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Research article

Enhancing Ionic Conductivity and Stability of Electrolyte Membranes: A Study on CQD-GO Composites for Aluminum-Air Batteries

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ABSTRACT

The development of efficient and sustainable energy storage technologies is crucial to address the depletion of fossil fuels and environmental pollution. This study focuses on the synthesis and characterization of a novel electrolyte membrane based on the integration of Carbon Quantum Dots (CQDs) derived from coconut-shell charcoal into a Graphene Oxide (GO) matrix for application in aluminum-air batteries. The CQDs were synthesized using a simple, low-cost, and environmentally friendly hydrothermal method, whereas the GO-based membrane was prepared via a solution casting technique. The physical and electrochemical properties of the resulting CQD-GO composite membranes were systematically investigated using various characterization techniques, including Fourier Transform Infrared Spectroscopy (FTIR), X-ray Diffraction (XRD), Electrochemical Impedance Spectroscopy (EIS), and Scanning Electron Microscopy (SEM). The results revealed that the incorporation of CQDs significantly enhanced the ionic conductivity, reduced the internal resistance, and improved the interfacial stability of the membrane compared with pristine GO. The synergistic effect between the CQDs and GO was attributed to the formation of hybrid conductive pathways and the enhanced ion mobility within the membrane structure. Furthermore, the CQD-GO membrane exhibited excellent stability in the electrochemical environment typically encountered in aluminum-air batteries. These findings highlight the potential of integrating biomass-derived CQDs with GO to develop high-performance electrolyte membranes for advanced energy storage applications, paving the way for more efficient and environmentally friendly aluminum-air batteries.

1. INTRODUCTION

In the current age, the decreasing availability of fossil fuels is coupled with their significant contribution to environmental pollution. Therefore, it is imperative to explore alternative renewable energy sources offered by nature, such as solar, wind, and hydropower. Although these renewable energies are plentiful in nature, the challenge lies in the inefficient and limited energy storage technology. Effective energy-storage solutions are essential for facilitating this shift. In particular,

batteries are vital for portable electronic devices and electric vehicles (EVs) because of their relatively high energy density and electronic efficiency. Currently, lithium-ion batteries (LIBs) dominate the market because of their high gravimetric and volumetric capacities and excellent energy efficiency. However, the energy density of current LIBs, approximately 100–200 Wh/kg, falls short of meeting the high-energy and power demands of EVs. Consequently, new strategies are necessary for developing energy storage and conversion systems with higher theoretical energy densities for future

applications[1]. One promising alternative under development is the aluminum-air battery, which boasts high energy density, environmental friendliness, and lower costs depending on the type of metal anode used[2]. This battery generates electrical energy through an electrochemical reaction between aluminum and oxygen from the air[3].

In this battery system, the electrolyte membrane is essential for keeping the anode and cathode apart, while also facilitating ion conduction. The electrolyte solution must strike a balance between sufficient ion concentration and high capacity for ion diffusion to achieve optimal performance. Additionally, the membrane must regulate the diffusion of oxygen from air into the cathode to prevent disruption of the electrochemical reaction. Graphene Oxide (GO) is a promising material for this purpose due to its porous structure, large surface area, and hydrophilic nature, which promote high ionic conductivity and effective oxygen diffusion [4]. Graphene oxide (GO) is a carbon-based material abundant in functional groups like hydroxyl, epoxide, and carboxyl, which aid ionic interactions and improve membrane stability in electrolytes. Moreover, the synthesis of GO is simpler than that of pure graphene, while still retaining excellent mechanical and electrical properties[5]

The efficiency of GO-based electrolyte membranes can be enhanced by incorporating additives such as carbon quantum dots (CQDs). These are tiny carbon particles with exceptional optoelectronic properties, large surface areas, and excellent electron-transfer abilities. Notably, CQDs derived from coconut-shell charcoal are eco-friendly, cost-effective, and sustainable, as they utilize waste biomass. CQDs contribute to boosting ionic conductivity, thermal stability, and enhancing the interfacial interaction properties of membranes [6].

Batteries serve as crucial energy storage devices and play a significant role in addressing both the present and future energy demands. Numerous studies have highlighted that incorporating Graphene Oxide into battery systems like zinc-air and lithium-air

batteries enhances electrolyte membrane performance by boosting ionic stability and lowering internal resistance[7].

The effectiveness of graphene-based materials and their derivatives in enhancing the ionic conductivity and electrochemical performance of metal-air battery systems is well documented. Research conducted by Shao et al. demonstrated that adding graphene oxide (GO) and reduced graphene oxide (rGO) to membrane structures can decrease internal resistance and enhance battery cycle stability by facilitating ion migration pathways and more efficient charge transfer[7].

Moreover, advancements in membrane research for metal-air batteries have yielded highly encouraging outcomes. The incorporation of carbon-based nanocomposites, such as graphene and fullerenes, has enhanced the performance of electrochemical energy storage systems, such as supercapacitors and batteries. Graphene is recognized for its excellent electrical conductivity, expansive specific surface area, and robust chemical stability, making it a promising candidate for the development of polymer-based solid electrolytes in batteries[8].

Furthermore, the inclusion of Carbon Quantum Dots (CQDs) has been shown to enhance the performance of electrolyte membranes in supercapacitors and metal ion batteries by improving ionic conductivity and electrode interface properties[9].

The inclusion of carbon-based materials, such as graphene and graphene oxide, has been shown to markedly enhance the electrical conductivity and interfacial stability of various energy storage systems. Hao et al. found that employing carbon layers, especially those based on graphene and graphene oxide, can improve electron transfer routes, strengthen active material adhesion, and lower internal resistance, collectively enhancing the cycle performance of energy devices, such as batteries and supercapacitors. These benefits are attributed to the high electrical conductivity, chemical stability, and corrosion resistance of graphene and its derivatives [10].

With the progress of energy storage technology, aluminum-air batteries have emerged as a significant area of research interest. Incorporating graphene-based materials into aluminum-air battery systems has demonstrated improved electrochemical performance, notably by enhancing the oxygen reduction reaction (ORR) activity and expanding the active surface area for electrochemical processes. Qin et al. revealed that applying graphene to SrCoO₃ perovskite materials notably boosts the specific surface area, optimizes electron transfer routes, and increases the discharge capacity of aluminum-air batteries. The integration of graphene with an oxide material structure can enhance conductivity and offer additional ion migration pathways, thus boosting the efficiency of energy storage systems[9].

Recent studies have indicated that integrating graphene with various dopants or nanomaterial composites can enhance the electrocatalytic activity and energy conversion efficiency of metal-air batteries, such as zinc-air and lithium-air batteries. Furthermore, modifying the graphene surface can optimize ion migration pathways and bolster the stability of the electrode structure, which is pertinent to improving the ion conductivity of the electrolyte membrane in aluminum-air batteries[11]. Graphene oxide serves as an affordable and sustainable proton exchange membrane, offering good ion conductivity and high chemical resistance, thus making it a potential material for applications in energy storage and conversion [12].

Additionally, combining graphene with carbon nanomaterials, such as carbon quantum dots (CQDs), has been reported to broaden the ion transport pathways and enhance the interface characteristics in electrochemical systems. This discovery underscores the potential of utilizing CQDs derived from biomass waste, such as coconut shell charcoal, and GO as functional materials to boost the ion conductivity of electrolyte membranes in aluminum-air batteries[13].

Moreover, integrating carbon-based nanoparticles can substitute liquid electrolytes with nanocomposite-based solid electrolytes, thereby

offering a safer alternative for improving ion transport. This is particularly significant for the use of Carbon Quantum Dots (CQDs) derived from coconut-shell charcoal, as their conductive properties provide an effective ion transfer route. When combined with Graphene Oxide (GO), which features a layered structure with a substantial surface area, the two can form a hybrid conductive pathway within the electrolyte membrane, enhancing the efficiency of aluminum-air batteries [14].

According to several studies, combining CQDs derived from coconut shell charcoal with Graphene Oxide (GO) is believed to create an efficient hybrid conductive pathway within the electrolyte membranes of aluminum-air batteries[9], [15], [16]. This integration is likely to enhance ion mobility, decrease internal resistance, and bolster the structural stability of the membrane, thereby directly enhancing the electrochemical performance and energy efficiency of aluminum-air batteries [16,17]

Despite the numerous benefits previously mentioned, it is crucial to highlight that research on the integration of Graphene Oxide (GO) and Carbon Quantum Dots (CQDs), particularly those sourced from coconut shell charcoal biomass in aluminum-air battery systems, remains scarce[10,18]. This suggests that there is still a significant opportunity to investigate the synergistic effects of GO and CQDs in enhancing the ion conductivity and stability of electrolyte membranes, making it a highly pertinent area for future research [19].

This study addresses the existing research gap by synthesizing Carbon Quantum Dots (CQDs) from coconut shell charcoal biomass using a straightforward, cost-effective, and eco-friendly approach [20]. Additionally, the synthesized CQDs were incorporated into a graphene oxide (GO)-based electrolyte membrane matrix to examine the alterations in the physical and electrochemical characteristics of the resulting membrane [21].

This study aims to assess several critical factors, including ionic conductivity, internal resistance,

membrane morphology, ion interactions at the membrane interface, and membrane stability within the electrochemical environment typical of aluminum-air battery systems[22]. The expectation is that combining CQDs derived from coconut shell charcoal with GO will result in membranes that outperform traditional membranes lacking nano-carbon materials [15],[23].

The findings of this research are anticipated to make substantial scientific contributions to advancing energy storage technologies, with a focus on more efficient and eco-friendly aluminum-air batteries[17],[18]. Moreover, incorporating materials derived from biomass waste, such as coconut-shell charcoal, aligns with sustainable development initiatives. This approach emphasizes the growing importance of utilizing local, cost-effective, and renewable resources in the evolution of future technologies[19],[24].

This study not only advances scientific knowledge in the realm of electrochemical materials but also positively influences environmental and resource sustainability [20,21]. By innovatively incorporating CQDs and GO into the electrolyte membrane, it is anticipated that the aluminum-air battery system will achieve notable enhancements in performance, particularly in terms of ionic conductivity, cycle stability, and mitigating corrosion effects, which have been significant obstacles in the development of this battery type [22].

2. MATERIAL AND METHOD

2.1. Materials

In this study, Graphene Oxide was used as the main matrix for creating membranes (Figure 1). PVA (MW ~60,000), sourced from Merck, was utilized as an additional polymer to enhance the physical properties of the membrane. To boost ionic conductivity, Hydrochloric Acid (hydrogen chloride) was added to the matrix. Carbon Quantum Dots (CQDs) were synthesized using coconut-shell charcoal activated carbon as a starting material through a microwave-assisted hydrothermal method. Sulfuric Acid (H₂SO₄) and Nitric Acid (HNO₃), also from Merck, were used for the

oxidation and functionalization of the CQDs production process. Nanofibers, used as composite materials, were prepared via electrospinning from Polylactic Acid PLA (MW ~ 60, 000) using Dimethylformamide and Acetone as solvents. The quality and purity of all the components in this study were meticulously checked to ensure consistency and dependability

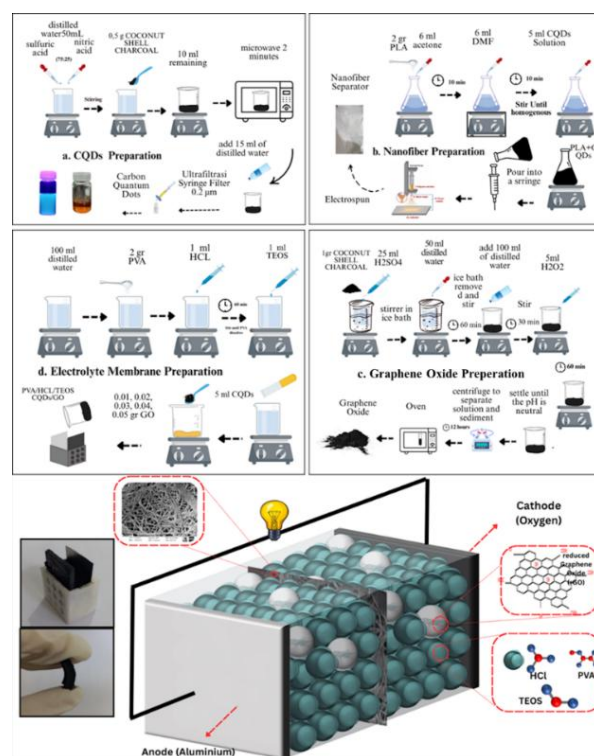


Figure 1. Aluminum air battery schematic

2.2. Preparation of Carbon Quantum Dots

The coconut shell charcoal was precisely weighed to 500 mg. Distilled water (20 mL) was added to a beaker and stirred while being heated with a magnetic stirrer. Sulfuric acid (H₂SO₃) and nitric acid (HNO₃), sourced from Merck, were introduced into a beaker at a 75:25 ratio. Following the addition of the strong acid, the coconut-shell charcoal was incorporated into the beaker, and the mixture was stirred for 10 min at a speed of 125 rpm. After this initial stirring, 3 mL of dimethylformamide (DMF; Merck) was added to the solution, which was then continuously stirred for another 20 min. Once stirring was complete, the solution was transferred to a 50 mL flask. The flask was placed in a microwave oven and heated for 2 min to accelerate the synthesis process. After microwave heating, 10

mL of distilled water was added to the flask and the mixture was stirred vigorously. The resulting solution was filtered through a 0.22 μm syringe filter to eliminate contaminants. The filtered Carbon Quantum Dots (CQDs) were subsequently stored in a sterile container for future use[25]

2.3. Nanofiber Preparation

Initially, 2 g of Polylactic Acid (PLA) was weighed using a digital scale. This was then dissolved in a 1:1 solution of Dimethylformamide (DMF) and Acetone, creating a total volume of 10 ml. The solvent mixture was transferred to a 50 mL flask and stirred with a magnetic stirrer while being gently heated for 5 minutes. Once the solvent was well-mixed, PLA was introduced into the solution. The dissolution of PLA took 30 minutes until the solution became uniform. This homogeneous solution was subsequently utilized in an electrospinning process to create nanofibers. The electrospinning setup includes several crucial components, such as a power source, syringe, brushed DC motor, and collector [26]. The nanofiber production was conducted at a feed rate of 0.3 ml/min, employing a high-voltage power source operating at 20 kV (10 mA). A syringe with a 38 mm diameter (18G x 1½") was used in this process.

2.4. Electrolyte Membrane Preparation

Polyvinyl Alcohol (PVA) was weighed to a maximum of 1.5 g. Once both matrices were ready, 100 mL of distilled water was poured into a beaker, stirred, and heated using a magnetic stirrer for 5 min at a speed of 100 rpm. Subsequently, 2 mL of hydrochloric acid was added to distilled water to aid in dissolving the matrix, and the measured PVA was then incorporated into the solution. To enhance the composite properties, 1 g of TEOS was added with continuous stirring. Stirring was sustained for 60 min at 150 rpm to ensure an even distribution. In this study, the electrolyte membrane formulation was developed in three distinct variations: (1) a base solution comprising PVA, HCl, and TEOS; (2) a base solution with an additional 5 mL of Carbon Quantum Dots (CQDs); and (3) a base solution with

an added 0.03 g of Graphene Oxide (GO). Each variation was crafted to assess the impact of additive materials on the structure and performance of the resulting electrolyte membrane. Carbon Quantum Dots (CQDs) at a concentration of 5 ml were gradually introduced into the solution. Once the stirring process was completed, the homogeneous solution was transferred to a Petri dish with a 10 cm diameter for drying. Initially, half of the solution was poured out, followed by the placement of a preformed nanofiber layer on top. The remaining solution was then applied to the nanofiber to ensure coverage across the surface. The resulting electrolyte membrane was dried at room temperature for 72 h. Similarly, Graphene Oxide (0.03 g) was added to the solution. After completing the stirring process, the homogeneous solution was transferred to a 10 cm diameter petri dish for drying. Initially, half of the solution was poured out, followed by the placement of a preformed nanofiber layer on top[27]. The remaining solution was then applied over the nanofiber, ensuring even coverage across the surface. The resulting electrolyte membrane was allowed to dry at room temperature for 72 hours.

2.5. Characterization

Fourier Transform-Infrared (FT-IR) spectroscopy is employed to detect molecular vibrations, analyze compounds, and determine absorption intensity at particular wavelengths. The FTIR analysis was conducted using a PerkinElmer Frontier C90704 Spectrum IR Instrument Version 10.6.1 [28]. X-ray diffraction (XRD) was performed at a wavelength of 1.54060 Å with copper (Cu) serving as the anode. The XRD analysis was executed at 40 kV and 30 mA. Electrochemical Impedance Spectroscopy (EIS) was conducted using a potentiostat device, covering a frequency range from 100000 to 0.01 Hz. Ionic conductivity was evaluated through impedance test data and calculated using the standard ionic conductivity formula [29], [30]. Cyclic voltammetry was carried out with a potentiostat device at a scan rate of 1 mV. This

analysis was performed at room temperature (32°C).

3. RESULTS AND DISCUSSION

3.1. Fourier-Transform- Infra-Red (FTIR)

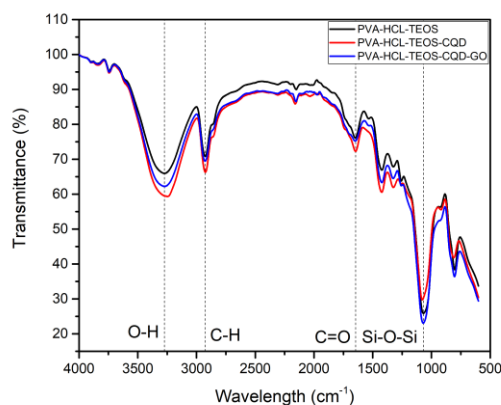


Figure 2. FTIR of three samples

Figure 2 shows the FTIR spectra of three sample compositions: PVA-HCL-TEOS, PVA-HCL-TEOS-CQD, and PVA-HCL-TEOS-CQD-GO. These spectra exhibit several characteristic peaks that represent the presence of major functional groups in composite materials.

The broad absorption peak appearing at around 3300 cm^{-1} indicates the stretching vibration of hydroxyl (–OH) groups and possibly also amine (–NH) groups, which are characteristic of PVA and hydrolyzed silica. The decrease in intensity in this region after the addition of CQDs and GO indicates the presence of hydrogen interactions between functional groups, such as between OH and oxygen functional groups in CQD/GO[31].

The absorption peak at around 2900 cm^{-1} is attributed to the symmetrical stretching of the C–H groups, which are part of the PVA polymer chain structure. The peak in the region of ~1600 cm^{-1} indicates the stretching vibration of the carbonyl group (C=O). This peak becomes more pronounced in the spectra of CQDs and GO, indicating the presence of carbonyl groups derived from surface oxygen on Carbon Quantum Dots (CQDs) and Graphene Oxide (GO) [32].

Furthermore, the peak appearing at around 1060 cm^{-1} is related to the stretching vibration of Si–O–Si groups, which is the result of silanol condensation during the sol-gel process using TEOS (Tetraethyl Orthosilicate). The addition of CQDs and GO causes the intensity of this peak to increase, indicating the interaction between the silica matrix and the reinforcing nanoparticles. Overall, the changes in the intensity and shape of the peaks in the FTIR spectrum confirm that the addition of CQDs and GO has an effect on the chemical structure and intermolecular interactions in the composite material, which has the potential to improve the mechanical properties and ionic conductivity of the electrolyte membrane [33].

3.2. X-Ray Diffraction (XRD)

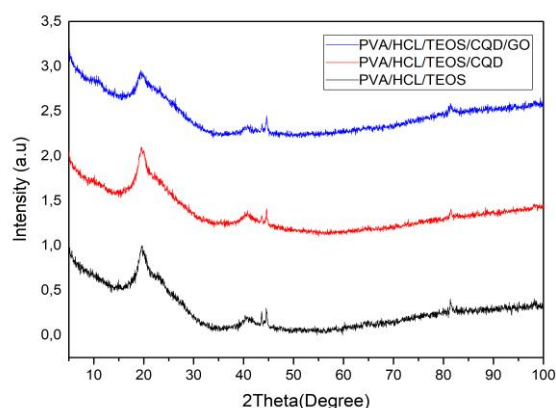


Figure 3. XRD of three samples

Figure 3 shows the results Based on the results of X-ray Diffraction (XRD) characterization of three membrane samples, namely PVA/HCL/TEOS, PVA/HCL/TEOS/CQD, and PVA/HCL/TEOS/CQD/GO. Based on X-ray Diffraction (XRD) characterization of PVA/HCL/TEOS, PVA/HCL/TEOS/CQD, and PVA/HCL/TEOS/CQD/GO membranes, a dominant diffraction peak consistently appeared around $2\theta = 19.3^{\circ}$ – 19.7° , which is attributed to the semi-crystalline nature of the PVA matrix. The incorporation of CQDs influenced the diffraction pattern by modifying peak intensity, although CQDs themselves are generally amorphous or poorly crystalline[34]. An additional peak at $2\theta = 11.11^{\circ}$ was observed in the

PVA/HCl/TEOS/CQD/GO membrane, corresponding to the typical diffraction peak of graphene oxide (GO), indicating its successful incorporation. Minor peaks between 40° – 45° and 81° – 98° suggest alterations in the crystalline structure, possibly due to TEOS-derived silica network formation and further material interactions.

3.3. Electrochemical Impedance Spectroscopy (EIS)

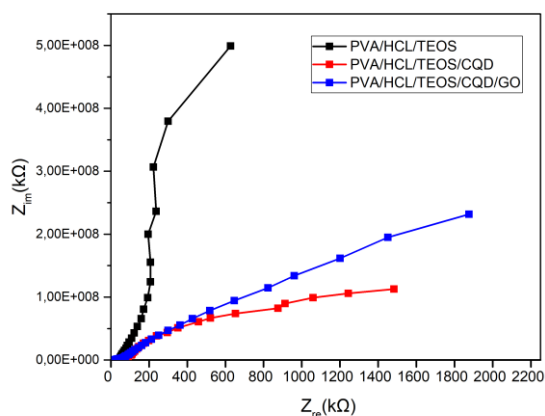


Figure 4. EIS of three samples

Figure 4 shows the results of the Electrochemical Impedance Spectroscopy (EIS) test on three samples: PVA/HCl/TEOS, PVA/HCl/TEOS/CQD, and PVA/HCl/TEOS/CQD/GO. The Nyquist graph displays the imaginary impedance value (Z_{im}) on the Y-axis and the real impedance (Z_{re}) on the X-axis. These data were measured over a frequency range of 100,000 Hz to 0.01 Hz with potentiostatic conditions at 0 mV DC and an AC signal voltage of 10 mV RMS for 20 minutes per sample.

Based on the graph, it can be seen that the PVA/HCl/TEOS curve (black) shows the highest total impedance value, which indicates the largest ionic resistance of the three samples. After the addition of Carbon Quantum Dots (CQD) (red curve), the impedance value decreased significantly, reflecting an increase in ionic conductivity in the membrane [25]. This decrease is more evident in the sample with the addition of Graphene Oxide (GO) (blue curve), where the impedance value is lower than the previous two samples, indicating that the combination of CQD

and GO is able to improve the ion conduction path in the membrane structure.

This decrease in impedance value can be attributed to the increased hydrophilic nature of the material due to the presence of functional oxygen groups on the surface of CQDs and GO, which facilitates the formation of conductive pathways for H^+ or OH^- ions [29]. This supports previous findings that the addition of CQDs and GO into a polymer matrix can create an intermediary network for more efficient ion transfer [15], [23]. Thus, these EIS data confirm that the combination of CQDs and GO in the PVA/HCl/TEOS system effectively lowers the ionic resistance of the membrane, making it a promising candidate for applications such as electrolyte membranes in fuel cells or aluminum-air batteries [8, 15].

3.4. Ionic conductivity

Ion Conductivity testing shows the battery conductivity produced by each membrane containing PLA, HCL, TEOS fibers with variations of CQD and Graphene Oxide.

Table 1. Ionic conductivity

Variation	$Z_{re}(\Omega)$	$Z_{im}(\Omega)$	Conductivity (mS/cm)
PVA/HCL/TEOS	627.2	4.9e8	8.02×10^{-9}
PVA/HCL/TEOS/CQD	1876.3	2.3e8	1.73×10^{-8}
PVA/HCL/TEOS/CQD/GO	1482.8	1.1e8	3.55×10^{-8}

Ion conductivity testing demonstrated the ion conductivity values produced by each PLA fiber-based membrane with varying membrane compositions. Based on the measurements, the PVA/HCl/TEOS membrane showed the lowest ion conductivity value at 8.02×10^{-3} mS/cm. This was due to the absence of additives such as Carbon Quantum Dots (CQDs) or Graphene Oxide (GO), which play a role in enhancing ion migration pathways [24].

After the addition of CQDs, the conductivity value increased to 1.73×10^{-2} mS/cm. This increase indicates that CQDs contribute to increasing the number of active functional groups and expanding the intermolecular contact surface area, allowing for

more efficient ion mobility. Koduru et al. (2018) reported room-temperature ionic conductivities of 1.56×10^{-4} mS/cm for PEO/PVP/NaIO₄ and 1.89×10^{-3} mS/cm for PEO/PVP/NaIO₄/GO [29], both markedly lower than the value obtained in this study (3.55×10^{-2} mS/cm). The superior conductivity of the PVA/HCl/TEOS/CQD/GO membrane can be attributed to the synergistic effect of incorporating CQDs alongside GO, which provides additional ion migration pathways, enhances interfacial interactions, and improves ionic transport compared to membranes containing only GO.

Therefore, the addition of these two materials has been shown to enhance the ion conduction capacity of the membrane, thus providing optimal performance for aluminium-air battery applications [30].

This study successfully synthesized and characterized a PVA membrane integrated with Carbon Quantum Dots and Graphene Oxide, demonstrating its feasibility as an alternative electrolyte membrane for aluminium-air batteries. The use of Carbon Quantum Dots significantly improved the structural, chemical, and functional characteristics of the membrane, as demonstrated by the increase in ionic conductivity [31]. FTIR characterization results verified the effective integration of hydroxyl groups, which further improved the membrane's characteristics. Furthermore, EIS testing showed a decrease in membrane impedance after the addition of CQDs, consistent with the increase in ionic conductivity.

4. CONCLUSION

Based on the analysis of bio-briquette characteristics from a mixture of rubber seed shell charcoal and sugarcane bagasse charcoal using different types of binders, two characteristic parameters were found to meet the standard values across all binder variations: moisture content and ash content. The optimum bio-briquette parameter for the mixture of rubber seed shell and sugarcane bagasse charcoal using various binders was achieved with a 90:10 ratio using tapioca flour as the binder. This composition demonstrated the best overall performance in terms of quality and

compliance with established standards.

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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