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ALKALINE DELIGNIFICATION OF COCOA (*THEOBROMA CACAO L.*) POD HUSK FOR CELLULOSE FIBERS EXTRACTION

Andri Cahyo Kumoro[1,2] ✉, Aji Prasetyaningrum[1], Kristinah Haryani[1], Ratnawati Ratnawati[1], Yumna Agustia Nursalsabila[1], Ziva Putri Yonanta[1], Misbahudin Alhanif[3]

[1] Department of Chemical Engineering, Faculty of Engineering, Universitas Diponegoro, Jl. Prof. Jacub Rais, Semarang, 50275, Indonesia
[2] Institute of Food and Remedies Biomaterials (INFARMA), Department of Chemical Engineering, Faculty of Engineering, Universitas Diponegoro, Jl. Prof. Jacub Rais, Semarang, 50275, Indonesia
[3] Department of Chemical Engineering, Faculty of Industrial Technology, Institut Teknologi Sumatera, Lampung Selatan, 35365, Indonesia
✉ *andrewkomoro@che.undip.ac.id*

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Abstract. As one of the cocoa (*Theobroma cacao L.*) processing by-products, cocoa pod husk contains about 26.4 % lignin-bound cellulose. Therefore, a delignification process is required to obtain high-purity cellulose by removing the lignin. This study aims to extract cellulose from a cocoa pod husk through alkaline delignification using caustic soda solution. In addition to alkaline solution concentration and liquid-to-solid ratio, the time and temperature of the alkaline delignification were studied in this research to determine the best delignification conditions based on the yield, purity, and characteristics of the cellulose produced. The resulting cellulose can be further used in numerous industrial applications.

Keywords: caustic soda, cellulose, cocoa, delignification, extraction.

1. Introduction

Cocoa *(Teobroma cacao L.)* is a tropical plant of the *Malvaceae* family and the genus *Theobroma*, which originates from the lower eastern equatorial slopes of the Andes of South America.[1, 2] Although *Teobroma* comprises more than 22 species, *Teobroma cacao L.* has been the most significant and broadly planted for its seed's economic benefit.[3] With a production capacity reaching 667,296 tons in 2023, Indonesia establishes itself as the 3rd largest cocoa bean producer globally after the Ivory Coast and Ghana, which produce 2,230,000 tons and 1,108,663 tons of cocoa beans, respectively.[4] Nonetheless, processing of fresh cocoa fruits to obtain one ton of dried cocoa beans will produce about 10 to 15 tons of cocoa pod, which is regarded as an undesirable waste, and hence underutilized.[5] Actually, a careful traditional procedure can transform cocoa pod husk (CPH) into a natural fertilizer by introducing CPH to soil that allows the recovery and recycling of nutrients in plant-suitable forms after decomposition.[6] Nevertheless, in many cocoa-producing countries, the pods are usually left at the cocoa plantation site and undergo natural decomposition, generating an unpleasant odor and aggravating the spread of plant diseases, specifically black pod rot caused by *Phytophthora spp.*[7] In fact, CPH comprises fibrous constituents, namely cellulose (19–26 %), hemicellulose (9–13 %), lignin (14–28 %), and 6–13 % pectin.[8] For that reason, CPH possesses a great potential as a non-wood source of cellulose that supports the movement against global deforestation. Cellulose bears numerous attractive industrial applications, namely cosmetics, food, biomaterials,[9] paper and textiles, biofuel,[10] and so on. Recently, utilization of biomaterials has widened from domestic product production to advanced goods, *i. e.*, batteries, clothing, and paints.[9] This specialty material has been proven to be suitable for inclusion in various products due to its unique attributes, including dimensions, durability, and low molecular weight.[10]

As cellulose exhibits a variety of industrial and economic benefits, its extraction from CPH has received considerable interest from researchers worldwide. In addition to the botanical characteristics of the raw CPH, the methods applied for extraction and separation exhibit significant effects on obtaining high yield and purity of cellulose.[11] To obtain high-purity cellulose, the chemical delignification process using alkali is the most effective method for application to various lignocellulosic biomass, which results in a higher yield and better cellulose characteristics compared with other chemical, mechanical, physico-chemical, and biological methods.[9,12–14] Ying *et al.*[15] indicated that alkaline pretreatment removed 52.5 % of lignin in sugarcane bagasse. Unlike acid hydrolysis, which often damages cellulose and creates unwanted by-products,[16] alkaline delignification offers a milder and easier process.[17] While organosolv methods produce high-quality results and useful lignin by-products, they require costly solvents and high-pressure equipment, making them less practical for large-scale use.[18] Biological methods, like using fungi and enzymes to remove lignin, are good for the environment but take a long time and don't remove enough lignin.[19] Mechanical processes, like steam explosions and milling, are frequently employed as pretreatments; nevertheless, the extreme conditions of steam explosions can induce substantial structural alterations, thereby impacting their applicability in specific uses.[20]

The chemical delignification of lignocellulosic biomass utilizing alkaline solution, specifically sodium hydroxide (NaOH) solution, is highly selective and efficient as a result of its strong capacity to cleave the phenolic bonds α-O-4, β-O-4, β-O-5, and β-β in lignin, subsequently followed by the cleavage of non-phenolic bonds β-O-4 and C-C bonds.[21] During alkaline delignification, the alkali destroys the lignin structure through the swelling of the CPH cell walls, lowering lignin crystallinity, and finally increasing the surface area of cellulose.[12] Alkali concentration, alkali type, pressure, particle size, temperature, liquid-to-solid (L/S) ratio, and delignification time influence the cellulose yield and characteristics.[12] Tao *et al.*[22] confirmed that sodium hydroxide (NaOH) concentration, L/S ratio, and reaction temperature significantly influenced the delignification of *(Achnatherum splendens)*. Rafidah *et al.*[23] reported that the concentration of NaOH had a significant influence on increasing cellulose content and reducing lignin content during the delignification of *Acacia mangium*. Optimal conditions were attained at a temperature of 70 °C, a delignification time of 3 hours, and a NaOH concentration of 5.50 % (w/v), resulting in a cellulose content of 47.66 %. Under these conditions, the removal efficiencies for hemicellulose and lignin were 36.16 % and 12.66 %, respectively.[23] Based on a process parameter screening experiment on cellulose extraction from Sengon (*Albizia chinensis*) sawdust employing alkaline delignification, Cahyani *et al.*[24] concluded that the NaOH concentration and L/S ratio had a significant effect on the cellulose produced compared to delignification temperature and time. The optimal conditions were achieved at a NaOH concentration of 2 % and an L/S ratio of 19.20:1, resulting in a composition of 52.49 % cellulose, 8.01 % hemicellulose, and 22.2 % lignin.[24] However, further statistical evaluation revealed that the influence of delignification time was more pronounced than that of the delignification temperature. A key advancement is mixing alkaline treatments with oxidizing agents such as H_2O_2, which creates alkaline peroxide pretreatments that make it easier to remove lignin and reduce polysaccharide degradation.[25] Amrillah *et al.*[26] performed alkaline delignification of CPH using a NaOH solution, followed by bleaching with 40 % H_2O_2, which yielded an optimal composition of 62.0 % cellulose, 8.9 % hemicellulose, and 23.9 % lignin. In addition, the study compared the effectiveness of NaOH and KOH as delignification agents. The results demonstrated that NaOH treatment produced a higher cellulose content (61.6 %) compared to KOH (50.9 %). The variation in optimal parameters among studies may be attributed to differences in the botanical sources of the biomass. Alkali solutions have lower corrosiveness compared to acidic solutions, and fractionation can be conducted at moderate conditions and atmospheric pressures. Therefore, employing this approach may obviate the necessity for expensive building materials and bespoke designs to mitigate corrosion susceptibility and extreme reaction circumstances.[23] Moreover, the leftover alkali solution that was used in the extraction process can be recovered and reused in the fractionation process that follows.[21] In addition, engineering a closed-loop system to recycle the alkaline reagent can also improve the sustainability of this method. Industrial-scale processing favoured alkaline delignification due to its cost-effectiveness, efficiency, and suitability.[27] Its versatility and efficacy across a wide range of biomass types highlight its important function in cellulose extraction.

Despite the increasing interest in cellulose extraction from diverse agricultural residues, only a few studies have thoroughly investigated the optimization of alkaline delignification conditions specifically for CPH by a systematic multi-variable analysis. This study distinguishes itself by analyzing the concurrent effects of NaOH concentration, L/S ratio, delignification time, and temperature – variables frequently investigated in isolation in prior research – on cellulose yield and purity. This research highlights CPH, a readily available but often overlooked waste from cocoa production, helping to reduce waste and support circular economy efforts in developing countries.

Recovering cellulose from CPH may result in the development of a commercial commodity and eventually generate more earnings for farmers.[24] Further conversion of CPH to highly valuable products, including biomaterials for both food and non-food applications, may serve as a strategic method to sustain cocoa production. Accordingly, this effort will support economic and welfare development of the cocoa-producing countries, which are mostly categorized as developing countries. This study aims to extract cellulose from CPH using an alkaline delignification method with a NaOH solution. To determine the optimal delignification conditions, the effects of NaOH concentration, L/S ratio, delignification time, and temperature were evaluated based on the yield, purity, and characteristics of the resulting cellulose.

2. Experimental

2.1. Materials

This investigation employed distilled (DI) water, NaOH 98 % purity and H_2SO_4 99.9 % purity from Sigma-Aldrich. Meanwhile, fresh cocoa pods husk (CPH) as the raw material was collected from a cocoa plantation enterprise in Semarang, Central Java, Indonesia.

2.2. Methods

2.2.1. Preparation of CPH

The CPH was cut into pieces using a sharp knife and further reduced in size using scissors to pieces measuring 1×1 cm^2. The CPH samples were subjected to drying in an electric oven for 8 hours at 120 °C to ascertain their moisture content and ensure prolonged storage prior to use. The initial contents of cellulose, hemicellulose, and lignin in the CPH were assessed using the Chesson-Datta method.[26, 28]

2.2.3. Delignification of CPH

The delignification process began by placing 25 grams of chopped CPH into a 2.5 % (w/v) NaOH solution in a beaker, using a L/S ratio of 10:1. The mixture was then heated and stirred at 40 °C for 10 minutes. After the reaction, the CPH was separated from the solution by filtration using Whatman No. 1001-185 filter paper. The solid residue was subsequently dried in an electric oven at 120 °C for 60 minutes prior to further analysis. Delignification experiments were conducted at various temperatures (50, 60, 70, and 80 °C), times (20, 30, 40, 50, 60, and 70 minutes), L/S ratio (6:1, 8:1, 12:1, and 14:1), and NaOH concentrations (5, 7.5, 10, and 12.5 % w/v). All experiments were performed in triplicate, and the reported data represent average values.

2.2.3. Analysis of cellulose, hemicellulose, and lignin

The Chesson-Datta method was employed to analyze the cellulose, hemicellulose, and lignin content in CPH.[26, 28] Briefly, one gram of CPH pulp from the CPH delignification process (weight A) was refluxed with 150 mL of DI water at 100 °C for 2 hours to dissolve the oligosaccharides and pectin. Then, the CPH pulp was filtered and rinsed with DI water until the filtrate achieved a neutral pH. The residue was dehydrated in an oven at 105 °C until a constant weight (weight B). The residue was subsequently refluxed with 150 mL of 0.5 M H_2SO_4 solution for 2 hours at 100 °C. Thereafter, the residue was filtered and washed with DI water until the filtrate reached a neutral pH, followed by drying in an oven at 105 °C to achieve a constant weight (weight C). A total of 10 mL of 72 % v/v H_2SO_4 solution was added to the residue, which was allowed to soak at the atmospheric temperature for 4 hours. Subsequently, the mixture was diluted to obtain 0.5 N H_2SO_4, followed by refluxing at 100 °C for 2 hours. Finally, the mixture was filtered to obtain solid residue, which was then rinsed with DI water until the filtrate reached a neutral pH. The resulting residue was subsequently dried in an oven at 105 °C until the attainment of constant weight (weight D). The residue and ashless filter paper were incinerated in a furnace at 600 °C for 5 hours. Subsequently, the mass of residual ash was measured and documented (weight E). The quantities of cellulose, hemicellulose, and lignin were determined using the following equations:

$$\text{Cellulose content (\%) or } C_C = \frac{(weight\ C - weight\ D)}{weight\ A} \times 100\ \% \tag{1}$$

$$\text{Hemicellulose content (\%) or } C_H = \frac{(weight\ B - weight\ C)}{weight\ A} \times 100\ \% \tag{2}$$

$$\text{Lignin content (\%) or } C_L = \frac{(weight\ D - weight\ E)}{weight\ A} \times 100\ \% \tag{3}$$

$$\text{Cellulose yield (\%) of } C_Y = \frac{(weight\ D - C_C)}{25} \times 100\ \% \tag{4}$$

2.2.4. Characterization of cellulose

The functional groups present in CPH and the cellulose obtained through alkaline delignification were identified using Fourier-transform infrared spectroscopy (FTIR). Spectra were recorded over a wavenumber range of 500–4000 cm^{-1}.[5] Meanwhile, the scanning electron microscopy (SEM) was utilized to analyze the surface morphology of both the raw CPH and the delignified cellulose samples, using a JEOL instrument (JEOL Ltd., Tokyo, Japan) operated at an accelerating voltage of 10 kV under high vacuum conditions, as previously described by Thakur and Nanda.[29] Prior to SEM analysis, the samples were mounted onto a specimen holder with the assistance of double-sided carbon tape and subsequently sputtered with platinum powder employing an ion sputter coater. Thereupon, they were coated with an ultra-thin film of gold at ambient temperature to achieve the required conductivity and prevent charging.

3. Results and Discussion

The raw CPH used in this study contained 30.16 % cellulose, 28.37 % hemicellulose, and 18.705 lignin. The alkaline delignification process was successfully carried out and proved to be an efficient method to obtain cellulose from CPH.

3.1. Influence of NaOH concentration

Fig. 1 displays the effect of NaOH concentration in the delignification solution on the cellulose, hemicellulose, and lignin content of the extracted cellulose. As expected, an increase in NaOH concentration from 2.5 to 5.0 % w/v leads to an increase in the cellulose content. Conversely, both the hemicellulose and lignin content diminish as the concentration of NaOH increases. As a consequence, the cellulose yield was also significantly raised from 32.51 % to 38.56 %. The results are plausible because a strong alkali, NaOH solution with a higher concentration, will possess a greater capacity to break the structural linkage between the lignin, hemicellulose, and cellulose. As a potent alkali, NaOH promotes the cleavage of ester and ether connections between lignin and carbohydrates, especially the β-O-4 linkages, which are predominant in lignin.[30] Moreover, NaOH interferes with the glycosidic bond and crystalline structure of the hemicellulose matrix, enhancing its solubility and facilitating its removal.[31] This dual action results in the liberation and purification of cellulose.

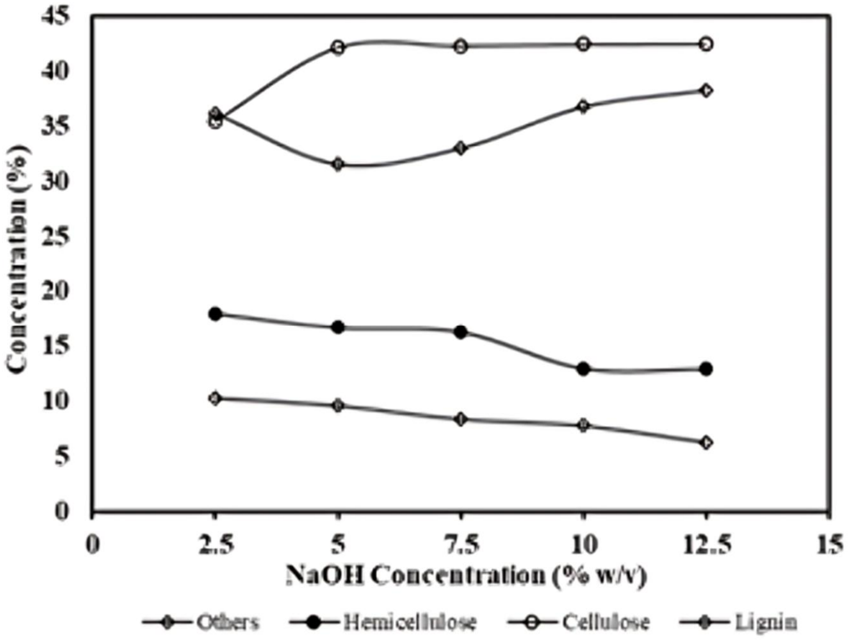

Fig. 1. Effect of NaOH concentration on the profile of cellulose, hemicellulose, and lignin

Nevertheless, when the NaOH concentration exceeded 5.0 % (w/v), the cellulose content of the extract did not exhibit a substantial rise (38.56 to 38.89 %). Predictably, both the hemicellulose and lignin content of the extract continued to decline with nearly the same pattern. At a higher concentration, the NaOH solution has a stronger capability to separate the hemicellulose and lignin from the lignocellulosic structure and further dissolve them.[32] Specifically, it can depolymerize lignin complexes through nucleophilic attack on the aromatic ring structure, increasing phenolic hydroxyl and methoxyl groups.[33] As a result, the cellulose content in the extract gradually increased. However, the rate of cellulose enrichment may also decrease due to saturation of the delignification capacity or possible degradation of amorphous cellulose under more severe alkaline conditions.[34]

The findings indicate that the optimum NaOH concentration for the delignification of CPH is 5 % (w/v). The same finding was previously reported by Radzi *et al.*[35] who investigated fiber preparation from oil palm empty fruit bunch (OPEFB) using alkaline pre-treatment. Moreover, the identical optimal NaOH content was also reported for the delignification of mature coconut (*Cocos nucifera L.*) coir.[36] A somewhat elevated optimal NaOH concentration (5.50 % w/v) was documented for the delignification of *Acacia Mangium* wood.[23] Meanwhile, Sayakulu and Soloi[37] revealed that the optimum NaOH concentration for delignification of OPEFB is 4 % (w/v).

3.2. Influence of liquid-to-solid (L/S) ratio

The effect of L/S ratio on the cellulose, hemicellulose, and lignin content in the extract is depicted in Fig. 2. As seen in Fig. 2, different L/S ratios rendered remarkably dissimilar results on hemicellulose, cellulose, and lignin contents. Hydrodynamically, a higher L/S ratio value will contribute to a higher probability of the CPH experiencing extensive contact with the NaOH molecules in the delignification solution. As a consequence, a larger number of NaOH molecules in the system will undergo reaction with the lignocellulosic substances of the CPH that extensively break the aryl-ester, carbon-carbon, and alkyl-alkyl bonds.[38] As a result, the lignocellulosic bond will be released, which is identified by the generation of a blackish brown solution.

As a strong alkali, NaOH molecules are capable of breaking lignocellulosic bonds and further destroying lignin.[35] In addition, they can also cause the lignocellulosic biomass to swell, enhance the internal surface area under relatively mild conditions, and subsequently alter monosaccharides and end groups of polysaccharides (1,4 glycosidic and hemicellulose bonds) into numerous carboxylic acids by breaking bonds from one end to the other end. Meanwhile, hemicellulose is more susceptible to bases than both lignin and cellulose.[36, 39] The hydrophilic nature of hemicellulose causes this fibrous material to undergo intensive interactions with water. In addition, its molecule possesses short and branched bonds that facilitate easy solvent penetration into it.[40] These unique characteristics promote the polysaccharide bond breakage, which leads to the easy solubilization of hemicellulose in water or hydrolysis. Meanwhile, the cellulose is made of β-D-glucopyranose units with (1–4) glycosidic interconnections. Therefore, there exists a negative correlation between the increase in L/S ratio with both hemicellulose and lignin contents.

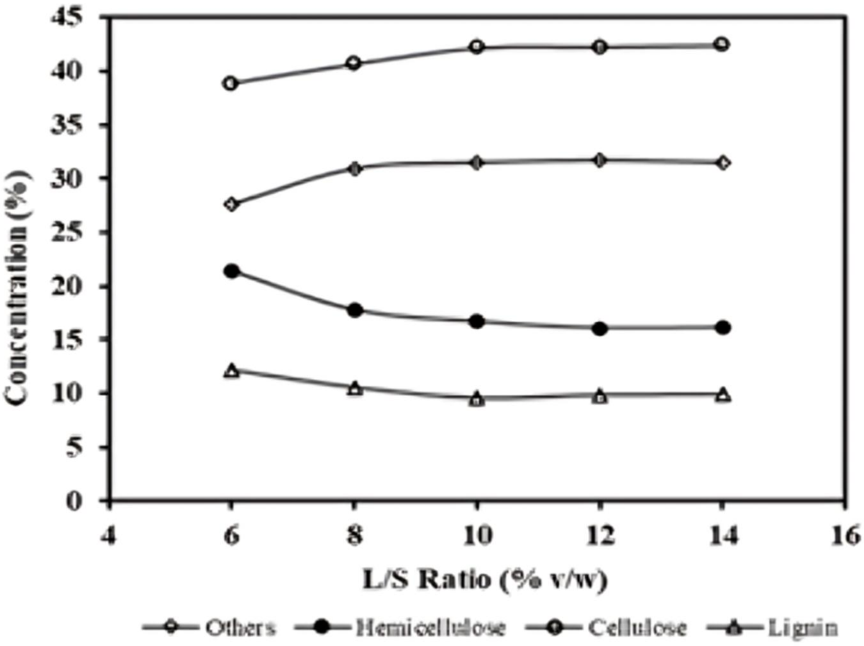

Fig. 2. Effect of liquid to solid ratio (L/S) on the profile of cellulose, hemicellulose, and lignin

On the other hand, cellulose molecules are linear and possess numerous hydrogen bonds, so their aqueous solubility is very low.[41] After the removal of hemicellulose and lignin from the extract, the cellulose concentration markedly rises from 38.85 to 42.17 % as the L/S ratio escalates from 6 to 10. Nonetheless, the cellulose concentration did not considerably increase when the L/S ratio exceeded 10. For this reason, the L/S ratio of 10 was determined as the optimum L/S ratio. Under this condition, the lignin removal was approximately 53 %.

The identical optimal L/S ratio was observed for the delignification of *Achnatherum splendens*, Brewer's wasted grain, and *Acacia mangium* wood.[42, 43] Whereas an optimum L/S ratio of 19.2 was reported for delignification of Sengon (*Albizia chinensis*) wood sawdust.[24] These differences can be linked with the botanical source of the lignocellulosic materials that vary in the fiber structure, the type of cellulose, and the surface composition in terms of cellulose and lignin.

3.3. Influence of delignification time

The effect of time on the alkaline delignification of CPH was examined using a 5 % (w/v) NaOH solution at a L/S ratio of 10:1 (v/w). The delignification temperature was maintained at 50 °C, while the time varied from 10 to 70 minutes. The research findings are depicted in Fig. 3.

As seen in Fig. 3, an increase in delignification time causes hemicellulose and lignin content to decrease in a nearly similar pattern. The reduction in both components was most pronounced during the first 30 minutes. However, beyond 30 minutes, extending the delignification time up to 70 minutes resulted in only a gradual decline in hemicellulose and lignin content. As expected, the reduction in hemicellulose and lignin content led to the rise of cellulose content in the extract. Surprisingly, the cellulose content slightly declined as the delignification time was extended to 70 minutes. Alkaline delignification conducted at temperatures ranging from 20 to 50 °C for an amount of time causes a swelling of the intracrystalline structure in the alkaline medium, transforming crystalline cellulose into Na-Cellulose and disrupting the hydrogen bonds inside the cellulose structure.[44] In fact, alkali cellulose possesses a higher reactivity compared to cellulose, and hence assists the NaOH molecules to penetrate more easily into the cellulose. This will sequentially facilitate reaction with the hydroxyl groups and cause cellulose loss. Based on Fig. 3, the optimal delignification time for CPH was 60 minutes, which successfully removed approximately 54.63 % of the lignin and produced a cellulose extract with a yield of 41.52 % and a cellulose content of 45.36 %. This optimal delignification time exists between the alkaline delignification times for Sengon *(Albizia chinensis)* wood sawdust (30 minutes) and *Acacia mangium* (180 minutes), representing softwood and hardwood, respectively.[23,24]

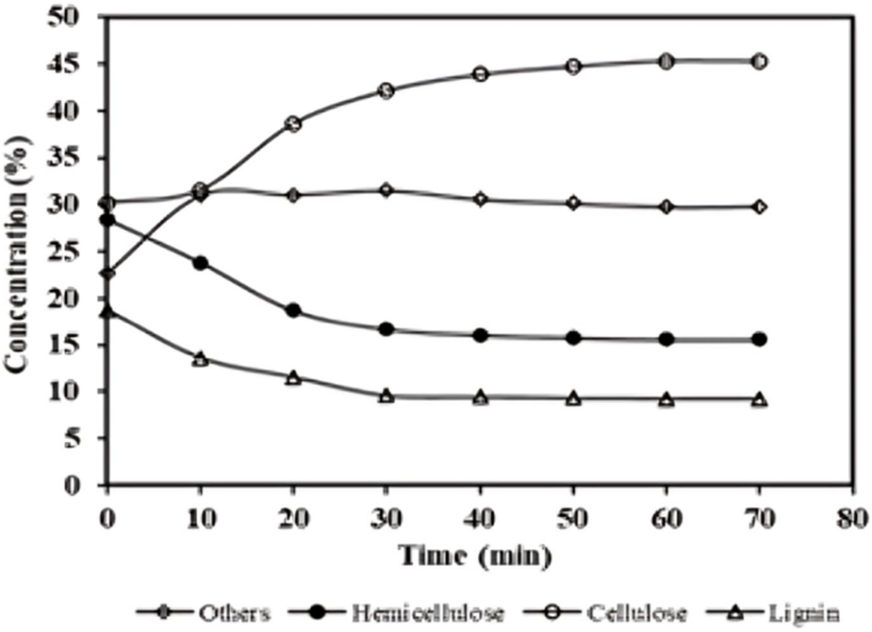

Fig. 3. Effect of delignification time on cellulose, hemicellulose, and lignin concentrations

3.4. Influence of delignification temperature

Several delignification experiments were carried out to see how the temperature affected the amounts of cellulose, hemicellulose, and lignin. A 5 % (w/v) NaOH solution and a 10:1 (v/w) L/S ratio were used to delignify CPH for an hour at temperatures ranging from 40 to 80 ºC. Fig. 4 displays the changes in cellulose, hemicellulose, and lignin compositions with delignification temperatures.

The hemicellulose and lignin content in the extracts after alkaline delignification declined from 17.51 % to 15.59 % and 9.70 % to 9.27 %, respectively, as the delignification temperature rose from 40 to 60 ºC. On the contrary, the cellulose content improved from 41.53 to 45.36 %. However, as the delignification temperature further rose from 60 to 80 ºC, the hemicellulose and lignin content in the extracts obtained continued to decline to about 15.45 and 9.21 %, respectively. Meanwhile, the cellulose content only slightly declined to 45.32 %, which indicates no significant influence of temperature when the delignification temperatures are beyond 60 ºC. Thus, 60 ºC was chosen as the best temperature for alkaline delignification of CPH using NaOH solution. This best temperature lies between the optimal temperatures for alkaline delignification of Sengon (*Albizia chinensis*) wood sawdust (50 ºC) and *Acacia Mangium* (70 ºC), which represent the softwood and hardwood.[23, 24]

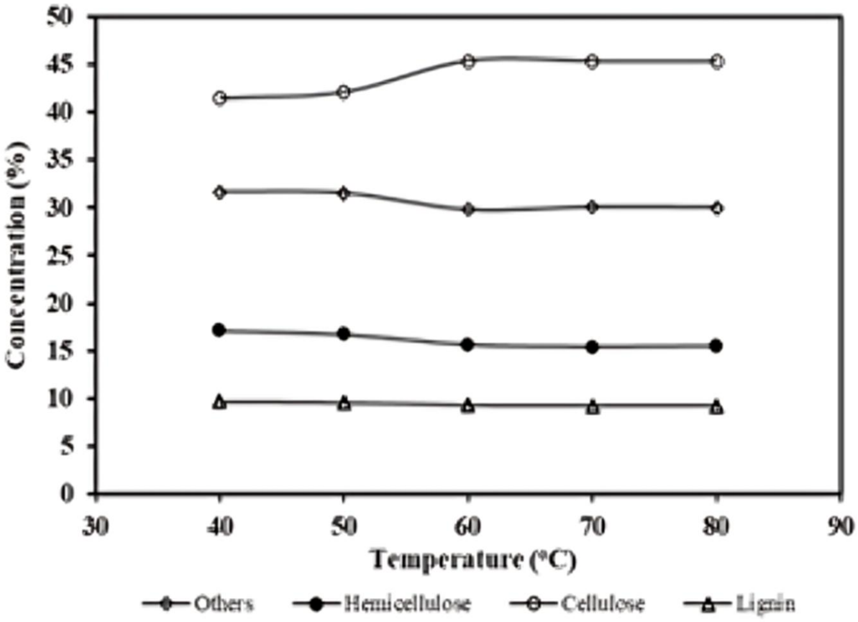

Fig. 4. Effect of delignification temperature on the profile of cellulose, hemicellulose, and lignin

The results of all the tests show that the best conditions for using NaOH solution to delignify CPH are a 5 % (w/v) NaOH concentration, a 10:1 (v/w) L/S ratio, a temperature of 60 ºC, and a delignification time of 60 minutes. Under this optimum operating condition, the cellulose yield obtained was approximately 41.52 % with a cellulose content of 45.36 %, and about 54.63 % lignin was successfully removed.

3.5. Morphology of CPH cellulose extract

Fig. 5 shows the results of morphological imaging of the cocoa pod surface before and after the delignification process using SEM with a magnification of 10,000×. As seen in Fig. 5 (A), the fibers in undelignified CPH are almost completely covered by rough and thick layers of white monosaccharides and polysaccharides (most probably pectin) and dark lignin that binds hemicellulose and cellulose to preserve the lignocellulosic content in this fibrous material. The SEM image of the raw CPH also demonstrates the lamella-shaped particles on its surface. Meanwhile, Fig. 5 (B) shows that the surface of the delignified CPH fibers has more cavities and numerous white-color layers of residual monosaccharides and polysaccharides that form small stalagmite-like structures. This indicates that most of the monosaccharides and pectin have been successfully removed from the CPH. As seen in Fig. 5 (B), the CPH also designated a high content of lignocellulosic material with linear fibrillar orderings that strengthen the resulting cellulose extract. The difference in microstructure on the CPH surface shown in Fig. 5 (A) and (B) proves that the caustic soda solution used in the delignification process successfully broke the lignin bonds in the lignocellulose structure and dissolved the lipids and the hot water-soluble carbohydrates and proteins, leaving pores in the CPH cell wall, reducing the lignin and hemicellulose content and subsequently increasing the cellulose content.[45]

3.6. Functional group CPH cellulose extract

FTIR spectroscopy was employed to identify alterations in the chemical structure of alkali delignified and raw CPH as shown in Fig. 6. A prominent absorption band is detected at around 3398 cm^{-1}, attributed to -OH stretching, characteristic of the α-cellulose structure.

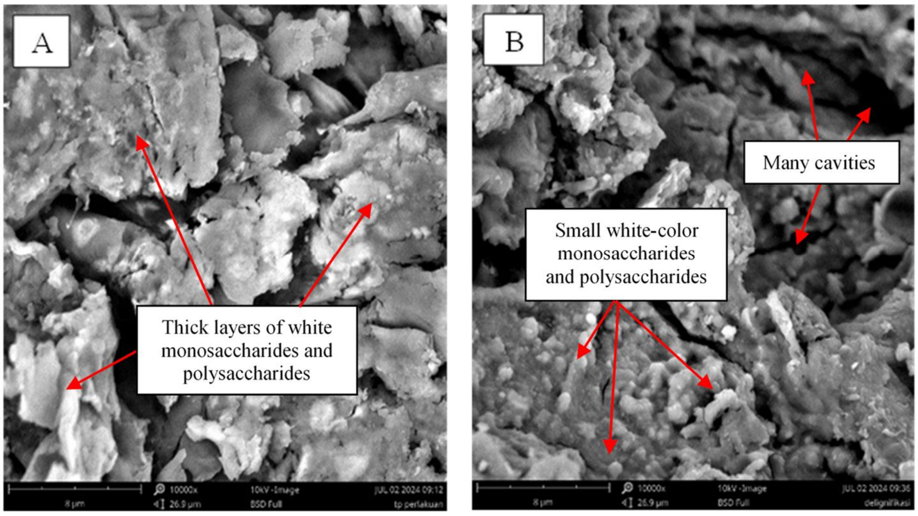

Fig. 5. Morphology of raw CPH (A) and CPH cellulose extract (B)

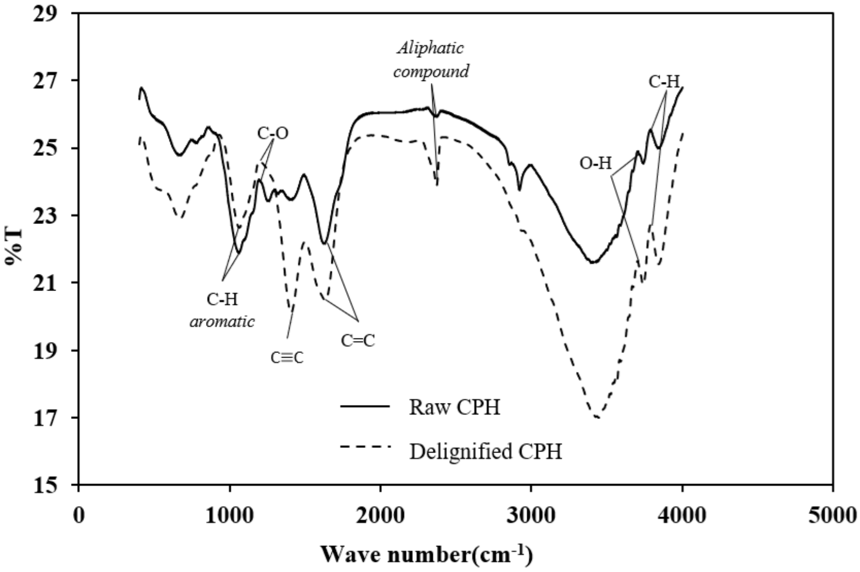

Fig. 6. FTIR spectra of raw and delignified CPH

The alkaline treatment caused reduced intensities of the bands at 1508 and 1270 cm^{-1} (associated with lignin) and the bands at 1730 and 1240 cm^{-1} (attributed to the acetyl functional groups), as illustrated in Fig. 6. The peak at 1740 cm^{-1} is assigned to the carbonyl ester component of hemicellulose. The molecular structure of hemicellulose includes both ester and acid functionalities. The diminished band intensities resulted from heating in the NaOH solution, leading to the cleavage of the β-O-4 linkage, the main bonds in lignin forming β-β′, β-5′, and β-1′ bonds at moderate heat treatment temperatures (130–170 °C),[46] and the elimination of acetyl groups associated with CPH due to delignification carried out under alkaline conditions.[47]

The efficiency of alkaline delignification of CPH employing NaOH solution to extract cellulose was confirmed by FTIR analysis, which indicated a significant reduction in the transmittance of the C=C aromatic group of lignin at wave numbers between 1600 and 1590 cm^{-1} following alkali treatment. The -OH group of cellulose and the -CH methylene group exhibited peaks at approximately 3398 and 2919 cm^{-1}, respectively. The stretching and deformation vibrations of aliphatic C–H bonds (–CH–, $–CH_2–$, $–CH_3$) at 2919 cm^{-1} are indicative of cellulose [48]. The presence of the cellulose was further confirmed by the absorption band at 1423 cm^{-1} (area integrated between 1405 and 1480 cm^{-1}), mostly attributed to the scissoring vibrations of the methylene group and the in-plane bending vibrations of the OH group.[49]

4. Conclusions

A delignification process has been successfully conducted using a NaOH solution to extract high-purity cellulose from CPH by removing the lignin. The effects of NaOH solution concentration, L/S ratio, delignification time, and temperature were investigated to identify the optimal delignification conditions based on cellulose yield, content, and characteristics. The optimum conditions for CPH delignification were determined to be a 5 % (w/v) NaOH solution, a 10 (v/w) L/S ratio, a delignification temperature of 60°C, and a delignification time of 60 minutes. Under these optimal conditions, the cellulose yield was approximately 41.52 %, with a cellulose content of about 45.36 % and a lignin removal of approximately 54.63 %. Morphological and functional group analysis collectively confirmed the successful alkaline delignification of CPH, demonstrating the efficient removal of pectin, lignin, and hemicellulose, which resulted in a more porous structure and an increased cellulose content, as evidenced by the characteristic absorption bands of cellulose. The cellulose that is made can then be further processed and used in many non-food and food-related ways, such as to make emulsifiers, stabilizers, coatings, films, gels, adhesives, thickening structures, veterinary foods, cosmetic and pharmaceutical excipients, pulp and paper, fibers, and fabrics.

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ЛУЖНА ДЕЛІГНІФІКАЦІЯ ЛУШПИНИ КАКАО (THEOBROMA CACAO L.) ДЛЯ ЕКСТРАКЦІЇ ЦЕЛЮЛОЗНИХ ВОЛОКОН

Анотація. *Лушпиння какао-бобів – один із побічних продуктів переробки какао (Theobroma сасао L.) містить близько 26,4 % лігнінзв'язаної целюлози. Тому для отримання целюлози високої чистоти необхідна делігніфікація, яка полягає у видаленні лігніну. Мета цього дослідження – вилучення целюлози із лушпиння какао-бобів за допомогою лужної делігніфікації із використанням розчину каустичної соди. Окрім концентрації лужного розчину та співвідношення рідини та твердої речовини, в цьому дослідженні було вивчено час і температуру лужної делігніфікації з метою визначення найкращих умов делігніфікації на основі виходу, чистоти та характеристик отриманої целюлози. Одержана целюлоза придатна для численних промислових застосувань.*

Ключові слова: *каустична сода, целюлоза, какао, делігніфікація, екстракція.*