E-ISSN: 2829-7687
Journal of Fibers and Polymer Composites
Vol. 3 No. 2 (2024): 91-100

Optical and Electrical Properties of OPEFB Alkali Cellulose and PVA Composite

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Abstract. *The oil palm empty fruit bunches (OPEFB) are a byproduct of oil palm plantation and abundantly available in Indonesia. It is necessary to process the biomass waste into a more useful and higher value substances, such as semiconductor. This research aims to extract alpha-cellulose from OPEFB and process it into alkali cellulose – poly vinyl alcohol (PVA) composite for potential photodetector application. PVA acts as mechanical binding agent with 5%, 7.5%, and 10% concentration. Spectroscopy-based measurement is used to characterize the composite's optical properties, while an inductance-capacitance-resistance meter (LCR meter) is used to measure the electrical properties such as conductivity and capacitance. Using UV-Vis spectroscopy with wavelength range of 400-900 nm, it is observed that alkali cellulose with 5% PVA has the highest optical absorbance. Using Kubelka-Munk equation and Tauc plot, the energy gap of the composite was calculated, with the lowest energy gap is 2,769 eV at 5% PVA. Using LCR meter between frequency of 5 Hz to 5 MHz, one can measure conductivity (specific conductance) of a material, where highest obtained electrical conductivity is* $2{,}65\times10^{04}$ *S/cm, which satisfy typical value of semiconductor characteristic. Measurement of Impedance also shows that composite with 7.5% PVA has the highest impedance at lower frequencies, then decreases to almost zero at frequency higher than 5000 Hz. These results demonstrate the potential of alkali cellulose-PVA composite as semiconductor sensors. These findings suggest the potential for developing low-cost, sustainable electronic devices.*
Keywords: *Alkali cellulose; OPEFB; photodetector; PVA; semiconductor.*

Type of the Paper: Regular Article

1. Introduction

Indonesia possesses immense potential in palm oil production. However, one of the primary challenges in crude palm oil (CPO) production is the suboptimal management of palm oil biomass waste, particularly empty fruit bunches (EFB). EFB is a waste product that requires innovative solutions to enhance its economic value and reduce environmental impacts [1]. One potential solution to address this issue is through alkali cellulose processing of EFB. This process can yield high economic value and stimulate the creation of new sustainable industries.

Cellulose is the most important component in EFB with the potential to be utilized as a

https://doi.org/10.55043/jfpc.v3i2.211
Received September 27, 2024; Received in revised October 15, 2024; Accepted Month October 17, 2024; Published October 29, 2024
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biomaterial. EFB consists of 57.2% spikelet, 21.2% stalk, 9.1% sepals, 5.1% thorns, and 5% other components [2]. Cellulose is an abundant natural biopolymer that is renewable, biodegradable, and non-toxic. In nature, cellulose is not found in its pure form, but is always associated with other polysaccharides such as lignin and hemicellulose [3].

The chemical composition of EFB reveals the presence of alpha-cellulose, a component that can be enhanced through the removal of lignin, hemicellulose, and extractives by the pre-hydrolysis kraft pulping method. Approximately 41.96-60.6% of fibers in EFB consist of alpha-cellulose [4]. Sodium hydroxide (NaOH) is identified as an ideal chemical reagent for the alkalization process [5,6]. The alkali cellulose derived from EFB exhibits significant potential for application as a semiconductor material following physicochemical modifications.

Materials exhibiting electrical conductivity values between 10^{-8} and 10^{3} Siemens per centimeter are classified as semiconductors. In addition to electrical conductivity, resistivity serves as a critical parameter in differentiating between conductors, insulators, and semiconductors. Resistivity, a measure of a material's opposition to electric current flow, is inversely proportional to conductivity: higher resistivity corresponds to lower conductivity. Semiconductors typically possess resistivity values ranging from 10^{-3} to 10^{8} ohm-centimeters, signifying their capability to regulate electric current flow effectively [7]. Cellulose-based composite can be employed to measure a wide range of parameters [8–14]. Given its biodegradable and biocompatible properties, cellulose stands out as an ideal material for the development of optical biosensors [15].

In this research, alpha-cellulose was extracted from empty fruit bunches of oil palm and subsequently converted into alkali cellulose using a chemical thermodynamic method. The alkali cellulose then combined with PVA as mechanical binding agent to create a composite. The composite can be fabricated into a light sensor or photodiode by utilizing its characteristics as a semiconductor material.

2. Materials and Methods

2.1. Raw Material Preparation

The preparation of raw materials for alkali cellulose production begins with the cleaning of 50 kg of empty fruit bunch (EFB) biomass. The EFB undergoes a fibrillation process using the water retting method, which involves immersing it in static liquid for 6 days until it decomposes into long fibers measuring 20-40 cm. These long fibers are then broken down using a crusher or grinder into short fibers measuring 5-10 cm, which are subsequently washed with dynamic liquid to remove impurities until they are clean and odorless. After that, the fibers are air-dried to achieve a uniform moisture content. Subsequently, raw material analysis is conducted based on TAPPI standards to determine the content of alpha-cellulose, holocellulose, hemicellulose, and lignin in the raw EFB. This process ensures that the raw material is ready for the next steps in alkali cellulose production, including pre-hydrolysis, pulping, bleaching, and alkalization, all aimed at producing high-quality alkali cellulose for further applications.

2.2. Dissolving Pulp

In the prehydrolysis stage [16,17], a rotary digester was used to mix 30 grams of TKKS fiber with 1 N H_2SO_4 solution at a fiber-to-solution ratio of 1:5 (w/v). The mixture was then heated gradually for 60 minutes, with the temperature being increased to 165°C. This heating was maintained at this temperature for 90 minutes to break down the lignin and hemicellulose present, as well as reduce the content of extractives. After the heating process was completed, the resulting product was separated and washed with water until a neutral condition was reached, ensuring that any residual acid and other impurities were completely removed before proceeding to the pulping and bleaching stages.

During the pulping process, TKKS is treated with a 15-25% NaOH and 30% Na_2S solution in a rotary digester, where the TKKS raw material is mixed with the solution and heated gradually for 90 minutes until reaching a temperature of 160°C. This temperature is then maintained for 120 minutes to ensure that lignin and hemicellulose are completely dissolved, resulting in cellulose in the form of pulp. After that, the resulting pulp is dried and filtered to separate cellulose fibers from the remaining solution and impurities. It then undergoes a bleaching process with alkaline extraction using H_2O_2 at a temperature of 70°C for 60 minutes. This bleaching process aims to increase the purity of cellulose by removing residual lignin and coloring matter, resulting in a whiter pulp. The dissolved pulp is tested according to TAPPI standards to determine the content of alpha-cellulose, lignin, holocellulose, and extractives after physical and chemical treatment. The content of alpha-cellulose is the main indicator in determining the best pulp quality.

2.3. Alkalization

The alkalization process begins by introducing the dried dissolved pulp into an oven, followed by mixing it with an 18% NaOH solution at a pulp-to-NaOH solution ratio of 1:14 (w/v). The mixture is continuously stirred at a temperature of 52°C for 8 minutes to ensure efficient alkalization reaction. The main objective of the alkalization process is to form alkali cellulose pulp, break hydrogen bonds between cellulose molecules, change the crystalline structure of cellulose into a more reactive form, and to dissolve hemicellulose in NaOH.

After the alkalization process, the resulting alkali cellulose is filtered using a 200-mesh screen to remove unwanted particles and then pressed at room temperature with a mass ratio of 1:3. This filtration and pressing aims to remove excess NaOH and residual contaminants, as well as to separate short-chain cellulose and dissolved hemicellulose, which are produced as filtrate.

The filtered alkali cellulose is then shredded using a shredding machine at room temperature until a small and homogeneous size is obtained. The shredded alkali cellulose is then placed in an aging drum and left for 5 hours at a temperature of 44°C. followed by testing of optical and electrical properties to ensure the quality and performance of the material in its application.

2.4. Composite fabrication and characterization

The alkali cellulose material obtained from alkalization method then combined with PVA to create a composite. PVA has a good mechanical property and chemically inert, providing good support for the alkali cellulose, which is a fine powder with very soft particles and has low cohesion. A binding agent needs to be added so the alkali cellulose can be deposited into an interdigital electrode and tested as semiconductor. Three variations of PVA (5%, 7.5%, and 10% concentration) are added to the alkali cellulose to create a composite. The PVA was dissolved in aquadest (1:1 w/v) and manually mixed with the alkali cellulose powder. The optical characterization used spectroscopy method, observing the absorbance of the composite material at ultraviolet and visible light spectrum. The composite then deposited into the surface of the interdigital electrode and left to dry. The interdigital electrode then given a short cable for positive and negative electrode, and is ready for LCR meter testing.

3. Results and Discussion

Oil palm empty fruit bunches (OPEFB) can be transformed into alkali cellulose through a series of physical and chemical processes. Prior to chemical treatment, the OPEFB undergoes mechanical cleaning, peeling, water retting, and drying to produce dry EFB fibers with a moisture content below 20%. For this study, whole OPEFB, including both the stalk and spikelet components, was used. Using the standard pre-hydrolysis Kraft pulping, alpha-cellulose was successfully extracted, and prepared for alkalization/mercerization process.

The alkali cellulose material formed a fine powder, with the particle very soft and has low cohesion. A binding agent needs to be added so the alkali cellulose can be deposited into an interdigital electrode and tested as semiconductor. Three variations of PVA (5%, 7.5%, and 10% concentration) are added to the alkali cellulose to create a composite. It has come to attention that using 5%-10% PVA, a gel-like composite is obtained and it is the best form to be deposited into the surface of interdigital electrode.

Spectroscopy method is used to measure the absorbance spectrum of the alkali cellulose composites. The result is depicted in Fig. 1. The absorbance is observed at the wavelength of 400 nm to 900 nm, both visible and near-infrared range. The absorbance peak is between 400 nm and 425 nm with 5% alkali cellulose composite has the highest absorbance value, and the pure alkali cellulose has the lowest. The increasing absorbance value probably caused by the optimized structure of alkali cellulose. While PVA itself is not a conducting polymer, it is a good binding agent, creating more homogenous alkali cellulose distribution. Therefore, creating a better absorbance response. Meanwhile, in the longer wavelength, the absorbance spectrum becomes less wavelength-dependent. The absorbance values of the composites are generally lower than the pure alkali cellulose in those longer wavelengths.

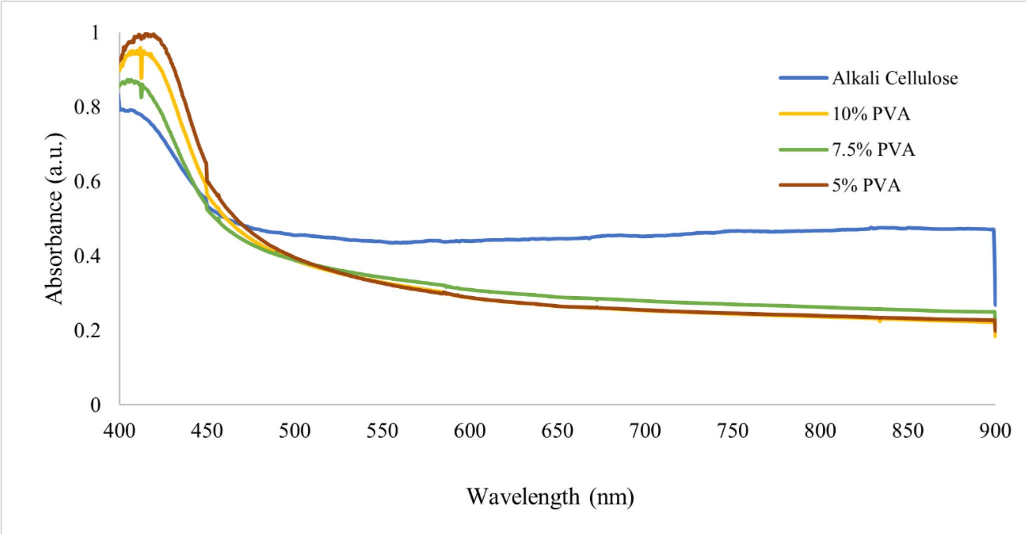

Fig. 1. Absorbance spectrum of alkali cellulose and its composites

The band gap energy of a material can be determined using the Kubelka-Munk method. This method involves converting the reflectance (%R) of the material into a factor (F(R)) using the following equation:

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

Where

$F(R)$ Kubelka-Munk Function

K absorption coefficient

S scattering coefficient

R reflectance

The band gap energy is then calculated by plotting the relationship between the energy (hv) and the square of the factor (F(R)hv). The linear part of this plot, known as the Tauc plot [18], is extrapolated to determine the band gap energy. The band gap energy of the 5%, 7.5%, and 10% composites are 2.769 eV, 2.773 eV, and 2.779 eV respectively. The energy gap of these composites are in the semiconductor range. Typical value of silicon energy gap is 1.2 eV, and Germanium 0,7 eV. This composite material has potential to be used as wide band gap semiconductor (2 eV – 3 eV), with application as implantable and wearable devices. Moreover, cellulose as biodegradable material has potential to be used as green electronics. Fig. 2 shows the correlation between the PVA concentration to the energy gap.

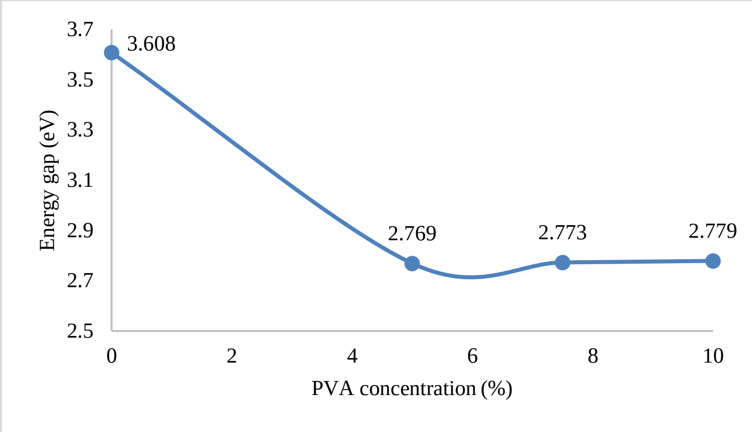

Fig. 2. The effect of PVA concentration to the energy gap of composite

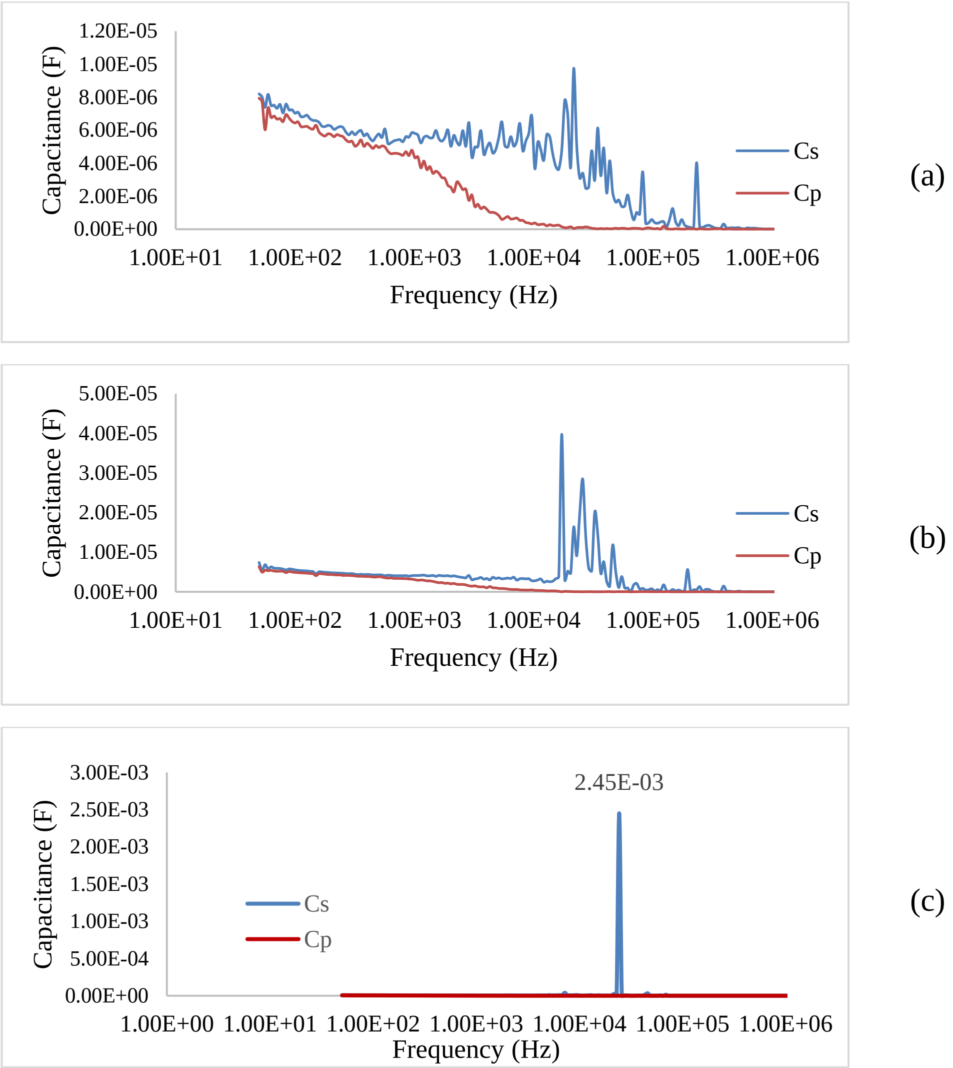

Fig. 3. Capacitance of Alkali cellulose composite (a) 5% PVA (b) 7.5% PVA (c) 10% PVA across frequency range of 5 Hz to 5 MHz, with Cs is series capacitance and Cp is parallel capacitance.

The addition of PVA significantly enhanced the electrical properties of the alkali cellulose. As with capacitance, conductivity, and impedance. Electrical properties of the composite was measured using an LCR meter within a frequency range of 5 Hz to 5 MHz. Fig. 3 presents a comparative analysis of series (Cs) and parallel capacitances (Cp) across the three composites. The data indicates a noticeable difference in the regularity of capacitance values between the two configurations. While the parallel capacitance exhibits a more consistent trend, the series capacitance displays a higher degree of variability. It is also observed that capacitance decreases with increasing frequency [19].

As depicted in Fig. 4, the composite's conductivity exhibited a direct correlation with the concentration of PVA. The conductivity value was calculated by multiplying the device thickness by the conductance and subsequently dividing by the device area [20]. The results showed that the composite with a 5% PVA concentration had the highest conductivity, reaching 2.65×10^{-4} S/cm. Composites containing 7.5% and 10% PVA concentrations exhibited lower conductivities, measured at 2.40×10^{-4} S/cm and 4.86×10^{-5} S/cm, respectively. The decreasing conductivity value can be attributed to the ion mobility inside the composite. PVA is used as matrix and it is not an electrical conductor by nature, so the excess PVA content can create denser composite or traps the ion using the hydroxyl group of the polymer, hence reducing the ion mobility. However, considering the typical conductivity range of semiconductors, 10^{-8}-10^{3} [21], it can be concluded that the alkali cellulose – PVA composite exhibited semiconductor properties.

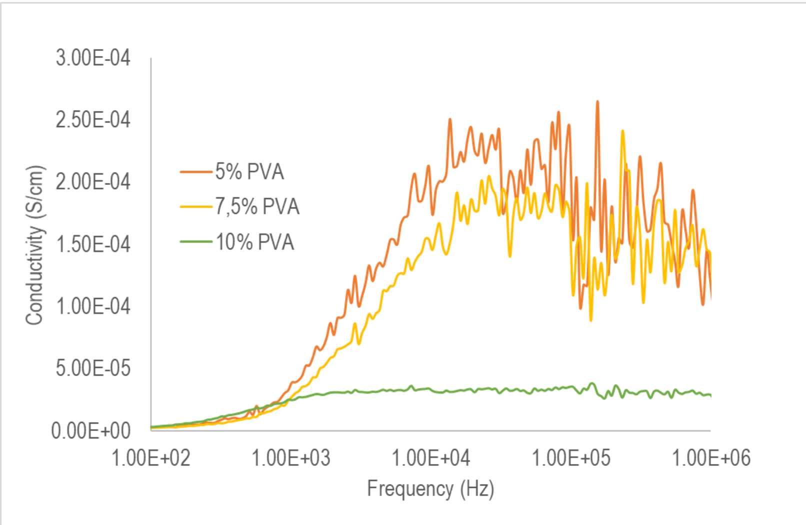

Fig. 4. Conductivity of Alkali cellulose - PVA composite

In addition to its effect on conductivity, the addition of PVA also influenced the impedance of the composite. Impedance is a complex number representing the measure of the opposition that a circuit presents to a varying current when a voltage is applied. It arises from the combined effects of resistance, inductance, and capacitance [22]. Impedance measurement is a powerful technique for characterizing and understanding the charge transport processes in complex materials [23]. Among the three concentration variations, the sample with 7.5% PVA exhibited the highest impedance. This was followed by the 10% PVA sample, while the composite with 5% PVA had the lowest impedance. As depicted in Fig. 5, impedance values exhibit a consistent decline as frequency increases. This trend is markedly pronounced in composites containing 5% and 7.5% PVA, where impedance drops significantly up to approximately 5 MHz Above this frequency, impedance values exhibit fluctuations but tend towards a constant value. Conversely, the composite with a 10% PVA concentration demonstrates a less steep impedance reduction, yet still exhibits constant fluctuations within the same frequency spectrum [24].

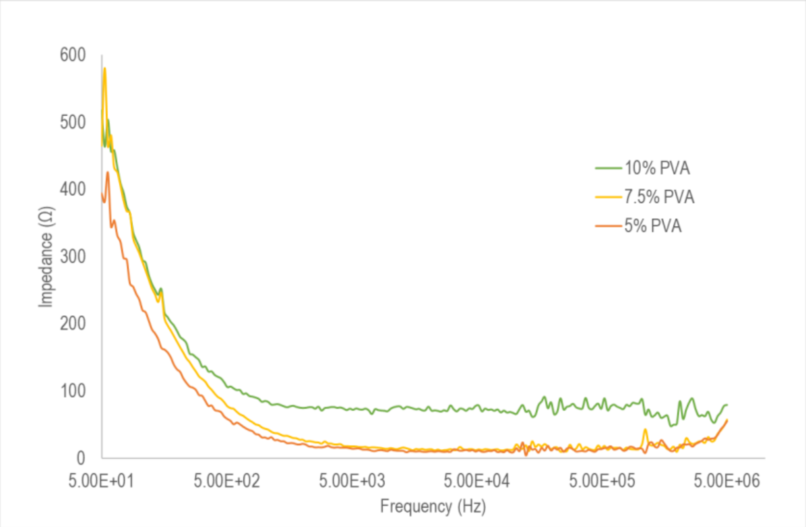

Fig. 5. Impedance of Alkali cellulose - PVA composite

Based on these results, the composite with 5% PVA concentration appears to be the most promising in terms of electrical properties. It offers a balance of high conductivity and relatively low impedance, making it suitable for applications requiring efficient charge transport.

4. Conclusions

The OPEFB alkali cellulose-PVA composites investigated in this study demonstrate promising potential as sustainable and low-cost semiconductor materials. Among the three variations, the composite with 5% PVA exhibited the most favorable properties, characterized by the highest optical absorbance, lowest band gap energy, and highest electrical conductivity. These findings suggest that alkali cellulose-PVA composites could be effectively utilized in the development of novel semiconductor sensors and other electronic devices. The combination of their natural origin, biodegradable nature, and desirable electrical and optical properties makes them attractive candidates for sustainable and environmentally friendly electronic technologies.

Data availability statement

Data supporting this result reported in this paper will be made available on request.

CRediT authorship contribution statement

Rima Adiati: Conceptualization, Formal Analysis, Writing – review and editing **Siti Nikmatin:** Conceptualization, Methodology **Irmansyah:** Supervision, Validation **Nazwa Nuradilla Putri:** Investigation, Writing – original draft **Siti Altirana Anandiwa:** Investigation, Writing – original draft

Declaration of Competing Interest

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.

Acknowledgement

This research is supported by IPB University community fund, year 2024 under the scheme of *"Penelitian Dosen* Muda*"* / Young lecturer's research, with contract number 23474/IT3/PT.01.03/P/B/2024.

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