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Enhanced Performance of Solid Polymer Electrolyte Separator Lithium Battery with Cellulose Acetate From Empty Palm Fruit Bunch Coated Al_2O_3-Polyacrylic Acid

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Abstract

As lithium battery technology improves, it becomes more important to have solid polymer electrolyte dividers that work better. The objective of this study is to enhance the efficiency of solid polymer electrolyte separators in lithium batteries. This research aims to expand the limits of innovation in hybrid separator development by utilizing empty palm fruit bunches (OPFEB) as a plentiful source of cellulose acetate. This approach enhances ion transfer by increasing the number of pores in the separator. However, there are challenges to achieving the desired levels of optimal ionic conductivity. In order to address these constraints, this study presents a novel Al_2O_3*-PAA inert ceramic oxide coating treatment that is applied to the separator by a spin coating technique. An electron microscope was utilized to observe the pore structure of the separator. Additionally, the separator underwent physical, mechanical, thermal, and cyclic voltammetry tests. The findings of this research indicate a significant increase in the physical properties, particularly the porosity and mechanical strength. The thermal shrinkage of the* Al_2O_3*-PAA coated separator is below 10% when exposed to a temperature of 140 °C for 30 minutes. The Cyclic Voltammetry test results demonstrate a pronounced loop curve, indicating an improvement in the ionic conductivity of the* Al_2O_3*-PAA coated separator. The findings of this study provide a method to enhance the efficiency of separator performance at high temperatures while maintaining safety and long battery life.*

Keywords: *Solid Polymer Electrolyte; Lithium Battery Efficiency; Cellulose Acetate; Empty Palm Fruit Bunch;* Al_2O_3*-Polyacrylic Acid*

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INTRODUCTION

The advent of lithium-ion (Li-ion) batteries has revolutionized energy storage, offering high performance and efficiency. Despite this, challenges in heat management critically impact battery dependability, safety, and durability [1-5]. The importance of battery temperature control for safety and performance has spurred numerous experiments and simulations to study battery pack temperature evolution [6]. While widely used, traditional polyolefin separators suffer from poor heat shrinkage and non-uniform wetting behavior.

In response to these challenges, Cellulose Acetate (CA)-based separators have emerged as a solution, addressing the limitations of traditional separators and enhancing lithium-ion battery performance. CA separators are known for their thermal qualities, conductivity, sustainability, adaptability, balance safety, efficiency, and performance needs [7-8]. Additionally, a global push for performance, safety, and sustainability in contemporary technologies is fueling the surge in research on cellulose-based lithium battery separators. Extensive research has been conducted on various cellulose sources, including natural (cotton, wood, bacteria) and regenerated (acetate, Lyocell fiber), to optimize separator properties [9-10].

Cellulose-based separators have shown promise in improving the mechanical strength of electrode materials and providing excellent wettability to electrolyte solutions [11]. The research on cellulose-based lithium battery separators is to innovate by developing a hybrid separator using empty palm fruit bunches (OPFEB) as a rich source of cellulose acetate. The use of OPFEB for cellulose-based lithium battery separators is a promising area of research. OPFEB, a readily available cellulosic biomass from palm processing facilities, contains a high percentage of alpha-cellulose, making it a potential source for cellulose production. The high alpha-cellulose content of OPFEB makes it a viable candidate for the development of cellulose-based separators for lithium batteries, contributing to both sustainability and performance [12-14]. This approach not only utilizes a sustainable resource but also aims to improve separator properties. Traditional cellulose separators are known to enhance ion transfer through the introduction of pores; however, they face limitations in achieving optimal ionic conductivity values[15].

To overcome these limitations, the research introduces a novel Al_2O_3. The research introduces a novel Al_2O_3-coated cellulose-based composite separator to overcome the limitations of traditional cellulose separators in achieving optimal ionic conductivity values. This approach has been shown to enhance the long-term operation of batteries, with the separator enabling high capacity retention over multiple cycles. Ceramic particle-based coating layers have been added to polyolefin-based separators to enhance their thermal stability and conductivity, thereby improving the safety and performance of lithium-ion batteries. These enhancements are often achieved with ceramic coatings. Ceramic particle-based coating layers are added to polyolefin-based separators because they have lower thermal conductivity than cathodes, anodes, or current tabs. Polyolefin separator thermal stability and conductivity are often improved by this coating [16-17].

The study introduces cellulose composite separators that are coated with Al_2O_3. These improve the long-term performance and ionic conductivity of lithium-ion batteries [18]. This ceramic coating improves polyolefin separators' thermal stability and conductivity, increasing battery safety and efficiency [19-20]. The Al_2O_3 coating reduces temperature rise by 20% and offers better thermal stability, mechanical properties, and liquid absorption than commercial separators. It also improves resistance to thermal shrinkage, oxidation, and puncture, contributing to safer, more efficient batteries [21-23]. The research has focused on enhancing the performance of lithium-based batteries. Several studies have investigated the use of Al_2O_3 in combination with various binders to prepare composite separators [24-25]. For instance, a study selected inorganic materials, such as SiO_2, ZrO_2, and TiO_2 to improve its performance in high-power lithium-ion batteries [26-28].

Nevertheless, limitation of the Al_2O_3 coating on the separator is its feeble cross-linking within the polymer composition of the separator. This deficiency hinders the battery's ion transfer capabilities, consequently promoting dendrite growth[29]. Polyacrylic Acid (PAA) has been successful in suppressing lithium (Li) dendrite growth, indicating its potential for Li-metal coatings to prevent dendrite formation [30]. Presents a study on sulfur-carbon (S/C) composite cathodes for lithium-sulfur batteries, using a crosslinked water-soluble PAA binder. The research focuses on improving the electrochemical performance of these batteries, particularly in terms of discharge capacity, cycling stability, and thermal stability [31-33]. Furthermore, partially neutralized PAA has been found to be an efficient binder for aqueous ceramic-coated separators in lithium-ion batteries, demonstrating its utility in battery technology [34]. Studies have demonstrated that incorporating cross-linked poly (acrylic acid) into Li-ion batteries enhances adhesion and electrochemical performance. This suggests that the use of PAA could potentially enhance the overall performance and durability of batteries [35].

In this study, cellulose acetate derived from an eco-friendly source, OPFEB, was utilized. However, this material has limitations in terms of conductivity and thermal shrinkage. To solve this problem, cellulose acetate from OPFEB will have Al_2O_3-PAA coated on it to create a separator. The aim is to obtain a separator with excellent ionic conductivity and thermal shrinkage values while also considering other properties such as mechanical and physical characteristics that are essential for a high-quality separator.

METHOD

Material

The materials employed in this investigation encompassed Empty Palm Oil Bunches (OPEFB), Sodium hydroxide (NaOH), sodium hypochlorite (NaOCl), sulfuric acid (H_2SO_4), deionized water (Aquades), glacial acetic acid, acetic anhydride, sodium acetate, aluminum oxide (Al_2O_3), polyacrylic acid (PAA), N, N-dimethylacetamide (DMAc), and polyvinylidene fluoride (PVDF). The equipment utilized in this research comprised a Grinding Machine, a 60 Mesh sieve, a three-neck flask, a hot plate, a desiccator, a digital balance, a dropping pipette, a Magnetic Stirrer, a Spin Coating apparatus, a Casting knife, a stirring rod, a measuring cup, an oven, an ultrasonication device, a glass substrate, a coagulation bath, a distilled water bath, and a petri dish.

Procedure for Making Cellulose Acetate

The procedure for producing cellulose acetate involves washing and subsequently drying the Oil Palm Empty Fruit Bunches (OPEFB). The dried OPEFB is then chopped into sizes ranging from 5 to 10 cm, ensuring that the moisture level does not exceed 10 %. The dried oil palm empty fruit bunches (OPEFB) were subjected to grinding using a grinding machine and afterwards sieved using a 60-mesh sieve [36]. A mixture consisting of 25 grams of oil palm empty fruit bunch (OPEFB) powder and a 2 % sodium hydroxide (NaOH) solution (w/v) was subjected to reflux at a temperature of 90 °C for a duration of 3 hours. The reflux filtrate that had been subjected to filtration was afterwards subjected to a reaction with 72 % (v/v) H_2SO_4. After the completion of the filtration process, the remaining residue was subjected to a further reaction. This residue was refluxed at a temperature of 70 °C for a duration of one hour using a mixture consisting of 1 % NaOCl and 1 gram of NaOH, with a ratio of 100 : 1 (volume to weight). The purpose of this reflux was to facilitate the bleaching process. The combination underwent filtration, and the remaining solid was immersed in a solution of 100 mL of 17.5 % sodium hydroxide (NaOH) for a duration of 30 minutes [37-38]. The combination underwent a subsequent filtration process and was subjected to multiple washes with distilled water until it reached a neutral pH. Subsequently, the mixture was subjected to a drying process in an oven for a duration of 24 hours at a temperature of 40 °C. Subsequently, the cellulose that had been isolated was subjected to a further drying process, lasting for a duration of 1 hour, at a temperature of 50 °C [39,40]. The cellulose extract obtained from the oil palm empty fruit bunch (OPEFB) was subjected to a three-stage synthesis process. These stages included activation, acetylation, and hydrolysis. The water content, acetyl content, yield, and functional groups of the cellulose acetate were assessed using FTIR analysis.

Procedure for the Fabrication of Solid Polymer Electrolyte

The process starts by dissolving PVDF at a total concentration of 3 % (3 g in 100 ml of deionized water), then mechanically stirring while heating on a hot plate at 50 ^{o}C for two hours until homogeneous. Next, 0.5 g of titanum oxide (TiO_2) and 1 mL of dimethylacetamide (DMAc) were added as a crosslinker, then stirred mechanically while heating using a hot plate at a temperature of 50 ^{o}C for 2 hours until a homogeneous solution was obtained. The solution is stored in a syringe container for further molding. This solution is used for SPE1 samples, while SPE2 samples add cellulose acetate to a 3 % based solution.

The next step is to prepare the Al_2O_3-PAA solution; the deionized water is then stirred mechanically while heating using a hot plate at a temperature of 50 ^{o}C for 2 hours until a homogeneous solution is obtained. Approximately 6.0 % by weight of PAA was added to the solution until homogeneous Al_2O_3-PAA was obtained. The Al_2O_3-PAA solution is then prepared in a syringe to be dripped onto a glass substrate that has been cleaned and there are SPE1 and SPE2 membranes installed on the top of the spin coat unit, rotated at a speed of 3000 rpm for 30 seconds coated with an Al_2O_3-PAA solution with a concentration of 2% and 4% wt, the composition of the ingredients can be seen in Table 1.

Table 1. SPE Membrane Concentration

Sample	PVDF (%)	CA (%)	Solution Al_2O_3-PAA(%)
SPE1	100		-
SPE2	50	50	
SPE3	40	40	20
SPE4	30	30	40

The SPE membrane was tested for surface morphology using SEM (Scanning Electron Microscopy), CV (Cyclic-Voltametry), the tensile strength test, the porosity test, and the thickness test. A surface morphology test was conducted to observe the surface of the electrolyte polymer membrane using a Hitachi TEM 3000 with 3000x magnification. CV (Cyclic-Voltametry) is used to analyze membrane ionic conductivity values. The tensile strength test of the membrane is carried out to determine the ability of the membrane to withstand maximum stress (tension) when the membrane is loaded by the L&W Tensile Tester. The porosity test is carried out to determine the membrane's ability to absorb a solution. Porosity can also describe the membrane's hydrophilicity toward water. The thickness test is carried out to measure the physical thickness of the membrane. The thermal shrinkage test of the SPE membrane was conducted at room temperature and 140 °C for 30 minutes [41].

RESULTS AND DISCUSSION

Cellulose Acetate Test Results Using FTIR

The results of the analysis of the cellulose acetate functional group from EFB show an important absorption peak in the infrared spectrum which can be seen in Figure 1. Specifically, the ester group originating from the C-O acetyl group exhibits absorption peaks at 1156.72 and 1242.39 cm^{-1} within the range of 1050-1300 cm^{-1}. Additionally, the C=O carboxylic groups demonstrates an absorption peak at 1642.44 cm^{-1}., falling within the range of 1690-1675/1650-1600 cm^{-1}. The presence of an absorption peak at a wave number of 3336.50 cm^{-1} suggests the presence of the O-H functional group within the range of 3200-3600 cm^{-1}. Additionally, another absorption peak at 2897.48 cm^{-1} indicates the presence of the C-H functional group. Based on the results of the FTIR spectrum, the compounds contained in cellulose acetate are the functional groups C-O, C=O, O-H and C-H. The FTIR analysis data of cellulose acetate was compared with commercial cellulose acetate which can be seen in Table 2. The absorption peak intensity of the TKKS acetyl cellulose acetate group is in the commercial acetyl cellulose acetate group.

Spectrometry results show the most indicative band around 1600 cm^{-1}. Resonance phenomena in carboxylic groups (C=O), which are formed during acid ionization. This group is related to ion transfer in lithium batteries and is used in the design of lithium-ion battery materials. For example, carboxylic groups can be used in the manufacture of cellulose-based separators for lithium-ion batteries, where the separators can influence electrolyte performance and ion transfer in the battery [12]. Also, carboxylate groups are used to make solid electrolyte membranes out of cellulose for lithium-ion batteries. These groups can change how well the membranes conductions ions and how stable they are electrochemically. Therefore, C=O groups have an important role in the applications of lithium-ion batteries and related materials [42].

Table 2. Comparison of OPEFB Cellulose Acetate IR Spectrum Results with Commercial Cellulose Acetate

Frequency Wavenumber Commercial CA	Frequency Wavenumber OPFEB CA	Functional Groups	Group Frequency Wavenumber (cm^{-1})
3479.28 cm^{-1}	3336.50 cm^{-1}	O-H	3200-3600 cm^{-1}
2944.99 cm^{-1}	2897.48 cm^{-1}	C-H	2850-3000 cm^{-1}
1744.87 cm^{-1}	1642.44 cm^{-1}	C=O	1690-1675/1650-1600 cm^{-1}
1232.72 cm^{-1}	1156.72 and 1242.39 cm^{-1}	C-O	1050-1300 cm^{-1}

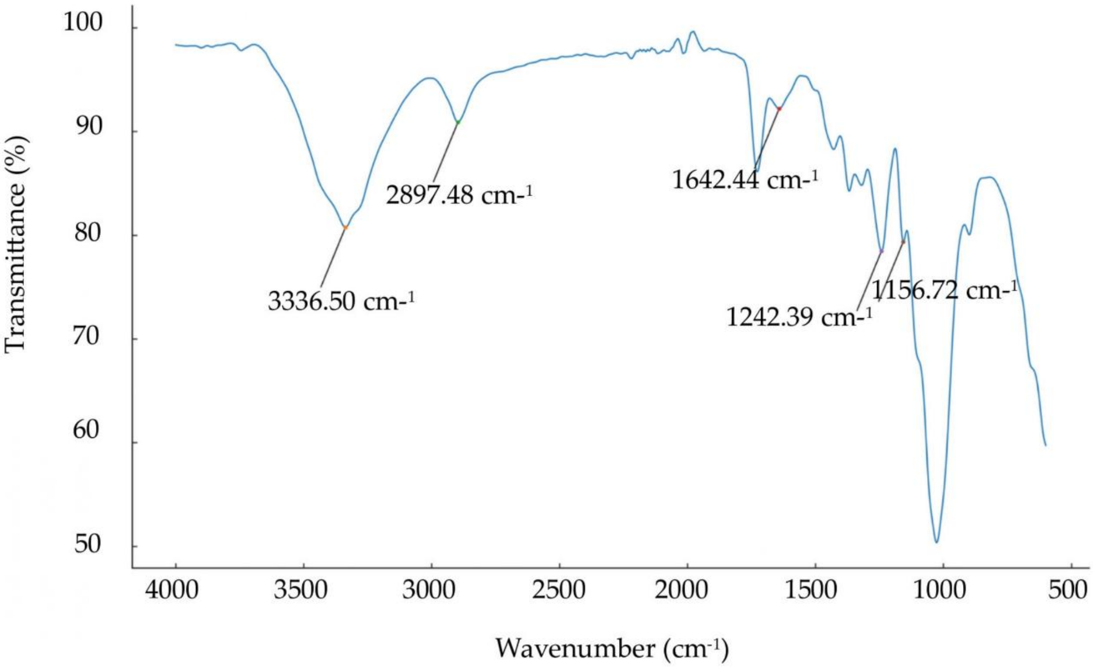

Figure 1. FTIR Spectra of Cellulose Acetate OPEFB

Test Results PSA (Particle Size Analyzer) of Cellulose Acetate Synthesis

The results of the cellulose acetate particle size test can be seen in Figure 2. Particle size can have an impact on the battery separator's absorption capacity. It is possible that larger particles show lower absorption capacity compared to smaller particles. Battery separators with a high adsorption capacity are considered a desirable characteristic because they allow for the maintenance of a sufficient amount of electrolyte to facilitate the electrochemical processes occurring within the battery. Cellulose acetate tested for particle dispersion showed an average particle size of 13.3922 μm. Utilization of materials to improve the performance of lithium battery separators generally involves manipulation of particle size, typically in the nanoscale range of 1-100 nanometers [29].

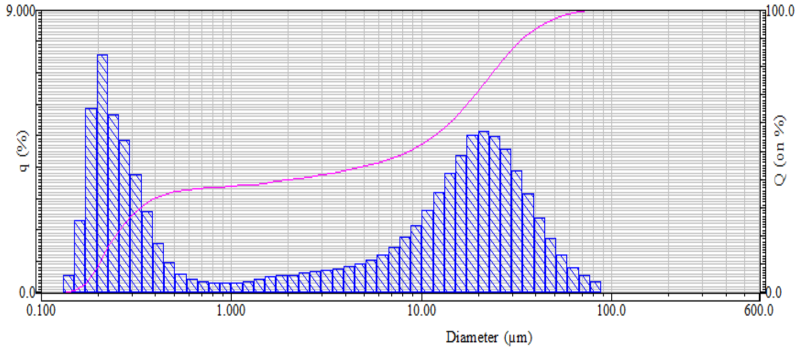

Figure 2. Particle Size Distribution of Cellulose Acetate OPFEB

Test Results CV (Cyclic Voltametry) of SPE

Cyclic Voltammetry (CV) testing is a common electrochemical inspection technique that is often used to examine and assess the electrochemical characteristics of various materials, including battery separators. The influence of CV loops on the electrical properties of battery separators was shown to yield important insights into the expected behavior of separators in battery applications. Cyclic voltammetry performed at a scan rate of 20 $mV \cdot s^{-1}$. The graph presented depicting the results of the CV test carried out on the membrane separator can be seen in Figure 3. The graph illustrates that the CV loop of Sample SPE4 is greater where this sample is a separator with Al_2O_3-PAA coated treatment. This indicates that the Al_2O_3-PAA coated treatment on SPE4, contributes to increasing the charge storage and release capacity during the electrochemical process. The electrochemical stability of the separator also improves ion transport and reduces resistance during charge-discharge cycles [43-44]. The Al_2O_3-PAA coated treatment can affect the ionization of surface carboxylic acid groups on the separator. As these groups are transformed to surface carboxylate groups by exposure to basic aqueous solutions, the surface becomes more hydrophilic, leading to changes in the free energy of the solid-liquid interface and the contact angle of the aqueous solution. Changes in surface hydrophilicity can influence the free energy of the solid-liquid interface. It can refer to the amount of energy required or released during the interaction between the separator surface and the electrolyte solution [45-46].

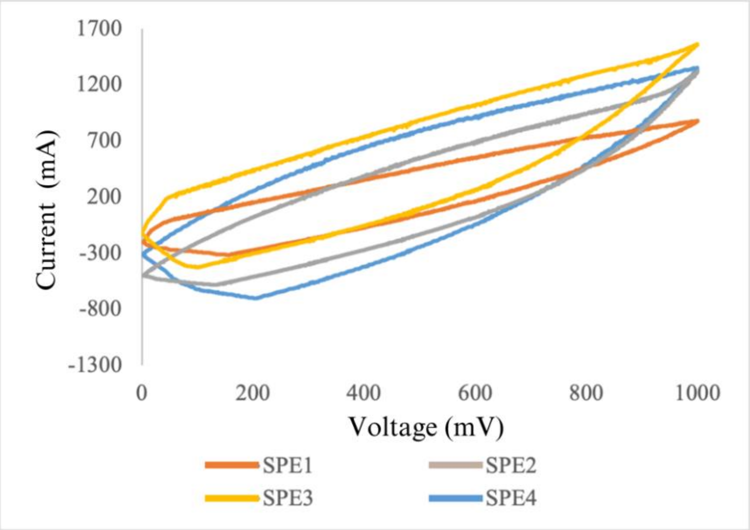

Figure 3. Cyclic Voltammetry Results of the SPE Membrane

Results of Porosity and Morphology Testing of Solid Polymer Electrolytes

The results of the investigation of the pore morphology of the SPE1 membrane without the addition of cellulose acetate revealed the existence of a compact top layer characterized by pores with irregular shapes, as seen in Figure 4(a), and the SPE2 sample consisting of PVDF/CA with an increased number of pores and a uniform appearance. Figure 4(b). The SPE2 membrane has a dense top layer, which is characterized by a higher level of pore uniformity, resulting in an increase in the number of pores of up to 71 % of the data in Figure 5 [35]. Figure 4(c) shows SPE4 with the Al_2O_3-PAA coating treatment, which has a larger pore surface area and shows a uniform pore size distribution as seen in the data in Figure 5.

For lithium-based battery dividers to move Li ions, they must have a certain amount of porosity. To keep the ion conductivity high, there should be enough liquid electrolyte caught in the separator's micropores and channels that connect to each other. Generally, dividers should have more than 40% porosity. Higher porosity leads to lower internal resistance and better electrolyte uptake, both of which are good for improving battery performance [47]. Increasing the porosity of a separator can increase its electrolyte absorption capability, thereby increasing its capacity to accommodate larger amounts of electrolyte. Facilitation of ion transport in lithium-ion batteries is an important aspect [48-49].

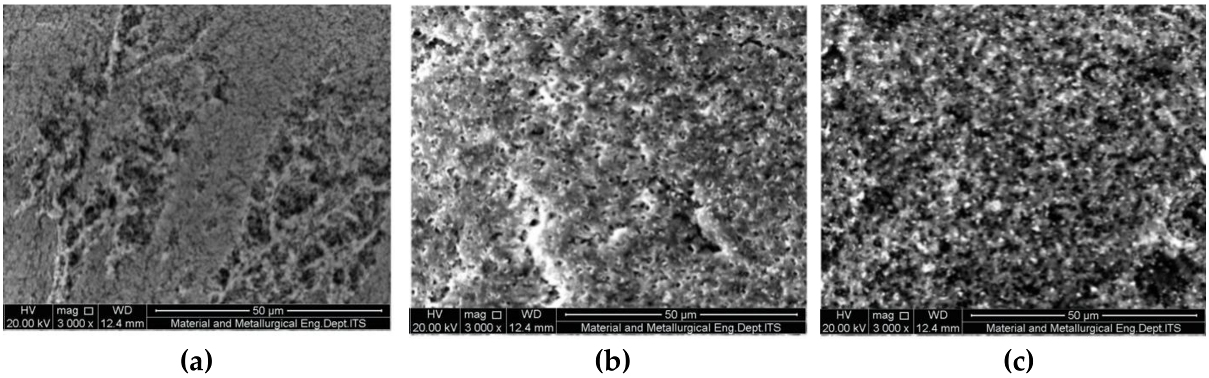

Figure 4. SEM Micrographs of SPE Membranes a) SPE1, b) SPE2, and c) SPE4, at 3000x Magnification

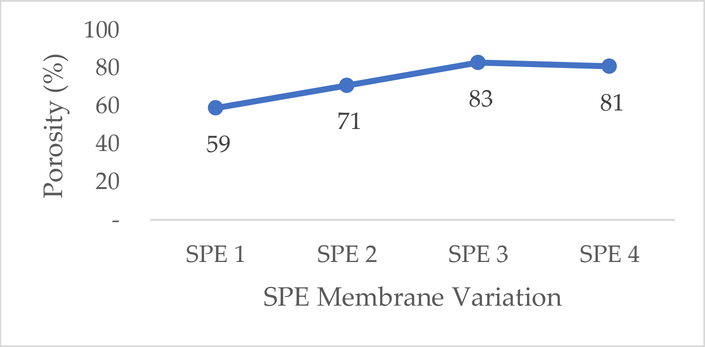

Figure 5. Porosity of SPE Membrane Variation

Thickness Test Results of SPE

The thickness test results for each sample obtained can be seen in Figure 6. The thickness of each Solid Polymer Electrolyte (SPE) membrane in Figure 6 was obtained with values between 30.5-39.3 μm. The thicker the membrane, the higher its tensile strength, the lower the elongation value and the lower the water vapor transmission speed. The thickness results of the membrane are still in the category of meeting the requirements based on the range of 25-50 μm[50].

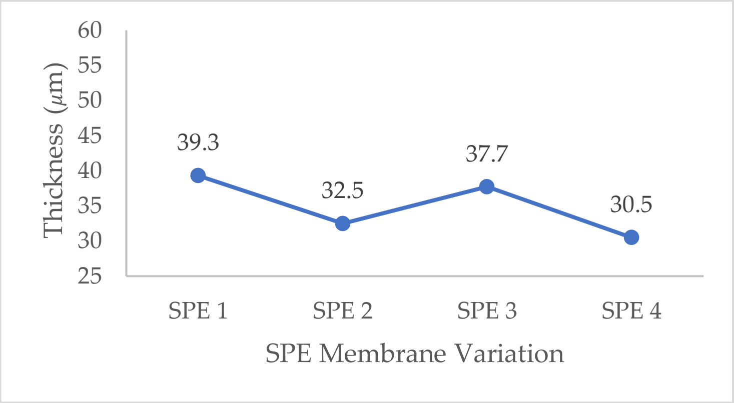

Figure 6. Thickness of SPE Membrane Variation

Tensile Strength Test Results of SPE

Tensile testing was carried out with an L&W Tensile Tester using ATSM D638-02a-2002. The tensile strength test results of the samples can be seen in Figure 7. The tensile test results show that the SPE4 sample, which received the Al_2O_3-PAA coating treatment, had the highest value. The Al_2O_3-PAA coating increases lithium-ion battery separator tensile strength. Al_2O_3-PAA coating enhances the tensile strength of lithium-ion battery separators through a synergistic effect. Aluminum oxide (Al_2O_3) provides mechanical strength, reducing the risk of separator damage. PAA contributes flexibility and adhesion, forming a protective layer that improves the overall mechanical integrity of the separator. The combined benefits of these materials result in increased tensile strength, making the coated separator more resilient to mechanical stress and enhancing the overall performance and safety of lithium-ion batteries [35].

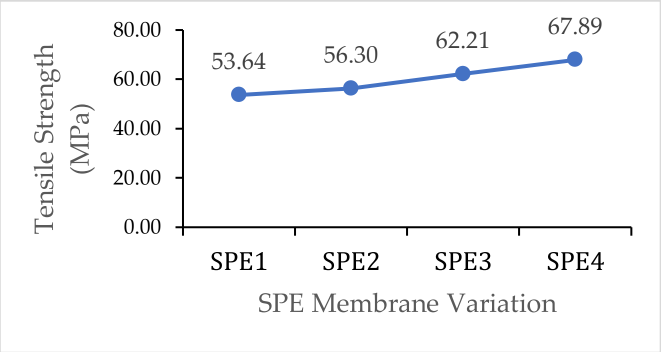

Figure 7. Tensile Strength of SPE Membrane Variation

Thermal Shrinkage Test Result of SPE

The Thermal Shrinkage Test Result for the Solid Polymer Electrolyte Separator Lithium Battery with Cellulose Acetate from Empty Palm Fruit Bunch Coated Al_2O_3-PAA provides valuable insights into the thermal stability and dimensional changes of the separator under varying temperature conditions. The test results can be seen in Table 3. The test results showed a significant increase in thermal shrinkage in sample SPE2, this can be caused by the amount of CA used in SPE2. Cellulose acetate has distinct thermal properties compared to PVDF. Its thermal expansion and contraction characteristics might differ, contributing to an overall increase in thermal shrinkage for SPE2. Another cause is the presence of water content in the added CA. The thickness test results also show that SPE2 also has a smaller value, which can also lead to greater thermal shrinkage. The presence of Al_2O_3-PAA in SPE4 may have optimized the structure and thermal stability of the separator, significantly reducing shrinkage. These results back up the idea that adding Al_2O_3-PAA can improve the thermal performance of the separator. This gives us a good reason to think about the composition when developing SPE in lithium batteries [51].

Table 3. Thermal Shrinkage **of** SPE Membrane Variation

SPE Membran	Thermal Shrinkage (%)
SPE1	23.2
SPE2	31.6
SPE3	7.3

SPE4	5.6

The results of our study were what we expected, but it's important to note that there are some problems. It's important to note that our research has only been used to explain the separator's properties, not for construction purposes. The improvements we've talked about here might be even better if we added separator compatibility tests with both anodes and cathodes to our analysis. This would give us a fuller picture of how they work in lithium battery systems. Furthermore, changes in the Al_2O_3-PAA coated need a deeper investigation covering a wider range of factors to find the best conditions. Also, the process of making cellulose acetate (CA) from empty oil palm bunches can lead to differences, which can change the general results. Because of this, it is important to be aware that our results may be different depending on how complicated the CA synthesis process is. This means that we need to think about the possible effects of the observed performance improvements in a different way.

CONCLUSION

The primary objective of this study is to create a lithium battery separator by utilizing cellulose acetate derived from empty palm fruit bunches. The research also entails a novel approach involving the application of an Al_2O_3-PAA hybrid coating. The purpose of applying cellulose acetate and Al_2O_3-PAA coatings is to enhance the thermal and mechanical characteristics of the separator. Testing revealed that the inclusion of cellulose acetate (CA) in SPE2 augmented the quantity of pores. The use of an Al_2O_3-PAA coating on SPE4 significantly improved the outcomes of both the cyclic voltammetry and thermal shrinkage tests. This improvement is expected to increase the ion conductivity and improve the heat resistance, hence boosting the efficiency and safety of lithium batteries.

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AUTHOR CONTRIBUTIONS

Delovita Ginting: conceptualization, supervising, validation, analysis, and writing. Fitra Perdana: Investigation, data collection, analysis, and writing of the original draft, Romi Fadli Syahputra: reviewing and editing the final manuscript; investigation; data collection; analysis; and writing of original draft. Noor Maizura Ismail: Investigation, analysis, and reviewing of the final manuscript.

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.

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