Semiconductor based on Oil Palm Empty Fruit Bunch Alkali Cellulose

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ARTICLE INFO	ABSTRAK
Kata kunci: TKKS; alfa-selulosa; alkali selulosa; semikonduktor	Tandan kosong kelapa sawit (TKKS) merupakan sumber biomassa yang berlimpah di Indonesia. Pengolahannya menjadi alfa-selulosa dan alkali selulosa menawarkan peluang ekonomi yang menjanjikan dan pengembangan sensor yang berkelanjutan. Studi ini berfokus pada ekstraksi alfa-selulosa dari TKKS menggunakan metode prehidrolisis kraft dan konversi selanjutnya menjadi alkali selulosa menggunakan larutan NaOH. Variasi 15, 20, dan 25% konsentrasi NaOH selama pulping menghasilkan kualitas pulp yang berbeda, seperti yang ditunjukkan oleh kandungan alfa-selulosa. Alkali selulosa yang dihasilkan menunjukkan sifat semikonduktor dengan pita celah energi 3,6 eV, menunjukkan potensi untuk aplikasi sensor cahaya atau fotodioda. Selain itu, dihitung pula pita celah energi pada komposit alkali selulosa - polivinil alcohol.
Keyword: *OPEFB;* *Alpha-cellulose;* *Alkali cellulose;* *Semiconductor*	***ABSTRACT*** *Oil palm empty fruit bunch (OPEFB) is a readily available biomass resource in Indonesia. Its processing into alpha-cellulose and alkali cellulose offers promising economic opportunities and sustainable sensor development. This study focuses on extracting alpha-cellulose from OPEFB using kraft hydrolysis method and converting it into alkali cellulose using NaOH solution. The 15, 20, and 25% variation of NaOH content during pulping resulting in different pulp quality indicated by alpha-cellulose content. The resulting alkali cellulose exhibits semiconductor properties with an energy gap of 3.6 eV, demonstrating its potential for light sensor or photodiode applications. Additionally, the optical energy gap of alkali cellulose-polyvinyl alcohol composites is investigated.*

1. INTRODUCTION

Indonesia's Oil palm is abundant. The problem of palm biomass from CPO production, especially the Oil Palm Empty Fruit Bunch (OPEFB), is something that requires a solution in product diversification [1]. Processing EFB into alpha-cellulose and alkali cellulose is a high economic added value that can stimulate the creation of new products and markets in a sustainable national sensor industry.

Cellulose is the main component of EFB which is divided into 57.2% spikelets, 21.2% stalks, 9.1% petals, 5.1% spines and 5% other components [2]. A total of 41.96-60.6% of the fibers that make up EFB are alpha-cellulose. Cellulose in its basic form in nature cannot be directly applied as a sensor. Some common forms of cellulose used as sensors are cellulose nanofiber, cellulose nanocrystals, and bacterial cellulose [3]. Cellulose is also often used as a matrix material for nanoparticle composites [4]–[10].

The unique characteristic of cellulose used as a sensor is the monoclinic crystalline phase where a # b # c with $\gamma \neq 90°$, $\alpha = \beta = 90°$. Meanwhile cellulose can be converted into alkali cellulose with phase 1 becoming phase 2. Various types of quantities can be measured by cellulose-based sensors. For example gas detection sensors [11], humidity[3], [7], and ultraviolet sensors [12]. Using calorimetric and electrochemical methods [13], cellulose can detect various elements, ions, and even complex compounds [12]. The biodegradable and biocompatible characteristics of cellulose are widely used as optical biosensors [14].

The mechanical properties of cellulose are used to measure physical quantities such as strain and pressure. Strain sensors, which are electro-mechanical sensors, are widely applied in building

infrastructure, mechanical parts of moving systems such as vehicles or industrial machines, as well as for activity monitoring [15] and human body health through wearable sensors [16], [17]. In its application as a strain sensor, cellulose can be used in pure form, composite, hydrogel [18], nanocellulose, and even as a framework for carbon nanoparticle material [6], [19].

Based on chemical composition, EFB contains alpha-cellulose which can be optimized by degradation of lignin, hemicellulose and extractive substances through kraft pulping [20]. Alkali cellulose produced from EFB has high potential to be used as a sensor material after undergoing physical-chemical treatment. NaOH is one of the optimum chemicals for alkalization processes [21], [22].

In this research, the alpha-cellulose is extracted from oil palm EFB. Alpha cellulose can be converted into alkali cellulose using chemical thermodynamic methods. In addition, the resulting alkali-cellulose can be fabricated into a light sensor or photodiode by utilizing its characteristics as a semiconductor material.

2. METHOD

2.1 Raw Material Treatment

Whole EFB biomass with a mass of 50 kg was separated from the palm fruit and then cleaned. Fibrillated EFB was obtained by soaking water (water retting) in a static fluid for 6 days until it broke down into long fibers measuring 20 - 40 cm. The long fiber then was broken down using a crusher/milling machine into short fiber measuring 5-10 cm. The fiber was then washed with dynamic fluid to remove impurities until it was clean and odorless. Next, the fiber was air-dried to obtain a uniform moisture. Raw material analysis was carried out based on TAPPI standards to determine the content of alpha-cellulose, holocellulose, hemicellulose and lignin in raw EFB.

To calculate the percentage of various parameter based on TAPPI standards, equations (1) to (4) is used:

$$\text{Extractives, \%} = [(\text{weight of extract} - \text{weight of black residue})/\text{weight of pulp}] \times 100 \qquad (1)$$

$$\text{Holocellulose, \%} = (\text{weight of holocellulose}/\text{weight of wood}) * 100\% \qquad (2)$$

$$\alpha\text{-cellulose, \%} = (\text{weight of alpha-cellulose} / \text{weight of pulp}) * 100\% \qquad (3)$$

$$\text{Acid-insoluble lignin, \%} = (\text{weight of lignin} / \text{weight of wood}) * 100\% \qquad (4)$$

2.2 Dissolving Pulp Production

The prehydrolysis kraft [23], [24] was carried out in three stages: prehydrolysis, pulping, and bleaching. In the pre-hydrolysis stage, a rotary digester was used to mix 30 grams EFB fiber with 1 N H_2SO_4 solution in a ratio of 1 part fiber : 5 part solution (w/v). Heating was carried out gradually over 60 minutes until it reached a temperature of 165°C and held for 90 minutes. The results were separated and washed with water until neutral.

During the pulping, EFB was treated with 15-25% NaOH and 30% Na_2S in a rotary digester. The EFB in the NaOH and Na_2S solution underwent gradual heating for 90 minutes to reach a temperature of 160°C and held in the state for 120 minutes. The product of the pulping process were dried and filtered. The pulp that passes through the filter from each process will then go through a bleaching process.

Bleaching was carried out by alkali H_2O_2 extraction at a temperature of 70°C for 60 minutes. Dissolving pulp was tested following TAPPI standards to determine the content of alpha-cellulose, lignin, holocellulose and extractives after physical and chemical treatment. The content of alpha-cellulose determined the best quality of pulp.

2.3 Alkalization   The alkalization process began with kiln-dried dissolving pulp being placed in a 18% NaOH solution with a ratio of 1 part pulp : 14 part NaOH solution (w/v). The mixture was continuously stirred at a temperature of 52 °C for 8 minutes. This alkalization process aimed to form an alkali cellulose slurry, breaking hydrogen bonds between cellulose molecules, changing the crystalline structure of cellulose 1, also separating and dissolving hemicellulose in NaOH. The resulting cellulose alkali was filtered using a 200-mesh gauze filter and continued with pressing at room temperature with a mass ratio of 1:3. The purpose of filtering and squeezing alkali cellulose is to remove excess NaOH,

removing short chain cellulose and hemicellulose which dissolves into filtrate. The alkali cellulose that was formed then shredded using a shredding machine at room temperature to obtain small and homogeneous alkali cellulose. The shredded alkali cellulose was then put into the aging drum and aged for 5 hours at a temperature of 44 °C. The aging process aimed to change cellulose phase 1 to 2, which optical and electrical properties was tested.

3. RESULT AND DISCUSSION

The processing of oil palm empty fruit bunches (OPEFB) into alkali cellulose is carried out physically and chemically. The pre-treatment steps, namely mechanical cleaning and peeling, water retting, and drying, are carried out until dry EFB fibers are formed with a water content < 20%. The oil palm EFB used in the research was the whole EFB, without separating the stalk and spikelet components. These pre-treatment steps still rely heavily on human power and natural drying using sunlight, as shown in Figure 1. There are several obstacles in the pre-treatment stage. Soaking the large EFB fibers takes a long time due to their size, further complicated by cloudy and unpredictable weather that extends the drying time to more than 6 days.

 (a) (b)

Figure 1. EFB pre-treatment process: (a) Palm fresh fruit bunches (FFB); (b) the process of separating the fruit from the FFB before soaking (water retting)

Table 1. Test results for EFB raw materials based on TAPPI standards

Parameter	Result (%)	Method
Extractive	7,60	SNI 8401 / TAPPI T 204 om-88
Holocellulose	71,59	TAPPI T 9m-54
Alpha-cellulose	46,62	TAPPI T 203-OS-61
Lignin	23,17	SNI 8429 / TAPPI T 222 om-88

3.1 Analysis of Raw Materials

Dried short fibers of EFB raw materials are tested for extractive, holocellulose, alpha-cellulose and lignin levels in accordance with TAPPI standards. The results are shown in Table 1. These results will be compared with the dissolving pulp to see the success of alpha-cellulose formation.

3.2 Analysis of Dissolving Pulp

The processing of EFB into dissolving pulp consists of prehydrolysis, pulping and bleaching stages, which comply to a kraft prehydrolysis method. The aim is to produce a high alpha-cellulose component from EFB and remove the lignin component. There were three variations of samples made, with an initial weight of TKKS each of 30 grams. Samples after pulping and bleaching are shown in Figure 2.

Figure 2. Dissolving pulp production: (a) sample after pulping process; (b) sample after the bleaching process

Table 2. Measured sample mass after each stage of dissolving pulp production

Stage	Sample	Init. mass (gr)	Final mass (gr)	Mass reduction (%)
prehydrolysis	A	30	28,58	4,73
	B	30	28,47	5,10
	C	30	28,34	5,53
pulping	A (15%)	24	16,34	31,92
	B (20%)	24	18,35	23,54
	C (25%)	24	13,22	44,92
bleaching	A	16,34	13,77	15,73
	B	18,35	15,65	14,71
	C	13,22	11,14	15,73

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Table 3. Test results for dissolving pulp based on TAPPI standards

Parameter	Result (%)			Method
	A	B	C	
Extractive	10	26	9	SNI 8401 / TAPPI T 204 om-88
Holocellulose	79	77	82	TAPPI T 9m-54
Alpha-cellulose	69	79	84	TAPPI T 203-OS-61
Lignin	5,63	5,14	5,69	SNI 8429 / TAPPI T 222 om-88

Table 2 shows the mass of EFB at each stage of dissolving pulp production. Samples A, B, and C used various NaOH treatments of 15, 20, and 25% respectively at the pulping stage, with the same treatment at the prehydrolysis and bleaching stages. The initial mass of the pulping stage was equalized at 24 grams as a control variable. The largest reduction occurs at the pulping stage.

After the bleaching stage, dissolving pulp is analyzed using TAPPI method, similar to the raw material. Table 3 shows the results. The result in Table 3 can be directly compared to the Table 1, to measure the success of the delignification and pulping process.

Extractives are undesirable contaminants during the production of dissolving pulp. Extractives can have a significant impact on the pulping process. They can interfere with the pulping chemicals, leading to incomplete delignification and poor pulp strength. Extractives can also deposit on equipment, causing problems with corrosion and fouling. The SNI standard used in this analysis is SNI 938:2017 with a value of 0.2%. Based on the test results, the extractive content of dissolving pulp samples did not meet SNI requirements, however, the lowest extractive content was sample C at 9%. The increasing value of extractives compared to raw material implies that there are some impurities contaminating the sample during the production of dissolving pulp or during storage.

Alpha-cellulose is one of the parameters representing the quality of a pulp. A high percentage of alpha-cellulose is necessary for the formation of microcrystalline cellulose[25]. As shown in Table 3, the dissolving pulp sample A, B, and C has 69, 79, and 84% composition of alpha-cellulose, respectively. Compared to the results from raw material, there has been a significant increase in alpha-cellulose percentage. This shows that the pulping process with a NaOH concentration from 15 to 25% effectively produces dissolving pulp, with 25% NaOH yielding the best quality pulp. However, alpha-cellulose in all samples does not meet Indonesia`s SNI standard for paper pulp, namely 94%.

The percentage of holocellulose slightly increased from 71,59% for raw material, to 79, 77, and 82% for dissolving pulp. Holocellulose itself is a water-insoluble carbohydrate fraction of wood materials that can be obtained from biomass by removing the lignin and extractives. Holocellulose is mostly comprised of hemicellulose (also identified as gamma-cellulose) and alpha-cellulose, although there may be some beta-cellulose in the composition. Theoretically, beta-cellulose is soluble in a 16.5% NaOH solution, and gamma-cellulose is soluble in a neutral solution[26]. This indicates these two types of cellulose should be removed in samples B and C, with 20 and 25% NaOH, proved by the equal percentage of holocellulose and alpha-cellulose in respective samples. That does not seem to be the case in this experiment.

The role of NaOH in the pulping stage also aims to remove the lignin component. This happens because, during pulping, the OH- ions are dissolved by the NaOH solution so that the bonds in the lignin structure are broken. Therefore, it can be said that NaOH plays a role in degrading and dissolving lignin and other impurities so that cellulose is released and can be separated from its bonds. The results of measuring lignin composition are also shown in Table 3. These results are still do not yet meet the established SNI standards, but lower than the 23,17% result of raw material.

3.3 Analysis of Alkali Cellulose

The production of alkali cellulose from alpha-cellulose is also known as the alkalization or mercerisation process. Cellulose I can be converted into cellulose II during this process. Alkalization is irreversible because of more stability of cellulose II compared to cellulose I [27]. In this process, a sample of dry dissolving pulp is soaked in a solution of 18% NaOH. The mixture is stirred at 52 °C for 8 minutes. The resulting alkali cellulose is then squeezed and blended to reduce the particle size. It is then fermented in an aging drum for 5 hours. The resulting material then oven-dried and tested for its optical and electrical properties.

The resulting material is a fine powder with very weak cohesion. The easily crumbled texture of alkali cellulose makes it difficult to press or print into thin plates. This changes the initial plan to install Tin contacts as electrodes. Several studies have been conducted to find materials that can help the fabrication of light sensors. Polyvinyl alcohol (PVA) was chosen as a binding agent in the hope that alkali cellulose can adhere to the surface of the interdigital circuit, while also lowering the energy gap of the composite.

The composite of PVA-Alkali cellulose is then characterized using spectrophotometry in the ultraviolet and visible light ranges to determine the band gap energy as optical properties. The calculation graph is shown in Figure 3. The results of UV-Vis characterization which obtained information on absorbance and transmittance at wavelengths of 200nm - 900nm were then converted into %R quantities. The band gap energy will be obtained using the Kubelka-Munk method by changing the %R quantity into a factor (F(R)) with the following equation:

$$F(R) = \frac{K}{S} = \frac{(1-R)^2}{2R} \tag{5}$$

The value of band gap energy is calculated from the linear relation between hv(eV) vs [F(R)hv]^2. Linear extrapolation is implemented in the Tauc plot[28] as shown in Figure 3. The result of these calculations is shown in Table 4.
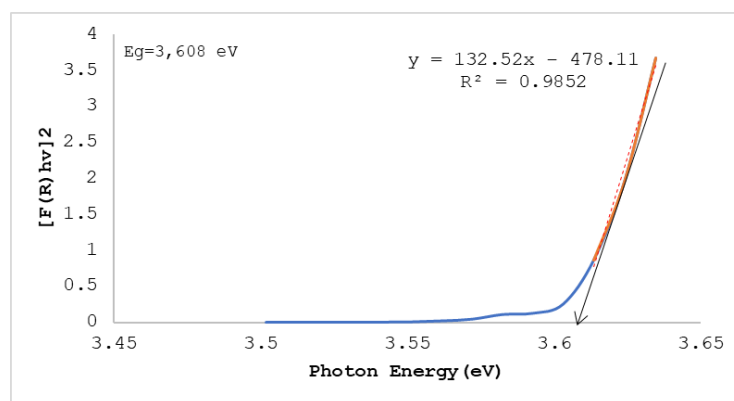
Figure 3. Band-gap energy calculation

Band gap energy is the amount of energy needed by electrons to move from the valence band to the conduction band. The smaller the valence band gap to the conduction band, the smaller the energy required by electrons so that it will be easier for electrons to move. Band gap energy determines whether a material is an insulator, semiconductor or conductor. The cellulose in its natural form behaves like an electrical insulator, while previous research shows that with certain treatment, the alkali-cellulose is a fast-ionic conductor with energy gap 0.49 eV and 0.68 eV at high temperature [29]. More examination on various cellulose structure [30] shows that the alpha-cellulose, a pure form of cellulose has the energy gap of 5,5 eV. The experiment result shows that alkali cellulose has a band gap energy of 3,60 eV. Combined with conducting polymer PVA, the band-gap energy decreases further into the semiconductor range with lowest energy gap is the sample with 5% PVA composite. This composite can be applied as part of photodiode sensor by deposition into interdigital circuit.

Although the energy gap is not as low as Silicon at 1,12 eV, there are possible application of alkali-cellulose semiconductor is in the soft electronics field. The Single Electron Transistor (SET) devices utilizes doped nanocellulose with 2,49 eV energy gap [10]. Wide Band Gap (WBG) materials are defined as materials possessing a band gap of 2.2 electron volts (eV) or higher, including diamond and the materials belonging to the IV–IV, III–V, and II–VI semiconductor families [19]. WBG electronics have proven to be a versatile technology for wearable and implantable devices, with applications in long-lasting electronics, energy harvesting, bioresorbable components, and optogenetic devices.

Table 4. Band-gap energy of Alkali cellulose – PVA composite

Sample		Band-gap energy (eV)
Alpha-cellulose [30]		5,5
Alkali Cellulose	0% PVA	3,608
	5% PVA	2,769
	10% PVA	2,773
	15% PVA	2,779

4. CONCLUSION

Alkali cellulose has been successfully extracted and processed from oil palm empty fruit bunch (OPEFB) through prehydrolysis kraft and alkalization method comprising a variety of NaOH concentration. The dissolving pulp shows increasing level of alpha-cellulose and decreasing level of lignin percentage, indicating successful delignification with best quality obtained by 25% NaOH pulping solution. Alkalization by 18% NaOH successfully creating alkali cellulose having semiconductor characteristic of 3,6 eV bandgap energy. Composite of alkali cellulose and PVA has been designed to be applied to sensor. However, further morphological examination should be carried out in the future to understand the effect of alkalization to the cellulose structure and how to best utilize alkali cellulose as sensors.

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