

The Effect of Reaction Time and Temperature on the Synthesis of Methyl Ester Sulfonate Surfactant from Palm Oil as a Feedstock using Microwave-Assisted Heating

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Abstract. Methyl ester sulfonate is an anionic surfactant that can be synthesized from palm oil as a raw material with the addition of sodium bisulfite and calcium oxide catalyst through transesterification and sulfonation process using microwave-assisted heating. The effect of microwave-assisted heating in the transesterification-sulfonation process was investigated in this study. The transesterification process was carried out using a microwave power of 300 watts for 10 minutes with an addition of a KOH catalyst of 1%. The transesterification process gave a result of palm oil methyl ester with a yield of up to 98% and density of 0.8546 gr/ml, and kinematic viscosity of 3.19 cSt. The sulfonation process is carried out using palm oil methyl ester and sodium bisulfite with a mole ratio of 1:3 and calcium oxide catalyst of 1.5% with the microwave power of 300 watts while varying the sulfonation time and temperature. The physicochemical properties of methyl ester sulfonate were analyzed, and the sulfonate group was characterized using FTIR. The optimum condition gave a yield of up to 98.68%, the density of 0.8657 gr/ml, viscosity of 3.75 cSt, pH of 2.12, and surface tension of up to 27.34 dyne/cm at a temperature of 100°C and sulfonation time of 40 minutes.

Keywords: Methyl ester sulfonate, Microwave, Palm Oil, Sulfonation, Transesterification

INTRODUCTION

Surfactants are amphipathic organic compounds that contain a hydrophilic group and a hydrophobic group (Khoshima and Dehghani, 2015) in their molecule, which allows them to interact with polar and nonpolar molecules (Mehling et al., 2007). Surfactants are widely used in the chemical industry, chemical studies, agriculture, and daily needs such as cosmetics, soap, shampoo, cleaning agents, pharmaceutical

products, etc. Aim of using surfactants to reduce surface tension on the interface between fluids and other liquids, liquids with other solids, or liquids with a gas phase (Bazel et al., 2014).

Most cleaning commodities contain petroleum derivatives as their main ingredients. However, consumption of cleaning products increases and natural reserves of petroleum decreases, the petroleum derivatives may soon suffer from a lack of raw materials (Xie et al., 2013) and

shortage of fossil resources (Alwadani and Fatehi, 2018). In addition, surfactants derived from petroleum derivatives have high toxicity levels and can irritate the skin (Bondi et al., 2015).

Generally, existing commercial surfactants are often based on slowly degrading compounds that can harm the environment and humans (Babu et al., 2015). The harmful effects of those substances to aquatic ecosystems consist of their adsorption onto the cell surface of lower microorganisms that affect the ecosystem's production-destruction characteristics and the aquatic environment's self-purification parameters (Stepanets et al., 2001). In recent decades, the detergent industry has met the challenge to produce cleaning products that have an important role in our daily lives, such as removing germs and other contaminants safely and effectively and caring for homes and property, thus making the environment healthier and more pleasant (Amous, 2016). Much attention is focused on agriculturally derived oleochemicals as alternative feedstocks to reduce costly petroleum-derived products' harmful environmental effects (Elraies et al., 2010).

One of the surfactants that can be made for this challenge is the anionic surfactant methyl ester sulfonate. Methyl ester sulfonate (MES) is an anionic surfactant made from renewable natural resources, such as vegetable oil and fats. MES can replace petroleum-derived surfactants because it has many advantages: they are easily degraded (Xie et al., 2013), good cleaning agents, easily dissolve in water, and inexpensive (Sánchez and del Valle, 2005), so they received great attention. From the economic and environmental point of view, MES is considered a valuable surfactant that is widely used in the chemical industry because

of their excellent surface activity and self-assembly behavior (Jin et al., 2016) and their good performance and high level of biodegradability in detergency (Asselah and Tazerouti, 2014) means it is also more stable and tolerant to hydrolysis at low or high pH (Alwadani and Fatehi, 2018).

One vegetable oil that has great potential that can be used as a raw material for anionic surfactant production is palm oil. Palm oil can be used as a raw material for producing MES because it is inexpensive, abundant, renewable, and biodegradable. In addition, palm oil contains fatty acids that can be converted to MES. The dominant fatty acids in palm oil are oleic acid at 47.7% and palmitic acid at 36.3% (Derawi et al., 2014).

MES can be made through the transesterification process of palm oil and the sulfonation process of methyl ester. Sulfonation is the process used to manufacture anionic surfactants by reacting methyl ester with sodium bisulfite as a substitute for SO_3 gas, which is very reactive. The use of sodium bisulfite in the sulfonation process of methyl ester has several advantages, which include producing MES with a brighter color, and can be used on a small scale process, and using simpler equipment (Hidayati et al., 2005).

Babu et al. (2014) have studied the characteristics of sodium methyl ester sulfonate for chemically-enhanced oil recovery using castor oil and chlorosulfuric acid as raw materials that produced surfactants that can reduce the surface tension by 38.4 dyne/cm (Babu et al., 2015). Jin et al. (2015) have made MES using raw materials from waste cooking oil and chlorosulfuric acid in the sulfonation process, which produced a surface tension of 32.3 dyne/cm with a reaction time of 3 hours and yields of up to 78 % (Jin et al., 2016). Research

on making surfactants using sodium bisulfite in the sulfonation process with raw material in the form of Itaconic acid has been carried out, which produced a surfactant with a surface tension of 25.02 dyne/cm with a reaction time of 5 hours and yields reached up to 84.01% (Xu et al., 2016).

Therefore, based on these researchs that has been done on the sulfonation process of making surfactant MES, they all require long reaction times as the reactants were heated using conventional heating methods. Alternative heating methods can be used to save energy consumption to produce MES, such as microwaves. The use of microwave technology as a heat source has long been developed, where the use of microwaves can shorten reaction times, make the process energy-efficient, save costs, and are also environmentally friendly (Nomanbhay and Ong, 2017). Additionally, microwave-assisted can reduce the reaction time, increase the product's yield, and improve product properties and quality (Ghasali et al., 2017). Microwaves can speed up reactions because they work by emitting waves directly to the reactants so that the heating process is faster and applied evenly throughout the entire surface, hence the heat transfer is more effective (Motasemi and Ani, 2012).

Since the microwave-assisted heating process can increase the rate of reaction and reduce the formation of by-products compared to conventional methods (De Souza, 2015), the former can be a good alternative in the production of MES. Hence, microwave-assisted methods can be developed to produce MES from palm oil.

This study aims to prepare methyl ester sulfonate surfactant from palm oil using sodium bisulfite with microwave-assisted heating. The density and viscosity of palm oil methyl ester were analyzed, and the

composition was determined using GCMS analysis. The sulfonate group in MES was characterized using FTIR, and the physicochemical properties such as density, viscosity, yield, pH, and surface tension were analyzed.

MATERIALS AND METHODS

Materials

The materials used in this study are palm oil (Bimoli, commercial), methanol (Merck, 98%), potassium hydroxide catalyst (Merck, 99,99%), sodium bisulfite (NaHSO_3), calcium oxide catalyst (Merck, 97%), sodium hydroxide (Merck, 97%), and distilled water.

Microwave Equipment

The main equipment used in this study was an Electrolux microwave EMM2308X model (power 150 – 800 Watt) with a 2-neck flask, condenser, stirrer, and thermocouple.

Experimental Procedure

Transesterification Process

The palm oil methyl ester transesterification process was carried out by reacting palm oil and methanol with a mol ratio of 1:9 at 60°C. The reaction was carried out for 10 minutes with the addition of 1% KOH catalyst using a microwave power of 300 watts. After the reaction is completed, the product is separated into methyl ester and glycerol. The methyl ester is purified by adding warm distillate water until the pH is neutral. The purified methyl ester is heated to reduce the water content in an oven at 100°C for 48 hours.

Sulfonation Process

The sulfonation process was carried out by reacting palm oil methyl ester (60 ml, 0.18

mol) with sodium bisulfite (57.01 g, 0.55 mol), which has been dissolved in distilled water (100 mL) with a mole ratio of 1:3. The reaction was carried out with the addition of a CaO catalyst of 1.5% wt. The mixture is then heated using a microwave at 300 watts of power with variations in temperature (80°C, 90°C, and 100°C) and reaction time (20, 30, 40, 50, and 60 minutes). After the sulfonation process is complete, the mixture is separated to isolate methyl ester sulfonate from unreacted sodium bisulfite and the remaining catalyst using a separating funnel. The MES, which was in the upper layer, was then purified with a methanol addition of 30% v/v and heated at 55°C for 10 minutes using a microwave at 300 watts of power. The purified MES is then neutralized by adding 20% NaOH until the pH is neutral. MES that has been neutralized are then characterized and analyzed to determine their physicochemical properties.

RESULTS AND DISCUSSION

Transesterification Process

The transesterification process of palm oil gave methyl ester yields of up to 98% and had a density of 0.8585 gr/ml and a kinematic viscosity of 3.149 cSt. It is in accordance with the standard SNI 7182:2015 biodiesel that has been determined, with a density of 0.85 - 0.89 gr/ml and kinematic viscosity of 2.3 - 6 cSt.

The difference in density and viscosity of palm oil and methyl ester is the breakdown of the glycerol chain contained in oil (Aziz, 2008) and the influence of the intermolecular forces in the oil (Santoso et al., 2019).

Table 1 shows the composition of the resulting palm oil methyl ester. Figure 1 displayed the results of GCMS methyl ester. From Figure 1, the results of GCMS palm oil

methyl ester show that the largest component in palm oil methyl ester is 9-Octadecenoic acid (methyl oleate) with a composition of 50.2865%, which is formed at 23.035 retention time, and also hexadecenoic acid (methyl palmitate) with a composition of 41.9157% at retention time 20.909.

Table 1. Fatty Acids Composition

Fatty Acid	Palm Oil (%)	Methyl Ester (%)
Caprylate	0	0.0409
Caprate	0	0.1433
Laurate	0.262	0.7982
Myristate	1.012	4.5129
Palmitoleate	0	1.9136
Palmitate	38.201	41.9158
Stearate	3.637	0
Oleate	45.962	50.2865
Linoleate	10.926	0.3889

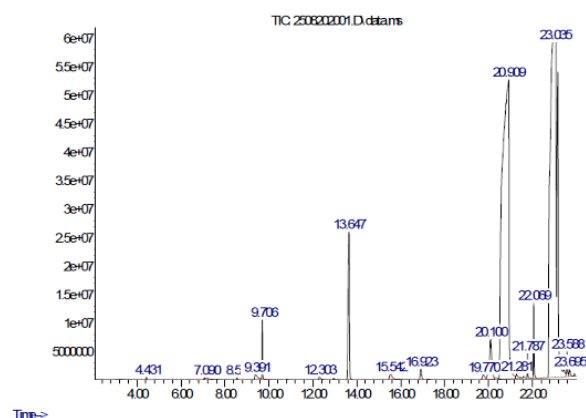


Fig. 1: GCMS of Methyl Ester

Sulfonation Process

Yield Methyl Ester Sulfonate

Figure 2 shows the effect of sulfonation time on the yield of methyl ester sulfonate that the longer the reaction time used in a process, the greater the yield obtained. It is because the longer the reaction time, the greater the contact between the reacting substances, leading to a greater yield of the

MES product obtained. However, a reaction time that is too long can reduce the yield of the product. This decrease in yield is because the energy required by the solvent is very large and can lead to the destruction of organic molecules contained in the methyl ester, which causes the interactions between molecules in the material to be irregular (Gude et al., 2013).

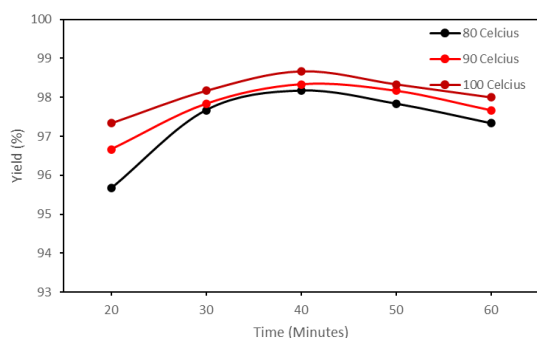


Fig. 2: The effect of sulfonation time on the yield of methyl ester sulfonate

The largest yield of 98.67% was obtained at a reaction time of 40 minutes with a sulfonation temperature of 100°C. In contrast, the lowest yield of 95.67% was obtained at a reaction time of 20 minutes at 80°C.

The density of Methyl Ester Sulfonate

The methyl ester sulfonate density obtained in this study has increased from 0.8640 gr/ml - 0.8657 gr/ml compared to the density of palm oil methyl ester, which is 0.85462. The binding of the SO₃ sulfonate group to the methyl ester molecule tends to cause the MES to have a larger molecular size so that it has a higher density than the raw material. The effect of sulfonation in relation to the density of methyl ester sulfonate is shown in Figure 3.

Based on Figure 3, it can be seen that the largest density of MES was obtained at a reaction time of 40 minutes with a sulfonation temperature of 100°C, which was 0.8657

g/ml, and the lowest density 0.8640 gr/ml was obtained at a reaction time of 20 minutes with a sulfonation temperature of 80°C.

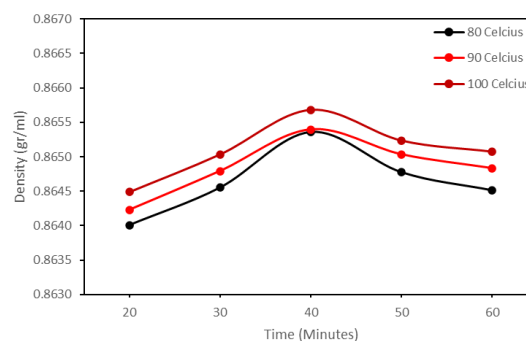


Fig. 3: The effect of sulfonation time on the density of methyl ester sulfonate

However, a long reaction time can reduce the product's density because the long reaction time in the sulfonation process will reduce the cohesive forces and increase molecular changes (Muhammed, 2016). It can cause the SO₃ group to be separated from the MES molecule, which results in a reduced mass of the MES volume.

The viscosity of Methyl Ester Sulfonate

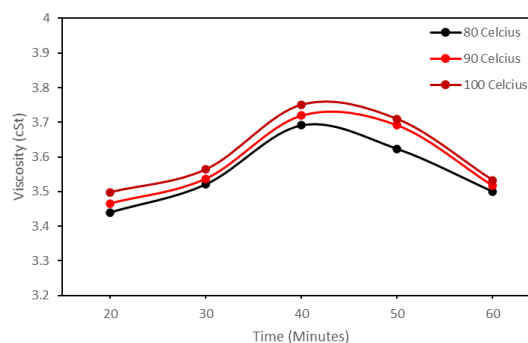


Fig. 4: The effect of sulfonation time on the viscosity of methyl ester sulfonate

Figure 4 shows the effect of sulfonation time on the viscosity of methyl ester sulfonate. From Figure 4, it can be seen that the MES viscosity has increased from a reaction time of 20

minutes to 40 minutes and then decreased from reaction time 40 minutes to 60 minutes.

The largest viscosity of MES was obtained at a reaction time of 40 minutes with a sulfonation temperature of 100°C, which was 3.7507 cSt. While the lowest viscosity, 3.4393 cSt, was obtained at a sulfonation time of 20 minutes with a sulfonation temperature of 80°C.

A longer reaction time used in a process causes the obtained viscosity to increase due to the greater contact between the reacting substances such that more methyl esters are converted to form MES so that the MES product's viscosity will be obtained also be greater. However, a long heating time can cause viscosity to decrease due to a complex set of cognate consequences. One consequence is that it reduces cohesive forces, which can, in turn, reduce shear stress. On the other hand, another result is that molecular interchange increases, leading to increased shear stress. Together, these seemingly contradictory effects cause the viscosity to decrease (Muhammed, 2016).

pH of Methyl Ester Sulfonate

The pH test was carried out on the MES surfactant to describe the presence of the sulfonate group bound to the strong acid methyl ester structure during the sulfonation process. The effect of sulfonation time on the pH of methyl ester sulfonate can be seen in Figure 5.

Based on Figure 5, it can be seen that the pH of the MES has decreased from the reaction time of 20 minutes to the reaction time of 40 minutes and has an increase in the reaction time of 40 minutes to the reaction time of 60 minutes. The largest pH of MES, which was 3.28, was obtained at a reaction time of 20 minutes with a sulfonation

temperature of 80°C. In contrast, the lowest pH of MES, which was 2.12, was obtained at a reaction time of 40 minutes at 100°C.

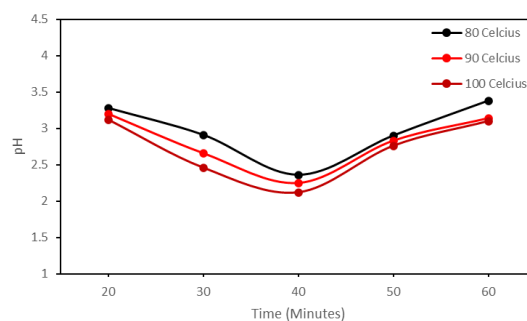


Fig. 5: The effect of sulfonation time on pH of methyl ester sulfonate

The pH tends to decrease with the increase in the length of the sulfonation time. It is caused by the longer the reaction time used, the more contact between the methyl ester and the acidic sodium bisulfite. It leads to more methyl esters converted to form MES and more sodium bisulfite bound to the methyl ester structure so that the pH of the MES product obtained will also be lower.

Surface Tension of Methyl Ester Sulfonate

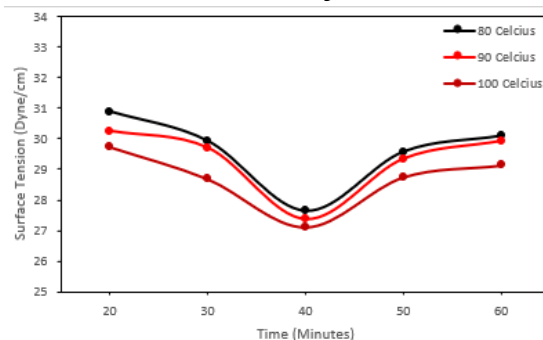


Fig. 6: The effect of sulfonation time on the surface tension of methyl ester sulfonate

Surface tension is the downward pull or force that causes the surface of the liquid to contract so that it looks elastic or tense. Surface tension is measured using a capillary tube by measuring the increase in fluid in the capillary tube. The low surface tension

indicates that there has been a sulfonation reaction where the binding of the sulfonate group to the methyl ester will increase the number of sulfonate groups that will be converted into surfactant MES. It was causing more MES molecules to be formed. Hence it will reduce surface tension to a larger extent.

From Figure 6, it can be seen that the use of higher temperatures causes the surface tension of the MES to decrease. The higher amount of surfactant molecules formed can make the surface tension decrease because the more surfactant molecules there are, the lower the cohesive water forces.

The largest surface tension of MES was obtained at a reaction time of 20 minutes with a sulfonation temperature of 80°C, which was 30.89 dyne/cm, while the lowest of the surface tension of MES was obtained at a reaction time of 40 minutes at 100°C, which was 27.103 dyne/cm.

The surface tension obtained from this study is lower than previous research using conventional methods conducted by Slamet et al., 2017 which is 35.70 dyne/cm with a

reaction time of 4.5 hours and Jin et al., 2016 at 32.3 dyne/cm for 3 hours, and higher than the research conducted by Xu et al., 2016 which was 25.02 dyne/cm with a reaction time of 5 hours.

FTIR Analysis of Methyl Ester Sulfonate

FTIR analysis is carried out to identify the MES structure formed and determine if the reaction is carried out as expected. The results of the FTIR analysis of MES from the sulfonation process are shown in Figure 7.

In the FT-IR spectrum in Figure 7, the absorption peak in the wavenumber area of approximately 1741.01 cm⁻¹ is a typical absorption of the carbonyl group (C = O) of the ester and is supported by the absorption peak of C-O-C in the wavenumber area of 1117.70 cm⁻¹ so that it can be concluded that the FTIR results of MES indicate the presence of an ester group. The peak of the absorption in the wavenumbers 1168.59 cm⁻¹ and 1194.89 cm⁻¹ shows a typical absorption from the sulfonate group (-SO₃H).

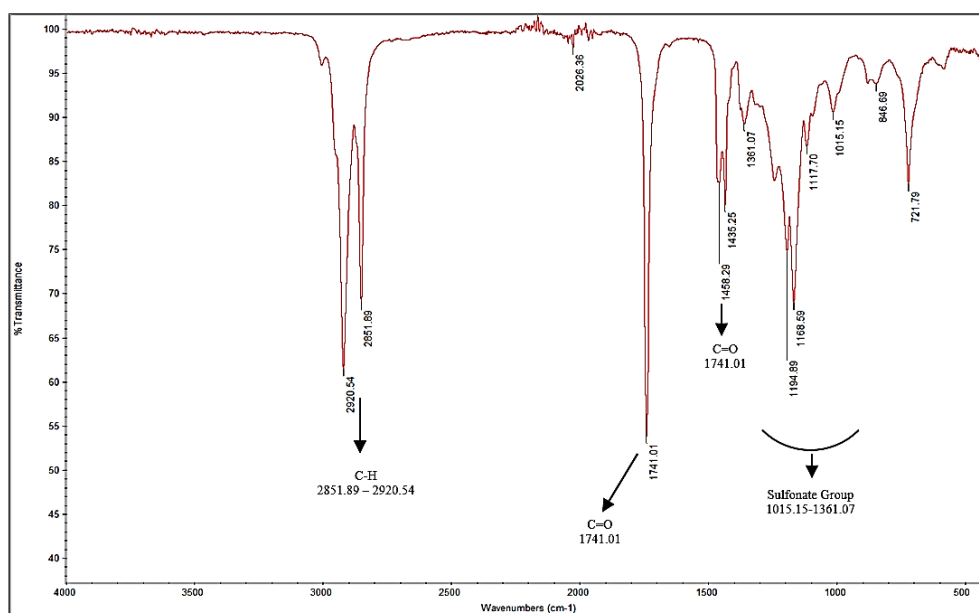


Fig. 7: Analysis of the sulfonate groups on the MES of palm oil methyl esters using FTIR.

CONCLUSIONS

Palm Oil was successfully synthesized into MES with microwave-assisted heating. The use of microwave-assisted heating can reduce the transesterification and sulfonation time. With microwave-assisted heating, MES can be produced within a shorter time with a surface tension of up to 30.89 dyne/cm – 27.38 dyne/cm. At the sulfonation time of 40 minutes and sulfonation temperature 100°C, the experiment obtained a yield of up to 98.68%, the density of 0.8657 gr/ml, the viscosity of 3.75 cSt, pH of 2.12, and surface tension of up to 27.34 dyne/cm, and these were the best conditions. The FTIR result shows that the sulfonate group is at a wavelength of 1168.59.95 cm⁻¹ and 1194.89 cm⁻¹, indicating that methyl ester was successfully converted to MES.

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