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To cite this article: Farhana Sultana Toma *et al* 2021 *IOP Conf. Ser.: Mater. Sci. Eng.* **1092** 012027

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Comparison between lignin extraction by alkaline and ultrasound-assisted alkaline treatment from oil palm empty fruit bunch

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Abstract. Lignin is the second most abundant terrestrial biopolymer after cellulose and is the largest renewable source of aromatic groups in nature. In this work, the study was carried out on the selective extraction of lignin from oil palm empty fruit bunch (OPEFB) using alkaline and ultrasound-assisted alkaline methods. OPEFB is available in high amounts as solid waste of palm oil industries, which is considered as lignocellulosic and has fairly high lignin content. The pretreatment of OPEFB for lignin production depends on the effect of particle size, liquid to solid ratio, the concentration of NaOH, reaction temperature, type of solvent, and sonication time. OPEFB was treated with 7.5%, 10%, and 15% NaOH at 90°C for 1.5h without ultrasound irradiation and with ultrasonic time for 10, 20, 30, and 40 min. Extracted lignin was characterized by FT-IR, UV-Vis, TGA, and elemental analysis. Considerably improved results were obtained by using the ultrasound-assisted time for 30 min. The percentage yield and purity of lignin extracted by the ultrasound-assisted alkali method was higher than that lignin fraction which was achieved by the alkali method, and their purity was improved with the addition of sonication time from 10 to 30 min.

Keywords: Oil palm empty fruit bunch (OPEFB), Lignin extraction, Alkaline pretreatment, Ultrasound-assisted treatment, Irradiation time, Yield % of lignin.

1. Introduction

Lignocellulosic biomass consists mainly of three polymers; cellulose, hemicellulose, and lignin together with small amounts of other components, like acetyl groups, minerals, and phenolic substituents. Among the three macromolecular components (cellulose, hemicellulose, and lignin), cellulose is the major component found in all plant cell walls [1]. There is a wide range of cellulose and lignin sources available including jute, cotton, OPEFB, hemp, pine straw, wheat straw, wood pulp, alfalfa, etc [2].

In addition to the low value-added lignin application, a wide variety of high-value industrial applications has been industrially recognized or established including uses as novel resources, polymeric, oligomeric, and monomeric feed stock. Lignin can be used in a wide range of applications,



such as the use of lignin in textile, agriculture, binders, adhesive, dispersants and composites. In recent years, lignin has been exploited in several polymer composite uses, for example, lubricants, plasticizers, stabilizing agents, surfactants, coatings, carbon fibre etc. Therefore, it is very important to extract lignin from different sources. Hence, the physical and chemical behavior of lignin will be changed with respect to the original source and extraction method used [2, 3]. Although many studies have been reported about extraction of lignin [3] but very few works has been done using ultrasound assisted alkaline method [4].

Oil palm (*Elacis guineensis*) is the prevailing cultivated crop in many countries, especially Malaysia and Indonesia being the two largest growers and producers of oil palm [5]. Palm fiber is produced from oil palm's vascular bundles in the empty fruit bunch (EFB). In Malaysia, about 15 million tons of EFB are produced per year and the large quantities of these residues are unutilized, which can be further used as raw material and convert these materials into value-added products have gained great interest, particularly in the papermaking industry and fiberboard industry [6]. Various types of biomass can be obtained from the oil palm plantation and palm oil mills, such as oil palm empty fruit bunch (OPEFB), oil palm fronds (OPF), oil palm mesocarp fiber (OPMF), oil palm trunks (OPT), leaves and roots. OPEFB is the biomasses waste from the crude palm oil industry and it has a molecular structure composed of 30–40 %wt of cellulose, 20–25 %wt hemicellulose, and 20–35 %wt of lignin, containing also some quantities of pectin, protein, extractives (nonstructural sugars, nitrogenous material, and waxes) and ash [7].

Lignin is a polymeric factor in biomass, portion as a linker between cellulose bundles and working to provide strength and severity to the cell walls. The structure of lignin is amorphous and highly branched by carbon-carbon bonds and ether bonds forming with three phenylpropanolic monomers (monolignols): p-Coummaryl alcohol, Coniferyl alcohol and Sinapyl alcohol [8]. Based on the structure of lignin and the corresponding properties, study on value-added application of lignin has drawn great scientific attention and some progress has been achieved to convert lignin into value-added products, including liquid fuels, carbon fiber and chemicals [9]. However, most of the expertise are distant from commercial practice and much more efforts are wanted in this extent. More specifically, lignin molecules have both hydrophilic functional groups and lipophilic carbon backbone, suggesting an amphipathic property to polar and nonpolar phases and great potential as a surfactant [10]. Furthermore, as a natural polymeric material providing mechanical supports in wood, lignin can be used as binder in wood composites [11].

There are various methods for lignin extraction from lignocellulosic biomass, such as soda pulping, oxidative delignification, organosolve process, acidic pretreatment, ultrasound-assisted treatment to efficiently extract lignin, nevertheless it is a major challenge to select the effective method to separate lignin and other components from OPEFB for further utilization. Many studies have shown that a high yield of lignin from the fiber can be obtained using an alkaline medium such as sodium hydroxide, ammonium hydroxide, potassium hydroxide, hydrogen peroxide [12]. Nowadays, ultrasonic-assisted extraction is gained more intension due to its effectiveness for the extraction of lignin from natural products [13]. The sonication effect of ultrasonic develops the dispersions of solvent and heat into cellular resources and therefore rises the mass transfer, resulting in requiring significant additional energy input [14]. The major effect of ultrasonic irradiation was improved through the softening effect by hydration that can make the fiber cell wall more flexible and shatter up the ultrasonic vibration which can increase the productivity to the selected amount as the effect of the breaking force by ultrasonication [12].

In the present work, lignin was extracted from OPEFB comparing two altered processes i.e alkaline and ultrasound-assisted alkaline treatment. The aim of this study is to compare the lignin properties isolated using both procedures and promising yields of lignin achieved. The alkaline treatment was carried out using 10% NaOH solution. In both processes, a similar concentration of Sulfuric acid (H_2SO_4) was used to find supernatants after both extraction processes to get lower pH through the object which increased the lignin precipitation. The isolated lignin samples were characterized by established procedures, as well as Fourier transform infrared spectroscopy (FTIR),

UV spectroscopy, and thermal stability test of the samples was studied by thermogravimetric analysis (TGA).

2. Materials and Methods

2.1. Materials

OPEFB samples were collected from the Palm Oil Mill plant (LKPP Corporation Sdn. Bhd). The chemicals used such as Sodium hydroxide (NaOH) solution, Sulfuric acid (H₂SO₄), and ethanol were purchased from Sigma Aldrich. All chemicals in the highest purity or reagent grade and were used without extra purification.

2.2. OPEFB pretreatment and lignin extraction

The OPEFB was cut into 2-3 cm lengths and was treated with hot water at 70-80°C for 2h, at the ratio of solid-to-liquid of 1:10 w/w. After that, OPEFB was cooled at 25°C and washed with normal water at the same above mentioned ratio. Finally, the sample was dried at 60°C in an oven for 16 h and stored at 4°C before the extraction procedure.

For alkali treatment, first, the OPEFB fiber was pulped by sodium hydroxide solution (7.5%, 10%, and 15% NaOH m/v) with solid to liquid ratio 1:10 (gm/ml) at 90 °C for 50, 70, 90 and 110 min, under constant stirring, then filtered to obtain the black liquor without any fibrous materials. The black liquor was acidified by sulfuric acid to pH 2, to precipitate the acidified lignin and washed by water and collected by centrifugation, and air-dried at 60°C.

For ultrasound assisted alkaline method, the fiber was treated with ultrasonic irradiation by sonication system provided with final power of 500 w and the time of sonication was 0, 10, 20, 30, and 40 min in (7.5%, 10%, and 15%) sodium hydroxide aqueous solution, the overall solid residue–liquid ratio was 1:10 (gm/ml). After the process was completed, the solid components were obtained by filtration. Then, the fiber was again washed with 0.1M NaOH to eliminate the residual lignin on the cellulose surface. The acid-insoluble lignins were formed by precipitation at pH 2 adjusted by H₂SO₄. The precipitates were centrifuged at 7000 rpm for 10 minutes. The precipitates obtained from both pretreatments known as acid insoluble lignin. Then the solid portion of lignins was washed with acidified water (pH 2) and dried at 60°C for 24 hours in a vacuum dryer.

The percentage of lignin yield extracted from both treatment were well-defined as follows [15]:

$$\% \text{ of lignin yield} = \frac{\text{mass of lignin extracted}}{\text{mass of initial biomass loading}} \times 100 \dots \dots \dots (1)$$

2.3. Characterization of lignin

The elemental analysis of C, H, N of the purified samples was performed on an Elemental Analyzer, a thermal conductivity detector was used; the oxygen content was determined by difference. The determination was performed using a calibration curve using acetanilide as standard. To evaluate the purity of the extracted lignin portions, UV spectrometer (UV 2600, Shimadzu Spectrometer) was employed at spectra of λ 200-700 nm. Fourier transform infrared spectra (FT-IR) were obtained on an FT-IR spectrometer (Perkin Elmer, Spectrum-100) with a KBr disc having 1% finely crushed lignin samples. In the frequency range of 400 cm⁻¹ to 4000 cm⁻¹, the spectra were obtained at a resolution of 5 cm⁻¹ in transmittance mode. Thermal analysis of acid-insoluble lignin preparations was performed using (Shimadzu TA-50WSI) thermal analyzer. The sample weighed between 6 and 8 mg. Both lignin samples were heated from room temperature to 700°C at a proportion of 10°C min⁻¹.

3. Result and Discussion

3.1. Yield, Purity, and Molecular Properties of extracted lignin

Figure 1 shows that the extension of treatment time tends to favor the lignin extraction yield. As expected, the lignin yield by alkali treatment for 90 min (20.1%) was much higher than 70 min (15.6%). Very low total lignin yield was obtained for the time of 50 min. However, there was no considerable yield % difference between 90 min and 110 min. This percentage was almost the same.

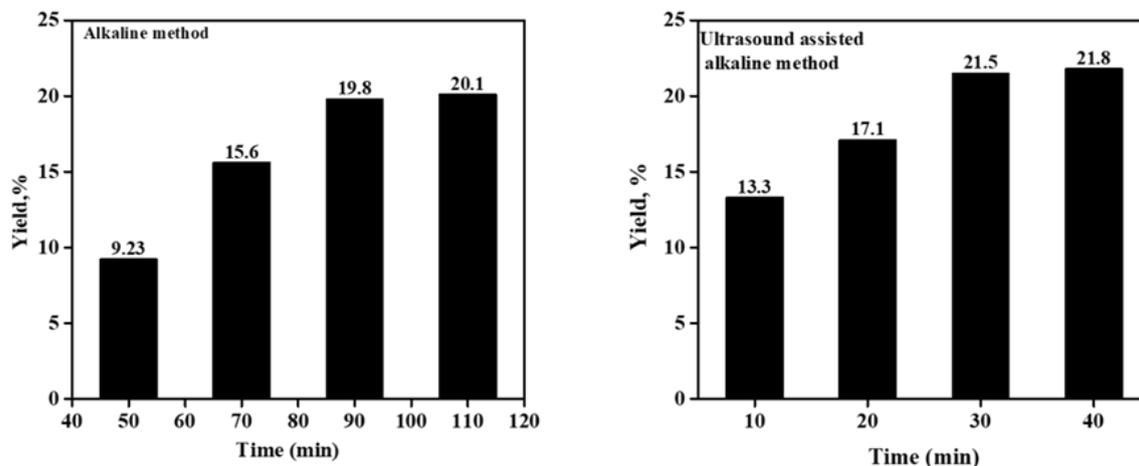


Figure 1. Effect of time on lignin extraction by alkali and ultrasound assisted alkali treatment.

Treatment of OPEFB using alkaline method with ultrasonic irradiation for 10, 20, 30, and 40 min, the total lignin obtained was 13.3%, 17.1%, 21.5%, and 21.8% respectively. Therefore, with the extension of ultrasonic time, the percentages of lignin yield were enhanced. The maximum lignin of OPEFB was released through the alkaline extraction at an ultrasonic time of 30 min. The contents of lignin from OPEFB of this study by using alkaline and ultrasound-assisted alkaline methods are 19.8% and 21.5%. RunCang Sun et al. studied the extraction of lignin fractions using ultrasound-assistance for 35 min and got 19% of total lignin [15].

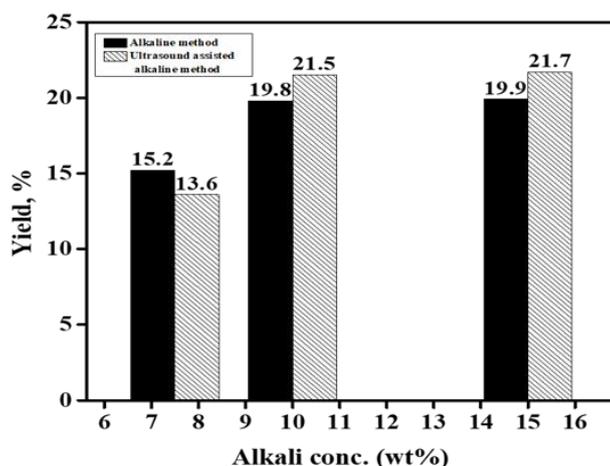


Figure 2. Effect of alkali concentration on lignin extraction by alkaline and ultrasound assisted alkaline method.

Figure 2 shows the effect of NaOH concentration used in the alkaline treatment expressed by the percentage of lignin yield. It is shown that the yield % of lignin increases gradually from 7.5% until 10% of NaOH and then until 15% of NaOH. This indicates that the amount of lignin extract from OPEFB increases gradually with increasing NaOH concentration. The extent of lignin removal with 7.5% NaOH is much lower than 10% NaOH concentration. It can be seen that when the NaOH concentration is 10% the yield lignin is 19.8% in the alkaline method. On the other hand, 21.5% lignin yield was obtained from the ultrasonic-assisted alkaline method. Thus, it can be understood that almost all of the lignin was removed in that condition.

The ultrasound-assisted alkaline extraction under the condition used has a better effect on the cleavage of the ether bonds of lignin from the cell walls of OPEFB than the alkaline treatment [16, 17]. However, it is proposing that more pure lignin fractions can be found at a moderately higher ultrasonic irradiation time such as 30 min when the polysaccharide degradation products were isolated [15, 18].

3.2. Elemental analysis

Lignin generally has a higher content of carbon and lower contents of oxygen and hydrogen. The decrease of O in lignin resulted from the elimination of cellulose and hemicellulose from biomass. Table 1 shows Carbon (C), oxygen (O), hydrogen (H), and nitrogen (N) contents of all the lignins according to CHNS analyzer. It was found that OPEFB lignin isolated by the ultrasonic-assisted extraction process provided lignin with higher carbon and lower oxygen contents (Table 1) compared to the alkali extracted lignin. Nitrogen content of lignin imitates impurity by the protein residues. Lignin from alkali extraction has high N content than ultrasound-assisted lignin. Besides, Ultrasound treatment produces a higher amount of phenolic groups in the lignin structure and therefore increases the activity of refined lignin. From elemental analysis it is understood that, ultrasound-assisted alkali lignin is more pure than alkali lignin.

Table 1: Elemental analysis of alkali and ultrasound-assisted alkali lignin

| Extraction method | Elemental amount (wt %) | | | | | Ash content (%) |
|------------------------------|-------------------------|------|------|------|-------|-----------------|
| | C | H | N | S | O | |
| Alkaline | 46.43 | 5.93 | 1.21 | 2.32 | 40.16 | 7.34 |
| Ultrasound Assisted alkaline | 51.26 | 4.29 | 0.64 | 2.07 | 39.44 | 5.82 |

3.3. Lignin ash content

In most challenging lignin applications, low ash contents are usually required. The ash content was determined gravimetrically by heating the lignin in a furnace at 800°C until constant weight. In this work, the ash content of ultrasonic-assisted extracted lignin was 5.82%, whereas, the alkali conditions produced lignin with high ash content of 7.34%. The low purity of alkali lignin is related to the high ash and carbohydrates impurities [19].

3.4. UV- Spectrometry analysis of lignin

UV spectrometry analysis has been used to semi-quantitatively define the purity of lignin. In this study, UV- vis absorption extents of the five lignin fractions were carried out with a dioxane/water mixture, which dissolved the lignin. Figure 3 illustrates the UV-vis spectra of the lignin fractions extracted by alkali and ultrasound-assisted alkali methods. The spectra of the lignin fractions are similar except for the amount of the absorption coefficient representations. The extracted acid-

insoluble lignin fractions were characterized by UV spectroscopy at λ 200-700nm which is presented in Figure 3. The absorbance λ at 280nm originated from non-conjugated phenolic hydroxyl groups in the lignin [20]. The presence of the second characteristic region of lignin absorption around λ of 300nm could be assigned to the presence of both ferulic acids and p-coumaric acids [15]. As shown in Figure 3, the highest absorption coefficient occurred in the ultrasound-assisted alkaline method, suggesting that the purest lignin preparation could be obtained when the ultrasound time was increased at 30 min. Compare to the ultrasound-assisted method alkali extracted lignin gives lower absorbance that was certainly due to co-precipitated non-lignin ingredients, for example, ash, polysaccharide degradation products, and salt.

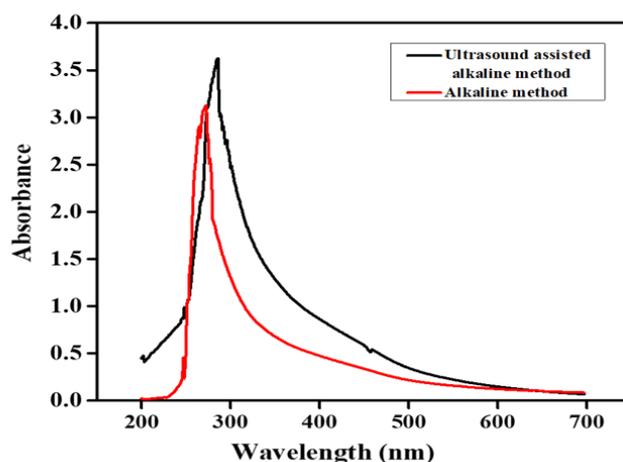


Figure 3. UV spectra of alkali lignin and ultrasound assisted alkaline extracted lignin

3.5. Spectroscopic Characterization

The spectrum clearly has shown the presence of various functional groups such as hydrogen-bonded O-H, carbonyl stretching-unconjugated ketone and carboxyl group, aromatic skeletal vibrations due to guaiacyl group, aromatic C-H in-plane deformation and aromatic C-H out of the plane, etc. [8]. The Infrared Spectrum of lignin function (Fig. 4) with and without ultrasound assistance is characterized by a broad peak at 3500 to 3000 cm^{-1} which has been given to the -OH stretching of phenolic compounds [20]. Signals with great intensity at 2926 cm^{-1} and 2924 cm^{-1} were signified C-H stretching in methylene and methyl groups, saindicating that lignin had abundant side-chain structures [21]. The absorption band at 1694 and 1670 cm^{-1} corresponds to conjugated carbonyl stretching in lignin [22]. Aromatic skeleton vibrations in lignin formations are given at 1517 and 1506 cm^{-1} . In both spectra, the bands at 1110 and 1108 cm^{-1} represent aromatic C-H plane deformation for syringyl and guaiacyl type. The relative intensity of the band at 1110 cm^{-1} , which belongs to non-etherified Ph-OH groups in lignin [23, 24]. Aromatic C-H out of bending appears at 798 and 783 cm^{-1} . An observation of the IR spectrum for acid-insoluble lignin products with and without ultrasonic-assistance (Fig. 4) shows peaks due to C-H deformation at 604-618 cm^{-1} . As we have shown in Figure 4 wherever the FTIR peak with and without ultrasonic extraction was not much different, the sonochemical vibration did not affect the structure of alkali lignin from OPEFB.

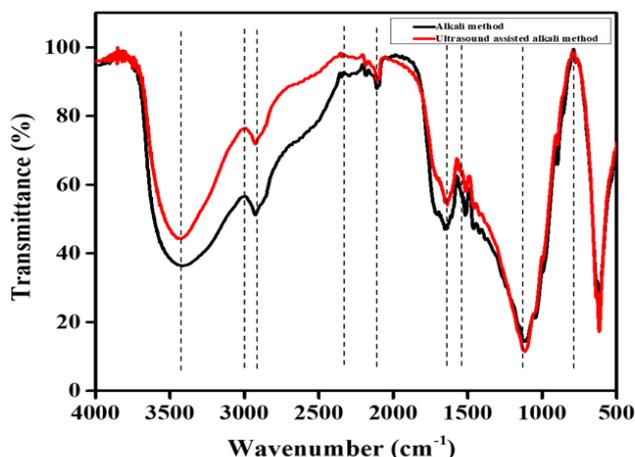


Figure 4. FT-IR spectra of alkali lignin ultrasound assisted alkali lignin.

3.6. Thermal Analysis

The thermal stability of the isolated lignin compound was studied by measuring the weight loss percentages concerning temperature using a thermogravimetric analyzer. The rate of change marks the maximum point of change in the degradation of the material [25]. The isolated lignin fraction was heated from (30–800) °C and the thermal degradation was determined by TGA. At temperature (30–80)°C, lignin degradation occurs as a result of moisture loss. This was not considered as a thermal result in the TGA discussion [26]. The thermal result of isolated lignin fraction was obtained at temperatures ranging from (166–521) °C. The maximum weight change was observed at 258 °C for alkali lignin and 303°C for ultrasound-assisted alkali lignin. Above 500°C, the degradation process is possibly related to the slow decomposition of some aromatic rings in lignin [27]. The weight loss counted at this temperature and showed that thermal stability is higher in ultrasound-assisted alkali extracted lignin than in alkaline extracted lignin.

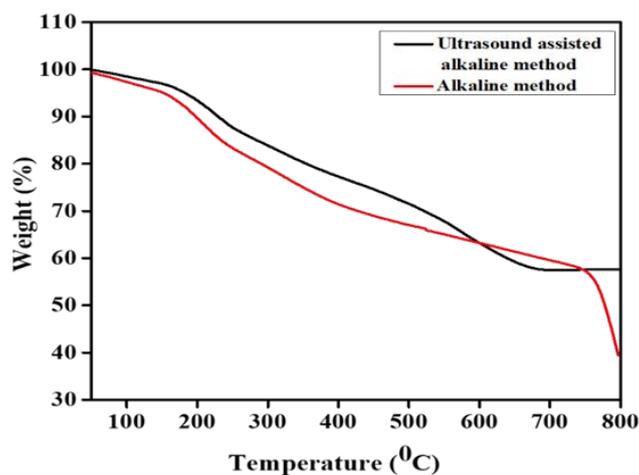


Figure 5. Thermal analysis of ultrasound assisted alkali extracted lignin for 30 min and alkali extracted lignin.

Table 2: Thermal analysis of lignins (TGA)

| Lignin | Temperature range (°C) | T _{max} (°C) | Residue (wt %) |
|-----------------------------------|------------------------|-----------------------|----------------|
| Ultrasound assisted Alkali lignin | 179-517 | 303 | 52.6 |
| Alkali lignin | 166-521 | 258 | 44.4 |

4. Conclusions

The alkaline pretreatment process requires lower pressure and temperature, but the reaction time was long compared to the ultrasound-assisted method. From the two methods, a higher yield of lignin with higher purity was obtained from the ultrasound-assisted alkaline treatment. There were no considerable differences in the structural features between the lignin fractions obtained by these two methods. The yield and purity of lignin extracted from OPEFB during ultrasound-assisted alkali treatment were higher compared to alkaline treatment. It also appeared from TGA, the thermal stability increased with increasing ultrasonic time from 10 to 30 min under certain conditions. Lignin treatment under ultrasound-assisted presented lower carbohydrates than that lignin which isolated with alkaline extraction method. Nevertheless, ultrasound alkali conditions did not cause any vital changes in lignin structure and composition. The ultrasound treatment in the lignin extraction process has also suggestively enhanced the removal of hemicelluloses to obtain high purity lignin. The total analysis indicated that it is possible to obtain high yield and pure lignin by ultrasound-assisted alkali treatment than that alkali-treated lignin. From an industrial point of view, it can be said lignin extraction by ultrasound-assisted extraction method is very significant and beneficial.

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Acknowledgement

The authors would like to thanks the Ministry of Higher Education Malaysia for providing funding for this project through TRGS grant (TRGS/1/2018/UMP/01/1/2).