Characterization of nata de soya electrolyte membrane as a capacitor separator using $(NH_4)_2SO_4$

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Abstract. Tempeh and tofu production continues to grow rapidly in Indonesia. This means that the volume of liquid waste is also increasing dramatically. The problem is that most of this waste is disposed of in the environment and has the potential to pollute nature. In this study, Nata de Soya was used as a capacitor separator after undergoing a soaking process. This material was soaked for 30 minutes in an ammonium sulfate ($(NH_4)_2SO_4$) electrolyte solution. Researchers tested four different concentration levels, namely 22.5%, 25%, 27.5%, and 30%, to see how it performed. Characterization was performed using Cyclic Voltammetry (CV) to measure its specific capacitance and Electrochemical Impedance Spectroscopy (EIS) to measure the level of ionic conductivity. The test results show that increasing the electrolyte concentration has a positive effect on performance. After 30 minutes of soaking, the 30% concentration sample showed the highest capacitance compared to other samples, at 13.35 F/g, and also had the highest ionic conductivity, at 0.382 S/m. Based on these results, Nata de Soya membranes treated with varying electrolyte concentrations, including the 30% sample, show potential to be further explored as separator materials for environmentally friendly energy storage applications.

1 Introduction

Tofu and tempeh are foods that are popular among Indonesians. They have long been recognized as healthy, nutritious, and inexpensive foods. Almost every city in Indonesia has a tofu and tempeh industry. According to data from the Central Statistics Agency (BPS) in 2024, the average weekly consumption of important foodstuffs per capita for tofu was 0.148 kg and for tempeh was 0.136 kg. The abundance of tofu and tempeh is in line with the rapid growth of the tofu and tempeh manufacturing industry. As the population grows rapidly, the waste produced will become environmental pollution if it is not handled properly[1].

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One way to process tofu and tempeh waste is to use it in the development of porous organic composite membranes. Solid waste from tofu pulp is often processed into food products or given to third parties for use as animal feed, while liquid waste, or whey, is left to flow into water sources around the factory[2]. The organic substances contained in whey can be used as a source of nutrients in the production of Nata de Soya[3].

The depletion of energy sources, increasing global warming, and excessive environmental pollution have triggered the development of environmentally friendly renewable energy resources [4]. A capacitor is a device or container that has the ability to store electrical charge[5],the charge can be stored and also released. Nata de Soya is an attractive choice because of its porous membrane characteristics.

The mixture of liquid waste from tofu and tempeh production is used to make Nata de Soya electrolyte membranes. Before being made into electrolyte membranes, the membranes are soaked in ammonium sulfate electrolyte solutions at different concentrations. Ammonium sulfate[6] was chosen because it is a compound that can ionize in water and produce ions that are conductive to electricity. Electrolytes are important because they have the ability to convert values into capacitance[7].

To characterize the Nata de soya electrolyte membrane separator, two characterization tests were performed, including Cyclic Voltammetry (CV) and Electrochemical Impedance Spectroscopy (EIS). The CV test was performed to understand the energy storage mechanism during the charging process. The EIS test is also important when analyzing electrical conductivity. EIS can provide information about charge transfer impedance, charging and discharging mechanisms in the system, using Nyquist plots[8].

2 Materials dan Methods

2.1. Nata de Soya Membrane Synthesis

Nata de soya is produced by fermenting liquid waste from tofu and tempeh using Acetobacter Xylinum bacteria at a pH of 4-5 for approximately 7-14 days to produce nata fibers. Next, Nata de soya is processed into membranes using cold-press and hot-press techniques, resulting in dense, thin, porous sheets. This study used ready-made Nata de Soya membranes with a sample size of 3x3 cm. The membranes were soaked in ammonium sulfate ($(NH_4)_2SO_4$) electrolyte solutions at varying concentrations, namely 22.5%; 25%; 27.5%; and 30%, for 30 minutes each. In preparing the solution concentration, we used the weight/volume (w/v) measurement method, which involves measuring the grams of solute in 50mL of distilled water solution.

2.2. Nata de Soya Electrolyte Membranes Synthesis

The electrolyte solution is prepared by dissolving ammonium sulfate ($(NH_4)_2SO_4$) in distilled water until it is mixed homogeneously. The nata de soya membrane is then soaked in the solution for 30 minutes so that the electrolyte ions can be absorbed and distributed into the membrane pore structure. After soaking, the membrane is dried at 75 °C for 15 minutes to reduce the water content while keeping the pores open, thereby supporting increased ionic conductivity when the membrane is used as a separator.

In the capacitor system, the nata de soya membrane acts as a separator, where the soaking treatment using $(NH_4)_2SO_4$ solution is intended to improve the internal properties of the membrane, particularly in terms of ion absorption and ionic conductivity. Meanwhile, cyclic voltammetry (CV) testing was conducted using H_2SO_4 solution as the supporting electrolyte (external electrolyte) to obtain a stable and reproducible electrochemical response during the measurement process.

2.3. Nata de Soy Electrolyte Membranes Characterization

The synthesized nata de soya membrane was tested using electrochemical impedance spectroscopy (EIS). EIS also measures ionic conductivity and resistance, and cyclic voltammetry (CV) to measure energy storage efficiency and specific capacitance.. The specific capacitance value was obtained from the area under the CV curve, indicating that the greater the membrane's capacity to store electrical charge, the higher the specific capacitance value.

$$S_c = \frac{\int_{v_1}^{v_2} i(V)dV}{mk\Delta V} \quad (1)$$

Where $\int_{v_1}^{v_2} i(V)dV$ representing the area under the closed CV curve where the voltage limits V1 and V2 are lower and higher, m represents the mass of the active separator material, k represents the scan rate in the CV curve, and ΔV is the potential window[9].

Testing the ionic conductivity of the soy milk membrane assesses its ability to conduct ions during the charging and discharging process. Membranes with high conductivity will reduce ionic resistance, allowing the capacitor to perform optimally. Ionic conductivity was calculated using the following formula:

$$\sigma = \frac{L}{A\ .\ Rs} \quad (2)$$

Where L is the thickness of the sample, A is the cross-sectional area of the sample, and Rs is the resistance of the solution[10].

Warburg impedance is used to evaluate ion diffusion resistance in Nata de Soya membranes, where lower Warburg values indicate faster and more efficient ion diffusion and charge transfer within the capacitor system.

$$Zw = \sigma\,(1 - j)\frac{1}{\sqrt{w}} \quad (3)$$

Where Zw is Warburg Impedance, σ is the Warburg coefficient from the linear regression plot of Z' vs $\frac{1}{\sqrt{w}}$, j is the imaginary number, 1 is the real number, and $\frac{1}{\sqrt{w}}$ is the angular frequency at 45° [11].

3 Results and Discussion

3.1. Cylic Voltammetry Analysis

The electrochemical properties in this study can be determined using a Metrohm Dropsens + EIS 0.01 MHz device at a frequency range of 10 kHz to 0.1 Hz with the following test configuration: nata de soya membrane is used as a separator with a size adjusted to the active electrode area. Cyclic voltamograms were recorded in H_2SO_4 (1.0 M) solution in the potential range of -0.15 to +0.15 V through Cyclic Voltammetry and Electrochemical Impedance Spectroscopy tests.

Cyclic voltammetry (CV) testing and measurement were conducted to evaluate the electrochemical properties and calculate the specific capacitance value of the $(NH_4)_2SO_4$ Nata de Soya electrolyte membrane in the samples produced. Figure 1 shows the CV curve on the Ammonium Sulfate $(NH_4)_2SO_4$ Nata de Soya electrolyte membrane separator at concentrations of 22.5%, 25%, 27.5%, and 30% after 30 minutes of immersion. Figure shows the CV curve at various scanning rates of 10, 20, 30, 40, and 50 mV/s. It can be seen that all CV curves have an almost identical shape, resembling a symmetrical ellipse without sharp redox peaks, indicating that the dominant charge storage mechanism is electric double-layer capacitance. The CV curves of $(NH_4)_2SO_4$ 30% 30 minutes form consistent curves that almost overlap each other, indicating that the electrode is able to maintain its capacitance stability even as the scanning rate increases.

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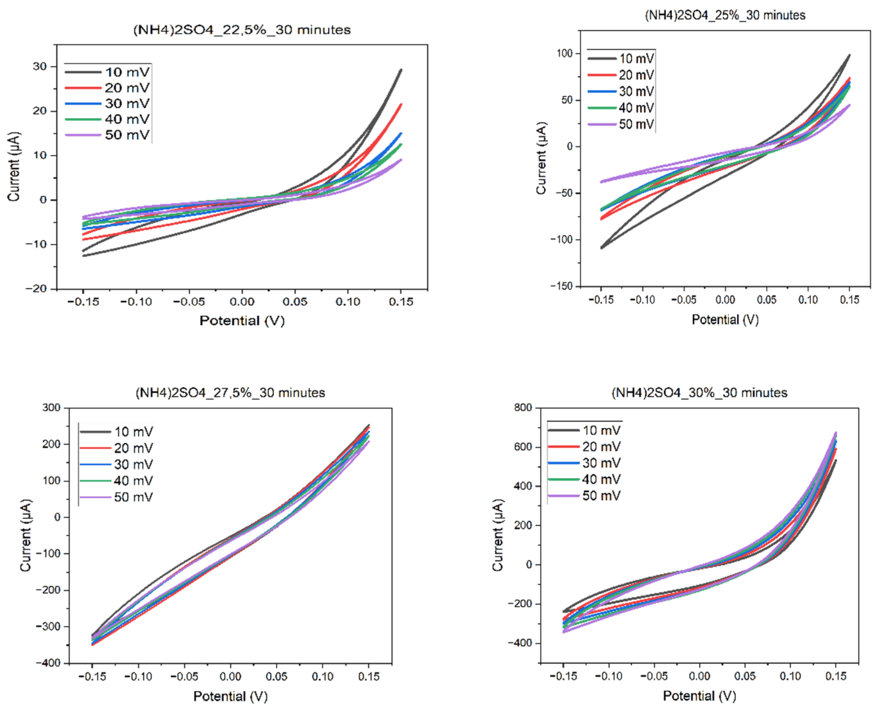


Fig. 1. Cyclic Voltammetry Separator Electrolyte Membrane $(NH_4)_2SO_4$ Nata de Soya

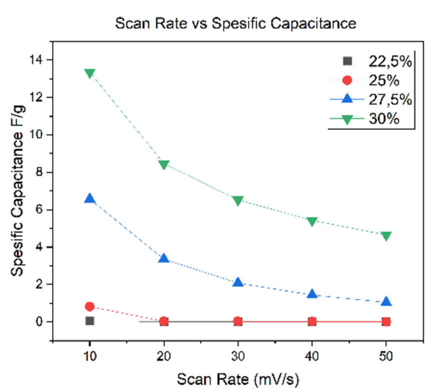


Fig. 2. Specific Capacitance vs. Scan Rate Curve of Electrolyte Membrane Separator $(NH_4)_2SO_4$

As shown in Fig. 2, the specific capacitance (Csp) of the Nata de Soya separator decreases with increasing scan rate from 10 to 50 mV/s for all electrolyte concentrations, with the highest values obtained at 10 mV/s due to sufficient ion diffusion time. The capacitance increases with higher $(NH_4)_2SO_4$ concentration, reaching a maximum of 13.35 F/g for the 30% sample at 10 mV/s. The large variation in capacitance among samples suggests that charge storage performance is influenced by electrolyte uptake and ionic transport within the separator. Furthermore, the steep increase in capacitance may be affected by normalization to the relatively small membrane mass; therefore, the values are mainly interpreted to compare performance trends rather than absolute capacitance.

3.2. Electrochemical Impedance Spectroscopy Analysis

Electrochemical Impedance Spectroscopy (EIS) testing was conducted on $(NH_4)_2SO_4$ Nata de Soya electrolyte membranes with varying concentrations of 22.5%; 25%; 27.5%; and 30% at a soaking time of 30 minutes. The EIS results included Nyquist plots, ionic conductivity, and warburg impedance. The resulting Nyquist curves were generally semicircular in shape with a diameter representing the charge transfer resistance (Rct), while the intersection point with the Z' axis indicated the solution resistance (Rs). In Figure 3, an increase in electrolyte concentration results in a smaller circle diameter, indicating decreased resistance and increased charge transfer efficiency. Thus, higher electrolyte concentrations tend to result in lower total resistance.

Figure 4 (a) shows that the membrane with a concentration of 22.5% has the highest Warburg impedance value, which is 1492.831 Ω. This value indicates a large ion diffusion resistance, which means that the membrane structure at that concentration is not yet able to support optimal ion movement. At higher concentrations, the Warburg impedance value decreases significantly to 97.58544 Ω at 25%, then increases slightly at 27.5% to 110.4619 Ω and increases again at a concentration of 30% to 187.5617 Ω.

The decrease in Warburg impedance at medium concentrations indicates an increase in ion diffusion efficiency within the membrane, while the increase at higher concentrations shows that an increase in electrolyte concentration is not always followed by improved ion transport, which is likely influenced by changes in pore structure or electrolyte saturation within the membrane.

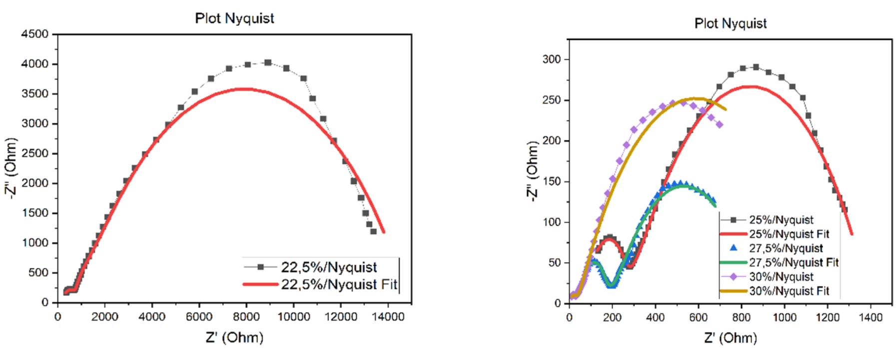
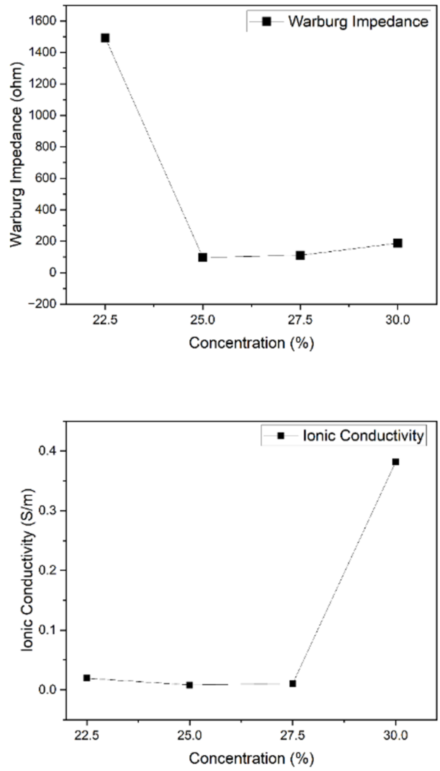


Fig. 3. Nyquist plots for 22.5%; 25%; 27.5%; and 30% at 30 minutes

The Electrochemical Impedance Spectroscopy (EIS) results show that the ionic conductivity (σ) of the Nata de Soya membrane does not increase linearly with the concentration of the $(NH_4)_2SO_4$ soaking solution. As shown in Fig. 4 (b), at a soaking time of 30 minutes, the ionic conductivity generally tends to be higher at increased electrolyte concentrations and reaches its maximum value of 0.38176 S/m at a concentration of 30%. However, intermediate concentrations of 27.5% and 25% exhibit lower conductivity values compared to both higher and lower concentrations, indicating that ionic transport is not governed solely by electrolyte concentration. This behavior may be related to differences in ion distribution within the membrane pores, partial pore blockage, or changes in membrane microstructure during the soaking process.



(a) (b)

Fig. 4. (a) Warburg Impedance and (b) Ionic conductivity of Nata de Soya membrane as a function of $(NH_4)_2SO_4$ concentration

3.3. Comparison with Previous Research

Based on previous research, various types of separator materials have been developed for supercapacitor applications with diverse ionic conductivity and specific capacitance characteristics. Polypropylene/PNIPAM-based separators exhibit a specific capacitance of 0.36 F/g at 25 °C[12], while PVDF separators have a value of 39.5 F/g[13]. Recent studies report that electrospun PVDF-based separator membranes can achieve ionic conductivity values of up to 1.5 S/m, attributed to enhanced electrolyte uptake and interconnected porous structures that facilitate efficient ion transport [14]. On the other hand, the solid polymer electrolyte PEGDGE/TETA/EMIBF has a lower conductivity of 0.02 S/m [15]. When compared to the results of this study, the nata de soya membrane shows an ionic conductivity of 0.38 S/m and a specific capacitance of 13.35 F/g, which are competitive values among polymer-based separators and other natural materials.

4 Conclusion

This study explored the electrochemical behavior of Nata de Soya membranes soaked in $(NH_4)_2SO_4$ solutions with concentrations ranging from 22.5%, 25%, 27.5%, and 30% for 30 minutes. The membrane treated with a 30% solution shows the most favorable performance among the samples, with a specific capacitance of 13.35267 F/g and an ionic conductivity of 0.38176 S/m. In contrast, the highest Warburg impedance, reflecting strong ion diffusion resistance, is observed for the 22.5% sample. Overall, these findings indicate that electrolyte concentration influences ionic transport in the membrane and highlight the potential of Nata de Soya as a promising separator material for further capacitor applications.

To improve causal interpretation and enable more reliable performance benchmarking, future studies should incorporate control samples, such as untreated or non-soaked Nata de Soya membranes, as well as commercial separator materials. The presenc e of these controls would allow a clearer assessment of the specific impact of electrolyte soaking and facilitate a more meaningful comparison between Nata de Soya membranes and established separator materials used in capacitor applications.

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