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Enhancing Current Density and Specific Capacitance through Tensile TOBC/PPY Nanocomposites

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Abstract— The researchers developed a bio-composite film material that serves as a substitute for metal. The materials used are TEMPO ((2,2,6,6-tetramethylpiperidine-1-oxyl)), Bacterial Cellulose, and Polypyrrole (Ppy). This research aimed to increase the current density and specific capacitance values of the material using the drawing method. Composite nanomaterials are made by oxidizing BC (Bacterial Cellulose) with TEMPO. The resulting TOBC (TEMPO Bacterial Cellulose) material was mixed with Ppy using the in situ method. The mixture is then drawn wet—measurement of current density and capacitance using Cyclic Voltammetry (CV) Testing. The results of the current density and specific capacitance increased by 542.74% and 754.79% after drawing the nanocomposite material. It is directly proportional to the effects of characteristic testing, which includes SEM, XRD, and FTIR. As a result of the withdrawal of the polypyrrole, it will be more evenly distributed in the composite material, absorbing and coating the nata de coco. When the TOBC/Ppy fibers are straighter and denser, they achieve higher current density and capacitance values, as concluded by the researchers.

Keywords: Bacterial Cellulose, Composite nanomaterials, Tensile method, Wet Tensile, Even distribution.

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1. Introduction

Acetobacter xylinum is used to ferment coconut water, producing nata de coco. Acetobacter xylinum breaks down the glucose in coconut water into carbon, which is subsequently transformed into cellulose fiber during Nata de Coco production [1][2]. Under the influence of Krebs cycle enzymes, acetobacter may quickly oxidize acetic acid to carbon dioxide and water. As a result, they cannot oxidize acetic acid, in contrast to other genera such as Gluconobacter [3][4]. Bacterial cellulose fiber also has a high tensile strength and is corrosion-resistant [5]. Often referred to as cellulose fiber (BC), bacterial cellulose has several benefits, including low cost and environmental friendliness. Regarding mechanical strength, the bacterial cellulose fibers in this study outperform cellulose fibers made from plants [6]. The bacterial cellulose must be mixed with a substance with high electrical conductivity to form a conductive membrane.

Composite materials are innovative materials that combine two or more materials with exceptional properties. To create composite materials with electrical characteristics, we conduct material mapping to identify substances with both solid mechanical and electrical properties. We aim to develop composites that can replace metals in various applications.

Innovative materials, known as composites, combine two or more materials with remarkable qualities. We perform material mapping to find materials with solid mechanical and electrical properties and then create composite materials with electrical characteristics. Our goal is to develop composite materials that can take the place of metal in a variety of uses.

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Building on the previous justification, Nata de Coco's excellent mechanical qualities make it a viable option for use as a composite material in this study. At the same time, its electrical conductivity needs to be improved. Consequently, polymer compounds such as polypyrrole, polyaniline (PANI), polythiophene (PTP), poly (3,4-ethylenedioxythiophene) (PEDOT), and poly (p-phenylenevinylene) (PPV) are required as electrical composite materials [7].

The use of polypyrrole is the main polymer compound that this study considers. A substance that can aid in oxidizing Polypyrrole and Nata de Coco is necessary for their successful combination.

TEMPO is the most commonly used technique for combining cellulosic materials because it binds functional groups and promotes their growth. When it comes to oxidizing bacterial cellulose (Nata de Coco), the use of TEMPO is intended to improve the membrane surface by smoothing it out, creating cavities for nanoparticles, maintaining homogeneity, and increasing pore density [8][9]. Based on prior research, the bio composite materials under investigation appear to have low capacitance and current density. The study shows polypyrrole cannot cover the bio composite fibers and fill in nano-gaps [10].

Bio composites are essential in today's materials landscape because of their strong mechanical and electrical properties, as the previous explanation shows. Still, there is potential to improve the electrical characteristics of the TOBC/Ppy bio composite material. This research aims to increase the values of specific capacitance and current density using a drawing-induced process. This technique promotes increased polypyrrole infiltration by aligning the nata de coco fibers and causing a pulling effect on the TOBC/Ppy bio composite. Thus, this alignment aims to improve specific capacitance and current density. SEM, XRD, and FTIR are examples of extensive characteristic tests that will validate these results. The desired outcomes are materials for electrical conductors that are strong, flexible, and environmentally benign. This bio composite material has the potential to be an excellent alternative to conventional electronic metals.

2. Method

The following research materials: Nata de Coco, acquired from an Indonesian production facility in Siteba, Padang, West Sumatra. Sigma-Aldrich, St. Louis, Missouri, USA is the source from where we obtained the Sigma-Aldrich brands Polypyrrole (Ppy), Sodium Bromide (NaBr), and TEMPO. The Metallurgy Laboratory, Department of Mechanical Engineering, Andalas University, has readily available technical-grade ferrous chloride (FeCl3) (Aloin brand), technical-grade sodium hypochlorite (NaOCl), deionized water (Aquadest), acetone, and hydrochloric acid (HCl).

The bacterial culture of cellulose obtained from Nata de coco is subjected to a series of treatments. It is first soaked in a 0.5% NaOH solution and then thoroughly rinsed with distilled water until the pH reaches 7, which removes any remaining materials and bacteria [10]. The BC membrane was soaked in acetone after the researchers used an Ampia grinder to grind it into thin sheets finely. The goal of acetone soaking and grinding is to reduce the water content of the membrane. After soaking, the BC membrane is divided into several follicles, each measuring 10 cm by 1.5 cm.

There are various steps involved in using TOBC to produce films: First, dissolve 0.1 grams of NaBr and 0.016 grams of TEMPO in 100 milliliters of distilled water. Stir the mixture for 30 minutes at 500 revolutions per minute until it dissolves completely. Then, add the nata de coco follicles to the mix and spin them for ten minutes at 200 rpm.

After that, add 1.6 grams of NaOCl and stir for an additional 10 minutes at 200 rpm. After rushing for ten more minutes, add 0.5% NaOH to bring the pH to 10. The solution should be heated to 70°C and held there for 30 minutes before cooling to room temperature. Lastly, use HCl to adjust the pH to 7.

To combine Ppy and TOBC to create a bio composite material, mix 1.83 ml of Ppy with 4 ml of FeCl3. Then, use the generated Ppy solution to immerse the TOBC.

Under ASTM D 882-02 guidelines, we conducted the tensile testing at a 5 mm/min speed. [11]. We sampled in an environment with high humidity (90 ± 5%). COM-TEN Testing Machine 95T series 5K is the instrument in use. Tensile strength, strain, and tensile modulus were the test results.

Cyclic Voltammetry (CV) is used to obtain current density and specific capacitance values. This test used a 1 M H2SO4 electrolyte solution at room temperature (27°C) and a 0-1500 mV voltage range. Two electrodes were used: An Ag/AgCl reference electrode and an Ag/Pt auxiliary electrode. The following is the equation to get the current density as equation 1 as follows [13].

$$J = \frac{I}{A} \quad (1)$$

Where I is the current strength (A), and A is the cross-sectional area of the wire (cm^2). Meanwhile, to get the specific capacitance value. It can be formulated as equation 2 as follows [15]:

$$Csp = \frac{Ic-Id}{v \times m} \quad (2)$$

Where Ic is the charge current (A), Id is the discharge current (A), s is the scan rate (mV/s), and m is the average mass (g).

Fourier transform infrared (FTIR) is a technique used to determine a compound's functional groups. One instrument used in FTIR testing is PerkinElmer. Samples were dried for 48 hours at room temperature (27^{o}C). The FTIR machine will then scan the pieces at a frequency of 500–4000 cm^{-1} and a resolution of 4 cm^{-1} [16].

An X-ray diffraction (XRD) device determines a material's crystalline level. One instrument used in XRD testing is PAN analytical Xpert PRO. Tests were conducted at 40 kV, 30 mA, and 25 ^{o}C [17]. The scanning angle of the sample will be 2θ = 10^o to 50^o. One can use Segal equation 3 [18] to determine the crystalline index:

$$CRI = \frac{(I_{hkl}-I_{am})}{I_{hkl}} \times 100\% \quad (3)$$

In cellulose, the maximum diffraction intensity of the crystal area is represented by I_{hkl}, and it is comparatively located at 2θ = 22.5^o. I_{am}, on the other hand, is the intensity of amorphous diffraction. It is approximately 2θ = 16^o in cellulose [19]. Meanwhile, the Debye Scherer equation 4 can be used to find the sample's crystal size.

$$D = \frac{0.9\lambda}{B\,Cos\,\theta} \quad (4)$$

Where θ is the peak formation's angular position, B is the Full Width at Half Maximum, D is the crystal size (Å), and ***E*** is the wavelength used for testing.

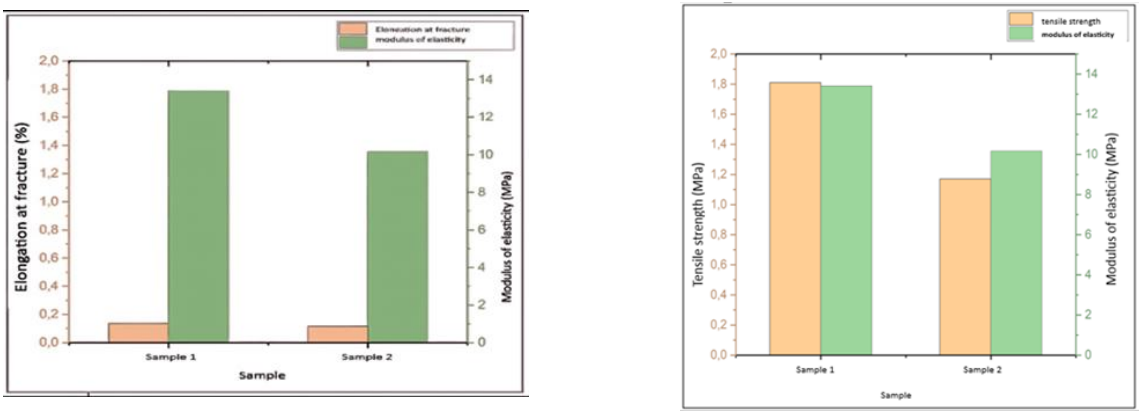
The sample surface will be described using scanning electron microscopy (SEM). One instrument used in SEM testing is the Hitachi S-4800 SEM. A 1 µm ruler was used with a 500-times magnification during the test.

3. Result and Discussion

3.1 Tensile Testing

From the results of the biocomposite tensile testing, the tensile strength, elongation at break (extension), and elastic modulus of the samples were obtained. Figures 1a and 1b show that the tensile strength and elongation increase with the material's elastic modulus tends to be stable. It means it has flexible and plastic properties [22]. Tensile sample 1 has a maximum tensile strength of 1.81 MPa and a maximum elongation of 0.13. Meanwhile, tensile sample 2 has a full tensile strength of 1.17 MPa and a maximum strain of 0.12. Differences in tensile

strength, extension, and elastic modulus values between the two samples occur because cellulose has inhomogeneous fibers [23].



a) b)

Figure 1. Tensile test graphs a) tensile strength and modulus of elasticity, b) maximum elongation and modulus of elasticity.

3.2. Measurement of Current Density and Specific Capacitance

From the CV test, data is obtained, which is plotted into a voltammogram curve. The following is the voltammogram curve from testing TOBC/Ppy nanocomposite samples without a tensile test and with a tensile test:

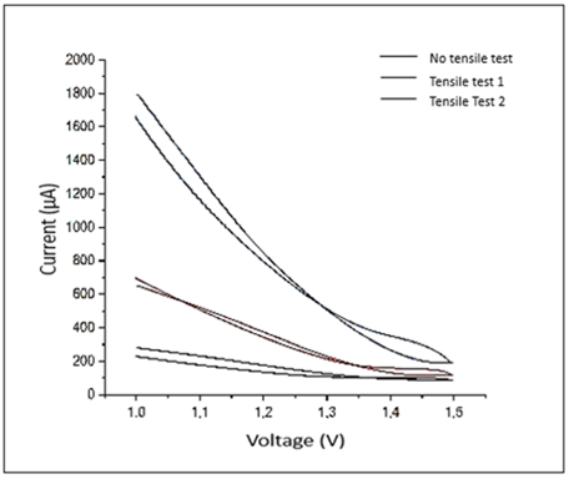


Figure 2. Voltamogram graph

The figure 2 shows the oxidation-reduction reaction on the surface of the TOBC/Ppy composite electrode [25]. The occurrence of electrochemical processes shows the movement of electrons through redox reactions. From the picture, it can be seen that Ic (charge current) is the current at the oxidation peak, and Id (discharge current) is the current at the reduction height. The current value used in equation 1 is the current Ic [26]. Following are the results of the cyclic voltammetry test table and the calculation of the current density value based on equation 1.

Table 1 Test results and current density calculations

No	Sample	A (cm^2)	E(V)	Ic (µA)	Id (µA)	Current Density (µA/cm^2)	Error (%)
1	No Tensile Test	2	1,00	278,63	86,39	139,3	4,15
2	Tensile Test 1	2	0,99	696,6	113,4	348,3	4,24
3	Tensile Test2	2	1,01	1790,7	189,6	895,3	4,08

Based on the table 1, the current density values without withdrawal, withdrawal 1, and withdrawal 2 were obtained at 139.3 µA/cm2, 348.3 µA/cm2, and 895.3 µA/cm^2. In withdrawal sample 1 and pulling sample 2, there was an increase of 150.03% and 542.74% without pulling. Meanwhile, to get the specific capacitance value, use equation 2. The following are the results of the cyclic voltammetry test table and the calculation of particular capacitance values:

Table 2 Test results and specific capacitance calculations

No	Sample	Scan rate (mV/s)	Massa (gr)	E(V)	Ic (µA)	Id (µA)	Csp (µF/gr)	Error (%)
1	No Tensile Test	50	0,028	1,00	278,63	86,39	137,3	4,15
2	Tensile Test 1	50	0,028	0,99	696,6	113,4	416,6	4,24
3	Tensile Test2	50	0,028	1,01	1790,7	189,6	1143,6	4,08

Based on the table 2, the specific capacitance values without withdrawal, withdrawal one, and withdrawal two were obtained at 137.3 µF/gr, 416.6 µF/gr, and 1143.6 µF/gr. In withdrawal sample 1 and retreat sample 2, there was an increase of 303.42% and 754.79% from without withdrawal. From this test, there are differences in the current density and specific capacitance values of the samples using the tensile test. It happens because cellulose bacteria have fibers not arranged regularly and different absorption intensities on their sides [27].

3.3. Fourier transform infrared (FTIR)

In Figure 3, it can be seen to determine the functional groups of natural fibers [28]. In the FTIR spectrum, region I, which can be seen in the image, is 3600-3050 cm^{-1} [29]. The wave absorption value of the TOBC/Ppy biocomposite sample without the tensile test was obtained (3286 cm^{-1}). It can be seen that the wave absorption value was more significant than the wave absorption value of the TOBC/Ppy biocomposite sample with the tensile test (3227 cm^{-1}). It means that models without tensile tests have higher humidity.

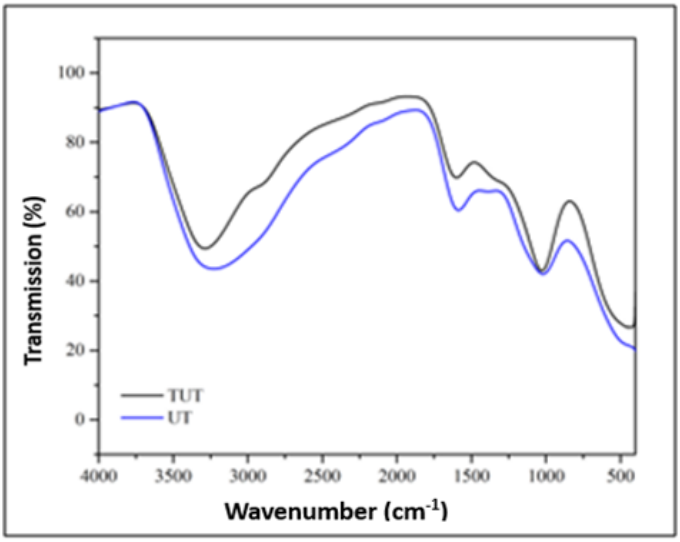


Figure 3 FTIR graph of TBC/Ppt nanocomposites

In region II, which can be seen in the figure 1800-1450 cm^{-1} [22]. The wave absorption value of the TOBC/Ppy biocomposite sample with the tensile test (1586 cm^{-1}) is close to the C=C (1540 cm^{-1}) and C-N (1467 cm^{-1}) stretching vibration wave absorption value. It means that the Ppy absorption value of the TOBC/Ppy biocomposite sample with a tensile test (1586 cm^{-1}) is better than the Ppy absorption value of the TOBC/Ppy nanocomposite sample without a tensile test (1599 cm^{-1}). The similarity of polypyrrole vibration characteristics (C=C and C-N stretching vibration wave absorption values) with TOBC/Ppy biocomposite samples using

tensile tests [30]. It can be interpreted that in the biocomposite sample using this tensile test, polypyrrole can coat and fill the nano-sized nata de coco fibers well.

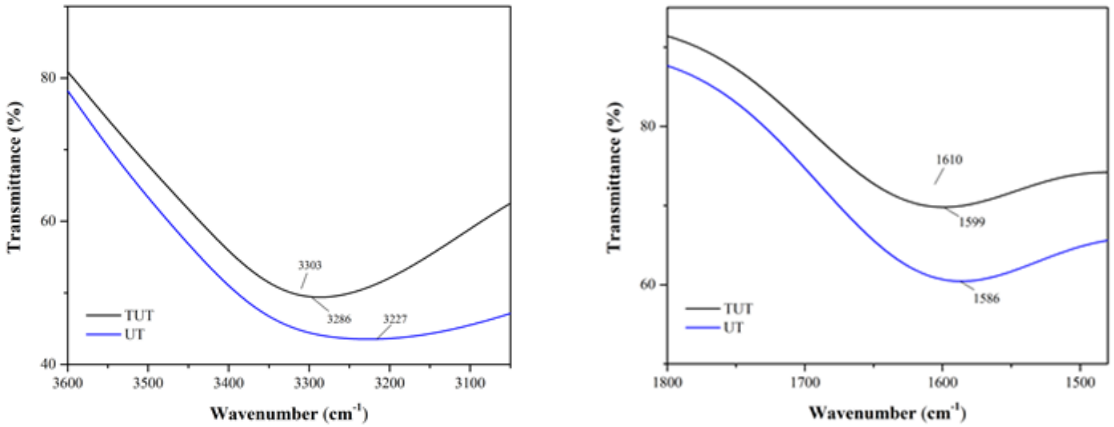
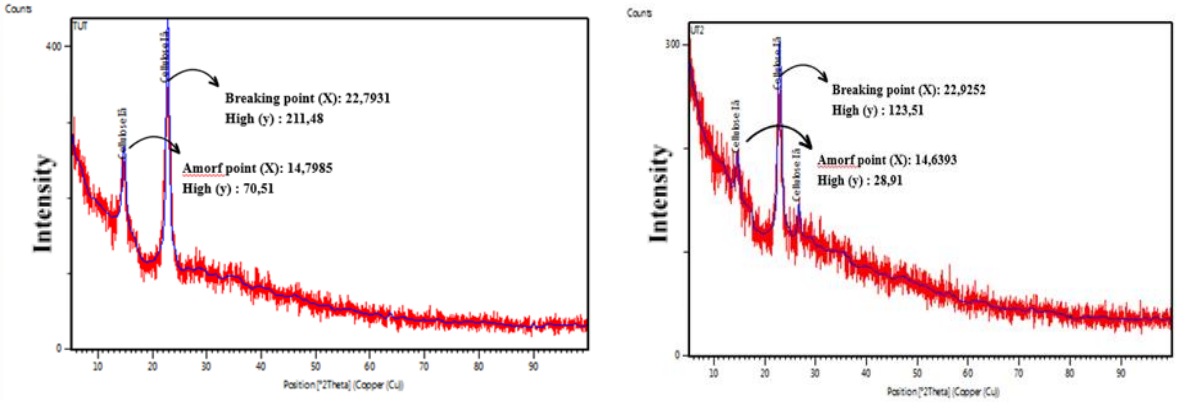


Figure 4 FTIR graph of OH (Water Content) a) and C=C (Polypyrrole Ring) b) *X-ray diffraction (XRD).*

Figure 5 shows the results of XRD testing without withdrawal and with withdrawal. In the picture, two prominent peaks approach 2θ = 22.5º and 2θ = 16º. It is consistent with previous research [19].



a) without tensile test b) with tensile test

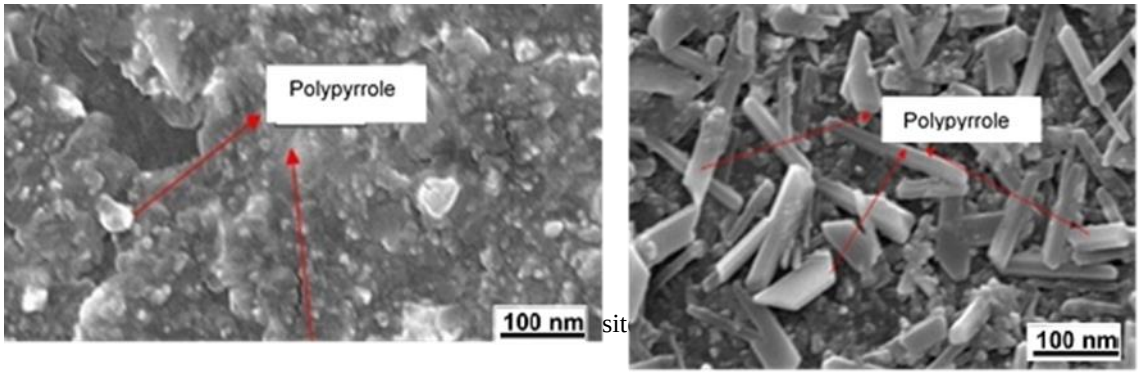
Figure 5 XRD graph of TOBC/Ppy bio composite

The researchers discovered the amorphous intensity location 2θ = 14.79º and the diffraction peak intensity position 2θ = 22.79º while testing the TOBC/Ppy biocomposite sample without performing a tensile test. In the meantime, 2θ = 22.92º was the diffraction peak intensity location and 2θ = 14.64º was the amorphous intensity position for tensile testing of TOBC/Ppy biocomposite samples. The crystallity index increased by 66.66% using the Segal method and increased to 76.59% upon withdrawal. The density between molecular bonds increases with increasing crystallinity index [31].

By using the Debye Scherrer technique, the 3.90154 Å starting size is reduced to 3.87936 Å. We can see that the particle size decreases after pulling [20], suggesting that it gets smaller.

3.4. Scanning Electron Microscopy (SEM)

The surface morphology of the TOBC/Ppy biocomposite is shown in Figure 6. The material in Figure 6a, which has not been stretched, shows an uneven distribution of polypyrrole. In contrast, Figure 6b shows the biocomposite material after pulling, which demonstrates a more even distribution of polypyrrole on the surface. [32].



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4. Conclusion

The test results showed that the nanocomposite material's current density value and specific capacitance value increased after being withdrawn. The characteristic test results are XRD, FTIR, and SEM tests, which validate the electrical properties test results. The XRD, FTIR, and SEM tests show an increased crystallinity index; polypyrrole has coated and spread evenly on the material. The test results indicate that the drawing method can improve the electrical properties of nanocomposites that will be used as fuel cell membranes.

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