# Electrical Conductivity of Conducting Polymer Composites based on Conducting Polymer/Natural Cellulose

Berlian Sitorus[1*], Mariana B. Malino[2]

[1] Department of Chemistry, Universitas Tanjungpura, Indonesia
[2] Department of Physics, Universitas Tanjungpura, Indonesia
Corresponding Email: [*] berlian.sitorus @chemistry.untan.ac.id

*Abstract –* **Merging each of the best properties of components into a composite design or hybrid architecture opens up opportunities to develop electroconductive materials as conducting polymer composite. This work deals with studying the electrical conductivity of conducting polymer composites made of cellulose extracted from two biomass: empty fruit bunch from oil palm and peat soil. Two kinds of conducting polymers have been used to fabricate the composites, i.e. polyaniline and polypyrrole, which are polymerized from their monomers, aniline and pyrrole. The novelty of this research is the using of biomass as the source of cellulose to produced conducting polymer composites by adding conducting polymer as filler into polymer matrix. We report experimental studies about the influence of monomer addition on the electrical conductivity of composites produced. The conductivity of the material was measured by using the Electrochemical Impedance System method. The experiments were carried out as a four-set experiment, using two different cellulose sources, EFB and peat soil, combined with aniline and pyrrole. The mass ratio variations of the monomer: cellulose are 1, 2, 3, and 4. The conductivities of the composites increased when more aniline or pyrrole was blended with the extracted cellulose from each source, either EFB or peat soil. The conductivity of composite PANI/EFB, which is $3.5 \times 10^{-3} - 1.1 \times 10^{-2}$ S/cm, is in the semiconductor range that makes the composites useful for many applications.**

*Keywords: Composite, conducting polymer, conductivity, empty fruit bunch, peat soil, polyaniline, polypyrrole*

## I. INTRODUCTION

Conducting polymer composite (CPC) is a composite consist of a matrix from either single or multi-phase blend polymer and conductive material as fillers. The conductive fillers can be carbonaceous, metallic, or conducting polymeric particles. The Chemistry Nobel Prize in 2000 recognized the importance of conducting polymers as it was rewarded for the innovation and development of conducting polymers [1-3]. Since then, the conducting polymers have attracted attention from academic and industrial development, such as applications in energy systems and electronics, organic light-emitting diodes, functional and electronic, optoelectronic devices, and energy storage as well as organic solar cells. Conducting polymers have been known as ideal matrices for embedding redox-active molecules. They can develop conductive materials that are easily processible, leading to introducing organic conductors in low dimensional with flexible and distinctive properties [4].

The CPCs usually contain a polymer matrix of one or more non-conducting polymers combines with filler from conductive materials dispersed in the matrix. The conductivity of CPCs is dominated by percolation theory, which describes the conducting phase of CPC formed by a network of the filler materials at a particular mass ratio. However, some of the shortcomings of CPCs include high dependence on processing conditions, mechanical instability, and an insulating surface layer over the conducting material [5].

A class of organic conducting polymers, also identified as intrinsically conducting polymers (ICPs), was established following the discoveries of conducting polymers. The transfer of electrons through the polymers themselves and the fully conjugated π-electron system spanning the polymer backbone's length determines the conductivity of ICPs. In contrast, extrinsically conducting polymers are based on the distribution of conductive particles such as metals in a non-conductive matrix polymer.

ICPs contain monomers capable of acquiring positive or a negative charge through oxidation or reduction, which contributes to the electrical conductivity in ICPs. The polymer doping oxidation generates an oxidative (p-type) while reduction produces reductive (n-type), both allow the conductivity tuning from semiconductors to metallic conductors [6]. Some examples of ICPs are polyacetylene (PA), polypyrrole (PPy), polythiophene (PT), and polyaniline (PANI). In this work, our interest is focused on the so-called intrinsically conducting polymers (ICPs), more specifically PANI and PPy.

The use of conducting polymers in the development of energy storage devices has received significant attention. Polyaniline, with a conductivity of 0.01-5 S cm$^{-1}$ is one of the most considered conducting polymers for this purpose because it has a relatively high electrical conductivity, good thermal properties, and environmental stability and is easy to fabricate [7]. Unfortunately, the mechanical properties of these polymers limit their commercial applications. Therefore, this PANI's

shortcomings need to be overcome by forming a polymer composite with a matrix of other materials to produce high conductivity and other superior properties.

Cellulose is a favorably crystalline material containing glucose units linked together in long chains; hemicellulose, as a polysaccharide, acts as a cementing matrix between micro-cellulose fibrils, forming the main structural component of the fiber cell [8]. As an abundant natural organic polymer, cellulose is the most promising group of materials for use as a matrix for conducting polymers. It can be obtained from plants, animals, and bacteria and has a unique combination of properties, including its flexible surface chemistry and its ability to bind to other materials, including a conducting polymer. Cellulose cannot be used directly to manufacture energy storage devices because it does not conduct electricity [9-12]. CPC's main benefit is that their electrical properties are close to the fillers, although the mechanical characteristics are most typical to plastics. The composites have numerous benefits over the traditional conductive materials, including their processability, flexibility, lightweight, ability to absorb mechanical shocks, and low cost [13]. CPC may be used as antistatic materials, switching devices, cables, transducers, and gas sensors. Moreover, CPC can be employed as a device for electromagnetic radiation shielding and electrostatic discharge [14].

### 1.2 Natural cellulose composites

Indonesia is the world's largest palm oil producer and responsible for almost 50% of global palm oil production in 2017 after a rapid expansion of oil palm plantations in recent decades [15]. In line with this, an enormous amount of Empty Fruit Bunch (EFB) also generates by the palm oil industry, approximately 40 million tons/year of EFB, which is about 20-30% of the weight of fresh fruit bunch [16-17]. However, it is not economical for further EFB processing in palm oil mills due to its low oil content. Thus, EFB is mainly treated as waste with low-value applications such as additional fertilizer or organic mulch in plantations [18-19]. According to [20], EFB typically comprises of cellulose (24–65%). Therefore, it is essential to utilize EFB and simultaneously use the resource for valuable products, at the same time to reduce its threat to the environment.

Researchers have shown that cellulose extracted from organic soils is similar to plant cellulose; they found it possible, from the cellulose content, to assess the proportion of undecomposed plant material in the peat [21]. A study performed for thirty samples from different peat soil sources depicted the cellulose content varies from 14.9 to 46.7 %, with an average of 25.4% [22]. Cellulose content from two different locations in Indonesia, Jambi and Central Kalimantan, was less than 10% [23]. Having different cellulose content makes EFB and peat soil has the potential as a source for cellulose-based products, such as for the fabrication of CPC.

Conducting composite is an alternative for solving the problem of electrical energy storage due to the CPCs' unique properties. In this research, the CPCs were fabricated by combining cellulose with PANI and PPy. The conductivities of the formed composites are then measured. The research results can be a source of insight for developing raw materials for making storage energy materials, such as batteries, based on organic materials with relatively economical and straightforward techniques and can also provide added value for agricultural waste.

## II. METHODOLOGY

### A. Materials

The palm oil EFB biomass waste was obtained from a local palm oil mill at Parindu, Sanggau District, West Kalimantan province, Indonesia, while the peat soil was taken from Kubu Raya Regency. Sodium hydroxide pellets, hydrochloric acid (37%), sulfuric acid (95–97%), ammonium persulphate (APS) were supplied by Merck, whereas aniline and pyrrole were acquired from Smart Lab. All chemicals and reagents were adopted as received without further purification.

### B. Pretreatment of the biomass and cellulose extraction

In this study, EFB and peat soil was chosen as the biomass source to fabricate CPCs. Pretreatment was started by washing to remove impurities before drying in an oven at 80 °C for 24 h. Size reduction was carried out using a commercial blender to turn the biomass into finer fiber form, which was then sieved to the particle size of less than or equal to 400 μm.

Afterward, in order to extract cellulose from EFB, the fiber was subjected to a chemically delignification process in a reflux system with 2% NaOH solution at 90ºC for three hours, followed by bleaching with a mixture solution of 1% NaOCl and NaOH. After cooling to ambient temperature, the resulting precipitated solid was filtered, washed thoroughly with distilled water to remove any possible remnants of chemicals, and dried 24h in an oven at 60ºC. The cellulose was kept in air-tight plastic bags and stored inside a desiccator to avoid contamination before the subsequent process.

### C. CPCs synthesis

The steps for the CPC synthesis were as the following. The cellulose obtained was mixed with aniline or pyrrole, converted to polyaniline by in-situ polymerization method, using ammonium persulfate $(NH_4)_2S_2O_8$ as an initiator for the polymerization process. Initially, the solution color was blue since the formed polyaniline was not protonated or in the emeraldine base form. Later the color turned green, which indicates that HCl has protonated the polyaniline to form conductive emeraldine salt.

The resulting PANI or PPy-coated cellulose fibers were filtered on a filter paper and washed thoroughly with distilled water to remove any free PANI or PPy. The coated fibers were then redispersed in ethanol and

similarly filtered and washed with water. This procedure was repeated several times to ensure the complete removal of any loosely bound or free polymer. Then the resulting cellulose fibers were dried.

### D. CPCs conductivity measurement

Impedance spectroscopy consists of a real component and an imaginary component, and they are measured as a function of the frequency. Generally, a small amplitude AC potential (sinusoidal form) is introduced to the system. The response is measured in the sinusoidal form at the same frequency but shifted in the phase. The Nyquist plot is one of the most used impedance spectra to understand the electrochemical responses [24].

About 1.5 g of the CPCs from each variation mentioned in (C) was inserted into a tubular copper tube. Nickel bars are added and positioned in the middle of the tube, followed by adding composites until the CPCs covered the whole tube. Then it is compacted using a manual press with the same force magnitude.

The CPCs electrical conductivity measurement was carried out using the two-electrode impedance technique on the Agilent E4890A LCR meter with 20Hz – 2MHz of frequency. The results data were obtained in the form of impedance values (Z) and impedance angles (θ). The data is then processed to obtain the resistance value, as in Equation (1).

$$Z_R = Z \cos \theta \qquad (1)$$

The conductivity (σ) is then obtained by converting $Z_R$, which is the real impedance using Equation (2).

$$\sigma = \frac{1}{Z_R} \times \frac{l}{A} \qquad (2)$$

Where

σ = conductivity (S/cm)

$Z_R$ = impedance (Ω)

$l$ = distance between electrodes/sample thickness(cm)

$A$ = cross-sectional area of the sample (cm$^2$)

### III. RESULTS AND DISCUSSION

The experiments were carried out as a four-set experiment, using two different cellulose sources, EFB and peat soil, and two kinds of monomers, i.e., aniline and pyrrole, then polymerized with conducting polymers PANI and PPy, respectively. The amount of aniline or pyrrole mixed with the extracted cellulose was varied into four. This led to four mass ratio variations of the monomer: cellulose, which was 1, 2, 3, and 4.

The synthesis was arranged by combining the conducting polymer and the extracted cellulose from natural sources, as follows:

1. Aniline with EFB (PANI/EFB)
2. Pyrrole with EFB (PPy/EFB)
3. Aniline with peat soil (PANI/peat soil)
4. Pyrrole with peat soil (PPy/peat soil).

These variations are summarized in Table 1.

**Table 1.** The variation of cellulose-monomer combination used to produce CPCs

| Monomer | Cellulose source | Conducting Polymer Composite |
|---------|------------------|------------------------------|
| Aniline | EFB | PANI/EFB |
| Pyrolle | | PPy/EFB |
| Aniline | Peat soil | PANI/ |
| Pyrrole | | EFB/PANI |

The present research aimed to examine the potential of conducting polymer composites based on two conducting polymers, PANI and PPy, combined with cellulose from two different sources, EFB and peat soil. Therefore, the discussions are divided into two parts in order to compare the electroconductivity of each cellulose-monomer combination.

Conductivities of the CPCs synthesized from cellulose extracted from two sources, EFB and peat soil, combined with two conducting polymers, are shown in Table 2 and Table 3. For each of the variations, four specimens were used. The data presented in both tables were obtained from four measurements of four CPCs packed in the copper tube for each variation. Thus, there were four CPCs (4 x 1.5 g CPCs inserted in a tubular copper tube) for every variation. All conductivities shown in both tables are the average values and followed by the standard deviations from four packed-CPCs measured for each of the variations. However, only the average conductivity values are exhibited in Figure 3 and Figure 3.

### A. Comparing Conductivity of CPC based on the conducting polymer

Electrical conductivity in conducting polymer involves positively charging carriers or electrons along polymer chains and the hopping of these carriers between chains. Although there are many papers on optimizing PANI and PPy, including their conductivity, it is not easy to compare because various research groups have used different conditions of preparation and characterization.

**Table 2.** The conductivity of CPCs using EFB as the cellulose source

| Mass ratio of monomer: cellulose from EFB | CPCs' Conductivity using conducting polymer (S/cm) | |
|---|---|---|
| | PANI/EFB | PPY/EFB |
| 1.0 | $(7.0 \pm 0.2) \times 10^{-3}$ | $(6 \pm 0.4) \times 10^{-3}$ |
| 2.0 | $(9.2 \pm 0.1) \times 10^{-3}$ | $(7.8 \pm 0.5) \times 10^{-3}$ |
| 3.0 | $(2.1 \pm 0.2) \times 10^{-2}$ | $(1.2 \pm 0.08) \times 10^{-2}$ |
| 4.0 | $(2.2 \pm 0.09) \times 10^{2}$ | $(1.1 \pm 0.1) \times 10^{-2}$ |

The values in Table 2 are owned by the composites made of extracted cellulose from EFB with PANI or PPy, with four mass ratios of monomer: cellulose. Table 3 compares the conductivity measured in the CPCs when the same ratio variations in Table 2 are used to obtain the composites of cellulose from peat soil with PANI or PPy as the conducting polymer.

From Table 2, it can be concluded that there was a clear trend of increasing conductivity when more aniline or pyrrole was added to the mixture during polymerization. Namely, the composites' conductivity was very much affected by the amount of monomer added to the cellulose. It is depicted that the conductivity for the CPCs cellulose from EFB conductivities leveled up as the mass ratio was raised.

**Table 3.** The conductivity of CPCs using peat soil as the cellulose source

| Mass ratio of monomer: cellulose from peat soil | CPCs' Conductivity using conducting polymer (S/cm) | |
|---|---|---|
| | PANI/peat soil | PPY/peat soil |
| 1.0 | $(5.2 \pm 0.07) \times 10^{-3}$ | $(3.6 \pm 0.5) \times 10^{-3}$ |
| 2.0 | $(7 \pm 0.25) \times 10^{-3}$ | $(6.4 \pm 0.2) \times 10^{-3}$ |
| 3.0 | $(2 \pm 0.14) \times 10^{-2}$ | $(9.3 \pm 0.2) \times 10^{-3}$ |
| 4.0 | $(2.3 \pm 0.1) \times 10^{-2}$ | $(1.0 \pm 0.1) \times 10^{-2}$ |

As can be seen from both Table 2 and Table 3, the reported conductivities of the composites increase when more aniline or pyrrole is blended with the extracted cellulose from each source, either EFB or peat soil. Both tables show that the quantity of conducting polymer in the composites increased along with the larger amount of monomers mixed with the extracted cellulose. It is apparent that the in-situ polymerization processes of the monomers were successfully coated the conducting polymers to cellulose.

Another aspect that we can see from Table 2 and Table 3 is comparing composites' results using the same cellulose sources but different monomers. Within the corresponding mass ratio, most of the conductivity values are comparable between PANI and PPy.

The polymerization of aniline carried out in this work was designed to be as straightforward. The synthesis was based on mixing aqueous aniline hydrochloride and APS solutions at room temperature, followed by the separation of PANI hydrochloride precipitate by filtration and drying. The most common green protonated emeraldine has conductivity on a semiconductor level of the order of $10^0$ S/cm, many orders of magnitude higher than that of common polymers, which is $10^{-9}$ S/cm, but lower than that of typical metals ($<10^4$ S/cm) [23]. Conductivity in PANI is the property of conducting polymers, whose nature is explained by the ability of PANI to form polarons, cation radicals. The polarons can eventually spread over the polymer chain to produce a polaron lattice [25].

The oxidation of aniline is exothermic, so the reaction mixture's temperature can be used to monitor the reaction's progress. Figure 1 displays a typical polymerization recorded when 0.2 M aniline hydrochloride oxidized with 0.25 M APS in 100 ml or 500 ml of aqueous medium [26]. The oxidation was started at room temperature. After an induction period, polymerization commences, and the reaction mixture's temperature increases; it passes through a maximum after the reaction is complete, and the medium cools down.
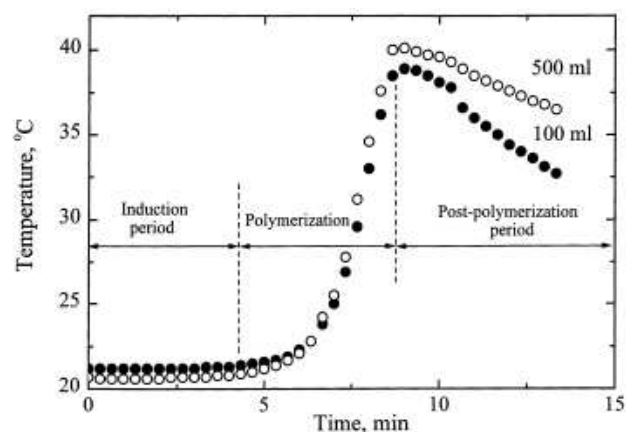


**Figure 1.** Temperature profile in the polymerization of aniline according to [25].

The polymers formed from PPy have a more irregular chain structure than PANI with a narrower crystalline area [27]. Conducting polymers with irregular chain structures can generate an electric current through a mechanism dominated by hopping. The conductive electrons are condensed in small areas surrounded by a much wider amorphous region. Thus, the conductive electrons have to hop from one delocalized position to another delocalized position with phonons' help to pass through via tunneling. However, when APS has been used as the oxidant, the conductivity of PANI reported in the literature is generally higher than that of PPy [28], as in the present case.

### B. Comparing Conductivity of CPCs based on the sources of cellulose used

Figure 2 and Figure 3 compare the conductivity values when the composites formed using the same conducting polymers. The former figure shows conductivity measured when the CPCs have PANI, whereas the latter shows the effect of PPy on those composites. The dependence of the conductivity on the monomer-to-cellulose mass ratio has common features for PANI and PPy. By comparing the two results depicted in Figure 2 and Figure 3, it can be seen that both monomers have a similar effect that more amount of monomer led to larger conductivities.

Figure 2 shows that the CPCs' conductivities obtained using aniline are only slightly different for the comparable aniline: cellulose mass ratio. The values are laid in the same order of magnitude. For example, is in the ratio of 1, PANI/EFB has a conductivity of $7 \times 10^{-3}$ S/cm while it is $5 \times 10^{-3}$ S/cm for PANI/peat soil. Nevertheless, PANI/EFB composite always has higher conductivities compare to their counterparts.
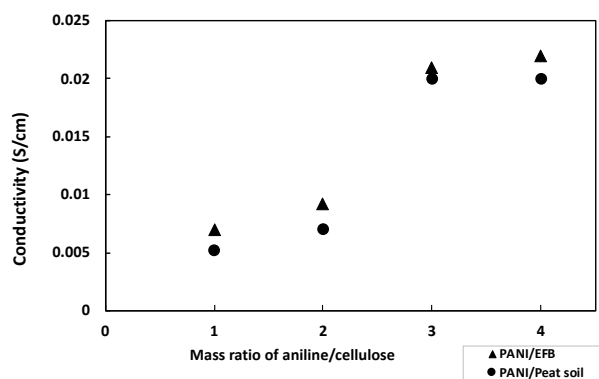
**Figure 2**. Conductivity comparison of CPCs using EFB and peat soil as cellulose sources and PANI as conducting polymer

A corresponding expression is depicted in Figure 3, showing the behavior of composites's formed from cellulose using pyrrole as the monomer. A similar trend like in Figure 2, related to monomer concentration, is also found in Figure 3. The conductivities of CPCs contained PPy became higher as more pyrrole was added to the mixture, polymerized and mixed with cellulose. In addition, the conductivities of PPy/EFB are also higher than PPy/peat soil.
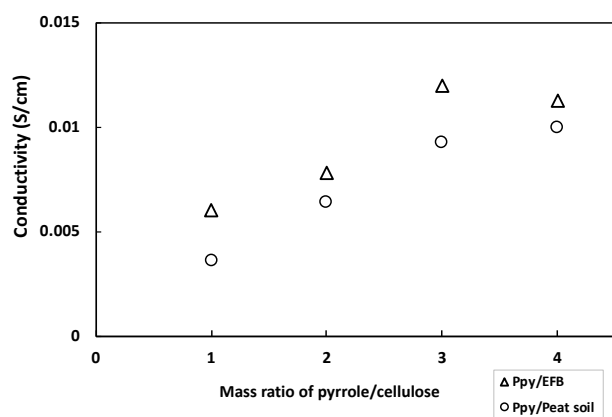


**Figure 3**. Conductivity comparison of CPCs using EFB and peat soil as cellulose sources and PPy as conducting polymer

An explanation for the higher conductivities in EFB can be described as the following. The morphological analysis using Scanning Electron Microscope (SEM) for the PANI/cellulose from peat soil evident that the PANI was covered in cellulose [29]. This condition causes a decrease in the polaron's mobility, thereby reducing the concentration of the space carrier limited. On the other hand, the micrograph from PANI/cellulose from EFB in [30], revealed that PANI's aggregate was dispersed on the cellulose's surface so that the cellulose chain layers did not hinder the movement of polaron. In other words, polaron is easier to move from one delocalized position (delocalized site) to another delocalized position to produce effective mobility that can increase the space carrier limited. Thus the polaron dispersed better in EFB than in peat soil and led to a higher conductivity than the corresponding composite. At this point, the significant value of CPCs arises in combining the electrical properties of conducting polymer with the filler's mechanical strength and the ease of fabricating the matrix.

## IV. CONCLUSION

In the present paper, the conducting polymer composites were synthesized by chemical-oxidative polymerization of two kinds of monomers, aniline and pyrrole, with natural cellulose from two different sources, EFB and peat soil activated by acid.

The electrical conductivity increased in both monomers and reached the maximum values at the mass ratio monomer: cellulose of four. The conductivity measurements show that the composite resulting from the incorporation of polyaniline or polypyrrole/cellulose from empty fruit bunches or peat soils is a material with conductivity in the semiconductor range. Overall, the highest conductivity value is owned by composites with a mass ratio variation of 4, namely $2.2 \times 10^{-2}$ S/cm for composites using cellulose from EFB and $2.3 \times 10^{-2}$ S/cm for composites with cellulose from peat soil. The conductivity measurement results also showed that the polyaniline/cellulose composite's conductivity value was higher than that of the polypyrrole-cellulose composite. The composites cellulose/PANI and cellulose/PPy would be promising materials in application due to their electrical conductivity.

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