EVOLUTION IN ELECTRICAL AND ELECTRONIC ENGINEERING
e-ISSN: 2756-8458

EEEE

Vol. 6 No. 2 (2025) 106-112
https://publisher.uthm.edu.my/periodicals/index.php/eeee

Study on Electrical and Physical Properties of Lithium Cobalt Oxide Thin Films for Renewable Energy Applications

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DOI: https://doi.org/10.30880/eeee.2025.06.02.013

Article Info

Received: 08 July 2025
Accepted: 24 September 2025
Available online: 30 October 2025

Keywords

Lithium cobalt oxide, Thin Films, RF Magnetron sputtering, Reactive ion etching, Surface morphology, Conductivity, Renewable energy storage

Abstract

The work described in this paper seeks to understand the impact of post-deposition treatments on the electrical, mechanical and physical properties of lithium cobalt oxide (LiCoO2) thin films in order to improve their applicability as cathode materials in lithium-ion batteries. The thin films of LiCoO2 were deposited on nickel substrates by RF magnetron sputtering at room temperature, and then thermal annealed at 200 °C temperature during 1 hour and reactive ion etching (RIE) at 100 W RF power, 20 sccm Ar flow, 100 mTorr working pressure, for the duration of 2 minutes. The characterization of surfaces by FESEM and AFM showed that annealing boosted the average grain size (34.2nm untreated to 39.0nm) and roughness (Ra 5.2nm to 7.4nm) and etching further promoted grain size to 42.3nm and roughness to 9.8nm. Local conductivity was found to increase sixfold as measured by conductive AFM, 0.3 nA (bare) to 1.9 nA (etched). Contact angle measurements showed an increase in wettability after annealing (~25) and a decrease in wettability after etching (~50). The combination of these morphological and electrical improvements leads to an increase in the electrochemically active surface area, optimization of lithium-ion transport pathways, and a decrease in interfacial resistance - leading to improved charge storage and cycling stability. These results indicate the performance of LiCoO2 thin films can be greatly enhanced by adjusting the surface structure by controlled annealing and etching to meet the requirements of advanced energy storage devices.

1. Introduction

Lithium cobalt oxide (LiCoO2) has long been a dominant cathode material due to its high energy density, layered structure, and stable electrochemical behavior [1]. Thin film forms of LiCoO2 are being explored for next generation micro-batteries due to their compact structure, improved interfacial properties, and design flexibility [2]. Despite its advantages, as deposited LiCoO2 films often suffer from poor crystallinity and limited electrical conductivity. To overcome these drawbacks, thermal annealing is typically applied to promote grain growth and phase stabilization [3]. Surface modifications techniques such as plasma etching have also proven effective in tuning surface roughness and enhancing active surface area [4]. The influence of nanoscale surface features on electrochemical performance has been widely reported, particularly in relation to conductivity and electrolyte interaction [5].

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In this study, LiCoO2 thins films were deposited by RF magnetron sputtering and subjected to post-deposition treatments including thermal annealing and reactive ion etching (RIE). The structural, morphological, and electrical changes were characterized using FESEM, AFM, C-AFM and contact angle measurements. The goal is to evaluate how these modifications improve film properties for energy storage application.

2. Methodology

2.1 Thin Film Deposition

LiCoO2 thin films were deposited on pre-cleaned nickel substrates using RF Magnetron Sputtering [6-7]. The sputtering process was conducted at room temperature with base pressure below 5×10^{-6} Torr. A working pressure of 5mTorr was maintained using a 90% Ar and 10% O_2 gas mixture. The RF power was set at 100W and the deposition was carried out for 30 minutes with substrate rotation at 10 RPM to ensure uniform film growth [8].

2.2 Post-Annealing Treatment

Following deposition, the films were subjected to thermal annealing in ambient air. The annealing process was performed at 200°C for 1 hour using a tube furnace. This step aimed to enhance film crystallinity, promote grain growth, and relieve internal stress [9].

2.3 Reactive Ion Etching

To further modify the surface morphology, the films were etched using RIE in a controlled plasma system. Argon gas was used at a flow rate of 20sccm with a chamber pressure of 100mTorr and RF power of 100W. The etching duration was fixed at 2 minutes. The etching process aims to increase surface roughness and activate the electrochemical surface area by introducing micro textures [10].

2.4 Characterization Techniques

The films were characterized to evaluate morphological, electrical and wetting properties. Surface morphology was studied using Field Emission Scanning Electron Microscopy (FESEM), while surface roughness and topography were analyzed using Atomic Force Microscopy (AFM). Local conductivity mapping was performed using Conductive AFM (C-AFM) in contact mode. Wettability behavior was assessed via contact angle measurement using deionized water droplets to evaluate surface energy changes before and after treatment [11]. The overall experimental process is summarized in the flow chart shown in Fig. 1.

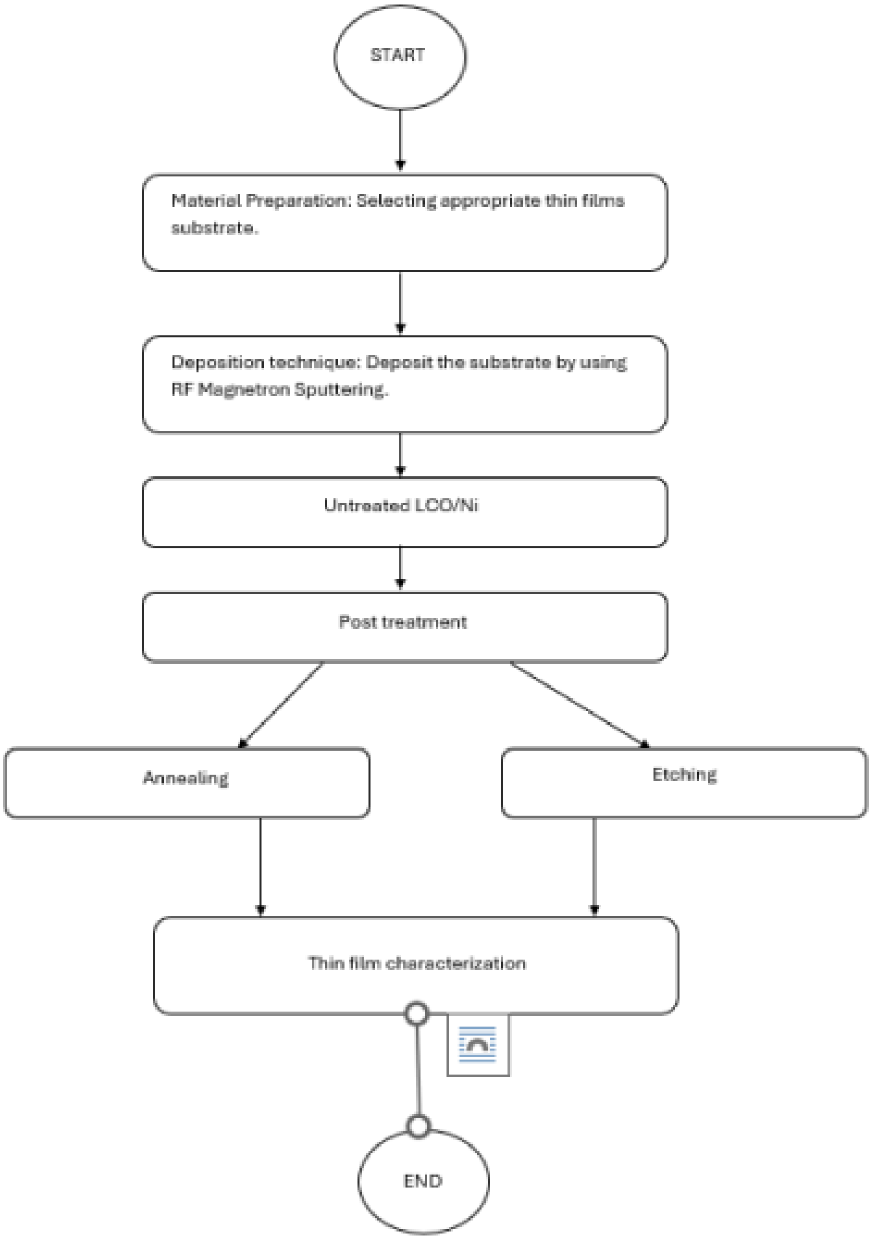
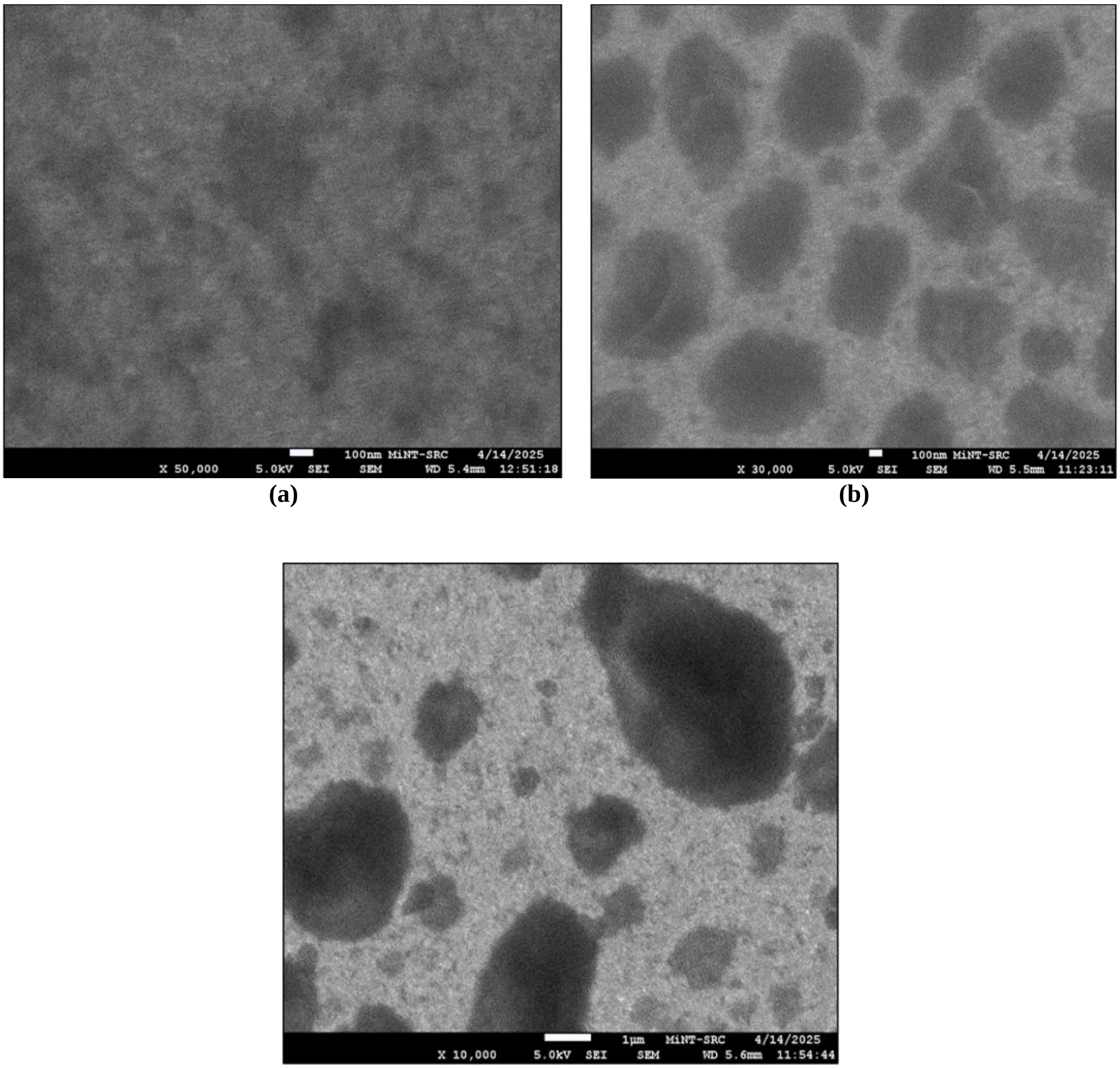


Fig. 1 *Experimental process flow for thin film preparation and treatment.*

3. Results and Discussion

3.1 Surface Morphology by FESEM

The morphological evolution of $LiCoO_2$ thin films shown in Fig. 2 was examined using FESEM for bare, annealed and etched samples. The untreated (bare) films showed densely packed grains with a relatively smooth surface and minimal porosity, indicating limited crystallinity. After annealing, grain boundaries became more defined and larger in size, suggesting enhanced crystallinity and growth due to thermal energy input. Upon etching, the surface became rougher with tiny cavities formed due to ion interactions, improving the film's reactivity and enhancing ion mobility during operation.



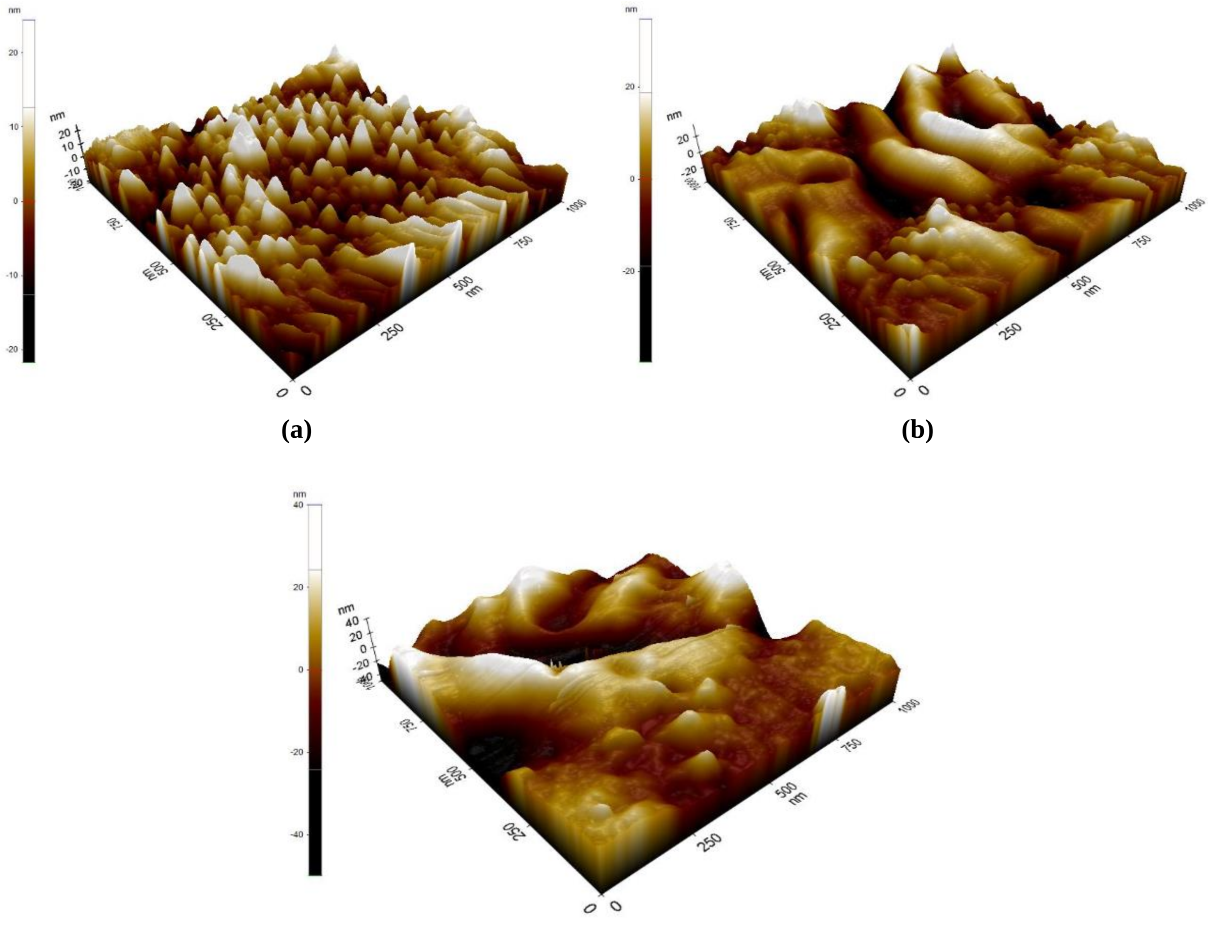
(a) **(b)**

(c)

Fig. 2 *FESEM images of LiCoO2 thin films: (a) Bare (b) Annealed (c) Etched*

3.2 Topographical Analysis by AFM

AFM was used to quantitatively assess the surface roughness (Ra) and topography of sample as shown in Fig. 3. The results revealed a consistent increase in roughness with each treatment step. The bare sample recorded a roughness of approximately 5.2nm, while the annealed sample increased to 7.4nm. The etched sample reached the highest roughness of 9.8nm. This increase in surface roughness contributes positively to the electrochemical performance by enlarging the effectiveness contact area with the electrolyte.



(a) **(b)**

(c)

Fig. 3 *AFM topography of LiCoO2 thin films: (a) Bare (b) Annealed (c) Etched*

3.3 Local Conductivity from C-AFM

The conductive properties of the films were analyzed using C-AFM. As depicted in Fig. 4, the current distribution maps showed a significant increase in local current response after each treatment. The bare film exhibited a low current response of around 0.3nA, which increased to 1.0nA for the annealed sample and further to 1.9nA for the etched sample. This six fold improvement indicates that both crystallinity from annealing and surface roughness from etching play crucial roles in enhancing local conductivity [10].

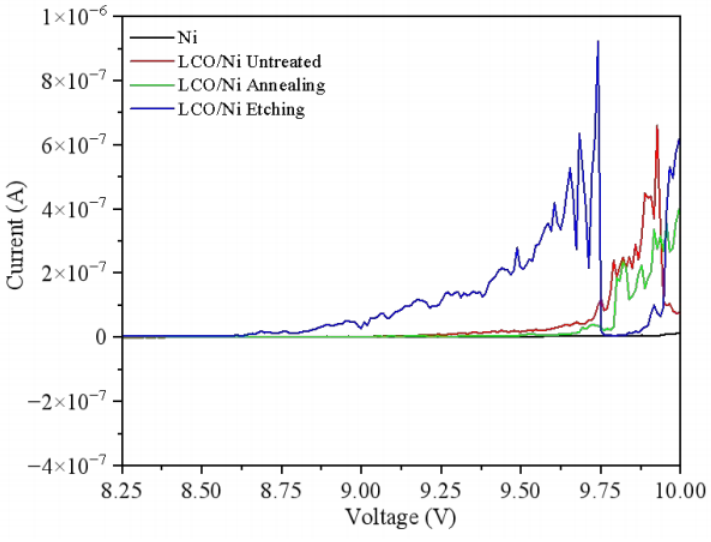
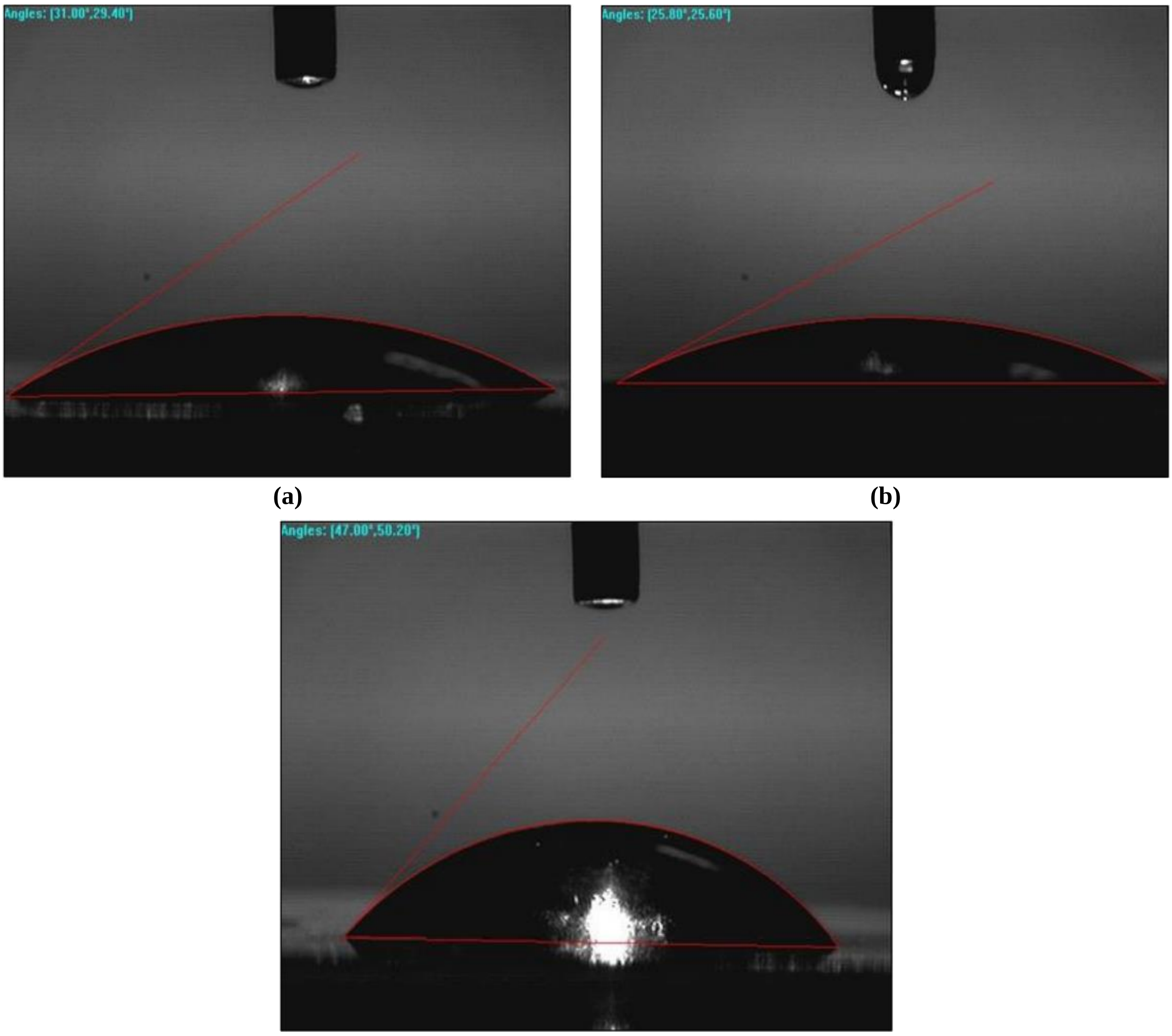


Fig. 4 **C-***AFM current maps of LiCoO2 thin films.*

3.4 Wettability Analysis

Surface wettability, an indicator of surface energy and electrolyte compatibility, was evaluated through contact angle measurements. As depicted in Fig. 5, the bare film had a contact angle of approximately 42°, indicating moderate hydrophilicity. Post-annealing, the angle decreased to around 25°, suggesting improved wettability. However, after etching, the angle increased to ~50°, likely due to increased roughness and the formation of microscale texture that reduces the solid-liquid contact area. While high wettability promotes electrolyte spreading, controlled roughness may help modulate electrolyte penetration depending on the application [13-14].



(a) **(b)**

(c)

Fig. 5 *Contact angle measurements: (a) Bare (b) Annealed (c) Etched*

4. Conclusion

This study demonstrates that post-deposition treatments significantly influence the structural, electrical, and surface properties of LiCoO2 thin films. Annealing improved crystallinity and increased grain size, while reactive ion etching further enhanced surface roughness and conductivity. These modifications contributed to better wettability, higher electrochemical surface area, and enhanced local charge transport. The combination of thermal and plasma-based treatments proved effective in optimizing thin film characteristics for application in lithium-ion batteries and other renewable energy storage devices.

Acknowledgement

The authors would like to thank Universiti Tun Hussein Onn Malaysia (UTHM) for their continuous support, guidance and access to laboratory facilities throughout this project.

Conflict of Interest

Authors declare that there is no conflict of interests regarding the publication of the paper.

Author Contribution

The authors confirm contribution to the paper as follows: ***study conception and design:*** *Syarifah Aqilah binti Syed Ahmad Tarmizi;* ***data collection:*** *Syarifah Aqilah binti Syed Ahmad Tarmizi;* ***analysis and interpretation of results:*** *Syarifah Aqilah binti Syed Ahmad Tarmizi;* ***draft manuscript preparation:*** *Syarifah Aqilah binti Syed Ahmad Tarmizi, Nafarizal bin Nayan. All authors reviewed the results and approved the final version of the manuscript.*

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