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Formation of Methyl Ester Sulfonate (MES) from Pure Coconut Oil Material as Surfactant

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A B S T R A C T

Research on the formation of methyl ester sulfonate from pure coconut oil as a surfactant raw material has been carried out. This research aims to study the optimum conditions for the transesterification reaction, as well as to obtain the optimum conditions for the sulfonation method in the synthesis of methyl ester sulfonate through variations in time and mole ratio. Where the initial stage of research goes through a transesterification process. Then observations were made of the products produced. The transesterification results showed that the optimum reaction conditions were observed at a time of 55 minutes, a stirring rotation of 500 rpm and a base catalyst amount of 2%. Under these conditions the yield of methyl ester laurate obtained was 60.81%. Meanwhile, the surfactant product, in this case methyl ester sulfonate, was identified at an absorption wave number of 1273; 365. and 1458.cm^{-1}.

Keywords: coconut oil, transesterification, methyl ester sulfonate, surfactan

1. INTRODUCTION

Surfactant is an organic compound, the molecule of which has at least one hydrophilic group and one hydrophobic group. The properties of surfactants, if added to a liquid, even at low concentrations, can reduce the surface and interfacial tension of the liquid (Watkins, 2001). Several applications of surfactants can be found in various industries, including the mining industry, paper industry, textiles, leather, rubber, plastics, metal industry, construction materials industry and many other applications (Hambali et al., 2013). The demand for surfactants throughout the world is increasing over time along with the increase in population and applications in daily life.

Various types of commercial surfactants are available on the market, but most commercial surfactants are made from petrochemical-based petroleum raw materials such as Linear Alkyl Benzene Sulfonate (LABS), where LABS has disadvantages because it cannot be renewed, is very expensive, cannot withstand high hardness and is not environmentally friendly. environment because it is difficult to degrade (Hidayati, 2009). Therefore, in this research, the use of raw materials from vegetable oil in making surfactants is very possible considering the availability of raw materials which is quite abundant, making it an alternative substitute for petrochemical-based surfactants. In this case, Methyl Ester Sulfonate (MES) will be made, which is an anionic surfactant based on vegetable oil, which contains C12 - C18 chain fatty acids, has more environmentally friendly properties and is biodegradable. And the production costs are also relatively cheaper compared to the production costs of petrochemical-based surfactants. Research on the manufacture of MES surfactants using crude palm oil (CPO) and palm kernel oil as raw materials was carried out by Hidayati et al (2012). Where this research places emphasis on temperature and reaction time on the performance of methyl ester sulfonate, from the results of this research it can be concluded that sulfonation of methyl ester carried out at temperatures above 120oC with a sulfonation time of more than 6 hours results in a decrease in the performance of the MES surfactant.

In this research, the manufacture of MES was carried out using pure coconut oil as raw material, where the process began with transesterification (methanolysis) to produce methyl ester. The resulting methyl ester is then subjected to a sulfonation process using sodium bisulfite, then purified with alcohol. The next stage is a neutralization process using sodium hydroxide solution. Information about the manufacture and characterization of methyl ester sulfonate surfactants from palm oil, both palm oil and palm kernel oil using long-chain methyl ester as raw materials, has been widely published. However, publications regarding the manufacture of methyl ester sulfonate from coconut raw materials, which generally have short to medium carbon chains as a surfactant, are still very limited. Based on this, it is necessary to conduct research to optimize sulfonation results, by varying the reaction time and mole ratio between methyl ester and sodium bisulfite in order to obtain methyl ester sulfonate with characteristics that qualify as a surfactant material for various applications.

2. MATERIALS AND METHODS

2.1 Tools and materials used

The tools used in this research are laboratory glassware including three-neck flasks, separating funnels, measuring cups, thermometers, Erlenmeyers, beakers, ball coolers (reflux coolers), dropper pipettes, glass stirrers, measuring flasks, and glass funnel. Other equipment besides glassware includes filter paper, aluminum foil, static rods, clamps, magnetic stirrer, bath, digital hot plate stirrer, stopwatch, digital analytical balance, pH meter. Several analytical instruments are also used, including: FT-IR, Gas Chromatography (GC),

The ingredients used in this research are pure coconut oil from Palu City, Methanol (CH_3OH) p.a. (E merck), Sodium Hydroxide (NaOH) p.a. (E merck), Hydrochloric Acid (HCl) p.a. (E merck), distilled water, $NaHSO_3$ (technical), Al_2O3 (technical) Na_2CO_3. (technical), Anhydrous Sodium Sulphate p.a. (E Merck), and Vaseline.

2.2 Methods

In this research, the optimum conditions used were variations of the 2% NaOH catalyst, a reaction time of 55 minutes and a rotation speed of 500 rpm based on the results of previous research, considering that these conditions can be used in industrial scale production. Where in these conditions the methyl ester product is produced which is dominated by methyl ester laurate. From these results, a sulfonation process is then carried out to obtain methyl ester sulfonate which is used in various applications in everyday life.

Preparation of Methyl Ester Sulfonate by Sulfonation Reaction

Methyl ester sulfonate is made by reacting the methyl ester with SO_3 from sodium bisulfite which is heated at a temperature of 60^oC and sulfonated in a reactor. In this research, variations in reaction times and reactant mole ratios were made. Variations in reaction time carried out were (3.5; 4.0; 4.5; 5.0; and 5.5) hours while variations in the mole ratio of sodium bisulfite to methyl ester were 1:1.2; 1:1.3 ; 1 :1.4 ; 1 : 1.5 and 1 : 1.6. . The process then continues with the addition of methanol to inhibit the formation of Na-salt (a by-product) and continues with the process of neutralizing the MES product by slowly adding NaOH solution until a neutral pH number is obtained.

3. Results and Discussion

Methyl ester sulfonate is made by adding methyl ester with sodium bisulfite which is heated at a temperature of 60^oC and sulfonated in a reactor. The result of sulfonation in the form of crude methyl ester sulfonate is then refluxed to separate the solid from the remaining $NaHSO_3$ and Al_2O_3 with MES solution. The process was then continued with the addition of methanol to inhibit the formation of Na-salt (a by-product) and continued with the process of neutralizing the MES product by slowly adding NaOH solution until a neutral pH value was obtained.

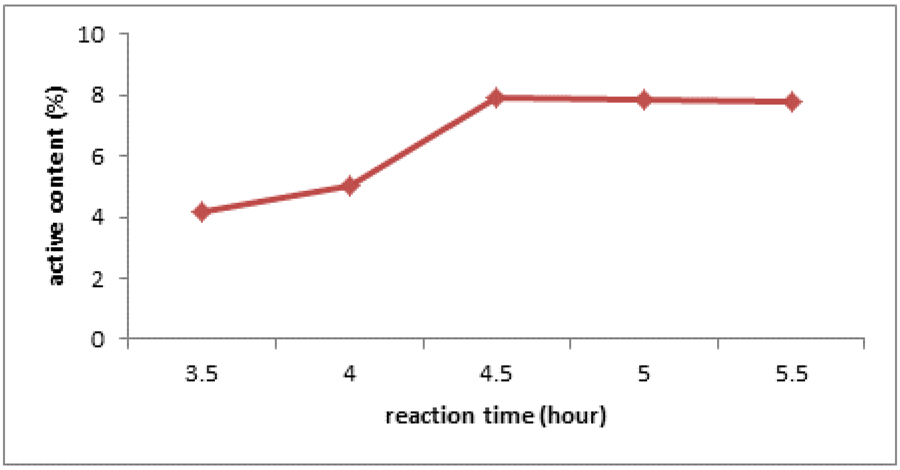

Figure 1. Effect of reaction time on the active ingredient content of MES

Active ingredients are one of the parameters that indicate the quality of a surfactant. This parameter has been applied to the surfactant industry, which is used to assess whether a type of surfactant has good performance or not. The higher the active ingredient value of a type of surfactant, the better its performance will be. Generally commercial surfactants are characterized by their active ingredient content. The results of the analysis of the active ingredient content in the MES surfactant samples by varying the reaction time and reaction temperature, obtained content data ranging from 4.16% to 7.38%. A recapitulation of the results of the analysis of the active ingredient content of MES resulting from the sulfonation process is presented in Figure 1 above.

The effect of increasing the reactant mole ratio (mole $NaHSO_3$) on the active ingredient content is that the increasing mole of NaHSO3 reactant added to ME causes the active ingredient content in the surfactant sample to also increase. As a result of the greater concentration of $NaHSO_3$, there is an excess of $NaHSO_3$ from the ideal mole ratio condition between NaHSO3 and methyl ester which triggers the formation of complex derivative products that have no charge, that is, $NaHSO_3$ does not bind to the molecule. The influence of the mole ratio on the active ingredient content of MES can be seen in Figure 2 below:

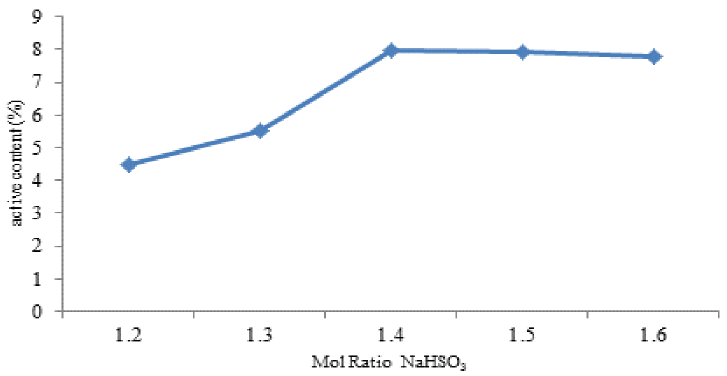

Figure 2. The influence of the mole ratio on the active ingredient content of MES

Figure 2 shows that the lowest active ingredient content in the MES sample occurred in reaction conditions with a $NaHSO_3$ reactant mole ratio (1:1.2) with a value of 4.50% and the highest active ingredient content in MES occurred at a reactant mole ratio (1:1.4). with an active ingredient value of 7.95%. The best active ingredient value obtained by Hidayati (2006) was 7.6%. The complex derivative products formed cause the possibility of the formation of sulfonate groups to be higher. This condition causes the cationic surfactant needed to neutralize the negative charge on the MES sample when measuring the active ingredient to be higher, after neutralization with NaOH solution the sulfonate groups are reduced. This condition causes the cationic surfactant needed to neutralize the negative charge on the MES sample when measuring the active ingredient to be lower, so that the measured active ingredient content of the MES sample becomes lower.

4. Conclusion

The results of the study of the conditions of the methyl ester sulfonate surfactant production process show that the best conditions are achieved with a reaction time of 4.5 hours, a reactant mole ratio of 1:1.4 and purification is carried out by adding methanol then neutralizing with NaOH solution until it reaches pH 7.

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