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# Surfactant Production of Methyl Ester Sulfonate from Virgin Coconut Oil using Aluminum Oxide with Microwave Assistance

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**Abstract.** As a surfactant, methyl ester sulfonate (MES) can be produced from virgin coconut oil (VCO) raw materials through the following stages: transesterification, sulfonation, and purification. The transesterification process was carried out to produce methyl esters by reacting VCO with methanol in a mole ratio of 1:41 using a 1% KOH catalyst at a microwave power of 300 W for 60 min. The effects of microwave power and mole ratios between methyl esters and sodium bisulfite in the sulfonation process were investigated. The sulfonation process was carried out using a 1% aluminum oxide catalyst. The purification process was carried out by reacting MES with 35% v/v methanol at 150 W of microwave power for 10 min. The resulting MES was analyzed using gas chromatography and Fourier transform-infrared (FT-IR) spectroscopy. The optimum conditions for surfactant production included a microwave power of 450 W and reactant mole ratio of 1:1, which resulted in a surface tension of 37.9 dyne/cm, pH of 4.21, density of 0.87 g/mL, and viscosity of 3.33 cSt. Based on the FT-IR analysis, the vibrational strain of the sulfonate group was detected at a peak value of 1014.42 for symmetrical S-O and 722.24 $cm^{-1}$ for asymmetrical S-O.

*Keywords:* Methyl ester sulfonate; Microwave; Sulfonation; Transesterification; Virgin Coconut Oil

## 1. Introduction

Surfactants are active ingredients in detergents, personal-care products, pharmaceuticals, mining, and the paper industry. In addition, surfactants have the characterisctic of reducing surface tension so that they can be used as flocculation and wetting agents, adhesives (Singh et al., 2007), the enhancement of oil recovery (Irawan et al., 2017), stabilization of emulsion (Mulligan, 2009), and foam production (Zhang et al., 2017). However, they are still primarily used as synthetic surfactants. Globally, the production of synthetic surfactants has reached 13 million tons/year, and it has been economically profitable. In fact, in 2014, the market research institute Ceresana, Germany, reported that the world market for surfactants has exceeded 33 billion US dollars and that the annual revenue for surfactants is estimated to increase by 2.5% by 2022 (I et al., 2016). Some commonly used synthetic surfactants include cetyltrimethyl ammonium bromide,

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sodium dodecyl sulfate, and polyoxyethylene sorbitan monolaurate, which are derived from fossil fuel compounds (petrochemicals) (Panda et al., 2020).

However, most commercial synthetic surfactants still have negative effects on human health and the environment because they are corrosive and toxic. Furthermore, the use of surfactants developed from petrochemical compounds can increase environmental problems, such as global warming, and the shortage of fossil resources (Alwadani & Fatehi, 2018). Thus, it is necessary to replace surfactant raw materials with materials sourced from natural materials (oleochemicals) because they are less toxic and can be more stably decomposed into the environment (Muntaha & Khan, 2015). In addition to the above two factors, global detergent manufacturers have increasingly focused on oleo-based feedstocks, such as surfactants developed from biodegradable vegetable oils, and have used them in enhanced oil recovery (Tulathammakit & Boonyarach, 2014). In fact, biodegradable materials for industrial production have become a research-intensive area due to the increasing demand to conserve limited petrochemical resources and the need to protect the environment from persistent petrochemical products (Soy et al., 2020).

The surfactants have hydrophobic tails and hydrophilic heads that have evolved into nonionic, anionic, cationic, and amphoteric surfactants, and have been widely distributed and used worldwide (Khoshsima & Dehghani, 2016; Rozaini et al., 2012). Based on these four types of surfactants, the most widely used anionic surfactants with active ingredients are derived from carboxylates, phosphates, sulfates, and sulfonates (Alwadani & Fatehi, 2018). In addition, the use of anionic surfactants as bio-based surfactants is less toxic, biodegradable, and compatible with humans and the environment (Adiwibowo & Slamet, 2018).

Sulfonate-based surfactants can be developed from vegetable oil, with soybean and coconut oils being the most popular raw materials used to derive oleochemical feedstocks, such as fatty alcohol and esters (Hill, 2001). Comparing the two sources, coconut oil has a higher fatty acid content, especially lauric acid (up to 44%–52%) (Sari, 2018). The derivative of the material is usually methyl ester sulfonate (MES), which exhibits suitable surface-active properties, excellent active ingredients as the main ingredients in laundry detergents, and good biodegradability (Tobori & Kakui, 2019).

Transesterification and subsequent sulfonation are the two main processes involved in developing this surfactant. Sheats and MacArthur (2001) reported that the sulfonation-transesterification process using falling film reactor equipment at a ratio of coconut oil and $SO_3$ of 1:1.2 was able to produce 83.6% methyl ester sulfonate product for a reaction time of 1.5 hours. Jin et al. (2016) produced MES from soybean oil, reused oil, and waste cooking oil (WCO) through the sulfonation process of methyl esters using chlorosulfonic acid reactants for 3 h at 60°C. Methyl ester was produced from WCO with a yield of up to 78%, which is slightly lower than the yield of soybean oil (i.e., 82%) but comparable with that of the reused cooking oil (i.e., 76%). The surface tension value of MES produced from WCO was 32.3 mN/m, which is slightly higher than that of soybean oil (i.e., 31.5 mN/m) but comparable with that of the reused oil (32.6 mN/m) (Jin et al., 2016). Furthermore, similar research was conducted by Slamet and Wulandari (2017), who studied MES manufacture from crude palm oil using a natrium bisulfite ($NaHSO_3$) reactant at 100°C. The optimum conditions for surfactant production included a reactant ratio of 1:1.5 with a sulfonation time of 4.5 h and a surface tension of 35.70 dyne/cm (Slamet & Wulandari, 2017). Several studies that have been conducted with conventional heating suggest that the process completion requires a long time and, thus, a process that is less time-consuming is urgently required.

Based on these studies, the technology that has recently received considerable attention is the use of microwaves. Ning and Niu (2017) reported that microwaves can speed up the

transesterification process by up to 30 times compared to conventional waves. This is because, as compared to conventional technology, microwave reactors produce longer wavelengths that focus directly on the sample; moreover, the reactions use less energy and are less time-consuming. This technology can effectively avoid material aggregation with the long reaction time required by conventional technology (Huang et al., 2020). Therefore, microwave radiation is a suitable method to speed up the reaction and facilitate a more effective heat-transfer process (Motasemi & Ani, 2012).

Hence, a transesterification–sulfonation microwave process needs to be developed to produce an MES surfactant from virgin coconut oil (VCO). It is also important to investigate parameters such as variation of reactants and microwave power to convert methyl esters to surfactants because no report has explained this so far. Therefore, we study the effect of microwave power (150, 300, 450, and 600 W) and variations in the mole ratios between methyl esters and sodium bisulfite (1:0.6, 1:0.8, and 1:1 (w/w methyl ester)) in producing an MES as a surfactant.

## 2. Methods

### *2.1. Materials*

The main ingredient used in this study was VCO, with the brand Barco produced by PT. Barco, Jakarta, Indonesia. As supporting materials, methanol ($CH_3OH$) (99.9% Merck 106498; transesterification reactant) and potassium hydroxide (KOH) (85.0% Merck 05033; transesterification catalyst) were used. As for the sulfonation process, the assembly used was Merck 98%–100.5% $NaHSO_3$ 06528 with Merck 01095 $Al_2O_3$ used as the catalyst.

### *2.2. Experimental Process*

This study was conducted in three successive stages: transesterification, sulfonation, and purification. Figure 1 shows a schematic diagram of the production process.

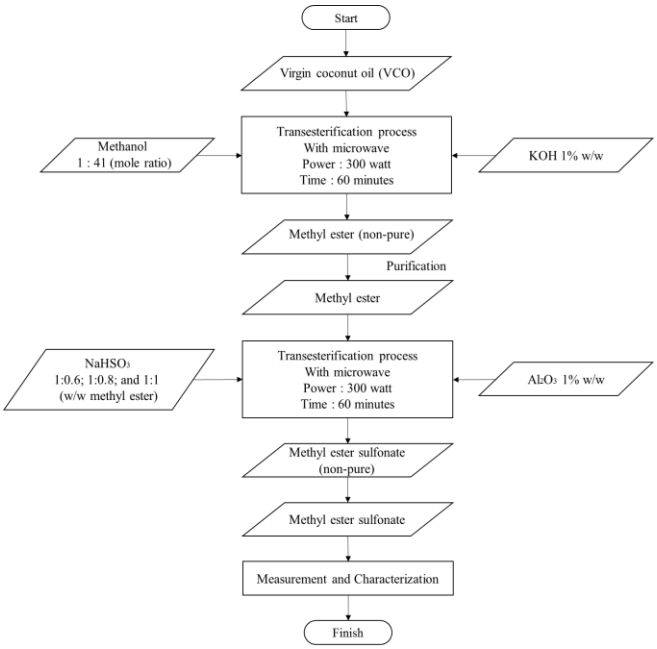


**Figure 1** Schematic diagram of MES production

#### 2.2.1. Transesterification

The transesterification process was carried out by reacting VCO with $CH_3OH$ with a mole ratio of 1:41 using a 1 wt% KOH catalyst with a transesterification time of 60 min at 300 W microwave power. Furthermore, the separation between methyl ester and glycerol,

which then forms the product of methyl ester, was purified by washing with warm water, and then allowed to stand for a few hours until the methyl ester separated from glycerol.

2.2.2. Sulfonation

The sulfonation of methyl ester was conducted by reacting the methyl ester obtained from the transesterification process with $NaHSO_3$ at varying reactant mole ratios of 1:0.6, 1:0.8, and 1:1 (w/w methyl ester) using a 1% $Al_2O_3$ catalyst (w/w methyl ester). The sulfonation process was conducted for 60 min under varying microwave power of 150, 300, 450, and 600 W.

2.2.3. Purification

The purification process was conducted to purify the resulting methyl ester sulfonate because it still contained di-salt, which can degrade the performance of the MES. Therefore, it was necessary to perform re-esterification by reacting the MES with 50% v/v methanol for 10 min using 150-W microwave power to remove the di-salt. Accordingly, distillation was conducted to evaporate the remaining methanol in the purification process.

*2.3. Measurement*

Conversion was calculated based on the mass balance of the MES, and physicochemical properties, such as viscosity, surface tension, pH, and density, were calculated using the Ostwald viscometer, Du-Nuoy Tensiometer, pH meter, and pycnometer, respectively.

*2.4. Characterization Product*

2.4.1. Gas Chromatography-Mass Spectrometry

A gas chromatography-mass spectrometry (GC-MS) analysis was performed by injecting 2 µL of the solution inside the inlet sample into the injection site with the merk of Agilent Technologies 7890B/5977B MSD.

2.4.2. Fourier-Transform Infrared Spectroscopy

A Thermo Scientific Fourier transform-infrared (FT-IR) spectrometer, Nicolet IS10, with a spectral range of 7800–350 $cm^{-1}$ was used to analyze functional groups of the surfactant (sample). One drop of the sample was dropped on the plate provided on the device, which was then pressed using a mechanical device. This pressure was maintained for several minutes, and then the plate was placed in the FT-IR sample holder.

## 3. Results and Discussion

*3.1. Characteristics of VCO*

The physical properties of the VCO used in this study (i.e., in terms of density, viscosity, and Free Fatty Acid (FFA) levels) are 0.91 g/mL, 28.82 cSt, and 0.01%, respectively.

Based on the data, VCO has an FFA level < 2%; thus, it can undergo the transesterification process directly without the need for an esterification process using a basic catalyst and without the saponification reaction (Oo et al., 2021).

*3.2. Transesterification*

The transesterification process is carried out by reacting the VCO with methanol at a ratio of 1:41 moles at 300 W of microwave power and 60 min of transesterification time. Then, the density, viscosity, and GC-MS analyses are performed to determine the characteristics of the methyl esters produced. The characteristics of the methyl esters are listed in Table 1.

**Table 1** Characteristics of methyl esters

| Parameters | Result | |
|---|---|---|
| | VCO | Methyl ester |
| Density (g/mL) | 0.91 | 0.86 |
| Kinematic Viscosity (cSt) | 28.82 | 3.01 |

Based on the comparison between the results of VCO and methyl ester, we observe decreases in the values of density and viscosity. This is because VCO still contains a large proportion of triglycerides, which is an arrangement of three fatty acids and glycerol. Through the transesterification process, the fatty acids are cut off and can react with methanol to form methyl esters, while glycerol breaks up and is discarded as a byproduct (Qadariyah, 2021). The density and kinematic viscosity values obtained are 0.86 g/mL and 3.01 cSt, respectively. These results agree with the ASTM D6751-2 standardization of methyl ester for density and kinematic viscosity values (0.87–0.90 g/mL and 1.9–6.0 cSt, respectively) (Kumar et al., 2020).

The composition of methyl esters can be determined using GC-MS analysis to identify the methyl ester components contained in the product. By identifying these components, their compositions are obtained relative to the total methyl ester. Figure 2 presents the chromatogram of the transesterified methyl ester product.

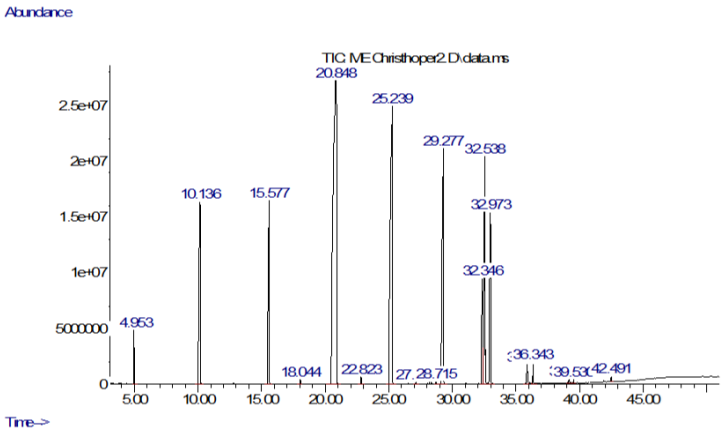


**Figure 2** Results of GC-MS analysis of methyl esters

Based on Figure 2, data collection is performed on the composition of fatty acid methyl ester, which is presented in Table 2.

**Table 2** Composition of methyl esters

| No | Methyl ester | Ret Time | Molecular weight(gr/mol) | Relative composition |
|---|---|---|---|---|
| 1 | Methyl laurate | 20.85 | 214.34 | 35.50% |
| 2 | Methyl Myristate | 25.24 | 242.40 | 19.15% |
| 3 | Methyl Oleate | 29.27 | 296.50 | 11.56% |
| 4 | Methyl Linoleate | 32.54 | 294.50 | 9.56% |
| 5 | Methyl Caprylate | 10.14 | 158.24 | 7.78% |
| 6 | Methyl Caprate | 15.58 | 186.29 | 7.34% |
| 7 | Methyl Stearate | 32.97 | 298.50 | 5.61% |
| 8 | Methyl Palmitate | 32.35 | 270.45 | 3.50% |
| | | Total | | 100% |

Table 2 shows that the most dominant compositions of fatty acid methyl esters are methyl laurate (35.50%) and methyl myristate (19.15%). By identifying the components contained in the methyl esters, their molecular weights can be estimated. The molecular weight of the methyl ester obtained is 229.13 gr/mol.

### *3.3. Sulfonation Process*

Sulfonation is the main process in MES manufacture, with the aim of converting methyl esters into MES using $NaHSO_3$ as the sulfonating agent. The analysis results can be described as follows:

#### 3.3.1. Viscosity

The resulting viscosity of the MES surfactant's was measured using an Ostwald viscometer. Figure 3 shows the effect of microwave power on surfactant viscosity at various reactant mole ratios.

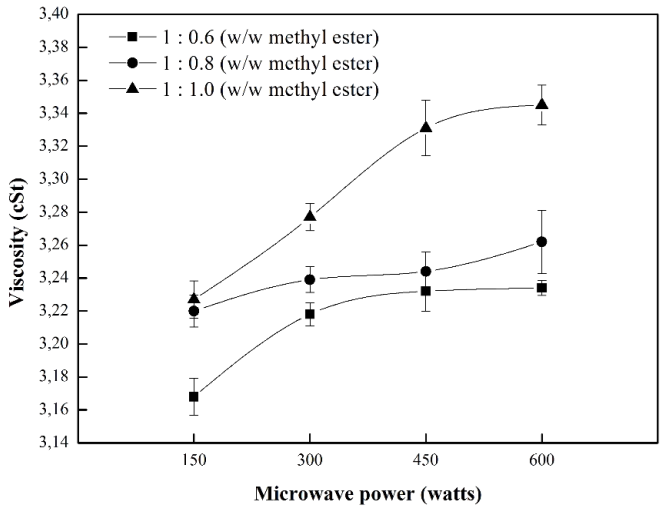


**Figure 3** Effect of microwave power on viscosity at various reactant mole ratios

The figure shows that the viscosity increases with the reactant ratio, with the highest viscosity recorded as 3.34 cSt, which occurs at 600 W of microwave power with a reactant mole ratio of 1:1, whereas the lowest viscosity is recorded as 3.17 cSt, which occurs at 150 W of microwave power with a reactant mole ratio of 1:0.6.

In addition, the higher the mole ratio of the reactants used, the higher is the viscosity value. An increased viscosity level suggests that during the sulfonation process, methyl ester is converted into MES.

The viscosity of a liquid is a fluid characteristic that is influenced by molecular size and intermolecular forces. The binding of sulfonate groups to methyl esters results in the surfactants having larger molecular sizes, so they have a higher viscosity than that of the raw material used for methyl esters. An increase in viscosity is also influenced by the reactant ratio and the sulfonation duration. The higher the reactant ratio and sulfonation duration, the greater is the likelihood of contact between substances, so the product has a high viscosity. This increase in viscosity is attributed to the binding of $SO_3H$ to the methyl ester structure.

#### 3.3.2. Surface Tension Analysis

The surface tension measurement of the MES surfactant is carried out using a Du-Nuoy tensiometer. Figure 4 shows the effect of microwave power on surfactant surface tension at various reactant mole ratios. According to the experimental results, the surface tension increases with the reactant mole ratio.

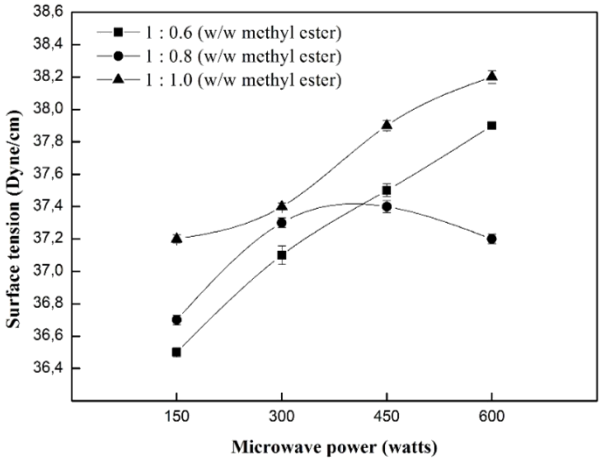


**Figure 4** Power effect on surfactant surface tension at various reactant mole ratios

As shown in the figure, the highest surface tension value is 38.2 dyne/cm, which is achieved at a reactant mole ratio of 1:1 at 600 W of microwave power. In contrast, the lowest surface tension value is 36.5 dyne/cm, which is obtained at the reactant mole ratio of 1:0.6 at 150 W of microwave power. The results clearly indicate that the increased surface tension due to the higher microwave power and reactant mole ratio used causes the formation of side-products other than the MES itself, which reduced the surface tension. These results agree with those reported by Hariani et al. (2016), who stated that the surface tension of MES ranges from approximately 30 to 40 dyne/cm.

3.3.3. pH Analysis

The pH measurements are carried out to determine the acidity of the MES surfactant produced in the sulfonation process. The resulting surfactant of methyl sulfonate is acidic, with pH values ranging from 4 to 5.04. Figure 5 shows the effect of microwave power on surfactant pH at various reactant mole ratios.

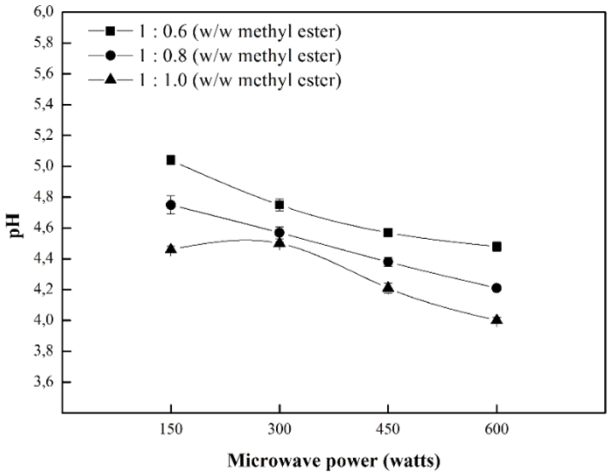


**Figure 5** Effect of microwave power on surfactant pH at various reactant mole ratios

The figure shows that the pH value obtained decreases with increasing mole ratio of reactants in the sulfonation process. The lowest pH value is 4, which is attained at a microwave power of 600 W and a mole ratio of 1:1. The highest pH is 5.04, which is achieved at a microwave power of 150 W and a mole ratio of 1:0.6. This acidic nature is attributed to

the H+ ion present in the $SO_3H$ group. The decrease in pH is attributed to the greater amount of $NaHSO_3$ used, thus increasing the possibility of the formation of sulfonate groups in the methyl ester reactant.

3.3.4 Density Analysis

Density is generally associated with viscosity, where the denser liquid tends to have a higher viscosity. In other words, the content of the material affects its density. The higher the microwave power and the reactant mole ratio used, the higher is the density of the resulting surfactant. Figure 6 shows the effect of microwave power on surfactant density at various reactant mole ratios.

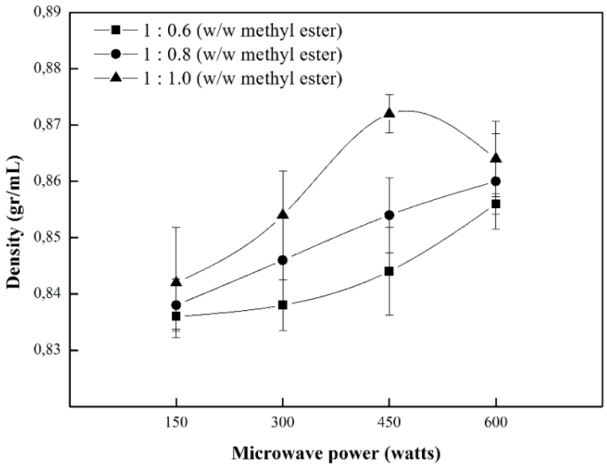


**Figure 6** Effect of microwave power on surfactant density at various reactant mole ratios

The measurement results show that the MES surfactant has density values ranging from 0.84 to 0.87 g/mL. As shown in Figure 6, the highest density is 0.87 g/mL, which is obtained at 450 W of microwave power with a reactant mole ratio of 1:1, whereas the lowest density is 0.84 g/mL, obtained at 150 W of microwave power with a reactant mole ratio of 1:0.6.

3.3.5. Analysis of FT-IR Spectroscopy

The sulfonate group analysis of the MES surfactant is carried out at a 1:1 mole ratio of reactants with 450 W of microwave power using FT-IR. According to Socrates (2001), the vibrational strain of the sulfonate group is detected at wavenumbers 1020–850 $cm^{-1}$ and 830–690 $cm^{-1}$. Figure 7 shows the FT-IR analysis result of MES at 450 W of microwave power and a reactant mole ratio of 1:1.

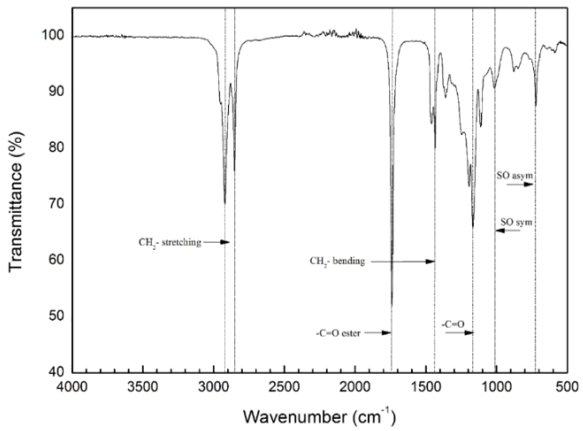


**Figure 7** FT-IR analysis results at 450 W of microwave power

As shown in Figure 7, the absorption band ν = 2921.75–2852.32 $cm^{-1}$ originates from $CH_2$ symmetric and asymmetric stretching vibrations. The $CH_2$ stretching vibrations indicate the presence of a linear alkane absorption band 1740.51 $cm^{-1}$ (-C=O stretching of ester), 1434.84 $cm^{-1}$ ($CH_2$ bending), 1164.84 $cm^{-1}$ of –C=O of carbonyl bond in unreacted methyl ester). This is attributable to the fact that the methyl ester groups are hydrolyzed by water and then become carboxylic groups. However, the carboxylic groups of fatty acids can be recovered by transesterification with excess anhydrous methanol and an acidic reagent. The vibrational strain of the sulfonate group is detected at peak values of 1014.42 and 722.24 $cm^{-1}$ with S-O symmetrical and S-O asymmetrical, respectively. This shows that the spectrum for the sulfonate group is observed around the wavenumber 1020–690 $cm^{-1}$. These results are in accordance with those obtained for the MES surfactant reported by Tulathammakit and Boonyarach (2014).

## 4. Conclusions

In this study, an MES surfactant was successfully developed from VCO using the $Al_2O_3$ catalyst through the sulfonation process using microwave radiation, which could reduce the sulfonation time. The MES production was affected by the reactant mole ratio and microwave power, where the optimum conditions included a reactant mole ratio of 1:1 and 450 W of microwave power with a viscosity of 3.33 cSt, density of 0.87 gr/cm, surface tension of 37.9 dyne/cm, and pH of 4.21. In addition, the results of the FT-IR analysis suggested that a sulfonate group was present in the sample at the absorption peak of ν = 1020–690 $cm^{-1}$. Moreover, in the last three years, 3050 papers have discussed this surfactant. This is due to the demerits of synthetic surfactants and the tendency of people to prioritize natural ingredients. Therefore, multidisciplinary research related to MES development is required to review the cost aspect so that a simpler method with a lower cost can be developed. In addition, it is necessary to further review the critical micelle concentration and hydrophilic–lipophilic balance to measure the strength balance of the hydrophilic and lipophilic groups of the surfactants formed. As a renewable and environmentally friendly bio-based anionic surfactant, substantial ongoing efforts are expected in the next few years to build green products and reduce the use of synthetic surfactants.

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