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Preliminary Investigation on Enhanced Hydrogen Generation via Carbon Nanodots–Sulfonated Polyoctahedral Silsesquioxane Layer Modified Anode in Water Electrolysis

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ABSTRACT

This study investigated the preliminary electrochemical performance of carbon anodes coated with a composite of Carbon Nanodots (CND) and Sulfonated Polyoctahedral Silsesquioxane (SPOSS) for water electrolysis. The coatings were applied in varying ratios of CND to SPOSS (1:0, 1:1, 0:1) and at different layer thicknesses (3 and 5 layers) simulating membrane-like interfaces for enhanced electrode–electrolyte interaction. Electrochemical performance was evaluated through hydrogen production rates in a 2 wt% NaOH electrolyte. Scanning Electron Microscopy (SEM) was used to confirm the uniformity and integrity of the coatings, while UV-Vis Spectroscopy was employed to assess any potential leaching of coating materials into the electrolyte. Results indicated that electrodes coated with CND:SPOSS (1:1) modification with three coating layers improved both catalytic activities, achieving the highest hydrogen production rate of 0.51 cm^3/s. Excessive coating thickness, particularly at five layers, led to aggregation observed in the SEM images, which resulted in a reduced hydrogen production rate and overall performance. The 3-layer CND:SPOSS (1:1) coating is optimal for hydrogen production performance due to its uniform, crack-free structure and enhanced electrochemical activity, while the 3-layer CND:SPOSS (0:1) coating is best for minimizing leaching, as it shows the least absorbance increase. These preliminary findings highlight the potential of nanocarbon-based surface modification as a membrane-inspired strategy to enhance hydrogen production efficiency in alkaline water electrolysis systems.

Keywords: Carbon Nanodots (CND), Sulfonated Polyoctahedral Silsesquioxane (SPOSS), Anode Layers, Alkaline Water Electrolysis, Hydrogen Generation

1.0 INTRODUCTION

The pursuit of sustainable and low-emission hydrogen production has led to renewed interest in alkaline water electrolysis, a well-established and scalable technique for generating hydrogen from water. However, its efficiency remains constrained by the sluggish kinetics of the oxygen evolution reaction (OER) at the anode, which demands significant overpotential and limits overall system performance [1]. Enhancing anode activity and durability in strongly alkaline conditions is therefore critical to advancing electrolysis technologies [2,3]. Carbon-based electrodes, widely appreciated for their conductivity, affordability, and chemical resistance, have emerged as promising candidates for surface modification strategies aimed at improving OER performance [4,5].

Among various nanomaterials, carbon nanodots (CNDs) have garnered

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attention due to their high surface area, excellent electron mobility, and catalytic properties [6]. Their ability to form stable, functional coatings makes them ideal for enhancing electrochemical interfaces. CNDs also possess unique optical properties, chemical stability, and biocompatibility, making them ideal for use in electrochemical applications [7, 8]. CNDs was also modified as an electrodes in detection of various organic materials in water [9, 10]. To further enhance the properties of CNDs, this study investigates their combination with Sulfonated Polyoctahedral Oligomeric Silsesquioxane (SPOSS), a material known for its stability and resistance to harsh environments [11]. POSS with different functional group other than sulfonated was also reported as an anode or electrolyte material applicable for lithium ion battery [12,13]. The nanocomposites materials would demonstrate a synergistic effect, combining light absorption, catalytic efficiency, and resistance to degradation [14]. Enhancing the anode layer through surface modification with nanostructured composite coatings such as CND and SPOSS offers a membrane-inspired approach to improving interfacial properties, catalytic activity, and ionic transport. CNDs has been reported as an electrolyte membrane component, typically by being embedded into a polymer matrix, to enhance proton conductivity, mechanical properties, and thermal stability [15,16]. This strategy mirrors the design principles of functional membranes, where tailored surface chemistry and morphology are critical for optimizing electrochemical performance and long-term stability in hydrogen production systems.

The primary objective of this study is to evaluate the impact of different CND:SPOSS ratios (1:0, 1:1, and 0:1) and coating thicknesses (1, 3, and 5 layers) on the electrochemical performance of the coated carbon anodes during alkaline water electrolysis. The study will assess the effect of these parameters on hydrogen production rates and electrode stability. By evaluating different CND:SPOSS ratios and coating thicknesses, this work aims to identify optimal configurations that balance catalytic efficiency, structural integrity, and chemical stability. The insights gained contribute to the broader development of membrane-integrated electrochemical systems for efficient and durable hydrogen generation.

2.0 METHODS

2.1 Preparation of Carbon Nanodots (CNDs)

In this study, the CNDs were synthesized through top-down or bottom-up methods [17] without modification. 1.0 g of citric acid was mixed with 0.35 g of 1, 2-ethylenediamine (EDA). This mixture was then dissolved in 1 mL of water. The solution was then put into a glass tube oven and heated at 240 °C for 30 min. A red-brown and foamy solid was formed. 10 wt. % of CNDs was suspended in the DMSO solution for further used.

2.2 Preparation of Sulfonated POSS

In this study, SPOSS was synthesized via a sulfonation process involving chlorosulfonic acid. The SPOSS was synthesized follows the method from previous study [18]. Octaphenyl-POSS (3 g, 2.9 mmol) were first dried in an oven at 80°C for more than 24 h to remove moisture content. The dried POSS were added to chlorosulfonic acid ($ClHO_3S$, 60 ml, 900 mmol) and was stirred for 24 h at 50°C in an oil bath under Ar gas conditions. Unreacted $ClHO_3S$ was removed by vacuum distillation at 110 °C. The crude products were washed with water several times and the excess water was evaporating under normal heating. The brownish liquid products designated as SPOSS was formed and was carefully stored in close container for further used.

2.3 Fabrication of Modified Carbon Anodes

The dip-coating method was chosen for this study to apply CND:SPOSS composite coatings onto carbon anodes. This technique involves the immersion of carbon rods into a slurry of CND:SPOSS and Polyvinylpyrrolidone (PVP) as a binder as shown in Figure 1. The PVP solution was prepared by dissolving 5 wt% of PVP in deionized water and stirring it for 24 hours to ensure complete dissolution. The slurry was then divided into three separate tubes, with varying 1wt% CND:SPOSS ratios (1:0, 1:1 and 0:1). The carbon rods (electrode dimensions, D=0.8 cm, L=5 cm, A= 6.79 cm^2) were dipped into the solutions for a fixed duration of 10 minutes, followed by drying at 130°C for 30 minutes. The coating process was repeated for 1, 3 and 5 layers to determine the effect of coating thickness on electrochemical performance [19].

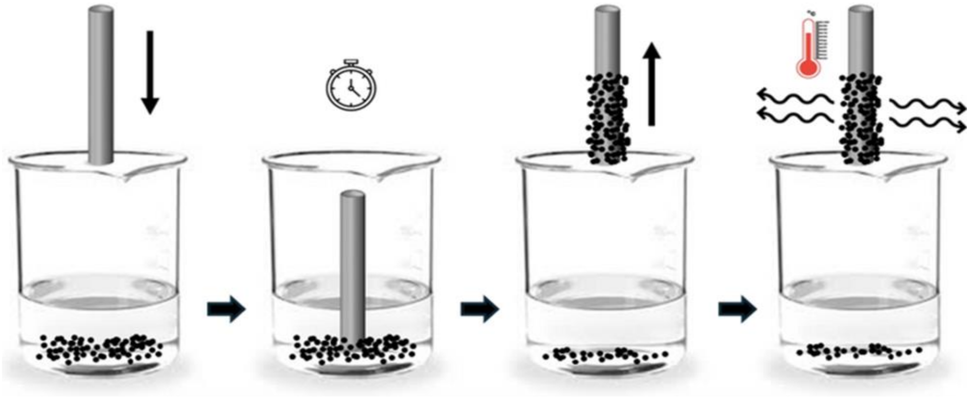

Figure 1 Modification of carbon anodes through dip coating technique

2.4 Characterization of Modified Carbon Anode

In this study, Scanning Electron Microscopy (SEM) was employed primarily to verify the successful deposition of CND:SPOSS nanocomposite coatings on the surface of carbon anodes. The SEM images was performed to compare the morphological structure of coated samples with different CND:SPOSS loadings also aids in provides essential evidence of the physical integrity and consistency of the dip-coated layers.

UV-Vis spectroscopy was employed to detect any leaching of nanomaterials into the electrolyte. This method works by measuring the absorbance of ultraviolet and visible light across a specific wavelength range as it passes through liquid samples, thereby revealing the presence of dissolved substances based on their spectral signatures.

2.5 Hydrogen Production Performance

Hydrogen Production was evaluated using a custom-designed electrolysis setup incorporating a water displacement gas collection system. The working electrode comprised carbon anodes coated with CND-SPOSS at varying concentrations and layer thicknesses, while the cathode remained unmodified. A 2 wt% NaOH aqueous solution was used as the electrolyte. The electrolysis was connected to 9 V applied voltage and experiment was conducted at room temperature. As electrolysis proceeded, hydrogen gas evolved at the cathode and displaced the electrolyte in a calibrated hydrogen collection chamber. The time taken to reach a fixed displacement height of 3 cm was recorded, with shorter durations indicating higher hydrogen evolution rates. This setup enabled consistent and comparative assessment of hydrogen production efficiency across different electrode configurations.

3.0 RESULTS AND DISCUSSION

3.1 Morphological Structure of Coated Carbon Anode

The SEM images of the carbon anodes coated with different CND:SPOSS ratios and layers reveal distinct morphological characteristics that influence the electrochemical performance. The anodes with 3 layers of CND:SPOSS 1:0 (Figure 2 (b)) show a relatively smooth texture, but with visible cracks, suggesting incomplete adhesion and possible structural weaknesses. These imperfections may limit the effectiveness of the coating in promoting efficient electrochemical reactions. In contrast, the 3 layers of CND:SPOSS 1:1 (Figure 2 (c)) present a more uniform and stable surface, with fewer cracks. This improved morphology is attributed to the balanced combination of CNDs and SPOSS, which enhances both the coating's mechanical integrity and electrochemical activity, making it more suitable for hydrogen production.

The 3 layers of CND:SPOSS 0:1 (Figure 2 (d)) coating, dominated by SPOSS, shows a rougher and more irregular surface. The larger particles of SPOSS reduce the available surface area for catalysis, which could hinder the anode's overall electrochemical efficiency. While SPOSS contributes to the mechanical stability of the coating, the lack of CNDs for better conductivity and active surface area could limit its hydrogen production performance. Meanwhile, for the 5-layer coatings, the CND:SPOSS 1:0 (Figure 2 (e)) sample exhibits a more uneven, porous surface, which could increase the surface area but at the cost of stability.

This might negatively affect the coating's long-term performance. On the other hand, the 5 layers of CND:SPOSS 1:1 (Figure 2 (f)) shows more dense and uniform coating, which combines both the high conductivity of CNDs and the stability of SPOSS, might lead to a more effective electrochemical surface for hydrogen production. The 5 layers of CND:SPOSS 0:1 (Figure 2 (g)) appear compact but with reduced catalytic surface area, which may enhance stability but limit performance.

SEM images for the single coatings are absent from this analysis because they showed little evidence of surface alteration. In fact, the microscopically examined surface of the one-layer samples appeared nearly identical to that of the unmodified carbon rod. This lack of improvement agrees with the work of Orozco-Barrera *et al.* [20] who reported similar findings on carbon-based electrodes modified with single-layer coating. Their investigation further pointed out that only multi-layer deposits meaningfully enhance surface features, increase the number of accessible active sites, and raise the overall electrochemical performance properties [21].

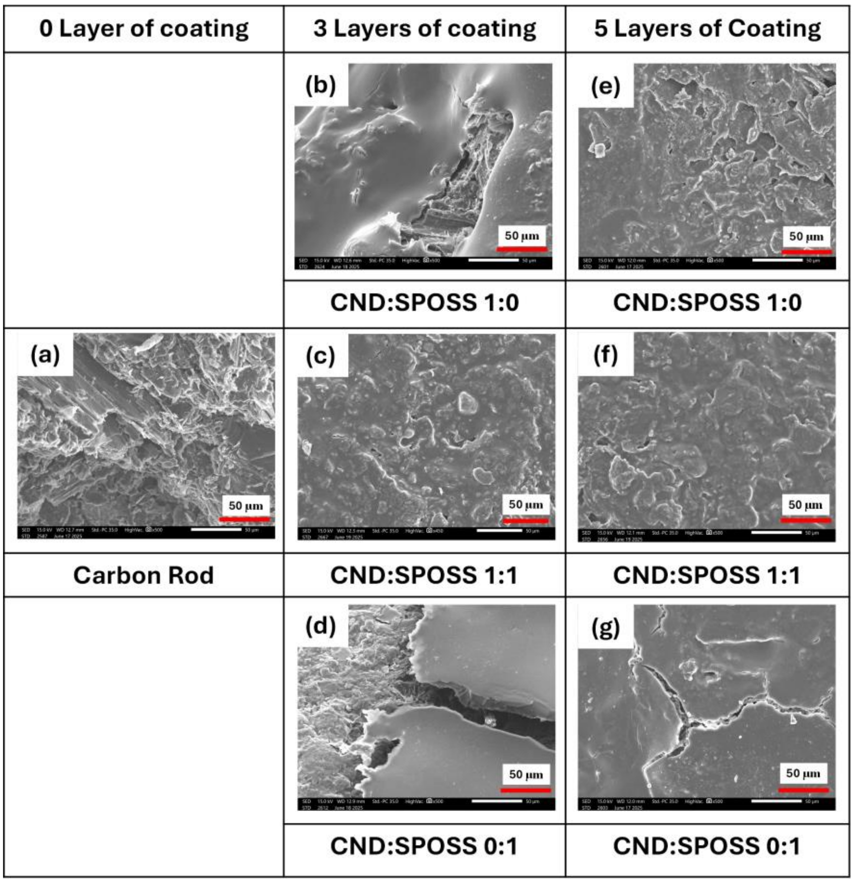

Figure 2 SEM image of (a) uncoated (carbon rod) and coated carbon anode with different layers and CND:SPOSS loadings (b) 3 layers of CND:SPOSS 1:0 (c) 3 layers of CND:SPOSS 1:1 (d) 3 layers of CND:SPOSS 0:1 (e) 5 layers of CND:SPOSS 1:0 (f) 5 layers of CND:SPOSS 1:1 (g) 5 layers of CND:SPOSS 0:1

3.2 Physical Properties of Coated Carbon Anodes

The UV-VIS absorbance measurements revealed that the NaOH electrolyte exhibited a characteristic peak at 289 nm wavelength, with an absorbance value of 1.1119. This peak served as the baseline reference for evaluating the optical properties of the CND:SPOSS coatings. When moving to the 5-layer coatings, the absorbance values increase further, suggests significant dissolution of the coating material, likely due to instability in the coating, or weak adhesion CND/SPOSS had with the electrode surface. CND:SPOSS (1:0) shows a value of 2.141, while CND:SPOSS (1:1) is slightly lower at 2.124, and CND:SPOSS (0:1) has an absorbance value of 2.0894. These increased absorbance values might suggest that there is leaching from the coating into the electrolyte after the electrolysis experiment. The coating's potential to leach can be influenced by the number of layers and the concentration of CND:SPOSS, as well as the conditions of the electrolysis experiment [22]. This leaching effect could contribute to the observed increase in absorbance after the experiment, which needs further investigation to determine its exact impact on the overall performance and stability of the anode.

Based on the UV-VIS absorbance data, the CND:SPOSS (1:0) coating with 3 layers exhibits the increasing absorbance (1.8113), followed by the CND:SPOSS (1:1) with 3 layers at 2.1391. Both of these coatings show a significant increase in absorbance compared to the NaOH baseline (1.1119), suggesting that leaching may be occurring. The CND:SPOSS (0:1) coating, with a ratio of 0:1, shows the lowest absorbance values in both the 3-layer (1.1417) and 5-layer (2.0894) coatings compared to the other coatings. This indicates less interaction or leaching of the material into the electrolyte. Low leaching is expected due to SPOSS's highly cross-linked, cage-like inorganic–organic hybrid structure. The least absorbance increase for this configuration, confirming minimal release. The CND-rich coatings showed higher absorbance, indicating greater leaching, likely due to weaker matrix encapsulation and higher surface exposure.

The leaching behaviour observed here is likely related to the dip-coating technique utilized for applying the CND/SPOSS to the electrodes [23]. In this method, the electrode is dipped multiple times in a solution while allowing it to dry in between each dip. However, insufficient drying time coupled with multiple steps may impair the ability of the PVP binder to fully bind the CNDs material onto the electrode surface. Chen *et al.* [24] found that similar leaching in electrocatalysts occurred due to instability in the catalyst structure during electrochemical cycling, leading to material dissolution and degradation of performance. Their study demonstrated that increased current density contributed to higher rates of leaching.

Future work should focus on fine-tuning the dip-coating setup to improve coating uniformity and minimize leaching. Minimizing leaching is critical for maintaining membrane integrity, preventing contamination of the electrolyte, and ensuring consistent electrochemical performance. One proposal is to substitute the current binder with a stronger formulation and then extend the drying time, thereby strengthening adhesion between the carbon-nitride layer and the electrode surface.

Among the three CND:SPOSS ratios tested, the 0:1 (SPOSS) coating shows the least absorbance increase, especially for the 3-layer coating. Therefore, in this particular study, it can be concluded that CND:SPOSS (0:1) with 3 layers is likely the optimal coating for minimizing leaching.

Table 1 UV-VIS absorbance values of CND:SPOSS coated carbon Anodes

Coating Layers	**Wavelength, nm**	**NaOH**	**CND:SPOSS 1:0**	**CND:SPOSS 1:1**	**CND:SPOSS 0:1**
3	289	1.1119	1.8113	2.1391	1.1417
5	289	1.1119	2.141	2.124	2.0894

3.3 Hydrogen Production Performance

The hydrogen production rates for each sample configuration, including various CND:SPOSS ratios (1:0, 1:1, and 0:1) and coating thicknesses (1, 3, and 5 layers), are presented in the Figure 3. The data were obtained through a custom-designed gas collection system based on water displacement, and the results reflect the hydrogen gas evolution rates at different experimental conditions.

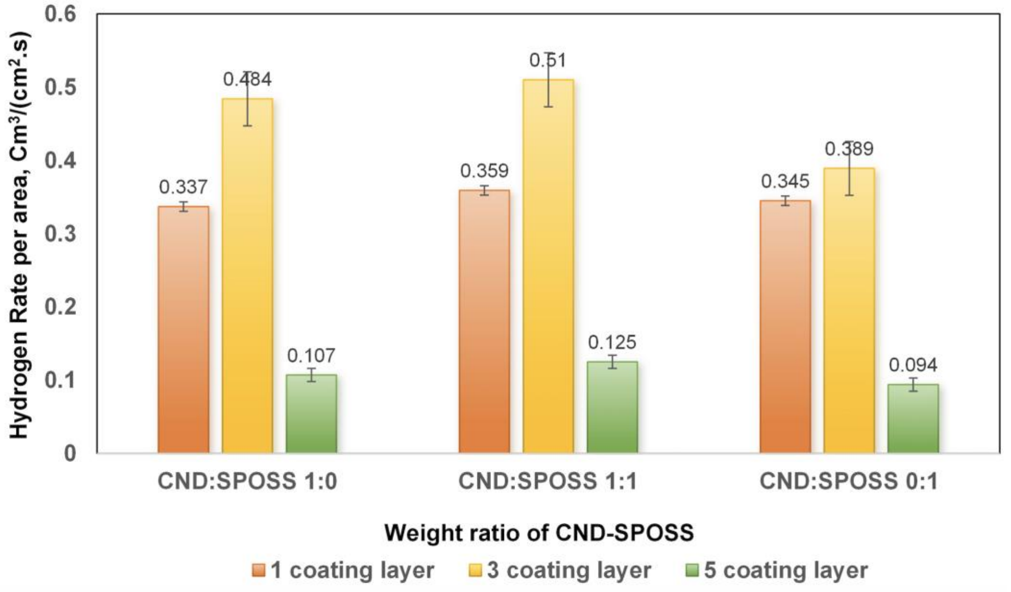

Figure 3 Hydrogen evolution rate

From Figure 3, it shows that the experimental results demonstrate that the CND-SPOSS composite coating significantly influences hydrogen rates over effective area of modified anode during electrolysis. Among the tested configurations, the CND:SPOSS 1:1 ratio with 3 coating layers exhibited the highest hydrogen evolution rate. This superior performance can be attributed to the synergistic combination of carbon nanodots (CNDs) and sulfonated polyoctahedral silsesquioxane (SPOSS), which together optimize both electron transfer and proton conduction pathways essential for efficient hydrogen evolution reactions [25].

The enhanced performance of the 1:1 composition stem from complementary properties of its components. CNDs contribute excellent electron conductivity and catalytic active sites, while SPOSS provides proton-conducting sulfonic acid groups that facilitate proton mobility [26,27]. This balanced combination creates an ideal environment for the hydrogen evolution reaction. Furthermore, the 3-layer coating thickness appears optimal, providing sufficient catalytic sites without introducing excessive resistance that could hinder charge transfer. Thinner 1-layer coatings showed reduced activity likely due to insufficient catalytic site density, while thicker 5-layer coatings may have suffered from increased ohmic resistance and limited electrolyte penetration to the electrode surface.

The pure component coatings (CND:SPOSS 1:0 and 0:1) demonstrated lower hydrogen production rates, highlighting the importance of the composite approach.

The pure CND coating (1:0) lacked proton-conducting capability, while the pure SPOSS coating (0:1) suffered from insufficient electron conductivity. These results emphasize that both electron transfer and proton conduction must be optimized simultaneously for efficient hydrogen evolution, as neither component alone provides the complete set of properties needed for optimal performance [28].

The findings suggest that coating thickness plays a crucial role in electrolysis performance. While increasing layers initially improves performance by providing more active sites, excessive thickness can become detrimental. The 3-layer coating represents a balance between maximizing catalytic surface area and maintaining efficient charge and mass transport. This observation aligns with previous studies showing that electrode modifications must carefully consider thickness effects to avoid compromising the benefits of surface modification [29].

4.0 CONCLUSION

The results demonstrated that the CND and SPOSS coatings significantly enhanced hydrogen production rates especially in CND:SPOSS (1:1) with 3-layer coating. The increase in hydrogen production was attributed to the enhanced surface area, catalytic properties of CNDs, and improved conductivity from SPOSS highlighting the importance of composite coatings. SEM analysis confirmed that all modified anodes were successfully coated, though with some variability in uniformity and adhesion. UV-VIS spectroscopy detected leaching across all coatings, with SPOSS showing the minimal release attributed to its highly cross-linked, cage-like inorganic-organic hybrid structure that improves chemical stability and mechanical robustness. To further enhance coating quality, optimizing dip-coating parameters could improve layer-by-layer uniformity, thereby minimizing leaching and defects. Overall, the integration of CND and SPOSS into electrode coatings presents a promising membrane-inspired strategy for enhancing both the efficiency and durability of electrochemical systems for hydrogen production, with future work directed toward long-term operational stability and scalable membrane fabrication.

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CONFLICTS OF INTEREST

The authors declare that there is no conflict of interest regarding the publication of this paper.

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