

Developing Sustainable Reinforcement PLA/NCC from Kapok to Improve Mechanical and Electrical Properties Composites with PVA Matrix



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ABSTRACT

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electrolyte membrane, electrospinning, PLA, fibers, nanocellulose crystals

This study investigates the enhancement of mechanical and electrical properties of composite electrolyte membranes through the incorporation of polylactic acid (PLA) fibers and nanocrystalline cellulose (NCC) derived from kapok. PLA fibers were prepared using electrospinning, while kapok NCC was synthesized through pulping, bleaching, and hydrolysis. Scanning electron microscopy (SEM) analysis revealed an average PLA fiber diameter of 597 nm and a kapok NCC grain size of 70.2 nm. The composite electrolyte membranes were prepared by combining polyvinyl alcohol (PVA), potassium hydroxide (KOH), glycerol, and varying amounts of PLA/NCC reinforcement. Tensile tests showed that the addition of 0.05 g kapok NCC improved the tensile strength by 87.6% and increased elongation. Impedance analysis demonstrated a decrease in impedance with the incorporation of PLA and kapok NCC fibers, indicating enhanced conductivity. The sample with 0.05 g kapok NCC (FKP5) exhibited the highest conductivity of 0.068 mS/cm. Cyclic voltammetry revealed more pronounced reduction and oxidation reactions in FKP5, while electrical power measurements showed a maximum power of 12.6 mW for FKP5. The improved mechanical and electrical properties of the composite membranes containing PLA and kapok NCC highlight their potential for use in sustainable batteries and other industrial applications.

1. INTRODUCTION

The use of composition materials has gained widespread acceptance across diverse sectors, including construction, aerospace, and biomedical industries. The ability of composite materials to combine the superior properties of various components makes them a promising solution. Additionally, the use of biodegradable materials has gained industry attention due to concerns regarding their environmental impact. The exploration and development of materials that can naturally decompose has become a promising strategy for reducing environmental pollution.

Poly(lactic acid) (PLA) is a biodegradable and relatively inexpensive polymer that can be produced from renewable resources with good thermomechanical properties and is considered a sustainable alternative to fossil-based plastics [1-4]. PLA has good thermal workability and mechanical properties, similar to conventional polyesters. PLA can be converted into fibers via electrospinning. Electrospinning produces fibers of different sizes and characteristics, ranging from micro-to nanofibers with various types and levels of porosity [5-8]. This method uses high tension to produce fibers [9]. The electrospinning method has the advantages of unique morphology and improved properties [10], such as increased surface area [11], as well as making fibers with high mechanical properties, so it is suitable as a method for

producing fibers as reinforcement in composites [12]. PLA has low toughness. Given its limitations, PLA can be reinforced with natural or synthetic fibers or nanoparticles to form composites with more excellent durability, strength, and recyclability [13, 14]. Nanocrystals such as cellulose nanocrystals (NCC) have demonstrated enhancement of the mechanical characteristics of polymer materials such as PLA [15]. NCC may serve as a reinforcing agent in PLA composites to enhance their strength and ductility [7]. NCC can be formed from various types of cellulose, one of which is Kapok cellulose [16-18]. Kapok fiber reinforcement has increased tensile and impact strengths and shows promise as an environmentally friendly material [19-22]. Polyvinyl alcohol (PVA) is preferred as the matrix because of its beneficial properties. PVA has good solubility in water; therefore, it is easy to mix and process with fibers [23]. PVA exhibits favorable characteristics in terms of elasticity and flexibility, allowing it to resist deformation [23]. Thus, KOH is a suitable electrolyte for use in supercapacitors and batteries. KOH-activated materials have shown excellent electrochemical performance in supercapacitors, attaining high specific cell capacitance values.

This research will contribute significantly to the advancement of current knowledge by deepening the understanding of the use of natural reinforcements, such as PLA (polylactic acid), NCC (nanocellulose crystals), and

kapok fiber, in combination with a PVA (polyvinyl alcohol) matrix. These natural materials are sustainable and biodegradable and exhibit unique properties that enhance the performance of composite materials. By exploring the synergistic effects of combining these reinforcements with PVA, the study aims to improve the mechanical properties, such as tensile strength and durability, and the electrical properties, such as conductivity of the resulting composites. This research offers an environmentally friendly alternative to traditional composite materials, which often rely on non-renewable, synthetic components [13, 24, 25]. Additionally, the superior performance of these bio-based composites, in terms of strength, flexibility, and conductivity, makes them suitable for various industrial applications, including electronics, automotive components, packaging, and energy storage devices like batteries. The findings of this study not only support the shift towards greener, more sustainable materials but also provide valuable insights for future research and development in the field of advanced composite materials.

2. METHODS

2.1 Materials

PVA, KOH, glycerol, NaOH, NaClO, and H₂SO₄ were procured from Merck, whereas acetone and DMF were obtained from Andeska Ltd. PVA, KOH, and glycerol were used in the production of electrolyte membranes. NaOH, NaClO, and H₂SO₄ were utilised in the synthesis of nanocrystalline cellulose (NCC). Acetone and DMF functioned as solvents in the synthesis of PLA fibers.

2.2 Characterization

2.2.1 Scanning electron microscopy (SEM)

Scanning Electron Microscopy (SEM) offers a remarkably adaptable and finely detailed approach to analyzing two-dimensional and three-dimensional materials, encompassing imaging and chemical examination. This methodology is noninvasive and applicable to examining materials ranging from nanoscale to microscale, rendering it an invaluable instrument for comprehensively assessing materials [26]. SEM was performed using a Hitachi S-3400N instrument to analyze the biomaterial composites' morphology, particle size, and membrane characteristics. This approach entails incorporating many components of a sample onto a metallic substrate. The Hitachi S-3400N device facilitated accurate analysis and distinct visual depiction of the membrane surface of the biomaterial composite.

2.2.2 Tensile strength

The concept of tensile strength is concerned with the ability of a substance or arrangement to endure deformation without enduring permanent and irreversible distortion. It indicates the maximum magnitude of tensile deformation that a given material can sustain before fracturing. The tensile strength value can vary based on the nature and composition of the substance [6]. ASTM D638 Type 5 is a standard used to determine the properties of a material by subjecting it to tension and measuring its strength and deformation during tensile testing.

2.2.3 Impedance

Impedance was measured using a Potentiostat Corrtest

E100 instrument. Impedance is the resistance of a circuit to the flow of alternating current, represented as $|Z|$. It is a complex number comprising both resistance (Z_{re}) and reactance (Z_{im}). Comprehending impedance is essential for the analysis of electrical circuits, particularly alternating current circuits, as it affects the circuit's response to varying frequencies. Eq. (1) can be employed to determine the resultant impedance.

$$|Z| = \sqrt{(Z_{re})^2 + (Z_{im})^2} \quad (1)$$

2.2.4 Ionic conductivity

Four NCC-containing membrane samples with compositions of 0, 0.01, 0.03, and 0.05 g were tested for ionic conductivity using Electrochemical Impedance Spectroscopy at room temperature with frequencies from 10 mHz to 100 kHz, DC voltages of 0 mV and AC voltages of 10 mV rms. The stabilization took over 10 minutes. The electrolyte had a surface area of 4 cm². Ionic conductivity of post-stabilization samples was calculated using Eq. (2).

$$\sigma = \frac{L}{R \cdot A} \quad (2)$$

The ionic conductivity (σ), membrane thickness (L), and measured resistance (R) are all factors that influence the electrolyte's surface area (A) in this equation. The aforementioned equation facilitates the calculation of ionic conductivity through the resistance values acquired from electrochemical spectrometry assessments. This work provides critical insights into the influence of NCC on the ionic conductivity of membranes, potentially enhancing their performance across diverse applications.

2.2.5 Cyclic voltammetry (CV)

Cyclic voltammetry (CV) is an electrochemical technique that observes the current while the electrode potential is incrementally increased and decreased in an electrolyte solution. An elevation in potential induces oxidation, generating an anodic current, whereas a lowering in potential leads to reduction and cathodic current. These current peaks convey insights into redox reactions and the electrochemical characteristics of substances within the electrolyte.

2.2.6 Electrical power

Electrical power is a quantity that indicates how quickly a device or electrical network consumes electrical energy. It is calculated based on the result of voltage (V) multiplied by electric current (I), with the unit being watts (W). Understanding electrical power is essential in determining electrical devices' efficiency and energy consumption.

2.3 Preparation of PLA fibers

A total of 1.25 g of PLA was added to a measuring cup, followed by the addition of solvent to ensure a homogeneous solution with a composition of 50% AC and 50% DMF. The concentration of PLA in the solvent was 12.5%. The mixture was then stirred at 100 rpm for 2 hours at a temperature of 50°C using a magnetic stirrer to dissolve the PLA. The subsequent step involves utilizing the electrospinning technique to fabricate fibers. This process employs electrohydrodynamics, wherein liquid droplets are charged to create a jet, which is then stretched and elongated to form fibers [27, 28]. As depicted in Figure 1, the electrospinning

apparatus consists of three main components: a high-voltage power source, a syringe pump, a spinneret, and a collector.

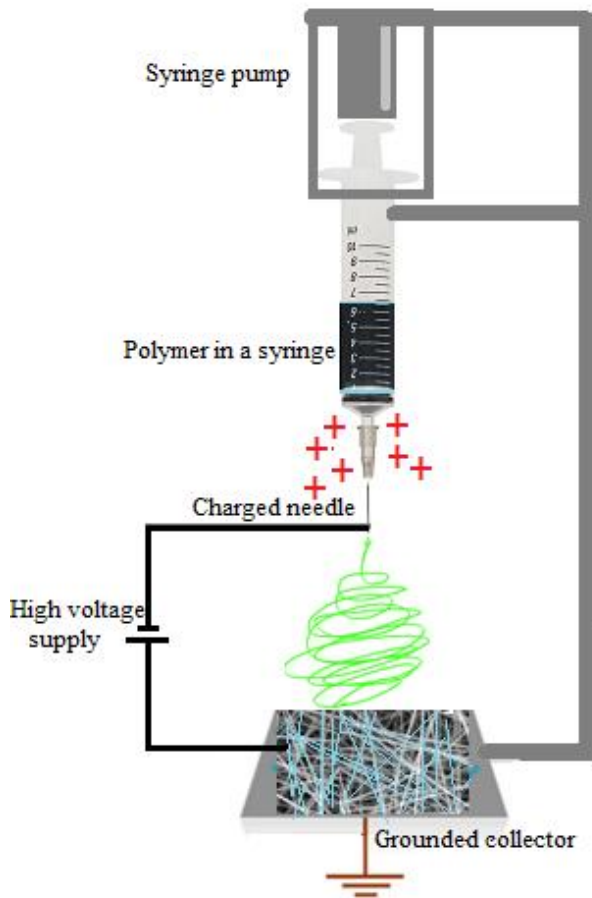


Figure 1. The basic setup of electrospinning

In this process, the electrospinning device is adjusted to a voltage of 20 kV, operating at a flow rate of 0.2 ml/min [10]. The resulting fibers will be gathered in the collector, after which the polylactic acid (PLA) fibers will be stored in an airtight container for future use or preservation.

2.4 Preparation of kapok NCC

Kapok NCC is produced through pulping, bleaching, and hydrolysis [29]. Pulping refers to the process of producing pulp. Kapok was immersed in distilled water and magnetically agitated at 60°C for 2 hours with 1.5% NaOH. This technique solubilizes lignin and removes it from cellulose fibers for refinement. The subsequent procedure was bleaching with 3.5% sodium hypochlorite following pulping. Kapok pulp was solubilized in two hours at 60°C using a magnetic stirrer. Bleaching eliminates color, stains, and impurities from materials or substrates. Hydrolysis occurs subsequent to bleaching. The treated kapok was combined with 66% sulfuric acid in 400 milliliters. The solution was magnetically agitated at 45°C and 300 revolutions per minute. The suspension was centrifuged at 3000 rpm for 15 minutes and repeated until achieving neutral pH [30].

2.5 Preparation of composite electrolyte membrane solution

A 4 g sample of polyvinyl alcohol (PVA) was solubilized in

100 mL of distilled water by heating to approximately 80°C. The resulting solution was magnetically stirred for 3 h, during which time, clarity and homogeneity were achieved. Approximately 2 g of Potassium hydroxide (KOH) was added to 20 mL of distilled water. The two solutions were then mixed together. Glycerol was incorporated into the PVA-KOH mixture with continuous agitation and heating to guarantee complete homogeneity. Subsequently, the PLA/NCC reinforcement was incorporated into the electrolyte membrane matrix.

3. RESULT AND DISCUSSION

3.1 Scanning electron microscopy (SEM) morphological

The morphology in Figure 2 displays a scanning electron microscopy (SEM) analysis of the polylactic acid (PLA) fibers. The SEM data for the PLA fibers were processed using the ImageJ software to determine the diameter of the fibers. Based on these data, the average diameter of the fibers was found to be approximately 597 nm. With such a small relative fiber diameter, PLA fibers can be classified as approaching nanofibers. According to the ISO/TS 80004-1 standard, nanoscale is approximately 1-100 nm [31]. The small dimensions of the PLA fibers contribute to an increase in the surface area, which enhances the mechanical strength, absorption capacity, and chemical resistance [32].

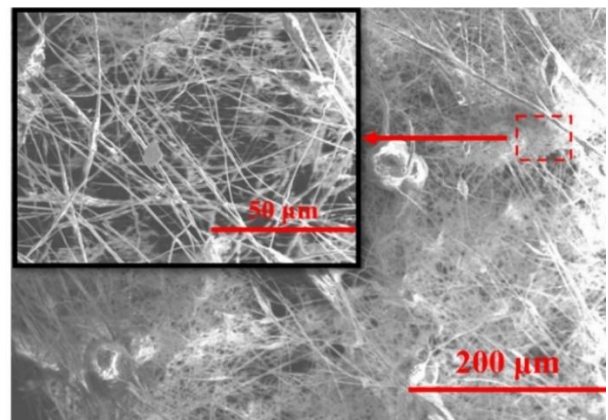


Figure 2. SEM result PLA fibers

The kapok NCC data were observed using a microscope optic SZX10 and processed using the ImageJ software to determine the NCC grain size. The average NCC grain size of 70.2 nm was observed. Based on the size of the NCC, kapok can be categorized as nanosized [31]. The grain size influences its tensile properties; the smaller, the stronger [33].

3.2 Tensile test

Figure 3 displays the tensile test results of four different samples: PVA/PLA/0 g NCC-kapok (FK0), PVA/PLA/0.01 g NCC-kapok (FKP1), PVA/PLA/0.03 g NCC-kapok (FKP3), PVA/PLA/0.05 g NCC-kapok (FKP5).

The tensile strengths of FKP0, FKP1, FKP3, and FKP5 were 3.72 MPa, 3.79 MPa, 4 MPa, and 6.98 MPa, respectively. From Figure 3, it is apparent that the FKP5 sample had the highest tensile strength. The inclusion of 0.5 g of kapok NCC resulted in a significant improvement of 87.6% in the tensile strength. The tensile strength of FKP1 increased by 1.9%,

while that of FKP3 increased by 7.5%. These findings highlight the potential of NCC as a powerful tool for improving polymer characteristics. By adding just 0.05 g, the use of NCC greatly enhances the tensile strength.

Furthermore, kapok NCC exhibited a significant increase in length and elongation [34]. In Figure 3, FKP5 demonstrated the greatest elongation, measuring 13.03 cm, while FKP1 and FKP3 showed lesser increases, measuring 8.5 cm and 8.71 cm, respectively. Overall, the elongation of the membrane containing kapok NCC experienced an increase in elongation compared to kapok non-NCC membranes with a value of 2.97 cm. In summary, this study indicates that including biodegradable materials such as PLA and kapok NCC in manufacturing composite materials can enhance their mechanical properties and mitigate environmental pollution.

The selection of this concentration is based on preliminary tests and previous literature, which indicate that variations in concentration can significantly influence the mechanical properties of composites. In a study by Sultana et al., a concentration range of 1-5 wt% nanocellulose was used to enhance the material's mechanical strength [35].

3.3 Impedance

Based on Figure 4, impedance analysis of the membrane system shows that the addition of kapok NCC positively influences the conductivity of the PVA/PLA membrane. Impedance (Z), which consists of resistance Z_{re} and reactance Z_{im} , indicates that increased Z_{re} can be caused by higher resistance or lower conductivity at a particular frequency. Observations showed an increase in Z_{im} , indicating the presence of greater capacitive or inductive elements with the addition of PLA/NCC-kapok fibers. Overall, the data shows a decrease in impedance, especially in the FKP5 sample with the lowest impedance of 3662.3 ohms, which reflects an increase in membrane conductivity due to the addition of PLA and kapok NCC fibers.

3.4 Ionic conductivity

Figure 5 illustrates the enhancement in conductivity values corresponding to the incorporation of kapok NCC in samples FKP0 through FKP5. Specifically, FKP0 has a conductivity of 0.048 mS/cm, but FKP1 marginally rises to 0.050 mS/cm. Nonetheless, a more pronounced enhancement in conductivity is shown in FKP3, registering at 0.060 mS/cm, whereas FKP5 exhibits the peak conductivity value of 0.068 mS/cm. This finding aligns with the research by Atifi et al., which showed a conductivity of around $2.34 \pm 0.28 \times 10^{-6}$ S/cm for solid polymer electrolyte (SPE) [36].

These results indicate that the addition of kapok NCC can enhance the conductivity of the sample, which may lead to improved material performance in specific applications, such as better electrical conductivity. The presence of kapok NCC facilitates the transfer of electrical charges in the material, which can explain the observed increase in conductivity.

3.5 Cyclic voltammetry

Figure 6 illustrates the intensity of the reduction and oxidation reaction peaks and the kinetics of electrochemical reactions when voltage is varied upward and downward. FKP5 exhibits a substantial enhancement in power and demonstrates an elevated intensity of electrochemical processes, resulting in

more pronounced reduction and oxidation reactions. Nevertheless, the voltammetry cycle concludes at a different point than its initiation due to the instability in the chemical reaction between the membrane electrolyte and aluminium, which serves as the working electrode. Aluminium is particularly susceptible to alkaline substances and easily experiences corrosion [37].

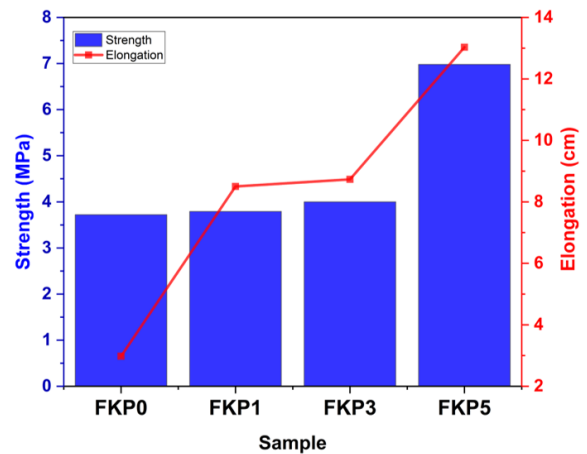


Figure 3. Graph tensile test of electrolyte membranes with various samples: FKP0, FKP1, FKP3, and FKP5

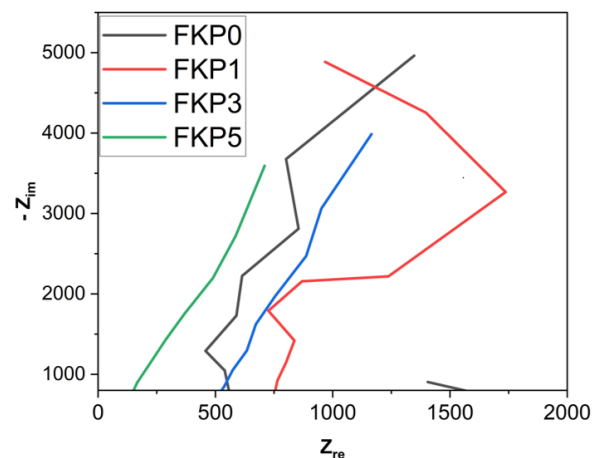


Figure 4. Nyquist plots of PVA/KOH/glycerol membranes with various samples: FKP0, FKP1, FKP3, and FKP5

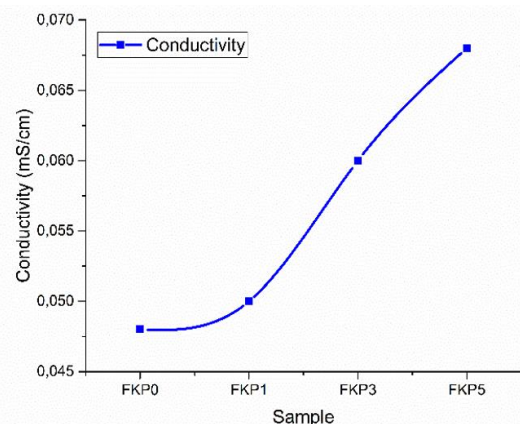


Figure 5. Ionic conductivity of PVA/KOH/glycerol membranes with various samples: FKP0, FKP1, FKP3, and FKP5

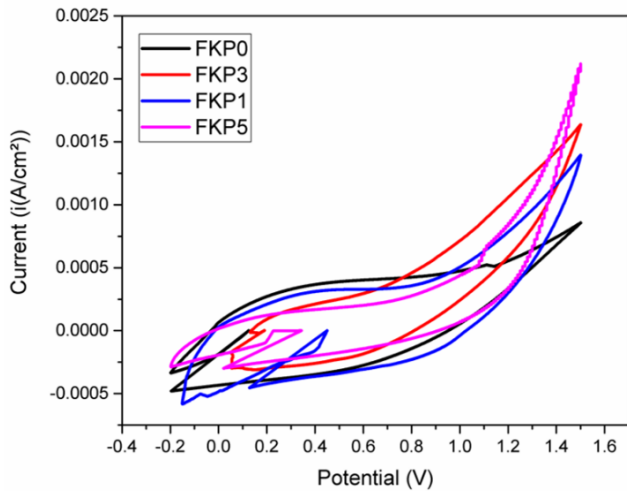


Figure 6. Cyclic voltammetry

3.6 Electrical power

Figure 7 illustrates the power enhancement in the PVA/KOH/Glycerol membrane corresponding to differences in kapok NCC, measured in milliwatts. FKP0 generates a minimal power of 5.1 mW. Nevertheless, FKP 5 provides a maximum power of 12.6 mW. The increase of kapok NCC inside the membrane mixture enhances the density of membrane particles, facilitating improved electron transport.

Porous membranes may exhibit elevated ionic conductivity due to their microporous architecture, facilitating ion movement akin to that in a pure liquid electrolyte [38].

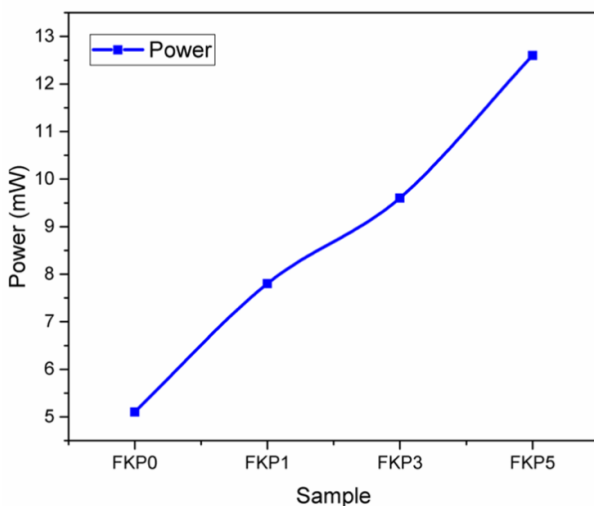


Figure 7. Power of PVA/KOH/Glycerol

4. CONCLUSIONS

The SEM analysis of the PLA fibers showed an average diameter of 597 nm, while the study of the kapok NCC revealed a paper NCC grain size of 70.2 nm. The addition of NCC increased the tensile strength of the samples by up to 87.6%. The highest elongation was observed for the FKP5 sample, and the addition of NCC increased the elongation of the samples. The sample impedance decreased with the addition of PLA and kapok NCC fibers. Decreasing

impedance will increase the conductivity of the sample. FKP5 has the highest increase in conductivity of 40.42%.

Aluminum batteries have seen limited development due to the susceptibility of aluminum anodes to corrosion, which occurs because the ionic nature of the electrolyte membrane destabilizes chemical reactions. Potential solutions to address this issue include protecting the aluminum anode through techniques such as coating.

The results of this study reveal that the addition of kapok NCC into the battery membrane can significantly improve its electrical and mechanical properties, which are essential to supporting battery performance for a longer and more efficient period. Therefore, the use of kapok NCC has great potential in industrial applications, especially in developing lighter, stronger, and more environmentally friendly sustainable batteries in the future.

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NOMENCLATURE

σ	Ionic conductivity, mS/cm
$ Z $	Impedance, Ω
Z_{re}	Impedance, Ω
Z_{im}	Reactance, Ω