

# Production of Sodium Lignosulfonate (SLS) Surfactant from Oil Palm Empty Fruit Bunches (OPEFB) Using Microwave

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**Abstract.** The aim of this study was to investigate the effect of using a microwave in synthesizing Sodium Lignosulfonate (SLS) surfactant made of Oil Palm Empty Fruit Bunches (OPEFB) which contains lignin as the content to be processed, especially in the sulfonation process. The observed variables are the NaHSO<sub>3</sub> concentration ranging from 0.1 M to 0.7 M, reaction time ranging from 30 minutes to 90 minutes, and microwave power ranging from 300 W to 750 W. on the yield and comparing the results of SLS synthesized with microwave to those obtained through conventional heating methods in the sulfonation process. The process encompassed several steps, including drying, milling, delignification, lignin isolation, and sulfonation using microwaves. Afterward, the SLS results were subjected to analysis through FTIR and HLB testing. The findings revealed a positive correlation between NaHSO<sub>3</sub> concentration, reaction time, microwave power, and the yield of SLS Product, with the optimal conditions being at the NaHSO<sub>3</sub> concentration of 0.7 M, microwave power of 600 W, and reaction time of 75 minutes. Moreover, the microwave method demonstrated greater efficiency in increasing the SLS Product yield by up to 24% compared to the conventional method.

## 1 Introduction

Surfactants are organic compounds characterized by having at least one hydrophilic (water-attracting) group and one hydrophobic (water-repelling) group. These molecules consist of a head and a tail. The head is hydrophilic, highly polar, and water-loving, while the tail is hydrophobic, nonpolar, and water-repellent. Surfactants find widespread applications as essential additives in various industries, including soap and detergent manufacturing, textiles, rubber and plastics, cosmetics, food processing, gypsum board adhesive, construction materials dispersion, and many more [1]. Given the extensive demand and utility of surfactants across industries, they hold high commercial value. The escalating demand for surfactants in Indonesia is intricately intertwined with the burgeoning detergent industry and the ongoing construction boom. Current statistics from the Indonesian Institute of Sciences (LIPI) indicate that Indonesia's annual surfactant consumption stands at approximately 95,000 tons, while domestic production capacity hovers around 55,000 tons, leaving a substantial gap that necessitates the importation of about 44,500 tons of surfactants as of 2006. Simultaneously, the surging pace of infrastructure development, spanning roads, bridges, housing, and edifices, has driven the need for construction materials, with concrete

being a primary choice. In this context, surfactants play a pivotal role as additives, particularly Sodium Lignosulfonate, used to enhance concrete properties.

Sodium Lignosulfonate (SLS) is an anionic surfactant with hydrocarbon groups in its tail region. This structural feature enhances the hydrophilic properties of SLS, making it readily soluble in water. As per ASTM standard C 494-79 (spec. for water reducing admixture for concrete), SLS is classified as a chemical belonging to the category of water reducing admixtures (WRA) or plasticizers. Global production of lignosulfonate salts has reached an estimated 965,000 tons annually, with nearly half dedicated to serving as dispersing agents in gypsum and cement paste [2]. Astonishingly, Indonesia remains entirely reliant on imports for lignosulfonate, primarily sourced from countries like Finland and Scandinavia, according to PT Fosroc-Indonesia (2005). This underscores the pressing need for local lignosulfonate production. One of the raw materials that can be used for the production of SLS is the waste from oil palm empty fruit bunches (OPEFB) obtained from oil palm trees.

Oil palm trees (*elaeis guineensis jacq.*) belong to the Gramineae plant family, monocotyledonae class, araceae family, and cocoideae order. The most crucial part of the oil palm tree is the Fresh Fruit Bunch (FFB). Each bunch contains 62-70% palm fruit, the source of palm oil production, while the remainder comprises the Oil Palm Empty Fruit Bunches (OPEFB), accounting for 23-30%. In the palm oil production process, OPEFB becomes a substantial solid waste. The primary components of OPEFB are cellulose, hemicellulose, and lignin, categorizing OPEFB as lignocellulosic waste [3].

Remarkably, prior research in Sodium Lignosulfonate production from OPEFB, as evidenced by studies conducted by [4] and [5], predominantly leaned on conventional heating methods, often entailing extended processing durations. The novelty of our research stems from embracing microwave-assisted heating, a departure from conventional conduction and convection-based methods. Microwave heating directly irradiates heat into the solution, bypassing intermediary processes, and is noted for its potential to curtail reaction times, conserve energy, reduce costs, and minimize environmental footprints [6]. Consequently, this study strives to manufacture Sodium Lignosulfonate surfactant from Oil Palm Empty Fruit Bunches (OPEFB) through microwave-assisted heating, delving into how microwave utilization influences Sodium Lignosulfonate (SLS) surfactant production via sulfonation. It scrutinizes variables such as  $\text{NaHSO}_3$  concentration, reaction time, and microwave power, culminating in a comparative analysis between the microwave approach and the conventional heating method.

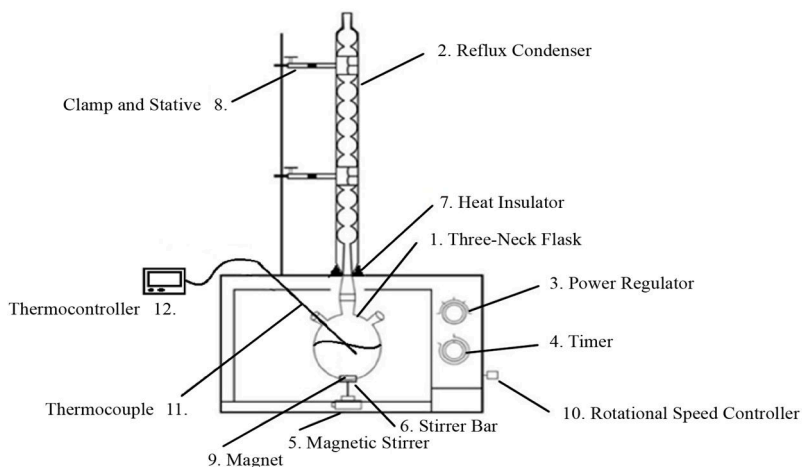
## 2 Material and Methods

### 2.1 Material

The raw materials used in this study are Oil Palm Empty Fruit Bunches (OPEFB), sodium hydroxide ( $\text{NaOH}$ ) technical, sulfuric acid ( $\text{H}_2\text{SO}_4$ ) technical, sodium bisulfite ( $\text{NaHSO}_3$ ) technical, methanol ( $\text{CH}_3\text{OH}$ ) technical, and aquadest ( $\text{H}_2\text{O}$ ).

### 2.2 Equipments

The equipment used is a batch reactor in the form of a 1 liter three-neck flask, a thermocouple and temperature controller, a reflux condenser, and Electrolux microwave EMM2308X with a range power of 150 watt – 750 watt.

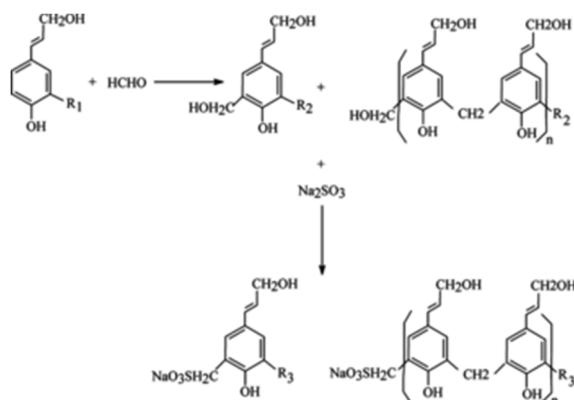


**Fig. 1.** Equipment scheme.

### 2.3 Delignification

The delignification process is carried out to extract black liquor with a high lignin content from the raw material, oil palm empty fruit bunches (OPEFB). Initially, OPEFB is dried by exposure to sunlight for approximately 3 days and then ground using a grinder until the size is  $< 0.5$  mm. The delignification process involves adding 10% NaOH to the OPEFB at a temperature of  $90^{\circ}\text{C}$  for 80 minutes, resulting in the formation of black liquor and pulp. The pulp is then filtered, and the black liquor is separated and collected.

### 2.4 Sulfonation



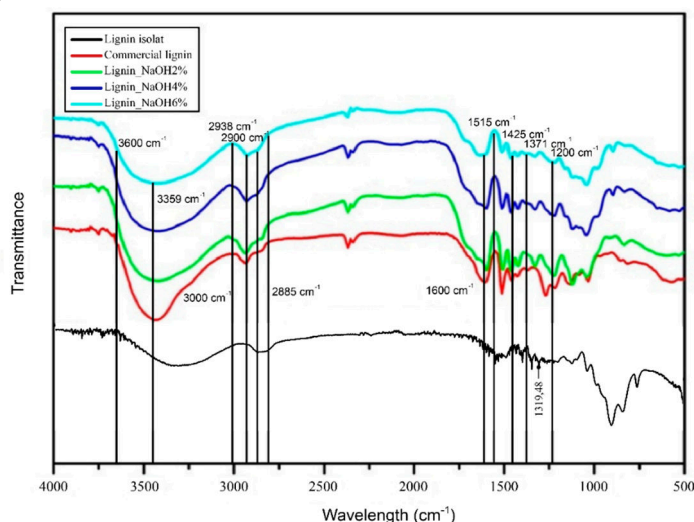
**Fig. 2.** Sulfonation reaction of alkali lignin [7].

The sulfonation process involves mixing the lignin precipitate with 250 ml of  $\text{NaHSO}_3$  at predetermined molarities. This mixture is then heated using a microwave to a temperature of  $90^{\circ}\text{C}$ , with power and time settings according to the specified variables, resulting in the formation of an SLS solution. The hydroxymethylation and the subsequent sulfonation reaction of alkali lignin from research conducted by [7] are schematically shown in Fig. 2.

In Fig. 2, condensed lignin was degraded into lower polymeric degree of lignin fragments by oxidation, producing more active site to react with formaldehyde, which further produces more active hydroxymethyl groups for sulfonation [7].

### 3 Results and Discussion

The delignification and isolation process yielded an average dry lignin isolate yield of 30% from the TKKS raw material used. The obtained lignin isolate from the Lignin Isolation process was then subjected to FTIR testing to analyze the functional groups contained within it. This was done to ensure that the product from the previous process was indeed lignin. The FTIR analysis results of the lignin isolate, when compared with the literature by [8], are shown in Fig. 3.

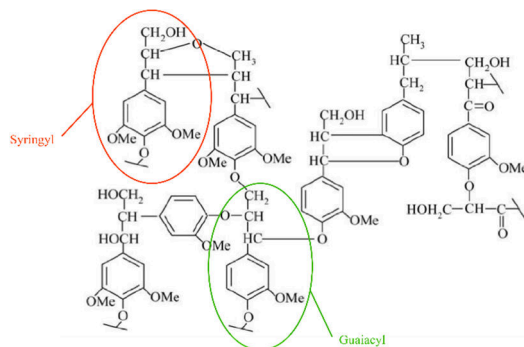


**Fig. 3.** FTIR analysis results of lignin isolate.

**Table 1.** FTIR spectrum reading on lignin isolate.

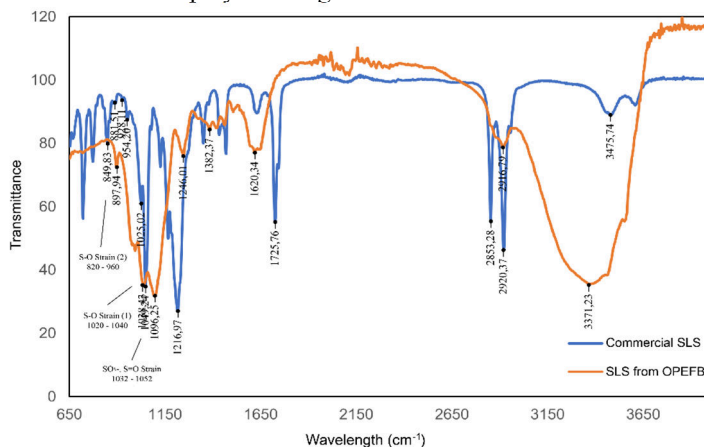
Absorption Band Range Standard (cm <sup>-1</sup> )	OPEFB Lignin Isolate Samples (cm <sup>-1</sup> )	Functional Groups
1330 – 1325	1319,48	Syringil ring vibrations [9]
1316 – 1336	1319,48	Guaiasil ring vibrations [10]

The oil palm used as the raw material for this lignin isolate is an angiosperm plant and falls into the hardwood category, according to [11]. Thus, lignin consists of guaiacyl and syringyl units in certain proportions, depending on the type of wood, wood age, growth location, and climate. Based on Fig. 3, it can be seen that the FTIR analysis of the lignin isolate in this study bears a resemblance to both commercial lignin and the lignin resulting from [8]. As indicated in Table 1, absorption in the region of 1319.48 cm<sup>-1</sup> suggests the presence of guaiacyl ring vibrations [10] and syringyl ring vibrations [9]. Fig. 4 provides an example of the lignin structure found in nature, which contains both syringyl and guaiacyl groups.



**Fig. 4.** Partial lignin structure [12].

Subsequently, the lignin isolate obtained through the delignification and lignin isolation processes was reacted with  $\text{NaHSO}_3$  to undergo the sulfonation process. The sulfonation process resulted in SLS products, which were then subjected to FTIR analysis. The FTIR analysis of the Sodium Lignosulfonate aimed to determine the presence or absence of sulfonate groups formed after the sulfonation reaction. The results of the FTIR analysis for sodium lignosulfonate are displayed in Fig. 5.

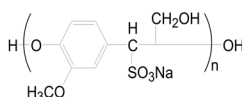


**Fig. 5.** FTIR testing results of Sodium Lignosulfonate (SLS) product.

**Table 2.** FTIR Spectrum Reading of Sodium Lignosulfonate Product.

Functional Groups	Standard Wavelength Range ( $\text{cm}^{-1}$ )	Wavelength ( $\text{cm}^{-1}$ )
$\text{SO}_3^-$ , S=O strain	1032-1052 [13]	1038,43
S-O strain (1)	1020-1040 [5]	1038,43
S-O strain (2)	820-960 [5]	897,94

According to [14], sulfonate groups form at a wavelength of  $1043 \text{ cm}^{-1}$ , which aligns with this study, where peaks were detected at a wavelength of  $1038.43 \text{ cm}^{-1}$ , indicating the presence of sulfonate groups. Additionally, according to [5], sulfonate groups can be detected by the presence of  $\text{SO}_3^-$ , asymmetric stretching of S=O, and S-O stretching in the infrared spectra. The structural diagram of sodium lignosulfonate is illustrated in Fig. 6, where sulfonate groups are indicated by the presence of  $\text{SO}_3^-$  bound to  $\text{Na}^+$ .

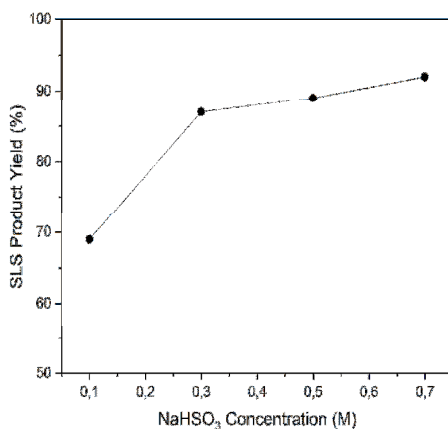


**Fig. 6.** Sodium Lignosulfonate structure [11].

### 3.1 The Effect of $\text{NaHSO}_3$ Reactant Concentration on Sodium Lignosulfonate Yield

The effect of  $\text{NaHSO}_3$  concentration was observed at the power level that has the highest yield, which was 600 W. The concentrations used were 0.1 M, 0.3 M, 0.5 M, and 0.7 M. As shown in Fig. 7, the curve on the graph indicate that the yield tends to increase with the rising concentration of the reactant.

The lowest concentration, which is 0.1 M, resulted in the lowest yield, which was 69%. Subsequently, as the reactant concentration increased to 0.3 M, the SLS yield also increased significantly, reaching 87%. When the concentration was further raised to 0.5 M, the yield showed a minor increase, resulting in an 89% SLS yield. Then, with a concentration of 0.7 M, the SLS yield experienced another increase, reaching the highest yield of 92%.

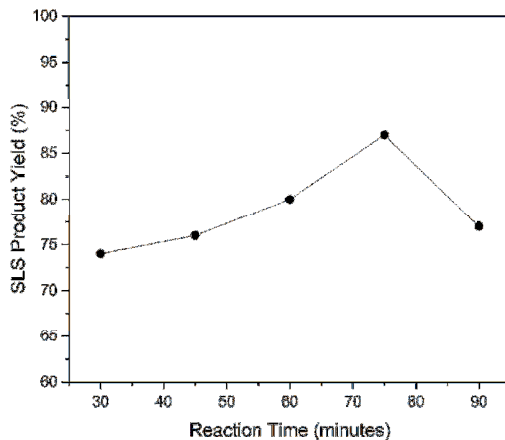


**Fig. 7.** Effect of  $\text{NaHSO}_3$  concentration on SLS product yield at 90°C and 600 W.

From Fig. 7, it can be observed that an increase in reactant concentration leads to a higher yield. In this study, the highest yield achieved was 92% with the highest  $\text{NaHSO}_3$  concentration of 0.7 M. These results align with a study conducted by [4] regarding the influence of  $\text{NaHSO}_3$  concentration on sodium lignosulfonate yield, with concentration variations of 0.1 M, 0.3 M, and 0.5 M. The highest yield was obtained at a  $\text{NaHSO}_3$  concentration of 0.5 M, with an SLS yield of 51.87%. The differences in yield between this study and the one conducted by [4] can be attributed to several factors, including the use of a 98% concentration in acid precipitation, whereas using concentrations of sulfuric acid greater than 20% results in lower yields and reduced lignin isolate purity due to the degradation of non-lignin components and excessive condensation reactions [14]. Additionally, the use of microwaves also contributes to the increased yield [6]. In this study, the highest molarity used was 0.7 M, which still showed an increase in yield, whereas in [4], the highest molarity was only up to 0.5 M.

### 3.2 The Effect of Reaction Time on Sodium Lignosulfonate Yield

The effect of reaction time was observed at the power level that has the highest yield, which was 600 W. The variations in reaction time used were 30 minutes, 45 minutes, 60 minutes, 75 minutes, and 90 minutes.



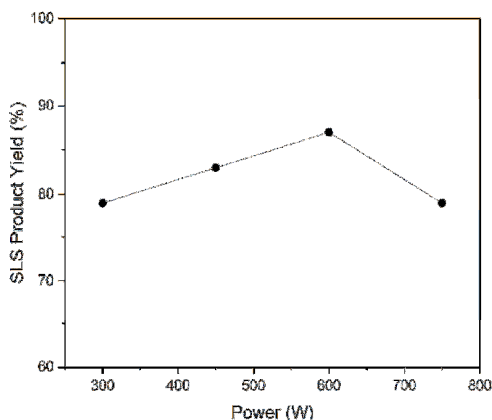
**Fig. 8.** Effect of reaction time on SLS product yield at 90°C and 600 W.

As shown in Fig. 8, when reacted for 30 minutes, the SLS yield obtained was 74%. Subsequently, when the reaction time was extended to 45 minutes, the SLS yield increased to 76%. When the reaction time was further increased to 60 minutes, the resulting SLS yield reached 80%. When the reaction time was further extended to 75 minutes, the SLS yield continued to increase, reaching 87%, which was the highest yield achieved at the 600 W power level. However, when the reaction was allowed to run for 90 minutes, the SLS yield decreased to 77%.

From Fig. 8, it can be observed that the longer the reaction time, the greater the yield obtained. This occurs because with increased reaction time, there is a higher likelihood of contact between the reactants and their bonding to form sodium lignosulfonate products, thus leading to a higher yield [6]. However, from the results of the research conducted, it can be observed that at 90 minutes of reaction time, there was a decrease in yield following an increase at 75 minutes of reaction time. This can be attributed to the fact that with longer reaction times, some of the already-formed products may undergo degradation due to the continued heating or reflux processes [5].

### 3.3 The Effect of Microwave Power on Sodium Lignosulfonate Yield

The Effect of Microwave Power on SLS Yield was observed at a reaction time of 75 minutes, selected based on the reaction time that has the highest yield, namely 75 minutes. The variations in microwave power used were 300 W, 450 W, 600 W, and 750 W.



**Fig. 9.** Effect of power on SLS product yield at 75 minutes reaction time.

As shown in Fig. 9, the curve indicates that the yield tends to increase with increasing power. In the study, at 300 W, an SLS yield of 79% was obtained. When the power was increased to 450 W, the resulting SLS yield increased to 83%. Then, as the power was raised to 600 W, the SLS yield continued to increase to 87%. However, when the power was increased to 750 W, the SLS yield decreased to 79%.

From Fig. 9, it can be seen that an increase in power tends to result in higher yield. This aligns with research conducted by [15] on Biodiesel Production from Coconut Oil with Base Catalyst via Transesterification Process Using Microwaves, where higher microwave power resulted in higher product yields. The similarity between these two studies lies in the use of microwave heating due to the presence of microwaves and polar molecules in the system, resulting in vibrations that are not dependent on reaction temperature [16]. However, at 750 W, a decrease was observed, indicating that increasing power is not an effective way to achieve higher sodium lignosulfonate yields.

### 3.4 Comparison of Sodium Lignosulfonate Product Yields from Conventional Heating Method and Microwave Heating Method

The production of lignosulfonate surfactants using conventional methods involves heating with a heating mantle, the addition of a reflux condenser, and a thermometer to control temperature. In line with the defined research scope, the study focused on the influence of the sulfonation process, so conventional heating was only performed during sulfonation. The operating conditions used for this sample were a  $\text{NaHSO}_3$  concentration of 0.7 M and a reaction time of 75 minutes. This was because the SLS produced using the conventional method would be compared to the highest yield from samples using microwave heating.

Sodium lignosulfonate produced using microwave heating yielded the highest, at 92%, under the operating conditions of a  $\text{NaHSO}_3$  concentration of 0.7 M, a reaction time of 75 minutes, and a microwave power of 600 W. Meanwhile, using conventional heating, the yield of sodium lignosulfonate obtained was 74%. This aligns with research conducted by [6], stating that microwave heating can increase reaction rates, yields, and product purity compared to conventional heating methods. This can be attributed to local temperature and pressure, as well as microwaves absorbing character to penetrate materials, thereby producing distributed volumetric heat sources [17].



### 3.5 Analysis of HLB (Hydrophilic-Lipophilic Balance) of Sodium Lignosulfonate Surfactant

Sodium lignosulfonate surfactant is one type of ionic surfactant. Ionic surfactants do not follow the weight percentage HLB (Hydrophilic-Lipophilic Balance) base, even though the hydrophilic part of sodium lignosulfonate surfactant has a low molecular weight, the presence of ionization provides excessive emphasis, making the product more hydrophilic. Therefore, the HLB value of sodium lignosulfonate surfactant must be determined using experimental methods [18].

The determination of HLB using experimental methods consists of mixing a surfactant with an unknown HLB in varying ratios with a surfactant with a known HLB, and then using this mixture to emulsify oil to the required HLB value, which is already known. The mixture with the best performance is assumed to have the same HLB value as the required HLB value of the oil used, thus allowing the HLB value of the unknown surfactant to be calculated. In practice, a large number of experimental surfactants must be prepared, and the average HLB value of the unknown can eventually be calculated. Such a procedure can be challenging and time-consuming, so HLB determination can be done using the water-solubility method, which in many cases is considered quite reliable [18].



**Fig. 10.** HLB testing results using water solubility.

**Table 3.** HLB by Dispersibility [18]

HLB by Dispersibility	HLB range
No dispersibility in water	1-4
Poor dispersion	3-6
Milky dispersion after vigorous agitation	6-8
Stable milky dispersion	8-10
Translucent to clear dispersion	10-13
Clear solution	13+

From Fig. 10, the water solubility of the sample indicates that it can dissolve in water, disperse well, and transmit light, approaching clarity. Therefore, in accordance with Table 3, the produced SLS has an HLB range of 10-13. This aligns with research conducted by [19] on the synthesis of sodium lignosulfonate from bagasse. The HLB analysis conducted by [19] obtained an HLB value of 11.62, which also falls within the range of this research, namely 10-13.

### 3.6 Physical Properties of Sodium Lignosulfonate Surfactant Products

The sodium lignosulfonate surfactant produced from each variable was then observed for its color to determine the result that closely resembles commercial sodium lignosulfonate.

**Table 4.** Physical properties of Sodium Lignosulfonate with 450 W power, 0.3 M NaHSO<sub>3</sub> concentration, and reaction times of 30, 45, 60, 75, and 90 minutes.

Reaction Time (minutes)	Power (W)	NaHSO <sub>3</sub> Concentration	Physical Properties
30	450	0.3 M	Has a pale grayish-brown color
45			Has a bright brown color
60			Has a pale grayish-brown color
75			Has a dark brown color similar to commercial SLS
90			Has a dark brownish-black color

**Table 5.** Physical properties of Sodium Lignosulfonate with 75 minutes reaction time, 0.3 M NaHSO<sub>3</sub> concentration, and power levels of 300, 450, 600, and 750 Watts.

Power (W)	Reaction time (minutes)	NaHSO <sub>3</sub> Concentration	Physical Properties
300	450	0.3 M	Has a light brownish color
450			Has a dark brown color similar to commercial SLS
600			Has a slightly darker brown color than commercial SLS
750			Has a dark-brownish black color

**Table 6.** Physical properties of Sodium Lignosulfonate with 600 W Power, 75 minutes reaction time, and NaHSO<sub>3</sub> concentrations of 0.1, 0.3, 0.5, and 0.7 M.

NaHSO <sub>3</sub> Concentration	Power (W)	Reaction time (minutes)	Physical Properties
0.1	600	75	Has a pale grayish-brown color
0.2			Has a slightly darker brown color than commercial SLS
0.3			Has a dark brown color
0.4			Has a light brown color

From Table 4 to Table 6, the sample that produced a color closest to commercial SLS was the sample with a 75 minutes reaction time, 450 watt power, and 0.3 M NaHSO<sub>3</sub> concentration, as shown in Fig. 12. Meanwhile, the properties of the produced SLS were compared to the properties of SLS in the National Indonesian Standard (Standard Nasional Indonesia or SNI) and are presented in Table 7.

**Table 7.** Properties of the SLS Product

Properties	SLS Product from OPEFB	Indonesian National Standard [20]
Density	1,25 g/ml	1,5 g/ml
pH	6	6-7
Water Content	6,4%	5%
Water Insolubility	0,34%	< 0,5%



**Fig. 11.** Commercial Sodium Lignosulfonate



**Fig. 12.** Sodium Lignosulfonate with 75 Minutes Reaction Time, 450 W Power, and 0.3 M NaHSO<sub>3</sub> Concentration

## 4. Conclusion

Production of surfactant sodium lignosulfonate from oil palm empty fruit bunches (OPEFB) was studied using microwave at reactant concentration range of 0.1-0.3 M, time reaction range of 30-90 minutes, power microwave range of 300-750 W. The concentration of the reactant NaHSO<sub>3</sub> on the yield of sodium lignosulfonate has a relationship; the higher the concentration of NaHSO<sub>3</sub> used, the higher yield has an increasing trend. Meanwhile, the effect of reaction time on sodium lignosulfonate yield is the higher the reaction time used, the higher the tendency of the resulting yield. Meanwhile, the effect of microwave power on the yield of sodium lignosulfonate is the higher the microwave power used, the resulting yield tends to be higher too. The best yield is obtained on NaHSO<sub>3</sub> concentration 0.7 M, microwave power 600 W, reaction time 75 minutes. The yield of sodium lignosulfonate was obtained with conventional method (74%) lower than sodium lignosulfonate yield using the microwave method (92%), so it was concluded that the microwave method can increase sodium lignosulfonate yield up to 24%.

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