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Optimisation of Tensile Strength by Means of Taguchi and Response Surface Methods For Silicone Dielectric Elastomer

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Abstract: Silicone dielectric elastomer (DE) can be utilised as a smart material to harvest energy from wave besides other applications such as actuator and sensor. Previous works have been conducted to increase permittivity and electrical break down without destroying the pristine softness of elastomer via incorporation of high permittivity filler, network reinforcing filler, copolymerisation, and micro capsulation. Among these works, incorporation of filler for enhancement of electrical and mechanical properties is desired due to not undergoing complex chemical synthesis and reaction, producing so-called a composite silicone DE. Chitosan, cellulose, and silica are fillers that commonly used as network reinforcing agents in silicone DE. Chemical-synthesised silica is commonly used in preparing silicone DE composite for a strong network. In this project, cellulose and chitosan are investigated for bio-based fillers as network reinforcing agents in silicone DE due to sustainability and environmental conservation, replacing chemical-synthesised silica. Based on this previous work, data of tensile strength of prepared film samples was used for optimising filler type, filler loading, and solvent concentration via Taguchi and Response Surface Method (RSM). The optimisation criterion of Taguchi was "the bigger the better", where high stress and strain at break are desired for good performance of silicone DE in wave energy harvester. The optimised tensile strength via Taguchi was compared to Response Surface Method (RSM) by means of Design-Expert software using central composite design to express the responses. The optimised tensile strength by Taguchi indicates that high stress and strain at break occur when utilising 1.00 % and 3.00 % chitosan, and when the silicone mixture was mixed with 60.00 % heptane. On the other hands, high stress and strain at break optimised by RSM is obtained by incorporating 3.00 % cellulose and 60.00 % of heptane in silicone dilution mixture. The optimised stress and strain at break via Taguchi and RSM are rather different and hence validation on tensile strength of sample films at abovementioned conditions need to be performed experimentally.

Keywords: Silicone Dielectric Elastomer, Bio-Based Filler, Tensile Strength, Taguchi, Response Surface Method

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1. Introduction

Malaysia's main energy sources depend heavily on three major fossil fuels: natural gas, coal, and petroleum. Burning fossil fuel, however, contributes to greenhouse gases (GHGs), environmental degradation, and climate change. The highly consumption and highly dependence of fossil fuels lead to depletion of these resources in future. Therefore, an alternative way to generate electricity needs to be planned to meet the current and future electricity demand in Malaysia. As moving towards developed country, Malaysia need a serious plan and strategy to protect energy resources so that the country can sustain developments in many areas such as engineering and technology, education, transportation, manufacturing industry, hospitalisation etc. Malaysia has renewable resources such as biomass, wind, small hydro, solar, and ocean resources, and hence has the potential to achieve sustainable energy [1].

Wave energy is one of the most resourceful and a constant source of energy in the ocean or the shoreline, among all renewable sources. The production of wave energy provides renewable and sustainable energy with a new perspective and methodology. The eastern coast of the Malaysian Peninsula has direct exposure to the South China Sea and is a potential location for Malaysia Peninsular to construct a wave power plant to host wave power devices. Aside from the east coast, the sea regions between Benyal of Bay (BoB) and the Strait of Malacca (MS) have also become a wave farm site for the production of wave power systems [2].

In this research, the optimisation of tensile strength for silicone dielectric elastomer (DE) consisting of different types of fillers e.g. chemical synthesised silica, bio-based silica, chitosan, and cellulose to seek the best silicone elastomer with high tensile strength to achieve high elongation to be utilised as wave energy harvester for the best performance in generating power. The optimised parameter is mainly based on stress and strain at break due to the nature of incorporated filler that serves as a network reinforcing agent. The design of experiment (DOE) will be based on three factors which are the type of filler, percentage of filler loading, and solvent percentage. Each factor also will be based on three levels, for instance, filler types are silica, chitosan, and cellulose. While for the levels of percentage of filler loading and solvent percentage are 1.00 %, 3.00 %, and 5.00 %, and 55.00 %, 60.00 %, and 65.00 %, respectively. The optimisation responses are stress at break, and strain at break. All these factors, levels, and responses were obtained from the previous study with the title of ‘Characterisation of Silicone Dielectric Elastomer With Incorporation Of Different Types of Fillers As Network Reinforcing Agents To Be Utilised In Wave Energy Harvester’ that done by Jumadi (2020). This study is chosen as a basis for the optimisation as the study provided complete raw data for factors and levels, and also has complete results for the responses.

The silicone DE is to be optimised as the silica network without reinforcement of silica fillers has low mechanical properties of end-linking linear polydimethyl siloxanes (PDMS) which are interlinked with medium-functional or even high-function cross-linkers without any filler [3]. Vudayagiri et al., (2014) has shown in it study that the reinforcement of the silica filler has improve the mechnical properties of the silicone DE where the inorganic oxide filler such as TiO_2 have been reinforced into silicone dielectric elastomer to enhance the relative permittivity and thermal stability. The cross-linker as shown in Figure 1 acts as the support system to the silicone polymer [3].

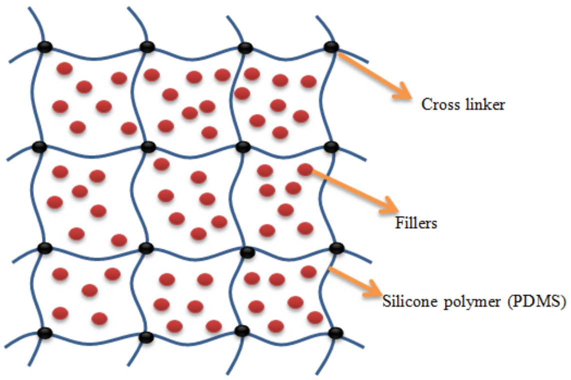


Figure 1: A diagram of network containing PDMS, cross linker and fillers[3]

1.1 Taguchi Method

Taguchi method is one of the common optimisation tools practiced by industrial engineers and chemists to maximise the yield of product at optimum conditions. Taguchi Method is a reliable, multiparameter mathematical technique that uses less tests to define and optimise the required response parameters. The Taguchi method includes parameter design, system design, and tolerance design procedures in order to achieve a stable process and result for the highest product quality. The purpose of the system design process is to evaluate the appropriate work levels for the design factors. The parameter design method defines the factor levels which can produce the best output in the analysed product or process. The technique of tolerance design is used to adjust the outcome of the parameter design by increasing the tolerance rates of the variables that significantly affect the product or process. Procedures for designing a parameter provide the three main steps [4,5].

The optimisation using the Taguchi method is based on two approach which is "the smaller is better" and "the bigger is better". 'The smaller is better" state is used when the responses of the system need to be optimised for minimum and it can be calculated by SN ratio in Eq.1. For the system that need to be optimise with the response in maximum, "the bigger is better" state is choose and it can be calculated by SN ratio in Eq. 2 [6].

$$SN_S = -10 \log \left[\frac{1}{n} \sum_{i=1}^{n} yi^2 \right] \qquad \textit{Eq. 1}$$

$$SN_L = -10 \log \left[\frac{1}{n} \sum_{i=1}^{n} \frac{1}{yi^2} \right] \qquad \textit{Eq. 2}$$

In this equation, yi indicated the value of the response and n indicated the trial number of experiment. The optimum value for the factor in the plot of Taguchi is the level which yield the highest value of sum of SN ratio [7].

In this research, only 'the bigger the better' is applicable in optimisation of mechanical properties of silicone DE film. Taguchi method was performed by using Microsoft Excel as a conventional method.

1.2 Response Surface Method

RSM is a commonly used mathematical and statistical approach to modelling and evaluate processes to maximise the response to the interest by different variables [8]. The process-affecting parameters are called dependent variables which also called the responses. RSM is a method that is based on surface placing. Therefore, the key goals of an RSM analysis are to understand the response surface topography including the maximum central, central, minimum, and ridge lines, and to identify the area where the most suitable response occurs. The RSM investigates a suitable relationship of approximation between the factors and response variables and identifies the optimal operating conditions for the characteristics of silicone dielectric elastomer. Box-Behnken designs (BBD) and central composite design (CCD) are two key experimental designs used in the methodology for the response surface [9]. In this study, the CCD is used as a methodology for the response surface. The outcome from RSM gives easily view of response surfaces from all angles with rotatable 3D plots, interactive 2D graphs, and point prediction to find the optimum condition.

2. Materials and Methods

The methodology focused on the optimisation of characteristics of silicone dielectric elastomers via two methods which were Taguchi method as a conventional method and RSM using Design Expert software. The factors and the levels for both optimisation method used in this research are the same as shown in Table 1.

Table 1: Factors, Levels, and Response for silicone DE samples with different fillers

	Factor			Response	
	Type of filler	Loading of fillers, %	Solvent, %	Stress at break, MPa	Strain at break, %
Level	Chemical synthesised silica	1	55		
	Chitosan	3	60		
	Cellulose	5	65		

The factors, levels, and responses for the optimisation of mechanical properties were obtained from the study done by Jumadi (2020) [9]. Based on the study, the factor chosen were three, which were (1) Type of filler, (2) Loading of fillers, and (3) Solvent. these factors have been chosen because they affect the tensile strength, ultimate elongation and hardness of the cured silicone [9]. For each factor, three levels were chosen from the study. The levels chosen for each factor also three where for the type of filler are chemical synthesized silica, chitosan, and cellulose. These three levels were chosen to identify the optimum type of filler for silicone DE film. The chemical synthesised silica was used as a reference sample. As for the loading of fillers, the levels were 1.00 %, 3.00 %, and 5.00 %. Filler enhances network of silicone DE, as a reinforcing agent. As for the solvent, heptane was used as a solvent to mix silicone premix with fillers for desired viscosity at concentration of 55.00 %, 60.00 % and 65.00 %. Previous work by Jumadi (2020), the responses chosen were stress and strain at break.

2.1 Taguchi Method

Taguchi design table or also known as design of experiment (DOE) table is a type of general fractional factorial design. The DOE is constructed based on the experimental result from previous studies. Based on the Table 2, the three factors and three levels resulting in the factorial design of is L_{27}. L_{27} is an orthogonal array where it was chosen due to the number of factors and levels obtained from the study and was meant for understanding the effect of three factors each having three-level values (3^3). The designation of DOE was essential as a guide for the combination of factors and levels. The DOE designation was based on the matrix outlined in Table 2. Table 3 displays the DOE of optimisation, where the designation was built on the basis of Table 2.

Table 2: Three levels and three factors in the experiment

Factor/level	0	1	2
A- Type of filler	Chemical synthesized silica	Chitosan	Cellulose
B - Loading of filler	1	3	5
C - Solvent	55	60	65

Table 3: Design of Experiment

Experiment set	Designation	Type of filler	Loading of fillers, %	Solvent %	Stress at break, MPa	Strain at break, %
A1	A0B0C0	Chemical synthesised silica	1	55	2.01	395.00
A2	A0B0C1	Chemical synthesised silica	1	60	32.86	788.00
A3	A0B0C2	Chemical synthesised silica	1	65	2.67	657.00
A4	A0B1C0	Chemical synthesised silica	3	55	1.88	314.00
A5	A0B1C1	Chemical synthesised silica	3	60	11.21	353.55
A6	A0B1C2	Chemical synthesised silica	3	65	8.21	367.00
A7	A0B2C0	Chemical synthesised silica	5	55	1.79	230.00
A8	A0B2C1	Chemical synthesised silica	5	60	2.24	275.00
A9	A0B2C2	Chemical synthesised silica	5	65	0.94	89.00
A10	A1B0C0	Chitosan	1	55	3.57	620.00
A11	A1B0C1	Chitosan	1	60	2.46	624.00
A12	A1B0C2	Chitosan	1	65	3.26	693.00
A13	A1B1C0	Chitosan	3	55	2.64	498.00
A14	A1B1C1	Chitosan	3	60	19.61	544.17
A15	A1B1C2	Chitosan	3	65	2.31	498.00
A16	A1B2C0	Chitosan	5	55	1.89	344.00
A17	A1B2C1	Chitosan	5	60	2.36	462.00
A18	A1B2C2	Chitosan	5	65	2.19	394.00
A19	A2B0C0	Cellulose	1	55	3.34	597.00
A20	A2B0C1	Cellulose	1	60	2.45	515.00
A21	A2B0C2	Cellulose	1	65	2.50	512.00
A22	A2B1C0	Cellulose	3	55	2.86	501.00
A23	A2B1C1	Cellulose	3	60	34.00	763.32
A24	A2B1C2	Cellulose	3	65	1.99	590.00
A25	A2B2C0	Cellulose	5	55	2.43	415.00
A26	A2B2C1	Cellulose	5	60	1.73	362.00
A27	A2B2C2	Cellulose	5	65	1.38	412.00

2.1.1 SN ratio

The Taguchi conventional statistic method was used to optimise two response in mechanical properties which were stress at break and strain at break. This study aims to obtain the highest stress at break and strain at break. Therefore, for the optimisation of the characteristics of the silicone dielectric elastomer, the “the bigger the better” criterion was used to calculate SN ratio to maximise the responses. “The bigger the better” criterion is selected because the responses of the optimisation in this study should be in the highest value. The optimum value for the factor in the plot of Taguchi is the level which yield the highest value of sum of SN ratio [7]. Using the expected “the bigger the better” SN ratio Eq. 2, the optimum levels of the chosen design parameters will be used as a last step of the Taguchi method.

2.2 Response Surface Method

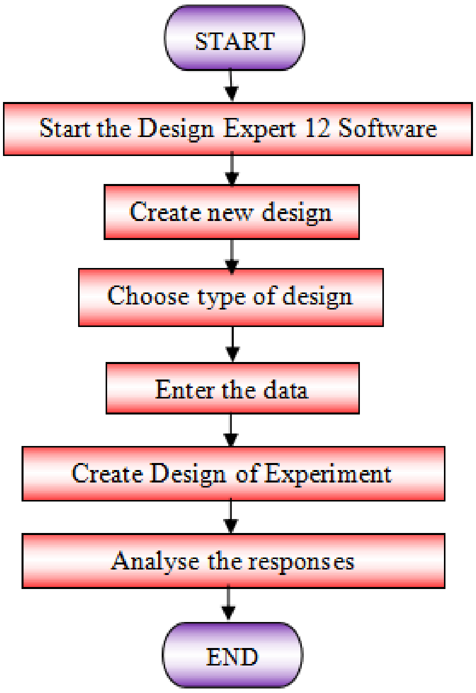


Figure 2: Flowchart of analysing data via RSM method

As shown in Figure 2, the step of optimisation using RSM start with by opening the the software and the 'File' on the left of the toolbar was clicked and 'New Design' is chosen. After that, the 'Response Surface' from the list of designs on the left that show the designs available for RSM was clicked. Then, then 'Central Composite' was chosen in the optimisation of mechanical characteristics of silicone DE film as the there were three factors where two factors were numeric factor (percentage loading of filler and solvent) and one factor was categoric factor (type of filler). The number of factors as mentioned before and the 'Name' of the factors for numeric factors were then keyed in. The low and high value of levels for both factors were keyed in where for 'Loading of filler' the low value is '1' and the high value is '5' while for 'Solvent' the low value is '55' and the high value is '65'. The center points was set to '3' and the alpha set as 'Face centered' and the number of runs which is 11 runs for each factor was shown in the figure. The 'Next' was clicked and, the 'Name' for the categoric factor, number of levels and the levels were keyed in. The type of factor chosen was 'Nominal'. After that the 'Next' was clicked again. The number of responses and the 'Name' and 'Unit' were keyed in. Lastly, the 'Finish' was clicked to complete the design specification process. The Design Expert then showed DOE with 33 runs. The values for responses can be entered either by opening the file of data of stress at break and strain at break or by manually entering the values. The data is now ready to be optimised by clicking the tabs such as 'ANOVA', 'Diagnostics', 'Model Graphs' and etc [11].

3. Results and Discussion

The results of optimisation study carried out by Jumadi (2020) on mechanical properties parameters such as types of filler, loading of filler, and solvent percentage is used as raw data for optimisation by means of Taguchi and RSM using Design Expert 12 software. For Taguchi, three levels and three factors of the Design of Experiment (DOE) were considered, because Jumadi (2020) emphasised only three factors in his studies, such as types of filler, loading of filler, and solvent percentage that can affect the responses. For three levels and three factors, the fractional factorial designs used the L_{27} orthogonal array. In the matrix, levels were represented by '0', '1', and '2' for each factor, where '0' is the minimum value and '2' is the maximum value as shown in Table 2.

3.1 Taguchi Method

Based on Figure 3, the percentage of solvent used has the greatest effect on the stress at break and followed by percentage loading of filler and type of filler. It is observed that the optimum condition of stress at break for three levels and three factors is at A0 (type of filler is chemical synthesised silica), B1 (loading of filler at 3.00 %), and C1 (solvent at 60.00 %). The value of stress at break at these conditions is 11.21 MPa. However, the objective of the optimisation is to find the optimum condition for sustainable filler, therefore, rather than choosing chemical synthesised silica, chitosan was chosen as an optimum condition for type of filler as it produced the second highest of stress at break as indicated in 'red circle'. Thus, the Taguchi method suggests that the preparation of silicone DE film is recommended to run under these (A1B1C1) conditions to obtain high stress at break.

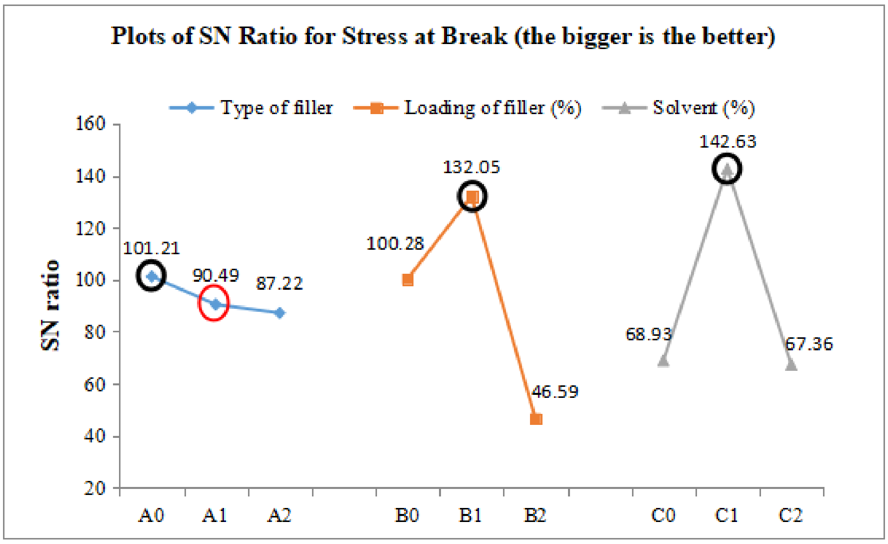


Figure 3: Plot of SN ratio for stress at break

Based on Figure 4, the percentage of loading of filler used has the greatest effect on the strain at break and followed by the type of filler and percentage of solvent. It is observed that the optimum condition of strain at break for three levels and three factors is at A1 (type of filler is chitosan), B0 (loading of filler at 1.00 %), and C1 (solvent at 60.00 %). The value of strain at break at these conditions is 624.00 %. Thus, the Taguchi method suggests that the preparation of silicone DE film is recommended to run under these (A1B0C1) conditions to obtain high strain at break.

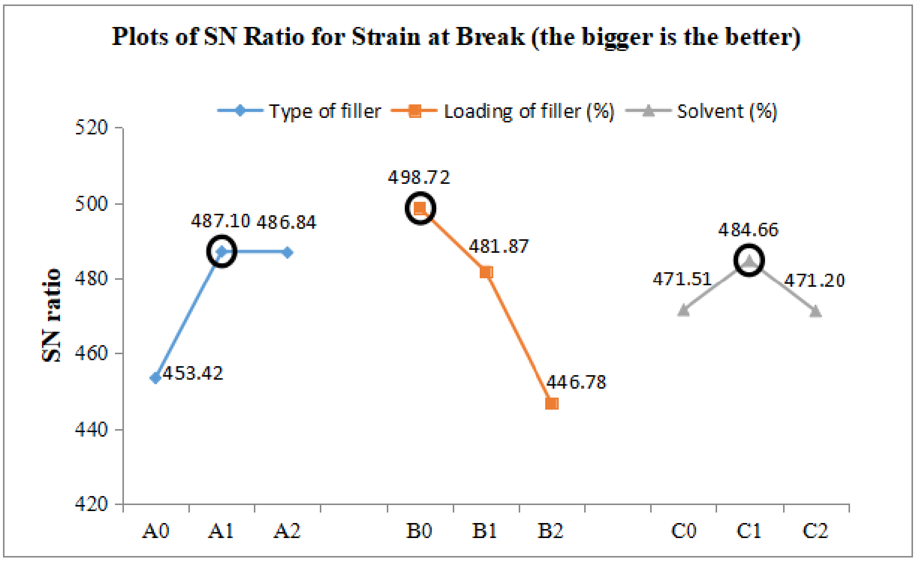
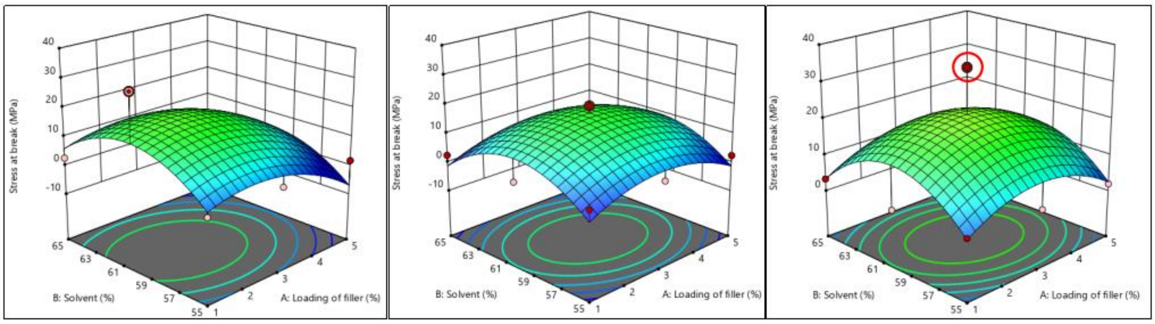


Figure 4: Plot of SN ratio for strain at break

3.2 Response Surface Method

3D plot below gives a better view to see where the optimum condition may occur. Based on Figure 5 and Table 4, the 3D plot and the table indicate that the highest value of stress at break is 34 MPa that occurs at loading of filler of 3.00 %, solvent of 60.00 %, and type of filler of cellulose as indicated by 'red circle'. This result is different to the optimum analysed by Taguchi method in terms of type of filler.



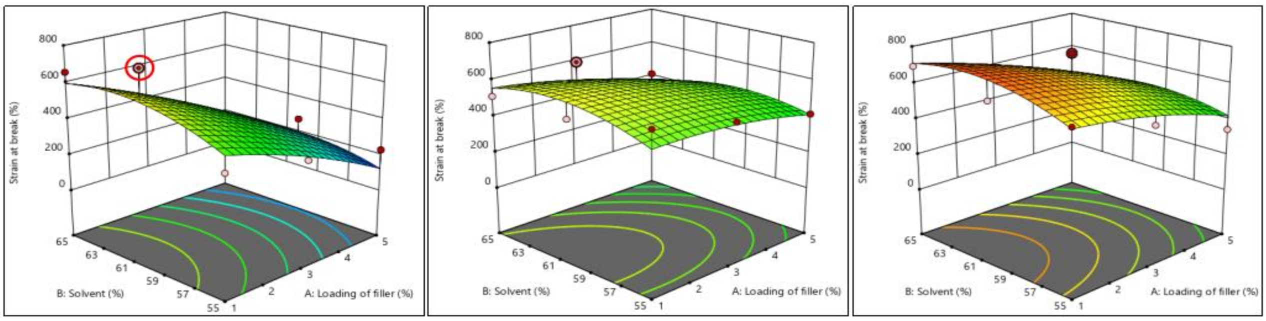
a) Chemical synthesised silica b) Chitosan c) Cellulose

Figure 5: 3D plot of loading of filler, solvent, and type of filler for stress at break by Design Expert

Table 4: The optimum conditions for every type of fillers based on 3D plot and its stress at break by Design Expert

Type of filler	Chemical synthesised silica	Chitosan	cellulose
Loading of filler (%)	1	3	3
Solvent (%)	60	60	60
Stress at break	32.86	19.61	34

3D plot below gives a better view to see where the optimum condition may occur. Based on Figure 6 and Table 5, the 3D plot and indicate that that the highest value of strain at break is 788.00 % that occurs at loading of filler of 1.00 %, solvent of 60.00 %, and type of filler of chemical synthesised silica as indicated by 'rectangular'. This result is different to the optimum analysed by Taguchi method in terms of type. However, as the chemical synthesised silica is used as control value, cellulose will be choosen as an optimum condition for type of filler as it produced the second highest value of strain at break as indicated in Table 5.



a) Chemical synthesised silica b) Chitosan c) Cellulose

Figure 6: 3D plot of loading of filler, solvent, and type of filler for strain at break by Design Expert

Table 5: The optimum conditions for every type of fillers based on 3D plot and its strain at break by Design Expert

Type of filler	Chemical synthesised silica	Chitosan	cellulose
Loading of filler (%)	1	3	3

Solvent (%)	60	65	60
Strain at break	788	590	763.2

3.3 Comparison in Taguchi and Response Surface Methods

Two statistical methods were used to optimise experimental data done by Jumadi (2020) in reinforcing sustainable bio-based filler into silicone DE-film. Based on previous analysis discussion on optimisation via Taguchi and RSM methods, optimum condition for silicone DE films with filler is non identical for stress and strain at break, as shown in Table 6.

Table 6: Overall optimum conditions for Taguchi and Response Surface Methods

Response	Optimisation Method	Taguchi Method	Response Surface Method
Stress at break	Type of filler	Chitosan	Cellulose
	Loading of filler (%)	3	3
	Solvent (%)	60	60
Strain at break	Type of filler	Chitosan	Cellulose
	Loading of filler (%)	1	3
	Solvent (%)	60	60

For stress at break, a film show the highest stress at type of filler of chitosan and cellulose which analysed by Taguchi and Design Expert, respectively. For strain at break, a film show the highest strain at loading filler of 1.00 % chitosan and 3.00 % cellulose which analysed by Taguchi and Design Expert, respectively. Both Taguchi and RSM methods show that the film was well prepared when diluting silicone mixture at 60.00 % of heptane. Based on these optimum conditions, the value of stress at break for Taguchi method and RSM were 19.61 MPa and 34 MPa, respectively. For the value of strain at break, the Taguchi method obtained 624.00 % while RSM obtained 763.20 %.

Here we can see that cellulose produced better strain at break compared to chitosan. This is probably due to the characteristics of the cellulose where it has a great dispersity where the cellulose fillers were spread homogenised, it also has good processability, high crystallinity, sustainable and biodegradable nature [12]. Other than that, the mechanical, water vapour barrier, and thermal properties of the film were also enhanced by the reinforcement of the cellulose fillers that lead to the production of high strain at break. This has been proved in the study conducted by Barros-Alexandrino et al. (2018), where the study compared the effects of cellulose and chitosan as reinforcement fillers on the mechanical, water vapor barrier, and thermal properties. From the study, cellulose is also found to be more efficient in enhancing tensile strength and strain at break compared to chitosan [13].

4. Conclusion

Herein, the optimisation of tensile strength for silicone DE was performed using Taguchi and response surface methods, for preparing film sample with high tensile strength. Chitosan and cellulose bio-based fillers have been compared to chemically synthesised silica based on their stress and strain at break. From Taguchi, the optimum conditions were shown by factor/level of A1B1C1 and A1B0C1, where it possessed highest stress at break (11.21 MPa) and highest strain at break (624.00 %), respectively. For RSM, the optimum condition was shown by sample with 3.00 % cellulose at 60.00 % heptane dilution, due to highest stress at break of 34 MPa and strain at break of 763.20 %.

Finally, both optimisation results are different and hence, for further research, a validation from both optimisation methods need to be carried out experimentally in preparing samples according to abovementioned sample condition (A1B1C1 and A1B0C1) and then these prepared sample need to be characterised the tensile strength for final verification.

Acknowledgement

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