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Characteristics of Lignin from Solid Waste Empty Palm Fruit Bunches (EPFB) using the Organosolv Method

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Abstract :
Oil palm empty fruit bunches are solid waste from the palm oil processing process that contains a significant amount of lignin and cellulose. Oil palm empty fruit bunches are widely found in Indonesia, especially in Riau Province. The processing of oil palm empty fruit bunches has not been effectively utilized. Therefore, the author isolated cellulose from oil palm empty fruit bunches using the organosolv method. The organosolv method is a type of isolation method that is environmentally safe than the kraft process, as it employs solvents in the form of organic chemicals, such as formic acid and acetic acid. This research was conducted by varying the ratio of the solvent solution using acetic acid and formic acid. Various solutions were employed, namely P1=20 ml formic acid+140 ml acetic acid, P2=40 ml formic acid+120 ml acetic acid, P3=60 ml formic acid+100 ml acetic acid, P4 = 80 ml formic acid+80 ml acetic acid, P5=160 ml formic acid, and P6=160 ml acetic acid. The best results were obtained in the P2 treatment with a variation of 40 ml formic acid + 120 ml acetic acid and obtained a high level of yield, namely in the P2 treatment it was 56.85%, the water content was 4.70%, the ash content was 4.84%, the acidity level (pH) was 4.67 and FTIR test results show wave peaks in treatments P3 and P6 28 waves.

Keywords: FTIR, isolation of lignin, organosolv, oil palm empty bunches

1. Introduction

Oil palm (Elaeis guineensis) is a plantation crop that dominates every region of Indonesia. The total area of oil palm plantations in Indonesia is recorded at 14,660,000 hectares, with Riau province being one of the regions with the largest area of oil palm plantations in Indonesia, namely 2,860,800 hectares (BPS, 2021). The size of the oil palm plantation is directly proportional to the amount of oil palm waste. The larger the oil palm plantation area, the greater the amount of waste produced. Solid waste from oil palm plantations consists of empty fruit bunches (EFB), fruit fibers, shells, tree trunks, and leaf sheaths. One type of waste from palm oil processing is empty fruit bunches, which account for 23–30% of fresh fruit bunches. Until now, empty fruit bunches have only been used as boiler fuel or compost (Ngadi and Lani, 2014).

Sudiyani et al., (2013) stated that empty palm fruit bunches (EPFB) contain cellulose (41–46.5%), hemicellulose (25.3–33.8%), and lignin (27.6–32.5%). The common use of EPFB today is as mulch in gardens, but the transportation costs per unit are quite high. Another use is as a raw material in the manufacture of organic fertilizer. EPFB is burned in incinerators so that the ash can be used as potassium fertilizer. However, the government has banned the incineration of EPFB due to its ineffectiveness in causing air pollution (Darnoko, 1995). Lignin, one of EPFB's chemical composition components, has potential for utilization.

Lignin is the main component of wood besides cellulose and hemicellulose. Lignin consists of polyphenol compound molecules that function as binders between wood cells, making them hard and rigid. Therefore, lignin can be used as an adhesive. Lignin has hydroxyl, carbonyl, and methoxy groups and has low solubility in water, making it potentially useful as an adhesive (Pramana et al., 2020). One method used in delignification is the organosolv method. The organosolv method is a way of separating fibers using organic

chemicals such as methanol, ethanol, acetone, acetic acid, and formic acid. This method is efficient, environmentally friendly, and produces optimal results (Taradita, 2013). This study used the organosolv method with acetic acid and formic acid.

Suhartati et al (2016), in their study of the physical and chemical properties of empty palm fruit bunches using the organosolv method with ethanol as the solvent, obtained the best lignin results, namely a moisture content of 3.07%, an ash content of 30.34%, and a lignin purity of 64.64%. Then, Mondylaksita *et al*., (2020) employed an organosolv method using ethanol acid-catalyzed as the solvent to recover high-purity lignin from empty palm fruit bunches, applying cooking time variations of 30, 60, 90, and 120 minutes under specific conditions. The best result was obtained at a cooking time of 90 minutes, yielding a lignin recovery of 64.94 ± 1.09% with a purity of 70.6 ± 4.9%. Next, Pramana et al., (2020) conducted research on the physical characteristics of lignin from EPFB fibers using the organosolv method with a cooking solution composition of acetic acid and formic acid at 60 °C, 85 °C, 100 °C, and 121 °C. The results showed that the highest treatment was obtained at of 85 °C with a lignin yield of 15.87%.

Although previous studies have explored the organosolv method using variations in solvents, temperature, or cooking time, only a limited number of investigations have specifically examined the effect of different ratios of formic acid and acetic acid cooking solutions under strictly controlled conditions. In fact, these two solvents possess distinct capabilities in breaking lignocellulosic bonds, and therefore their combination and proportions may potentially yield more optimal lignin quality and recovery. This research gap forms the basis for the necessity of the present study, which aims to complement and extend earlier findings on optimizing lignin isolation from empty palm fruit bunches (EPFB) using the organosolv method.

1. Method

Research Design

The method used was an experimental one with a non-factorial Completely Randomized Design (CRD) consisting of 6 treatments and 3 replicates, resulting in 18 experimental units. The treatments in this study were modified from the study by Pramana et al., (2020). The study combined formic acid cooking solution with acetic acid as the variable. This study used two variables, namely fixed variables and independent variables, where the fixed variables were temperature, cooking time, and catalyst concentration (HCl). The independent variable was the ratio of the cooking solution, while the fixed variables were the cooking temperature of 85℃, catalyst concentration (1% HCl), and cooking time (4 hours). The following are the cooking solution ratios used:

P1 = 20 ml formic acid + 140 ml acetic acid
P2 = 40 ml formic acid + 120 ml acetic acid
P3 = 60 ml formic acid + 100 ml acetic acid
P4 = 80 ml formic acid + 80 ml acetic acid
P5 = 160 ml formic acid
P6 = 160 ml acetic acid

Table 1. Lignin Isolation Formulations

Ingredient (g)	Amount					
	P_1	P_2	P_3	P_4	P_5	P_6
Formic acid	20	40	60	80	160	-
Acetid acid	140	120	100	80	-	160
H_2O	40	40	40	40	40	40
Total	200	200	200	200	200	200

Time and Location of the Study

This research was conducted at the Agricultural Product Processing Laboratory, Agricultural Product Analysis Laboratory, Faculty of Agriculture, and Science and Materials Laboratory, Faculty of Mathematics and Natural Sciences, as well as the Analytical Chemistry Laboratory, Faculty of Engineering, University of Riau, Pekanbaru, for three months.

Materials and Tools

The main raw material used is empty palm fruit bunches (EPFB), while the chemicals used for isolation and analysis are HCl, formic acid, acetic acid, and H_2O, as well as ethanol/alcohol, H_2SO_4, pH paper, and distilled water. The equipment used for material preparation includes a machete, a large tray for drying materials, a grinder, and an 80 mm sieve. The equipment used for product analysis includes a hot plate, round-bottom flasks, a condenser, a 400–600°C oven, a water bath, aluminum cups, porcelain cups, and other analytical tools.

Research Procedure

Fiber Production

This stage of research was conducted to obtain fiber from empty palm fruit bunches (EPFB) produced by the palm oil industry. To obtain the fiber, the EPFB were cleaned of any remaining palm fruit skin and then dried in the open air (sunlight) for one week. The dried EPFB fibers are cut into small pieces to be ground using a grinding machine and then sieved with an 80 mesh sieve.

Lignin Isolation

The EPFB powder sample is then mixed with a cooking solution consisting of formic acid, acetic acid, and water (according to the treatment) and 0.1% HCl catalyst is added to the container. Cooking is carried out at a temperature of 85℃ and maintained at the maximum temperature for 4 hours. After cooking, filtering is carried out to separate the fiber from the black liquor. The black liquor was then concentrated using a rotary vacuum evaporator for 3 hours, after which it was dried in an oven to obtain lignin.

Observation

Moisture Content

The determination of moisture content was based on Sudarmadji et al., (1997). A 2 g sample is weighed and placed in a porcelain dish of known weight. Before use, the porcelain dish is dried in an oven at 100°C for 10 minutes. The dish and sample are then dried in an oven at 105°C for 2 hours, cooled in a desiccator for 20 minutes, and weighed. The sample and dish were heated again in an oven for 30 minutes, cooled again, and weighed. This treatment was repeated until a constant weight was obtained (the difference between consecutive weighings was less than 0.2 mg). The moisture content was then calculated using the formula:

$$\text{Moisture content} = \frac{\text{Initial weight} - \text{Final weight}}{\text{Sample weight}} \times 100\%$$

Ash content

The determination of ash content refers to Sudarmadji et al., (1997). The determination of ash content is closely related to the mineral content of a material. A sample weighing 2 g is placed in a porcelain dish of known weight. Before use, the porcelain dish is dried in an oven at a temperature of approximately 100℃ for 10 minutes. The sample in the porcelain dish is then incinerated in a furnace at a temperature of 600°C for 2 hours until the sample turns into a whitish ash, then cooled in a desiccator for 30 minutes and weighed. The ash content is calculated using the formula:

$$\text{Ash content} = \frac{\text{Ash weight}}{\text{Sample weight}} \times 100\%$$

Yield

Yield measurement refers to the National Standardization Agency (2006). In this study, lignin isolate was obtained from the filtrate of palm kernel shell powder extraction. The filtrate is a cellulose precipitate whose yield will be measured. The precipitated filtrate was then separated and concentrated to obtain lignin. The calculation of cellulose and lignin yield refers to the weight of the raw material (empty palm fruit bunches) before pretreatment (cutting, sorting, sun drying, grinding). The formula for cellulose and lignin yield is as follows:

$$\text{Yield} = \frac{\text{Weight of filtrate}}{\text{Weight of empty palm fruit bunches}} \times 100\%$$

Acidity (pH)

The determination of acidity (pH) refers to Muchtadi et al., (2010), using a pH meter. Before measuring the pH of transparent soap, the device was first calibrated using pH 4 and 9 buffer solutions. pH measurements were taken before and after the EPFB cooking process at varying organic acid compositions (according to treatment). The numbers shown on the pH meter represent the pH of the cellulose and lignin isolation solution using the organosolv method.

Chemical Structure (FTIR)

This chemical structure analysis was used to identify the lignin structure and observe changes in specific functional groups in lignin at each given stage, namely the spectrum of raw materials and lignin resulting from hydrolysis using the organosolv method. The KBr 1 ultra-thin pallets method was used to obtain the IR spectrum. The samples were ground and mixed with KBr (sample/KBr ratio, 1/100) to prepare the pellets. The experiments were conducted in the range of 500–4000 cm-1 with a resolution of 4 cm-1 and a total of 32 scans for each sample.

Data Analysis

The data obtained were analyzed statistically using variance analysis with a completely randomized design. If the data showed Fcount > Ftable, a follow-up test was conducted using Duncan's Multiple Range Test (DMRT) at a 5% level to determine the differences in each treatment. The software used for data analysis was IBM SPSS Statistics.

2. Results and Discussion

Moisture Content

Moisture content is the water content found in a material. The principle of determining moisture content is based on weighing the sample. The results of the analysis of variance show that the variation in the cooking solution has a significant effect on the moisture content of the lignin produced. The average moisture content of lignin after further testing with DMRT at 5% can be seen in Table 2.

Table 2. Average Moisture Content of Empty Palm Fruit Bunches

Treatment	Moisture Content (%)
P1 = 20 ml formic acid + 140 ml acetic acid	4,70[a]
P2 = 40 ml formic acid + 120 ml acetic acid	6,26[c]
P3 = 60 ml formic acid + 100 ml acetic acid	6,30[c]
P4 = Formic acid 80 ml + acetic acid 80 ml	6,37[c]
P5 = Formic acid 160 ml	6,84[d]
P6 = Acetic acid 160 ml	5,30[b]

Note: Numbers followed by different lowercase letters indicate significant differences according to the DMRT post-hoc test at the 5% level.

Based on Table 2, it can be seen that the water content obtained in this study shows that treatment P1 is significantly different from each treatment, while procedures P2, P3, and P4 are significantly distinct from treatments P1, P5, and P6. The addition of formic acid and acetic acid solutions will affect the water content of the lignin produced. This is because the organic acid solution used is polar, which can affect the moisture content obtained.

Based on Table 2, it can be explained that the moisture content of empty palm fruit bunches using the organosolv method obtained values in the range of 4.70-6.84%. The high and low moisture content is due to lignin being hydrophobic (water-resistant), which inhibits lignin from easily binding water, so that the higher the lignin content in the empty fruit bunches, the lower the moisture content (Suhartati et al., 2016). This is because the addition of formic acid and acetic acid causes lignin to dissolve in the cooking solution, thereby affecting the water content.

Based on Table 2, it can be seen that the lowest moisture content was obtained in treatment P1 with a cooking solution of 20 ml formic acid + 140 ml acetic acid, resulting in a moisture content of 4.70%, while the highest moisture content was obtained in treatment P5 with a cooking solution of 160 ml formic acid, resulting in a moisture content of 6.48%. The high and low moisture content produced was caused by the cooking solution used, namely formic acid and acetic acid, as well as the material used, namely empty palm oil bunches containing lignin.

Ash Content

Ash content refers to the inorganic residue remaining after the combustion of an organic material. The ash content is influenced by the type of raw material used. The results of the analysis showed that the variation in the cooking solution, namely formic acid and acetic acid, had a significant effect on the ash content of the lignin produced. The average lignin ash content after further testing with DMRT at 5% can be seen in Table 3.

Table 3. Average Ash Content of Empty Palm Fruit Bunches

Treatment	Ash Content (%)
P1 = 20 ml formic acid + 140 ml acetic acid	6,67[d]
P2 = 40 ml formic acid + 120 ml acetic acid	6,44[d]
P3 = 60 ml formic acid + 100 ml acetic acid	6,30[c]
P4 = Formic acid 80 ml + acetic acid 80 ml	5,10[a]
P5 = Formic acid 160 ml	4,84[a]
P6 = Acetic acid 160 ml	5,85[b]

Note: Numbers followed by different lowercase letters indicate significant differences according to the DMRT post-hoc test at the 5% level.

Based on Table 3, it can be seen that the ash content obtained from this study in treatment P1 was not significantly different from treatment P2, but was significantly different from treatments P3, P4, P5, and P6. Treatment P2 was not significantly different from treatment P1, but was significantly different from treatments P3, P4, P5, and P6. Treatment P3 was significantly different from treatments P1, P2, P4, P5, and P6. Treatment P4 was significantly different from P1, P2, P3, and P6, but similar to P5. Treatment P5 was significantly different from treatments P1, P2, P3, and P6, but similar to treatment P4. Treatment P6 was significantly different from all treatments.

Based on Table 3, it can be explained that the lignin ash content of empty palm fruit bunches using the organosolv method yielded values ranging from 4.84 to 6.67%. The addition of cooking solutions such as formic acid and acetic acid had a significant effect on the ash content of empty palm fruit bunches. According to Feringgo (2019), the use of formic acid solvent and a small amount of acetic acid resulted in a lower ash content in the material. This is because the use of cooking solutions in the form of acetic acid and formic acid affects the ash content produced, so the addition of cooking solutions in the form of formic acid and acetic acid has a significant effect on the ash content of empty palm fruit bunches.

Formic acid and acetic acid solutions in the organosolv method can help remove minerals and ash from lignin. The raw materials used contain high mineral content, such as empty palm fruit bunches. This is in line with Wanrosli et al., (2007), who found that empty palm fruit bunches have a fairly high ash content, so the lignin produced from empty palm fruit bunches has a relatively high ash content. The acids used can dissolve minerals and facilitate their separation from lignin during the extraction process. Several factors influence the ash content produced, such as acid concentration, temperature, and contact time, which can also affect ash removal efficiency during the organosolv process (Ancastami et al., 2020).

Yield Analysis

Yield is the ratio of the weight of the extract produced to the weight of the sample as raw material. A higher yield value indicates that more extract is produced (Dewatisari et al., 2018). The results of the analysis of variance show that the variation in the cooking solution has a significant effect on the lignin yield produced. The average lignin yield after further testing with DMRT at 5% can be seen in Table 4.

Table 4. Average Lignin Yield of Empty Palm Fruit Bunches

Treatment	Yield (%)
P1 = 20 ml formic acid + 140 ml acetic acid	50,67[c]
P2 = 40 ml formic acid + 120 ml acetic acid	56,85[d]
P3 = 60 ml formic acid + 100 ml acetic acid	48,32[b]
P4 = Formic acid 80 ml + acetic acid 80 ml	43,60[a]
P5 = Formic acid 160 ml	38,24[a]
P6 = Acetic acid 160 ml	49,50[bc]

Note: Numbers followed by different lowercase letters indicate significant differences according to the DMRT post-hoc test at the 5% level.

Table 4 shows that the yield values for treatment P1 are significantly different from those for treatments P2, P3, P4, and P5. Treatment P3 is similar to treatment P6, and treatment P4 is similar to treatment P5. The results of this study show that the highest yield value was obtained in treatment P2, which used 40 ml of formic acid + 120 ml of acetic acid, with a yield value of 56.85%, while the lowest yield value was obtained in treatment P5, which used 160 ml of formic acid, with a yield value of 38.24%. Based on the results shown in Table 4, it can be explained that the use of formic acid and acetic acid can affect the yield of lignin from empty palm fruit bunches. This impact is thought to be due to the chemical hydrolysis of polysaccharides during the cooking process (Hidayati et al., 2017).

According to Wanrosli et al., (2007), the amount of formic acid used can dissolve lignin into organic solvents. Based on treatment P5, with 160 ml of formic acid as the cooking solution, the lowest result was obtained with a value of 38.24%. Based on this, it can be explained that formic acid and acetic acid play an important role as solvents in the organosolv method. Both can dissolve lignin, preserve it from raw materials, and separate it from cellulose and other components.

The use of formic acid and acetic acid in accordance with the requirements of the organosolv method will help obtain lignin efficiently. The use of an appropriate formic acid and acetic acid solution occurs in treatment P2, where formic acid and acetic acid are mixed in a ratio of 40:120. Both have a very effective role so that a high lignin yield can be obtained. Hidayati et al., (2017) state that an increase in chemical concentration will decrease pulp yield. The higher the concentration of chemicals used in the cooking solution, the lower the yield of the pulp produced.

Acidity Analysis

Acidity (pH) analysis was conducted before and after treatment, where before treatment, namely cooking solution variation, the acidity level of empty palm fruit bunches was 7.8-8 (Harahap et al., 2020). The analysis of variance showed that the variation in cooking solution had no significant effect on the pH value of the lignin produced. The average pH value of palm oil empty fruit bunches (EPFB) can be seen in Table 5.

Table 5. Average pH of Empty Palm Fruit Bunches

Treatment	pH
P1 = 20 ml formic acid + 140 ml acetic acid	3,33
P2 = 40 ml formic acid + 120 ml acetic acid	4,67
P3 = 60 ml formic acid + 100 ml acetic acid	4,33
P4 = Formic acid 80 ml + acetic acid 80 ml	3,00
P5 = Formic acid 160 ml	4,00
P6 = Acetic acid 160 ml	3,67

Note: Numbers followed by different lowercase letters indicate significant differences according to the DMRT post-hoc test at the 5% level.

Table 5 shows the average pH scores of cellulose with a range of 3.00-4.67. The average pH values indicate that there are no significant differences between the treatments (P1, P2, P3, P4, P5, and P6). This is because the pH values of empty palm fruit bunches (EPFB) are almost the same in each treatment. The highest pH value was obtained in treatment P2, which used 40 ml of formic acid + 120 ml of acetic acid as a solvent,

with a pH value of 4.67. After treatment with the cooking solution, namely formic acid and acetic acid, the pH value of palm oil empty fruit bunches tended to be acidic, ranging from 3.00 to 4.67. This variation was due to the cooking solution used. The use of an organic acid cooking solution caused the pH value of the resulting palm oil's empty fruit bunch lignin to decrease, making it acidic.

The use of the organosolv method with acidic cooking solutions, such as acetic acid and formic acid, causes a decrease in the pH of empty palm fruit bunches, resulting in pH values that are not significantly different. In addition, the use of temperature and cooking time also affects the acidity level. This is because the longer the cooking time, the more the acid solution penetrates into the empty palm fruit bunches, resulting in acidic lignin. Not only that, the low pH value obtained through isolation is due to the washing process because the use of ethanol can only clean the remaining lignin but cannot restore the pH caused by the lignin isolation process, so the pH obtained is acidic.

FTIR Chemical Analysis

Fourier transform infrared spectroscopy (FTIR) analysis of lignin is a common technique used to characterize the chemical structure of lignin molecules. Lignin is a complex aromatic polymer found in plant cell walls along with cellulose and hemicellulose. Fourier-transform infrared (FTIR) chemical analysis can be used to observe any changes in a molecular bond qualitatively, but this method is only done under certain conditions, so it can be used as a basis for analytical testing information that can be modified in the use of raw materials. The following are the FTIR results of lignin from empty palm oil fruit bunches in treatment P1, as shown in Figure 1.

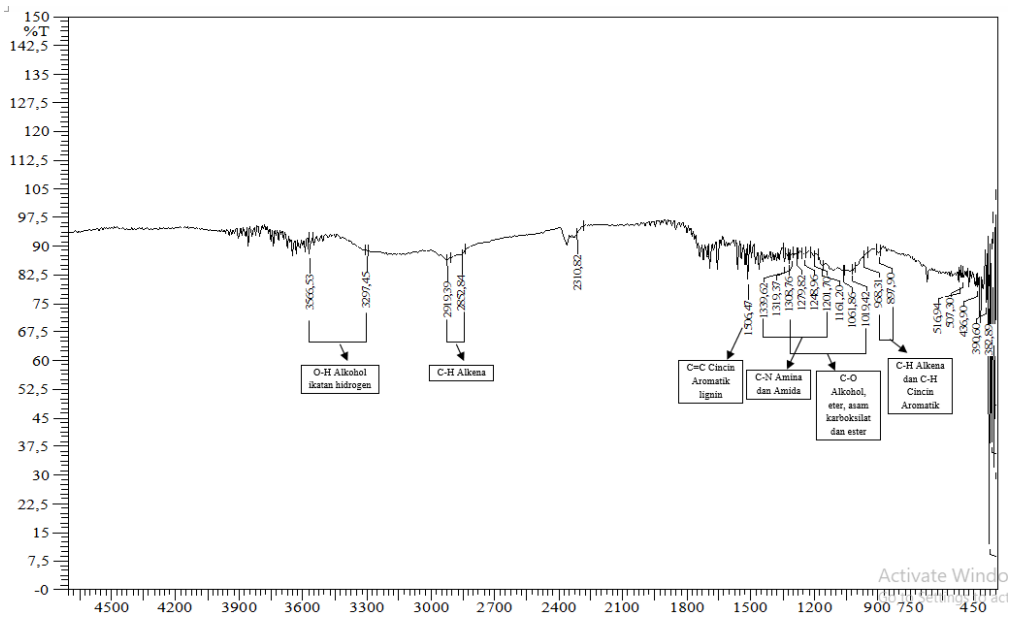

Figure 1. FTIR graph of lignin P1

The image in treatment P1 shows a variation of 20 ml formic acid + 140 ml acetic acid cooking solution. Lignin content can be analyzed based on the absorption of lignin aromatic ring functional groups (C = C). The C = C group appears at a wavelength peak of 1506 cm^{-1}. Not only that, at a wavelength of 897–968 cm^{-1}, the C-H group, which is an alkenic and aromatic ring in nature, can be seen. At a wavelength peak of 3297–3566 cm^{-1}, the O–H group, which has hydrogen bond alcohol properties, can be seen. The C-O group, which is an alcohol, also appears at the 1019-1308 cm-^{1} wavelength peak. Not only that, the C-N amine and amide groups are also visible at the 1201-1339 cm^{-1} wavelength peak. Based on this information, it is clear that Figure 4 represents lignin, which contains aromatic ring groups that are its main components. Next is the FTIR graph of empty palm oil bunches in treatment P2, which can be seen in Figure 2.

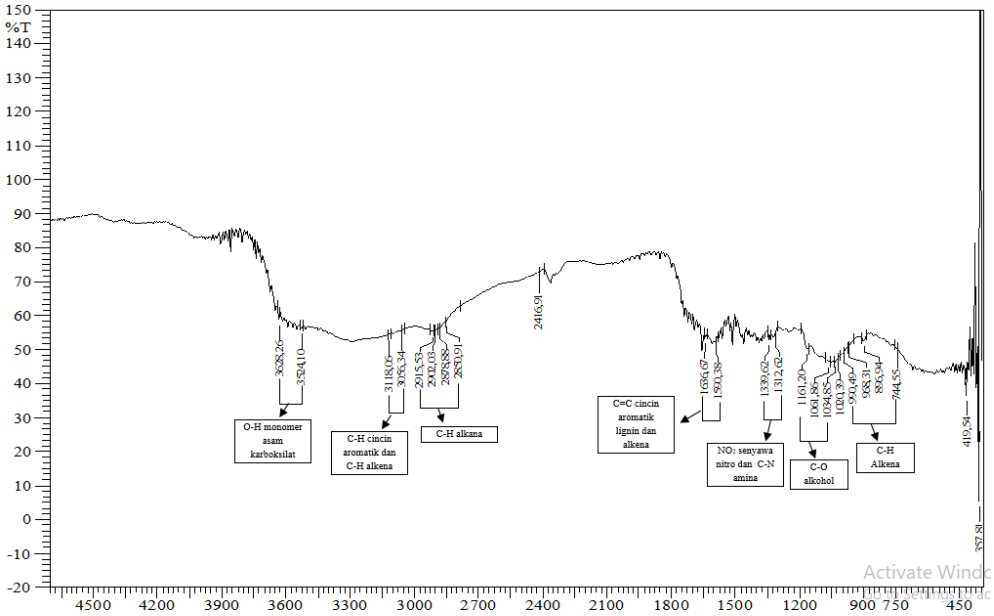

Figure 2. FTIR graph of lignin P2

It can be seen that the lignin graph results in treatment P2 with a variation of 40 ml formic acid + 120 ml acetic acid cooking solution have a wavelength peak that indicates the presence of an alkena C-H group with a wavelength peak of 744-990 cm^{-1}. followed by a C-O group with a wavelength of 1034-1161 cm^{-1}, and then a NO_2 group with nitro compound properties and an amine C-N group appearing at a wavelength peak of 1312-1339 cm^{-1}. There is also a C=C group, which is an aromatic ring compound of lignin with a wavelength of 1590-1636 cm^{-1}. Not only that, but C-H groups with alkane compounds also appear at a wavelength peak of 2850-2915 cm^{-1}. C-H groups are also visible, but they have aromatic ring and alkene compounds, which appear at a wavelength peak of 3056-3118 cm^{-1}, hydroxyl O-H groups are also visible with an absorption wavelength of 3524-3628 cm^{-1}. The FTIR P2 graph shows variations in the cooking solution consisting of 40 ml of formic acid + 120 ml of acetic acid. In this case, it can be seen that the addition of formic acid and the reduction of acetic acid in the cooking solution will affect the lignin yield obtained. This variation is indicated by the difference in the number of functional group peaks seen in the resulting IR.

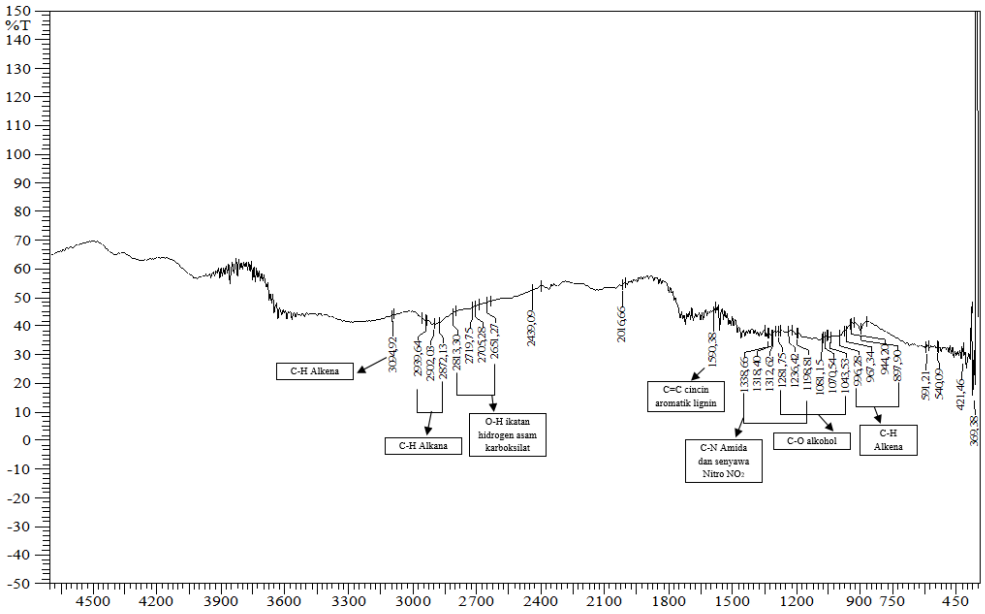

Figure 3. FTIR graph of lignin P3

The IR results of lignin shown in Figure 3 are the P3 treatment with a variation of 60 ml formic acid + 100 ml acetic acid cooking solution, which can be seen at the wavelength peak of 897-996 cm^{-1}, indicating the presence of C-H, which has an alkene compound type. In addition, absorption waves with C-O alcohol groups appear in the absorption region of 1043-1281 cm^{-1}. C-N groups with amine compounds and NO_2, which are nitro compounds, are seen at the wave peaks of 1198-1338 cm-1. Treatment P3 with isolated empty palm oil bunches also shows C=C groups with aromatic ring lignin compounds appearing at a wavelength of 1590 cm^{-1}. The O-H functional group, which is a hydrogen bond, is visible at a wavelength peak of 2651-2813 cm^{-1}, while at a wavelength peak of 2872-2939 cm^{-1}, C-H groups with alkane properties are also visible, as well as C-H groups with alkene properties, but with a different absorption length, namely at a wavelength peak of 3094 cm^{-1}. Based on this, the result is lignin, which has aromatic ring functional groups.

The many other groups found in the graph are impurities contained in the lignin produced. The addition of formic acid and the reduction of acetic acid in the solution will affect the lignin results obtained. While it may exhibit many wavelengths, it also contains numerous other impurity components. Lignin compounds are generally identified by the presence of several constituent groups, such as O-H, aromatic C-H, aromatic C=C ring, and alkane and alkene C-H groups.

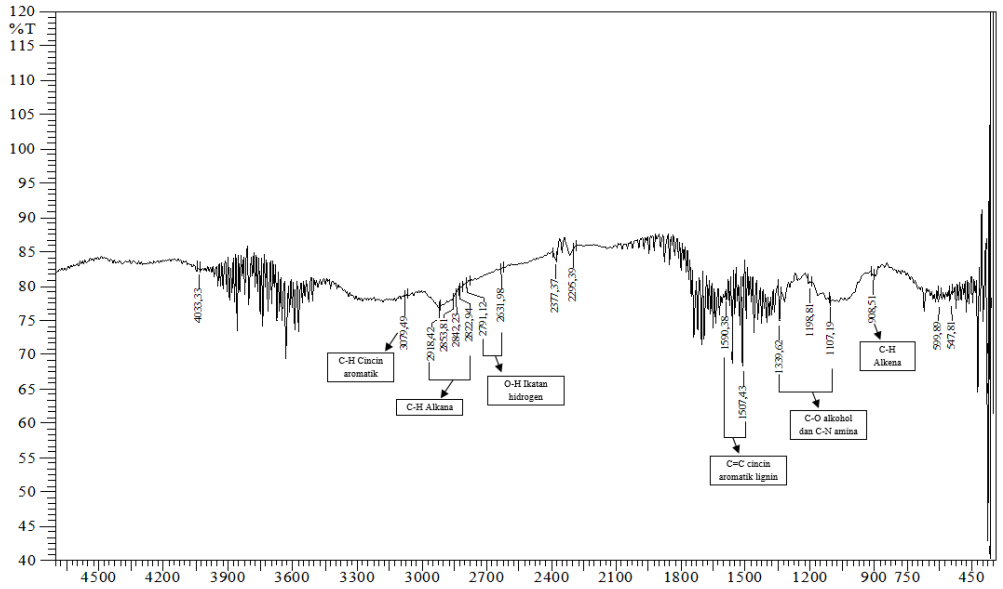

Figure 4. FTIR graph of lignin P4

Treatment P4 with 80 ml of formic acid + 80 ml of acetic acid showed a wavelength peak of 885-983 cm^{-1}, which is a C-H group with an alkene compound type and is a C-H with an aromatic ring property. A C-O group with an alcohol compound type and C -N groups of amine compounds at the 1107-1339 cm^{-1} wavelength peak, as well as C=C aromatic ring groups of lignin at the 1507-1590 cm^{-1} wavelength peak, and C-H groups of alkane compounds appearing at the 2631-2791 cm^{-1} wavelength peak. C-H groups with alkenes appear in the 2822-2918 cm^{-1} wavelength peak, and C-H groups, which are aromatic ring compounds, reappear in the 3079 cm^{-1} wavelength peak.

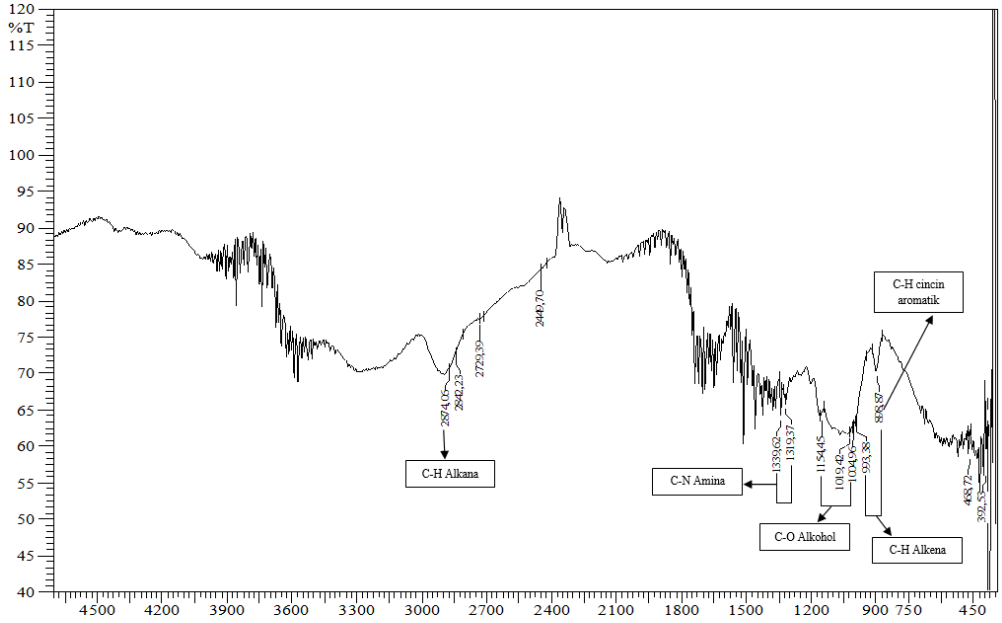

Figure 5. FTIR graph of lignin P5

Treatment P5 with a cooking solution variation of 160 ml formic acid shows a difference from the other graphs. This variance is because the use of different cooking solutions affects the FTIR peak results. Based on Figure 5, it can be seen that the C–H group, which is an alkene compound and has an aromatic ring, is present at the 808–993 cm^{-1} wavelength peak. followed by C–O groups with alcohol compounds appearing at a wavelength peak of 1004–1154 cm^{-1}, and C–N groups with amine compounds appearing at a wavelength peak of 1319–1339 cm^{-1}. Based on this, it can be explained that in treatment P5 with a variation of 160 ml of formic acid cooking solution, there is no evidence of lignin components. This phenomenon is because only aromatic C–H is visible in FTIR, while functional groups such as C=C and O–H are not yet visible. The absence of lignin functional groups is due to the excessive use of formic acid, which causes lignin to dissolve into the cooking solution. The addition of a cooking solution will affect the amount of lignin degraded, and if the addition is excessive, it will accelerate the cooking time (Kusumo et al., 2020).

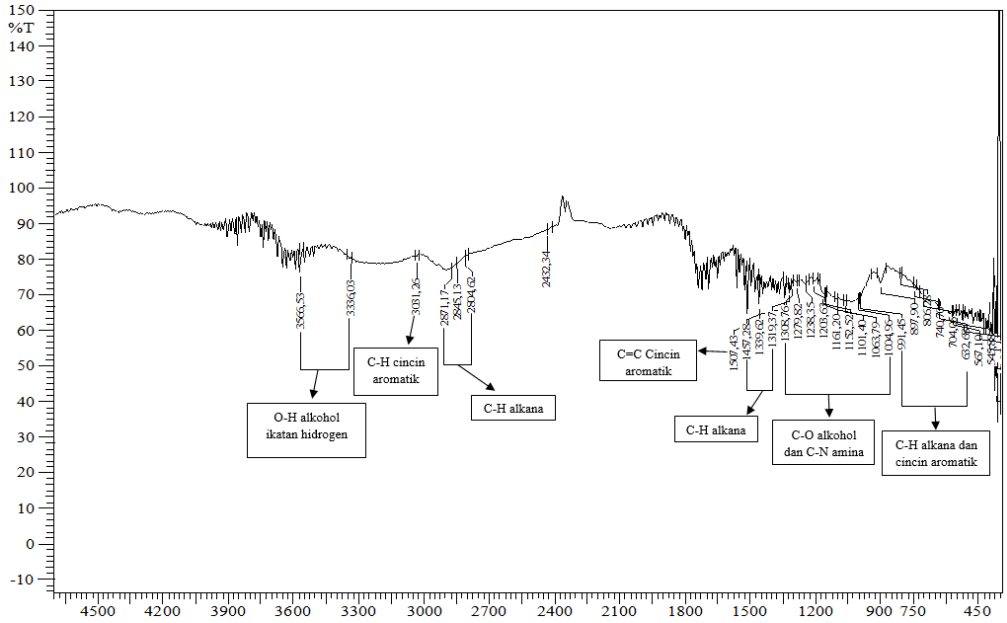

Figure 6. FTIR graph of lignin P6

The FTIR graph of lignin in treatment P6, with 160 ml of acetic acid, shows the presence of C–H functional groups with alkenes and aromatic compounds appearing at 632–991 cm^{-1}, as well as C–O groups with alcohol compounds and C–N amine compounds visible at the 1004–1308 cm^{-1} wavelength peak. There was also a C=C functional group with a lignin aromatic ring compound type that appeared at a wavelength of 1507 cm^{-1}, followed by a C–H group with an alkane compound type that appeared at a wavelength peak of 2804–2871 cm^{-1}. This was followed by another C–H group, but this time in an aromatic compound, at a wavelength peak of 3031 cm^{-1}, as well as an O–H group with a hydrogen bond at a wavelength peak of 3336–3566 cm^{-1}.

Based on graphs P1, P2, P3, P4, P5, and P6, it can be seen that there are differences in the number of wave peaks that appear in each group. It can be observed that treatment P5 has the fewest wave peaks compared to the others. This can be explained by the fact that the addition of formic acid affects the shape of the resulting graph, indicating that the use of excess formic acid will degrade the lignin into solution so that the resulting lignin is not optimal (Kusumo et al., 2020). Kusumo et al., (2020) also stated that, in general, FTIR results characterized by the presence of lignin must contain functional groups such as aromatic C–H, aromatic ring C=C, O–H hydrogen bonds, and methyl C–H. The lignin isolates obtained in this study can be said to have produced standard lignin compounds, based on the relevance of the general functional groups found in lignin. Therefore, it can be concluded that these compounds are indeed lignin (BSN, 2015). As can be seen in Table 6.

Table 6. Identification of FTIR Functional Groups of Lignin

Wavenumber (cm^{-1})						Standard Absorption Band Range (cm^{-1})	Information
1	2	3	4	5	6		
897	744–896	897	–	898	632–897	690–900	C–H aromatic
2852–297	2850–2519	2872–2939	–	2874	2804–2871	2850–2970	C–H alkane
1201–1339	1312–1339	1198–1281	1107–1339	1319–1339	1004–1339	1180–1360	C–N stretching
3297–3566	3524	–	–	–	3336–3566	3200–3600	O–H stretching
1019	1061–1161	1043–1281	1107–1339	1004–1154	1004–1308	1050–1300	C–O ether
1506	1590	1590	1507–1590	–	1507	1500–1600	C=C aromatic ring

Treatments P1, P2, and P6 can be considered lignin because they meet the established standards. Next, the number of wave peaks that have been observed as lignin functional groups can be calculated. Treatment P1 has 22 wave peaks that are part of lignin compounds, while treatment P2 has 22 wave peaks that are part of lignin compounds, and treatment P3 has 28 wave peaks that are part of lignin but lack O-H functional groups with hydrogen bonding properties. Furthermore, treatment P4 has 18 wave peaks that are compounds included in the characteristics of lignin. Treatment P5 has 13 wave peaks that are included in the characteristics of lignin, while treatment P6 has 28 wave peaks that are part of the characteristics of lignin. This variance is due to variations in the use of formic acid and acetic acid, resulting in significant differences. In line with Rambe et al. (2020), during cooking, a rapid reaction occurs where the carbohydrate lignin bonds are broken, causing the released lignin to dissolve in the cooking solution, as well as a slow reaction where condensation and repolymerization occur, causing the lignin to be insoluble in the cooking solution. The appearance of several absorbance peaks, in addition to the general groups, is caused by multiple factors, including the imperfect purity of lignin, which leads to the presence of various impurity groups within the lignin isolate. This results in significant variations in the structure and composition of lignin, as well as differences in lignin measurement techniques depending on the solvent used.

3. Conclusion

The conclusion that can be drawn is that variations in the ratio of cooking solutions using the organosolv method had a significant effect on water content, ash content, and yield, but no significant effect on pH values. The best ratio of cooking solution was found in treatment P2 (40 ml formic acid + 120 ml acetic acid), which produced the highest yield of 56.85%. The lowest moisture content was obtained in treatment P1 (4.70%), while the lowest ash content was observed in treatment P5 (4.84%). The acidity level (pH) in treatment P2 was 4.67. The FTIR analysis showed absorption peaks in treatments P3 and P6 at 28 wave numbers. An increase in formic acid and a decrease in acetic acid reduced lignin production, as indicated by the FTIR chemical analysis.

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