Lignin Isolation from Oil Palm Empty Fruit Bunches (OPEFB) by Acidic Method

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Abstract: The isolation of lignin from oil palm empty fruit bunches (OPEFB) has been done. Firstly, OPEFB was immersed in NaOH 2% and 4%. Then, the filtrate was acidified by using H_2SO_4 5N to obtain the lignin. SEM morphology of isolated lignin showed rough surfaces. While, chemical structures of the lignin were analyzed by using FTIR and UV-visible. Aromatic ring vibrations of the phenylpropene (C9) skeleton appeared at 1600 cm^{-1}, 1515 cm^{-1}, and 1425 cm^{-1}. In addition, the presence of aromatic rings/non-conjugated phenolic groups in lignin structure was observed at 280 nm absorption.

1 INTRODUCTION

Biomass is the most abundant natural bioresources in the world. It is known as lignocellulosic material as it consists of three main molecules namely cellulose, hemicellulose, and lignin (Ma'Ruf, Pramudono and Aryanti, 2017). Oil palm empty fruit bunches (OPEFB) are composed of cellulose (41.3-46.5%), hemicellulose (25.3-32.5%), and lignin (27.6-32.5%). OPEFB have been reported for their uses in pulp production, fertilizers for oil palm plantations, carbon fibre precursors, and electrospun nanocomposite (Gea *et al.*, 2020)(Misran *et al.*, 2020). However, the isolation of lignin from OPEFB is still limited.

Lignin has several functional groups such as methoxy, carbonyl, carboxyl, and hydroxyl (phenolic and alcoholic components). The chemical structure of lignin is presented in Figure 1. The phenolic compounds in lignin can potentially be used as macromolecular in toughening agents for epoxy resin, surfactant, and carbon fibre precursors. Lignin-based carbon fibre is known to have tensile strength and modulus of 0.51 GPa and 28.6 GPa respectively (Baker, Gallego and Baker, 2011).

There are several methods to isolate lignin from biomass, such as using acid and alkali solvent, ionic solution and organic solvent, and alkaline hydrogen

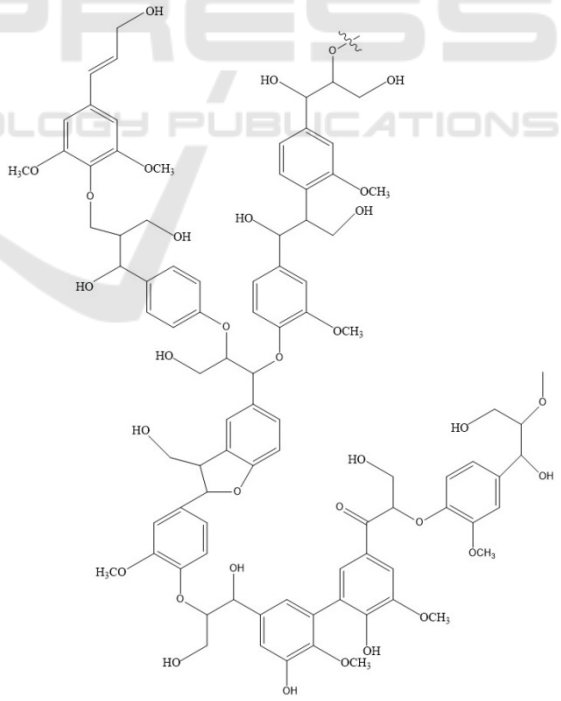

Figure 1. The potential chemical structure of lignin (Gregory, 2007)

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peroxide (H_2O_2) (Ma'Ruf, Pramudono and Aryanti, 2017). The isolation of lignin using acid solvent method has some advantages, such as low-cost, efficient production, and low temperature requirement.

In this study, we extracted lignin from OPEFB using H_2SO_4 at room temperature. The isolated lignin was characterized for its morphology and chemical structures.

2 EXPERIMENTAL

2.1 Materials

Oil palm empty fruit bunches (OPEFB) were obtained from PTPN IV Adolina, North Sumatera. $NaOH_{(s)}$ and $H_2SO_{4(l)}$ 97% were purchased from Merck, Germany.

2.2 Isolation and Purification of Lignin

The fibres of OPEFB were immersed in different concentration of NaOH, such as 2% and 4%. The immersion process was done at room temperature for 24 h. After that, OPEFB were filtered and acidified by using 5 N H_2SO_4 to reach pH 2. The acidified solution was washed with distilled water and filtered. The solid parts were collected and centrifuged at 8000 rpm for 5 min. Finally, the isolated lignin was dried in a vacuum oven at 50 °C for 4 h. The products were coded as lignin2% and lignin4%.

2.3 Characterization

2.3.1 FTIR

The functional groups in isolated lignin were investigated by using FTIR Spectrometer (FTIR, Nicolet 380, Thermo Scientific, Boston, MA, USA). The sample was analyzed in a disk form with 100:1 KBr to sample ratio. FTIR instrument was operated in transmission mode with 400–4000 cm-1 wavelengths, 4 cm-1 resolution, and 100 scans.

2.3.2 UV-Visible

The aromatic rings in lignin were investigated by using ultraviolet/visible spectrophotometer (UV 1800 series, Shimadzu Scientific Instrument, Kyoto, Japan). The instrument was operated with absorbance between 250 and 400 nm wavelengths.

2.3.3 Scanning Electron Microscopy

Sample morphology was analyzed by using Scanning Electron Microscopy (SEM, Hitachi TM3030, JEOL, Ltd., Tokyo, Japan) operating at 20 kV. The sample was first coated with a thin layer of gold before analysis to reduce charges.

3 RESULTS AND DISCUSSION

3.1 FTIR Analysis

The functional groups present in the isolated lignin were analyzed by using FTIR spectral analysis. FTIR spectra for the polymers are presented in Figure 2. Aromatic ring vibrations of the phenylpropene (C9) skeleton could be seen to have appeared at 1600 cm^{-1}, 1515 cm^{-1}, and 1425 cm^{-1}. The absorption between 3600 and 300 cm^{-1} was attributed to hydroxyl groups in the aromatic and aliphatic structures. In addition, C-H stretching in methyl and methylene groups, as well as C-H stretching in aromatic methoxy group were observed at 2938 cm^{-1} and 2885 cm^{-1} respectively (Chen *et al.*, 2016; Abdelaziz and Hulteberg, 2017).

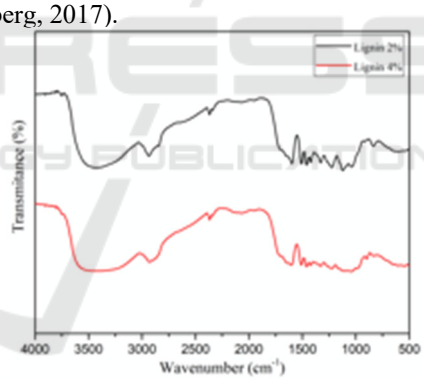

Figure 2 FTIR spectra of isolated lignin immersed in NaOH 2% and NaOH 4%.

3.2 UV-visible Analysis

UV-visible analysis (Figure 3) was used to investigate the presence of aromatic rings/non-conjugated phenolic groups in the structures of lignin. From Figure 3, there was an absorption at approximately 280 nm, which indicated aromatic rings in lignin (Gea *et al.*, 2020).

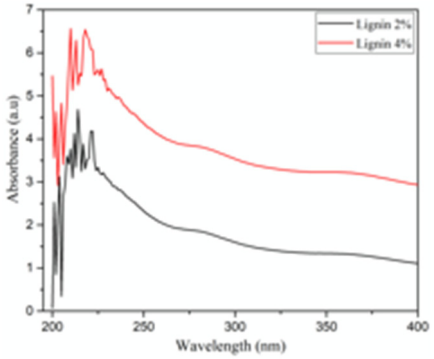

Figure 3. UV-visible of lignin immersed in NaOH 2% and NaOH 4%.

3.3 Scanning Slectron Microscopy Morphology

Surface morphology of isolated lignin is shown in Figure 4. Before analysis, isolated lignin was dried at 80 ºC for 5 h in a vacuum oven to remove moisture and water content. Then, the sample was coated with a thin gold to reduce charging during analysis. As seen in Figure 4, the morphology of isolated lignin was rough and flaky. This finding result was different by a previous study that reported lignin with smooth and uniform morphology in powder form (van de Pas *et al.*, 2011). These different results could be caused by different instrument and isolation procedure used. Furthermore, NaOH 4% produced a smoother surface morphology than NaOH 2%.

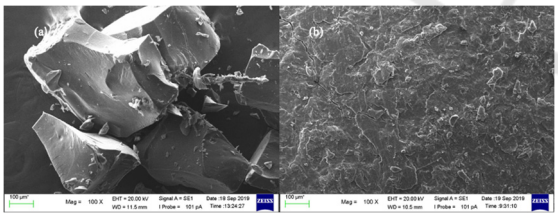

Figure 4. Surface morphology of isolated lignin immersed in (a) NaOH 2% and (b) NaOH 4%, with magnification of 100x

4 CONCLUSIONS

The isolation of lignin from OPEFB was done by using acidic solvent (H_2SO_4). OPEFB were immersed in NaOH 2% and 4% before lignin extraction. FTIR and UV-visible analysis showed that lignin had aromatic structures. Meanwhile, SEM analysis confirmed that lignin had rough surface morphology.

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