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# Laboratory Optimization Study of Sulfonation Reaction toward Lignin Isolated from Bagasse

*Rini Setiati, Septoratto Siregar and Deana Wahyuningrum*

## Abstract

Bagasse is scientifically defined as waste from the extraction of sugarcane liquid after the grinding process. Bagasse is biomass which is used as raw material to be processed into surfactants. Bagasse fiber cannot be dissolved in water because it consists mostly of cellulose, pentosane and lignin. The optimum conditions for obtaining the highest yield and the best conversion of bagasse to lignin were achieved when used 80 mesh bagasse and 3 M NaOH as a hydrolysis agent. Then lignin is reacted with 0.25 sodium bisulfite to the surfactant sodium lignosulfonate. Lignin and sodium lignosulfonate were further characterized using a FTIR spectrophotometer to determine the components contained therein. The lignin component consists of phenolic functional group elements, aliphatic and aromatic groups, ketone groups, aren functional groups, amine groups and alkyl groups along with standard lignin components. Likewise with lignosulfonates, with indicator components consisting of C=C alkenes, Sulfate S=O, C=O carboxylic acids and S-OR esters. The NMR test was resulted the monomer structure of SLS surfactant bagasse. The results indicate that the lignin isolation process from bagasse has been successfully. Likewise, the sulfonation of lignin to lignosulfonate is also successful.

**Keywords:** bagasse, FTIR, isolation, lignin, lignosulfonate, sulfonation

## 1. Introduction

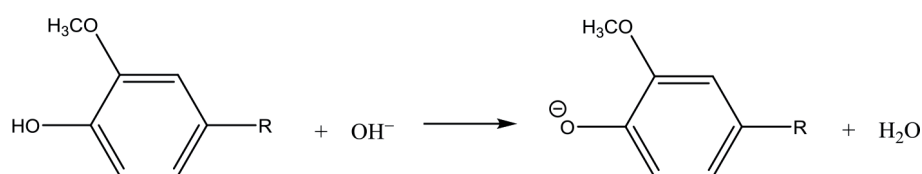
In specific, bagasse is scientifically defined as a waste of sugarcane liquid extraction after milling process and is in a fibrous form. Bagasse is one of the biomass resources that is widely used as a boiler fuel in sugar factory, source of animal feed, material of paper, cement and brick reinforcement material [1, 2]. The amount of bagasse production each year is abundant, easily obtained, and economical. Based on the data from Indonesian Sugar Farm Research Center (P3GI) [3], bagasse amounts to approximately 32% of milled sugarcane weight or about 10.2 million ton/year or mill/season all around Indonesia. Furthermore, bagasse contains 48–52% water, sugar (approximately 3.3% in average), and fiber at an average of 47.7% [4, 5]. Bagasse fiber is unable to be dissolved in water because mostly it consists of cellulose, pentosane, and lignin [6]. Bagasse waste could be used as a raw material in producing surfactant due to its high lignin content, which is estimated to be approximately 25% [7]. Lignin can be separated

from bagasse waste by lignin isolation method and hydrolysis process using sodium hydroxide (NaOH) solution [8, 9]. The process also depends on creating bagasse surface enlargement by minimizing the size of its fiber in order to have the better yields of the isolated product. Lignosulfonate is a derivate of lignin that can be produced by reacting lignin with sodium bisulfite (NaHSO<sub>3</sub>) at certain reaction conditions via the electrophilic addition reaction [10, 11]. The presence of double bonds within the lignin structure has made lignin to be available for the addition reaction using various electrophilic substances, for instance, the hydrogen sulfite (–HSO<sub>3</sub>) group of sodium bisulfite [12]. Thus, the product is categorized as sodium lignosulfonate (SLS) surfactant [13]. In addition, lignosulfonate is one of the variants of anionic surfactant that is often utilized in a chemical injection process of enhanced oil recovery (EOR) in the oil industry [14]. Therefore, the high lignin content in bagasse have made bagasse to be an eligible candidate to produce surfactant and became the aim of this research, which is to produce the lignosulfonate surfactant via sulfonation reaction of lignin previously isolated from bagasse. Based on the observations and search for existing patents, what have been found are patent Nos. 2,837,435 and 4,304,361 regarding the use of bagasse as a raw material for building needs, cutting of bagasse fiber for growing media needs, methods for producing bio-aromatic-based chemicals, bio-based aromatic fuels, and lignin residues [15, 16]. Whereas, the No. 8529731 was found to contain the process of fractionation of bagasse into cellulose, hemicellulose (xylene), and lignin with high-purity  $\alpha$ -cellulose, which is a useful raw material for the manufacture of cellulose esters such as cellulose triacetate and cellulose plastics [17]. Amri [18] has shown research on sodium lignosulfonate surfactant which has characteristics of water solubility, hygroscopic, and color properties as well as the polydispersity properties of sample SLS which are generally in accordance with commercial SLS.

The lignin isolation method (hydrolysis) can excite lignin with acid, resulting in acid lignin as shown in **Figure 1**.

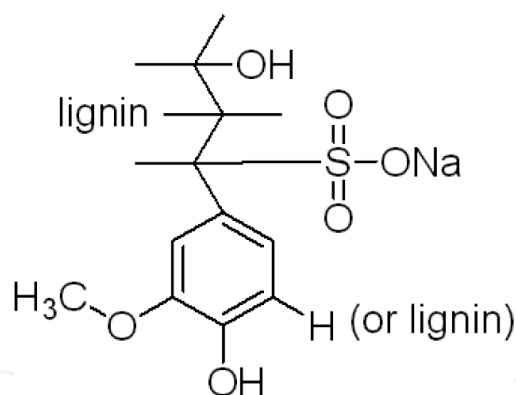
Isolation of lignin is generally carried out using sulfuric acid or hydrochloric acid. Under acidic conditions, the charged lignin will become neutral. Lignin will not dissolve in water and will settle. The resulting solid can be separated by filtering. To change the nature of water-insoluble lignin, lignin can be modified through the sulfonation process to become lignosulfonate [20]. Sulfonation is intended to change the hydrophilic nature of the less polar lignin into a more polar/water-soluble lignosulfonate salt by inserting the sulfonate group and its salt into the lignin hydroxyl group so that the lignosulfonate salt has a structure as a surface-active agent or surfactant [19]. The sulfonate group in the lignosulfonate is a hydrophilic group that causes the lignosulfonate to have an amphipathic structure (surfactant). **Figure 2** shows the structure of the lignosulfonate.

The existence of the sulfonate group can be determined by the general formula R-SO<sub>3</sub>Na which is a simplification of the sulfate R-O-SO<sub>3</sub>Na [21]. The R group is a group of C<sub>8</sub>-C<sub>22</sub> aromatic carbon atoms which is a hydrophilic group, while the hydrophobic group consists of carboxylates, sulfonates, phosphates, or other organic acids. The sulfonation process is the core process for producing



**Figure 1.**

*The reaction of lignin and NaOH in the delignification process [19].*



**Figure 2.**  
*Lignosulfonate structure.*

lignosulfonate salts. The reaction occurs between lignin and sulfite salts. There are several types of sulfite salts that can be used in this process, including using sodium bisulfite ( $\text{NaHSO}_3$ ) in addition to other ingredients such as  $\text{Na}_2\text{SO}_3$ ,  $\text{NaOH} + \text{CH}_2(\text{OH})\text{SO}_3\text{Na}$ ,  $\text{HCHO} + \text{NaOH}$ ,  $\text{C}_2\text{C}_{14} + \text{C}_1\text{SO}_3\text{H}$ , or  $\text{SO}_3^{2-} + \text{CH}_2\text{O}$  [22].

Several studies on the manufacture of sodium lignosulfonate that have been tried include raw materials for oil palm empty bunches [23], oil palm shells [24], palm frond biomass [18], and bagasse [25, 26]. The results of this study were limited to the manufacture of sodium lignosulfonate products which were correlated with the size of bagasse powder and the concentration of sodium bisulfite. Lignosulfonates, as a result of lignin sulfonation, are currently widely used as emulsifiers in iron ore processing, oil field chemicals, and pesticide formulas [27] as well as dust emission control and stabilizer for the fertilizer industry, animal feed industry, gypsum agent wallboard dispersant, oil well drilling mud additive, brick reinforcement, cement, and mortar [28].

## 2. Materials and methods

In this study of sulfonation reaction toward lignin isolated from bagasse, this study used bagasse as the main raw material, with chemical reagents being sodium hydroxide, sulfuric acid, sodium bisulfite, and distilled water. Range and specifications are used in the bagasse lignin isolation process consisting of bagasse size 40, 60, and 80 mesh; sodium hydroxide concentration 0.6, 2, 3, 6, 8, and 10 M; and sodium bisulfate concentration 0.25 M. The equipment used in the process of lignin isolation and surfactant sulfonation consists of a sieve shaker; hot plate magnetic stirrer; two- or three-neck flask; condenser; beaker glass 200, 500, and 1000 mL; measuring cup 250 mL; thermometer; rod mixer; burette; gloves; glasses; mask; fume hood; pH meter paper; Buchner funnel; Whatman paper; watch glass; oven; digital balance; 250 and 500 mL reagent bottles; 10-mL vial bottle; and desiccator. The mechanism process of the lignosulfonate surfactant occurs through two reactions, namely, hydrolysis and sulfonation [29]. Hydrolysis is a reaction to break down lignin molecules into smaller molecules so that they can dissolve in water. Sulfonation is a reaction between bisulfite ions and lignin molecules. Previous research results reported that the surfactant methyl ester sulfonate (MES) could be synthesized from the direct sulfonation of palm kernel oil methyl ester using sodium bisulfite solution. The important from this previous research is the sulfuric acid concentration factor which affects the value of the decrease in surface tension, the decrease in interface tension, the stability of the emulsion, and the color of the surfactant [30].



The method of processing bagasse into liginosulfonate is carried out through two processes, namely, the isolation process of lignin from bagasse and the sulfonation process of lignin into sulfonates. The bagasse from the sugar factory was previously sifted coarsely and then to oven to dry completely. Then the oven bagasse is sieved again with a sieve shaker to obtain a particle size of bagasse with a certain mesh, namely, 40 mesh, 60 mesh, 80 mesh, and 100 mesh. **Figure 3** shows the bagasse that has been dried and then sieved using a sieve shaker to become a fine powder (**Figure 4**) [31].

The method used in this study is a development from previous researchers who modified liginosulfonate from lignin. In his research, lignin isolation was carried out using NaOH reaction by heating at a temperature of 60–100°C for 3–10 hours [20]. In this research, the lignin isolation process begins by inserting the bagasse that has been sieved with a sieve shaker into the reaction flask and reflux directly in sodium hydroxide solution at a various concentration for 5 hours at a temperature of 90–100°C. The result of reflux of NaOH is then filtered, diluted, and neutralized by adding dropwise concentrated sulfuric acid ( $H_2SO_4$ ) to pH = 2 and allowed to stand for at least 8 hours until a precipitate appears, then filtered, and dried in an oven at 70°C. In this filtering process, it is accompanied by rinsing with distilled water because lignin does not dissolve in water and this rinsing with distilled water will dissolve the remaining glucose that may still be present in the results of the lignin isolation. The precipitate obtained is lignin isolated from bagasse and after drying using a vacuum oven, it becomes a brown powder.

The lignin isolation process starts with 5 gram of dry bagasse powder of each mesh size which is put into a three-neck flask, then NaOH is added until the bagasse is submerged and heated for 5 hours using a hot plate magnetic stirrer at a



**Figure 3.**  
*Bagasse.*



**Figure 4.**  
*Mesh of bagasse [31].*

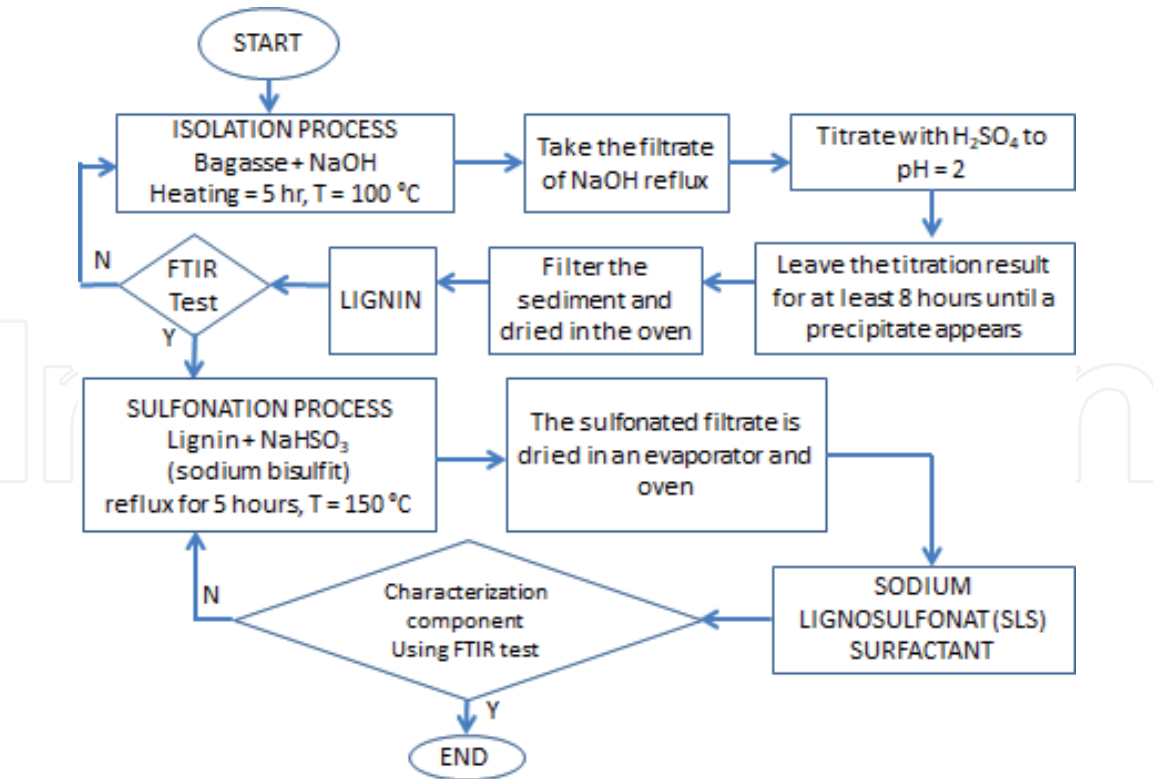
temperature of 90–100°C. The reflux filtrate which still contains NaOH is taken and diluted with water at a volume ratio of 1:1. The solution is then added dropwise to H<sub>2</sub>SO until it reaches pH = 2, then this solution is left to stand to get a precipitate for at least 8 hours. The precipitate that is formed is filtered and then dried in an oven. The structure of isolated lignin product was determined through FTIR spectrophotometric measurements which were then compared with the standard lignin FTIR spectrum. In the lignin isolation process, optimization was also carried out using the concentration of NaOH used, namely, with a concentration range of 2, 3, 6, 8, and 10 M. Each NaOH concentration is used in the lignin isolation process by varying the size of the bagasse mesh.

The synthesis of bagasse into sodium lignosulfonate begins with the preparation of bagasse powder which will be isolated to separate the lignin from the bagasse. After lignin is formed, a Fourier transform infrared (FTIR) [32, 33] test must be carried out to ensure the presence of lignin-forming components. The standard lignin used is commercial lignin from the lignin product of Aldrich and Kraft. If the component has not been formed, it must return to the isolation process again with changes to the variables used. There are three components of the main functional groups as indicators of lignin formation, namely, the phenolic O–H functional groups, the aliphatic and aromatic –CH– stretching groups, and the C=C aromatic functional groups. In the lignin isolation process, the variables used are NaOH concentration, duration of the isolation process, and temperature in the isolation process. This looping process is carried out continuously until the lignin component is obtained that is in accordance with the existing commercial lignin standards. If the lignin formed meets the component requirements, it can be continued to the sulfonation process. The result of this sulfonation process is a brown powder of sodium lignosulfonate (SLS) surfactant. This product must also perform component characterization using the FTIR test. If the FTIR test results do not show any lignosulfonate-forming components, then a looping process is carried out until the sulfonation process produces a lignosulfonate component that matches the standard lignosulfonate. The standard lignosulfonates used are Patricia and Aldrich standards [34].

The components of the lignosulfonate that must be present include the stretching vibration of the alkene functional group –C=C–aromatic, the stretching vibration of the sulfonate functional group S=O, the bending vibration of the C=O functional group carboxylate group, and the bending vibration of the S-OR ester functional group. At this stage, it can be said that the synthesis process is complete, as illustrated in **Figure 5**. The process of synthesis of bagasse into sodium lignosulfonate surfactant as a whole can be seen in **Figure 5**.

The sulfonation process is a procedure in the form of adaptation and modification from research conducted by Ari [25] and Furi [26]. A total of 8 gram of isolated bagasse lignin was put into a three-neck flask, then sodium bisulfite solution was added, and then heated (refluxed) at 150°C for 5 hours. The reaction product is cooled and precipitated and further dried in a vacuum oven. From this sulfonation process, it produces a surfactant called sodium lignosulfonate (SLS). The structure of SLS surfactant was determined through FTIR, LCMS, and NMR spectrophotometric measurements [36]. The FTIR test results were then compared with the main components of the commonly used commercial lignosulfonate [34]. If it is in accordance with the components forming the SLS surfactant, this product can be said to have been successfully obtained. If it is not accordance with the standard components that should be present in the lignosulfonate, the sulfonation process is repeated with different parameters.

Furthermore, the lignosulfonate monomer structure test was carried out using gas chromatograph mass spectrum (GCMS) and nuclear magnetic resonance



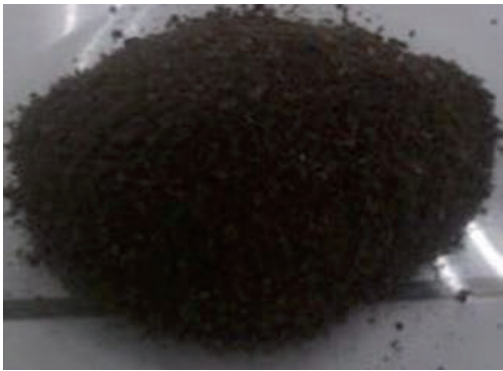
**Figure 5.**  
*Schematic synthesis of bagasse into sodium lignosulfonate [35].*

(NMR). The structure of the lignosulfonate monomer is needed in order to help see the suitability of the use of the surfactant lignosulfonate against the crude oil to be injected by the lignosulfonate.

### 3. Results and discussion

The lignin isolation process has been carried out several times with variations in the concentration of NaOH and the size of the mesh bagasse. Variations in bagasse mesh sizes used were 40 mesh, 60 mesh, 80 mesh, and 100 mesh, and the concentration of NaOH was 2, 3, 6, 8, and 10 M. **Figure 6** shows the results of lignin isolation in the form of a dark brown powder.

From the experiment as many as 15 variations, only four variations met the requirements, namely, lignin results above 60% and they had lignin-forming components, namely, lignin (80–3), lignin (60–8), lignin (40–10), and lignin (80–10). The results of lignin recovery can be seen in **Table 1**. In this table, it can be seen that



**Figure 6.**  
*Lignin from bagasse isolation.*



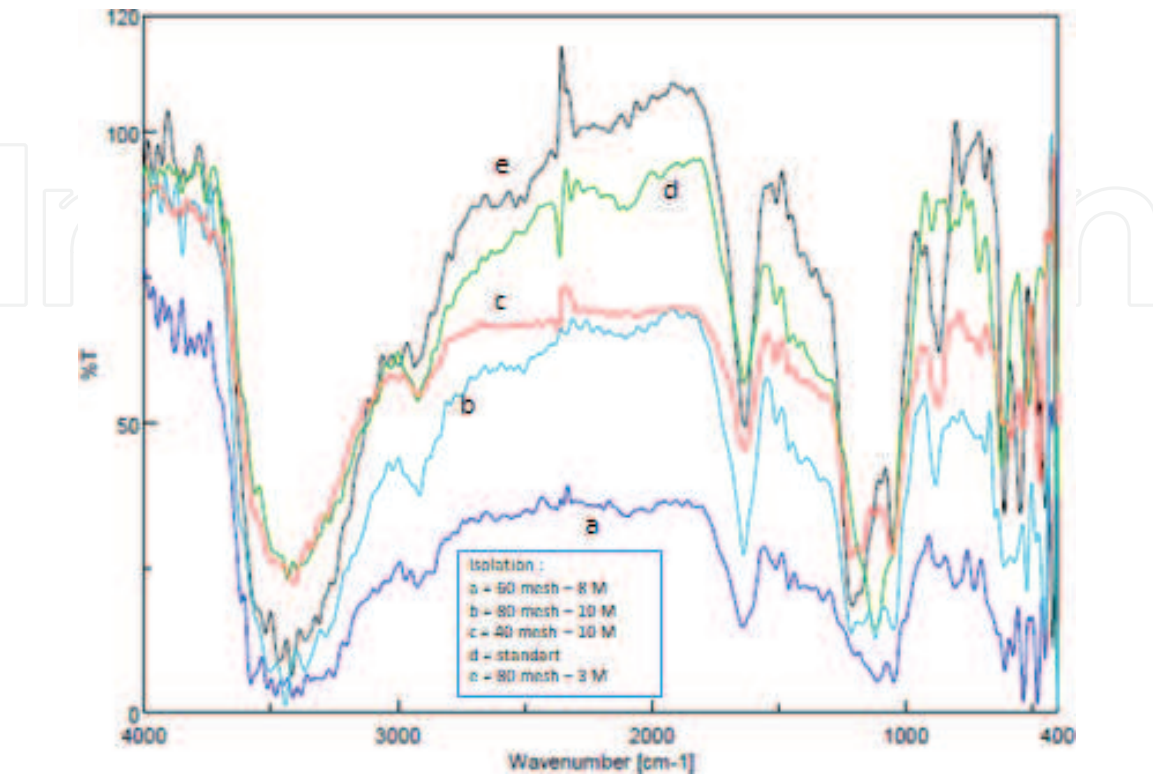
No.	Concentration of NaOH (M)	Lignin (%)			
		mesh 40	mesh 60	mesh 80	mesh 100
1	2	22.46	20.66	48.60	18.00
2	3	63.36	34.36	61.80	22.22
3	6	32.26	13.07	24.43	35.80
4	8	66.80	75.73	38.36	24.30
5	10	62.85	51.80	63.79	26.10

**Table 1.**  
*Results of lignin isolation at variations in bagasse size and NaOH concentrations.*

the highest percentage of lignin recovery occurs in the lignin isolation process with a concentration of 3 M NaOH—40 mesh size of 63.36%, 8 M NaOH—60 mesh size of 75.73%, and 10 M NaOH—80 mesh size 63.79%.

Based on the results of the percentage lignin obtained and the results of the lignin functional group absorption test, it turns out that not all research variations have three indicators of the lignin-forming functional groups. The lignin results were compared by looking at the percentage transmittance value; the best lignin results were lignin (80–3), which is bagasse lignin processed with 80 mesh size variations using NaOH 3 M. Lignin (80–3) is then compared with lignin commercial standards which are lignin of Aldrich and Kraft. **Figure 7** shows the FTIR test results on the sample result of isolated and sample of standard lignin.

**Figure 7** shows the combined FTIR results for the four most lignin isolation processes, which produce lignin yields of more than 60%. The four variations of lignin isolation are represented as curve a, curve b, curve c, and curve e. This FTIR graphic overlay is then combined with the standard lignin FTIR results, namely, curve “d” at this figure (**Table 2**).



**Figure 7.**  
*FTIR test results on lignin isolation.*



No.	Typical functional group vibrations in lignin structure	Wave number (cm <sup>-1</sup> )			
		Standard	Bagasse lignin (80–3)	Aldrich lignin	Kraft lignin
1.	Stretch the phenolic O-H	3200–3550	3405.67	3436.62	3414
2	Aliphatic and aromatic stretch groups –CH–	2900	2919.70	2930.17	2926.01
3.	Stretch the arena–C=C	1500–1600	1511.92	1599.14	1614.42
4.	Amine C–N	1000–1250	1100		
5.	Alkyl C–H	600–700	650		

**Table 2.**  
*Comparison of the typical absorption peak wave numbers of bagasse lignin with commercial standard lignin FTIR spectrum by Aldrich and Kraft [35].*

Based on the reference, standard lignin consists of five main components, namely, phenolic O–H functional groups at wave number 3200–3550 cm<sup>-1</sup>, aliphatic and aromatic –CH– stretching groups at wave number 2900 cm<sup>-1</sup>, the C=C aromatic functional groups at wave number 1500–1600 cm<sup>-1</sup>, amine C–N, and alkyl C–H [37]. There are three main components that are the same as Aldrich lignin and Kraft lignin, namely, phenolic, aliphatic aromatic, and arenas.

In **Figure 7**, for the four curves that have a shape similar to the standard curve, curve “e” (colored black) shows peaks at phenolic, aliphatic, and aromatic wavelengths. So that based on the overlay of the FTIR results, it can be said that the most similar to the standard conditions is the “e” (black) curve which is the result of 80 mesh lignin isolation with 3-M NaOH reagent.

The selected lignin was then continued for the sulfonation process with several variations in the concentration of sodium bisulfite. The sulfonation process has been done with various variations in the concentration of sodium bisulfite and sulfonation time. The best results were achieved in the sulfonation process with a concentration of 0.25 M sodium bisulfite and a sulfonation time of 5 hours. Sulfonation process repeated three times and compare to find spectrum that compose liginosulfonate. The final result of the sulfonation process is liginosulfonate in the form of a light brown powder, as shown in the figure below **Figure 8 (Table 3)**.

From the result of FTIR test, liginosulfonate has been formed, indicated by difference a wavelength spectrum of liginosulfonates and a wavelength spectrum of lignin. The sulfonation process was done in 3 repetitions and the results were tested again by FTIR. With 3 repetitions of the process, the results are almost the same, so you



**Figure 8.**  
*Sodium liginosulfonate surfactant from bagasse.*

No.	Functional groups in the structure of lignosulfonates	Wave numbers (cm <sup>-1</sup> )		
		SLS standard (Patricia)	SLS standard (Aldrich)	SLS bagasse
1.	Stretch alkene =C=C	1630–1680	1608.34	1635.34
2.	Stretch Sulfonate S=O	1350	1365	1384.64
3.	Carboxylate C=O	1000–1300	1187.94	1114.64
4.	Ester S-OR	500–540	499.83	462.83

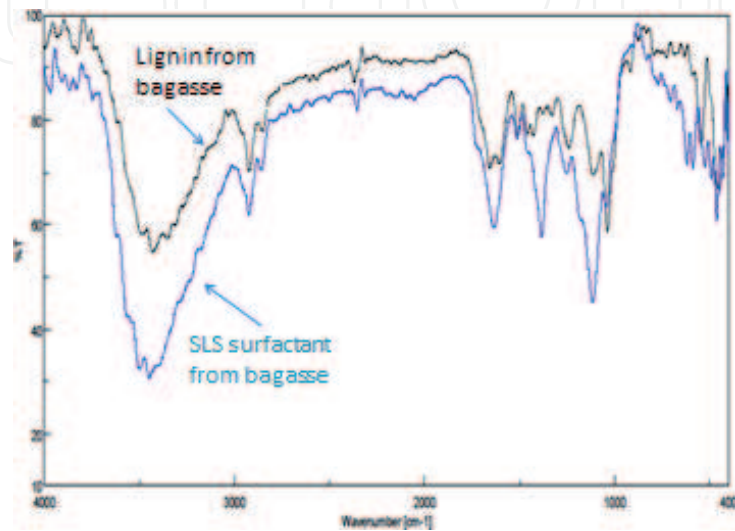
**Table 3.**  
*Comparison of the FTIR spectrum of SLS surfactant-synthesized bagasse and the FTIR spectrum of SLS standard Patricia and Aldrich.*

can say the process is correct. To ensure the perfect result of the sulfonation process, a comparison was made with other lignosulfonates [34]. The standard lignosulfonate used for comparison were SLS Aldrich and SLS Patricia. From the FTIR results, the spectrum of SLS surfactant synthesized bagasse and sodium lignosulfonate standard spectrum, the absorption peak and its wave number in the FTIR spectrum of SLS surfactant synthesized from bagasse showed conformity with the spectrum of FTIR standard. This shows that the sulfonation process of lignin to lignosulfonate has been successfully.

In **Figure 9**, it is clear that there is a difference between the FTIR results of lignin and surfactant, where on the blue curve line, as in the surfactant FTIR curve, there is a shift in the absorption peak that occurs, especially at a wavelength of 1635.34 cm<sup>-1</sup> as a function of the alkene group, at a wavelength of 1384.64 cm<sup>-1</sup> as a function of the sulfate group, at a wavelength of 1114.65 cm<sup>-1</sup> as a function of the carbolic acids group, and at a wavelength of 462.832 cm<sup>-1</sup> as the ester functional group.

Some of the peaks read on FTIR showed lignin and lignosulfonate bagasse components. The lignin component consists of phenolic functional group elements OH, aliphatic and aromatic groups –CH–, C=O ketone groups, arena functional groups –C=C–, CN amine groups, and CH alkyl groups with similarity values for standard spectrum wavelengths, such as those shown in **Table 4 (Figure 10)**.

Likewise for lignosulfonates, with indicator components consisting of C=C alkenes, sulfate S=O, C=O carboxylic acids, and S-OR esters, with spectrum



**Figure 9.**  
*Overlay of FTIR surfactant—Lignin from bagasse.*

Indicator	Component	Wavelength (cm <sup>-1</sup> )
Lignin	Phenolic O-H	3400
	Aliphatic and aromatic -CH-	2910
	Ketone C=O	1450
	Arena -C=C	—
	Amine C-N	1100
	Alkyl C-H	650
Lignosulfonates	Alkene C=C	1635.34
	Sulfate S=O	1384.64
	Carboxylic acids C=O	1114.65
	Ester S-OR	462.832

**Table 4.**  
*FTIR of lignin and lignosulfonate bagasse.*

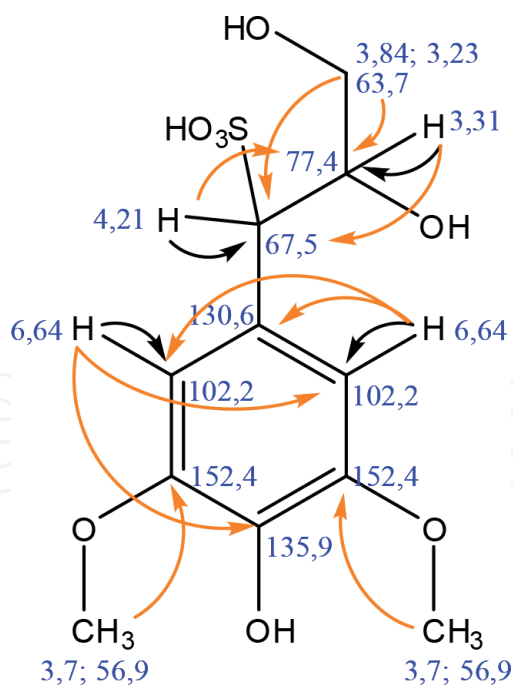


**Figure 10.**  
*Sugarcane becomes lignosulfonate [35].*

wavelengths close to the standard spectrum wavelength values. Lignin from bagasse can be completely synthesized into sodium lignosulfonate surfactant completely with lignosulfonate components consisting of alkene, sulfonate, carboxylate, and ester.

Furthermore, from the results of the NMR test, the components form the lignosulfonate. In the HMQC data, it can be seen that the proton nuclei are directly correlated with carbon-13 (13C) or have one bond (1J<sub>C, H</sub>) so that their own pairs can be known with certainty. The broad singlet signal on the  $\delta$  H 6.64 ppm chemical shift (2H, bs, H<sup>-3</sup>, and H-5) correlates directly with carbon at  $\delta$  C 102.2 ppm (C-3 and C-5). In addition, the HMQC spectrum also indicates the presence of methylene protons bound to C-9, methane bound to oxygen, and sulfate bound to C-8 and C-7, respectively.

From the HMBC spectrum, it can be seen that there is a correlation between protons and carbon with a distance of two bonds (2 J) to three bonds (3 J), which can be seen in **Figure 3**. From the HMBC data, it can be seen that there is a correlation between H-3 and H-5 with C-5/C-3, C-1, and C-7; H-7 correlates with C-8 and H-9 correlates with C-8 and C-7. These data support the existence of phenyl propanoid compounds as the basis for lignosulfonates [38]. The correlation between HMQC and HMBC can be seen in **Figure 11**. With the results that look like this, it shows that the isolation process of lignin from bagasse has been successful. Likewise, the sulfonation of lignin to lignosulfonate has also been successful.



**Figure 11.**  
 NMR test results—HSQC and HMBC correlation of bagasse lignosulfonate  $H_4S_4$  isolates.

## 4. Conclusions

Based on the results of the lignin sulfonation process on lignin sulfonation optimization, several conclusions can be drawn, namely:

1. Bagasse as biomass is a raw material that can be processed into lignosulfonate surfactants. The lignosulfonate obtained from bagasse is processed in two stages, namely, the lignin isolation process using sodium hydroxide and the sulfonation process using sodium bisulfite.
2. Based on the FTIR test, the lignin-forming components were shown by the presence of phenolic functional groups O–H, aliphatic –CH– and aromatic stretching groups, and C=O ketone functional groups, while the lignosulfonate-forming components were indicated by the presence of alkene groups, sulfate groups, and carbocyclic acids and ester functional groups, each with a spectrum wavelength corresponding to the standard spectrum.
3. Based on the results of the NMR test, the presence of phenyl propanoid compounds as the basis of the lignosulfonate compounds indicates that the sulfonation process has reached the expected target, namely, the formation of lignosulfonates completely.



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