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Cellulose and lignin purified from *Metroxylon sagu* palm fronds by a new technology with 2-methylantraquinone cooking and peroxymonosulfuric acid bleaching

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Abstract

The demand for high-purity cellulose, optimization of wood utilization, and environmentally friendly processes has increased in dissolving pulp (DP) production. Sago palm fronds (SPF), an abundant agricultural waste in Indonesia, hold great potential as a raw material for cellulose, hemicellulose, and lignin production. This study aimed to explore the production of from SPF by employing a combination of prehydrolysis, soda cooking with 2-methylantraquinone (MAQ) as a green additive (PHS-MAQ), and totally chlorine-free (TCF) bleaching with peroxymonosulfuric acid (P_{sa}). Furthermore, lignin was recovered from the black liquor of PHS-MAQ. The results showed that prehydrolysis at 150 °C for 3 h, followed by soda-MAQ cooking at 160 °C for 1.5 h using 0.03% of MAQ, 23% active alkali (AA), and a five-stage bleaching with oxygen (O), P_{sar} , alkaline extraction with hydrogen peroxide (E_p , P_{sar} , and E_p) successfully produced high-purity cellulose as DP, with properties of 94.3% α -cellulose content, 89.9% ISO brightness (SNI ISO 2470–1:2016), 9.1 cP viscosity, and 0.13% ash content. Moreover, the soda-MAQ cooking method exhibited superior delignification compared to prehydrolysis kraft (PHK) and prehydrolysis soda (PHS) processes in a range of kappa numbers of 9.4–22.6 at 17–25% AA. The inclusion of MAQ increased pulp yields by 4.6–4.9% and decreased kappa number by 1.6–3.1 compared to the PHS without additives at similar AA. Lignin was separated from the PHS-MAQ, with yields of 69–77%. This work demonstrated the suitability of SPF processed by PHS-MAQ cooking and P_{sa} bleaching for the preparation of viscose rayon and cellulose derivatives. The lignin recovery could be an attractive biorefinery process in modern pulp mills.

Keywords Sago palm fronds, Cellulose, Lignin, Prehydrolysis soda cooking, 2-Methylantraquinone, Pulp

Introduction

Dissolving pulp (DP), also referred to as dissolving cellulose, is an essential raw material comprising high-purity cellulose and unique properties which has recently attracted significant attention due to its high demand and diverse applications. The global production of DP increased at an average annual rate of 3.9% between 2007 and 2018, with China as the leading country followed by India, Indonesia, Germany and United States [1]. Supply issues with cotton limit its use as the raw material in DP production. The predominant application of wood in DP

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production poses numerous challenges including forest resources depletion, global warming, and rising prices of virgin wood fibers. These backgrounds emphasize the need to explore alternative non-wood-based raw materials to optimize wood usage, develop sustainable alternatives, and enhance industrial competitiveness.

Sago palm (*Metroxylon sagu*), a promising tree crop known for being a valuable starch source, is extensively grown in Southeast Asia and Pacific Island countries [2, 3] (Fig. 1). Approximately 85% of its global cultivation is found in Indonesia, in which 95% of sago region is located in Papua and West Papua [4]. Despite its benefits in food and several other industries, however, significant amounts of waste, i.e., sago palm fronds (SPF) are generated during the harvesting and starch extraction processes leading to environmental issues [5]. According to Jamaluddin et al. [6], SPF is a valuable raw material containing numerous holo- and α -cellulose and long fibers with high length-to-diameter ratios (L/D) suitable for pulp and paper production.

A past study achieved a 45.0% pulp yield from SPF using non-prehydrolysis soda anthraquinone (PHS-AQ) pulping with an 18% alkali charge and without bleaching

[7]. Prehydrolysis is a process prior pulping in DP production that allows a high cellulose content (minimum 94.0%) and low hemicellulose content (<6.0–7.0%) [8, 9]. This enables the conversion of separated hemicelluloses (hexoses and pentoses) into valuable products such as furfural, xylitol, and ethanol, which are often referred to as potential biorefinery units [9]. The same study [10] investigated non-PHS-AQ followed by bleaching of SPF using three sequences and 80.0% ISO pulp brightness (ISO 3688:1999, [11]) was obtained. Another source conducted non-PHS delignification of SPF and reported a 28.0% pulp yield, 79.1% brightness level, and 68.4% final cellulose content after bleaching with alkaline extraction in the presence of hydrogen peroxide [12]. However, currently, there are no reports on producing high-purity cellulose as dissolving pulp from SPF for textile fibers (viscose rayon and lyocell) and other cellulose derivatives, incorporating prehydrolysis and purification steps.

AQ has been considered a low-cost pulping additive for mill applications [13]. AQ, a redox catalyst first introduced in 1972 by Bach and Fiehn and explained in Anita et al. [14, 15], is employed in kraft and soda pulping to accelerate delignification and increase pulp

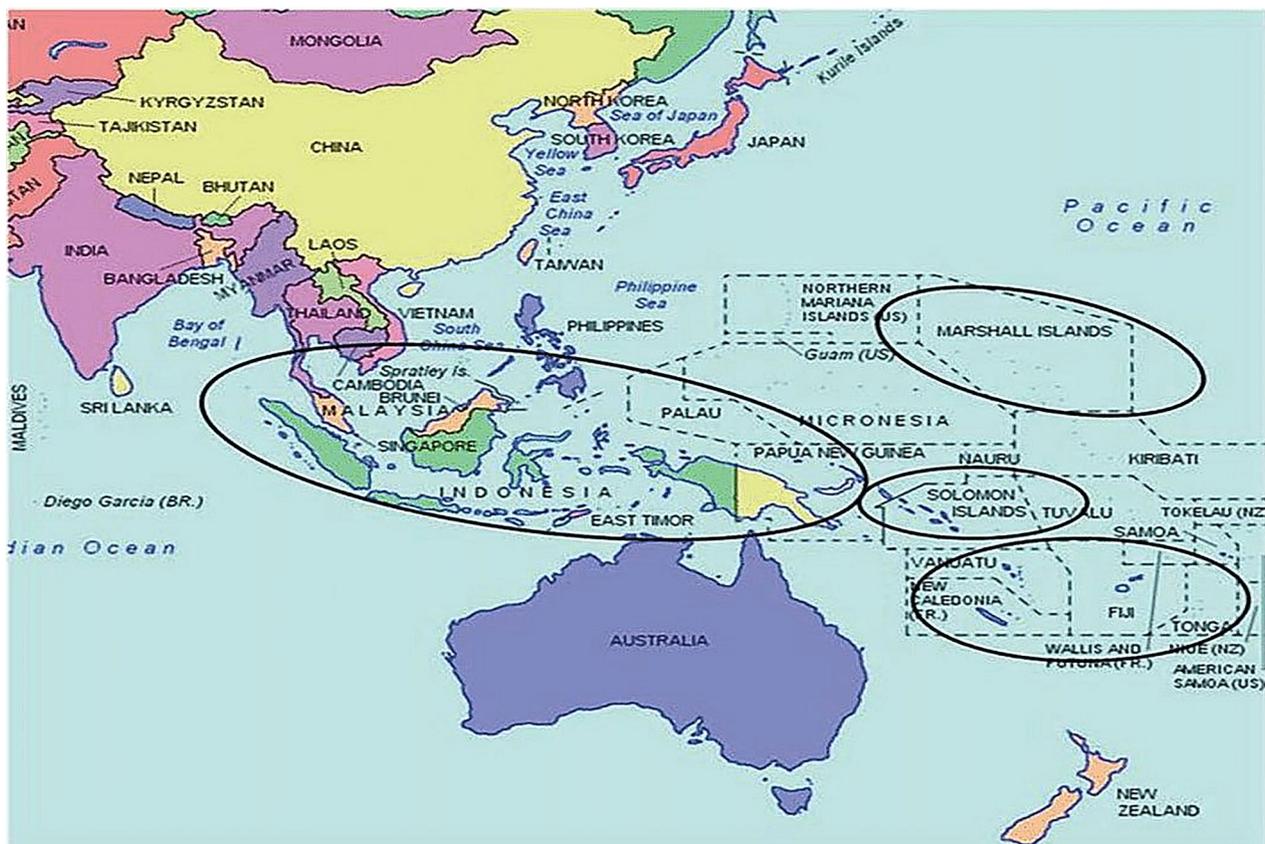


Fig. 1 Sago palm plantation countries indicated by black circles (Source: Konuma [2]. Licenced under CC BY 4.0)

yield at lower temperatures or chemical charges [13]. In comparison to the kraft process, the non-sulfur (soda) cooking method in combination with AQ offers remarkable benefits for biorefinery [16]. AQ is very useful in alkaline pulping, specifically when cooking non-woody materials [17]. However, its carcinogenic potential as evidenced by tests on mice leading to kidney and urinary bladder cancers, raises health concerns [18]. Impurities in AQ, such as 9-nitroanthracene, 1-hydroxyanthraquinone, and 2-hydroxyanthraquinone, can be harmful to human health. 2-methylanthraquinone (MAQ) has been reported as a safe additive with non-carcinogenic and anti-cancer or anti-tumor properties [14, 15, 19–25].

To explore the feasibility of producing dissolving grade pulps, this study assessed a combination of prehydrolysis followed by soda-MAQ (PHS-MAQ) cooking and totally chlorine-free (TCF) bleaching on SPF using peroxymonosulfuric acid (P_{sa}). In a past study, P_{sa} was successfully applied to produce high-quality dissolving pulp from *Nypa fruticans* pulp [8]. The chemical composition of SPF and the properties of the resulting DP were characterized. The advantages of using MAQ as a green additive in PHS cooking compared to PHS cooking without additives and commercial prehydrolysis kraft (PHK) processes were investigated. The delignification efficiency of the PHS-MAQ pulps and their ability to produce purified cellulose through bleaching were evaluated. Lignin, a by-product and an important raw material for biopolymers, was isolated from PHS-MAQ black liquor and characterized. To our knowledge, this is the first evaluation of SPF for dissolving pulp and lignin production.

Experimental

Materials

As the raw material for dissolving pulp, SPF was obtained from Muntai village in the Banten district on the Indonesian island of Bengkalis. Furthermore, its chips with 0.5 to 1.0 cm lengths were prepared after thorough washing and 90% sun drying, following established procedures [8, 26, 27]. The pulping catalyst, MAQ from Alfa Aesar, India, was generously provided by the University of Tsukuba, Japan. Peroxymonosulfuric acid (P_{sa} or H_2SO_5) used for bleaching was synthesized by combining 98% sulfuric acid (Wako Pure Chemical Industries, Ltd.) and 50% hydrogen peroxide aqueous solution (Mitsubishi Gas Chemical Company, Inc.) in a molar ratio of 1:3 at 70 °C based on established methods [28, 29].

Prehydrolysis and cooking of SPF

Prehydrolysis and cooking experiments were conducted in a stainless-steel reactor with a working volume of 300 mL, employing the conditions described in previous studies [8]. During prehydrolysis performed at 150 °C for

3 h, a liquor-to-solid ratio of 7 L/kg was used, incorporating distilled water as the liquor. For cooking, a temperature of 160 °C, cooking time of 1.5 h, liquor-to-solid ratio of 7 L/kg, and active alkali (AA) dosages between 15 and 25% were applied to the wet solid residues without any washing. Duration for the prehydrolysis and cooking were total time required for these processes (including the time to achieve the set temperatures, i.e., 30–40 min). The optimal times for the prehydrolysis (3 h) and cooking (1.5 h) were selected based on previous studies by Evelyn et al. [8]. Additionally, 0.03% of MAQ dosage based on the wood weight was used [30], and the results were compared to PHS without MAQ addition and PHK method with 30% sulfidity. The pulp generated was disintegrated using FRANK-PTI GmbH, followed by sieving, washing, and drying at 105 °C to obtain the yield.

TCF bleaching

A five-stage bleaching process, namely O– P_{sa} – E_p – P_{sa} – E_p , was used and the specific conditions for each stage were as follows [8]:

- (1) Oxygen (O) bleaching: O_2 pressure of 0.5 MPa, NaOH dosage of 2.0%, temperature and time of 115 °C and 60 min, and a pulp consistency (PC) of 30%.
- (2) Peroxymonosulfuric acid (P_{sa}) bleaching: H_2SO_5 dosage of 0.2%, temperature and time of 70 °C and 70 min, pH 3.0, and PC of 10%.
- (3) Alkaline extraction with hydrogen peroxide (E_p) bleaching: H_2O_2 dosage of 2.0%, NaOH dosage of 1.4%, temperature and time of 70 °C and 60 min, and PC of 10%.

The bleaching procedure with P_{sa} and peroxide was performed with slight modifications according to established methods [26, 29]. Only PHS-MAQ pulps with a kappa number less than 10 and adequate brightness and viscosity that were subjected to TCF bleaching. It is advisable to produce pulp with a kappa number around 10 to obtain bleaching properties utilizing this TCF sequence [26]. The brightness and viscosity values after each stage of TCF bleaching were determined as described in the chemical analysis of wood and pulp.

Chemical analysis of wood and pulp

The chemical analysis of wood meal prepared from chips was carried out using the following procedures [8]: holocellulose (TAPPI T249 cm-00) [31], α -cellulose (TAPPI T203 cm-99) [32], pentosan (SNI-01–1561-1989) [33], acid-insoluble lignin (Klason lignin, TAPPI T222 om-02) [34], acid-soluble lignin (UV spectrophotometry at the wavelength of 205 nm, TAPPI T222 om-011) [35], extractives (SNI 8401: 2017) [36], and ash

(TAPPI 211 om-07) [37] content. Correction for ash and extractive values were not made in this work, however comparison was carried out to similar works and studies with ash and/or extractive-free.

The pulps were characterized in terms of yield, kappa number (TAPPI T236 om-06)[38], brightness (SNI ISO 2470–1:2016) [39], and viscosity (TAPPI T230 om-08) [40] after cooking. Additionally, the bleached dissolving pulps were subjected to α -cellulose (TAPPI T203 cm-99), extractives (SNI 8401: 2017), and ash content (TAPPI 211 om-07) analyses.

Lignin precipitation and characterization

Black liquors generated from PHS-MAQ cooking at AA of 21, 23, and 25% were used for lignin precipitation. Furthermore, the precipitation procedure, initially described by H'ng et al. [41] and Chem et al. [42] and modified by Utami et al. [9] was followed. An illustration of the precipitation and purifying processes is presented in Fig. 2. The supernatant obtained after centrifugation steps, S-1 and S-2, were mixed and labeled as S-3. Pn and Pa represented the precipitate derived from S-3, while their weights were calculated as yields based on the wood lignin weight in the black liquor according to Eq. (1) and

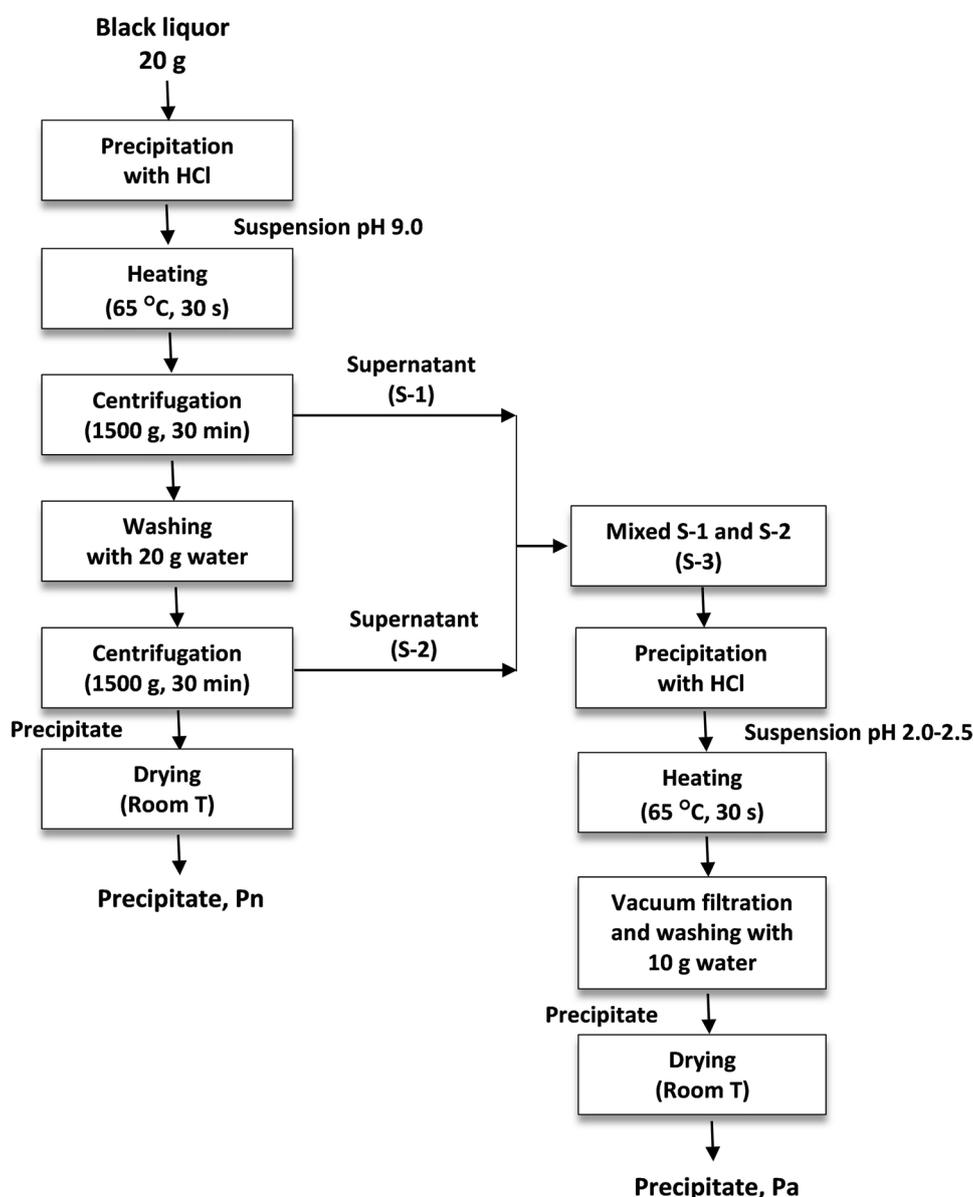


Fig. 2 The procedure for lignin precipitation at pH 9.0 (initial) and pH 2.0–2.5 (final) from PHS-MAQ cooking black liquor

Table 1 Chemical composition of SPF used in the study and its comparison with literature

Components (%)	SPF (this study)	SPF[33]	Nypa fronds[8]	Nypa fronds[36]	Vine stem[38]	Grape vine stalks[39]	Rice Straw [40]	Okra Stick [37]	Sugarcane Bagasse [34]	Oil palm fronds [45]
Holocellulose	61.5 ± 1.8	62.6	61.3	61.5	65.0	60.0	65.4	70.6	74.8	82.2
α-Cellulose	36.7 ± 1.4	35.5	37.3	35.1	35.0	36.0	38.5	34.3	47.4	47.6
Pentosan	24.8 ± 1.2	27.1	24.0	26.4	30.0	24.5	19.1	16.9	–	–
Acid-insoluble lignin	19.2 ± 0.5	18.3	17.5	17.8	28.1	39.6	12.7	25.2	20.4	15.2
Acid-soluble lignin	2.6 ± 0.1	–	0.8	1.9	–	–	–	–	–	–
Ash	6.3 ± 0.1	–	16.5	11.4–11.7	3.9	3.9	17.2	0.6	1.7	0.7
Extractives	7.4 ± 0.1 (Alc:DCM)	–	1.5 (DCM)	1.9 (Ac)	11.3 (Alc:benz)	1.8 (DCM)	1.1 (DCM)	2.9 (Ac)	3.2 (Alc:Ac)	1.4 (Alc:benz)

DCM: dichloromethane; Alc: benz alcohol; benzene; Ac: acetone

The composition for cellulose (holo and α-cellulose) and lignin in Tamunaidu et al. [46] was ash-free

The holocellulose values in Jahan et al. [47, 50] and α-cellulose value in Jahan et al. [50] were extractive-free

Eq. (2). The mass of precipitated lignin at pH 9 was much less than at pH 2–3 thus was assumed zero, thus Pa used in all discussion.

$$\begin{aligned} & \text{Lignin mass in black liquor} \\ &= \text{lignin mass in SPF} \\ & - \text{lignin mass in hydrolysate} \\ & - \text{lignin mass in pulp,} \end{aligned} \quad (1)$$

$$\text{Yield of precipitated lignin} = \left(\frac{\text{Mass of precipitated lignin at pH2} - 3}{\text{Weight of hydrolysate} + \text{wood samples}} \times \text{lignin mass in black liquor} \right) \times 100\%. \quad (2)$$

Samples of the precipitated lignin were determined for Klason lignin and ash content using the previously described procedure. Samples were also sent to an accredited laboratory for characterization of the presence of atomic functional groups or bonds within molecules using Fourier transform infrared spectroscopy (FT-IR, Shimadzu IRPrestige-21, with absorption spectra in the 4650 to 450 cm^{-1} regions) and morphology analyses using Scanning electron microscopy (SEM, Hitachi Flexsem 1000). The characteristics of precipitated lignin were compared to lignin obtained from *Eucalyptus pellita* [9] and literature data.

Results and discussion

Chemical characteristics of SPF

Table 1 presents the percentage of holocellulose, α -cellulose, lignin, alcohol–dichloromethane (DCM)-soluble extractives, and ash in SPF used in this study, along with a comparison to previous studies (determined with ash and extractive or non-ash and extractive-free). The chemical composition, i.e., α -cellulose, holocellulose, pentosan, and acid-insoluble lignin of SPF in this work is in agreement to SPF used in other study, also with non-ash and extractive-free [43]. The α -cellulose content of studied SPF was $36.7\% \pm 1.4$, making it suitable for pulp and paper production due to being close to or greater than the required level, i.e., 40.0% [44, 45]. The identified α -cellulose content in this study was comparable to the values of SPF analyzed by Sunardi et al. [43] as well as other non-wood plants such as *Nypa* palm fronds (NPF), grapevine stalks, vine stem, rice straw, and okra stick, determined with ash and extractive or non-ash and extractive-free (34.5–38.5%) [8, 43, 46–51]. SPF in this work exhibited a relatively high amount of holocellulose ($61.5 \pm 1.8\%$), showing similar results to those of the aforementioned non-wood plants, except for okra stick. Holocellulose content $> 60.0\%$ of non-wood materials is acceptable for pulp and paper production [48, 52]. Okra sticks, oil palm fronds, sugarcane bagasse (SB),

some bamboo, softwoods, and most hardwoods exhibited higher holocellulose (> 70.0 – 82.2%) and α -cellulose (> 40.0 – 56.0%) content than the non-wood materials [43, 53–55].

The investigated SPF contained $24.8 \pm 1.2\%$ pentosan, $19.2 \pm 0.5\%$ acid-insoluble lignin, $2.6 \pm 0.1\%$ acid-soluble lignin, $7.4 \pm 0.1\%$ alcohol-DCM-extractable substances (extractives), and $6.3 \pm 0.01\%$ ash. These values fell within the range of chemical characteristics observed in many

non-wood plants [6, 8, 43, 45–51, 55]. Pentosan and lignin were valuable by-products separated from the fiber matrix during DP production. Pentosan contained in hemicellulose could be extracted in the prehydrolysis step before kraft, alkaline sulfite, or soda cooking, for furfural production. Dissolved lignin from the sulfur-free soda cooking liquor was applicable as raw materials for biopolymers, fuels, and chemicals. The acid-insoluble lignin content in the SPF used was similar to that of SPF (both are non-ash free), NPF, and SB reported in previous studies [6, 8, 43, 45]. This value was higher than in oil palm fronds and rice straw (12.7–15.2%) [51, 54], but lower compared to vine stem [48], grapevine stalks [49], and okra stick [47].

The ash content of SPF ($6.3\% \pm 0.01\%$) in this study fell between that of NPF and other non-wood plants. With regard to extractive, the content of this component in compared non-wood plants seems to be affected by the solvent used. For example, except in oil palm fronds, high extractive values ($\geq 2.9\%$) were observed when alcohol-DCM, alcohol–benzene, alcohol–acetone, or acetone was used as the solvent including SPF in this work with $7.4 \pm 0.13\%$. Other investigators have reported similar amounts of extractives, ranging from 6.1–8.4% in whole and depleted SB in alcohol–benzene mixture [56]. The presence of high extractive and mineral contents, such as silica in ash, might complicate the utilization of DP for creating cellulose derivatives. This could equally cause various operational issues including scaling, causing difficulties in the cleaning work of the equipment. Although in the past sodium silicate has been used as stabilizer in oxygen bleaching, its use has been replaced by non-silicon stabilizer, i.e., phosphonates and macromolecule compounds [57]. Thoroughly washing the biomass before processing to remove sand could reduce the amount of silica in the pulp [58]. In previous studies, 98% of ash content in NPF was successfully removed by using prehydrolysis, soda-anthraquinone cooking, and totally chlorine-free bleaching processes similar to those used in this study. SPF possesses a reasonable amount of α -cellulose,

hemicellulose, and lignin, increasing its potential as raw materials for the production of cellulose derivatives, bio-fuels, and chemicals. Therefore, the suitability of this biomass for high-purity cellulose and lignin was examined in the current study.

Effect of cooking methods and MAQ addition on pulp characteristics

PHS-MAQ cooking (0.03% MAQ), PHK, and PHS cooking with no additives were conducted at varying AA levels between 17 and 25%, yielding the results presented in Fig. 3. The combination of prehydrolysis with MAQ additive in soda cooking of SPF improved delignification, as indicated by a kappa number of 5.2–7.3 points compared to PHK cooking at the same AA levels of 17, 23, and 25%. These results were also consistent with another study in which PHS-MAQ cooking (0.1% MAQ) of *Acacia crassicaarpa* at AA levels between 17 and 23% generated a smaller kappa number of 2.0–14.4 points than PHK cooking [14].

The pulp yields from PHS-MAQ cooking at different AA levels of 15–25% ranged from 20.3 to 22.5% as indicated in Fig. 3. To date, no literature was found on PHS cooking employing SPF as well its comparison to PHK-MAQ cooking. In a past study, Tze et al. [10] reported higher yields of 42.3–44.9% at 16–18% AA but with PHS-AQ cooking and no prehydrolysis step applied compared to PHS-MAQ cooking in this study. Arnata et al. [12] applied enzymatic hydrolysis followed by soda cooking of powdery SPF at 100 °C for 1–3 h with 5–15% AA and obtained only 11.1–13.0% yields. The pulp yields described earlier in this work for PHS-MAQ cooking were around similar values to the pulp yields of

19.9, 21.1, and 21.5% derived from PHK cooking at the same AA levels of 17%, 21%, and 25%, indicating lower AA to achieve the same yields. Francis et al. [59] found that non-PHS cooking of sugar maple with 0.075–0.116% MAQ was superior (i.e., 1.2–1.7% higher pulp yields) than non-PHK cooking alone. These results indicated the benefits of PHS cooking with MAQ additive than commercial PHK process utilizing SPF such as enhanced delignification and comparable yield at lower AA used, suggesting a better and more economical process.

PHS cooking without MAQ addition showed the lowest performance compared to the other two processes, namely PHS cooking with 0.03% MAQ and PHK cooking at similar AA levels of 17, 21, and 25%. In comparison to PHS without the additive, the PHS-MAQ process was able to increase yields by 4.6–4.9% and reduce kappa numbers by 1.6–3.1 points for PHS without additives. AQ has been renowned for its benefits in the pulp and paper industries, such as incremental pulp production, energy reduction, and protection of wood resources [12]. This material was considered a safe cooking additive in the non-sulfur process due to not generating toxic H₂S and being suitable for biorefinery processes [14]. Jamaluddin et al. [7] stated 50% reductions in the kappa number and a 1.2–1.6% increase in pulp yields after non-PHS-AQ cooking of powdery SPF compared to using non-PHS alone at 14–18% AA. Similarly, several studies reported that non-PHS cooking with 0.075–0.2% AQ addition produced better results than PHS cooking alone, such as increased screen pulp yields and enhanced delignification rates for other non-wood materials (wheat and rye straw, cotton stalks, and bast kenaf fibers) [60–62]. Methyl anthraquinone or MAQ is methylated derivative of anthraquinone which is regarded a safe additive in pulping, slowly replacing AQ. According to Anita et al. [30], the inclusion of 0.03% MAQ in non-PHK cooking led to a reduced kappa number (3.0–4.2 points) than ordinary non-PHK cooking. The 2-MAQ addition in PHS-MAQ cooking (0.1% MAQ) of *A. crassicaarpa* reduced the kappa number in PHS without MAQ by 9.9–10.6 at AA levels between 20 and 23% [14]. Based on these backgrounds and results, SPF pulps processed by 3-h prehydrolysis and 1.5-h soda-MAQ cooking at AA levels between 15 and 23% were further checked for brightness and viscosity as discussed in the following section.

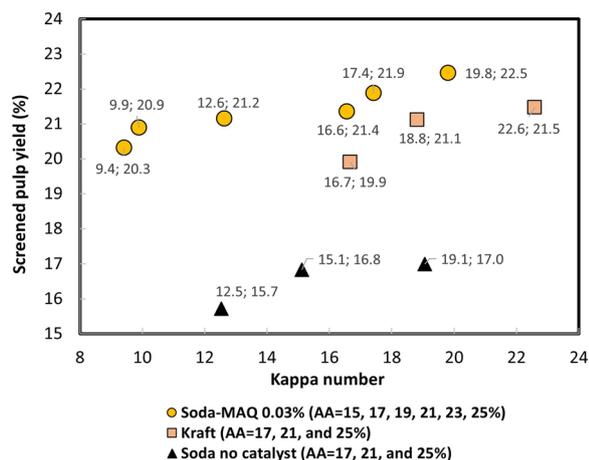


Fig. 3 Screened pulp yields and kappa number of SPF after PHK (with 30% sulfidity), PHS-MAQ and PHS cooking (prehydrolysis: 150 °C-3 h, cooking: 160 °C-1.5 h, MAQ dosage: 0.03%, liquor-to-solid ratio: 7 L/kg)

Effect of active alkali on the brightness and viscosity of soda-MAQ unbleached pulps

Figure 4 displays the brightness and viscosity profiles of PHS-MAQ pulps when applying different doses of active alkali (15 to 25%). The results showed that the pulp brightness elevated from 18.3 to 33.6% ISO with increasing AA dosage up to 23% and a kappa number

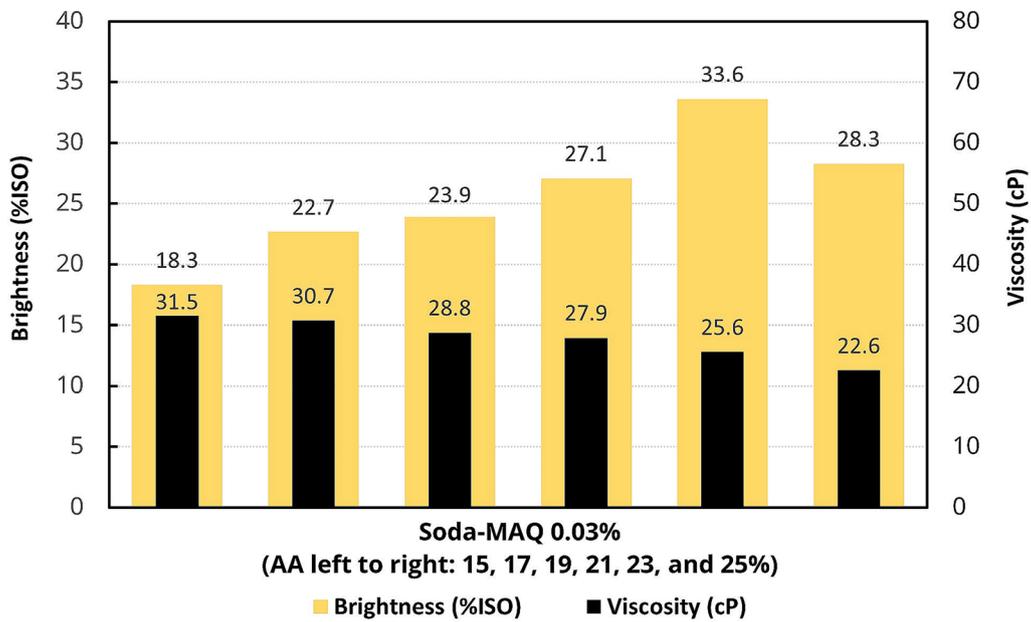


Fig. 4 Brightness and viscosity profiles of SPF pulp after PHS-MAQ cooking under various AA levels (prehydrolysis: 150 °C-3 h, cooking: 160 °C-1.5 h, MAQ dosage: 0.03%, liquor-to-solid ratio: 7 L/kg, active alkali: 15–25%). Yellow and black filled bars present the brightness and viscosity values, respectively

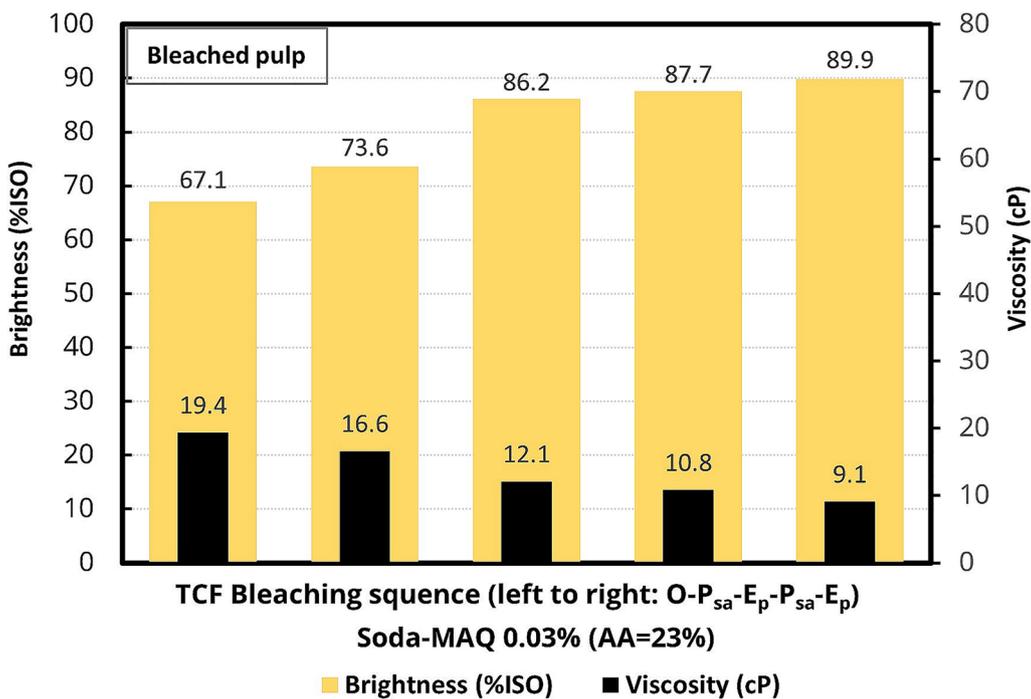


Fig. 5 Brightness and viscosity profiles during the TCF bleaching of SPF pulp (TCF-bleaching sequence: O-P_{sa}-E_p-P_{sa}-E_p). Yellow and black filled bars present the brightness and viscosity values, respectively



Fig. 6 Transformation of SPF unbleached pulps into pure cellulose during five-stage TCF bleaching at 23% AA dosage (O: oxygen; P_{sa}: peroxymonosulfuric acid; E_p: alkaline extraction with hydrogen peroxide)

of 9.9 points was recorded. Similarly, the increase of ISO brightness from 22.7 to 23.9% with the decrease of the kappa number from 17.4 to 16.6 accompanying the AA increase from 17 to 19%. Meanwhile, the brightness was mainly influenced by the presence of lignin measured as a kappa number of 9.4 points, a further increase in AA dosage from 23 to 25% even lowered the brightness value. These results suggest that ISO brightness of pulp is not only affected by the lignin content. Niskanen [63] and Maryana et al. [26] reported that high AA dosage could alter the light scattering coefficient, leading to lower brightness due to fiber softening. The results in this study aligned with those obtained by Maryana et al. [26] where the brightness of unbleached SB elevated upon AA application from 12 to 16%, but decreased after increasing AA from 17 to 19%. Moreover, viscosity representing the degree of polymerization (DoP) of cellulose, was assessed using an equation obtained from the viscosities as centipoises (cP) in this case determined using copper ethylenediamine (CED) as a solvent and a capillary viscometer [64]. Concerning the viscosity values of PHS-MAQ unbleached pulps, a decreasing trend from 31.5 to 22.6 cP (corresponding to DoP values of 1677–1448) was observed as the AA levels increased from 15 to 25%, indicating the degradation of SPF cellulose fibers after the increase in AA concentration. The pulp at 23% AA showed superior characteristics thus was further processed to bleaching.

Effect of TCF-bleaching sequence on the brightness and viscosity of PHS-MAQ pulp

In prior investigations, the use of TCF-bleaching procedures involving oxygen (O), peroxymonosulfuric acid (P_{sa}), and alkali extraction in the presence of hydrogen peroxide (E_p) has been demonstrated to be effective in producing DP from both wood and non-wood materials. These pulps have been shown to meet the requirements of the Indonesian National Standard (SNI) for α -cellulose

($\geq 94\%$), brightness ($> 88\%$), and viscosity (> 6.2 cP) [8, 29, 65]. In this study, a five-stage sequence of TCF bleaching (O–P_{sa}–E_p–P_{sa}–E_p) was employed to obtain the required DP. During the bleaching process, the pulp brightness gradually increased from 67.1% to 73.6%, 86.2%, 87.7%, and 89.9% ISO following the stages of O, first P_{sa}, first E_p, second P_{sa}, and second E_p, respectively (Fig. 5). At the same time, the viscosity decreased from 19.4 to 9.1 cP. Figure 6 demonstrates the transformation of unbleached pulp brightness until it meets the required standard.

Previous studies have reported similar observations in the properties of SB and *Eucalyptus globulus* pulps following the application of a bleaching sequence. For SB pulps produced through PHS-AQ cooking at AA 16%, the brightness level increased from 70 to 89.1% ISO, while the viscosity decreased from 19.3 to 6.4 cP after the O–P_{sa}–E_p–P_{sa}–E_p bleaching sequence [26]. Similarly, *E. globulus* pulps obtained from PHS-AQ cooking at AA 18% exhibited a similar trend, with an increase in brightness level from 71.2 to 88.4% ISO and a decrease in viscosity value from 31.5 to 6.0 cP after the same bleaching sequence [54]. Notably, there is a gap in the existing literature regarding the combined effects of PHS-MAQ and this TCF bleaching on pulp properties.

The final bleached pulp exhibited favorable properties, with a brightness of 89.9% ISO, α -cellulose content of 94.3%, and ash content of 0.13% (Fig. 5 and Table 2). These properties indicate the suitability of the bleached pulp for the production of viscose rayon and other cellulose derivatives. Additionally, the low pentosan content, i.e., 0.6% (Table 2) suggests that most of pentosan was removed in the prehydrolysate and cooking liquor. The extraction of pentosan from these liquors creates a platform for the production of value-added products such as biofuels, biochemicals, and biomaterials. Lignin dissolved in the PHS-MAQ cooking black liquor can also be developed in future into a promising biorefinery

Table 2 Chemical composition of SPF raw material and pulp

Components	α -Cellulose (%)	Pentosan (%)	Extractives (%)	Ash (%)
Raw material	36.7 ± 1.4	24.8 ± 1.2	7.4 ± 0.1	6.3 ± 0.01
Pulp before the TCF bleaching*	78.5 ± 1.1	3.5 ± 0.7	3.1 ± 0.1	3.1 ± 0.02
Pulp after the TCF bleaching*	94.3 ± 0.9	0.6 ± 0.1	0.6 ± 0.1	0.13 ± 0.01

*Calculated values were based on the pulps

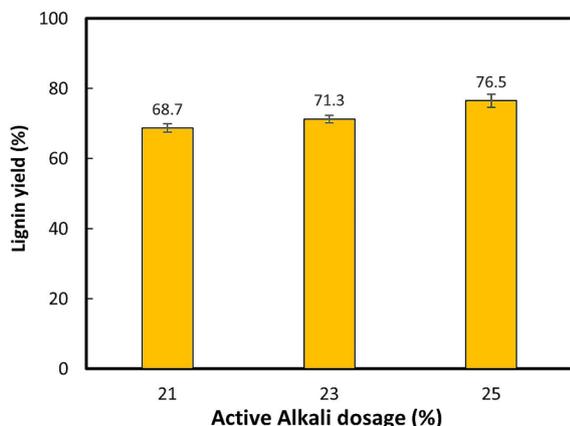


Fig. 7 The yields of precipitated lignin obtained from PHS-MAQ cooking black liquor at AA levels between 21 and 25%

since the lignin can be readily precipitated using a suggested technique such as lignoboost process [8].

SPF lignin precipitation and characterization

Acid precipitation is one of the effective methods to separate lignin from black liquors, hence hydrochloric acid was employed in this study to separate the dissolved lignin. The yields of precipitated lignin (Pa) obtained from the PHS-MAQ cooking of SPF at AA levels of 21–25% were between 68.7 ± 1.2% and 76.5 ± 1.8% based on the wood lignin weight in the black liquor as indicated in Fig. 7. Using the same precipitation lignin protocol, Utami et al. [14] obtained 85.6–88.8% of lignin yield from PHS-MAQ cooking of *A. crassicarpa* and *E. pellita*. Hardwoods typically contain a higher initial lignin content compared to non-wood materials like SPF, thereby

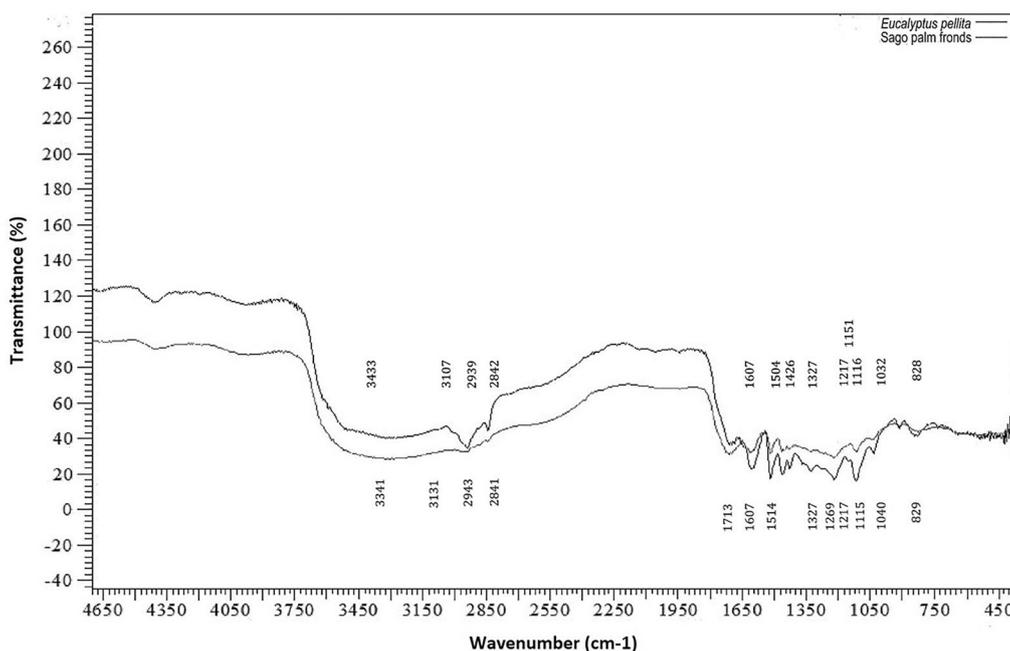


Fig. 8 The FT-IR spectra of precipitated lignin obtained from PHS-MAQ cooking black liquor: SPF versus control (*E. pellita*)

Table 3 FT-IR characteristic bands assignments for SPF precipitated lignin compared to hardwood

Absorption band location (cm ⁻¹)*	Type of vibration in different absorption bands	SPF wavenumber (cm ⁻¹)	<i>E. pellita</i> 's wavenumber (cm ⁻¹)	Birch's wavenumber (cm ⁻¹) [68]
3500–3100	Stretching vibrations of alcoholic and phenolic OH groups involved in hydrogen bonds	3341–3131	3433–3107	3273–3393
2937 and 2850	Stretching vibrations of C–H bonds of the methyl and methylene groups	2943 and 2841	2939 and 2842	2937 and 2928
1715–1710	Non-conjugated carbonyl	1713	–	1712–1715
1665–1655	Conjugated carbonyl	–	–	–
1605–1595, 1515–1500 and 1430–1425	Aromatic ring vibrations	1607, 1514, and 1425	1605, 1518, and 1426	1590–1607, 1515–1420
1340–1330	Vibrations of syringyl rings and stretching vibrations of C–O bonds	1327	1327	1328
1272–1220	Vibrations of guaiacyl rings and stretching vibrations of C–O bonds	1269–1217	1217	–
1150–1140	Deformation vibrations of C–H bonds in guaiacyl rings	–	1151	–
1125	Deformation vibrations of C–H bonds in syringyl rings	–	–	–
1085	Deformation vibrations of C–O bonds in secondary alcohols and aliphatic ethers	–	–	–
1130–1035	Deformation vibrations of C–H bonds in the aromatic rings and deformation vibrations of C–O bonds in primary alcohols	1115–1040	1116–1032	–
945 and 870–850	Deformation vibrations of C–H bonds in associated with aromatic rings	829	828	890–830
430–600	Stretching vibrations of C–S bonds	–	–	600–430

* Band interpretation was based on several previous studies [67–72]

SPF and *E. pellita* lignin were obtained from PHS-MAQ cooking and precipitated with hydrochloric acid, whereas birch lignin was obtained from soda-AQ cooking and precipitated with sulfuric acid [68]

often resulting in higher yields than non-woods. Depending on pH and wood species, Hubbe et al. [66] reported a wide range of lignin recovery (16–91%) in soda black liquor using acids as the precipitation agent.

The FT-IR spectra of the precipitated lignin and their characteristic band assignments derived from PHS-MAQ cooking black liquor of SPF (compared to *E. pellita* and birch) are shown in Fig. 8 and Table 3, respectively. Common FT-IR spectra between 4650 and 450 cm⁻¹ (obtained from previous studies with precipitated and commercial lignin) were used for the interpretation of SPF lignin [67–72]. In general, the FT-IR spectrum of SPF followed 75% patterns of those lignin from *E. pellita* (precipitated with HCl), but only showed 45% similar patterns to the lignin from birch (precipitated with H₂SO₄-literature data). Each lignin IR spectrum has a wide band from 3500 and 3100 cm⁻¹ due to OH stretching vibrations. The absorption bands at wavenumbers 2943 cm⁻¹ and

2841 cm⁻¹ were vibrations indicating the presence of methyl C–H groups [67–71], while 1713 cm⁻¹ signified non-conjugated carbonyl [68]. The bands at 1607 cm⁻¹ and 1514 cm⁻¹ demonstrated aromatic ring functional groups [68–70], 1327 cm⁻¹ and 1217 cm⁻¹ suggested syringyl and guaiacyl ring functional groups, respectively [70, 71], while 1115 cm⁻¹ indicated ether groups [71]. The presence of absorption bands at 1035–1130 cm⁻¹ was identified in the absorption spectra, which is caused by the deformation vibrations of C–H bonds in the aromatic rings or C–O bonds in primary alcohols [71, 72]. Both SPF and *E. pellita* did not show C–S stretching (characteristic bands at 430–600 cm⁻¹) as commonly observed for precipitated lignin using sulfuric acid.

SEM images at 10× magnification reveal different morphology, i.e., size and shape between SPF (non-wood) lignin and *E. pellita* (hardwood) lignin although these lignins were precipitated utilizing the same method

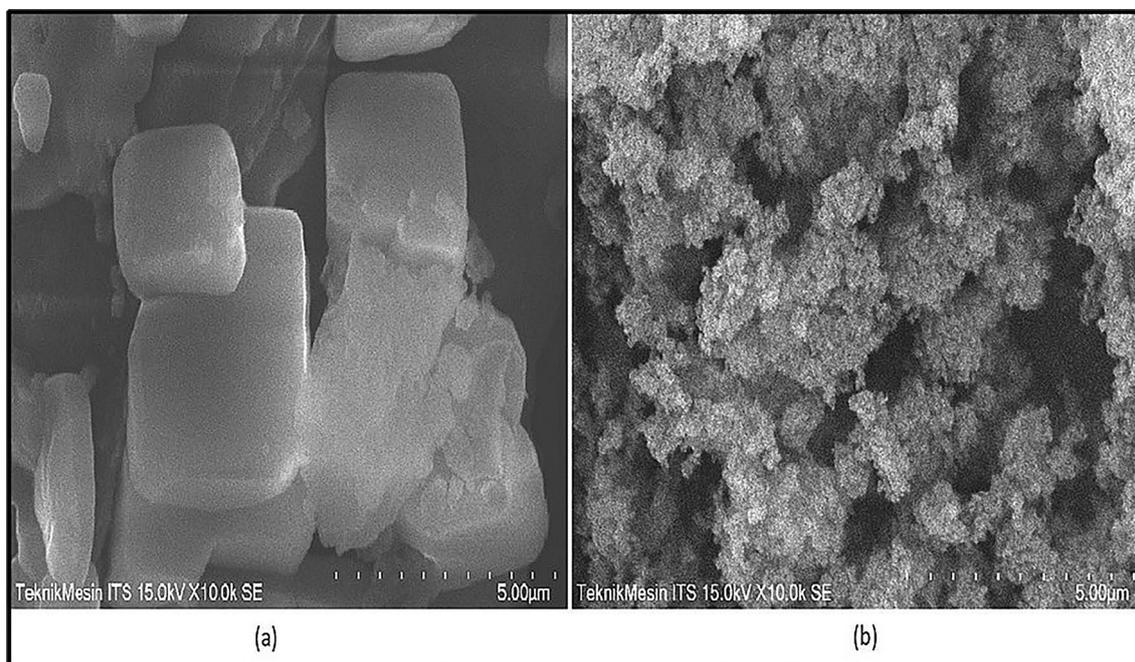


Fig. 9 The SEM images of precipitated lignin (10x magnification): SPF (a), and *E. pellita* (b)

(Fig. 9). Lignin derived from *E. pellita* showed an irregular shape and more compact structure than lignin derived from SPF. Wide cavities in the morphology of SPF lignin, indicating degradation of the material [70, 73]. Further studies will be needed to investigate SPF lignin using other analytical techniques to obtain more insights into the morphology, distribution, and behavior of the obtained lignin.

Conclusions

In conclusion, this study demonstrated the successful production of high-purity cellulose as dissolving pulp from SPF through a combination of prehydrolysis, soda-MAQ cooking, and five-stage TCF bleaching. Among the various cooking methods tested, the prehydrolysis soda-MAQ process exhibited superior performance, leading to lower kappa number and/or higher yield at the same active alkali levels compared to prehydrolysis kraft and soda cooking. The obtained dissolving grade cellulose possessed excellent properties, including 89.9% ISO brightness, 94.3% α -cellulose content, 9.1 cP viscosity, and 0.13% ash content, fulfilling the requirements for applications in viscose rayon and cellulose derivatives. The FT-IR spectrum of SPF lignin exhibited almost similar structure to the *E. pellita*'s lignin functional groups and the SEM results revealed different size and shape to the hardwood lignin.

Abbreviations

DP	Dissolving pulp
SPF	Sago palm fronds
AQ	Anthraquinone
MAQ	2-Methylantraquinone
PHS	Prehydrolysis soda
PHS-AQ	Prehydrolysis soda anthraquinone
PHS-MAQ	Prehydrolysis soda methylantraquinone
PHK	Prehydrolysis kraft
TCF	Totally chlorine-free bleaching
AA	Active alkali
O	Oxygen
Ep	Alkaline extraction with hydrogen peroxide
P _{sa}	Peroxymonosulfuric acid
PC	Pulp consistency
DCM	Dichloromethane
L/D	Length-to-diameter ratios
FT-IR	Fourier transform infrared spectroscopy
SEM	Scanning electron microscopy
CED	Copper ethylenediamine
DoP	Degree of polymerization

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Author contributions

E designed and performed the experiments, analyzed the data, and largely contributed to the writing of the manuscript. All authors contributed to the interpretation and discussion of the results. All authors have read and approved the final manuscript.

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Availability of data and materials

The datasets used and analyzed in the current study are available from the corresponding author upon reasonable request.

Declarations

Ethics approval and consent to participate

Not applicable.

Consent for publication

Not applicable.

Competing interests

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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