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# Investigation of the Base Oil Properties in Optimizing the **Demulsification**

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Abstract. In this research, the correlation of the physical properties from different types of base oil- Group I and Group II base oils - to the demulsification process with variables such as mixing speed (500-1500 rpm), water content (20-80%) and volume of formulated surfactant (1 - 5 ml) were investigated. The water-in-oil emulsion was prepared using a formulated surfactant, and the demulsification was observed via a Bottle Test method. The optimization of demulsification was obtained from the correlation of the respective variables using the Central Composite Design (CCD) for Response Surface Methodology (RSM). Results showed that the Group I base oil emulsions have a lower viscosity and higher density than the Group II base oil, which has higher emulsion stability. The higher demulsification was observed by reducing the mixing speed, increasing the water content, and reducing the volume of the formulated surfactant for both groups of the base oil. The optimum of the water separation w obtained from the Group I base oil was 46.73% at 515 rpm, 76% water content, and 1 ml volume of the formulated surfactant. Meanwhile, 99.29% of water separation was found for the Group II base oil at 520 rpm, with 75% water content and 2 ml volume of the formulated surfactant.

#### 1. Introduction

The demand for base oils as lubricants for machinery for transportation and automobile engines worldwide has constantly been growing over the years. Nowadays, lubricants are made up of various base oils and chemical additives, consisting of 90% base oil and 10% synthetic additives [1]. Lubricant base oils are manufactured through the refining of petroleum crude oil. The overall global demand for petroleum base oils was estimated to be 35 Mt in 1990, and it has been relatively steady since then [2]. The base oil composition contains saturated hydrocarbon molecules (n-paraffins, isoparaffins, and naphthenes), sulfur, and nitrogen. Products produced from the base oil with or without additives are based on specific applications. Base oil is derived from crude oil through a series of processes such as distillation, aromatic extraction, de-waxing, and finishing [2].

There are three types of base oil: mineral, synthetic, and bio-based. Mineral oil is made from petroleum refining, whereas synthetic oil is artificial. Natural oils such as sunflower oil and coconut oil are examples of bio-based base oil. The base oil is categorized into five groups (Group I, Group II, Group III, Group IV, and Group V) by the American Petroleum Institute (API) according to their sulfur, VI (Viscosity Index), and composition. Group I base oils are fractionally distilled petroleum that has been refined further using solvent extraction; Group II base oils are processed through hydroprocessing; Group III processed from isomerization process. Groups IV and V are such as polyalphaolefins (PAO)

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and the remaining base oils, respectively. Group I, II, and III are categorized under mineral, whereas Group IV and V are synthetic [6].

Emulsions are colloidal dispersions in which a liquid is distributed in a continuous liquid phase consisting of a different composition. The dispersed and continuous phases are akin to the internal and exterior phases, respectively. Emulsions are thermodynamically unstable systems that split over time, reducing the interfacial area between oil and water phases [7]. The emulsions are categorized into three types: primary, secondary, and multiphase emulsions [8]. In primary emulsions, only two phases are present in the emulsion: either Water-in-Oil (W/O) or Oil-in-Water (O/W). The phases in the emulsions differ depending on whether the liquid is in the dispersed or continuous medium. For secondary emulsions, there are three phases of liquid in the emulsion, which are either Water-in-Oil-in-Water (W/O/W) or Oil-in-Water-in-Oil (O/W/O). In this type of emulsion, there is only one continuous phase and two dispersed phases. Meanwhile, for the multiphase emulsions, there is an occurrence of having a double emulsion disperse in the inner droplet of the liquid, which is also known as W/O/W or O/W/O.

Numerous studies have been conducted on various factors for the demulsification of W/O emulsions. Abduhraman et al. [9] claimed that the agitation speed of the emulsion would influence the viscosity and droplet size. When high agitation speed is applied to the emulsion, the size of the droplets in the emulsion will decrease. The reduction of droplet size increases the viscosity, which increases the stability of the emulsion. This is due to hydrodynamic interactions' increased contribution due to increased particle surface area and decreased distance between droplets [10][11]. Due to this reason, the rate of flocculation and coalescence has slowed down. According to Kokal, Al-Ghamdi and Meeranpillai, [12], a reasonable degree of mixing is essential for emulsification as it increases the chances of the collision of water droplets. However, excessive agitation should be avoided as further emulsification may occur.

Al-Sabagh et al. [13] stated that water separation increased as the water content increased. This is because the repulsion of the interfacial layer in the W/O emulsion was dependent on the pressure in the dispersed and continuous phases; when the water content is high, the internal pressure of the water droplet will be higher than the external pressure of the oil droplet. Thus, it leads to the breakup of the interfacial layer and increases the coalescence rate due to the higher collision rate among the droplets. The higher coalescence rate is akin to the higher demulsification. The concentration of surfactant used affects demulsification as well. According to Al-Sabagh et al. [13] and Norela et al. [14], the highest demulsification efficiency was observed at a higher surfactant concentration. This is because more surfactant adsorbs at the interface between the oil membrane and the internal phases as surfactant concentration increases, increasing the strength and stability of the adsorption layer. The concentration of surfactant also affects the viscosity of emulsion [15]. As the surfactant concentration increases, the emulsion viscosity increases, and the emulsion becomes more stable.

The formation of emulsion causes many issues in the industry, such as restricted flow pressure, corrosion in pipelines, and failure of pumps. Therefore, an efficient demulsification is required to produce a higher quality of the base oil to satisfy the growing demand. Numerous studies have been conducted on the demulsification of crude oil emulsions to solve the problems [3][4]. However, there is limited research on the base oil compared to the crude oil emulsion.

Thus, a correlation between the base oil properties and demulsification was conducted by varying the parameters included mixing speed, water content, and volume of the formulated surfactant in preparing the W/O base oil emulsion using Groups I and II. The model equations in optimizing the demulsification also were proposed, and the experimental works were conducted using Response Surface Methodology (RSM). In this research, a formulated surfactant (PI 2019006769), a non-ionic surfactant, is used to separate the base oil emulsion. The formulated surfactant is produced using raw materials such as sunflower oil and polyethylene glycol (PEG). The conventional raw material used in producing surfactant is oleic acid instead of sunflower oil [5]. Therefore, the invention of this surfactant is economically competitive because of the usage of a new raw material instead of the conventional raw material. In fact, the surfactant also has an impact on reducing the cost of fuel by 30% and emissions by 60%.

#### 2. Methodology

#### 2.1. Materials

The base oils, Group I (SN500) and Group II (GS600N) were supplied by Lubetech Sdn Bhd, Klang, Selangor. The formulated surfactant (PI 2019006769) was invented and obtained by Abduhraman H. Nour, Universiti Malaysia Pahang.

#### 2.2. Physical characteristics of base oil

The viscosity of the base oil was tested using a viscometer (Brookfield DV-II+Pro, USA), and the spindle used was type 61. The spindle was immersed into 500 ml of the base oil, and the viscosity was obtained by measuring the torque on a vertical shaft that turns a spindle at room temperature, 28°C. The spindle was cleaned thoroughly before measuring the following base oil.

Density of the base oil was measured in a pycnometer (ASTM D1418). About 25 ml of the base oil was inserted in a pycnometer- a glass flask with a capillary hole and a close-fitting ground glass stopper. After the top-filled pycnometer was closed, the hole would release extra liquid, which allowed the given volume of the liquid to be obtained with high accuracy. First, the mass of the pycnometer was measured and recorded. The pycnometer was filled with distilled water and weighted. Any excessive liquid was wiped off with tissue paper before measuring the volume of the base oil. The volume of the pycnometer was measured using Equation 1. The procedure was repeated with another base oil. The density of the base oil was calculated using Equation 3. The detailed calculation and equations involved are shown as follow:-

Volume of pycnometer, V= 
$$\frac{\text{Mass of distilled water, m}_{\text{H}_2\text{O}}}{\text{Density of distilled water, }\rho_{\text{H}_2\text{O}}}$$
 (1)

$$V = \frac{\text{Mass of base oil, } m_L}{\text{Density of base oil, } \rho_L}$$
(2)

$$\rho_{\rm L} = \frac{m_{\rm L}}{m_{\rm H_20}} \rho_{\rm H_20} \tag{3}$$

#### 2.3. Preparation of emulsion

Emulsions were prepared by adding the respective volume of water, base oil, and formulated surfactant. In the preparation of the emulsion, the varied parameters were mixing speed (500 to 1500 rpm), water content (20-80%), and volume of formulated surfactant (1-5mL). The formulated surfactant was inserted into the base oil and stirred for 1 minute at 500 rpm. Water was gradually added into the solution and stirred for 5 minutes using a mechanical stirrer to ensure the solution was well-mixed.

#### 2.4. Demulsification

The demulsification process was determined through gravity settling or known as the Bottle Test. The prepared emulsion was poured into a measuring cylinder before the water separation was observed by the layer of water formed at the bottom of the measuring cylinder. The separation of water from the emulsion was calculated as shown in the following Equation.

Water separated (%) = 
$$\frac{\text{Volume of water, } V_w}{\text{Initial volume of water, } V_o} \times 100\%$$
 (4)

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#### 2.5. Optimization of demulsification

Central Composite Designs (CCD) of Response Surface Methodology (RSM) was chosen for the optimization of the demulsification parameters in this research. The analysis was done by using Design Expert 8.0.6 software (Stat-Ease, Inc., Minneapolis, USA). The investigated independent variables were listed and tabulated in Table 1. The selection of the variables' ranges was based on literature research. The analysis was applied using the second-order polynomial equation as shown in Equation 5 [16]. The analysis of variance (ANOVA) was performed to analyze the significance of the result.

Table 1. Experimental Independent Variables with Coded Levels for Central Composite Design.

Independent Variable	Coded Levels				
_	-1	0	1		
Mixing Speed (rpm)	500	1000	1500		
Water Content (%)	20	50	80		
Volume of Formulated Surfactant (ml)	1	3	5		

$$y = \beta_o + \sum_{i=1}^k \beta_i x_i^2 + \sum_{i=1}^k \beta_{ii} x_i^2 + \sum_{i=1}^{k-1} \sum_{j=2}^k \beta_{ij} x_i j_i + \varepsilon$$
(5)

Where y represents the response and  $\beta_o$ ,  $\beta_i$ ,  $\beta_{ij}$ ,  $\beta_{ij}$  represent the constant term, linear coefficient, quadratic influence, and cross-product coefficient, respectively.  $x_i$  represents the variable value, and  $\varepsilon$  represents the statistical error. The response, y, is the percentage of water separated, the x variable value, i.e., mixing speed, water content, and volume of formulated surfactant.

#### 3. Results and discussion

#### 3.1. Effect of physical properties of base oil

Table 2 shows the physical properties of base oil in Groups I and II. The physical properties of the base oil were identified to correlate with the demulsification process.

Table 2. Physical	properties	of Groups I	and II base oil.
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	Group I	Group II	
Viscosity (cP)	20.5	200.4	
Density (g/ml)	0.87913	0.86563	

From Table 2, Group I base oil has a lower viscosity than Group II. The density of Group I was slightly higher than Group II. Figure 1 shows the demulsification for both base oil from Group I and II at constant mixing speed, 1000 rpm, 50% water content, and 3 mL of the formulated surfactant. The figure displays that Group II has a higher percentage of water separated, about  $79.5 \pm 0.25\%$ , than Group I base oil, with  $38.0 \pm 0.45\%$ . In other words, the emulsions prepared from the Group I base oil was more stable than emulsions prepared from Group II base oil. This could be due to the lower viscosity of Group I base oil, which indicates that there were more interactions between droplets and surfactant molecules than in Group II oil, which has a higher viscosity. The formulated surfactant may be more readily adsorbed onto the surface of the oil droplet, resulting in a strong interfacial film in a low viscosity base oil emulsion [17]. As more formulated surfactant molecules are adsorbed onto the droplet's surface, the emulsion becomes more stable, resulting in less separation of water was observed.

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Figure 1. Percentage of water separated of the different type of base oil at constant mixing speed of 1000 rpm, water content of 50%, volume of formulated surfactant of 3 ml.

Another reason might be the different manufacturing processes between Groups I and II base oils. Group I base oils undergo solvent refining, which is a much simpler process than Group II. On the other hand, group II has a more complex hydroprocessing process [18][19][20][21]. This explains the higher density of Group I base oil as heavier molecules such as asphaltenes are present in the oil. Asphaltenes, resins, and paraffin wax that act as a natural emulsifier to emulsions are likely to present in Group I base oil [22] [23].

# 3.2. Effect of mixing speed

The demulsification efficiency has been investigated at different mixing speeds of 500, 1000, and 1500 rpm. Figure 2 shows the result of the separation of water with varying mixing speeds for Groups I and II at constant 50% water content and 3 mL volume of the formulated surfactant.



Figure 2. Effect of mixing speed in Groups I and II base oil on demulsification

The figure depicted that increasing the mixing speed results in a lower percentage of water separation. At 500 rpm, the maximum and minimum percentages of water separated for Group I base oil were  $43.0 \pm 0.29$  % and  $30.4 \pm 0.21$ %, respectively. Meanwhile, the water separated for Group II within the

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ranges of  $89.0 \pm 0.40\%$  and  $69.0 \pm 0.64\%$ , respectively. The water separation of emulsions was more significant when the mixing speed was increased, resulting in a more stable emulsion. Similar findings were obtained in studies conducted by Ashrafizadeh et al. [24] and Mohyaldinn et al. [25]. As increasing the agitation speed, the droplets in the emulsion become smaller indicated the increase of the interfacial area. Thus, the higher interaction between droplets and surfactant molecules leads to slower flocculation and coalescence.

#### 3.3. Effect of water content

The demulsification efficiency was investigated at different water contents of 20%, 50%, and 80%. Figure 3 shows water separation with varying water content for Groups I and II at a constant mixing speed of 1000 rpm and 3 mL volume of the formulated surfactant.



Figure 3. Effect of water content between Groups I and II base oil on demulsification

Based on Figure 3, the trend of percentage of water separated was observed to be increased with the water content for both Groups I and II. The highest and lowest water separation of Group I base oil was obtained at  $41.6 \pm 0.53\%$  and  $17.0 \pm 0.06\%$ , whereas for Group II was at  $86.0 \pm 0.12\%$  and  $35.0 \pm 0.17\%$ , respectively. A higher water content emulsion is less stable and separates more. According to Abdulredha et al. [26], increasing the dispersed phase volume improves separation efficiency and average coalescence by increasing the entropy for intense collision between single-phase droplets. When the dispersed phase (water) has a larger volume than the continuous phase (base oil), the coalescence process occurs faster due to an increase in the collision rate of the droplets. Research conducted by Othman et al. [27] showed the size of the droplet in the emulsion increases with increasing water content. In addition, the higher water content enhances the capability of water droplets to aggregate and eliminates the interfacial film that surrounds the droplets, leading the droplets to coalesce by creating a single and bigger unit of droplets. Furthermore, when the water content increases, the number of hydrogen bonds also increases, decreasing droplet distance.

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#### 3.4. Effect of volume of formulated surfactant

Figure 4 shows the result of the separation of water with a varying volume of formulated surfactant from 1 to 5 mL for Groups I and II at a constant mixing speed of 1000 rpm and 50% water content.



**Figure 4.** Effect of volume of formulated surfactant between Group I and II base oil at a constant mixing speed of 1000 rpm and 50% water content.

From Figure 4, the percentage of water separated from emulsion decreased by increasing the volume of the formulated surfactant. The highest water separation obtained from Groups I and II were  $43.6 \pm 0.38\%$  and  $90 \pm 0.20\%$ , respectively. Meanwhile, the minimum percentages of water separated were  $33 \pm 0.12\%$  for Group I base oil and  $65 \pm 0.32\%$  for Group II base oil. The presence of an emulsifier with hydrophilic (oil soluble) and lipophilic (water-soluble) properties causes the dispersion of water molecules into the continuous oil phase [27]. When the concentration of surfactant increases, the formation of larger droplets during coalescence is inhibited, resulting in less water separation. Similar findings were supported by Sohn et al. [28] and Qian et al. [29] in investigating the effect of droplet size as increasing the concentration of the emulsifier (surfactant). They discovered that the concentration of the surfactant significantly influences particle size. They stated that more surfactant was present to surround the droplets formed during high-pressure homogenization.

#### 3.5. Optimization of Group I base oil

Table 3 shows the model has an F-value of 74.70 and *p*-value of 0.0001, indicating a high significance of the model. The variables A, C, AC, BC, and B<sup>2</sup> are significant as *p*-value is less than 0.05. Other than these variables, they are considered insignificant. Furthermore, raising the F-value of factors increases their effect on the response [30]. For example, water content with an F-value of 473.58 had the most significance on demulsification, whereas the volume of formulated surfactant with a value of 18.02 would have the least significance. Other than that, the model's goodness-of-fit was also assessed using coefficients of determination R<sup>2</sup> (correlation efficiency) and R<sup>2</sup><sub>adj</sub> (adjusted coefficients of determination). The R<sup>2</sup> value of 0.9853 demonstrated that it was highly reliable in predicting the percentage of water separation, with the model explaining 98.53% of the response variability. As the R<sup>2</sup> value is very close to 1, it demonstrates a high degree of agreement between actual and anticipated results [31].

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Source	Sum of Squares	DF	Mean Square	F-value	Probability>F	
Model	3050.87	9	338.99	74.70	< 0.0001	significant
А	217.16	1	217.16	47.85	< 0.0001	-
В	2149.16	1	2149.16	473.58	< 0.0001	
С	81.80	1	81.80	18.02	0.0017	
AB	12.50	1	12.50	2.75	0.1280	
AC	24.50	1	24.50	5.40	0.0425	
BC	60.50	1	60.50	13.33	0.0045	
$A^2$	7.08	1	7.08	1.56	0.2401	
$\mathbf{B}^2$	222.98	1	222.98	49.13	< 0.0001	
$C^2$	5.682*10-5	1	5.682*10-5	1.252*10-5	0.9972	
Residual	45.38	10	4.54			
Lack of Fit	37.17	5	7.43	4.53	0.0615	not
						significant
Pure Error	8.21	5	1.64			
Total	3096.25	19				
	$R^2 = 0.9853$			Pred $R^2 = 0$	0.9022	
	Adj $R^2 = 0.9722$			Adeq Preci	ision = 31.626	

Table 3. ANOVA for Quadratic Model for Group I base oil.

The experimental results (percentage of water separated) were correlated using the second-order polynomial shown in Equation 6.

 $R1=37.95-4.664+14.66B-2.86C-1.25AB-1.75AC+2.75BC-1.60A^2 - 9.00B^2 - 4.545 \times 10^3 C^2$  (6)

where A, B and C are the coded values of the mixing speed, water content, and volume of formulated surfactant, respectively.

The plot of predicted against the actual percentage of water separated results in Figure 5 showed similar experimental and anticipated results.



**Figure 5.** Predicted versus actual values of the percentage of water separated for Group I base oil.



**Figure 6.** RSM 3D plot of the percentage of water separated for Group I on the influence of mixing speed and water content at volume of formulated surfactant of 3 mL.

**Figure 7.** RSM 3D plot of the percentage of water separated for Group I on the influence of mixing speed and volume of formulated surfactant at water content of 50%.



**Figure 8.** RSM 3D plot of percentage of water separated for Group I on the influence of water content and volume of formulated surfactant at a mixing speed of 1000 rpm.

Figure 6 showed the percentage of water separated increased as decreasing and increasing the mixing speed and water content, respectively. From the figure, a higher water separation could be observed between 62% to 80% of the water content and 500 to 750 rpm. Meanwhile, Figure 7 depicted a correlation between the mixing speed and volume of formulated surfactant at a constant water content. The higher water separation could be observed as decreasing both mixing speed and volume of the formulated surfactant. The number of surfactants adsorbed onto the droplets in the dispersed phase was lower as reducing the volume of formulated surfactant into the solution. Thus, it also reduced the stability of the emulsion because a fragile interfacial film forms around the droplet. In Figure 8, as the water content was greater than 68%, the demulsification was highly efficient along with the surfactant than the surfactant volume.

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## 3.6. Optimization of Group II base Oil

Table 4 shows that the model has an F-value of 79.51 and a p-value of 0.0001, indicating a highly significant model. Values less than 0.05 indicated the model terms were significant. Variables A, B, C, AB, AC, BC, and B<sup>2</sup> are considered significant, whereas  $A^2$  and  $C^2$  are insignificant. Similarly, water content with an F-value of 465.92 has the most significant response, while the volume of formulated surfactant with a value of 27.19 has the least. The R<sup>2</sup> value for this model is 0.9862, indicating that the model's prediction of experiment outcomes is correct. Figure 9 depicts the predicted against the actual percentage of water separated. Equation 7 describes the model equation for Group II base oil-water separation.

 $R2 = 10.38 - 3.25A + 14.36B - 3.02C + 0.16AB + 0.31AC - 3.26BC + 2.24A^{2} + 6.29B^{2} + 1.19C^{2}$ (7)

Source	Sum of Squares	DF	Mean Square	F-value	Probability>F	
Model	12912.99	9	1434.78	79.51	< 0.0001	significant
А	1080.77	1	1080.77	59.89	< 0.0001	·
В	8407.68	1	8407.68	465.92	< 0.0001	
С	490.56	1	490.56	27.19	0.0004	
AB	77.88	1	77.88	4.32	0.0645	
AC	104.84	1	104.84	5.81	0.0367	
BC	144.16	1	144.16	7.99	0.0180	
$A^2$	5.94	1	5.94	0.33	0.5787	
$\mathbf{B}^2$	1096.70	1	1096.70	60.78	< 0.0001	
$C^2$	24.26	1	24.26	1.34	0.2732	
Residual	180.45	10	18.05			
Lack of Fit	137.90	5	27.58	3.24	0.1114	not significant
Pure Error	42.55	5	8.51			-
Total	13093.44	19				
	$R^2 = 0.9862$			Pred $R^2 =$	0.9227	
	Adj $R^2 = 0.9738$			Adeg Prec	sision = 31.465	

Table 4. ANOVA for Quadratic Model for Group II base oil.



Figure 9. Predicted versus Actual values of the percentage of water separated for Group II base oil.

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Figure 10 has a similar pattern as Group I base oil emulsions. The water separation was increased by decreasing the mixing speed from 1500 to 500 rpm and increasing the water content from 20% to 80%. A similar finding was observed by varying the volume of formulated surfactant, in which the maximum separation was obtained as reducing the volume, as shown in Figures 11 and 12.



**Figure 10.** RSM 3D plot of the percentage of water separated for Group II on the influence of mixing speed and water content at volume of formulated surfactant of 3 mL.

**Figure 11.** RSM 3D plot of the percentage of water separated for Group II on the influence of mixing speed and volume of formulated surfactant at water content of 50%.



**Figure 12.** RSM 3D plot of percentage of water separated for Group II on the influence of water content and volume of formulated surfactant at mixing speed of 1000 rpm.

#### 3.7. Prediction of optimum condition of percentage of water separation

The desired goal was set to within the range, and the response was set to the maximum. At 515 rpm, 76 percent water content, and 1 mL volume of formulated surfactant, the optimum condition for the percentage of separated water was 46.73% for Group I. At 520 rpm, 75 % water content, and 2 mL volume of formulated surfactant, Group II achieved a 99.29 % of the water separation. Table 5 shows the results of a subsequent experiment with the optimal values to validate the model's ability to predict the highest percentage of water separation. The results showed a good agreement between predicted and experimental findings at optimal values, with the error percentage being less than 10%, indicating that the model has high validity.

Type of		Parame	ters	Percentage of	water separated (%)	Error
Base Oil	Mixing	Water	Volume of	Experimental	Predicted	(%)
	Speed	Content	formulated			
	(rpm)	(%)	surfactant (ml)			
Group I	515	76	1	46.5	46.73	0.49
Groun II	520	75	2	95 3	99 29	4 02

Table 5. The comparison of predicted and experimental data for the optimum condition of the percentage of water separated.

# 4. Conclusion

The physical properties between Groups I and II revealed a difference in emulsion stability, which affects demulsification efficiency. The emulsions produced from the two base oil samples had distinct model equations that correlated the percentage of water separated to the emulsion preparation variablessuch as mixing speed, water volume, and surfactant volume. The predicted optimum water separation (demulsification) for Group I was 46.73% at 515 rpm, 76.0% water content, and 1 mL volume of formulated surfactant. Meanwhile, Group II, which has a higher viscosity than Group I, showed the highest water separation, 99.29% at 520 rpm, the water content of 75%, and volume of formulated surfactant of 2 ml. In conclusion, the higher viscosity of the base oil has lower stability of the emulsion and higher demulsification due to the small dispersed water droplet onto the base oil.

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