

Production of Sodium Lignosulfonate Surfactant from Oil Palm Empty Fruit Bunches Lignin using Ultrasound-assisted Method

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Abstract. Global demand for surfactants is increasing, but petroleum-based materials still dominate the market. Given the environmental factors involved, the use of biomass-based surfactants is becoming increasingly popular, with lignin from oil palm empty fruit bunches as one example. However, the process still relies on conventional methods that are time-consuming and inefficient. An ultrasound-assisted intensification approach can reduce processing time. Therefore, this study aims to investigate the production of sodium lignosulfonate surfactants from oil palm empty fruit bunches via delignification and sulfonation using an ultrasonic homogenizer, and to analyze the effects of reaction time and ultrasonic power on the surfactant's characteristics. Lignin was first isolated via alkaline delignification at 70°C for 60 min, followed by acid precipitation with 98% sulfuric acid at pH 2. The sulfonation process was carried out in a batch reactor at 90°C and atmospheric pressure in an aqueous solution with an initial pH of 4–5, using ultrasonic power of 210–297 W and reaction times of 50–90 min. The results showed that lignin composition by precipitation was 89.16%, with the highest sodium lignosulfonate surfactant yield of 57.14% at 297 W and 70 min during sulfonation. The substance has strong hydrophilic properties and potential as an emulsifier, as evidenced by its dark brown color, pH of 6.6, and HLB value of 10–13.

Keywords: Lignin, Oil Palm Empty Fruit Bunches, Sodium Lignosulfonate, Ultrasound

INTRODUCTION

Global demand for surfactants is increasing sharply due to their widespread use across sectors, such as healthcare, food, biotechnology, pharmaceuticals, and cosmetics (Zargar and Srivastava, 2023). This demand is projected to increase to \$81.7 billion by 2030 (Zargar and Srivastava, 2023).

Asia accounts for the largest share of global surfactant consumption (Branna, 2012).

This increasing demand for surfactants is still dominated by oleum-based or petrochemical-based surfactants such as ethoxylated alcohols (as cleaning agents in detergents), nonylphenol ethoxylates (industrial applications) (Canter, 2004), polyethylene glycol esters (emulsifiers), all of

which are derived from petroleum (Reznik *et al.*, 2010), and sulfonated aromatic compounds (synthesized from aromatic hydrocarbons) (Nikitchenko, 2014). However, surfactants based on these materials pose significant environmental challenges due to high CO₂ emissions and poor biodegradability (Tesser *et al.*, 2020; Veeramanoharan and Kim, 2024). To address this issue, increasing attention is being given to the development of surfactants from sustainable raw materials such as animal oils, palm oil, and other renewable resources (Qadariyah *et al.*, 2024b). Surfactants derived from oleochemicals exhibit lower toxicity, greater biodegradability, and improved stability under extreme conditions (Aqif *et al.*, 2024; Lopes *et al.*, 2019; Marcelino *et al.*, 2020).

Among the available biomass raw materials, oil palm empty fruit bunches are a very promising candidate because they are a waste product that has not been fully utilized (Akbar *et al.*, 2024; Suhartini *et al.*, 2022), yet have a very high lignin content of up to 25% (Sugiwati *et al.*, 2021). Lignin, the main component of OPEFB, has a complex aromatic structure rich in hydroxyl and phenolic functional groups, enabling its chemical modification into amphiphilic compounds (Ibrahim *et al.*, 2012; Putra *et al.*, 2024). One of the most widely developed forms of modification is the conversion of lignin into sodium lignosulfonate (Li *et al.*, 2024; Mu'izzah *et al.*, 2023), which is commonly used as a polymer electrolyte membrane, oil-water separation material, and waterproof coating due to its thermal stability and superior hydrophobic properties (Li *et al.*, 2024). This anionic surfactant exhibits good dispersion and emulsification, properties, making it a viable alternative to petrochemical surfactants (Xia *et al.*, 2024).

In general, the production of sodium lignosulfonate involves three main stages, namely pretreatment, precipitation, and sulfonation (Thungphotrakul *et al.*, 2019). The pretreatment stage is carried out to break down the lignocellulose structure and facilitate the release of lignin from the biomass matrix. This is usually achieved by using an alkaline solution (Thungphotrakul *et al.*, 2019). Next is the precipitation stage, where lignin forms a solid. This is usually done at a pH of up to 2 with 98% H₂SO₄ (Kim *et al.*, 2016). The final and most important stage involves converting the isolated lignin into sodium lignosulfonate (Priyanto *et al.*, 2018). This sulfonation process generally requires a relatively high temperature of around 95-100 °C (Gao *et al.*, 2019; Sudrajat *et al.*, 2020), and takes 2-4 hours with conventional heating (Abdelrahman *et al.*, 2024; Inwood *et al.*, 2018; Thungphotrakul *et al.*, 2019). Therefore, intensification efforts are needed to optimize the time and energy required during the conventional heating process.

Amaliah *et al.* (2021) converted virgin coconut oil into methyl ester sulfonate-based surfactants using ultrasound-assisted heating. The methyl ester from the transesterification process was sulfonated by reacting it with a 1:1% v/v NaHSO₃ reactant at 65°C for 30 minutes at a frequency of 40 kHz. This resulted in a surfactant with a viscosity of 4.04 cSt, a yield of 98%, and a surface tension of 32.028 dyne/cm (Amaliah *et al.*, 2021). This research contributes to optimizing time and energy through intensive heating, but the use of virgin coconut oil as a surfactant base conflicts with the food sector (Prasanna *et al.*, 2024; Zhao *et al.*, 2024). Therefore, Qadariyah *et al.* (2024) reported the use of empty palm fruit bunches as a base for sodium lignosulfonate-based

surfactants using microwaves. The process was carried out at 600 W, 75 minutes, and a 0.7 M NaHSO₃ concentration, resulting in a 24% increase in surfactant yield compared to conventional heating (Qadariyah *et al.*, 2024a).

Furthermore, microwave-based SLS production typically requires a reaction time of 60–75 minutes at 80 °C (Qadariyah *et al.*, 2024a; Setiati *et al.*, 2019), whereas the conventional process operates at even higher temperatures and longer reaction times (Abdelrahman *et al.*, 2024). Although microwave-assisted sulfonation offers significant advantages in efficiency, speed, and yield, it also has disadvantages, including the risk of excessive heat risk and higher equipment costs (Zhou *et al.*, 2024). Meanwhile, Anwar *et al.* (2018) reported that ultrasound technology can reduce processing time by half compared to microwaves at a lower temperature of 60°C (Anwar *et al.*, 2018). Daghooghi-Mobarakeh *et al.* (2020) reported that heating using ultrasound can reduce energy input by up to 24% and process time by 23.8% compared to conventional methods (Daghooghi-Mobarakeh *et al.*, 2020). This reinforces that, with energy efficiency comparable to microwaves and conventional heating, ultrasound offers the advantage of faster processing times (Amaliah *et al.*, 2021; Devine and Leadbeater, 2011). This advantage provides a strong foundation for the study. In addition, studies on the intensification of ultrasound for the utilization of lignin from empty palm fruit bunches as a surfactant have not been reported, particularly regarding ultrasonic operational parameters, such as power and irradiation time, as well as the conversion and yield of sodium lignosulfonate. Therefore, this study aims to investigate the production of sodium

lignosulfonate surfactants from oil palm empty fruit bunches via delignification and sulfonation using an ultrasonic homogenizer, and to analyze the effects of reaction time and ultrasonic power on the surfactant's characteristics.

MATERIALS AND METHOD

Materials

Oil Palm Empty Fruit Bunches with a lignin content of 25,08 % were purchased from PT Kodeco Agrojaya Mandiri (KAM), Kabupaten Tanah Bumbu, South Kalimantan. Sodium hydroxide (NaOH) with 98% purity, sulfuric acid with 98% purity, and methanol with 100% purity were purchased from Chemical Indonesia Multi Sentosa in Surabaya.

Methods

Alkali Pretreatment of Oil Palm Empty Fruit Bunches

Alkali pretreatment was carried out in a 1 L reactor (AKICO Co., Ltd., Japan). As much as 50 g of TKKS powder was added to 10 g of solid NaOH (98% purity) (equivalent to 20% of the fiber mass), which had been dissolved in 500 mL of distilled water.

The reactor containing the sample solution was operated at a temperature of 70 °C, pressure of 1 atm, ultrasound power of 117 watts (13%), and reaction time of 58 minutes with an ultrasonic homogenizer (Biosafer 900-92) operating at a fixed ultrasonic frequency of 20 kHz in batch mode, with a maximum power of 900 W (220 V/50 Hz power supply). The ultrasonic probe was immersed directly into the reaction mixture at a fixed central position, approximately 2 cm below the liquid surface, to ensure uniform cavitation throughout the system. After the desired time, the product was a black liquor

containing pulp. The product was filtered to obtain the black liquor filtrate. Then, the filtrate was added to distilled water at a 1:1 volume ratio.

Acid Precipitation

The second stage of precipitation was carried out in a reactor with an internal volume of 1 L (AKICO Co., Ltd., Japan). The sulfuric acid (98%) was added gradually to the sample solution until the pH reached 2. The mixture was homogenized for 60 minutes using a shaker (VRN-210, 220 V/50 Hz). After precipitation, the sample was separated by centrifugation at 2500 rpm for 10 minutes using a centrifuge (80-2, maximum speed 4000 rpm). Filter paper was then used to separate the precipitate from the liquid phase. After that, the solid remaining on the filter paper was dried in an oven for 3 hours at 100°C. The mass of the dried precipitate will be measured in grams.

Sulfonation of Lignin to Sodium Lignosulfonate

The third stage of sulfonation was carried out in a reactor with an internal volume of 1 L (AKICO Co., Ltd., Japan). The lignin load was 10 g, the water added was 300 mL, and the mixture was treated with sodium bisulfite at a reagent ratio of 1:0.6. NaOH catalyst (10%) was added until the solution pH reached 4 to 5. The reactor was operated at a temperature of 90 °C, pressure of 1 atm, reaction time of 50–90 minutes, and ultrasonic power of 210–297 W (23%, 28%, and 33% of maximum power based on the instrument settings) using the ultrasonic homogenizer setup as shown in Figure 1 by the ultrasonic probe at a central position (2 cm below the liquid surface).

The sodium lignosulfonate product solution was evaporated in an oven at 100 °C

for 3 hours to reduce its water content. The concentrated solution was filtered under reduced pressure to separate the unreacted lignin. The solution product was treated with methanol to bind the unreacted sodium bisulfite. The pure product was oven-dried at 100 °C for 2 hours to produce SLS in solid form. Next, the SLS yield (%) was calculated using Eq. (1).

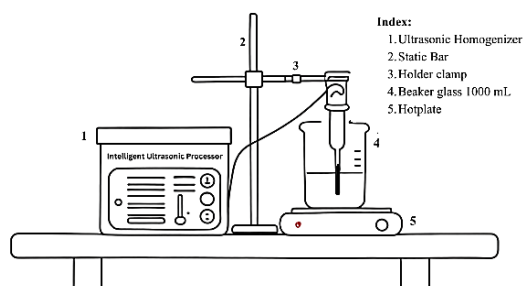


Fig. 1: Ultrasonic Homogenizer Schematic Diagram

$$\text{Yield (\%)} = \frac{\text{mass of dried SLS product (gr)}}{\text{lignin of after precipitation (gr)}} \times 100\% \quad (1)$$

Last, FTIR was used to analyze the solid product to identify chemical compounds and functional groups in the sodium lignosulfonate (SLS) sample, and it was also analyzed by GC-MS to identify and quantify the chemical components in the sodium lignosulfonate (SLS) sample.

Physicochemical Analysis of Sodium Lignosulfonate

The physicochemical analysis of the surfactant is conducted using pH measurements, visual observation, and the hydrophilic–lipophilic balance (HLB). The Hydrophilic–Lipophilic Balance (HLB) value of the sodium lignosulfonate surfactant was determined using the water solubility method with a semi-quantitative approach. This method was chosen as a simpler alternative compared to the experimental method of

mixing standard surfactants, which generally requires more time and complex procedures (Smejkal *et al.*, 2024).

A total of 0.1 g of sodium lignosulfonate was dissolved in 10 mL of distilled water, then homogenized by vortexing until thoroughly mixed. Next, the solution was visually observed to assess solubility and dispersibility in water. The HLB value range is determined by the visual characteristics of the resulting solution, using the dispersibility classification shown in Table 1.

Table 1. HLB Score Range Based on *Water Solubility* (Forster *et al.*, 2021)

Dispersibility in Water	Value
No dispersibility	1-4
Low dispersibility	3-6
Poorly dispersed (after vigorous agitation)	6-8
Stable milky dispersion	8-10
Translucent dispersion	10-13
Clear solution	>13

RESULTS AND DISCUSSION

The experiment was conducted in three main stages: pretreatment of oil palm empty fruit bunches (OPEFB), lignin precipitation, and lignin sulfonation using sodium bisulfite with sodium hydroxide as a catalyst in a batch system. Lignin conversion was determined based on the ratio between the initial amount of lignin and the lignin residue remaining after the reaction. At the same time, yield was defined as the ratio of the product mass to the initial lignin mass. The products identified in this study were acid-precipitated lignin and sodium lignosulfonate resulting from the sulfonation reaction.

The dried acid precipitation precipitate was identified as lignin. The solid lignin obtained was then weighed to determine the

lignin yield. The calculation results showed that the lignin yield produced was 2.2419% (w/w). Although the initial lignin content of OPEFB was 25.08% (w/w), the lignin yield obtained after the alkali pretreatment and acid precipitation processes was much lower. This was due to lignin degradation during pretreatment, incomplete delignification, and material loss during filtration and washing. A similar phenomenon was also reported during the separation of lignin from lignocellulosic biomass in previous studies (Harahap *et al.*, 2019; Zanirun *et al.*, 2015). From the precipitation process, 10.0976 g of solid lignin was obtained, which, based on composition analysis, contained 89.16% lignin, while the rest was non-lignin components originating from other lignocellulose fractions. Chen *et al.* (2014) stated that pretreatment with NaOH was highly effective at breaking down the lignocellulosic structure, resulting in significant lignin degradation. For example, pretreatment with 1% NaOH at 100°C for 40 minutes reduced the lignin content by 50-70% (Chen *et al.*, 2014). Thus, this demonstrates that NaOH can achieve significant lignin removal and improve biomass conversion. The lignin content obtained from NaOH pretreatment is high and consistent with the high lignin content reported in the literature, ranging from 70.6 to 91% (Maryana *et al.*, 2023; Mondylaksita *et al.*, 2020; Zanirun *et al.*, 2015).

Lignin obtained from precipitation is reacted with sodium bisulfite at a reagent ratio of 1:0.6. This ratio is chosen because a high reagent ratio will enhance the occurrence of growth, thereby facilitating the entry of sulfonate groups and making the substitution of hydroxyl groups on the benzyl carbon of lignin more complete (Syahbirin *et al.*, 2010). After that, a 10% NaOH solution

was added until the solution reached a pH of 4-5. 10% NaOH was used as a catalyst to create basic conditions that enhance the reactivity and openness of the lignin structure, thereby facilitating sulfonation. This concentration is sufficient to activate lignin without causing excessive degradation (Jia *et al.*, 2015). Meanwhile, the initial pH is raised to 4-5 from the previous precipitation stage, as this pH range is conducive to the lignin sulfonation reaction. This is because at that pH, the equilibrium of bisulfite (HSO_3^-) and sulfite (SO_3^{2-}) ion species in the solution is in optimal proportions, where bisulfite (HSO_3^-) acts as the most reactive sulfonation agent (Manurung *et al.*, 2024). In addition, this deactivates excess base and stops further reactions. This process also helps dissolve inorganic impurities and carbohydrates, resulting in SLS products with better purity (Syahbirin *et al.*, 2010).

This process involves sulfonation using an ultrasonic homogenizer at 90 °C. Hapsoro Aruna *et al.* (2018) state that this temperature is the optimum condition for the synthesis of sodium lignosulfonate, and it is also the temperature commonly used in the sulfonation process of lignocellulosic biomass (Hapsoro Aruno *et al.*, 2018). The resulting sodium lignosulfonate product is filtered and separated using a vacuum pump, and then 3 mL of methanol is added to remove any unreacted sodium bisulfite. After the reaction is complete, the SLS product formed is isolated and purified to remove unreacted lignin residues and excess sodium bisulfite.

Effect of Reaction Time

The effects of reaction time on sodium lignosulfonate yield were observed over the range 50-90 minutes. The relationships between sulfonation time and sodium

lignosulfonate surfactant yield at the ultrasonic powers used are 23% (210 W), 28% (256 W), and 33% (297 W), as shown in Figure 2.

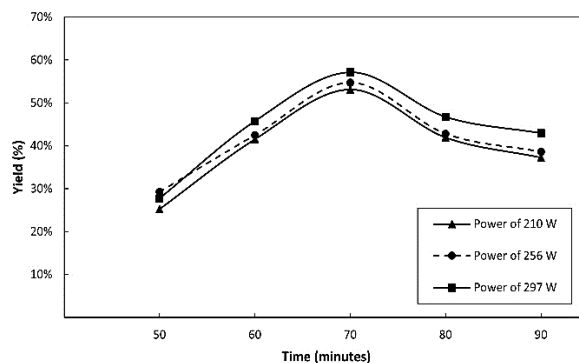


Fig 2: Surfactant yield as a function of reaction time at various ultrasonic powers.

Figure 2 shows the relationship between sulfonation time and sodium lignosulfonate surfactant at ultrasonic powers of 210 W, 256 W, and 297 W. From figure 2, the yield of sodium lignosulfonate surfactant increases from 50 minutes to 70 minutes and decreases from 70 minutes to 90 minutes. Using higher ultrasonic power also increased the yield of sodium lignosulfonate surfactants. The highest yield of sodium lignosulfonate surfactant was obtained at a reaction time of 70 minutes, an ultrasonic power of 297 W, and a temperature of 90 °C, the yield was 57,14% (wt). This is consistent with research by Qadariyah (2024), which discovered that a process's yield increases with response time. The extent of contact between the reacting chemicals increases with reaction time, so more sulfonate groups bind or react with lignin (Qadariyah *et al.*, 2024a). Lignin sulfonation is a critical step in converting it into an effective surfactant. The degree of sulfonation directly affects the yield and efficiency of the surfactant product. Optimal reaction conditions and additional chemical modifications can further improve the

properties and applications of lignin-based surfactants (Nita *et al.*, 2019; R Setiati *et al.*, 2018).

This is because the longer the reaction time, the greater the mechanical fragmentation due to prolonged ultrasonic cavitation (Liu and Zhu, 2011). Low-frequency ultrasound (usually 20–40 kHz) is dominated by the acoustic cavitation mechanism (Bai *et al.*, 2018). The ultrasonic cavitation process involves the formation, oscillation, and collapse of small bubbles in a liquid medium, thereby generating extremely strong physical forces such as microjets, shock waves, and turbulence (Shi *et al.*, 2012). These mechanical forces, particularly shear forces and shock waves, are capable of disrupting and may lead to bond cleavage within the lignin chains (Papadaki *et al.*, 2004) which are fragmented into compounds with very low molecular weights (<5000 Da) (Liu *et al.*, 2020; Ren and Fang, 2005) that can easily be more soluble during the separation/purification process of the sodium lignosulfonate product (Stewart *et al.*, 2016). These fragments must still be separated from the surfactant because their presence can reduce or degrade the surfactant's properties, particularly its ability to lower surface tension and form micelles (Chen *et al.*, 2025). This explains the observed decrease in yield beyond the optimal reaction time.

Furthermore, based on Figure 2, 42.86% of the lignin has not been converted into sodium lignosulfonate. This can be explained from a stoichiometric perspective because the sulfonation reaction requires bisulfite ions (HSO_3^-) to attack reactive sites on lignin (Sun *et al.*, 2013). Still, the complex and heterogeneous structure of palm empty fruit bunch lignin limits the number of accessible sites, so not all bisulfite ions succeed in forming sulfonate groups (Abel *et al.*, 2024).

Additionally, side reactions such as mechanical fragmentation due to ultrasonication for more than 70 minutes result in lignin fragments with very low molecular weight that are difficult to separate due to their high solubility, while purification processes through evaporation or precipitation also fail to recover the entire product (Liu *et al.*, 2020; Ren and Fang, 2005). Thus, although lignin conversion is relatively high, the yield of isolated SLS is lower due to product loss from limited reactive sites, excessive fragmentation, and inefficient separation.

Effect of Ultrasound Power

Ultrasonics generate alternating high- and low-pressure waves that form rapidly collapsing vacuum bubbles, a process known as cavitation. This bubble collapse can break down the lignin structure and increase the contact surface area between sodium bisulfite and lignin. The effect of ultrasonic power on sodium lignosulfonate yield was observed at various powers of 210 W, 256 W, and 297 W at a constant frequency of 20 kHz.

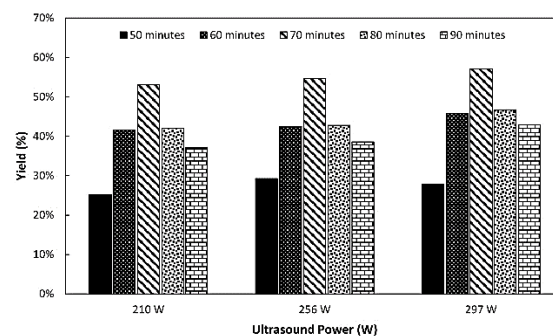


Fig. 3: Surfactant yield as a function of ultrasonic power at various reaction times.

Figure 3 shows that the yield value of sodium lignosulfonate surfactant increased from 210 W to 297 W. Longer reaction times also increase the yield of the sodium lignosulfonate surfactant. The highest yield of

sodium lignosulfonate surfactant was obtained at a reaction time of 70 minutes and a power of 297 W, corresponding to 57.14% (wt). This is according to research by Amaliah (2021), which found that the power of the equipment used in the sulfonation process can affect the yield of the resulting surfactant (Amaliah *et al.*, 2021). The sulfonation process using high-power ultrasonics can increase product yield. This is because the cavitation generated from ultrasonics effectively breaks down the lignin structure and expands the reaction surface (Grewell *et al.*, 2013; Li *et al.*, 2022; Mao *et al.*, 2017). The cavity effect can cause cracks and cavities on the lignin surface, thus increasing its surface area and reactivity (Mao *et al.*, 2021). Cavitation, caused by ultrasonic waves, also generates localized high temperatures and pressures, which disrupt the molecular structure of lignin. This process increases the surface area and exposes reactive sites, thereby facilitating chemical reactions such as sulfonation (Chen and Jiang, 2009; Özkan and Kuyumcu, 2006). Ultrasonic waves enhance sodium bisulfite's ability to fulfill its crucial role as a sulfonation agent, namely, by inserting hydrophilic groups into the lignin structure. This results in amphiphilic molecules comprising hydrophilic sulfonate groups and a hydrophobic aromatic backbone, thereby enabling sodium lignosulfonate to reduce surface tension and stabilize emulsions (Ruwoldt, 2020; R. Setiati *et al.*, 2018).

In addition, according to Anwar (2018), the use of ultrasonics at shorter reaction times and lower temperatures can yield higher sodium lignosulfonate yields than conventional methods. Ultrasonics can generate turbulence, thereby increasing the diffusion rate of the reagent into lignin pores (Anwar *et al.*, 2018). Zhou *et al.* (2014) reported that increasing the ultrasonic power

from 100 W to 300 W resulted in an increase in the phenolic content of sodium lignosulfonate from 0.69% to 0.92%, indicating more effective lignin breakdown (Zhou *et al.*, 2014).

FTIR and GC-MS Characterization

FTIR analysis was carried out on lignin samples and sodium lignosulfonate surfactant samples obtained from the sulfonation process with the highest yield, namely, a reaction time of 70 minutes and an ultrasonic power of 297 W, resulting in 57.14 wt%, as follows.

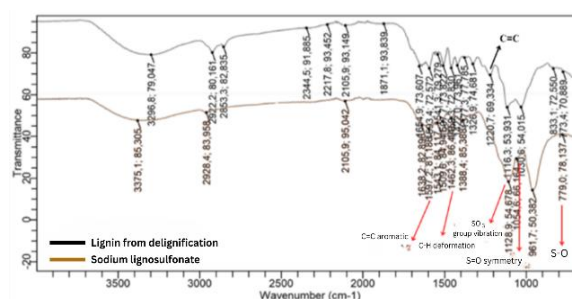


Fig. 4: FTIR Graph of Lignin and Sodium Lignosulfonate Surfactant

Figure 4 shows the lignin functional groups at absorption wavenumbers of 1220,7031 cm^{-1} . In the wavenumber region 1200-1300 cm^{-1} , the presence of aromatic C=C bond in the lignin structure is indicated. This is consistent with the general understanding of lignin's aromatic nature (Shen *et al.*, 2008). The effectiveness of lignin sulfonation is demonstrated by the creation of sulfonate groups (SO_3^-), which is revealed by the appearance of an absorption peak at wave number 1128,9 cm^{-1} in the FT-IR spectra for sodium lignosulfonate from empty fruit bunches (Ji *et al.*, 2013; Nita *et al.*, 2019). Another study confirmed the presence of sulfonate groups in lignosulfonates with a peak around 1192 cm^{-1} (Mu'izzah *et al.*, 2023). In addition, there is a symmetric S=O

stretch at a wavenumber of $1054,6\text{ cm}^{-1}$ and S-O stretches at wavenumbers of $779,0$ and $961,7\text{ cm}^{-1}$. The sulfonation reaction mechanism of lignin isolate EFB involves substituting SO_3 and attacking the -OH; C=, and guaiacyl (methoxyl) groups found in lignin (Calegari *et al.*, 2019).

The loss of -OH, C=C, guaiacyl groups, and the formation of sulfonate groups (SO_3^-) indicate that the sulfonation process has been successful and Sodium lignosulfonate has been formed. The sodium lignosulfonate surfactant formed has similarities to the commercial and standard sodium lignosulfonate from Aldrich, as shown in Table 2.

Table 2. Comparison of wavenumbers of sodium lignosulfonate

This study (cm^{-1})	Sodium lignosulfonate from Black Liquor (cm^{-1}) (Priyanto <i>et al.</i> , 2018)	Functional Group
779.0; 961.7	750 – 1,000	Range S-O
1,054.6	1,005 – 1,055	Range S=O
1,128.9	1,120 – 1,230	Vibration groups SO_3
1,462.3	1,460 – 1,470	Deformation C-H (asymmetric)
1,509.6 – 1,597.2	1,505 – 1,600	Aromatic ring vibrations
2,928.2	2,820 – 2,940	Range C-H

Based on the table 2 shows that the vibration range of the sulfonate group (SO_3) in sodium lignosulfonate from EFB and commercial sodium lignosulfonate is shown at wave numbers $1120\text{--}1230\text{ cm}^{-1}$, the S-O group at wave numbers $750\text{--}1000\text{ cm}^{-1}$, and the symmetric S=O group at wave numbers $1005\text{--}1055\text{ cm}^{-1}$ (Nita *et al.*, 2019; Priyanto *et al.*, 2018).

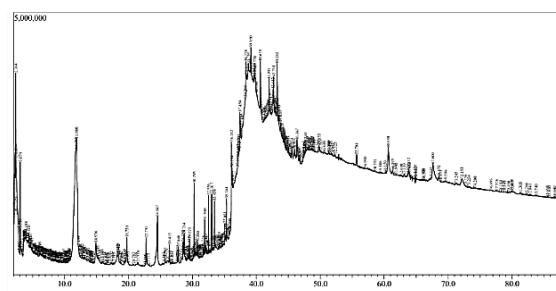


Fig. 5: Chromatograph of Sodium Lignosulfonate Surfactant of Oil Empty Fruit Bunches

Based on Figure 5, the chromatogram shows several peaks with retention times ranging from 2.226 to 87.609 minutes. The data shown in Figure 5 are summarized in Table 3, with the identified compounds grouped into several major compound groups to facilitate interpretation. Overall, identified compounds (excluding the "other compounds" category) accounted for approximately 34.57%, while the remainder (65.43%) consisted of minor peaks with areas below 0.01%.

Table 3. Compounds Content List of Sodium Lignosulfonate Surfactant Based on GC-MS Characterization

Compound Group	Residence Time (min)	Area (%)	Compound Name	Total Area (%)
Phenol and guaiacol derivatives	11.900	2.38	Phenol	4.3
	14.936	0.15	2-methoxy-phenol	
	19.750	0.15	4-vinylphenol	
	22.791	0.15	2-Methoxy-4-vinylphenol	
	33.456	0.16	Syringylacetone	

Compound Group	Residence Time (min)	Area (%)	Compound Name	Total Area (%)
Sterols & triterpenoids	45.995	0.73	Dimethoxy stilbene	5.85
	49.588	0.58	2,6-dimethoxyphenol	
	52.323	0.83	beta-Sitosterol acetate	
	58.551	1.65	3alpha,5alpha-Cyclo-ergosta-7,9(11),22t-triene-6beta.-ol	
	59.386	1.69	Stigmasta-3,5-dien-7-one	
	60.638	1.68	14-BETA-H-PREGNA	
Alcohol and Aldehyde	34.576	0.02	cis-9-Tetradecen-1-ol	6.34
	44.48	0.25	4-DECANOL	
	45.851	0.22	1-Heptadec-1-ynyl-cyclopentanol	
	49.101	1.38	13-Tetradecenal	
	49.835	0.82	13-Octadecenal	
	50.64	2.01	Hydroxy - tetradeca - hexadiene	
	64.992	1.51	Z,Z-3,13-Octadecediene-1-ol	
	82.29	0.13	6-Tridecanol	
Amides and ether-containing compounds	39.055	1.42	Dodecyl nonyl ether	3.57
	39.15	0.82	Nonanamide	
	41.991	0.71	Octadecenamide (Z)-	
	42.069	0.62	9-Octadecenamide	
Fatty acid and Esters	36.112	0.39	Hexadecanoic acid ethyl ester	10.89
	38.299	2.64	9,12-Octadecadienoic acid	
	38.466	1.67	(E)-9-Octadecenoic acid ethyl ester	
	39.834	1.08	Oleic acid ethyl ester	
	40.639	1.61	Glycidyl Palmitate	
	40.811	1.03	8-Nonenoic acid	
	41.155	0.49	Tridecanedioic acid, diethyl ester	
	41.210	0.48	Pentadecanoic acid, methyl ester	
	72.153	1.5	Linoleyl Palmitate	
Sulfur-containing compounds	2.364	1.12	L-Alanine 3-sulfo-	1.76
	42.228	0.34	1-Undecanol, 11-mercapto-	
	2.901	0.09	Aminomethanesulfonic acid	
	4.225	0.13	L-Cysteine sulfinic acid	
	4.304	0.06	Cysteic acid	
	10.680	0.02	Dimethyl trisulfide	
Sugar derivative	15.337	0.03	Anhydro-sugar	1.86
	35.735	0.08	1,5-Anhydro-4-O-acetyl-2,3,6-tri-O-methyl-D-mannitol	
	39.215	1.75	Methyl 2,6-anhydro- α -D-altroside	
Other compounds (<0.01%)				65.43
TOTAL				100

Table 3 shows that sulfur-containing compounds accounted for 1.76% of the total, comprising alanine-sulfo derivatives, sulfinic acid, sulfonic acid, and dimethyl trisulfide, confirming that the sulfonation reaction was successful. The sulfonate group ($-\text{SO}_3\text{H}$) was successfully incorporated into the lignin structure, which is a key step in the formation of the surfactant sodium lignosulfonate (Gupta *et al.*, 2025; Mousli and Tazerouti, 2007).

In addition, Table 3 also confirms the presence of phenol and guaiacol groups, with phenol being the most abundant compound at 2.38%. The compounds in this group are lignin degradation products representing the three main structural units of lignin, namely p-hydroxyphenyl (H), guaiacyl (G), and syringyl (S) (Del Río *et al.*, 2022; Vanholme *et al.*, 2019). This demonstrates that the ultrasonic method can depolymerize empty fruit bunch lignin into simpler phenolic fragments (Liang and Shiping, 2009).

In addition to the compound groups discussed above, Table 3 also shows the presence of several other groups, including fatty acids and their esters (10.89%), alcohols and aldehydes (6.34%), sterols and triterpenoids (5.85%), and amides and ethers (3.57%). Fatty acids and esters, such as linoleic and oleic acid derivatives, originate from the lipid components of biomass residues formed during the production process. This is consistent with the findings reported by Mei *et al.* (2020), who noted that sodium lignosulfonate may contain esters resulting from dissolution in methanol (Mei *et al.*, 2020) to remove sodium bisulfite. Alcohol and aldehyde groups are typically intermediate products of thermal and oxidative degradation during GC–MS analysis (Songara *et al.*, 2016). Meanwhile, sterols and triterpenoids are characteristic compounds of

plant biomass, indicating that some non-lignin components remain in the system (Seki *et al.*, 2015; Valitova *et al.*, 2016). Amide and ether compounds can form as secondary reactions during the chemical treatment process (Downey *et al.*, 2011).

In addition, sodium lignosulfonate (SLS) is a complex, non-volatile macromolecule; therefore, it cannot be directly detected as an intact molecule via GC–MS analysis without derivatization (Xueqing *et al.*, 2010; Yang *et al.*, 2015). Therefore, the compounds identified in this analysis more accurately represent molecular fragments derived from the SLS structure, either from thermal degradation during analysis or as byproducts of side reactions during sulfonation (Qiu *et al.*, 2010).

Furthermore, Figure 6 also shows a broad peak (broad hump) in the retention time range of approximately 35–50 minutes, indicating the presence of a mixture of complex compounds with a broad molecular weight distribution (Li *et al.*, 2025). This phenomenon is commonly observed in the analysis of lignin and its derivatives, which tend to undergo partial fragmentation into oligomers during the heating process in the GC system (Wu *et al.*, 2019). The presence of this broad peak reinforces the indication that the basic structure of lignin is still present in the form of complex fragments, even though it is not identified as a single compound in the mass spectrum (Chen *et al.*, 2018).

Physicochemical Analysis

Table 4 compares the physicochemical properties obtained in this study with those of commercial samples and SLS obtained by conventional methods. In terms of color review, this study obtained a darker brown color than the commercial and conventional methods, which showed a lighter brown

Table 4. The Comparison of Physicochemical Analysis of Sodium Lignosulfonate Surfactant

Parameter	Test Method	Commercial (SLS Aldrich) (Syahbirin <i>et al.</i> , 2010)	Conventional (R. Setiati <i>et al.</i> , 2018; Syahbirin <i>et al.</i> , 2010)	This study
Colour	Visual Observation	Light brown	Light Brown	Dark brown
pH	pH meter	7-7.5	6.3 – 7.24	6.6
HLB	Water solubility	-	11.62	10-13

color. Yang *et al.* (2014) stated that dark-colored SLS generally has a higher molecular weight content (Yang *et al.*, 2014). Additionally, the dark color obtained in this study is also caused by the presence of impurities such as many sugar derivatives and organic compounds with high yields, like fatty acids and esters (around 10.89%), alcohols and aldehydes (around 6.34%), as well as sterols and triterpenoids (around 5.85%), as shown in the GC-MS results. Although there is a color difference compared to commercial products, several previous studies have shown that the color range of SLS varies from light brown to dark brown or black, depending on the production process and its purity level (Ruwoldt, 2020).

Table 4 also shows the pH value of this study (6.6), which falls within the range of SLS using the conventional method of 6.3–7.5, but below the range of commercial SLS of 7–7.5. Syahbirin *et al.* (2010) stated that SLS obtained from biomass has a stronger acidic characteristic value, as demonstrated using eucalyptus lignin (Syahbirin *et al.*, 2010). Meanwhile, the difference in pH values relative to commercial products, which tend to be higher, can be caused by several factors, including the product's purity and the operating conditions of the sulfonation process. Yan *et al.* (2010) state that the behavior of lignosulfonate in indicates shows that the surface charge and the degree of molecular aggregation of SLS increase with rising pH due to the ionization of the

sulfonate and phenolic hydroxyl groups. Thus, the pH value of 6.6 obtained in this study is consistent with the characteristics of SLS produced by the biomass sulfonation process and falls within the neutral-to-slightly acidic pH range commonly reported for SLS solutions (Ruwoldt, 2020; Yan *et al.*, 2010).

Lastly, in the HLB test, SLS in this study showed its solubility in water and dispersion, and it is light-permeable, thus categorized within the range of 10–13. Setiati *et al.* (2018) also reported that the HLB value of the SLS surfactant, determined by the conventional method, was 11.62. The HLB value, which is classified as medium to high, indicates that the hydrophilic groups (water-loving) in the sodium lignosulfonate molecule are relatively more dominant compared to its lipophilic groups (oil-loving). This is consistent with the basic structure of SLS, an anionic polymer with many sulfonate groups ($-\text{SO}_3^-$) that ionize in water, making it readily dispersible and forming a stable solution (Ruwoldt, 2020; R. Setiati *et al.*, 2018).

CONCLUSIONS

Sodium lignosulfonate surfactant was successfully produced from oil palm empty fruit bunch lignin using an ultrasonic-assisted heating method. The success of this synthesis was evidenced by the formation of sulfonate groups (SO_3^-) in the lignin structure, indicated by a total sulfur content of 1.76%, with L-

Alanine-3-sulfo- (1.12%) as the primary indicator. Optimal operating conditions were achieved at an ultrasonic power of 297 W, a reaction time of 70 minutes, and a pressure of 1 atm, yielding 57.14% sodium lignosulfonate. Physicochemically, the product exhibits a dark brown color, a pH of 6.6, and an HLB value in the range of 10–13, indicating good hydrophilic properties.

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