



OIL SPILL DISPERSANT FORMULATION FROM PALMITOYL-DEA AND METHYL ESTER SULFONATE

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ABSTRACT

Surfactants are chemical compounds that can interact with solvents to reduce surface tension. Palmitoyl-Diethanolamide surfactant (Palmitoyl-DEA) and methyl ester sulfonate (MES) can be applied in dispersing oil in seawater as Oil Spill Dispersant (OSD). An OSD formulation derived from a mixture of two surfactant solutions, namely Palmitoyl-DEA and MES, was found. Diethanolamide non-ionic surfactant solution and anionic methyl ester sulfonate surfactant solution in various concentrations were mixed according to their respective ratios at 50°C for 60 minutes. The OSD product obtained was then analyzed for density, surface tension, and emulsion stability. OSD products at a concentration of 4% MES and 2% DEA with a 2:1 ratio obtained the best analytical results. The OSD product has excellent density, surface tension, and emulsion stability (> 80%). Furthermore, testing of OSD products was carried out for oil pollution in seawater. The results of simulation tests using OSD products for handling petroleum pollution in seawater (15 mL/L) show an excellent ability to disperse oil (> 90%). The oil and OSD dispersed in seawater showed a more significant increase in COD and BOD values.

Keywords: oil spill dispersant, palmitoyl-DEA, methyl ester sulfonate, surface tension, emulsion stability.

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INTRODUCTION

Oil Spill Dispersant (OSD) is a product that can decompose waste oil into small granules so that it can be dispersed naturally. Modern OSD is a mixture of non-ionic and anionic surfactants. The surfactant in OSD products carries out the adhesion force in the presence of the R group on the surfactant. At the same time, the OSD criteria include low toxicity for mammals, low toxicity for aquatic environments, and readily biodegradable and bio-accumulating [1, 2, 3].

Surfactants are unique active ingredients because they have hydrophilic and hydrophobic sides and can interact with solvents to reduce surface tension. Surfactants can combine moieties between different phases, such as air-water. The unique properties of this surfactant are due to its amphiphilic structure, which means that one surfactant molecule contains polar hydrophilic and non-polar hydrophobic groups [4, 5].

Mechanistically, surfactants reduce the oil's surface tension, increasing the contact area between the oil and the bacteria. In addition, surfactants can also increase the solubility of oil by forming micelles in the aqueous medium. Surfactants in OSD products function to carry out adhesion forces in the presence of R groups on surfactants [6, 7].

Alkanolamide is an amide compound widely used in the chemical, cosmetic, and automotive industries. This compound has detergency properties. These detergency properties are fundamental in living systems where aqueous and fatty phases must coexist. Due to its nature, alkanolamide can act as a surfactant. Alkanolamide-type surfactants are still needed to produce cheap,

environmentally friendly, biodegradable surfactants from renewable sources [8, 9].

Amidation is the reaction of forming an amide compound from a fatty acid or methyl ester with an amine compound. Diethanolamine is a provider of the amine group, which will replace the methoxy group on the methyl ester so that diethanolamine is formed with methanol as a side product. Amines can only react with esters at high temperatures. At low temperatures, the reaction will run very slowly, even with the help of a catalyst [10, 4].

The amidation of fatty acids is a necessary reaction in which fatty alkanolamides are formed as products. The application of this product is related not only to surfactants but also to high-value drugs that exhibit several biological effects, such as anti-carcinogenic and activity against Alzheimer's disease [11].

THEORETICAL BASIS

Diethanolamide (DEA) is a non-ionic surfactant with no charge but a high affinity for water. DEA (Diethanolamide/Ethanol N-alkyl amides) is a non-ionic surfactant that can be processed using the amidation method, which is the product of the reaction between alkanol amines and fatty acid vegetable oils/methyl esters. Amidation is a reaction to form amide compounds [12, 13].

Palmitic acid is a long-chain saturated fatty acid with a melting point of 64°C. This high palmitic acid content makes palm oil more resistant to oxidation (rancidity) than other oils. Palmitic acid is the most common saturated fat found in animals and plants. Palmitic acid is a raw material widely used in various



fields of the oleochemical industry [14, 15]. Palmitic acid has been widely used as a valuable and effective additive for manufacturing pulmonary surfactants (PS) such as Survanta and Surfaxin. In addition, palmitic acid also has biological, physical, and chemical properties, which are well-known for its simple chemical structure, allowing it to be manufactured at low cost [16].

CaO catalyst is the best heterogeneous catalyst with high catalytic activity and basicity. It has low solubility in methanol and is easier to use because it does not require excessive washing water [17]. CaO catalyst is the preferred heterogeneous base catalyst for transesterification reactions and can be derived from various natural resources. The CaO catalyst has a high degree of basicity. This catalyst is also inexpensive, non-corrosive, and has a low risk of environmental impact [18, 19].

Solvent polarity is data used as a consideration in selecting a solvent for a starting method and understanding the effect of a solvent on a chemical process. In general, polarity is referred to as the ability of the solvent to dissolve the solute and can be measured by the physical properties of the solvent. Hexane-isopropanol is used as a solvent in the manufacture of surfactants. Hexane is liquid, flammable, and colourless. Isopropanol has an alcohol-like odour, is colourless, is liquid, and is suitable as an ingredient or a mixture of solvents [8, 11].

Many parameters, including the following, determine surfactants' chemical, physical, and biological properties.

Interfacial Surface Tension (IFT)

Interfacial tension (IFT) is one of the fluid-fluid interfaces' most important physical properties. Without added surfactant, the IFT between two immiscible liquids is an intrinsic property independent of the surface's size or geometry. In the addition of surfactants, this does not happen. It has been proven theoretically and experimentally that IFT depends on surfactant partitioning between the bulk phases and the interface. The distribution of surfactants is determined by geometric factors such as volume fraction and the specific interface area of the two fluids [20].

Hydrophobic-Lipophilic Balance (HLB)

The HLB scale classification is based on the relative polarity of surfactant molecules caused by their hydrophilic and lipophilic groups. The dual character of surfactants will act as a bridge between two substances that are insoluble in each other. The HLB value balances a surfactant's lipophilic and hydrophilic properties. This value describes the relative ratio of polar and non-polar groups in a surfactant related to the solubility of the surfactant in polar and non-polar solvents. Lipophilic surfactants will have low HLB values and be more water soluble [21, 22].

Infrared (IR) Spectroscopy

Infrared light has a longer wavelength than UV-Vis, so it has lower energy with a wave number between

600-4000 cm^{-1} . Infrared light can only cause vibrations in the bond, either in the form of stretching or bending. A molecule's vibrational energy is specific, meaning a specific wave number. However, in practice, IR spectroscopy is more intended to determine the presence of the main functional groups in a sample which is obtained based on the wave number for the vibration [23].

MATERIALS AND METHODS

Materials

The materials used include palmitic acid ($\text{C}_{16}\text{H}_{32}\text{O}_2$), diethanolamine ($\text{C}_4\text{H}_{11}\text{NO}_2$), calcium oxide (CaO), 2-propanol ($\text{C}_3\text{H}_8\text{O}$), hexane (C_6H_{14}), acetone, potassium hydroxide (KOH), phenolphthalein, isopropanol, and hydrochloric acid. (HCl). All were obtained from E Merck.

Methods

The synthesis of this palmitoyl-diethanolamine surfactant will observe the following variables. The independent variable is the mole ratio of the substrate (MES: DEA) is 1:1, 2:1, and 3:1. DEA concentration (DEA: water) is 2, 4, 6 %; and the concentration of MES (MES: Methyl Ester) was 4, 8, 12%.

The fixed variable is the concentration of CaO 5% (w/w palmitic acid); reaction time is 4 hours; reaction temperature is 65 °C; the type of mixed solvent (w/w) is 2-propanol: hexane (1:1); raw material ratio (diethanolamine: palmitic acid) (w/w) is 4:1; the ratio of solvent (solvent mixture : substrate) (v/w) is 3 : 1; and the stirring speed is 250 rpm. The analysis carried out was a qualitative analysis using FT-IR spectroscopy, and the characteristics of the resulting surfactants were a decrease in surface tension (surface tension), pH, and density. Quantitative analysis is done by COD and BOD analysis of OSD products.

Palmitoyl-DEA Surfactant Preparation Procedure

Palmitic acid is mixed with diethanolamine in the first beaker glass, and a solvent mixture of 2-propanol and hexane is added into the second glass. Put the calcium oxide (CaO) catalyst into the second glass and mix it with the first.

The mixture in the beaker glass is put into a stirred tank and, heated and reacted according to the time and speed of a specific stirrer motor. The resulting product is then purified using acetone, and the solvent is evaporated using a rotary evaporator.

OSD Formulation Process

The OSD formulation process is carried out through the following stages. Palmitoyl diethanolamine (DEA) is dissolved in water at 2, 4, and 6%. MES surfactant is dissolved with methyl ester at a concentration of 4, 8, and 12%. The MES and DEA solutions are mixed with a substrate ratio (MES: DEA) of 1:1, 2:1, and 3:1. Each of the above steps is carried out at 50 °C, 400 rpm, for 1 hour.



OSD Performance Test

Put 1 liter of water and 10 ml of oil in a container. 5, 10, 15 mL of OSD was added and then stirred at 10 rpm. The amount of oil remaining on the water's surface was separated using a separatory funnel to take the water fraction. The oil dispersing efficiency was calculated by OSD based on the volume of dispersed oil divided by the volume of the initial crude oil, which was added 10 ml. COD, BOD, pH, and viscosity measurements were analyzed for the dispersed oil in the separated water simulation.

RESULTS AND DISCUSSIONS

Palmitoyl-DEA is one of the surfactants that can be obtained from vegetable oils and their derivatives. Palmitoyl-DEA is obtained from the reaction between diethanolamine and palmitic acid and is widely used in food, cosmetics, and medicine. Palmitoyl-DEA surfactant has good characteristics. Namely, it is easy to decompose in nature, does not damage the skin, and has a low level of toxicity [14]. Palmitoyl-DEA is a non-ionic surfactant widely used as a stable emulsifying agent. These compounds are also used in pharmaceutical, cosmetic, and household needs, such as shampoos and detergents, foam control agents, fuel additives, and corrosion inhibitors [4, 24].

Palmitoyl-Diethanolamide Surfactant Analysis

Palmitoyl-DEA surfactant formulated in Oil Spill Dispersant (OSD) products needs to be analyzed first. The analysis performed on palmitoyl-DEA surfactant was FT-IR analysis, acid number, saponification number, and HLB.

FT-IR Spectroscopy Analysis

FTIR spectrophotometer is a tool that can be used to identify compounds, especially organic compounds, both qualitatively and quantitatively. FT-IR spectroscopy is used to analyze the absorption of functional groups of compounds in the reaction product. The analysis was carried out by looking at the spectrum's shape, namely by looking at the specific peaks that indicate the type of functional group the compound has [23]. The results of the FT-IR analysis of palmitoyl-DEA surfactants can be seen in Figure-1.

The spectrum in Figure 1 shows that in palmitoyl-DEA, a wave number of 3301.24 cm^{-1} indicates the presence of O-H groups. This functional group can be seen in the vibration spectrum, which is a carboxylic acid compound, where the carboxylic acid synthesized in the manufacture of this surfactant is palmitic acid. In waves, 1199.01 cm^{-1} , 1307.41 cm^{-1} , and 1341.04 cm^{-1} indicate the presence of C-N functional groups. The functional group indicated by the vibrational spectrum is an amine compound, in which amine compounds are also synthesized to manufacture palmitoyl-DEA.

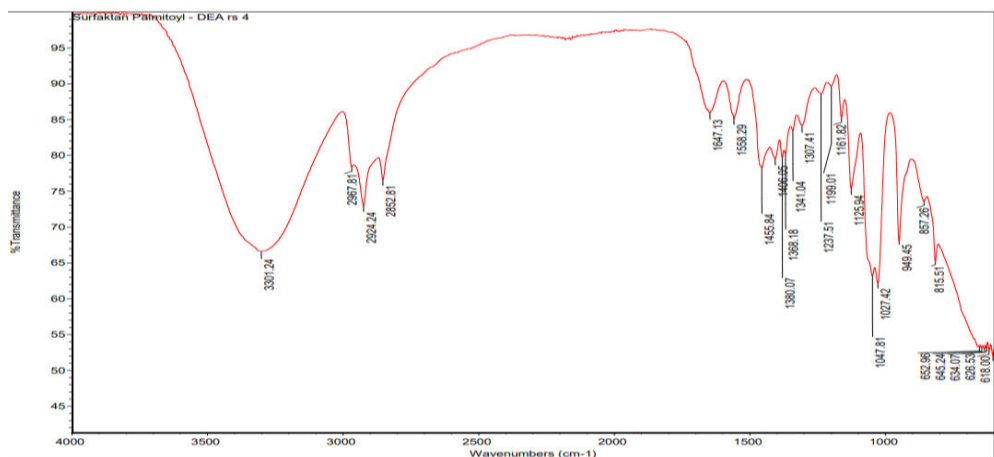


Figure-1. FT-IR spectrum of Palmitoyl-DEA surfactant.

Acid Number Analysis

The acid number is a measure of the amount of free fatty acids. The acid number is calculated based on the molecular weight of the fatty acid. The acid number is the milligrams of 0.1 N KOH used to neutralize the free fatty acids in one gram of oil or fat [21].

Based on the analysis of the acid number of the surfactant DEA, with a product weight of 2 grams, and using KOH as a titer, an acid number value of 14.025 is obtained. Palmitoyl-DEA surfactant can be used in industry. On an industrial scale, it is expected that the acid number of a product must remain low because if the acid

number is high, it will affect the corrosivity of industrial machines.

Saponification Number Analysis

The saponification number is affected by the molecular weight. The higher the molecular weight, the lower the saponification number, and vice versa. The saponification number can roughly determine the molecular weight of oils and fats. The saponification number is the number of milligrams of KOH required to saponify one gram of fat or oil [22]. Based on the results of the research that has been done, the value of the



palmitoyl-DEA saponification number is 4.2075. Analysis of the saponification number of the palmitoyl-DEA surfactant product, together with the acid number, can be used to determine the HLB value of the resulting surfactant product.

HLB Value Analysis

The HLB value is usually determined first to determine the use of surfactants. From the results of this research, the HLB value of surfactant products was obtained, which was measured using the acid number and saponification number approach [21]. The HLB value of the surfactant product obtained is 14. The HLB value indicates that palmitoyl-DEA surfactant can be used as an industrial material widely used in detergent and pulp and paper production applications.

OSD Product Analysis

The Oil Spill Dispersant (OSD) product produced in this study is a formulation between two surfactants, namely the anionic surfactant Methyl Ester Sulphonate (MES) and the non-ionic surfactant palmitoyl-DEA (abbreviated as DEA). The OSD product produced in this study was analyzed for its density, surface tension, and emulsion stability to determine the best OSD product [25]. The OSD formula chosen for further analysis was a formula that had an emulsion stability value of >80%. The OSD formula is a mixture of 4.8.12% MES solution and 2.4.6% DEA solution at a mixing ratio of 2:1. The mixing process produced 7 OSD formulas.

Effect of MES: DEA Ratio on Density

Density is the ratio of the mass of a substance in a specific volume to the influence of temperature. Based on the research that has been done, the density value of the OSD product is obtained at each run. The effect of MES concentration on density in Figure-2 shows that the greater the MES concentration in OSD, the greater the density. This is presumably due to the influence of micelle formation on each OSD product produced. The physical properties of density are related to the molecular weight contained in the OSD product. The greater the surfactant concentration, the greater the molecular weight; therefore, the density value of the resulting OSD product increases [3].

The effect of DEA concentration on density is also shown in Figure-2. An increase in DEA concentration from 2% to 6% slightly decreased the density of the OSD product. This figure also shows that the best concentration of DEA to be formulated into OSD is 4%. Figure-3 shows the effect of the MES: DEA ratio on the density of OSD products. The maximum OSD density is obtained at the MES: DEA ratio of 3:1. This is in line with the observation at the beginning of the study that if the MES concentration is increased, the density will also increase. It can be concluded that at a high MES:DEA ratio of 3:1, the maximum density will also be obtained.

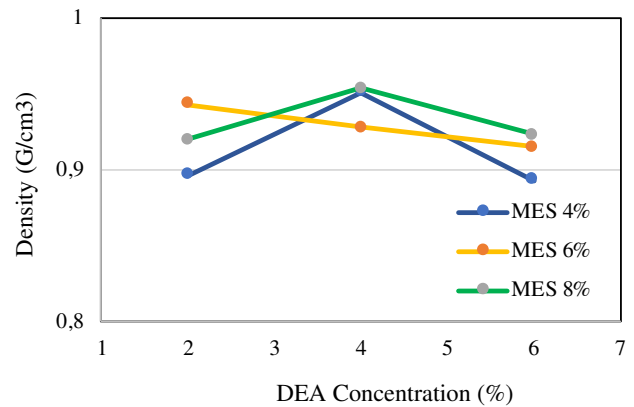


Figure-2. Effect of palmitoyl-DEA concentration on OSD density.

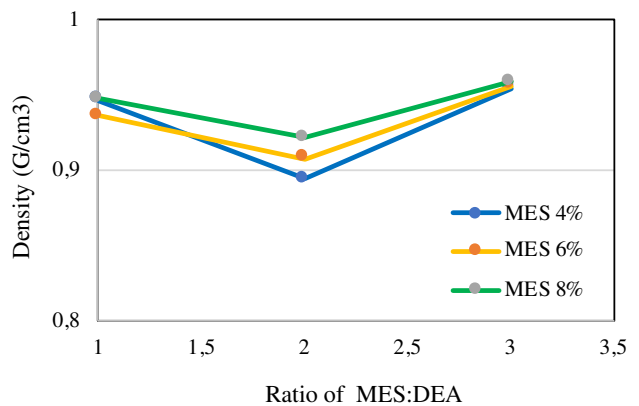


Figure-3. The effect of the MES: DEA ratio on OSD density.

Effect of MES: DEA Ratio on Surface Tension

The surface tension of a liquid is the tendency of the surface of a liquid to tighten so that the surface seems to be covered by an elastic layer. Surface tension can also be interpreted as the ability or tendency of a liquid to always be able to go to a state with a smaller surface area. The surface tension of the liquid in the capillary tube is affected by adhesion and cohesion. Where adhesion can cause liquids close to the wall to rise; meanwhile, cohesion can cause the liquid in the middle to rise as well [1].

Observation of the effect of DEA concentration on the surface tension of OSD products is shown in Figure-4. Of the three changes in MES concentration, it can be seen that the highest surface tension is obtained if the DEA concentration is 4% and the MES concentration is 8%. The surface tension at various MES: DEA ratios is given in Figure-5. In general, it is found that the greater the MES: DEA ratio, the higher the surface tension value. However, in this experiment, things fluctuated; this was thought to be due to the influence of the ratio of DEA to water in each experiment.

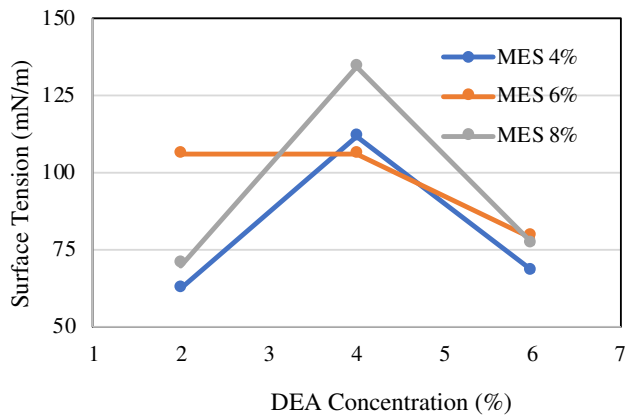


Figure-4. Effect of palmitoyl-DEA concentration on surface tension.

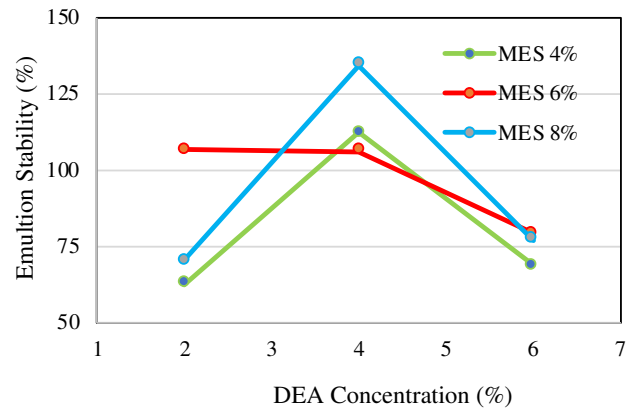


Figure-6. Effect of DEA concentration on the stability of the OSD emulsion.

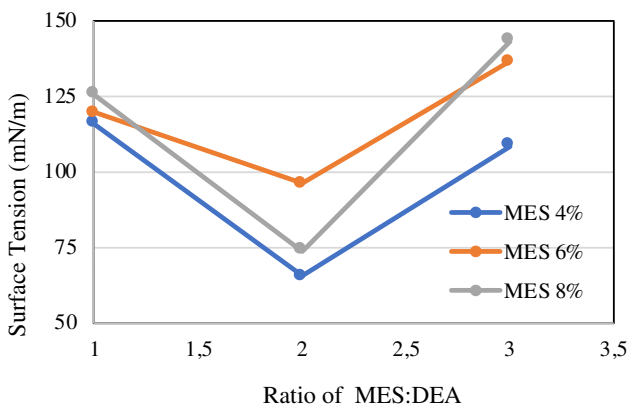


Figure-5. The effect of the MES: DEA ratio on surface tension.

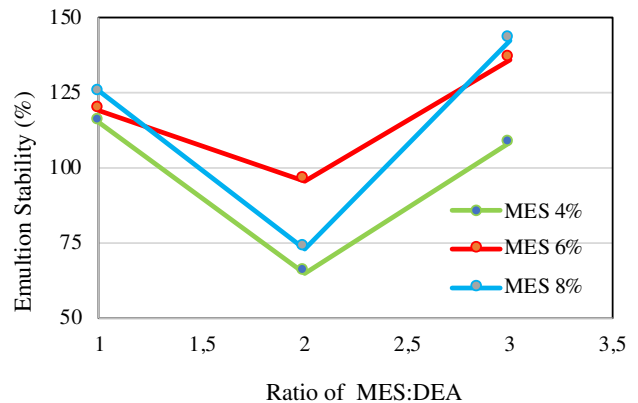


Figure-7. The effect of the MES: DEA ratio on the stability of the OSD emulsion.

Effect of MES: DEA Ratio on Emulsion Stability

Emulsion stability is the effort of the emulsion to maintain foam/bubbles. Emulsion stability can be characterized by separating the oil and water layers [2]. In this study, emulsion stability was measured for OSD products by calculating the size of the foam before and after the residence time. The residence time used is 24 hours. The effect of DEA concentration on emulsion stability is shown in Figure-6. Fluctuating results were also obtained, but overall, it has good emulsion stability, especially at 4% DEA concentration. Figure-7 shows the effect of the MES: DEA ratio on the emulsion stability of OSD products. From the measurement results, OSD products with stable emulsion properties were obtained consisting of 4, 8, and 12% MES solutions mixed with 2, 4, and 6% DEA solutions with a ratio of 2:1, while OSD products at a ratio of 1:1 and 3:1 has less stable emulsion properties.

OSD Performance Test

Oil Spill Dispersant with the best analysis results, its performance was tested on a seawater and oil mixture. In this study, the seawater used came from Belawan Port, while the oil used was pertalite gasoline. From the OSD performance test results, pH, COD, and BOD analyses were carried out to determine the feasibility of using OSD products in everyday life.

pH Analysis

The acidity and base values of a liquid can be determined by the liquid's pH level. The pH analysis carried out in this study used pH indicator paper. The pH values obtained for OSD 5 ml, 10 ml, and 15 ml were 7. Meanwhile, the pH value for seawater used was 7. Based on the results of the pH analysis, it can be stated that the OSD product with a ratio of 1:2 is suitable for use. This is because the pH value will not cause damage to aquatic ecosystems.

Effect of the Amount of Addition of OSD on COD

Chemical Oxygen Demand, often abbreviated as COD, indicates a liquid's chemical oxygen demand. Based on the regulation of the state minister for environment number 19 of 2010 concerning the quality standard of wastewater for oil and gas and geothermal businesses and



activities, it states that the maximum COD content is 160 mg/L using the measurement method SNI 06-6989:2-2004 or SNI 06-6989:15-20. Figure-8 shows that as the volume of OSD product increases in seawater with a fixed oil volume of 10 ml, the COD level also increases. This shows that more OSD is dispersing in water.

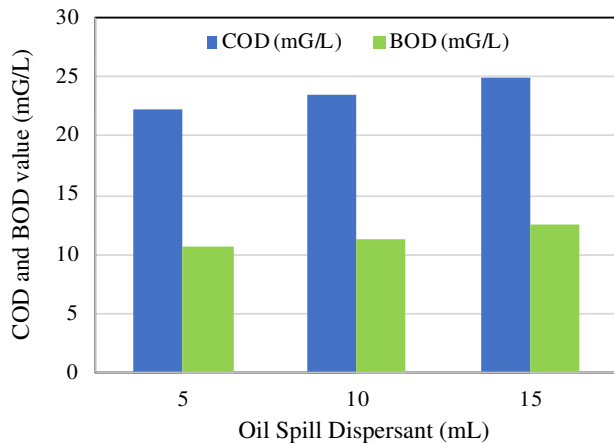


Figure-8. Effect of adding OSD to COD and BOD values.

Effect of the Amount of addition of OSD on BOD

Biochemical Oxygen Demand, also known as BOD, is a number that indicates the biochemical oxygen demand of a liquid. The effect of OSD performance with seawater polluted by oil on the BOD value tested based on SNI 06-6989.72:2009 can be seen in Figure-8. The state minister's regulation for environment number 19 of 2010 concerning the quality standard of wastewater for oil and gas and geothermal businesses and activities states that the maximum BOD content is 80 mg/L using the SNI 06-2503-1991 measurement method. Figure-8 shows that with the addition of 10 mL/L of oil in each sample tested. The data obtained experienced an increase in the BOD value for each additional OSD volume; this shows that OSD can disperse oil in seawater.

In this study, the highest COD test result was 24.9 mg/L, and the highest BOD test result was 12.6 mg/L, so the biodegradability could reach 50.6%. Thus, microbes in these waters can better biodegrade the application of OSD products to seawater pollution by oil additions.

CONCLUSIONS

The palmitoyl-DEA surfactant that has been prepared has a characteristic acid number of 14.025; the saponification number was 4.2075, and the HLB was 14. The best Oil Spill Dispersant (OSD) formulation was obtained by mixing 4% methyl ester sulfonate (MES) solution in methyl ester solvent and 2% palmitoyl-DEA solution in water, with a volume ratio of 2:1. The OSD product shows excellent stability, with good physical-chemical properties including a density of 0.89644 g/cm³, surface tension of 62.46123 mN/m, and a pH of 7. The simulation test results using the best OSD product in handling oil pollution in seawater (15 mL/L) showed an

excellent ability to disperse the oil. The resulting performance test results consisted of the highest COD test value of 24.9 mg/L, the highest BOD of 12.6 mg/L, pH 7, and the percentage of oil dispersion was 50.6%.

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