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Characteristics of Purified Lignin Obtained from *Metroxylon Sagu* Palm Fronds, Oil Palm Fronds, *Cratoxylum arborescens*, and *Eucalyptus pellita* via Soda-Methylanthraquinone Pulping

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ABSTRACT. Lignin is a valuable material that can be obtained from the black liquor generated during dissolving pulp production from lignocellulosic materials. Lignin can be used to produce biopolymers, fuels, value-added chemicals, and other functional materials. This study investigates the isolation and characterization of lignin from four lignocellulosic biomass sources i.e. *Metroxylon sagu* palm fronds (SPF), oil palm fronds (OPF), *Cratoxylum arborescens*, and *Eucalyptus pellita* using environmentally friendly soda–methylanthraquinone (soda–MAQ, 160 ºC for 1.5 h) pulping followed by acid precipitation with hydrochloric acid (pH 2.0-2.5). The lignin yield varied among the materials (68.3-88.8%), with *E. pellita* showing the highest recovery. SEM analysis revealed distinct differences in lignin morphology, where *E. pellita* lignin exhibited a more condensed and homogeneous structure compared to the more fragmented forms from non-wood biomass. FT-IR spectra confirmed the presence of guaiacyl, syringyl, and hydroxyl phenol units in all samples. EDX analysis supported the natural lignin composition, especially in *E. pellita*, OPF, and *C. arborescens*. Among the four sources, lignin from *E. pellita* demonstrated the greatest potential for value-added applications, followed by *C. arborescens* and OPF. These findings provide valuable insight into lignin recovery from diverse biomass and its suitability for further bioproduct development.

Keywords: Biomass, characterization, dissolving pulp, lignin, soda-methylanthraquinone.

INTRODUCTION

Lignin precipitation has become a topic of discussion by many researchers worldwide especially due to the rapid growth of the pulp and paper industry and the conversion of paper-grade pulp to dissolving pulp. Lignin is dissolved in the black liquid known as black liquor which is produced during pulping of biomass raw materials. Black liquor consists of about 10-50% heavy lignin (Ksibi, 2003), 65-85% solids (Ramesh et al., 2013), and is composed mainly of water, organic and inorganic substances (Kaabi et al., 2018; Marklund et al., 2007). Recovery of lignin from black liquor (LignoBoost) in pulp mills for its subsequent use as fuels or in the production of specialty chemicals e.g. composites, adhesives, and barriers, is thought to increase pulp mill capacity and creation of a completely fossil-fuel-mill concept (Vakkilainen & Välimäki, 2009). According to Sinhal and Kamboj, 40% cost of the new pulp and paper industry is mainly for installing and running of recovery furnaces for burning organic chemicals for steam generation (Sinha & Kamboj, 2019). Appropriate chemical processing and screening procedures are needed to isolate lignin from the black liquor (Fox, 2006). The structure and physicochemical properties of the obtained lignin are affected by various factors i.e. isolation methods, isolate types, temperatures, and raw material sources (Bykov, 2008; Ibrahim et al., 2004; Minu et al., 2012; Sahoo et al., 2011; Tejado et al., 2007; Toledano et al., 2010a, 2010b). Chemicals such as alkali and acid (Minu et al., 2012; Tejado et al., 2007), physical (Toledano et al., 2010a, 2010b), biological, and combined (Mosier et al., 2005) methods have been listed for lignin isolation. Acidic solutions such as hydrochloric acid (HCl) and sulfuric acid (H_2SO_4) have been commonly used for precipitation since lignin is insoluble in acid solutions (Priyanto et al., 2019).

High-purity cellulose (often referred to as dissolving pulp) can be produced in three stages i.e. prehydrolysis, cooking, and bleaching. During prehydrolysis, most of the hemicellulose is removed (Evelyn et al., 2022), whereas the main portion of lignin is desired to be removed during the cooking stage followed by their final removal through the bleaching stage. Recovery of lignin from the soda cooking black liquor is considered simpler than from the kraft-cooking black liquor (Utami et al., 2023). The purpose of this study is to compare the yield, functional groups, chemical properties, and morphology of lignin obtained from pulping of different raw materials i.e. *Metroxylon sagu* palm fronds (SPF), oil palm fronds (OPF), geronggang (*Cratoxylum arborescens*), and *Eucalyptus pellita*.

EXPERIMENTAL SECTION

Materials and Equipment

Muntai village in the Bantan district on the Indonesian island of Bengkalis is the location for sago palm fronds (SPF) collection, whereas a 5.5-year-old geronggang (*Cratoxylum arborescens*) wood was obtained from BP2TSTH (Development Institute of Forest Plant Fiber Technology), Kuok, Kampar Regency, Indonesia. Oil palm fronds (OPF) were collected from an oil palm industry in Sumatera, Indonesia. Lignin from *E. pellita* was obtained from a past study (Utami et al., 2023), and used to compare isolated lignins from the three wood materials. Chips preparation for SPF, OPF, and *C. arborescens* was performed according to previous studies by Evelyn et al. (2022, 2023) and Chairul et al. (2023). MAQ (Alfa Aesar, India) as the pulping catalyst was provided by the University of Tsukuba, Japan. SEM and FT-IR analyses were performed to characterize the precipitated lignin obtained from the extraction process.

Prehydrolysis and Cooking Conditions of Wood Materials

Three biomass (SPF, OPF, and *C. arborescens*) were prehydrolyzed and soda-cooked in a 300 mL stainless-steel reactor, as described in Evelyn et al. (2022). Prehydrolysis and cooking conditions were 150 °C for 3 h and 160 °C for 1.5 h (at active alkali or AA of 21-25%) with a liquor-to-solid ratio of 7 L/kg. Distilled water was used as the liquor in the prehydrolysis. During the soda cooking of pulp (without any washing), 0.03% of MAQ dosage based on the wood weight was added, and the resulting black liquor was used to produce lignin. With respect to *E. pellita*, the conditions were described previously (Utami et al., 2023).

Lignin Precipitation Protocol from Cooking Black Liquor

In the first experiments, 3 M sulfuric and hydrochloric acid (H_2SO_4 and HCl) were compared as lignin precipitation agents of SPF via soda-MAQ cooking at AA 21-25%. Then, black liquors obtained from pulp cooking of all biomass using 23% active alkali were used in all subsequent lignin precipitation and purification experiments, employing HCl instead of H_2SO_4. Hydrochloric acid requires a lower dosage and produces cleaner lignin precipitates with minimal ionic interference (Duret et al., 2025). Protocol for the lignin precipitation from the black liquor of prehydrolysis soda pulping was performed according to the method described previously and modified by Utami et al. (2023). Briefly, 20 g of black liquor was mixed with the acid to obtain a suspension at pH 9.0, followed by heating at 65 ºC for 30 s. Then, two-stage centrifugations (at 1500g for 30 min) were carried out to produce supernatants which labelled as S-1 and S-2 for each stage, which were further mixed (labelled as S-3). The pH in S3 was adjusted to 2.0-2.5 with 3 M HCl, heated to 65 ºC-30 s, followed by separation using vacuum filtration and drying at room temperature (P1=precipitate from S-3). P2 represents precipitates obtained after the second step of centrifugation and drying (at room temperature). The weights of P1 and P2 were calculated as yields based on the wood lignin weight in the black liquor. **Figure 1** shows the steps of precipitation and purification of lignin utilizing hydrochloric acid.

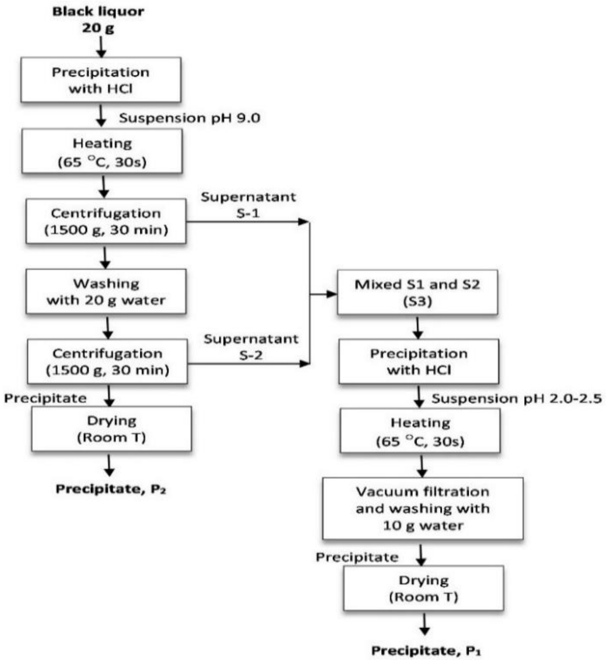


Figure 1. The steps of precipitation and purification of lignin from the soda-MAQ cooking black liquor

Characterization of Precipitated Lignin

Samples of extracted lignins were sent out to an accredited laboratory to carry out the characterization of lignins using Fourier Transform Infra-Red Spectroscopy (FT-IR, Shimadzu IRPrestige-21) and Scanning Electron Microscopy equipped with Energy-Dispersive X-ray (SEM-EDX, Hitachi Flexsem 1000). The absorption spectra of FT-IR are in the 4650 to 450 cm^{-1} regions. Scanning Electron Microscopy (SEM) was used to observe the surface morphology, while Fourier Transform Infrared Spectroscopy (FT-IR) was employed to identify the functional groups of the precipitated lignin.

RESULTS AND DISCUSSION

Effect of Acid Types on the Lignin Precipitation

Some of the main factors affecting the separation of lignin from black liquor are pH, acid type, temperature, and the concentrations and types of metal ions (Hubbe et al., 2019). Weak and strong acids have been attempted for lignin isolation, with effectiveness (including side effects) and cost as the key considerations. Strong acids mainly H_2SO_4 can lower the pH low enough to protonate the phenolic as well as carboxylic acid functional groups that bind to lignin (Hubbe et al., 2019). In this section, black liquors of SPF pulping at 21-23% AA were used to compare the lignin purified using H_2SO_4 and HCl (**Table 1**). As expected, based on the wood lignin weight, the yield of precipitated lignin was higher using H2SO4 (84.1-90.8%) than HCl (68.3-76.5%), and the highest yields were observed at the highest active alkali used. Similar results i.e. higher yield with H_2SO_4 than HCl (11.96-12.95 vs. 10.49-11.87 g/L) were also observed by other investigators with wheat straw (Domínguez-Robles et al., 2016). Ibrahim et al. (2004) reported that the precipitated lignin obtained from the black liquor of oil empty fruit bunch was affected by the acid type applied i.e. 1.45-1.51 g/100mL for phosphoric acid vs. 1.42-1.48 and 1.35-1.37 g/100mL for H_2SO_4 and HCl, respectively. According to previous studies, the use of H_2SO_4 resulted in a higher yield of precipitated lignin compared to HCl due to its stronger acidifying capacity as a diprotic acid, which lowers the pH more effectively and reduces lignin solubility (Weerasai et al., 2024; Zhu & Theliander, 2015). Additionally, the presence of sulfate ions enhances lignin aggregation and precipitation efficiency. Lignin yields between 85.6 and 88.8% were obtained from hardwood's black liquor (*Acacia crassicarpa* and *E. pellita*) using similar cooking and lignin precipitation methods Utami et al., (2023).

Effect of Wood Species on the Yield of Precipitated Lignin

Precipitated lignin obtained from the soda-MAQ cooking black liquor and different raw materials i.e. SPF, OPF, and *C. arborescens* were characterized for yields (and compared to *E. pellita*), as shown in **Figure 2**. *C. arborescens*, and *E. pellita* wood chips have the highest lignin recovery. Therefore, it can be assumed that these two hardwood materials are good candidates for lignin precipitation due to the higher content of lignin in wood materials since during the pulping process, more lignin is available to dissolve into the black liquor (Jardim et al., 2022).

Table 1. The precipitated lignins obtained from SPF using acids

Active alkali in soda- MAQ cooking (%)	Lignin yield (%)	
	Sulfuric acid	Hydrochloric acid
21	84.1	68.7
23	85.8	71.3
25	90.8	76.5

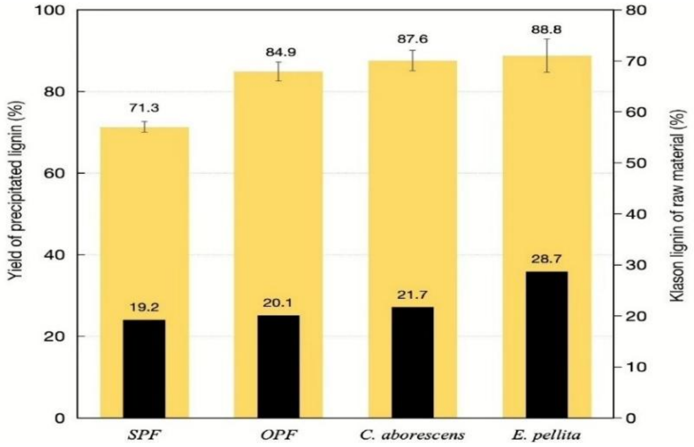


Figure 2. The yields of HCl precipitated lignin obtained from soda-MAQ cooking (AA=23%) black liquor with different raw materials (SPF, OPF, *C. arborescens*, and *E. pellita*). Yield data for *E. pellita* was taken from Utami et al. (2023) at AA=17%.

This fact is supported by 28.7% of klason lignin available in *E. pellita* compared to ≤21.7% in *C. arborescens*, OPF, and SPF. As suggested in a past study, lignin recovery in hardwood is also affected by high syringyl–guaicyl or S/G ratio of the native lignin (Jardim et al., 2022). The reported S/G ratio of *E. pellita* clones ranges from 2.6 to 3.6 (Diniz et al., 2019). However, no data on the S/G ratio of *C. arborescens* has been found in the literature, and it is likely to be lower than that of *E. pellita*. In the case of non-wood materials, additional factors such as inorganic content including extractives and ash can significantly influence the yield of lignin recovery (Do et al., 2020; Risanto et al., 2014). Despite its relatively high ash content (14.6%) compared to SPF (6.3%), OPF have shown promising lignin recovery yields of up to ~85%. For all materials tested, the lignin purity (measured as Klason lignin) obtained through the soda- MAQ pulping process ranged between 60% and 64.5%.

Lignin Characterization by SEM-EDX

The morphology and size of hydrochloric acid's precipitated lignins observed by SEM at similar magnification (2×) are shown in **Figure 3**. As mentioned earlier, hydrochloric acid was used in all subsequent lignin precipitation experiments due to its several advantages, including reduced sulfur content, which is more favorable for the environment. Despite its smoother surfaces, non-wood lignins (SPF and OPF) exhibited irregular shapes and uneven size distribution from 1 to 100 µm compared to hardwood lignins (*C. arborescens* and *E. pellita*). Bigger sizes of OPF lignin than SPF lignin were observed. Lignin obtained from *C. arborescens* showed a less compact block structure than OPF lignin at the same magnification. Lignin that was precipitated from *E. pellita* showed the most different structure than the others, showing small and round shapes particles with two major different sizes. Nonetheless, the SEM observations for OPF is similar to the one reported by other authors (also precipitated using HCl) (Hidayati et al., 2020), whereas *E. pellita* lignin resembles the same morphology to lignin reported in a past study with *Eucalyptus grandis*(Intapun et al., 2021).

Figure 4 shows the morphology and size of the precipitated lignins under SEM at high magnifications to provide a better understanding of the fractionated lignins. Lignin from OPF (non-wood) and *C. arborescens* (hardwood) exhibited the same size and smooth morphology by SEM (250×magniffication, **Figures 4b and 4c**). Although SPF (at 200 × magnification, **Figure 4a**) also seemed to have a similar structure to OPF and *C. arborescens*, it shows smaller sizes suggesting a more disturbed structure. SEM observation of *E. pellita* confirms the distinct morphology of precipitated lignin compared to other similar hardwoods, showing a more condensed and homogeneous structure, showing a more more condensed and homogeneous structure of this lignin (Intapun et al., 202; Suota et al., 2021; Zhang et al., 2022). EDX determination was further carried out to find the main composition of precipitated lignin from the four materials (SPF, OPF, *C. arborescens*, and *E. pellita*) and results are shown in **Figure 5**. In exception to SPF lignin, the chemical composition of lignin mainly includes carbon (C) and oxygen (O), corresponding to the natural structure of lignin with a high weight ratio of carbon and oxygen are 44.2-45.2% and 37.9-39.6% by weight, respectively. The C and O content of OPF in this study is higher than the composition reported in another study with OPF and formacell process (23.3% for both C and O elements) (Hidayati et al., 2020). Sodium and chloride were the dominant inorganic elements observed in all lignins. To the author's knowledge, none has been reported with SPF and *C. arborescens.*

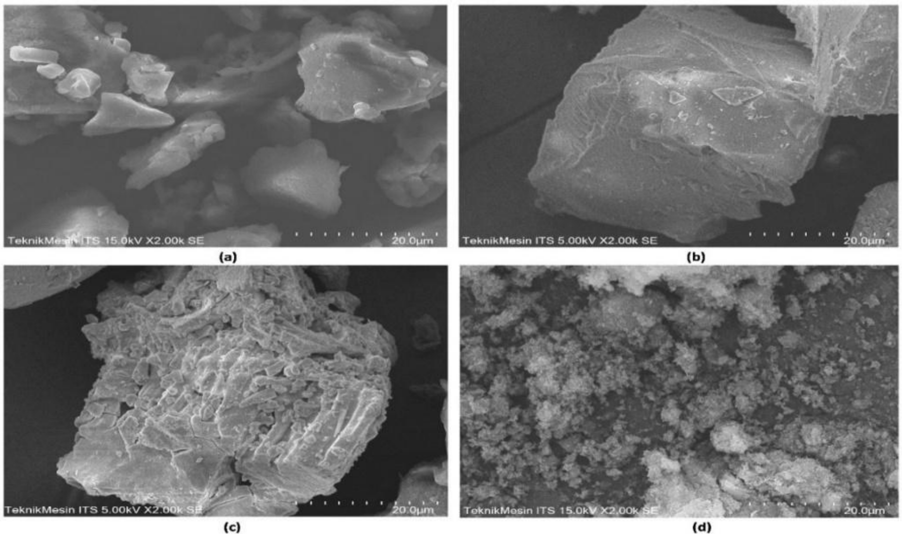


Figure 3. The SEM images of precipitated lignin (2 × magnification): SPF (**a**), OPF (**b**), *C. arborescens* (**c**), and *E. pellita* (**d**). *E. pellita* lignin was supplied by Utami et al. (2023).

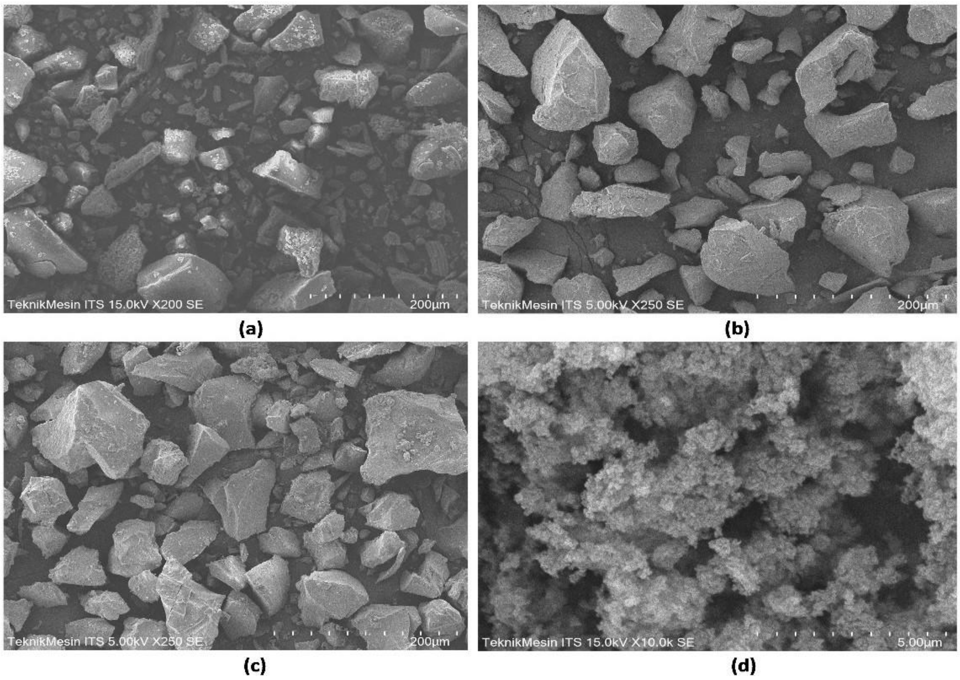
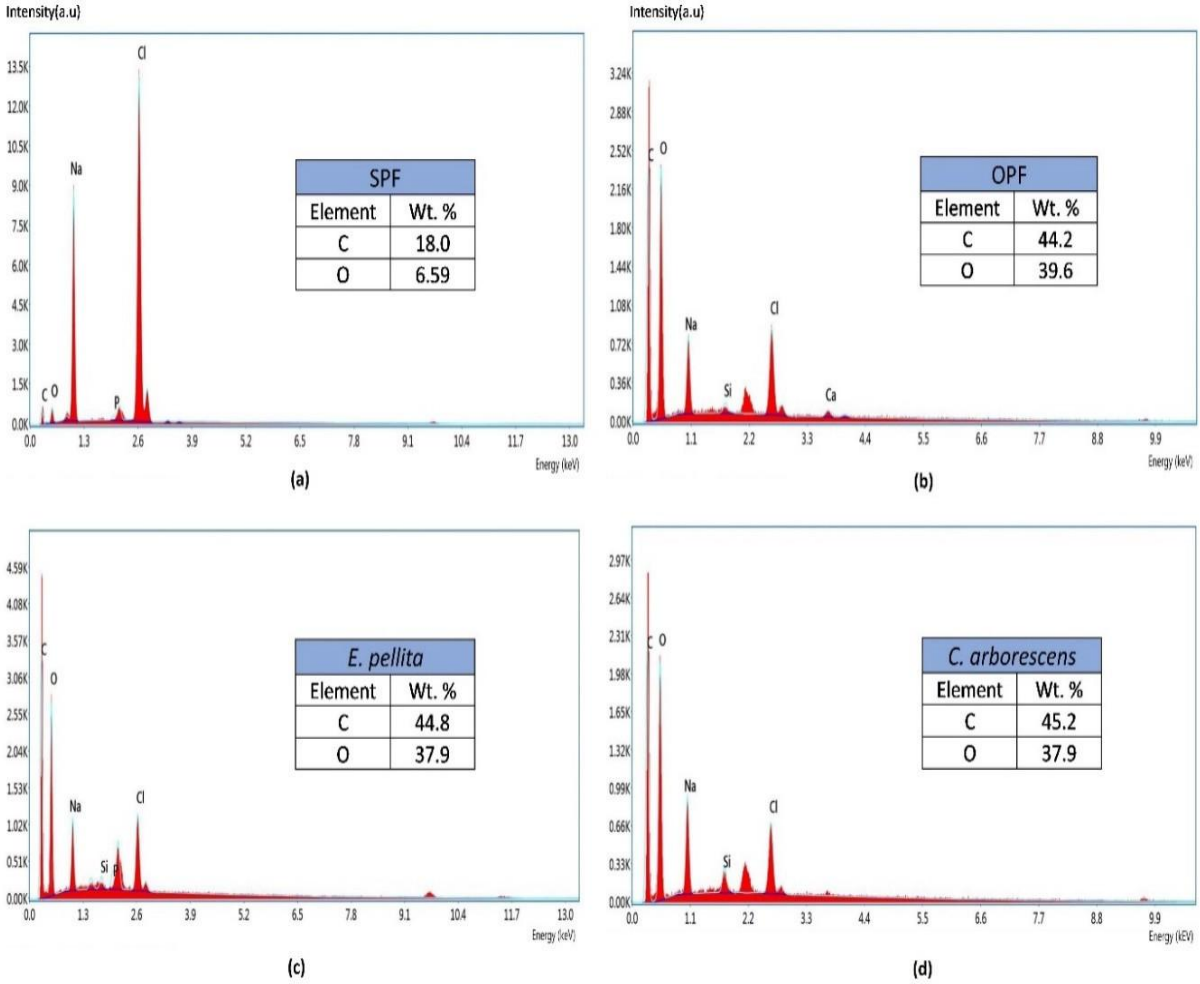


Figure 4. The SEM images of precipitated lignin (different magnification): SPF-200× (**a**), OPF-250× (**b**), *C. arborescens*-250× (**c**), and *E. pellita*-10× (**d**). *E. pellita* lignin was supplied by Utami et al. (2023).



SPF	
Element	Wt. %
C	18.0
O	6.59

OPF	
Element	Wt. %
C	44.2
O	39.6

E. pellita	
Element	Wt. %
C	44.8
O	37.9

C. arborescens	
Element	Wt. %
C	45.2
O	37.9

Figure 5. The EDX spectra of SPF (**a**), OPF (**b**), *E. pellita* (**c**), and *C. arborescens* (**d**) lignins precipitated using hydrochloric acid.

Lignin characterization by FTIR

FTIR spectra of isolated lignins from soda-MAQ cooking displayed nearly identical patterns based on normalization of the FTIR data, as shown in **Figure 6** and **Table 2**. The functional groups of these lignin samples correspond to the assignment FTIR spectrum of lignin reported previously by several authors (Bhat et al., 2009; Domínguez-Robles et al., 2016; Hidayati et al., 2020; Ibrahim et al., 2004; Inkrod et al., 2018; Kumar et al., 2016; Sameni et al., 2016; Shi et al., 2019). All lignin from different wood species presented the vibration of O—H stretching at 3340.71–3463.81 cm^{-1}, and C—H stretching in —CH_2— and —CH_3 groups at 2917.46–2943.37 and 2841.15–2843.20 cm^{-1}, respectively, as well as the C=O group stretching of carbonyl group that showed peaks at 1693.10–1712.79 cm^{-1} (Inkrod et al., 2018; Sameni et al., 2016). All of the following FT-IR spectra can also be observed in all lignin samples: aromatic skeletal vibrations (S>G) (1597.13–1606.70 cm^{-1}), aromatic skeletal vibrations in G>S (1507.43–1518.04 cm^{-1}), C-H deformations in —CH_2— and —CH_3 group (1453.43–1459.61 cm^{-1}), aromatic skeletal vibrations (1417.74–1426.42 cm^{-1}), S ring breathing (1321.30–1327.08 cm^{-1}), and C—C and C—O stretch, G condensed > G (1216.17–1217.14 cm^{-1}), aromatic C—H in-plane deformation (S) (1114.86–1117.80 cm^{-1}), aromatic C—H in-plane deformation (G>S) (1031.96–1039.63 cm^{-1}), —CH=CH— out-of-plane deformation (trans) (912.33-962.52 cm^{-1}), and aromatic C—H out-of-plane deformation (S + H) (823.64–829.39 cm^{-1}) (Domínguez-Robles et al., 2016; Hidayati et al., 2020; Ibrahim et al., 2004; Kumar et al., 2016; Sameni et al., 2016; Shi et al., 2019).

FT-IR strong absorption bands at around ~1600, ~1329, and 1119 cm^{-1} represent syringyl units (Inkrod et al., 2018; Kumar et al., 2016;). The lignin extracted from SPF showed no peak of C-O stretch in ester groups with HGS and weak intensity of aromatic C-H out-of-plane deformation (S + H) approximately at around 830 cm^{-1}, as opposed to the other three wood materials with strong intensities. These bands suggested that the lignin from OPF, *C. arborescens*, and *E. pellita* contained both of guaiacyl and syringyl units, while SPF consisted mainly of guaiacyl group (Sameni et al., 2016). Peaks of aromatic ring vibrations at ~1600, ~1500, and ~1420 cm^{-1} indicate occured delignification in wood materials (Inkrod et al., 2018). The weak intensity at ~1360 seemed to indicate that the process insignificantly cleaved the linkage between α-O-4′ and β-O-4′ in the isolated lignin macromolecules (Shi et al., 2019). OPF and *C. aborescens* wood showed peaks at 724.30–733.95 cm^{-1}, which has been observed in other study with palm fiber/waste (743-773 cm^{-1}) (Bhat et al., 2009; Inkrod et al., 2018). To conclude, these assignments confirm the typical lignin structure in all samples and support the qualitative presence of syringyl, guaiacyl, and phenolic hydroxyl groups across the lignins studied.

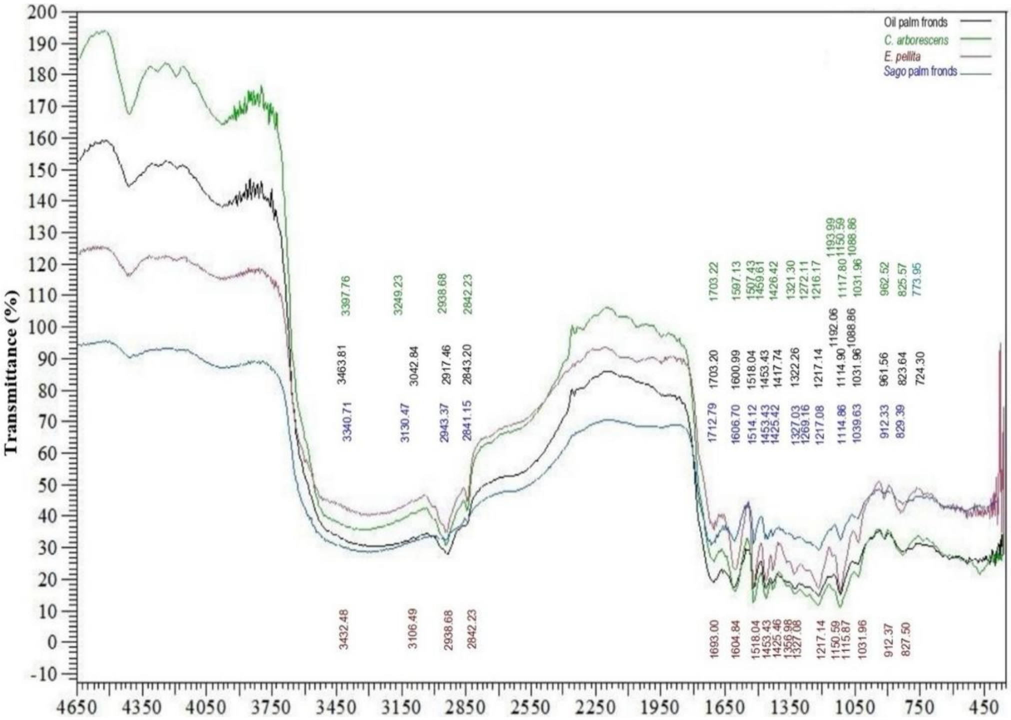


Figure 6. FT-IR spectra (wavenumber of 4650 to 450 cm^{-1}) of SPF, OPF, *C. arborescens*, and *E. pellita* lignin precipitated using hydrochloric acid. *E. pellita* lignin was supplied by Utami et al. (2023) from soda-MAQ cooking at AA=17%.

Table 2. FT-IR characteristic bands assignments of four materials (SPF, OPF, *C. arborescens*, *E. pellita*)*

Assignment	SPF	OPF	*C. arborescens*	*E. pellita*
	Wave number (cm-1)			
O-H stretching	3340.71	3463.81	3397.76	3432.48
C-H stretching in -CH_2- and -CH_3	2943.37, 2841.15	2917.46, 2843.20	2938.68, 2842.23	2938.68, 2842.23
C=O stretching, unconjugated	1712.79	1703.20	1703.22	1693.10
Aromatic skeletal vibrations (S > G)	*1606.70*	*1600.99*	*1597.13*	*1604.84*
Aromatic skeletal vibrations (G > S)	*1514.12*	*1518.04*	*1507.43*	*1518.04*
C-H deformations in -CH_2- and -CH_3	1453.43	1453.43	*1459.61*	*1453.43*
Aromatic skeletal vibrations	1425.42	1417.74	*1426.42*	*1425.46*
Aliphatic C-H stretch in CH_3, not in OCH_3	-	-	-	1356.98
S ring breathing	1327.03	*1322.26*	*1321.30*	*1327.08*
G ring breathing	1269.16	-	1272.11	-
C-C and C-O stretch, G condensed > G	*1217.08*	*1217.14*	*1216.17*	*1217.14*
C-O stretch in ester groups (HGS)	-	1192.06	1193.99	1150.59
Aromatic C-H in plane deformation (S)	*1114.86*	*1114.90*	*1117.80*	*1115.87*
Aromatic C-H in plane deformation (G > S)	*1039.63*	*1031.96*	*1031.96*	*1031.96*
-CH=CH- out-of-plane deformation (trans)	912.33	*961.56*	*962.52*	*912.37*
Aromatic C-H out-of-plane deformation (S + H)	829.39	*823.64*	*825.57*	*827.50*
C-H bond in the aromatic ring from G	-	*724.30*	*733.95*	-

*Band interpretation was based on several previous studies (Bhat et al., 2009; Domínguez-Robles et al., 2016; Hidayati et al., 2020; Ibrahim et al., 204; Inkron et al., 2018; Kumar et al., 2016; Sameni et al., 2016; Shi et al., 2019). *E. pellita* lignin was obtained from a previous study (Utami et al., 2023). G guaiacyl unit, S syringyl unit, H hydroxyl phenol unit; Italics show peaks' strong intensities.

CONCLUSIONS

This study demonstrated the isolation of lignin from black liquor obtained via environmentally friendly soda–methylanthraquinone (soda–MAQ) pulping of various lignocellulosic materials, including sago palm fronds (SPF), oil palm fronds (OPF), *Cratoxylum arborescens*, and *Eucalyptus pellita*, using acid precipitation. While lignin purity remained relatively consistent, the efficiency of lignin isolation was likely influenced by the inherent lignin content of each biomass type. SEM analysis revealed noticeable differences in lignin particle size and morphology, particularly between SPF and *E. pellita*. The chemical compositions of lignin from OPF, *C. arborescens*, and *E. pellita*, as determined by EDX, reflected characteristics of natural lignin. FT-IR spectra of all four materials confirmed the presence of guaiacyl, syringyl, and phenolic hydroxyl units.

Among the four biomass sources, *E. pellita* lignin showed the highest potential for application due to its superior recovery, homogeneous morphology, and complete functional group profile. This was followed by *C. arborescens*, which also exhibited good yield and structural integrity. OPF showed moderate potential, while SPF lignin appeared least favorable due to lower recovery and more fragmented morphology. These findings contribute valuable insights into lignin recovery and support its potential valorization into high-value bioproducts.

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