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Characterization of Methyl Ester Sulfonate (MES) from Mahogany (*Swietenia macrophylla* King) with Variations in H_2SO_4 Concentration and Sulfonation Duration

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ABSTRACT. Methyl ester sulfonate derived from mahogany (*Swietenia macrophylla* K.) oil has been characterized. The research began by synthesizing mahogany methyl ester (ME) in 4 stages: pressing, degumming, esterification, and transesterification. The next process was synthesizing methyl ester sulfonate (MES) also in four stages: sulfonation, bleaching, neutralization, and drying. The reactant for MES synthesis in this study was H_2SO_4 with a mole ratio of 1:6 and variations in the concentration of H_2SO_4 (70%, 75%, 80%, 85%, and 90%) as well as variations in the duration of sulfonation (45, 60, 75, 70, and 105 minutes) to determine the characteristics of the synthesized MES including density, acid number, and emulsion stability. The effect of the combination of treatment variations was analyzed using the two-way ANOVA test and the least significant difference (LSD) test. This research showed that MES from mahogany seed oil from a combination of variations in treatment has a density ranging from 0.91 to 0.97 g/mL where the LSD test at $\alpha = 0.05$ produces three different MES density groups due to variations in the concentration of H_2SO_4 namely A (70 % and 75%), B (80% and 85%), and C (90%). The resulting MES acid numbers ranged from 4.69 – 17.74 mgKOH/g sample with three different groups of MES acid numbers due to variations in the concentration of H_2SO_4, namely A (85 and 90%), B (75% and 80%), and C (70%). The stability of mahogany oil-based MES emulsion ranged from 0.000 – 0.975 and two different MES emulsion stability groups were obtained due to variations in the concentration of H_2SO_4, namely A (80% and 85%) and B (70%, 75%, and 90%). FTIR spectrophotometer showed the presence of S=O groups at wavenumber 1172 cm^{-1} and S–O groups at wavenumbers 972.12 cm^{-1} and 879.54 cm^{-1} proved that MES was successfully synthesized.

INTRODUCTION

Detergent is a product used as a clothes cleaner, both at the industrial and household levels, which has the potential to cause pollutions in the environment. It contains active ingredients, such as linear alkylbenzene sulfonate (LAS), alpha-olefin sulfonate (AOS), primary alcohol sulfate (PAS), alcohol ethoxy sulfate (AES), and others, which harm the environment and living things because of their difficulty to be decomposed by microorganisms (Kang *et al*., 2013). The solution to overcome this problem is to produce detergents that are easily biodegradable so that their impacts on the environment can be overcome.

Surfactants are differentiated by raw material for making it, namely petrochemical surfactants and oleochemical surfactants (Diana *et al*., 2011). Most of the surfactants require raw materials from petroleum (petrochemicals), whose availability is decreasing because they cannot be renewed. On the other hand, vegetable oil-based (oleochemical) surfactants such as methyl ester sulfonate (MES) exhibit many advantages of being nontoxic, being a low excitant to humans, and demonstrating the more excellent performance of environmental compatibility, emulsion, and foamability (Xie *et al*., 2013). Anionic surfactants such as MES can be prepared by the sulfonation process of fatty acid methyl ester (FAME) with chemical reagents containing sulfate or sulfite groups (Ortega, 2021).

One source of natural raw materials that can be used for MES production is oil from mahogany (*Swietenia macrophylla* King) seeds, whose oil content is about 52.5% greater than the oil content of jatropha seeds that is only about 30% – 50% (Daryono *et al*., 2014). Generally, MES can be synthesized from methyl ester through four stages, namely sulfonation, bleaching, neutralization, and drying. Synthesizing methyl ester (ME) is also carried out in four stages, namely pressing, degumming, esterification, and transesterification.

Several studies on mahogany seeds have been carried out include isolation and characterization of mahogany seed oil (Mursiti *et al*., 2013) and the use of mahogany seed oil as an alternative raw material for ME synthesis (Mohan *et al*., 2016). Meanwhile, research on the synthesis of MES has also been carried out with raw materials of palm oil, soybean oil, castor oil, and used cooking oil (Bantacut *et al*., 2014; Putra *et al*., 2018; Nugroho *et al*., 2019 and Hidayati *et al*., 2012). Previous studies showed that the synthesis of MES from the ME raw material of mahogany seed oil has never been carried out. Therefore, it is necessary to study the sulfonation of methyl ester from mahogany oil (*Swietenia macrophylla* K.) as a biodegradable surfactant.

METHOD

The materials used were mahogany seeds and all chemicals (reagent grade) were purchased from Merck (Germay) such as methanol (CH_3OH) pa, phosphoric acid (H_3PO_4) pa, potassium hydroxide (KOH) pa, sulfuric acid (H_2SO_4) pa, sodium hydroxide (NaOH), potassium iodide (KI), phenolphthalein (pp), xylene (C_8H_{10}), hydrochloric acid (HCl), filter paper, universal indicators, aluminum foil, tissue, and distilled water.

Sample Preparation and Treatment

Mahogany seeds as much as 3.97 kg used in this research were obtained from Kendari, Southeast Sulawesi. The mahogany seeds obtained are aerated at room temperature for about three days and then crushed in a blender to form a powder. The obtained mahogany seed powder was then pressed by a hydraulic press machine to obtain mahogany oil (Damayanti and Bariroh, 2012).

Degumming

The quality of mahogany oil obtained from pressing was then improved by degumming process, through the addition of 20% H_3PO_4 as much as 0.3% by weight of mahogany oil at a temperature of 80 °C, and then the mixture was left for 15 minutes until a precipitate occurred, and two phases were formed. The following process was separation with a separating funnel, and then the oil was washed with distilled water at a temperature of 60 °C until the pH was neutral while stirred using a magnetic stirrer. The final step was separating water and oil with a separating funnel. The remaining water was removed through the drying process in an oven at a temperature of 105 – 110 °C (Astam *et al*., 2019).

Esterification

Esterification was carried out by reacting mahogany oil and methanol with a mole ratio of 1:15 in the presence of 1% concentrated sulfuric acid (H_2SO_4) as a catalyst. The stage began by inserting the mahogany oil obtained from the degumming process in a two-neck flask equipped with a condenser and heater. The sample was then heated to a temperature of 65 °C simultaneously with the addition of methanol and sulfuric acid (H_2SO_4) slowly while stirred using a magnetic stirrer for one hour. The reaction mixture was then cooled and then put into a separating funnel and allowed to stand for 24 hours to form two phases. The lower phase was taken and then washed with warm distilled water until pH neutral (Mohan *et al*., 2016).

Transesterification (Methanolysis*)*

Transesterification was carried out by reacting the esterified oil with methanol with a molar ratio to the oil of 6:1 with 1% KOH (w/v) catalyst of the oil weight (Damayanti and Siti, 2012). The stage began by inserting the esterified oil in a two-neck flask equipped with a condenser and heater. Then the sample was heated to a temperature of 60 °C while added with methanol and KOH slowly and stirred using a magnetic stirrer for one hour. The reaction was then stopped, and the mixture was cooled and then put into a separating funnel and allowed to stand for 24 hours to form two phases. The upper phase, namely ME, was taken and then washed with warm distilled water until pH neutral. It was then heated at a temperature of 105 – 110 °C to remove residual water (Musta *et al*., 2017).

Producing Methyl Ester Sulfonate (MES)

Synthesizing MES was carried out in three stages, namely sulfonation, purification, and neutralization. Sulfonation is the main stage in the overall manufacture of MES. ME of mahogany oil was heated at a temperature of 50 – 55 °C and added with sulfuric acid (H_2SO_4) with various concentrations of 60%, 70%, 80%, and 90% with a molar ratio of ME: H_2SO_4 of 1:1.4. The reaction was carried out in a three-neck flask equipped with a condenser

with various sulfonation times: 60, 70, 80, 90, and 105 minutes. The next process was the purification process using 40% methanol and 1% H_2O_2 then put into a separating funnel and let stand for 2 hours to be neutralized with 20% NaOH at 55 – 60 °C and dried to produce MES in granular form (Hidayati *et al*., 2012).

MES Characterization

Density Test

The MES density obtained was determined using a pycnometer. The process began by weighing the empty pycnometer, inserting the sample into it, and weighing it again. This treatment was carried out three times (Saputra, *et al*., 2017).

$$P = \frac{m1-m2}{Vp} \quad (1)$$

Note:

m_1 = pycnometer mass with sample (g)
m_2 = pycnometer mass (g)
V_ρ = pycnometer volume (mL)
ρ = density (g/mL)

Acid Number Test

The acid number test was carried out after first standardizing the potassium hydrogen phthalate solution where 0.6 grams of potassium hydrogen phthalate were dried at 105 °C and 50 mL of distilled water was added and shaken, and followed addition of 3 drops of phenolphthalein indicator. The solution was then titrated with 0.1 N KOH until turning pink in color. After that, the amount of KOH used was calculated (in milligrams).

$$C_{KOH} = \frac{W_{C_8H_5KO_4} \times P_{C_8H_5KO_4} \times 1000}{V_{KOH} \times MW_{C_8H_5KO_4}} \times 100\% \quad (2)$$

Note:

C_{KOH} = KOH titrant molarity (mol/L)
V_{KOH} = KOH titrant volume (mL)
W ($C_8H_5KO_4$) = weight of potassium hydrogen phthalate (g)
P ($C_8H_5KO_4$) = potassium hydrogen phthalate purity (%)
MW($C_8H_5KO_4$) = MW of potassium hydrogen phthalate (2014.22 g/mol)

The acid number test was carried out by weighing 10 grams of oil and putting it in a 200 mL erlenmeyer with the addition of 50 mL of 95% neutral alcohol, then heating it for 10 minutes in a water bath while stirring. The solution was then titrated with 0.1 N KOH with 2 – 4 drops of 1% phenolphthalein indicator in alcohol until turning pink in color. The final step was to calculate the KOH used to neutralize free fatty acids in 1 gram of oil (in milligrams) (Tubino and Ariceti, 2013).

$$\text{Acid Value} = \frac{56.1 \times A \times N}{G} \quad (3)$$

Note:

A = amount of KOH for titration (mL)
N = normality of the KOH solution (N)
G = sample weight (gram)
56.1 = molecular weight of KOH (g/mol)

Emulsion Stability

Emulsion stability was measured by mixing xylene with water in a ratio of 6:4. The mixture was then shaken for 5 minutes using a vortex mixer. The separation of the emulsion between toluene and water was measured based on the length of separation between the phases and then 1 mL of surfactant was added. The volume between the phases before adding the surfactant was compared with that after adding the surfactant (Hidayati *et al*., 2012).

$$\text{Emulsion Stability} = \frac{Vb-Va}{Vb} \times 100\% \quad (4)$$

Note:

V_b = volume of water phase before emulsification (mL)

V_a = volume of water phase after emulsification (mL)

Identification using FTIR Spectrophotometry

The compositions of the synthetic mahogany oil and methyl ester sulfonate (MES) compounds were identified with a scientific IR Buck M500 brand FTIR spectrophotometer with a wavelength of 4000 – 400 cm^{-1} with specifications for instrument conditions: scan of 32 seconds/scan, resolution of 4, and pressure of 80 Torr. The interpretation of the obtained IR spectrum data was carried out by comparing the absorption of functional groups in the IR spectrum of the sample with the literature wavenumbers (Astam *et al*., 2019).

RESULTS AND DISCUSSION

Mahogany Seed Oil Preparation

Mahogany oil was obtained after going through the stages of peeling the skin, drying, smoothing, and pressing. Drying the mahogany seeds was done by aerating them at room temperature for three days to minimize the moisture content. Mahogany seeds were mashed in a blender to make it easier when pressing. Pressing was carried out with a 20-ton hydraulic press (Damayanti and Bariroh, 2012).

Mahogany seed oil obtained through a pressing process that still contained various impurities was purified by degumming, which aimed to separate the oil from sap containing phosphatides, protein, residue, carbohydrates, water, and resin (Qiqmana and Sutjahjo, 2014). After the addition of phosphoric acid (H_3PO_4) pa, the system was left idle, and a phosphatide compound was formed and easily separated from the oil. After the degumming process, the mahogany seed oil became clearer and golden yellow (Figure 1).

(a) (b)

Figure 1. (a). Pressed mahagony oil and (b) Degumming mahagony oil.

Esterification and Transesterification

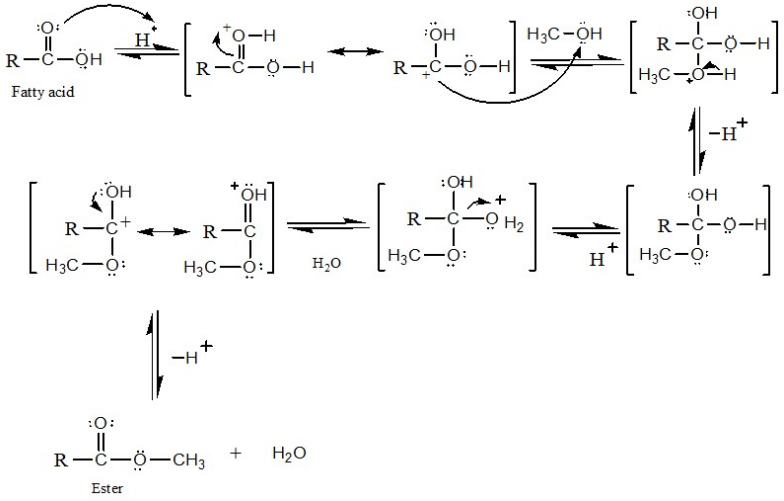

Figure 2. The esterification mechanism.

Vegetable oil contains more than 2% free fatty acids, must be esterified first (Pratigto and Istadi, 2019). Free fatty acids will react with an alkaline catalyst in transesterification, producing soap and damaging the expected

methyl ester product (Habibi *et al*., 2010). The formation of a sufficiently large amount of soap can inhibit the separation of glycerol from the methyl ester and result in the formation of an emulsion during the washing process. Esterification is used as a preliminary process to convert free fatty acids into methyl esters, thereby reducing their levels in vegetable oils. Transesterification with alkaline catalysts serves to convert triglycerides to methyl esters (Hasatan *et al*., 2012). The mechanism of the acid-catalyzed esterification reaction involves the exchange of a hydroxyl group from a carboxylate with an alkyl group from alcohol and the formation of a water molecule (Hikmah and Zuliana, 2010). Transesterification which is generally called alcoholysis is the conversion step from triglycerides (vegetable oils) to alkyl esters, by reacting with alcohol and producing a by-product: glycerol (Damayanti and Bariroh, 2012). The esterification and trans-esterification mechanism can be seen in Figure 2 and Figure 3.

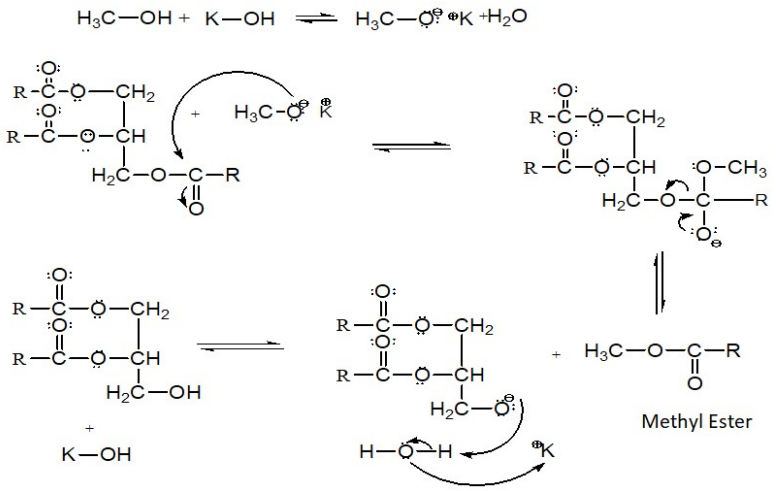

Figure 3. The trans-esterification mechanism.

Sulfonation

Sulfonation is a chemical reaction that involves the incorporation of sulfonic (–SO_3H) groups into a molecule or ion, including reactions involving sulfonyl halide groups or salts derived from sulfonic acid groups, such as the incorporation of –SO_3 into organic compounds (Rachim *et al*., 2012). The reaction product between methyl ester and H_2SO_4 is sulfonic acid methyl ester (MESA). The MESA obtained was then added with methanol and hydrogen peroxide as a catalyst. The purpose of adding methanol is to purify the MESA yield and reduce salt formation. Methanol is reactive and can reduce the occurrence of methyl group substitution in the MES structure (Iman *et al*., 2016). The prediction reaction mechanism for the formation of MES can be seen in Figure 4.

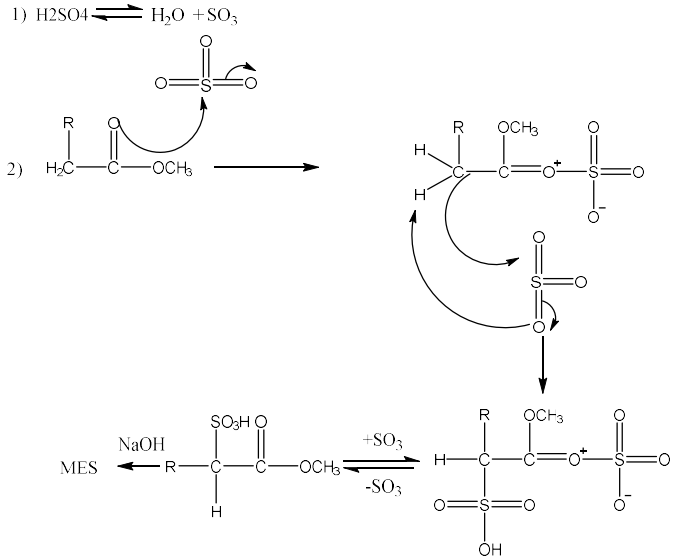

Figure 4. The prediction reaction mechanism for the formation of MES.

Characteristics of Methyl Ester Sulfonate (MES) Yielded

Density

Density means mass per unit volume of an object whose value affects its solubility in saltwater and petroleum (Putra *et al*., 2018). The density test of the synthesized MES was carried out with two treatment combinations, namely variations in concentration (70%, 75%, 80%, 85%, and 90%) and variations in the duration of sulfonation (45, 65, 75, 95, and 105 minutes). The MES density was determined by the pycnometer method, and the results can be shown in Figure 5, which shows that the MES density of mahogany seed oil in all combinations of treatment variations is in the range 0.91 – 0.97 g/mL. The MES density obtained was smaller than that on the market of 1.16 g/mL (Sampepana *et al*., 2015). However, the MES density of the synthesized mahogany seeds was close to the density of water, namely 1 g/mL, which indicates a good level of MES solubility in the solvent (Bantacut and Wahyudi, 2014).

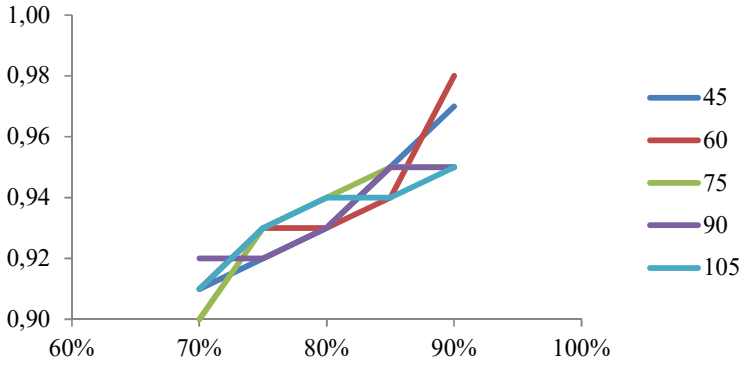

Figure 5. MES density vs concentration H_2SO_4 in various duration of sulfonation.

Figure 5 also shows that in general there is an increase in MES density due to the increasing concentration and duration of sulfonation. However, the results of statistical analysis with two-way ANOVA showed that at α = 0.05 there was no significant difference in the density of the synthesized MES due to the long variation of sulfonation treatment ($F_{score} = 0.19 < F_{tab} = 3.01$) but there was a difference in MES density due to variations in concentration ($F_{score} = 22.41 > F_{tab} = 3.00$). Further tests with the LSD test at α = 0.05 resulted in 3 different MES density groups due to variations in concentration, namely A (70% and 75%), B (80% and 85%), and C (90%).

Acid number

The acid number is the amount (in milligrams) of KOH needed to neutralize one gram of fat or oil. It is used to determine the amount of free fatty acids present in oil/fat. It is determined by titration with a standard KOH solution using the phenolphthalein indicator (Iman *et al*., 2016). The results of the analysis of the MES acid number of mahogany seed oil can be shown in Figure 6 which shows that the resulting MES acid number is in the range 4.69 – 17.74 mgKOH/gram sample. This MES acid number of mahogany seed oil partly corresponds to the MES of used cooking oil of 17.72 mgKOH/gram sample (Hidayati *et al*., 2012).

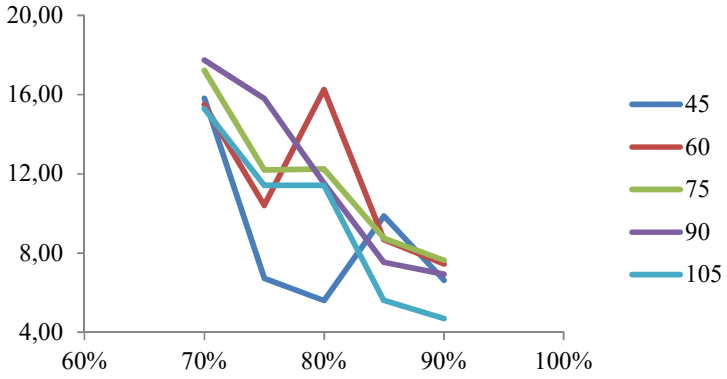

Figure 6. MES acid number vs concentration H_2SO_4 in various duration of sulfonation.

Figure 6 also shows that in general, there is a decrease in MES acid number due to increased concentration and duration of sulfonation. The results of statistical analysis with two-way ANOVA showed that at α = 0.05 there was no significant difference in the value of the MES acid number as a result of the treatment due to variations in sulfonation duration (F_{score} =1.73 < F_{tab} = 3.01) but there was a difference in the synthesized MES acid number due to variations in concentration (F_{score} = 13.09 > F_{tab} = 3.00). Further tests with the LSD test at α = 0.05 resulted in 3 groups of different MES acid numbers due to variations in concentration, namely A (85% and 90%), B (75% and 80%), and C (70%).

Emulsion Stability

The surfactant can act as an emulsifier when two different phases can mix homogeneously. The surfactant will combine two phases with different degrees of polarity. Emulsion stability is measured between water and xylene (Iman *et al*., 2016). MES is said to be good if having a high emulsion stability value. The MES emulsion stability of mahogany oil obtained ranged from 0.000 – 0.975 as shown in Figure7. The stability of the emulsion partially obtained was following that of the MES from used cooking oil of 0.8944 (Hidayati *et al*., 2012).

Figure 7 also shows that the stability value of the MES emulsion fluctuates if the concentration and duration of sulfonation are changed. The results of statistical analysis with two-way ANOVA showed that at α = 0.05 there was no significant difference in the value of the MES emulsion stability results from the synthesis due to variations in sulfonation duration (F_{score} =1.73 < F_{tab} = 2.31) but there was a difference in the stability of the synthesized MES emulsion due to variations in concentration (F_{score} =12.93 > F_{tab} = 3.00). Further tests with the LSD test at α = 0.05 resulted in 2 different groups of MES emulsion stability due to variations in concentration, namely A (80% and 85%) and B (70%, 75%, and 90%).

The addition of surfactants to an emulsion system aims to increase the stability of the dispersion of the phases by reducing the interface stress; the surfactant which acts as an emulsifier will form a thin layer that will cover the emulsified particles and prevent them from recombining (Hidayati *et al*., 2012).

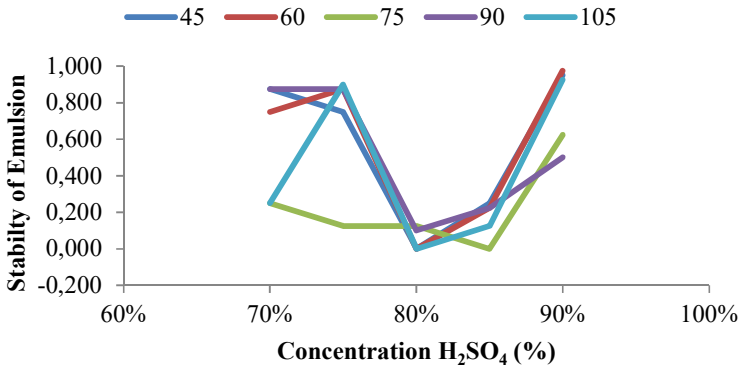

Figure 7. MES Stabillity versus concentration H_2SO_4 in various duration of sulfonation.

Identification of MES using Spectrophotometer (FTIR)

The FTIR spectrum of mahogany oil in Figure 8 shows the absorption of functional groups, including the absorption of C–CH_3 groups at wavenumbers 2924.09 cm^{-1} and 2854.65 cm^{-1}, absorption of groups C=O at wavenumber 1743.65 cm^{-1}, absorption of C=C group at wavenumber 1658.78 cm^{-1}, absorption of CH_2 groups at wavenumber 1435.04 cm^{-1}. Absorption at wavenumber 1373.32 cm^{-1} indicates O–C–O bond vibrations, C–OH bonds at wavenumber 1033.85 cm^{-1}, and absorption of O–H groups at wavenumber 3471.87 cm^{-1}. The interpretation of the FTIR spectra shows the presence of the absorption of functional groups that show the characteristics of the unsaturated fatty acid compounds that make up mahogany seed oil. The absorption of functional groups found in mahogany seed oil obtained is following the absorption of functional groups revealed by Mursiti (Mursiti *et al*., 2013).

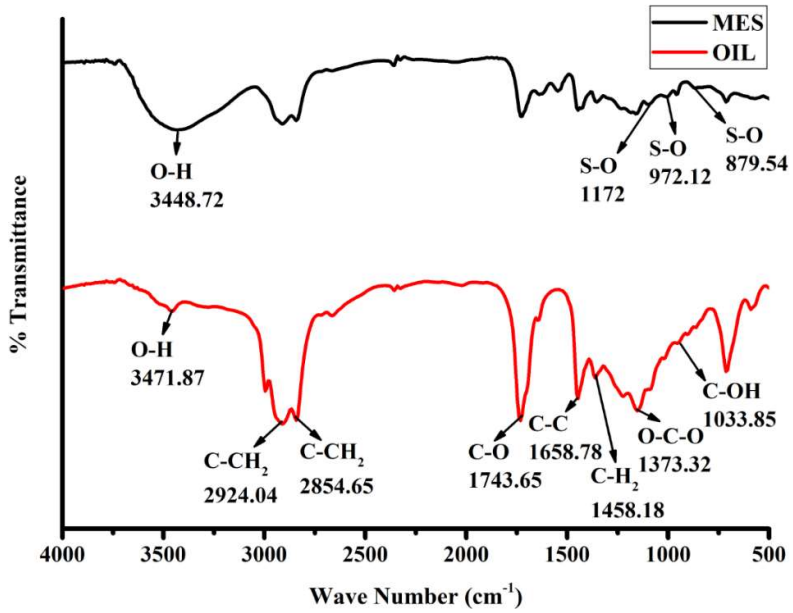

Figure 8. FTIR Spectra of mahogany oil and methyl ester sulfonate.

The FTIR MES spectra in Figure 8 show that there is a peak that is different from the FTIR ME spectra: in the 1172 cm^{-1}, it is suspected that there are S=O bonds, namely the sulfate group on the methyl ester molecule. Besides, there also appears a new peak in the range 879.54 cm^{-1} and 972.12 cm^{-1} (Table 1) that is S–O group absorption but with single bonds. The absorption is following studies (Xiyan and Shumei, 2016; Iman *et al*., 2016). He (2014) in Babu *et al*., (2015) stated that the O-H bonds contained in MES are caused by water absorption which indicates that the MES surface has changed from hydrophobic to hydrophilic.

Table 1. Interpretation of FTIR Spectra of Mahogany Seed Oil and MES.

Vibration type	Wavenumber (cm^{-1})			
	Mahogany Seed Oil	**Mahogany Seed Oil (Mursiti, 2013))**	**Methyl Ester Sulfonate**	**Methyl Ester Sulfonate** (Babu, 2015)
O–H	3471.87	3550 – 3200	3448.72	3550 – 3200
$C–CH_3$	2924.04 and 2854.65	3000 – 2800	-	-
C=O	1743.65	1740 – 1720	-	-
C=C	1658.78	1620 – 1680	-	-
CH_2	1435.04	1470 – 1450	-	-
O–CO	1373.32	1320 – 1210	-	-
C–OH	1033.85		-	-
S=O			1172	1120 – 1230
S–O			972.12 and 879.54	902.69 – 1006.84 and 750 – 1000

CONCLUSION

MES synthesized from mahogany oil in all combinations of treatment variations had densities in the range of 0.91 – 0.97 g/mL; the LSD test at $\alpha = 0.05$ showed three different MES density groups due to variations in concentration, namely A (70% and 75%), B (80% and 85%), and C (90%). The resulting MES acid numbers ranged from 4.69 – 17.74 mgKOH/gram sample with three different groups of MES acid numbers due to variations in concentration, namely A (85 and 90%), B (75% and 80%), and C (70%). The mahogany oil-based MES emulsion stability ranged from 0.000 – 0.975, and two different MES emulsion stability groups were obtained due to variations in concentration, namely A (80% and 85%) and B (70%, 75%, and 90%). FTIR spectrophotometer

showed the presence of S=O groups at wavenumber 1172 cm^{-1} of S–O groups at wavenumbers 972.12 cm^{-1} and 879.54 cm^{-1}, which proved that MES was successfully synthesized.

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