Effects of Operating Conditions on the Production of Sodium Stearoyl 2-Lactylate

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Abstract. Emulsifier is added in food products to maintain the shape of the food and stabilize it in an emulsion system. One of the emulsifiers commonly used in bread is sodium stearoyl-2-lactylate (SSL). SSL is the reaction product of stearic acid and lactic acid, with the addition of sodium hydroxide. This study aims to determine the effect of temperature, mole ratio, reaction time, and water vapor elimination in the manufacturing operation of SSL. The variations in this study include the composition ratio of stearic acid, lactic acid, and sodium hydroxide (2.2: 1: 1; 2.4: 1: 1; 2.6: 1: 1), as well as reaction conditions that include temperature (140-200 °C) and reaction time (3-5 hours). The analysis showed that the acid and saponification values of SSL were inversely related to the increase in temperature and reaction time. The acid and saponification values of the products for the reaction with a mole ratio of 2.6: 1: 1 (lactic acid: stearic acid: NaOH) at 180° C and the reaction time of 5 hours show the lowest acid and saponification values, which were 150.96 mg KOH / g and 246.41 mg KOH / g, respectively. The reaction conversion was inversely proportional to the acid number with a conversion range of 61.73% - 67.79%.

Keywords: emulsifier, stearic acid, lactic acid, SSL.

1. Introduction
Emulsifiers are one of the additives that are widely used in the chemical industry, one of which is the food industry. The role of emulsifiers is very important in the manufacture of certain food ingredients, for example bread, pasta, and other foods that require baking (bakery products) [1]. Apart from helping to improve the texture of food, this substance can also increase the shelf life of food because the emulsifier is able to maintain stability in an emulsion system. Sodium stearoyl-2 lactylate (SSL) is an anionic emulsifier that is widely used in the bakery industry [1] because of its ability to improve texture and increase the organoleptic value of taste and texture in general. Apart from that, SSL can also be used in the manufacture of cakes, pasta and noodles [2].

SSL can be made by mixing lactic acid, stearic acid, and sodium hydroxide [3]. Lactic acid comes from the fermentation of sugar substrates, such as sugar beet, while the stearic acid used in the manufacture of SSL is saturated fatty acids obtained from the processing of palm oil. The role of emulsifiers is broad and important makes many industrial sectors in Indonesia need emulsifiers in their processing, however this is not balanced with the production of emulsifiers by domestic industries due to various limitations. SSL is an example of an emulsifier that is still being imported today. Along with the increasing consumption of bread which has an impact on the growth of the bread and cake business, SSL manufacturing has high potential and usability if the operating conditions can be known.
2. Methods
SSL esterification was carried out using an experiment set up (Figure 1) with a reaction volume of 500 mL. SSL was made by mixing lactic acid in a liquid phase with commercial grade stearic acid. This mixture was stirred without heating, then NaOH is added. The esterification reaction was then carried out in a three-neck flask. Nitrogen was introduced during the reaction using a sparger to avoid oxidation of reactants and products, and to remove water. The variations in this study include the composition ratio of stearic acid, lactic acid, and sodium hydroxide (2.2: 1: 1; 2.4: 1: 1; 2.6: 1: 1), as well as reaction conditions that include temperature (140-200 ° C) and reaction time (3-5 hours). The final product obtained from this esterification was SSL powder. The sample was then analyzed for the acid number and saponification number. According to Franzke and Kroll [4], acid value and saponification value is the most important parameter of SSL.

![Image of experiment setup](image)

Figure 1 Experiment Setup for SSL esterification.

The procedure to determine acid number was started by weighing about 1 gram of the substance (SSL) in Erlenmeyer flask. Note the weight of the sample in gram (c). Heat until the substance has completely melted, cool; 25 mL of ethanol is added to Erlenmeyer flask. The solution will be refluxed for one hour, cool. Add 2-3 drops of phenolphthalein indicator to the flask. Titrate with potassium hydroxide (0.1 M), constantly shaking the contents of the flask until a pink color, which persists for 15 seconds, is obtained. Note the number of mL (a) required. As the next step, do another titration step in blank sample. Add 25 mL of ethanol to Erlenmeyer flask. Reflux for one hour, cool. Add 2-3 drops of phenolphthalein indicator to the flask. Titrate with potassium hydroxide (0.1 M), constantly shaking the contents of the flask until a pink color, which persists for 15 seconds, is obtained. Note the number of mL (b) required for blank sample. Calculate the acid value from the following formula:

\[
\text{Acid Value} = \frac{56.1 \times (a-b) \times N \text{ KOH}}{c}
\]  

(1)

The desired acid number for SSL products is within the range of 60-80 mg KOH/gr.

The procedure to determine saponification number is defined as two stages; titration with blank sample and titration with SSL sample. 25 mL of potassium hydroxide in 0.5 M ethanol are added to Erlenmeyer; add 2-3 drops of phenolphthalein. Add 0.5 M HCl is added to the burette. Titrate with hydrochloric acid (0.5 M), constantly shaking the contents of the flask until a clear color, which persists for 15 seconds, is obtained. Note the number of mL hydrochloric acid (a) required for the sample. Secondly, weigh about 10 grams of SSL in Erlenmeyer flask. Note the weight of the sample in gram (c). Add 25 ml of ethanolic potassium hydroxide (0.5 M); reflux for 1 hour; cool; add 2-3 drops of phenolphthalein. Titrate with hydrochloric acid (0.5 M), constantly shaking the contents of the flask until a clear color, which persists for 15 seconds, is obtained. Note the number of mL hydrochloric acid (b) required for the sample. Calculate the acid value from the following formula:

\[
\text{Saponification Value} = \frac{56.1 \times (a-b) \times N \text{ HCl}}{c}
\]  

(2)

3. Result and discussion
3.1 The Effect of Temperature Variation, Mole Ratio, and Time on Acid Number and Conversion

Based on Figures 2, 3, and 4, an increase in temperature causes a decrease in the acid number. This result occurs because the higher the reaction temperature, the lower the free fatty acid content produced. According to Srinivasan et al. [5], the SSL synthesis reaction takes place in an endothermic reversible state which shows better results at high temperatures. At temperature variations, the acid number values of the three figures with the lowest fixed variable mole ratio are at the reaction temperature of 180°C, while the lower temperature indicates a higher acid number. This is consistent with the theory of endothermic reactions. In an endothermic reaction, the higher the temperature, the reaction will shift towards the product, so that the product acid number is lower.

Figure 2 Acid value of SSL as a function of temperature and reaction time at a reactant mole ratio of 2.2:1:1.
The acid value obtained at a temperature of 140-180°C is not in accordance with the standard, namely 60-130 mg KOH/g sample (Commission Regulation (EU) No 231/2012)[6]. This is due to the presence of excess water in the product formation reaction. The water content comes from the initial stage reaction, namely the polymerization reaction of lactic acid into poly-lactides. The polymerization process that occurs is polycondensation, which is a reversible reaction. The presence of water in the reaction product mixture under certain conditions causes the reaction to shift towards the reactants. In
Figures 2, 3, and 4, the longer the reaction time, the lower the acid number. The longer reaction time allows greater contact between substances, so that the acid number obtained is lower.

3.2 The Effect of Temperature Variation, Mole Ratio, and Time on Saponification Value

Figures 5 to 7 show that the highest saponification value was achieved at a mole ratio of 2.4:1:1, which is consistent with the trend of acid values. This may occur because water hammering consistently happened in the flask of reactant mole ratio of 2.4:1:1. The hot plate temperature is lowered in that specific flask to avoid water hammering, resulting in anomaly in the data at mole ratio of 2.4:1:1.

Figures 5 to 7 also show that the saponification values of the SSL decreased with an increase in time and temperature. This trend is the same as the trend in the acid number, as shown previously. The saponification value may indicate the average length of the fatty acid chains. The lower the saponification value, the longer the chain the fatty acid has. A higher saponification value indicates that there are more fat and free fatty acids that can be soaped in the sample. The desired saponification value for SSL product is within the range of 190-230 mg KOH/gr. With the acid number, the saponification value is useful in providing information about the quantity of glyceride. The trend of the acid and the saponification values together indicates that in one gram of sample there is approximately the same quantity of glyceride.

![Figure 5](image.png)

**Figure 5** Saponification value as a function of reactant mole ratio at 140°C.
3.3 Order Reaction & Reaction Rate Constant Determination

In order to know whether the experiment fits certain reaction order, the experiment model is matched with zero, first, second order reaction model. In this case, the experiment model was based on the total lactic acid left in the product at third, fourth, and fifth hour of reaction. Based on the modelling of the reaction on Figure 8, the reaction order is 1, therefore reaction concentration affects reaction rate. The coefficient determination ($R^2$) of this experiment is 0.9969 ($R^2$ value is close to 1).
Table 1 shows that the temperature increases, the rate increases too. In first order reaction, reaction rate will increase as lactic acid concentration is increased. Reactant mole ratio should affect reaction rate, but the data shows that reaction rate lowered at mole ratio 2.4:1:1. The anomaly may happen due to water hammering that was already explained earlier.

![Graph](image.png)

**Figure 8** Modelling Result Comparison (For Reaction at 140°C with reactant mole ratio of 2.2:1:1).

**Table 1.** Reaction Constant at Every Temperature.

| Run | Temperature (°C) | Mole Reactant Ratio | Reaction rate constant (1/hour) |
|-----|------------------|---------------------|---------------------------------|
| 1   | 140              | 2.2:1:1             | 0.8092                          |
| 2   | 160              | 2.2:1:1             | 0.8169                          |
| 3   | 180              | 2.2:1:1             | 0.8364                          |
| 4   | 140              | 2.4:1:1             | 0.7136                          |
| 5   | 160              | 2.4:1:1             | 0.7935                          |
| 6   | 180              | 2.4:1:1             | 0.8195                          |
| 7   | 140              | 2.6:1:1             | 0.8364                          |
| 8   | 160              | 2.6:1:1             | 0.8417                          |
| 9   | 180              | 2.6:1:1             | 0.8543                          |

**3.4 The Effect of Temperature on the Colour of the Product**

Table 2 shows the effect of reaction condition on the colour of the SSL. Total colour difference (ΔE*) was obtained from the calculation based on the L*, a*, and b* values of the sample. The higher ΔE*, the higher the total color difference between the sample and the standard. Table 1 shows that the reaction temperature significantly affects the colour of the product. It was found that the ΔE* increases as an increase in the temperature. The product obtained at 140°C was generally has clear white in colour, while the product obtained at 160°C was generally yellowish in colour. In addition, the product obtained at 180°C had a slight brown colour.
Table 2. Total color difference for SSL as a function of reaction time, reactant mole ratio, and temperature.

| Mole Ratio | Duration (hours) | $\Delta E^*$ |
|------------|-----------------|-------------|
|            | 140°C | 160°C | 180°C |
| 2.2:1:1    | 3     | 1.49  | 2.14  | 11.17 |
|            | 4     | 1.84  | 2.77  | 12.13 |
|            | 5     | 1.06  | 2.33  | 11.15 |
| 2.4:1:1    | 3     | 1.17  | 2.06  | 16.49 |
|            | 4     | 0     | 3.25  | 17.34 |
|            | 5     | 1.75  | 4.66  | 22.81 |
| 2.6:1:1    | 3     | 1.44  | 4.47  | 15.15 |
|            | 4     | 1     | 2.73  | 15.28 |
|            | 5     | 1.54  | 2.21  | 14.95 |

4. Conclusions
In this study, the operating conditions of the SSL production has been evaluated. It was found that the higher the reaction temperature, the lower the acid and saponification values, which indicates the higher the reaction conversion. The increase in temperature also affects the total colour difference of the product. In addition, the longer the reaction time, the acid and saponification values. It was also found that the mole ratio affects the reaction rate of SSL formation because the SSL synthesis reaction follows a first-order reaction.

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