Modulus (MPa)
Chitosan	1	55	3.34 ± 0.38	597 ± 84	1.14 ± 0.01
		60	2.45 ± 0.12	515 ± 24	0.99 ± 0.04
		65	2.50 ± 0.19	512 ± 44	1.06 ± 0.12
	3	55	2.86 ± 0.08	501 ± 10	1.54 ± 0.14
		65	1.99 ± 0.17	590 ± 21	1.05 ± 0.08
	5	55	2.43 ± 0.28	415 ± 37	1.63 ± 0.10
		60	1.73 ± 0.25	362 ± 22	1.55 ± 0.15
		65	1.38 ± 0.30	412 ± 114	1.30 ± 0.09

Table 3: Tensile results for samples with chemical synthesised silica

	Loading of filler (%)	Solvent (%)	Stress at Break (MPa)	Strain at Break (%)	Young's Modulus (MPa)
Chemical synthesis	1	55	2.01 ± 0.28	395 ± 36.63	0.98 ± 0.06
		60	32.86 ± 2.71	788 ± 85.24	8.81 ± 2.39

		65	2.67 ± 0.07	657 ± 33.72	0.97 ± 0.08
		55	1.88 ± 0.09	314 ± 70.71	1.21 ± 0.27
	3	65	8.21 ± 11.38	367 ± 107.45	3.20 ± 4.17
		55	1.79 ± 0.18	230 ± 47.18	2.10 ± 0.13
	5	60	2.24 ± 0.25	275 ± 7.07	1.87 ± 0.13
		65	0.94 ± 0.02	89 ± 22.63	1.68 ± 0.32

Table 4: Tensile results for samples with cellulose

	Loading of filler (%)	Solvent (%)	Stress at Break (MPa)	Strain at Break (%)	Young's Modulus (MPa)
Cellulose		55	3.57 ± 0.13	620 ± 24	1.23 ± 0.34
	1	60	2.46 ± 0.12	624 ± 49	0.92 ± 0.15
		65	3.26 ± 0.05	693 ± 45	0.99 ± 0.05
		55	2.64 ± 0.38	498 ± 53	1.10 ± 0.05
	3	65	2.31 ± 0.40	498 ± 128	1.25 ± 0.03
		55	1.89 ± 0.39	344 ± 34	1.36 ± 0.15
	5	60	2.36 ± 0.11	462 ± 29	1.31 ± 0.21
		65	2.19 ± 0.16	394 ± 18	1.64 ± 0.26

The heptane solvent was used in silicone mixture for better mixing. In this studies, different percentage of heptane used during mixing had been identified to affect the homogeneity of sample such that filler dispersion in silicone matrix depends on the viscosity of silicone mixture. Hence concentration of silicone mixture by adding heptane affect the tensile strength.

Therefore, comparison of the different percentage of solvent for all samples with different types of filler were made. The loading of filler with 1% from cellulose possesses the highest value of stress at break with value 3.57 MPa, meanwhile the loading filler with 3% and 5% from chitosan possesses the highest value of stress at break with value 2.86 MPa and 2.43 MPa with respectively. Next, the percentage of solvent is increased to 60% which is shows the different results for stress versus strain shown in Figure 3. Based on the figure 3, the loading of filler with 1% and 5% from cellulose possesses the highest value of stress at break with value 2.46 MPa and 2.36 MPa with respectively. After that, the percentage of solvent is up to 65% to show another different result for stress versus strain shown in Figure 4. Regarding to the figure 4, it shows that cellulose possesses the highest value of stress at break among the other fillers which indicates with value 3.26 MPa for 1%, 2.31 MPa for 3% and 2.19 MPa for 5%. Sample with 65% of solvent possesses the stiffness and hence it destroys the inherent softness of elastomer for prepared films.

The other results for the mechanical testing i.e. percentage strain at break shows in Figure 2 that loading of filler with 1% cellulose possesses the highest value of strain at break with value 620% meanwhile both loading filler 3% and 5% chitosan possesses highest value of strain at break with value 501% and 415% with respectively. Figure 3 shows different results by indicates that cellulose possesses highest value of strain at break with value 624% for loading of filler 1% meanwhile loading of filler 5% chitosan possesses highest value of strain at break with value 462%. Next, Figure 4 indicates that loading of filler 1% cellulose possesses the highest value of strain at break with value 693% meanwhile both loading of filler 3% and 5% chitosan possesses the highest value of strain at break with value 590% and 412% with respectively. This finding verifies samples with cellulose and chitosan as bio-based filler can withstand high stress before breaking. For strain at break, both filler added into the sample possess enhanced elongation when high loads are applied. The increased strain and high stress at break indicate that cellulose and chitosan filler has a high potential in substituting silica for a better network reinforcement.

However, the plotted data of chemical synthesized silica shows sudden decrement, indicated by the red circle that might be due to the nonhomogeneous filler particle in silicone DE matrices, as shown in Figure 2, 3 and 4 for sample with solvent percentage of 55%, 60% and 65%, respectively. The non-homogeneity indicates not well dispersion of filler. This nonhomogeneous filler in the silicone DE matrix is indicated by the appearance of lumps on the film.

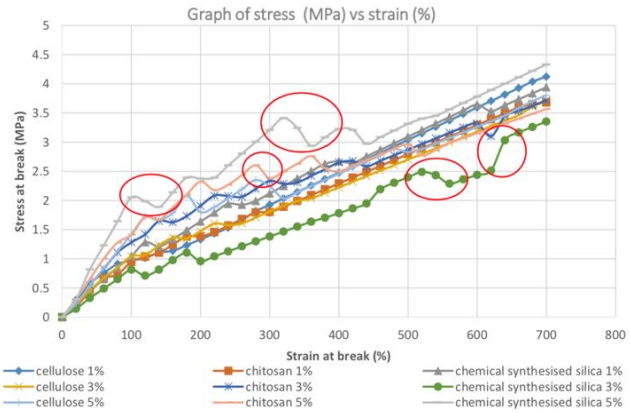

Figure 2: A comparison plot of stress versus strain for percentage solvent of 55%

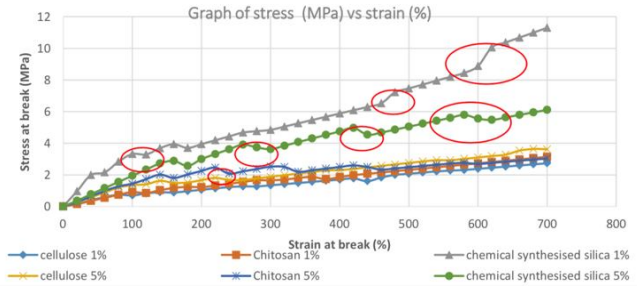

Figure 3: A comparison plot of stress versus strain for percentage solvent of 60%

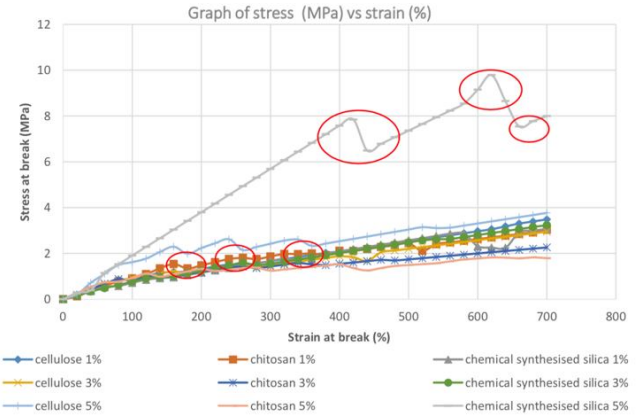

Figure 4: A comparison plot of stress versus strain for percentage solvent i.e. 65%

3.3. Optimization analysis by using RSM

Tensile strength of samples were optimised using Response surface methodology (RSM) by means of Design Expert 12 software. Optimisation on the factors (loading of fillers (%), solvent (%) and type of fillers) were employed by central composite design (CCD). The output responses of optimisation were stress at break (MPa), strain at break (%) and Young's Modulus (MPa). Table 6 shows optimised parameters using RSM for sample with 1% chemical synthesised silica filler and 58% solvent. However, based on actual sample, sample with 1% cellulose filler and 55% solvent possesses desired stress and strain at break (3.26 MPa and 693%, respectively), as shown in Table 5. To verify the optimised sample via RSM, sample of abovementioned parameter i.e. 1% chemical synthesised silica and 58% solvent need to be replicated and verified its properties.

Desired factors from optimisation via RSM are determined by high response (red zone) i.e. high stress and strain at break, as shown in Figure 5 and 7. Sample with chitosan does not possesses high stress and strain at break, as the surface is almost green zone (refer to Figure 6). The surface of optimisation plot for sample containing synthesised silica and cellulose show peaks with red zone, indicating those parameters are favourable for silicone DE, as shown in Figure 5 and 7.

Table 5: Actual tensile strength characterisation using tensile machine

Loading of fillers (%)	Solvent (%)	Type of filler	Stress at break (MPa)	Strain at break (%)
1	65	Cellulose	3.26	693
1	55	Chitosan	3.34	597
1	65	Chemical synthesised silica	2.67	657

Table 6: Optimised parameters generated from RSM

Loading of fillers (%)	Solvent (%)	Type of filler	Stress at break (MPa)	Strain at break (%)
1.000	64.242	Cellulose	4.849	666.329
1.488	55.000	Chitosan	3.770	580.492
1.000	57.999	Chemical synthesised silica	8.628	657.000

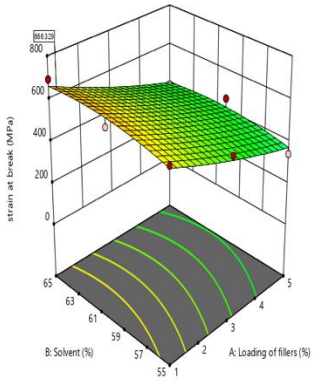

Figure 5: Cellulose (Loading of filler: 1%, Solvent percentage: 65%)

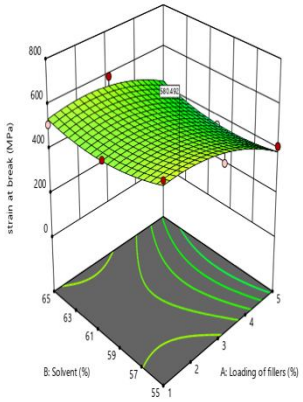

Figure 6: Chitosan (Loading of filler: 1%, Solvent percentage: 55%)

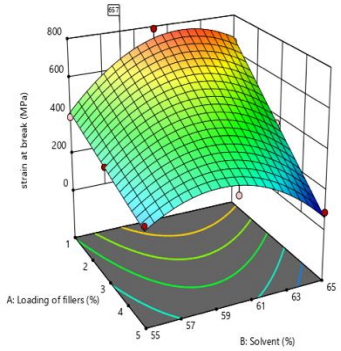

Figure 7: Chemical synthesized silica (Loading of filler: 1%, Solvent percentage: 65%)

4. CONCLUSION

As conclusion, the silicone DE film containing bio based fillers i.e. chitosan, cellulose to enhance the mechanical properties. Each fillers i.e. cellulose, chitosan, chemical synthesised silica were successfully prepared by varying filler loadings at 1wt %, 3wt% and 5wt % and each loading of filler were prepared in different solvent percentage i.e. 55%, 60%, 65%. These bio-based fillers were compared with chemical synthesized silica based on the characterisation of film with respects to mechanical properties and surface morphology so at the end bio-based of fillers replace the silica that currently used and it harmful to the environment. The mechanical properties in terms of stress at break (σ), strain at break (%) and Young's Modulus (Y) of prepared silicone DE films were investigated.

Based on the data of mechanical testing for each different type of fillers with respects to stress and strain relationship indicates that each different type of fillers i.e. chitosan, cellulose, chemical synthesized silica has highest strain means it indicate the fillers can be stretch when applied in generator application. This studies indicates that 1wt % cellulose loading with 65% solvent percentage in the silicone DE shows the highest strain at break of 693 %, stress at break of 3.26 MPa followed by 3wt % chitosan loading with 65% solvent percentage indicates the strain at break of 590 % and 1wt % chemical synthesised silica loading with 65% solvent percentage indicates the strain at break. However, chemical synthesized silica shows sudden decrement that might be due to the nonhomogeneous filler particle in silicone DE matrices. Hence, highest of strain at break enhance the film easy to be stretch when applied on the generator application.

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