

A HIGH-PERFORMANCE LI-ION BATTERY SEPARATOR FABRICATED FROM PP/POLYURETHANE/NANO CHITOSAN COMPOSITE WITH ENHANCED THERMAL STABILITY AND HOMOGENEOUS MORPHOLOGY

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ABSTRACT

This study investigates the modification of lithium-ion battery (LIB) separators using polypropylene (PP), polyurethane (PU), and nano chitosan through a casting method, focusing on the effects of composition ratio and immersion duration on separator thickness and thermal stability. Nine sample variations with PP: PU: Nano Chitosan ratios of 4:2:4, 4:3:3, and 4:4:2 were prepared and immersed for 1–3 days. The separators were characterized using thickness measurements, thermal shrinkage analysis, Thermogravimetric Analysis (TGA), Fourier Transform Infrared Spectroscopy (FTIR), and Scanning Electron Microscopy (SEM). The 4:3:3 composition demonstrated the most optimal performance, producing separator thicknesses of 23–25 μm with the lowest thermal shrinkage (0.5–2.1%), meeting commercial LIB separator standards. TGA results showed that the PCS5 sample (4:3:3, 2-day immersion) exhibited superior thermal resistance, with a degradation onset of 370.10 $^{\circ}\text{C}$ and peak decomposition at 429.73 $^{\circ}\text{C}$. FTIR confirmed the successful incorporation of urethane and hydroxyl/amine functional groups, indicating chemical modification of the polymer matrix. SEM observations revealed a dense and uniform morphology with well-distributed pores, supporting enhanced ionic transport. Overall, the 4:3:3 formulation with a 2-day immersion significantly improves the physical, thermal, and structural properties of the separator, demonstrating strong potential for high-performance LIB applications and contributing to ongoing advancements in battery safety and efficiency.

Keywords: Lithium-Ion Battery, Separator, Polypropylene, Polyurethane, Nano Chitosan, Thermal Stability, Casting Method.

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INTRODUCTION

Lithium-ion batteries (LIBs) have been extensively utilised in portable electronic devices, electric vehicles, and energy-storage systems because of their high energy density, long cycling durability, and minimal self-discharge characteristics. The separator serves as a critical component in LIBs, acting as an insulating barrier that prevents physical contact between the electrodes while still allowing ions to move during the charge–discharge processes.¹ However, most commercial separators based on polyolefins such as polypropylene (PP) still exhibit limitations, including low electrolyte affinity, poor thermal stability, and significant shrinkage at elevated temperatures, which may compromise battery safety.² Recent studies have explored polymer blending and functional additives to improve separator performance, particularly in terms of porosity, thermal resistance, and mechanical stability.³ Polyurethane (PU) has been reported to enhance flexibility and compatibility in polymer matrices, while nano chitosan—an abundant biopolymer with polar functional groups—can act as a pore-forming agent that improves electrolyte wettability and structural homogeneity. Despite these advantages,

comprehensive studies involving the combined use of PP, PU, and nano chitosan in separator development remain limited. Specifically, the influence of composition ratio and immersion time on the structural, thermal, and morphological characteristics of the resulting membranes has not been extensively investigated. This gap forms the basis of the present research.⁴ The development of safer and more stable separators also aligns with global sustainability initiatives, particularly SDG 7 (Affordable and Clean Energy) and SDG 9 (Industry, Innovation, and Infrastructure), by supporting advancements in energy-storage materials with improved operational safety. The introduction of nano chitosan, derived from renewable biopolymer sources, additionally supports SDG 12 (Responsible Consumption and Production) through the utilization of environmentally friendly materials. In this study, LIB separators were fabricated using PP, PU, and nano chitosan through the casting method with variations in composition ratio and immersion time. The resulting membranes were evaluated for thickness, thermal shrinkage, thermal stability (TGA), chemical structure (FTIR), and surface morphology (SEM). This work aims to determine the optimal formulation capable of providing improved dimensional stability, thermal resistance, and chemical interactions suitable for LIB separator applications. Recent literature emphasises the importance of separator materials that maintain structural integrity under high-temperature conditions to ensure safe battery performance.⁵

EXPERIMENTAL

Material and Methods

Polypropylene (PP), polyurethane (PU), nano chitosan, acetic acid, and distilled water were used as the primary materials in this study. All materials were used as received. The PP/PU/nano chitosan composite was prepared by melting 4 g of PP at 195 °C until fully liquefied. PU and nano chitosan were added according to the composition ratios of 4:2:4, 4:3:3, and 4:4:2 (PP: PU: Nano Chitosan). The mixture was stirred continuously at 70 °C for 2 h to obtain a homogeneous polymer solution. Separator films were fabricated through the casting method by pouring the homogeneous mixture onto an ITO glass substrate and placing a second ITO glass plate on top to obtain a uniform film. The cast films were allowed to solidify at room temperature and subsequently immersed in distilled water for 2–4 days to remove residual solvents and promote pore development. After immersion, the films were dried at 40 °C for 6 h, detached from the substrate, and cut into the desired dimensions for further testing. Thermal shrinkage was measured by heating the separator samples at 100 °C for 1 h and recording the initial and final masses. TGA analysis was carried out from room temperature to 900 °C at a heating rate of 20 °C/min under a nitrogen atmosphere to determine the onset, peak degradation temperature, and mass-loss profile. SEM analysis was performed to observe the surface morphology, pore distribution, and material homogeneity of the separator films. FTIR spectra were recorded in the range of 500–4000 cm⁻¹ to identify functional groups and evaluate chemical interactions between PP, PU, and nano chitosan.

General Procedure

Separator Manufacturing Procedure

a. Preparation of PP/PU/Nano Chitosan Composite

Four grams of PP were melted at 195 °C, followed by the addition of PU and nano chitosan according to the selected composition ratios. The mixture was continuously stirred at 70°C for 2h until a homogenous polymer solution was obtained. This blended solution acted as the precursor for separator fabrication.

b. Fabrication of Separator Films (Casting Method)

The homogenous blend was poured onto an ITO substrate and covered with another clean ITO plate to form a thin film. The film was allowed to harden at ambient conditions and subsequently soaked in distilled water for 2–4 days to facilitate solvent removal and pore formation. The films were then dried at 40°C for 6h, removed from the substrate, and cut to the desired dimensions for further characterisation.

Detection Method

General requirements for separators for lithium-ion batteries

Parameters	Requirements	Description
Thickness (μm)	< 25	-
No MacMulin	< 8	The ratio of the separator's resistance when saturated with

		electrolyte relative to the resistance of the electrolyte alone.
Gurley	~ 25/mile	The duration needed for air to permeate through the separator.
Pore Size (μm)	< 1	-
Porosity (%)	~ 40	-
Leakage Strength (g/mil)	>300	-
Melt Integrity (oC)	>150	-
Chemical Stability	Sufficient time	-
Heat Stability	< 5% depreciation	-
Tensile Strength	< 2%	-
Slope	< 2	-

Source: ⁵

Casting Method Separator Manufacturing

After the stirring process, the polymer solution was poured onto an ITO glass substrate and covered with another ITO glass plate to form a thin film via the casting method. The film was allowed to solidify and then soaked in deionised water for 2–4 days to remove residual solvents and enhance pore formation. After soaking, the film was dried in an oven at 40 °C for 6 hours to remove remaining moisture and stabilize its structure. The dried separator was then detached from the mold, trimmed to the required size, and prepared for characterization.

Thermal Shrinkage Test

The thermal shrinkage test was performed to evaluate the separator's stability at elevated temperatures, a critical factor for lithium-ion battery safety. Samples were heated at 100 °C for 1 hour, and the dimensional change was recorded. According to battery safety standards, shrinkage should remain below 5%. Low shrinkage indicates good dimensional stability, reducing the risk of internal short circuits under thermal stress.

Thermal Stability (TGA) Test

Thermogravimetric Analysis (TGA) was conducted to measure mass loss during heating from room temperature to 900 °C at a rate of 20 °C/min under nitrogen. The sample, placed in an inert crucible, was monitored using a high-precision microbalance. The TGA curve provides the onset temperature, peak degradation temperature, and total mass loss, indicating the separator's thermal resistance and degradation behaviour.

Scanning Electron Microscope (SEM) Test

SEM was used to observe the surface morphology and film thickness of the PP/PU/Nano Chitosan separator. The technique scans the sample surface with a high-energy electron beam, and images are produced from detected secondary or backscattered electrons. SEM provides high-resolution visualisation (1–5nm), allowing detailed analysis of pore structure, uniformity, and surface characteristics.

Fourier Transform Infrared (FTIR) Spectroscopy Testing

FTIR spectroscopy was conducted in the range of 500–4000 cm^{-1} to identify functional groups and chemical interactions within the PP/PU/Nano Chitosan separator. Absorption peaks corresponding to –OH, –NH, C–H, C=O, and C–O–C vibrations were analysed. The spectra indicated the formation of urethane bonds from PU and functional contributions from nano chitosan, confirming successful chemical modification and integration of all components in the composite separator.

Analytical Discussion

SEM analysis revealed that the incorporation of nano chitosan increased pore density, while higher PU content improved surface smoothness and uniformity. The composition 4:3:3 yielded a balanced morphology with interconnected pores and good surface homogeneity, favourable for electrolyte uptake. FTIR spectra confirmed the presence of characteristic PP, PU, and chitosan functional groups. Shifts in

the –OH and –NH absorption bands indicated hydrogen bonding interactions and improved compatibility between PU and nano chitosan, supporting successful composite formation. TGA analysis showed that higher PU content increased the onset and peak degradation temperatures due to the thermal stability of urethane linkages, whereas higher chitosan content slightly reduced the onset temperature. All compositions, however, maintained thermal integrity above 150°C, meeting separator safety requirements. Thermal shrinkage remained below the critical limit of 5%, demonstrating good dimensional stability at elevated temperatures. Increased PU contributed to reduced shrinkage, while nano chitosan enhanced pore formation without compromising thermal safety. Overall, the combination of PP, PU, and nano chitosan produced separators with suitable pore structures, chemical compatibility, and thermal performance for lithium-ion battery applications.

RESULTS AND DISCUSSION

Effect of Soaking Time on the Thickness of Lithium-Ion Battery Separator

After the vacuum drying process, thickness testing is performed to ensure that the separator layer has optimal dimensional stability after moisture loss. Thickness testing is important to assess the dimensional changes of the separator that occur during the drying process and to ensure that the separator still meets the desired thickness specification, which has a direct impact on the performance and durability of the lithium-ion battery. The thickness of the resulting separator can vary depending on the composition ratio of Polypropylene, Nano Chitosan and Polyurethane, as well as the length of soaking time. This difference in thickness occurs because the ratio of Polypropylene, Nano Chitosan and Polyurethane affects the formation of the separator layer. Figure-1 shows measurements of the thickness of the separator film taken from 9 samples with varying ratios of PP:PU: Nano Chitosan and immersion times ranging from 2 to 4 days. The measurement results indicate that variations in the composition ratio have a significant effect on the thickness of the separator film produced. Generally, thickness increases with higher nano chitosan content in the composition, but decreases again when polyurethane content dominates. At a sample of PCS₁, PCS₄, aPCS₇, the film thickness ranges from 25–26 µm. This value is quite high and exceeds the commercial separator thickness limit of <25 µm, which could affect the active volume efficiency within the battery cell. This high thickness is due to the high proportion of nano chitosan, which increases the viscosity of the solution and forms a thick but less uniform film structure.⁸ The sample of PCS₂, PCS₅, and PCS₈ shows thickness results that are close to the SNI standard, which is 23 µm for all immersion days. This value is closest to the standard for commercial lithium-ion battery separators and indicates good dimensional stability. The balanced composition of PU and nano chitosan at this ratio provides optimal effects in the solution, resulting in a film with uniform thickness and a dense structure. Meanwhile, the PCS₃, PCS₆, and PCS₉ ratio produces a thinner thickness, ranging from 21–22 µm. Excessively low thickness risks reducing the mechanical strength of the separator, making it susceptible to structural damage during the immersion process or when the separator is used in a battery cell. Although below the standard thickness limit, excessive thinness does not always provide an advantage, as it can reduce the separator's resistance to electrode and electrolyte pressure. From the overall results, it can be concluded that the PCS₂, PCS₅, and PCS₈ ratio provides the most optimal thickness and are sufficiently thin to meet LIB separator criteria while still maintaining good mechanical strength. This makes this ratio the best composition in terms of separator thickness.

Effect of composition ratio on the thermal stability of the separator. Thermal shrinkage in lithium-ion battery (LIB) separators occurs when the separator undergoes dimensional changes, such as shrinkage or deformation, due to exposure to high temperatures during battery operation. The separator, which functions to separate the positive and negative electrodes while allowing the movement of lithium ions through its microscopic pores, is susceptible to shrinkage due to the nature of its material. Excessive shrinkage can damage the structural integrity of the separator, reduce its ability to separate the electrodes properly, and risk causing a short circuit between the positive and negative electrodes, which can trigger a fire or explosion. Thermal stability of the separator is one of the important parameters in determining the safety and performance of lithium-ion batteries. In this study, thermal stability was tested through thermal shrinkage testing, which involved comparing the initial mass and final mass of the separator film after heating at 40°C for 6 hours.

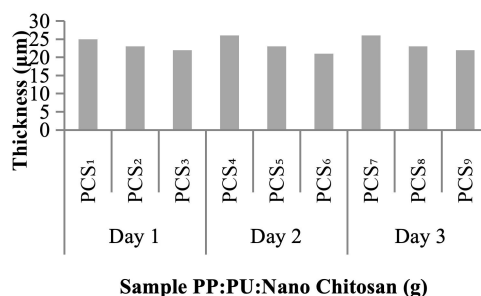


Fig.-1: Graph of Thickness (μm) versus Sample Comparison Based on Days of Immersion

The shrinkage values obtained indicate the extent of dimensional changes and mass loss caused by heat treatment, with the standard for lithium-ion battery separators typically setting a maximum shrinkage limit of 5%. Based on the test results of 9 samples, the thermal shrinkage of all composition ratios and soaking times was still within safe limits (<5%). However, there were significant differences between each variation. Samples PCS₂, PCS₅, and PCS₈ showed the lowest and most stable shrinkage values, ranging from 1.2% to 2.02% across all immersion days. These results indicate that the 4:3:3 ratio exhibits the best resistance to high temperatures, suggesting the stability of the formed separator structure. The balanced combination of PU and nano chitosan at this ratio enables the formation of a film with a dense morphology that is not easily shrunk by heat. Meanwhile, in samples PCS₁, PCS₄, and PCS₇, thermal shrinkage showed an increasing trend over time during immersion, reaching up to 1.98%. This may be due to the high content of nano chitosan, which, although it plays a role in pore formation and enhances absorbency, can cause dimensional instability when used in excess due to its high hydrophilicity. On the other hand, samples PCS₃, PCS₆, and PCS₉, which contain a large amount of PU, showed shrinkage up to 2.05%, approaching the maximum threshold. This indicates that films with excessive PU content tend to be more elastic but less resistant to thermal deformation if not balanced by the reinforcing structure of nano chitosan.

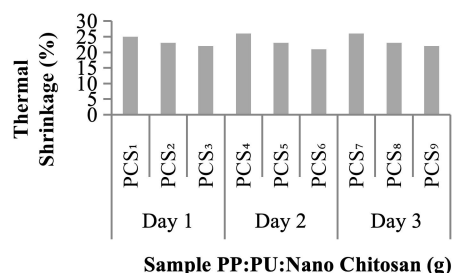


Fig.-2: Graph Showing the Effect of Immersion Time on Thermal

Effect of Composition Ratio on the TGA Analysis of the Separators

Thermogravimetric Analysis (TGA) on LIB separators is used to assess the thermal stability of separator materials, measure mass changes with temperature changes, and determine the degradation point of the material. Mass loss during thermal testing occurs due to decomposition processes, namely the breaking of chemical bonds. Figure-3 shows the TGA test results for the ratio of polypropylene, polyurethane, and nano chitosan, with the highest value in the previous test being at a sample ratio of 4:3:3. The TGA analysis results for the three samples show significant differences in terms of thermal stability and material weight loss during the heating process. Figure-3(b) (PP:PU: Nano chitosan with a sample PCS₅ ratio of 4:3:3 day 2) shows the most superior performance with a decomposition onset of 370.1 °C and a midpoint of 429.7 °C, which is higher than Fig.-3(c) (PP: PU: Nano chitosan with a sample PCS₈ ratio of 4:3:3 day 3) decomposition onset of 359.7 °C and midpoint of 425.7 °C, as well as Fig.-3(a) (PP: PU: Nano chitosan with a sample PCS₂ ratio of 4:3:3 day 1) decomposition onset of 322.3 °C and midpoint of 354.3 °C. This indicates that Sample PCS₅ has better thermal resistance because it is able to delay the degradation process to a higher temperature. In addition, the degradation range of PCS₅, which reached

502.4 °C, was also wider than that of PCS₂, which only lasted up to around 390 °C. Meanwhile, sample PCS₈ did reach a final temperature of nearly 505 °C, but this was accompanied by a greater weight loss. In terms of weight loss, PCS₅ only experienced a weight loss of -27.78%, which is much smaller than PCS₂ (-103.54%) and PCS₈ (-180.15%). This low percentage of mass loss indicates that the material structure of PCS₅ is relatively more stable and able to maintain its integrity when heated at high temperatures. Conversely, the high mass loss in PCS₂ and PCS₈ indicates that most of the material components are more easily decomposed, which is certainly a serious disadvantage for lithium-ion battery separator applications. In the context of separator performance, large weight loss can cause excessive porosity or even structural damage, which has the potential to reduce battery safety and reliability. Thus, when comparing the combination of onset parameters, midpoint, degradation temperature range, and especially weight loss rate, it can be concluded that PCS₅ has the best thermal stability and physical resistance. This is crucial for lithium-ion battery separators, which are required to operate under extreme conditions without significant degradation.

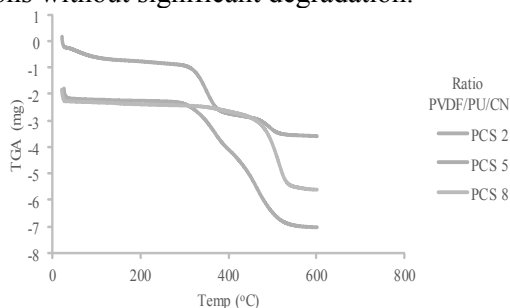


Fig.-3: (a) TGA PCS₂ (4: 3: 3, Day 1) (b) TGA PCS₅ (4: 3: 3, Day 2) (c) TGA PCS₈ (4: 3: 3, Day 3)

Morphological characteristics of separator surfaces based on scanning electron microscope (SEM). SEM testing on lithium-ion battery separators is conducted to observe the morphology and surface structure in detail at the microscopic scale. Through this testing, it can be observed whether the separator surface has a smooth or rough texture, or whether there are structural defects such as cracks, uneven pores, or deformations. Additionally, the thickness of the separator layer can be evaluated, as optimal thickness is crucial to prevent short circuits between electrodes and avoid increased internal resistance, which could reduce battery performance. SEM was used to analyse the distribution of constituent materials (PP, nano chitosan, and polyurethane) to ensure the homogeneity of the separator layer. Homogeneity is very important for maintaining mechanical stability and ion permeability. In this study, samples PCS₂, PCS₅ and PCS₈ with the optimal ratio of 4:3:3 were tested because they were considered balanced in terms of mechanical properties, thermal stability, and ionic conductivity. The SEM results are expected to provide an overview of the separator structure and support data from TGA and thermal stability tests. SEM test results on the three lithium-ion battery separator samples showed significant differences in surface morphology. In Figure-3(c) (PCS₈ ratio of 4:3:3 day 3), the surface morphology appears relatively dense, but there is still particle agglomeration in some areas and uneven pore distribution. This condition does support mechanical strength, but material inhomogeneity has the potential to reduce the electrochemical stability of the separator. Meanwhile, Fig.-3(a) (PCS₂ ratio of 4:3:3 day 1) shows a surface with large and uneven pores accompanied by light and dark areas, indicating a less homogeneous material distribution. Pores that are too large can increase permeability, but at the same time reduce mechanical strength and increase the risk of lithium dendrite penetration, which can trigger battery cell failure. On the other hand, Fig.3-(b) (PCS₅ ratio of 4:3:3 day 2) shows the most uniform and smooth surface morphology among the three samples. The surface appears dense with an even pore distribution and no significant agglomeration. This indicates that at this ratio, there is an optimal balance between polypropylene (PP), polyurethane (PU), and nano-chitosan. PP provides mechanical strength, PU acts as a compatibiliser that improves matrix uniformity, while nano-chitosan at an optimal concentration acts as a pore nucleation agent, resulting in small, homogeneous pores without agglomerates. The combination of these three materials creates a dense separator structure that remains finely and evenly porous. This morphology is highly desirable because it not only supports mechanical

stability but also ensures good ion permeability and reduces the risk of short circuits due to lithium dendrite growth. Therefore, the sample in Fig.-3(b) can be considered the best result compared to the other two samples.

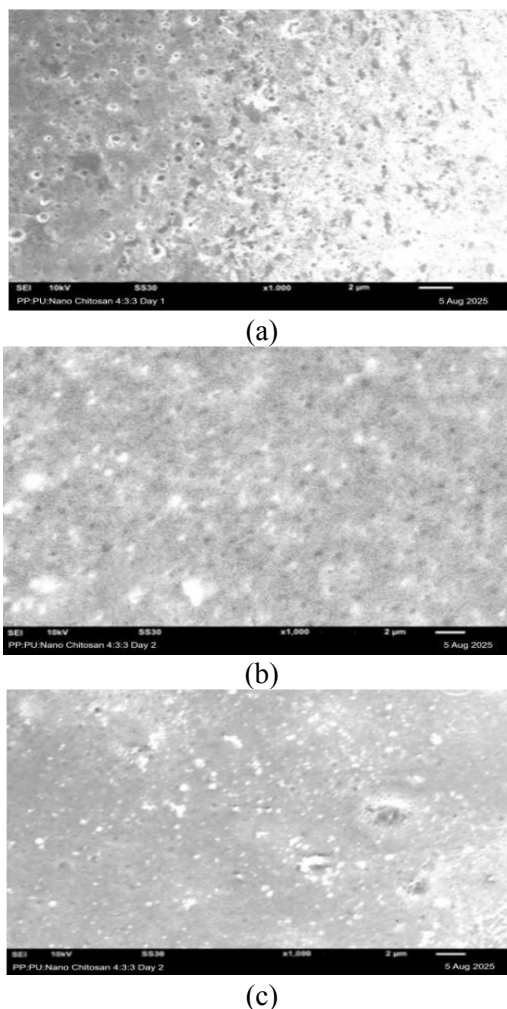


Fig.-4: (a) SEM PCS₂ (4:3:3, Day 1) (b) SEM PCS₅ (4:3:3, Day 2) (c) SEM PCS₈ (4:3:3, Day 3)

Structural and chemical characteristics of lithium-ion battery (LIB) separators based on Fourier Transform Infrared (FTIR) spectroscopy. Fourier Transform Infrared (FTIR) spectroscopy analysis is used to identify functional groups and changes in the chemical structure of lithium-ion battery separator materials. The FTIR spectra of pure PP and PP:PU: Nano chitosan composites show significant differences, indicating the success of the material modification. In pure PP, the FTIR spectrum (a) shows a characteristic polypropylene absorption peak at a wavenumber of 2958 cm⁻¹ associated with the asymmetric stretching of C–H from the –CH₃ group, as well as a peak at 1730 cm⁻¹ indicating the presence of minor carbonyl (C=O) stretching due to surface oxidation. In addition, the band at 1450–1400 cm⁻¹ is related to C–H bending deformation, while the band at 1160–1070 cm⁻¹ represents skeletal C–C strain from the polymer backbone. These characteristics are consistent with literature reports on the relatively simple characteristic spectrum of polypropylene and indicate the hydrophobic nature of the material. In contrast, the PP:PU: Nano chitosan spectrum shows new absorption bands as well as shifts in existing bands. The broad peaks around 3217–3780 cm⁻¹ represent the stretching of –OH and –NH groups originating from the amino groups of chitosan and the urethane groups in PU, which are not found in pure PP. The presence of an intense band at 1625 cm⁻¹ indicates C=O stretching from urethane bonds (–NH–CO–O–), while the band at 1155–1012 cm⁻¹ indicates C–O–C stretching from ether groups in PU and contributions from chitosan. In addition, the absorption band at 867–667 cm⁻¹ indicates glycosidic vibrations from chitosan, reinforcing the evidence of interaction between the polymer matrix

and biopolymer additives. A comparison of the two spectra confirms that the addition of PU and nano chitosan to PP not only results in morphological changes but also modifies the chemical structure of the separator material. The presence of polar groups $-OH$, $-NH$, and $-C=O$ increases hydrophilicity, which has a positive implication on the electrolyte absorption and ionic conductivity of the separator. These results are consistent with previous studies stating that the introduction of polar groups through polymer modification can increase the compatibility of the separator with liquid electrolytes and reduce the internal resistance of the cell.

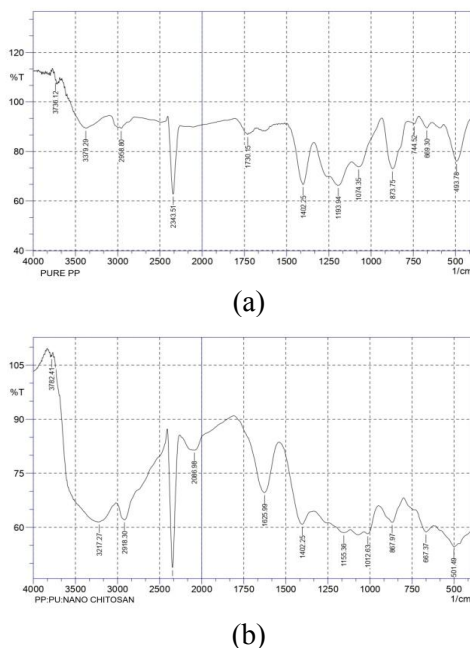


Fig.-5: (a) FTIR (Pure PP), (b) FTIR (PP: PU: Nano Chitosan)

CONCLUSION

This study demonstrates that the modification of lithium-ion battery separators using a composite of Polypropylene (PP), Polyurethane (PU), and Nano Chitosan has a significant influence on their physical, thermal, structural, and morphological characteristics. Among all variations, the PP: PU: Nano Chitosan ratio of 4:3:3 with a 2-day immersion time (PCS5) showed the most optimal performance. The optimised separator achieved a uniform thickness of approximately 23 μm , meeting commercial standards, and exhibited excellent thermal stability with shrinkage values below 2.02%. TGA results confirmed superior thermal resistance, with an onset degradation temperature of 370.1 $^{\circ}\text{C}$ and minimal mass loss of 27.78%. SEM analysis validated a dense and homogeneous surface morphology, ensuring safe ion transport and reducing the potential for short circuits. FTIR analysis confirmed successful chemical modification through the presence of urethane, hydroxyl, and amine groups, enhancing electrolyte compatibility. Overall, the PCS5 formulation provides a balanced and reliable separator suitable for lithium-ion battery applications. This outcome contributes to global sustainability efforts, particularly SDG 7 (Affordable and Clean Energy) by supporting safer and more efficient energy storage materials, and SDG 9 (Industry, Innovation, and Infrastructure) by advancing innovative engineered materials for next-generation battery technologies.

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CONFLICT OF INTERESTS

The authors declare that there is no conflict of interest.

AUTHOR CONTRIBUTIONS

The present work is related to *SDG-7: Affordable and Clean Energy*. All the authors contributed significantly to this manuscript, participated in reviewing/editing and approved the final draft for publication. The research profile of the authors can be verified from their ORCID ids, given below:

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