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THE EFFECT OF TEMPERATURE AND STIRRING SPEED ON THE PHYSICOCHEMICAL PROPERTIES OF OIL SPILL DISPERSANT FROM SOYBEAN OIL

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ABSTRACT

Oil spills are a form of marine pollution resulting from operations or accidents. Oil spill dispersant (OSD) can deal with oil spills. This research examines the effect of temperature and stirring speed on the OSD formulation using methyl ester sulfonate (MES) and natural soybean oil surfactant. OSD has nine variations of stirring speed (400 - 600 rpm) and temperature (40 - 60 ^{o}C). Oil spill dispersant characterization was done by testing density, pH, viscosity, and surface tension. The resulting OSD product has a density of 1.0022 g/mL - 1.0179 g/mL, a pH of 5.3-5.9, a surface tension of 29.465 dyne/cm-44.888 dyne/cm, and a viscosity of 1.261-2.747 cP. These characteristics meet wastewater quality standards for petroleum industry businesses and activities.

Keywords: oil spill dispersant, soybean oil, methyl ester sulfonate, temperature, stirring speed.

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INTRODUCTION

Oil spills are a form of marine pollution resulting from operations or accidents. Oil spills due to operations can occur due to periodic activities at an activity or installation, which only occur in small amounts frequently, such as waste disposal from tank washing, drains, and spills when unloading cargo. Meanwhile, spills resulting from accidents are unexpected and can cause significant environmental losses and loss of life [1].

Petroleum spills on the sea surface can cause pollution to the ecosystem, such as the death of marine biota, sea animals, and the like, as well as sea birds. This needs to be addressed to prevent environmental impacts from oil spills. The increasing need for sustainable technologies due to increasing interest in environmental protection and strict environmental control has led to the adoption of alternatives to surfactants derived from petroleum sources [2].

Petroleum-based surfactants are reported to have environmental problems such as low biodegradability, high toxicity, low lightness, and eutrophication, and are harmful to various aquatic microorganisms. Thus, there is a need to increase the conservation of petroleum resources and accelerate the development of new, environmentally friendly surfactants produced from biomass that have excellent biodegradability, ecological suitability, low toxicity, softness, and high interfacial activity [3].

Soybean oil is the most significant agricultural source of lecithin, where it is present at levels of approximately 1-3%. Lecithin is the most valuable byproduct of the soybean oil production industry due to its multiple uses in food and industrial goods. The global lecithin market is estimated to be around 130,000 metric tons annually. Lecithin functions as a dispersal agent. It is also used for adhesion, as an antioxidant, biodegrading, and dispersing agent, and as an emulsifier, stabilizer, viscosity modifier, and penetration agent. The unique characteristic of lecithin is its dual lipophilic and hydrophilic parts, resulting in its ability to attract water and fat, a characteristic that results in its good ability to be used as an intermediary between water and oil [4, 5]. Molecular structure of phospholipid-based surfactants is shown in Figure-1.

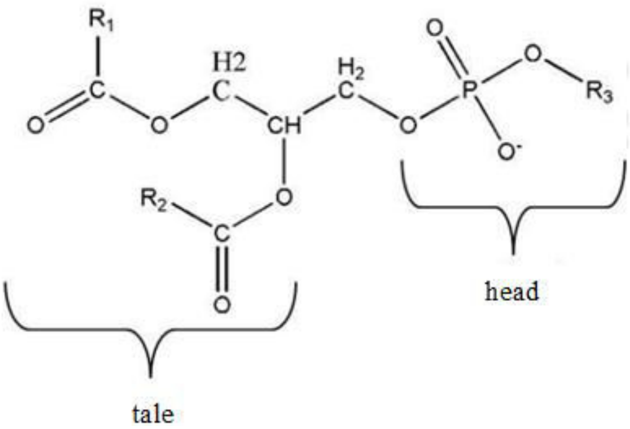

Figure-1. Molecular structure of phospholipid-based surfactants [6].

Methyl esters sulfonate have (MES) attractive properties for a variety of wetting applications and excellent detergent power with the low viscosity of their aqueous solutions; suitable for cleaning formulations, such as phosphate-free detergent powder; excellent water hardness stability allows them to be used in complex water areas; excellent ability to dissolve calcium soap scum; excellent skin compatibility which makes them potentially

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excellent for hand dishwashing formulations and body care products; viscous formulation, in mixture with nanoparticles. Recent studies on the rheological behavior of mixtures of MES and betaine solutions and the deposition of oil droplets on solid surfaces can be used in a broader range of applications [6].

Surfactants are amphiphilic compounds that have hydrophilic groups (like water) and hydrophobic groups (do not like water) so that surfactants can bind with water and oil at the same time. Based on the synthesis method, surfactants can be classified into cationic, anionic, zwitterionic, and non-ionic [8]. Surfactants can function as solvents for hydrophobic molecules, reduce surface tension, are suitable emulsifiers, and can be implemented in the pharmaceutical, cosmetic, paint, nanotechnology, petroleum recovery, bioremediation, chemical transformation, and other industries [9, 10]. When the process reduces the tension at the oil and water interface, an absorption process will occur at the surface area of the liquid-liquid interface. The decrease in interfacial tension causes a layer to form in the liquid phase area and the emergence of dispersed water droplets in the oil phase area [11].

Phospholipids are a type of surfactant with a lipid subclass consisting of a glycerol core with positions 1 and 2 connected to two fatty acyl groups via ester bonds. The third glycerol position is connected to a phosphate group. The phosphate group bears two ester bonds with a glycerol backbone and a polar head group, such as choline, ethanolamine, inositol, serine, and glycerol. The polar head groups correspond to the phospholipid subspecies: phosphatidylcholine (PC), phosphatidylethanolamine (PE), phosphatidylinositol (PI), phosphatidylserine (PS), and phosphatidylglycerol (PG). Structurally, all phospholipids are amphiphilic molecules, serving as the molecular basis for their biological functions and as emulsifiers in food products, cosmetics, or pharmaceuticals. In general, phospholipids are excellent surface-active amphiphilic molecules widely used as emulsifiers, wetting agents, solvents, and the main components of liposomes [12].

In the development of bio-based surfactant production, the primary natural resources used for the synthesis of this type of surfactant are fatty acids [13], amino acids [14], fatty acid methyl esters [15], carbohydrates, and triacylglycerols of vegetable oils (coconut oil, peanut oil soil, soybean oil, and corn oil) [16, 17]. From the description presented, it is necessary to observe the effect of temperature and stirring speed on the synthesis of oil spill dispersants based on soybean oil and methyl ester sulfonate in the diethylene glycol ethyl ether (dEGEE) solvent system.

Interfacial Tension Interfacial Surface Tension (IFT)

In general, the interfacial surface tension of surfactant mixtures can be attributed to the electrostatic interactions between ionic and polar head groups, including ion-dipole interactions (between non-ionic and ionic hydrophilic groups), steric interactions (between bulk groups and hydrophobic groups, respectively), as well as hydrogen bonds (between surfactant molecules) [18]. Surfactant molecules are the main chemicals needed to reduce Interfacial Surface Tension (IFT) values because their role as surface active agents can change surface or interface behavior to increase crude oil mobility. The mechanism for reducing Interfacial Surface Tension (IFT) is carried out by analyzing surfactant adsorption at the interface, interface thickness, interfacial energy, and the formation of hydrogen bonds between the surfactant head group and water molecules [19].

Hydrophilic-Lipophilic Equilibrium (HLB)

Hydrophilic-lipophilic balance (HLB) measures the degree of hydrophilicity or lipophilicity of a surfactant. It is related to the balance between the hydrophilic and lipophilic parts of the molecule. HLB values assigned to surfactants according to their chemical structure are often used to determine their potential use as emulsifiers and predict their emulsifying properties [20]. The HLB value is the molecule's polar to nonpolar site ratio. If it is lower than the polar value, it indicates a higher HLB value and produces an oil-in-water emulsion. In contrast, if it is higher than the nonpolar value, indicating a lower HLB value, it will usually produce a water emulsion in oil.

Hydrophile-lipophile balance (HLB) is an efficient way to select suitable surfactants for a particular application. HL) is categorized on a scale of 0-20, with 0 representing completely lipophilic molecules and 20 accounting for strongly hydrophilic molecules. HLB value of 3.5-6 corresponds to W/O emulsifier, and HLB value of 8-18 corresponds to O/W emulsifier. The HLB value of a wetting agent is in the range of 7-9. Hydrophilic surfactants (soluble in water) have an HLB value of> 10, while lipophilic surfactants have an HLB value of less than 10 (soluble in oil) [21].

MATERIALS AND METHODS

Materials

The ingredient used is soybean oil from Barco. N-hexane (C_6H_{14}), acetone (C_3H_6O), methyl ester sulfonate (MES), and diethylene glycol ethyl ether ($C_6H_{14}O_3$) from E Merck. Analytical materials in the form of phenolphthalein, isopropanol (C_3H_7OH), Potassium Hydroxide (KOH), neutral alcohol (C_2H_5OH), and hydrogen chloride (HCL) were also obtained from E Merck.

Synthesis of Lecithin

The synthesis of lecithin from soybean oil was obtained and modified from research conducted by Estiasih *et al*. [23, 24]. A total of 250 g of soybean oil was filtered and put into a glass beaker. Add 3% (v/v) distilled water from soybean oil to the glass beaker and homogenize. Please place it in a three-neck flask, then heat it at 70 °C and a stirring speed of 200 rpm for one hour. The mixture was centrifuged at 300 rpm for 30 minutes.

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Then, the lecithin sludge formed from the liquid phase was separated by filtering. The lecithin sludge was dried in a vacuum oven at 25 °C for one hour and then in a desiccator for one hour.

Lecithin Purification

The lecithin purification procedure to produce deoiled lecithin was adopted and modified from research conducted by Li and Guo [12]. At a substrate: solvent (w/v) ratio of 1 5, the crude lecithin was dissolved in hexane, allowed to stand for 30 minutes, and centrifuged at 4500 rpm for 10 minutes. The separated residue is separated by filtration. Hexane was separated using a rotary evaporator at a temperature of 70 °C. Acetone was added with a ratio (w/v) of 1: 1.5. Followed by centrifugation at 3000 rpm for 15 minutes. The deoiled lecithin formed was separated by filtration and dried in an oven at 70°C.

Oil Spill Dispersant Formulation

The oil spill dispersant synthesis procedure was adopted and modified from research [25]. Deoiled lecithin at a concentration of 4% was dissolved with diethylene glycol ethyl ether (dEGEE). Methyl ester sulfonate (MES) at a concentration of 8%. Dissolved with distilled water. The lecithin and MES solutions were mixed with a lecithin: MES ratio of 1 1. Placed in a three-neck flask, and stirred with varying stirring speeds of 400, 500, and 600 rpm. Heated with varying mixing temperatures at 40°C, 50°C, and 60°C. Mixing time was 1 hour. Surface tension, pH, viscosity, and density tests were carried out.

RESULTS AND DISCUSSIONS

The results and formulations of the nine variations carried out can be seen in Table-1. The preliminary formulation of the Oil Spill Dispersant was made by mixing the constituent ingredients until homogeneous and observing the effect of temperature and stirring speed on the ongoing process. The physicochemical properties in this research include the density, pH, viscosity, and surface tension of oil spill dispersants.

The Effect of Variables on pH

The pH value was obtained by measuring the oil spill dispersant for all formulations using a pH meter at 29^{o}C. The effect of variations in temperature and stirring speed on the pH of the oil spill dispersant can be seen in Figure-2. In formulation 1, with a temperature of 40^{o}C and a stirring speed of 400 rpm, the resulting pH was 5.5. In formulation 2, with a temperature of 40^{o}C and a stirring speed of 500 rpm, the resulting pH was 5.6. In formulation 3, with a temperature of 40^{o}C and a stirring speed of 600 rpm, the resulting pH was 5.6. In formulation 4, with a temperature of 50^{o}C and a stirring speed of 400 rpm, the resulting pH was 5.5. In formulation 5, with a temperature of 50^{o}C and a stirring speed of 500 rpm, the resulting pH was 5.7. In formulation 6, with a temperature of 50^{o}C and a stirring speed of 500 rpm, the resulting pH was 5.5. In formulation 7, with a temperature of 60^{o}C and a stirring speed of 400 rpm, the resulting pH was 5.5. In formulation 8, with a temperature of 60^{o}C and a stirring speed of 500 rpm, the resulting pH was 5.7. In formulation 9, with a temperature of 60^{o}C and a stirring speed of 600 rpm, the resulting pH was 5.7.

Table-1. Variations in oil spill dispersant formulations.

Run	Substrate Ratio		Lecithin Concentration		MES Concentration		Temp. (^{o}C)	Stirring speed (rpm)
	Lecithin (mL)	MES (mL)	Lecithin (Gr)	dEGEE (mL)	MES (Gr)	Water (mL)		
F1	40.0	40.0	1.6	40.0	3.2	40.0	40	400
F2	40.0	40.0	1.6	40.0	3.2	40.0	40	500
F3	40.0	40.0	1.6	40.0	3.2	40.0	40	600
F4	40.0	40.0	1.6	40.0	3.2	40.0	50	400
F5	40.0	40.0	1.6	40.0	3.2	40.0	50	500
F6	40.0	40.0	1.6	40.0	3.2	40.0	50	600
F7	40.0	40.0	1.6	40.0	3.2	40.0	60	400
F8	40.0	40.0	1.6	40.0	3.2	40.0	60	500
F9	40.0	40.0	1.6	40.0	3.2	40.0	60	600

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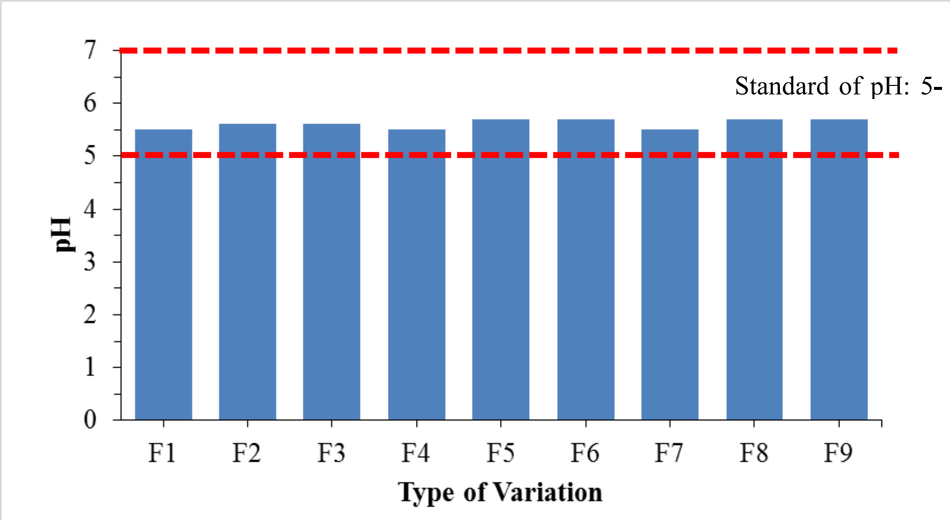

Figure-2. Effect of temperature variations and stirring speed on the pH of oil spill dispersant.

For ionic and non-ionic surfactants, increasing the temperature and stirring speed will affect the adsorption level of the surfactant so that the pH of the oil spill dispersant, which is a mixture of the two surfactants, will increase [26, 27]. Based on the results of research that has been carried out, the results of research regarding variations in temperature and stirring speed are directly proportional to the increase in pH of the oil spill dispersant. According to the Ministry of Industry standards regarding oil spill dispersants, the pH of oil spill dispersant products is 5-7. The resulting product has a pH of 5.5 - 5.7. Thus, the entire product produced is by the standards set by the Ministry of Industry regarding oil spill dispersants.

The Effect of Variables on Density

The density value was obtained by measuring the oil spill dispersant for all formulations using a pycnometer and calibrating the pycnometer volume using distilled water at 29°C. The results of measuring the density of each formulation are shown in Figure-3. The effect of variations in temperature and stirring speed on the density of the oil spill dispersant can be seen in Figure-3.

The density of oil spill dispersant can decrease with the increase in operating temperature and stirring speed due to the level of homogeneity of the mixture with increasing stirring speed and operating temperature [28, 29]. If the stirring speed increases, more particles are contained in the oil spill dispersant. They touch each other and react with each other, resulting in a decrease in density; this is also influenced by the presence of molecular interactions in the oil spill dispersant, which are more optimal and play an important role in increasing or decreasing the density of the oil spill dispersant [30]. The decrease in the density value of the oil spill dispersant, which is influenced by variations in temperature and stirring speed, which makes the density value close to the density value of water, shows that there are several advantages obtained, such as being easier to stabilize the interfacial tension of water, and forming an emulsion. It will be more efficient if applied to increase the thickness layer at the oil-water interface [31]. According to the Ministry of Industry standards regarding oil spill dispersants, the density of oil spill dispersant products is 0.8-1.2 g/mL. The resulting product has a 1.01615 g/mL density - 1.03064 g/mL. Thus, the entire product produced is by the standards set by the Ministry of Industry regarding oil spill dispersants. The results obtained were that the best density value in the preliminary oil spill dispersant was formulation 2 (F2) with an operating temperature of 40°C and a stirring speed of 500 rpm.

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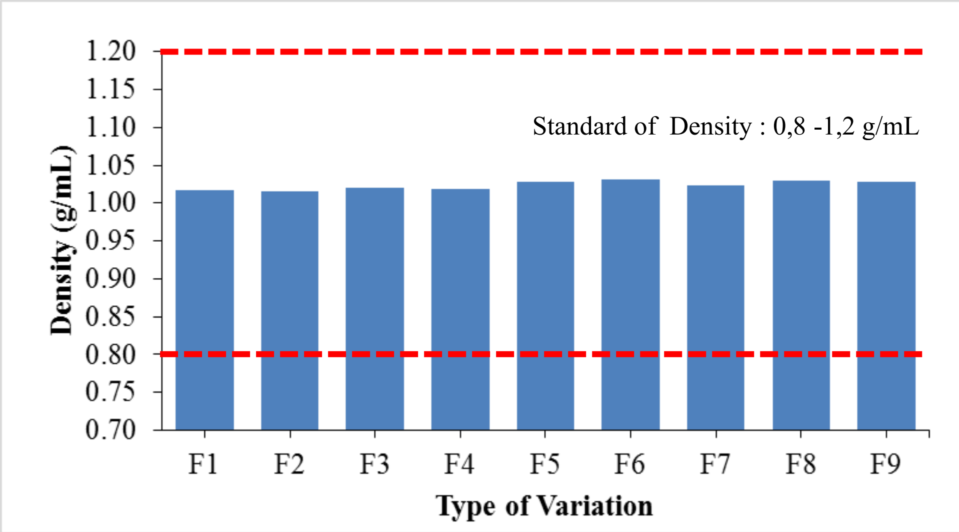

Figure-3. Effect of temperature variations and stirring speed on oil spill dispersant density.

The Effect of Variables on Viscosity

The viscosity value was obtained by measuring the oil spill dispersant for all formulations using an Ostwald viscometer. The results of measuring the viscosity of each formulation are shown in Figure-4. An increase in operating temperature will increase the viscosity of the oil spill dispersant introduction; this is due to the increase in temperature when mixing soy lecithin surfactant with MES surfactant, causing a reduction in the water content contained in the oil spill dispersant due to the use of distilled water as an MES surfactant solvent (methyl ester sulfonate). In addition, with variations in increasing stirring, the stirring speed will be inversely proportional to the particle size, resulting in minor changes in the surfactant mixture's particle size that result in a more stable emulsion system. The more stable an emulsion is, the better the viscosity value will be [32]. According to IPIECA (International Petroleum Industry Environmental and Social Issues) standards regarding oil spill dispersants, the viscosity of oil spill dispersant products is a maximum of 250 cP. The resulting product has a viscosity of 2.051 cP - 2.451 cP. Thus, the entire product produced is by the standards set by IPIECA (International Petroleum Industry Environmental and Social Issues) regarding oil spill dispersants. Based on the results of research conducted on the introduction of oil spill dispersants, it was found that the results obtained were still fluctuating. In some formulations, they were by theory, such as in formulation 2 with a temperature of 40°C and a stirring speed of 500 rpm, formulation 5 with a temperature of 50°C and a stirring speed of 500 rpm, and formulation 9 with a temperature of 60°C and a stirring speed of 600 rpm.

The Effect of Variables on Surface Tension

Furthermore, the surface tension value was obtained by measuring the oil spill dispersant for all formulations using a du Nouy tensiometer.

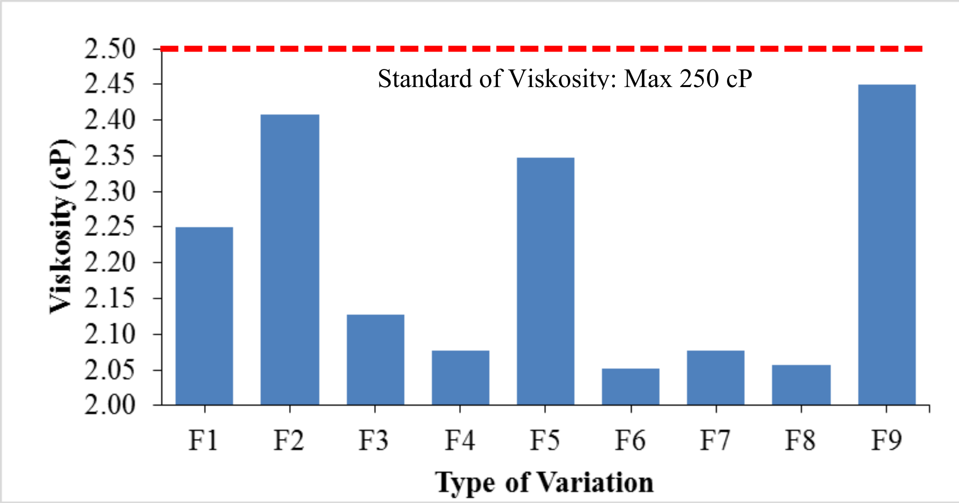

Figure-4. Effect of temperature variations and stirring speed on oil spill dispersant viscosity.

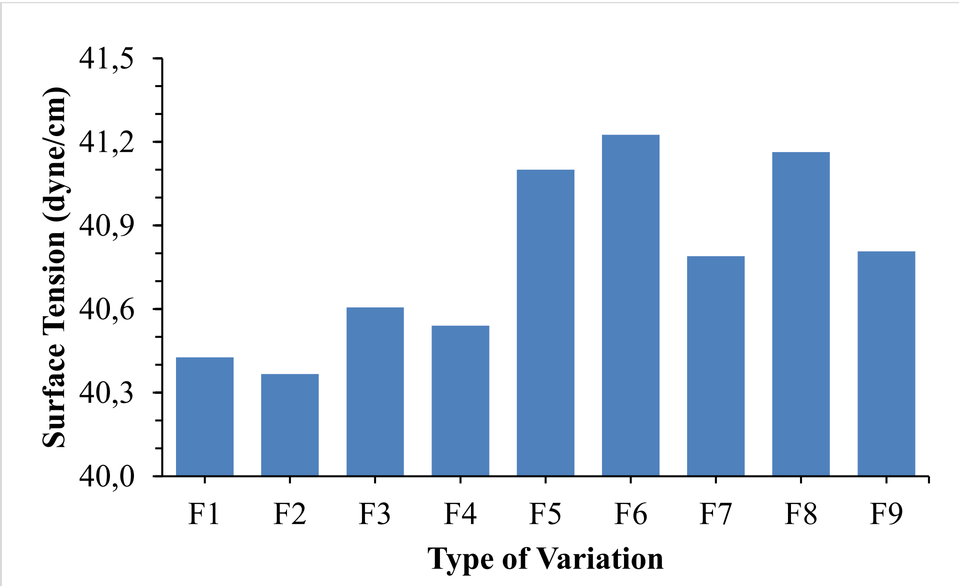

Figure-5. Effect of temperature variations and stirring speed on the surface tension of oil spill dispersant.

The effect of variations in temperature and stirring speed on the surface tension of the oil spill dispersant can be seen in Figure-5. In formulation 1, with a temperature of 40ºC and a stirring speed of 400 rpm, the resulting surface tension of 40.427 dynes/cm. In formulation 2, with a temperature of 40ºC and a stirring speed of 500 rpm, the resulting surface tension was 40.367 dyne/cm. In formulation 3 with a temperature of 40ºC and a stirring speed of 600 rpm, the resulting surface tension was 40.606 dyne/cm.

The smaller the surface tension value of the oil spill dispersant, the better the quality; oil spill dispersants with a minor surface tension can easily break down oil molecules into tiny droplets that microorganisms can degrade [33]. The surfactant adsorption and desorption rate at the air-liquid interface influences the effect of stirring speed on reducing the surface tension of oil spill dispersant. Higher stirring speeds can cause faster adsorption and desorption, resulting in a more significant decrease in surface tension. This is caused by increased exposure of the liquid to air, allowing more surfactant molecules in the oil spill dispersant mixture to reach the interface and reducing surface tension. The stirring speed accelerates the expansion and loss of foam, affecting the stability and bubble size, which is related to the surface tension of the oil spill dispersant [34]. The effect of temperature on the surface tension of oil spill dispersants is caused by increased dynamics of the molecules that make up the oil spill dispersant, caused by higher heat energy [35].

Based on the results of surface tension measurements on the preliminary oil spill dispersant

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product, it is known that increasing the temperature and stirring speed affects the surface tension value. At a temperature of 40ºC, the tension value is smaller than at other temperature variations and from variations in stirring speed at temperature, producing a lower surface tension value. The result is that a stirring speed of 500 rpm reduces the surface tension of the oil spill dispersant. By varying the stirring speed, it is necessary to find the optimum speed because if the stirring speed is too low, it can cause the mixture of the two surfactants not to mix evenly. In contrast, doing it at an excessive stirring speed will damage the critical micelle concentration (CMC) point in the oil spill. Dispersant.

The resulting Oil Spill Dispersant product is given in Figure 6. Based on the results of determining several specifications of the physicochemical properties of oil spill dispersant in preliminary research, the optimum temperature and speed that will be used in optimizing oil spill dispersant are obtained, namely at a temperature of 40ºC with a stirring speed of 500 rpm.

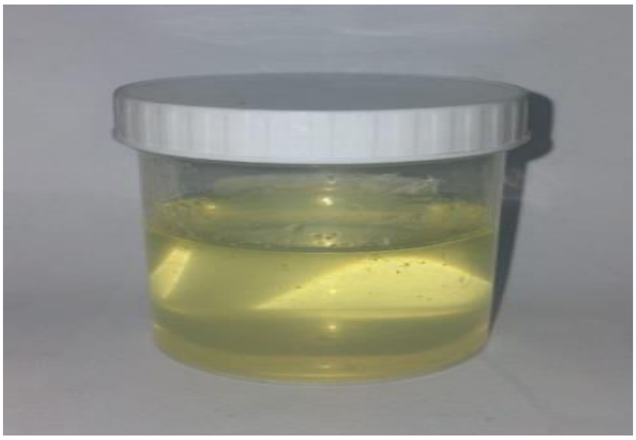

Figure-6. Oil spill dispersant products produced [6].

Application of the Taguchi Method

The Taguchi Method is applied to identify and interpret the factors influencing a response, such as the influence of the Noise Ratio (SNR) factor, which is part of this method. Table-2 shows the results of calculating each factor's effect value of the Signal Noise Ratio. Figure-7 shows the primary influence of SNR on the influence of temperature and stirring speed on density. The temperature factor has a more significant influence, compared to the stirring speed, on the resulting OSD in obtaining the most significant density. From the average of each factor, the most significant value was chosen to be suggested as the proposed design, so that from the analysis in Table-2, it is recommended that the proposed design be at level 3, namely a temperature of 60oC and a stirring speed of 600 rpm.

Figure-8 shows the main effects of SNR on the influence of both variables on pH. In line with density observations, a temperature of 60ºC and a rotation speed of 600 rpm are also recommended for OSD formulation design. However, Table-2 shows that rotation speed has a more significant influence than temperature on increasing the pH of the product.

Figure-9 shows the primary influence of SNR on viscosity observations. It can be seen that a temperature of 40ºC and a rotation speed of 500 rpm provide the most significant viscous gain for the resulting OSD product. Apart from that, according to Table-2, rotation speed significantly influences the viscosity obtained more than temperature.

Table-2. Response table for Signal to Noise Ratios (SNR). Larger is better.

Level	Temp. (ºC)	Stirring speed (rpm)
Response for Density		
1	0.1535	0.1706
2	0.2236	0.2123
3	0.2296	0.2237
Rank	1	2
Response for pH		
1	14.91	14.81
2	15.01	15.07
3	15.01	15.07
Rank	2	1
Response for Viscosity		
1	7.075	6.578
2	6.665	7.101
3	6.800	6.861
Rank	2	1

CONCLUSIONS

The conclusion that can be obtained is that the best stirring speed and temperature is 500 rpm with a stirring temperature of 40ºC with a density value of 1.01615 g/mL, a viscosity of 2.407 cP, a pH of 5.6, and a minor surface tension of 40.36697 dyne/cm. The combination of soy lecithin surfactant substrate and MES (methyl ester sulfonate) surfactant using diethylene glycol ethyl ether and water solvent at the optimization stage can produce an oil spill dispersant that meets the requirements and standards of the Ministry of Industry of the Republic of Indonesia regarding dispersant products in terms of pH, density, and form.

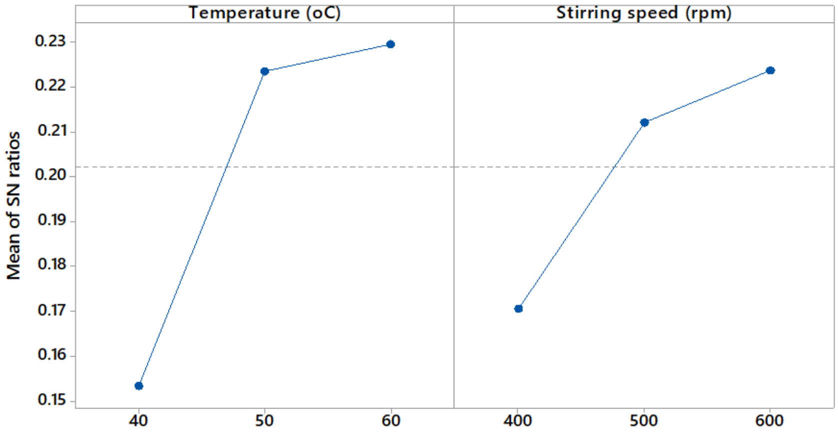

Figure-7. The main effect of SNR on density.

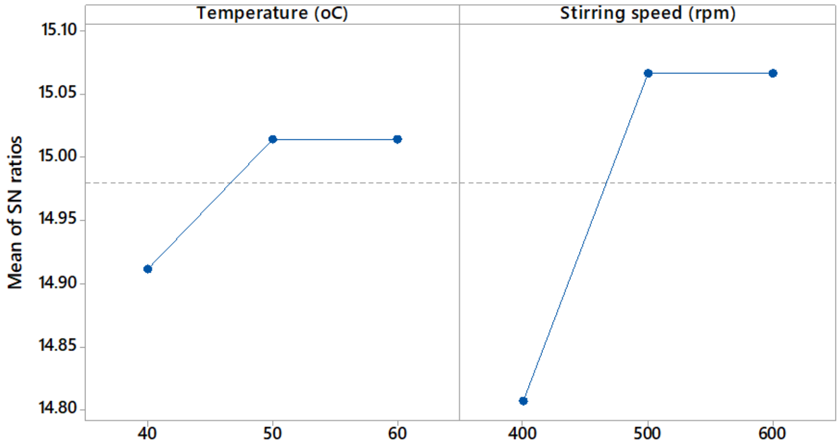

Figure-8. The main effect of SNR on pH.

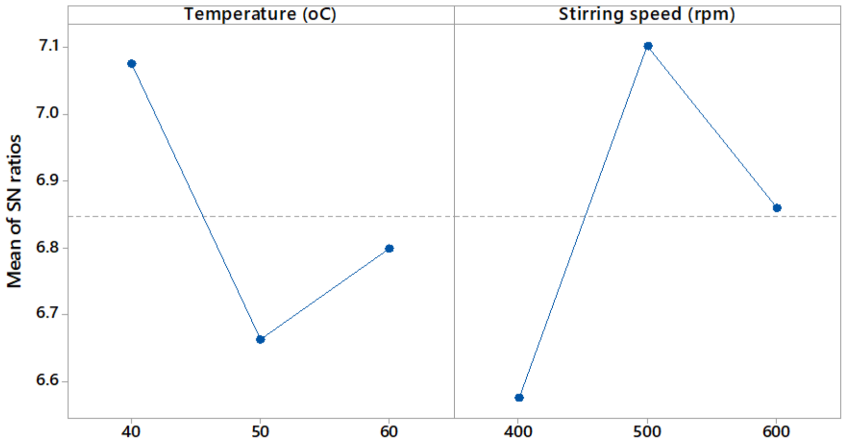

Figure-9. The main effect of SNR on viscosity.

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