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Optimisation of Droplet Size and pH Via Taguchi and Response Surface Method (RSM) For Stabilised Cosmeceutical Cream Containing Virgin Coconut Oil (VCO)

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Abstract: The demand for herbal/natural-based cream and cosmetic is high due to safer alternatives, good skin result and many other advantages. This high demand of natural-based cream is resulted from reported case of bad effect by the consumers who utilise cream that contains harmful ingredients such as mercury, hydroquinone, lead, etc. They use these type of cream for many purposes such as beauty, wound, irritation, skin allergy and weight loss. In Malaysia's market, many of these harmful creams do not go a proper clinical test and this has led risk to the consumers. In this project, a stable water-based cream containing virgin coconut oil (VCO) as an oil phase is used as a base cream for cosmeceutical wound healing cream when incorporating an extract of *Chromoleana odorata*. Previous works had shown a stable cream i.e. no phases separation when incorporating 1.00 % extract of *Chromoleana odorata* into VCO cream in a ratio of 90.00 % water and 10.00 % VCO. This project focused on the optimisation of oil droplet size in water-oil emulsion and pH when varying parameters such as ratio oil to water, emulsifier concentration and homogenizer speed via statistical methods i.e. Taguchi and Response Surface Method (RSM) by means of Design Expert software. A stable cream possesses microsized droplet and a good cream is suitable with human skin pH ranging from 5.5 to 6.5. Therefore, Taguchi criterion of droplet size was "the smaller the better" and while pH between 5.5 to 6.5 was chosen. The optimised droplet size and pH 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 droplet size and pH by Taguchi indicates that small droplet size and low level of pH occur when utilising 10.00 %, 15.00 % oil and 5:95 ratio oil to water, and when the percentage of emulsifier was at 1.00 % and 5.00 %. On the other hands, same percentage of oil and ratio oil to water as Taguchi method is obtained for small droplet size and low level of pH optimised by RSM while percentage of emulsifier used was 1.00 % and 3.00 %. The optimised droplet size and pH via Taguchi and RSM are rather different and hence validation on both droplet size and pH at abovementioned conditions need to be performed experimentally.

Keywords: Cosmeceutical Cream, Taguchi, Response Surface Method, Virgin Coconut Oil, Microsized Droplet

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1. Introduction

The demand for herbal or natural-based cosmetics, compared to the non-natural-based ones, is high nowadays. Natural-based cosmetics are safe to use and possess no adverse effects to the consumers [1]. Various types of pharmaceutical products in the market such as cosmetic, baby cream, skin care, antiseptic cream, etc., however, their content could contain harmful ingredients which could give severe side effect e.g. irritation, severe allergy, discoloration, skin cancer etc. These harmful products usually do not an approval from National Pharmaceutical Regulatory Agency (NPRA) and therefore the products do not pass the clinical test [2]. This situation is due to illegal imported product and then these products mainly skin care and cosmetic being re-branded as another brand in the market. Due to many cases regarding of bad effect from using these harmful cosmetics and skin care, users are more alert on to choose safe products such as organic- and natural-based products. Common ingredients in the harmful products consists of hydroquinone, lead, mercury, etc [3]. Hence natural-based products are extensively studied. In this project, water-based cream which contains virgin coconut oil and 1.00 % polymeric emulsifier were prepared previously with different ratio of oil to water, various emulsifier loading and different homogenizer speed. Herein, droplet size and pH of previously prepared cream from two previous studies which are Nazarudin (2020) [4] and Jalal (2019) [5] were optimised for desired properties i.e. microsized droplet and pH 5.5 to 6.5, respectively. The optimised outputs (droplet size and pH) were optimised via Taguchi and Response Surface Method (RSM) by means of Design Expert software. Droplet size and pH of cream were optimised based on ratio of oil to water, emulsifier loadings, and homogenizer speed, which were known as the factors in design of experiment (DOE). Optimisation was done to find the perfect formulation between oil, water and emulsifier in order to produce a light-weight, stable and safe cream which possessed a small droplet size and healthy skin pH.

1.1 Taguchi method

Taguchi uses orthogonal arrays (OAs) to minimize the time and cost of experiments in analyzing all factors, and uses the signal-to-noise ratio (S/N) to analyze the experimental data and find the optimum combination of parameters. Taguchi suggests using the S/N ratio to calculate the quality characteristics that deviate from the values desired. The optimal levels of the process factors are determined on the basis of an analysis of the S/N ratio [6]. Taguchi approach includes procedures for device design, parameter design, and tolerance design to achieve a stable process and result for the highest quality of products [6]. The main confidence of Taguchi 's techniques is the use of parameter design, which is a product or process design engineering method that focuses on determining the parameter (factor) settings that produce the best levels of a quality characteristic (performance measure) with minimum variation [6]. Taguchi designs provide a powerful and effective process design method that operates consistently and optimally across a variety of conditions. To determine the best design, a strategically designed experiment is required which exposes the process to different levels of design parameters. Taguchi's approach to design of experiments is easy to adopt and applied to users with limited statistical knowledge; thus, it has become widely popular in engineering and scientific community. Taguchi have specified three situations which are include the larger the better, the smaller the better and on-target, minimum-variation [6].

1.2 Response surface method

Response surface method (RSM) is a commonly used mathematical and statistical method for modelling and analysing a process where various variables affect the response of interest, and the purpose of this approach is to optimise the response [7]. Surface response is a method that is based on surface positioning. The RSM investigates a suitable relationship of approximation between input and output variables and identifies the optimal operating conditions for a system under study or a region of factor field that meets operating requirements [8]. Central composite design (CCD) has been selected as one of the most popular RSM design. CCD optimisation will allow a wide range of parameters to be screened as well as the function of each factor. Furthermore, CCD can also evaluate a single variable, or the cumulative effect of the response variables [9]. The RSM is done on Design Expert software. Design Expert is a software component designed to assist in the design and analysis of multifactor experiments [10]. This software is used in this study to help designing an experiment to observe how a characteristic such as droplet size varies with changes in percentages of ingredients such as water and oil.

2. Materials and Methods

The methodology focused on the optimisation of droplet size and pH level via Taguchi method and RSM. The selected factors and levels were based on previous study by Nazarudin (2020) [4] and Jalal (2019) [5]. For cream prepared by Nazarudin (2020) [4], VCO as the oil phase, distilled water (DI) water as the aqueous phase and the emulsifier were used to produce the base cream samples. The ratio of DI water to VCO and loading of emulsifier were selected as controllable factors in DOE. For each respective factor, three levels were selected in this study, as both factors and levels shown in Table 1. Based on the selected factors and levels, these factors compromising ratio of water to oil and emulsifier loading were optimised to obtain the desired properties i.e. microsized droplet and healthy skin pH. Then, the optimisation tools on these factors were executed via Taguchi and RSM by means of Design Expert software.

Table 1: Design of experiment (DOE) containing three factors and three levels for prepared VCO cream by Nazaruddin (2020)

	Factor			Response*	
	VCO %	Distilled water (%)	Emulsifier (%)	pH	Droplet size (µm)
Levels	5	85	1		
	10	90	3		
	15	95	5		

For VCO cream prepared by Jalal (2019) [5], Taguchi and RSM were used to optimise pH and droplet size of oil in oil-water emulsion. Compared to work done by Nazaruddin (2020), the prepared creams were optimised based on three factors i.e. ratio oil to water, emulsifier loading and homogenizer speed, where each factor contained three level quantitative measurements and resulted in 27 orthogonal arrays (denoted as L_{27}), as shown in Table 2.

Table 2: Design of experiment (DOE) containing three factors and three levels for prepared VCO cream by Jalal (2019)

	Factor			Response*	
	Ratio Oil to Water	Emulsifier (%)	Speed of homogenizer (rpm)	pH	Droplet size (µm)
Levels	5:95	1	5000		
	10:90	3	7500		
	15:85	5	10000		

2.1 Taguchi method

In this research, the Taguchi was used to optimise the formulation of a base cream containing virgin coconut oil (VCO) for microsized droplet size and skin pH. The criterion of 'the smaller the better' was applicable in optimisation of pH level and droplet size. The value of pH also was selected as 'the smaller the better' because from previous pH data, most of the pH level for prepared creams were far from the maximum range of skin pH (5.5 to 6.5) where the range of the pH for the prepared cream were from 6.0 to 7.5. Therefore, the smaller value was chosen so that the value will near to skin pH.

A Taguchi orthogonal array for 3 levels and 3 factors should be 27. However, for the experimental data from Nazarudin (2020) [4], the DOE was not based on orthogonal arrays as practised for Taguchi; instead, the optimisation was performed based on the number of samples generated by DOE via Design Expert software. Number of cream samples generated by Design Expert is rather random than formulated number. Hence based on DOE generated by Design Expert, the experiment done only consisted of 15 sets, as shown in Table 3.

Table 3: Operational parameters for Taguchi optimisation

Experiment	Designation	VCO (%)	Distilled water (%)	Emulsifier (%)	pH level	Droplet size (μm)
1	A0B0C0	10	90	3	6.73	5.413
2	A0B0C2	15	90	3	7.44	2.582
3	A0B1C1	10	90	1	6.60	3.126
4	A0B2C0	5	95	1	7.26	3.477
5	A0B2C2	5	85	1	6.99	2.174
6	A1B0C1	5	85	5	6.98	4.111
7	A1B1C0	5	90	3	7.16	2.129
8	A1B1C1	10	85	3	6.89	2.684
9	A1B1C2	15	95	3	6.94	2.458
10	A1B2C1	5	95	5	7.54	2.378
11	A2B0C0	15	85	5	7.57	4.621
12	A2B0C2	10	90	5	6.05	2.605
13	A2B1C1	15	95	1	7.28	5.334
14	A2B2C0	15	85	1	5.92	4.360
15	A2B2C2	10	95	1	7.39	3.262

Whereas for the study done by Jalal (2019) [5], a standard L_{27} (3^3) orthogonal array was selected for three levels and three factors due use of Taguchi optimisation tool. The experimental layout for the three factors using the standard L_{27} (3^3) orthogonal array is shown in Table 4.

Table 4: Operational parameters for Taguchi optimisation

Experiment	Designation	Ratio of Oil to Water (%)	Emulsifier (%)	Speed of homogenizer (rpm)	pH level	Droplet size (μm)
1	A0B0C0	5:95	1	5000	6.80	17.9
2	A0B0C1	5:95	1	7500	6.87	12.2
3	A0B0C2	5:95	1	10000	6.92	9.3
4	A0B1C0	5:95	3	5000	6.96	9.8
5	A0B1C1	5:95	3	7500	6.71	6.5
6	A0B1C2	5:95	3	10000	6.95	6.4
7	A0B2C0	5:95	5	5000	6.87	6.9
8	A0B2C1	5:95	5	7500	6.77	7.2
9	A0B2C2	5:95	5	10000	6.99	6.3
10	A1B0C0	10:90	1	5000	6.97	15.1
11	A1B0C1	10:90	1	7500	6.79	16.3
12	A1B0C2	10:90	1	10000	7.24	10.6
13	A1B1C0	10:90	3	5000	7.11	9.6
14	A1B1C1	10:90	3	7500	7.08	13.1
15	A1B1C2	10:90	3	10000	7.04	13.9
16	A1B2C0	10:90	5	5000	7.13	13.7
17	A1B2C1	10:90	5	7500	6.99	12.6
18	A1B2C2	10:90	5	10000	6.98	9.0
19	A2B0C0	15:85	1	5000	7.03	17.4

20	A2B0C1	15:85	1	7500	7.13	24.3
21	A2B0C2	15:85	1	10000	7.14	17.7
22	A2B1C0	15:85	3	5000	7.14	20.2
23	A2B1C1	15:85	3	7500	7.16	20.9
24	A2B1C2	15:85	3	10000	7.06	18.0
25	A2B2C0	15:85	5	5000	7.19	11.0
26	A2B2C1	15:85	5	7500	7.15	12.2
27	A2B2C2	15:85	5	10000	7.13	11.4

For criterion of 'the smaller the better', the formula that will be used to calculate Signal-Noise (S/N) ratio to minimize droplet size and loss tangent is shown in *Eq.* 1 below.

$$S/N = -10\log\frac{1}{n}\left[\sum_{i=1}^{n} y_i^2\right] \qquad Eq.\ 1$$

Where y_i is the independent variables and n is the number of replicates. In the Taguchi plot, the level which gives the highest value of sum of S/N ratio is the optimum value for that particular factor.

2.2 RSM via *Design Expert 12*

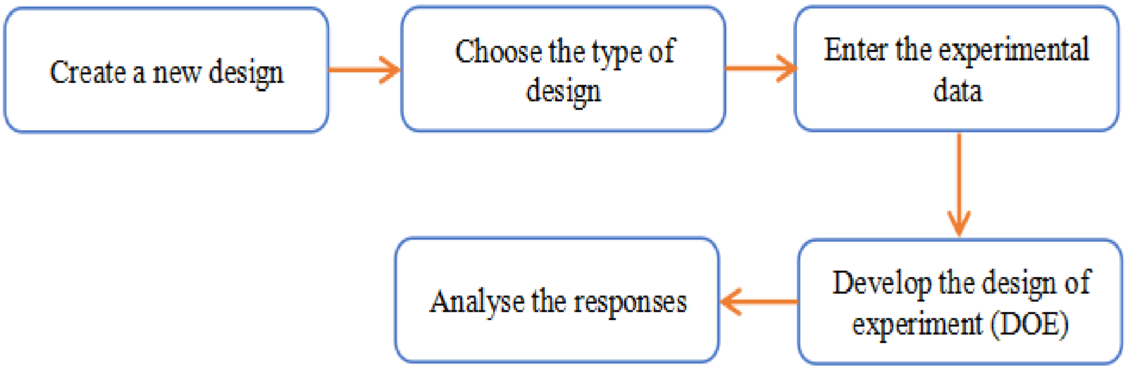


Figure 1: Flowchart of RSM by means of Design Expert software

RSM was used to optimise the formulation of a base cream containing VCO for small droplet size and skin pH. RSM analysed by Design Expert 12 using its own generated DOE samples which contained random number of samples. The outcome from RSM will easily give the 3D view in term of response surface 360 with rotatable 3D plot for finding optimum condition.

As shown in Figure 1, the optimisation using RSM method start when a new design is created. The type of design is chosen. In this study, Central Composite design (CCD) was choosen. The factors and levels based on Table 1 and Table 2 were keyed in as well as the responses. The Design Expert then will display the DOE with 15 runs for the study done by Nazarudin (2020) [4] and 20 runs for the study done by Jalal (2019) [5]. Then, the experimental data for the responses will be entered and the data can be analysed.

3. Results and Discussion

The results of optimisation taken from the previous studies done by Jalal (2019) [5] and Nazarudin (2020) [4] on formulation of base cream which are percentage of VCO, percentage of DI water, percentage of emulsifier and homogenizer speed using two statistical methods were discussed intensively. These two statistical methods include one conventional statistic methods and one statistical software methods. The conventional statistic method was Taguchi while the statistical software methods was Design-Expert 12. The results from these two studies then will be compared.

3.1 Taguchi method

Based on relative stiffness, percentage of emulsifier, percentage of water and oil has the same effect on pH level of base cream. It is obvious to observe that the optimum condition of pH level for data from study done by Nazaruddin (2020) [4] is at A2 (percentage of oil at 15.00 %), B0 (percentage of distilled water at 85.00 %) and C0 (percentage of emulsifier at 1.00 %) as shown in Figure 2. The value of pH level at these conditions is 7.57 as shown in Table 3. Thus, Taguchi method suggests that the formulation of base cream is recommended to run under these conditions in order to obtain low pH level. The optimum condition for the data from Jalal (2019) [5] is at A0 (ratio of oil to water at 5:95), B0 (percentage of emulsifier at 1.00 %) and C1 (speed of homogenizer at 5000 rpm) as displayed in Figure 3. Therefore, the value of pH level at these conditions is 6.87 as displayed in Table 4.

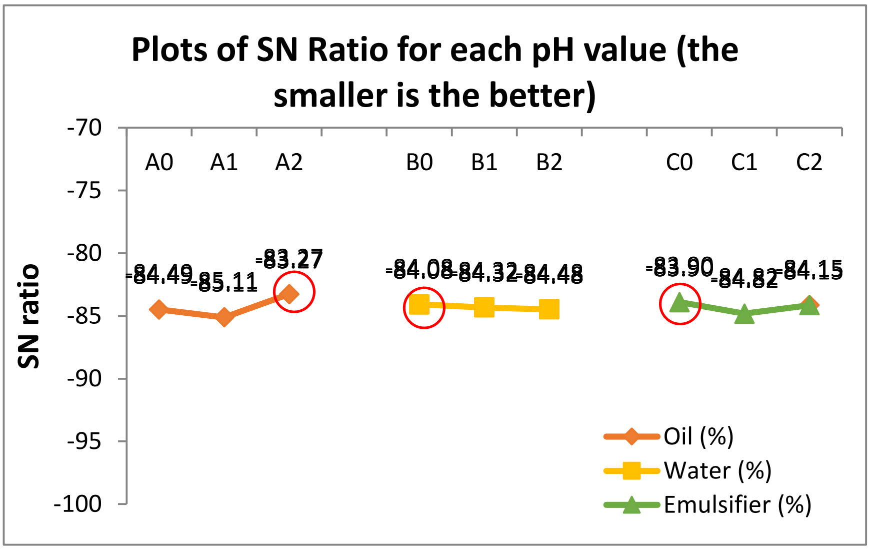


Figure 2: Plots of SN Ratio for each parameter in determining optimum condition of pH level (Nazaruddin, 2020)

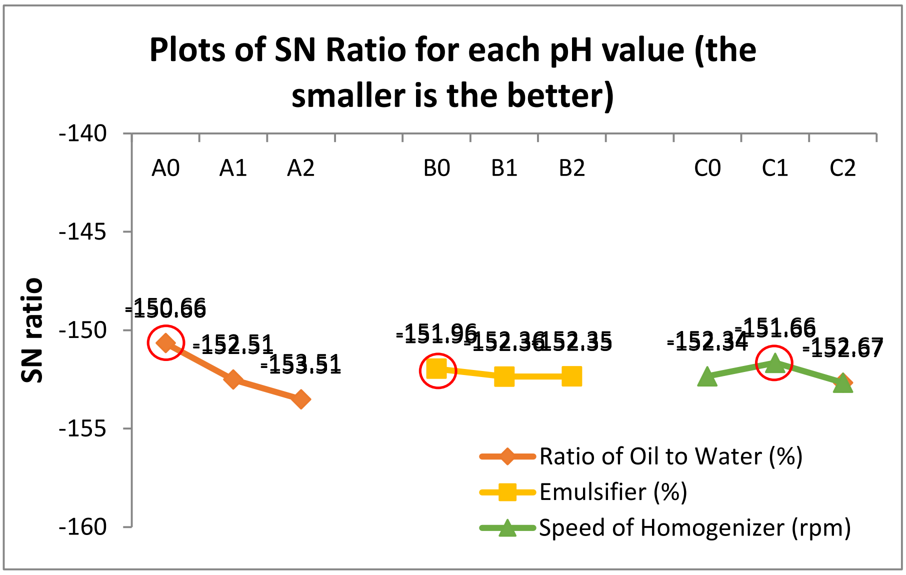


Figure 3: Plots of SN Ratio for each parameter in determining optimum condition of pH level (Jalal, 2019)

Based on relative stiffness, percentage of emulsifier, percentage of water and oil has the same effect on droplet size of base cream. It is obvious to observe that the optimum condition of droplet size for data from Nazarudin (2020) [4] is at A1 (percentage of oil at 10.00 %), B1 (percentage of distilled water at 90.00 %) and C2 (percentage of emulsifier at 5.00 %) as shown in Figure 4. The value of droplet size at these conditions is 2.459 µm as shown in Table 3. Thus, Taguchi method suggests that the formulation of base cream is recommended to run under these conditions in order to obtain small droplet size. The optimum condition for the data from Jalal (2019) [5] is at A0 (ratio of oil to water at 5:95), B2 (percentage of emulsifier at 5.00 %) and C2 (speed of homogenizer at 10000 rpm) as displayed in Figure 5. Therefore, the value of droplet size at these conditions is 6.3 µm as displayed in Table 4.

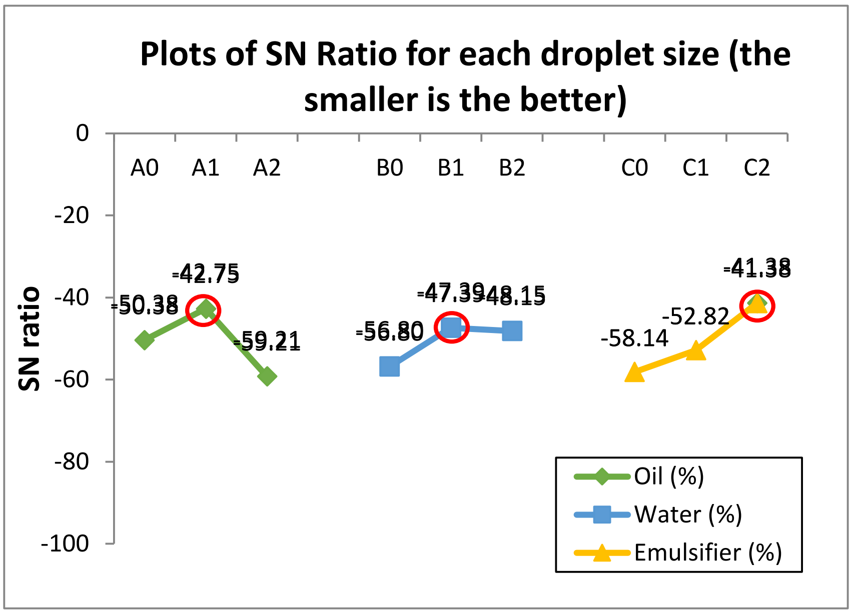


Figure 4: Plots of SN Ratio for each parameter in determining optimum condition of droplet size (Nazarudin, 2020)

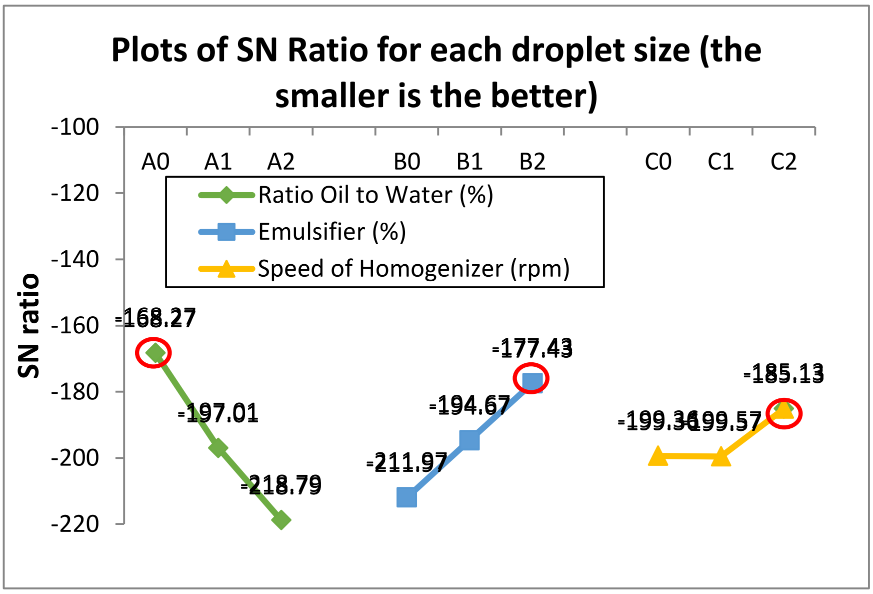
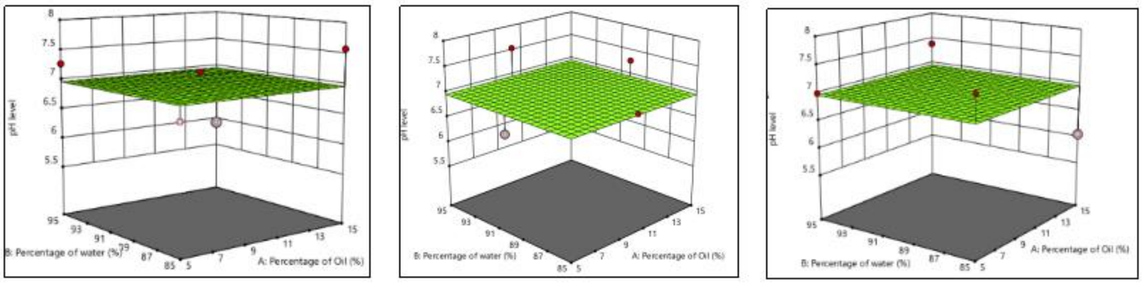


Figure 5: Plots of SN Ratio for each parameter in determining optimum condition of droplet size (Jalal, 2019)

3.2 Response surface method

3D surface plots above give a better view to see where the optimum condition may occur. Based on Figure 6 and Table 5, the percentage of emulsifier at 3.00 %, percentage of oil at 5.00 % and percentage of water at 90.00 % shown the pH level at 6.596 and the percentage of emulsifier at 5.00 %, percentage of oil at 15.00 % and percentage of water at 85.00 % shown the pH level at 6.046. 3D surface plots shows that the optimum of pH level occurs at percentage of emulsifier of 1.00 %, percentage of oil of 15.00 % and percentage of water of 95.00 %. This results is however contradict with the optimum condition analysed by Taguchi in terms of percentage of distilled water.



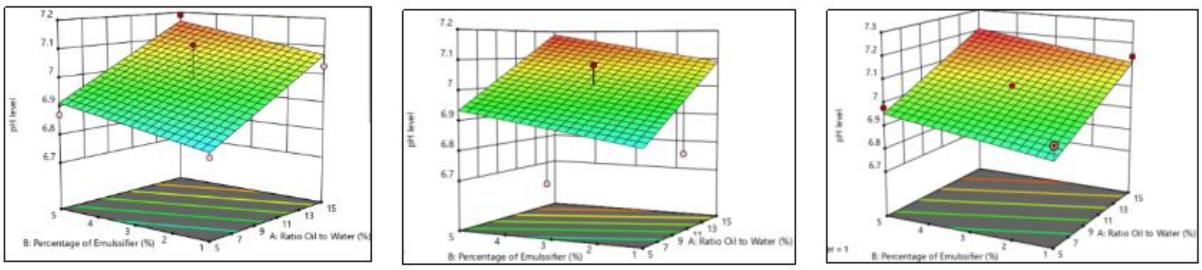
(a) (b) (c)

Figure 6: 3D surface plot pH level for (a) percentage of emulsifier at 1% (b) percentage of emulsifier at 3 % (c) percentage of emulsifier at 5 % (study done by Nazarudin, 2020)

Table 5: The optimum conditions for every percentage of emulsifier based on 3D surface plot and its pH level by Design Expert

Percentage of emulsifier (%)	1	3	5
Percentage of oil (%)	15	5	15
Percentage of water (%)	95	90	85
pH level	5.920	6.596	6.046

3D surface plots above give a better view to see where the optimum condition may occur. Based on Figure 7 and Table 6, speed of homogenizer at 5000 rpm, ratio of oil to water of 5:95 and percentage of emulsifier of 1.00 % shown the pH level of 6.8 and the speed of homogenizer at 10000 rpm, ratio of oil to water of 5:95 and percentage of emulsifier of 1.00 % shown the pH level of 6.92. 3D surface plots show that the optimum of pH level occurs at speed of homogenizer at 7500 rpm, ratio of oil to water of 5:95 and percentage of emulsifier of 3.00 %. These results however contradict with the optimum analysed by Taguchi which the optimum condition occurs at ratio of oil to water at 5:95, percentage of emulsifier at 1.00 % and speed of homogenizer at 5000 rpm.



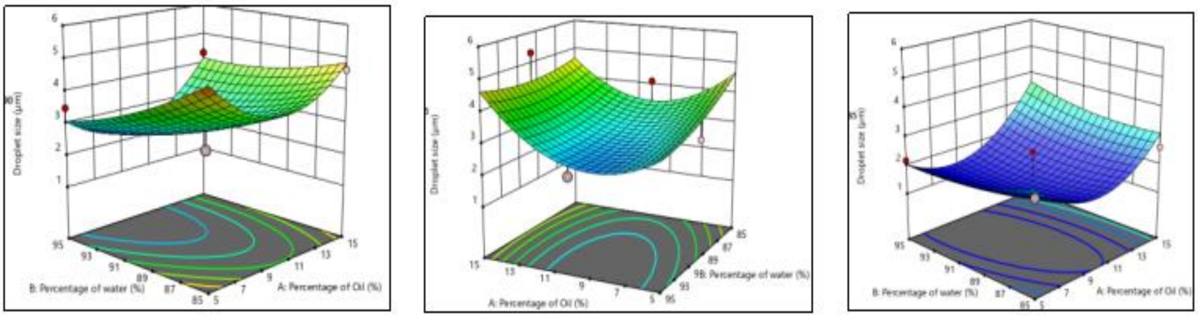
(a) (b) (c)

Figure 7: 3D surface plot of pH level for (a) speed of homogenizer at 5000 rpm (b) speed of homogenizer at 7500 rpm (c) speed of homogenizer at 10000 rpm (Jalal, 2019)

Table 6: The optimum conditions for every percentage of emulsifier based on 3D surface plot and its pH level by Design Expert

Speed of homogenizer (rpm)	5000	7500	10000
Ratio oil to water (%)	5:95	5:95	5:95
Percentage of emulsifier (%)	1	3	1
pH level	6.80	6.71	6.92

3D surface plots give a better view to see where the optimum condition may occur. Based on Figure 8 and Table 7, the percentage of emulsifier at 3.00 %, percentage of oil at 10.00 % and percentage of water at 95.00 % shown the droplet size of 2.378 μm and the percentage of emulsifier at 3.00 %, percentage of oil at 5.00 % and percentage of water at 85.00 % shown the droplet size of 2.582 μm. 3D surface plots shows that the optimum of droplet size occurs at percentage of emulsifier of 1.00 %, percentage of oil of 10.00 % and percentage of water of 90.00 %. This result is however contradicting with the optimum analysed by Taguchi in terms of percentage of emulsifier.



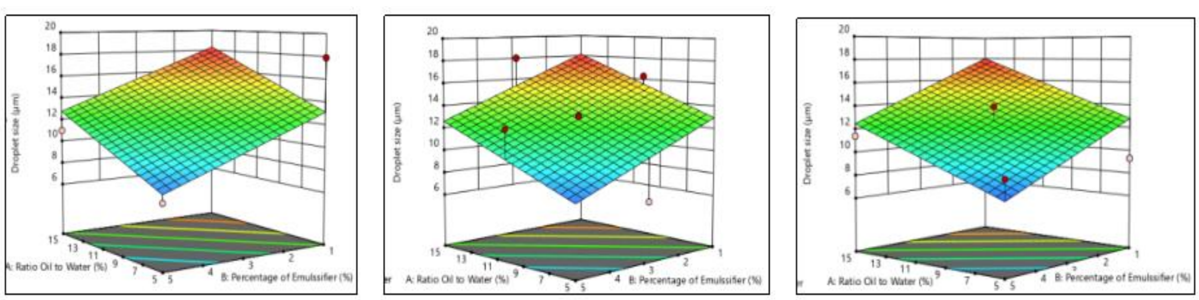
(a) (b) (c)

Figure 8: 3D surface plot of droplet size for (a) percentage of emulsifier at 1 % (b) percentage of emulsifier at 3 % (c) percentage of emulsifier at 5 % (Nazarudin, 2020)

Table 7: The optimum conditions for every percentage of emulsifier based on 3D surface plot and its droplet size by Design Expert

Percentage of emulsifier (%)	1	3	5
Percentage of oil (%)	10	10	5
Percentage of water (%)	90	95	85
Droplet size (μm)	2.129	2.378	2.582

3D surface plots above give a better view to see where the optimum condition may occur. Based on Figure 9 and Table 8, speed of homogenizer at 5000 rpm, ratio of oil to water of 5:95 and percentage of emulsifier of 5.00 % shown the droplet size of 6.8 μm and the speed of homogenizer at 10000 rpm, ratio of oil to water of 5:95 and percentage of emulsifier of 5.00 % shown the droplet size of 9 μm. 3D surface plots shows that the optimum of pH level occurs at speed of homogenizer at 7500 rpm, ratio of oil to water of 5:95 and percentage of emulsifier of 3.00 %. These results however contradict with the optimum analysed by Taguchi which the optimum condition occurs at ratio of oil to water at 5:95, percentage of emulsifier at 5.00 % and speed of homogenizer at 10000 rpm.



(a) (b) (c)

Figure 9: 3D surface plot of droplet size for (a) speed of homogenizer at 5000 rpm (b) speed of homogenizer at 7500 rpm (c) speed of homogenizer at 10000 rpm (Jalal, 2019)

Table 8: The optimum conditions for every percentage of emulsifier based on 3D surface plot and its droplet size by Design Expert

Speed of homogenizer (rpm)	5000	7500	10000
Ratio oil to water (%)	5:95	5:95	5:95
Percentage of emulsifier (%)	5	3	5
Droplet size (μm)	6.9	6.5	9.0

3.3 Discussions

Table 9 below shows the overall condition for both Taguchi and Response Surface Method (RSM) based on the analysis that had been done. It is shown that both methods do not show the same optimum condition for both responses which are pH level and droplet size. For the study done by Nazarudin (2020) [4], the optimum condition for pH level was different at percentage of water while for the droplet size, the optimum condition was different at the percentage of emulsifier for both Taguchi method and RSM. For both pH level and droplet size for the study done by Jalal (2019) [5], percentage of emulsifier and speed of homogenizer gave different optimum conditions for both Taguchi method and RSM. The outcome responses gave different optimum conditions due to the criteria constraints that had been determined in the model experiments design.

Table 9: Overall optimum conditions for Taguchi and Response Surface Methods

Study done by	Responses	Factors	Taguchi method	Response Surface method
Nazarudin (2020)	pH level	Percentage of oil (%)	15	15
		Percentage of water (%)	85	95
		Percentage of emulsifier (%)	1	1
	Droplet size	Percentage of oil (%)	10	10
		Percentage of water (%)	90	90
		Percentage of emulsifier (%)	5	1
Jalal (2019)	pH level	Ratio oil to water (%)	5:95	5:95
		Percentage of emulsifier (%)	1	3
		Speed of homogenizer (rpm)	5000	7500
	Droplet size	Ratio oil to water (%)	5:95	5:95
		Percentage of emulsifier (%)	5	3
		Speed of homogenizer (rpm)	10000	7500

4. Conclusion

The optimisation using Taguchi method for the study done by Nazarudin (2020) [4] found that the condition of 15.00 % of oil, 85.00 % of distilled water and 1.00 % of emulsifier are the optimum condition for a healthy pH level at 7.57 while for a small droplet size at 2.459 μm, the optimum condition is with 10.00 % oil, 90.00 % water and 5.00 % emulsifier. Besides, for the study done by Jalal (2019) [5], the optimum condition for a healthy pH at 6.87 was occur at 5:95 ratio of oil to water, 1.00 % of emulsifier and 5000 rpm whereas the optimum condition for a small droplet size occur at 5:95 ratio of oil to water, 5.00 % emulsifier and 10000 rpm with a size of 6.3 μm.

For the optimisation using RSM, the study done by Nazarudin (2020) [4] found that the condition of 15.00 % of oil, 95.00 % of distilled water and 1.00 % of emulsifier are the optimum condition for a healthy pH level at 5.922 while for a small droplet size at 2.129 μm, the optimum condition is with 10.00 % oil, 90.00 % water and 1.00 % emulsifier. Besides, for the study done by Jalal (2019) [5], the optimum condition for a healthy pH at 6.71 and small droplet size with a size of 6.5 μm. occurred at 5:95 ratio of oil to water, 3.00 % of emulsifier and 7500 rpm.

In order to verify the result of optimisation via Taguchi and RSM, a validation test should be done on optimised samples. This validation can be performed experimentally. Besides Taguchi and RSM, other optimisation tools can be used such as Minitab and ANOVA.

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