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MODIFICATION OF NANO CHITOSAN AND POLYURETHANE ON THE THICKNESS AND THERMAL STABILITY OF LITHIUM ION BATTERY SEPARATORS

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ABSTRACT

This study aims to modify lithium-ion battery separators using a combination of polypropylene (PP), polyurethane (PU), and nano chitosan, and to evaluate the effects of varying composition ratios and soaking time on thickness and thermal stability. The separators were fabricated using the casting method with variations in the PP:PU:Nano Chitosan ratio (4:1:5, 4:2:4, 4:3:3, 4:4:2, and 4:5:1) and soaking times of 1–5 days. Characterization included thickness (Digital Thickness Gauge), thermal stability (thermal shrinkage test and TGA), and surface morphology (SEM). The results showed that a 4:3:3 ratio with 3 days of soaking produced the best performance, with a thickness of 23–25 µm, low thermal shrinkage (0.5%–2.1%), degradation onset at 370.10 °C, degradation peak at 429.73 °C, and a dense and uniform morphology with well-distributed pores. This formulation is recommended as a potential separator for lithium-ion battery applications.

Keywords: Battery, Lithium-ion, Nano Chitosan, Polypropylene, Polyurethane, Separator

1. INTRODUCTION

1.1 Background

Lithium-ion batteries (LIBs) are one of the widely used energy storage technologies in modern life because they have good cycle performance, high energy density, and low self-discharge rates. One important component in LIBs is the separator, which serves as a physical barrier between the cathode and anode to prevent short circuits while also facilitating the movement of lithium ions during the battery charging and discharging processes (Perdana, 2021).

Commercial separators are generally made from polymers such as polyethylene (PE), polypropylene (PP), and polyurethane (PU). PU has good dielectric and mechanical properties as well as low cost, but it is weak in thermal stability and electrolyte compatibility (Cheng et al., 2022). Meanwhile, PP has low polarity and limited electrolyte absorption. This can reduce the risk of electrolyte leakage, but it also causes thermal shrinkage at high temperatures, which can compromise the performance of the separator (Arifin, 2017).

To overcome these limitations, the separator was modified by adding functional fillers such as nano chitosan. Nano chitosan has a structure that allows the formation of uniform pores, improves thermal stability, and supports better ion transfer. Therefore, this study modified PP- and PU-based separators by adding nano chitosan using the casting method. The main objective of this study is to examine the effect of soaking time and material composition variations on the thickness and thermal stability of the separator, as well as to analyze the surface morphology using a Scanning Electron Microscope (SEM).

2. RESEARCH METHODS

Research methodology

2.1 Research Place

This research was conducted in the Basic Chemistry and Analytical Chemistry Laboratory of the Chemical Engineering Department at Lhokseumawe State Polytechnic.

2.1 Tools and Materials

2.2.1 Tools used

Equipment used in this research includes beaker glass, measuring pipette, measuring glass, spatula, hot plate and magnetic stirrer, ITO glass, casting equipment, vacuum oven, optical tissues, ultrasonic device, analytical balance, digital thickness gauge, a set of Thermogravimetric Analysis (TGA) equipment, as well as a set of Scanning Electron Microscope (SEM) equipment.

2.2.2 Materials used

The ingredients used in this research include polypropylene (PP), distilled water, acetic acid, polyurethane (PU), and nano chitosan.

2.3 Experimental Treatment Design

2.3.1 Fixed Variables

- Synthesis Time: 2 Hours
- Synthesis Temperature: 70°C
- Drying Temperature: 40°C
- Drying Time: 6 Hours
- Polypropylene Weight: 4 grams

2.3.2 Independent Variables

- Polyurethane Weight : Nano chitosan : (1:5, 2:4, 3:3, 4:2 and 5:1) grams
- Soaking Time : (1, 2, 3, 4 and 5) Days

2.3.3 Dependent Variable

1. Lithium Ion Battery Separator Thickness Test
2. Thermal Shrinkage Test
3. Thermal Resistance Test (TGA)
4. SEM Test (Scanning Electron Microscopy)

2.4 Experimental and Testing Procedures

2.4.1 Synthesis Process of PP/PU/Nano Chitosan Separator Film

1. Weigh 4 grams of polypropylene (PP) and variations of polyurethane with Nanokitosan weights: (1:5, 2:4, 3:3, 4:2, and 5:1) grams.
2. In a glass beaker, add the PP and dissolve it at a temperature of 195°C.
3. Add the Nanokitosan solution and polyurethane to the PP solution. Stir until all components are evenly mixed.
4. Place the solution mixture on a hot plate and set the temperature to 70°C.
5. Stir the solution using a magnetic stirrer for 2 hours to achieve good homogenization.
6. After stirring, pour the solution onto the surface of the ITO glass, cover it with a smooth ITO glass on top to form a thin film layer (casting).
7. Let the mixture harden and form a thin film. Once the film is formed, immerse the layer in deionized water (aquadest) with immersion variations from 1 to 5 days.
8. After soaking, place the mold containing the solution in an oven at 40°C to dry the separator film for 6 hours.
9. Once dry, remove the separator film from the mold and it is ready for testing.

3. RESULTS AND DISCUSSION

3.1 Research Results

Table 3.1 Data from Test Results and Observation Analysis

Soaking Time (Days)	Sample PP:PU:Nano Kitosan (gram)	Massa Awal (g)	Massa Akhir (g)	Thermal Shrinkage Test (%)	Thickness Test (µm)
1	4:1:5	0,259	0,257	0,77	29
	4:2:4	0,254	0,252	0,78	25
	4:3:3	0,250	0,248	0,80	23
	4:4:2	0,246	0,244	0,81	23
	4:5:1	0,242	0,239	1,23	21
2	4:1:5	0,256	0,252	1,56	29
	4:2:4	0,253	0,250	1,18	25
	4:3:3	0,249	0,246	1,20	23
	4:4:2	0,245	0,242	1,22	22
	4:5:1	0,241	0,237	1,65	20
3	4:1:5	0,255	0,250	1,80	28
	4:2:4	0,251	0,247	1,59	26
	4:3:3	0,24	0,24	1,61	23

Soaking Time (Days)	Sample PP:PU:Nano Kitosan (gram)	Massa Awal (g)	Massa Akhir (g)	Thermal Shrinkage Test (%)	Thickness Test (µm)
		8	4		
	4:4:2	0,244	0,240	1,63	21
	4:5:1	0,240	0,234	2,50	20
4	4:1:5	0,258	0,252	2,32	28
	4:2:4	0,252	0,247	1,98	26
	4:3:3	0,247	0,242	2,02	23
	4:4:2	0,243	0,238	2,05	22
	4:5:1	0,243	0,236	2,88	20
5	4:1:5	0,257	0,249	3,11	27
	4:2:4	0,253	0,246	2,76	26
	4:3:3	0,248	0,242	2,41	23
	4:4:2	0,244	0,238	2,45	21
	4:5:1	0,241	0,232	3,73	21

3.2 Discussion

From the research results on lithium-ion battery separators using Polypropylene, Nanocellulose, and Polyurethane. Polypropylene serves as the main material for making lithium-ion battery separators. Polyurethane functions as a flexible binder material that can enhance the elasticity and mechanical strength of the separator. Meanwhile, nano chitosan acts as a functional filler that can improve the porosity and homogeneity of the film structure.

3.2.1 The Effect of Soaking Time on the Thickness of Lithium-Ion Battery Separators

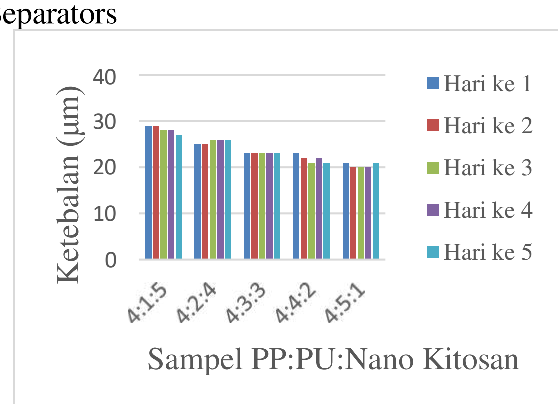


Figure 3.1 Thickness Graph (µm) Based on Sample Ratio by Soaking Days

After the vacuum drying process, a thickness test was conducted to evaluate the dimensional stability of the separator. Measurements on 25 samples with variations in the PP:PU:Nano Chitosan ratio and soaking times of 1–5 days showed that the composition significantly affected the film thickness. Thickness increased in ratios with high nano chitosan content (4:1:5 and 4:2:4) with values of 27–30 µm and 25–27 µm, exceeding the commercial separator standard (<25 µm). The 4:3:3 ratio produced a thickness of 23–25 µm, closest to the standard, indicating dimensional stability and uniform structure. Conversely, the 4:4:2 and 4:5:1 ratios resulted in thin films (21.5–23.5 µm and 20–22 µm), which could reduce mechanical strength. Thus, the 4:3:3 ratio was considered the most optimal in meeting the lithium-ion battery separator thickness criteria.

3.2.2. The Effect of Composition Ratio on the Thermal Stability of the Separator

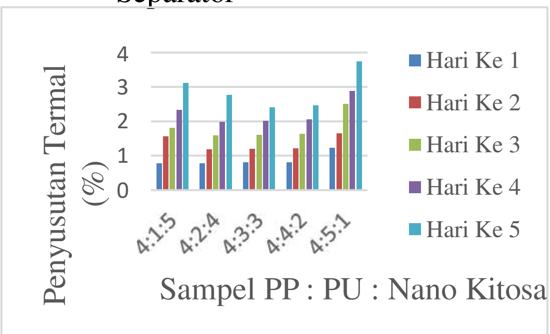


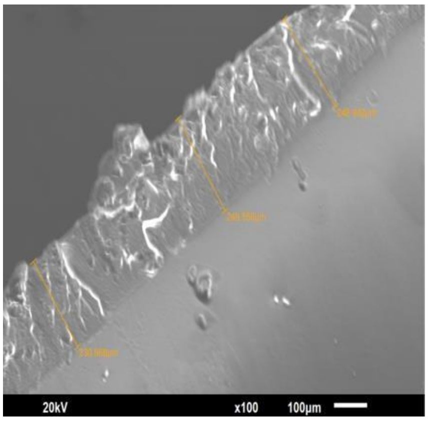
Figure 3.2 Graph of the Effect of Soaking Time on Thermal Shrinkage

Thermal shrinkage in lithium-ion battery (LIB) separators occurs due to exposure to high temperatures, which can trigger dimensional changes and potentially reduce the structural integrity of the separator. The thermal stability of the separator was evaluated through thermal shrinkage testing at 40 °C for 6 hours. Based on the test results on 25 samples, all compositions showed shrinkage of less than 5%, thus still meeting LIB separator safety standards.

The 4:3:3 ratio (PP:PU:Nano Chitosan) produces the lowest shrinkage results (±0.5–2.1%) and remains stable throughout the soaking period, indicating high resistance to thermal deformation. In contrast, the 4:1:5 and 4:2:4 ratios experienced shrinkage of up to 2.8%, while the 4:5:1 ratio reached 3.3%, close to the standard limit. This suggests that an excess of nano chitosan tends to increase dimensional instability due to its hydrophilic properties, whereas an excess of PU reduces resistance to thermal deformation. Therefore, the 4:3:3 ratio is considered the most optimal in maintaining the thermal stability of the separator.

The selected sample (4:3:3, soaked for 3 days) was then tested using Thermogravimetric Analysis (TGA). The test results showed initial degradation at 211.06 °C, onset at 370.10 °C, mid-point at 429.73 °C, and endset at 502.41 °C with a mass loss of 27.78%. This confirms that the PP/PU/Nano Chitosan-based separator has good thermal stability, capable of withstanding temperatures up to 500 °C, and is highly suitable for LIB applications operating at temperatures below 100 °C.

3.2.3. Surface Morphology Characteristics of Separators Based on Scanning Electron Microscope (SEM)



Picture. 3.3 SEM Test Results of PP, PU, and Nano Chitosan 4:3:3

SEM testing was conducted on the best sample with a 4:3:3 ratio to observe the surface morphology of the separator. The observation results showed an uneven surface texture due to phase imbalance during the casting process, with relatively large and evenly distributed pores. Although the dispersion of chitosan and PU in the PP matrix has not fully met commercial separator standards, the resulting morphology still supports the application of lithium-ion battery separators through a combination of good mechanical strength and sufficient porosity for ion diffusion.

4. CONCLUSION

4.1 Conclusion

Based on the results of the research that has been carried out, the following conclusions can be drawn:

1. The composition of Polypropylene (PP), Nano Chitosan, and Polyurethane (PU) as well as the soaking time affect the physical and thermal properties of lithium-ion battery separators. The optimal ratio was obtained at 4:3:3 with a soaking time of 3 days, resulting in a thickness of 23–25 µm and the lowest thermal shrinkage values of 0.5%–2.1%. Furthermore, the Thermogravimetric Analysis (TGA) results showed a degradation onset temperature of 370.10 °C and a degradation peak at 429.73 °C, indicating that this composition has good thermal stability and meets the criteria for lithium-ion battery separators.
2. The surface morphology characteristics of the separator from SEM analysis at a 4:3:3 ratio show that the film surface structure has an uneven texture due to phase imbalance during casting. The pore sizes are still large and evenly distributed across the entire film surface.

4.2 Suggestions

Based on the conclusions drawn, the following suggestions are given:
It is recommended to perform electrochemical testing, such as ionic conductivity and electrolyte absorption, to directly evaluate the performance of the separator in a lithium-ion battery cell.

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