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Nitrogen Surface Passivation of Biomass-Based Carbon Quantum Dots: A Pathway to Enhanced Colloidal Stability and Electrochemical Capacitance

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ABSTRACT

The conversion of organic biomass wastes into carbon quantum dots (CQDs) offers a sustainable pathway to reduce environmental pollution and valorize renewable resources. However, pristine biomass-derived CQDs typically exhibit low electrical conductivity, a wide band gap, and poor dispersion stability, limiting their practical applicability in energy storage systems. This study aims to address these challenges through nitrogen doping, which enhances charge transport, narrows the band gap, and improves colloidal stability. Two synthesis routes; direct carbonization and in-situ nitrogen modification were systematically compared to determine the most effective approach. Characterization using photoluminescence, UV-Vis spectroscopy, and zeta potential analysis confirmed that nitrogen incorporation effectively improved particle dispersion and surface passivation. The resulting nitrogen-doped CQDs (NCQDs) exhibited markedly higher conductivity of 1.7 mS cm^{-1} and dispersion stability, leading to a substantial enhancement in the electrochemical performance when integrated into activated carbon (AC/NCQD) composite electrodes. The optimized in-situ modification route yielded the greatest improvement, achieving a specific capacitance increase exceeding 150 % relative to pristine AC. These findings highlight that the in-situ nitrogen modification route offers a promising strategy for enhancing the functionality of biomass-derived CQDs, paving the way for sustainable, high-performance materials in next-generation energy storage devices.

1. Introduction

Sustainable energy development requires technologies that minimize environmental harm, utilize renewable resources and improve overall energy efficiency [1]. Renewable energy sources such as solar, wind, hydrothermal, and geothermal power are inherently intermittent, creating a critical need for efficient energy storage systems (ESS) that can store and deliver energy according to demand. Among these, electrochemical energy storage systems—particularly batteries and supercapacitors (SCs)—are promising due to their high energy and power densities, fast charge-discharge rates, and long cycle life [2].

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Biomass-derived activated carbon (AC) has emerged as a cost-effective and environmentally friendly electrode material for SCs because of its wide availability and sustainability advantages. SCs are widely recognized for their exceptional power density, extended cycle life, and rapid charging capability, which makes them suitable for applications in portable electronics, backup power systems, telecommunications, and hybrid electric vehicles [3]. Depending on their charge-storage mechanism, SCs are classified as either electric double-layer capacitors (EDLCs) or pseudocapacitors, primarily determined by the type of electrode material employed [4].

Despite extensive research into carbonaceous materials, metal oxides, and conducting polymers, AC remains the predominant material for commercial SC electrodes [5]. Biomass naturally contains lignin ($\approx 27\%$), cellulose ($\approx 43\%$), and hemicellulose ($\approx 20\%$), offering high carbon content and low ash content (0.2–10%), which are advantageous for electrochemical applications [3]. Carbonization of biomass via pyrolysis or hydrothermal carbonization removes volatile matter and moisture, forming micropores and creating a limited specific surface area (SSA). Subsequent activation increases SSA and optimizes pore size distribution, enhancing ion diffusion and electrode conductivity [3]. Nevertheless, pristine AC electrodes often exhibit low specific capacitance and limited energy density, which restricts their performance in high-power applications.

To address these limitations, strategies such as heteroatom doping and nanomaterial integration have been explored. Carbon nanomaterials, including carbon nanotubes (CNTs), carbon nanofibers (CNFs), and graphene exhibit high electrical conductivity, thermal stability, and mechanical strength [5]. However, these materials face challenges in scalability, cost, packing density, and compatibility with current electrode fabrication methods, limiting their practical application in commercial SCs.

Recently, carbon quantum dots (CQDs); 0-dimensional carbon-based nanomaterials have gained attention as electrode additives due to their tunable physicochemical properties, large surface area, and excellent electron transport pathways. However, pristine CQDs tend to aggregate, reducing their effective surface area and electrochemical performance. Nitrogen doping has been shown to enhance electron mobility, improve colloidal stability, and reduce the band gap energy of CQDs. For instance, N-doped CQDs derived from reed straw and melamine biomass achieved a specific capacitance of 202.8 F g^{-1} at 1 A g^{-1} , with excellent cyclic stability, retaining 96.3% capacitance after 5000 cycles [7].

Despite these advances, a clear research gap remains: few studies have directly compared different NCQD synthesis methodologies or systematically evaluated their integration with industrial-grade AC electrodes. Therefore, this study aims to synthesize nitrogen-doped CQDs via two green methodologies—(A) in-situ modification and (B) coating—and investigate their chemical, structural, and electrochemical properties when incorporated into AC electrodes. This approach enables a direct comparison of NCQD performance, providing insight into how nitrogen doping and synthesis method influence electrode behavior for the development of sustainable, high-performance energy storage devices.

2. Materials and Methods

2.1 Preparation of Precursor Material

The study used banana peel as a precursor material, focusing on reducing the moisture content. To achieve this, the shells were first dried and then ball ground into a fine powder. The final product was banana peel powder, which was the main material for further research.

2.2 Preparation of CQDs

To synthesize Carbon Quantum Dots (CQDs), 0.06g of prepared banana peel powder is then mixed with 80ml of water. The mixture is then transferred into an autoclave chamber and goes through a hydrothermal process at the temperature of 200 °C. The brown liquid is then filtered with microfilter adapted on a syringe. The final product liquid should be close to a clear solution.

2.3 Modification of CQDs with Nitrogen-based Dopant

To synthesize Nitrogen doped Carbon Quantum Dots (NCQDs), repeat the same process of synthesizing CQDs but with adding 1 % (w/w) of Ethylenediamine with 80ml water. The hydrothermal process was then continued similarly like the process of synthesizing CQDs.

2.4 Integration of CQDs and N-CQDs into AC

Activated carbon (AC) is submerged into 40ml of NCQDs. The mixture was capsulated and sonicated for 2 hr with 75% intensity. Afterwards, the mixture is filtered and dried off at the temperature of 50 °C for 6 hr.

2.5 Preparation of Supercapacitor Electrode Materials

3 types of electrode materials were synthesized with the usage of industrial grade activated carbon, carbon black as conducting material and polyvinylidene fluoride (PVDF) as binder were mixed in the mass ratio of 8:1:1. The mixture is mixed with the help of N-Methyl-2-pyrrolidone (NMP) as the suitable solvent. A slurry mixture is then obtained and coated on a stainless steel collector via blade coating methodology. The process is the repeated with the change of AC into AC/CQDs and AC/NCQDs as in Figure 1.

2.6 Electrochemical Analysis Preparation

Each of the SC's electrode materials were examined via cyclic voltammetry (CV), electrochemical impedance spectroscopy (EIS) and galvanostatic charge-discharge (GCD). The electrochemical performance analysis was done at room temperature with 2-electrode cell configuration. The CV test was performed in the potential window of 0.0V to 1.0V vs. SCE at different scan rates (5,10,30,100 mV/s). In the aspect of EIS, the analysis was recorded in a frequency range of 0.1 Hz to 104 Hz at 0.1 V amplitude. Finally, the GCD test was prepared at the specification of 0.0 V to 1.0 V (constant of 25mA).

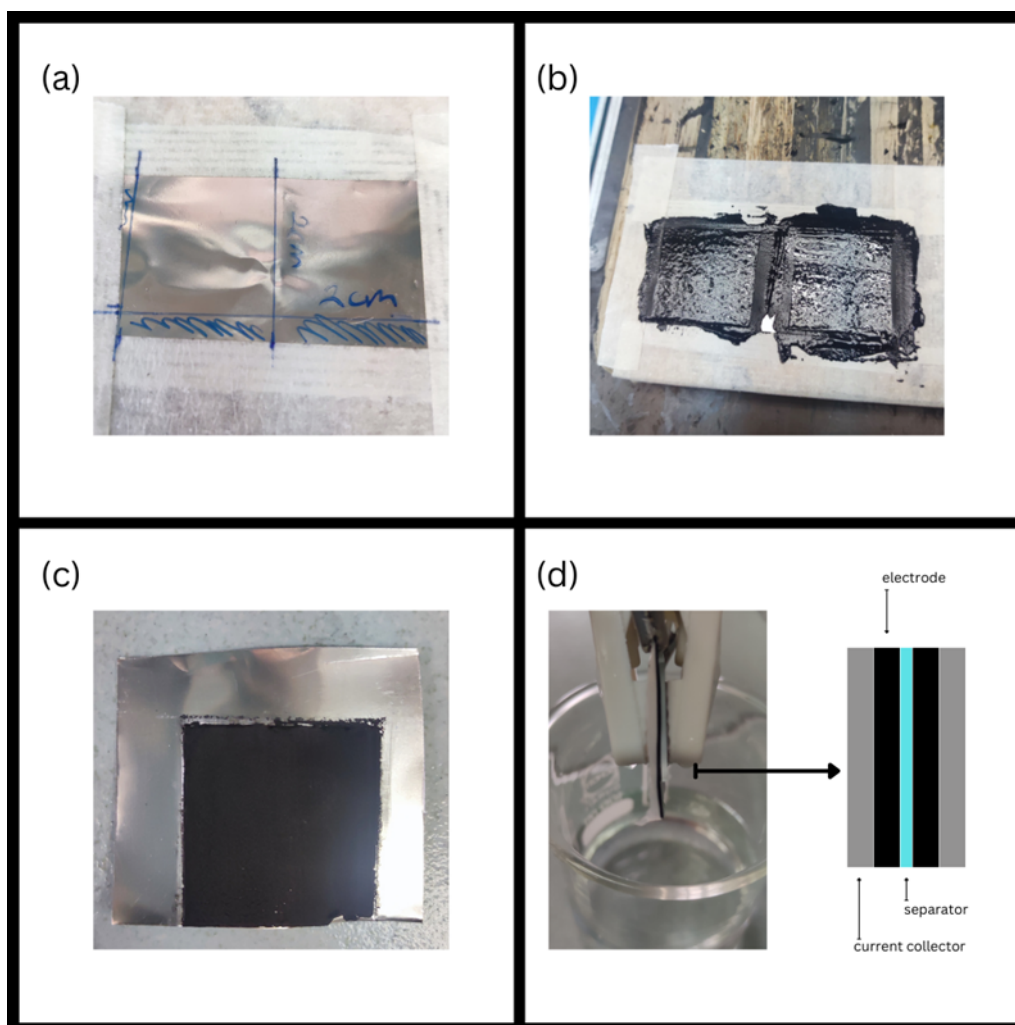


Fig.1. (a-d) Step by step process of preparing activated carbon-based supercapacitor electrode

3. Results

3.1 Chemical and Physical Analysis

Figure 2 shows the FTIR spectra confirming that both synthesis methodologies successfully integrated nitrogen bonding onto the CQDs. The FTIR peaks at 1477 cm^{-1} and 823 cm^{-1} correspond to C–H and C=C vibrations, respectively [8], which are typical of the amorphous carbon structure of CQDs. In contrast, additional peaks at 700 cm^{-1} and 1560 cm^{-1} represent nitrogen-related bonding, validating the successful incorporation of N-functional groups onto the CQDs [9]. Both in-situ and coating methods exhibit these characteristic nitrogen peaks, confirming that nitrogen atoms were effectively integrated into the carbon structure through both approaches [10].

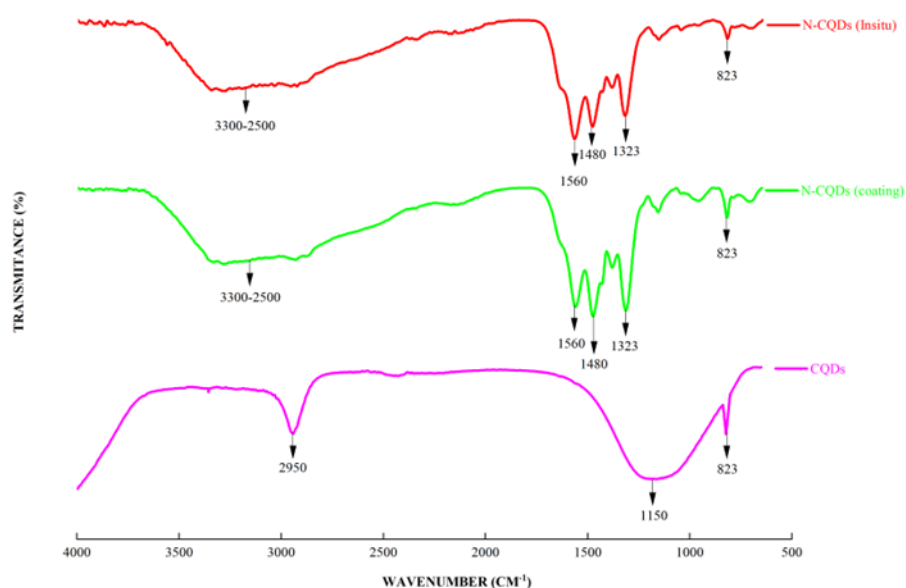


Fig. 2. FTIR result of Nitrogen doped Carbon Quantum Dots (NCQDs) compared with two different methodologies

The presence of nitrogen bonding within the CQDs structure was further verified through ¹Proton Nuclear Magnetic Resonance (¹H NMR) spectroscopy, a fundamental tool for detecting chemical bonding environments in carbon-based nanomaterials. As shown in Figure 3, a broad resonance peak between 4.6 and 4.7 ppm corresponds to sp²-hybridized carbon, while an additional resonance at 2.7 ppm is exclusively associated with the amine group in nitrogen-doped CQDs (NCQDs). This distinct chemical shift confirms the successful attachment of nitrogen-containing moieties, reinforcing the FTIR findings and validating the presence of nitrogen functionalities within the NCQDs structure [11].

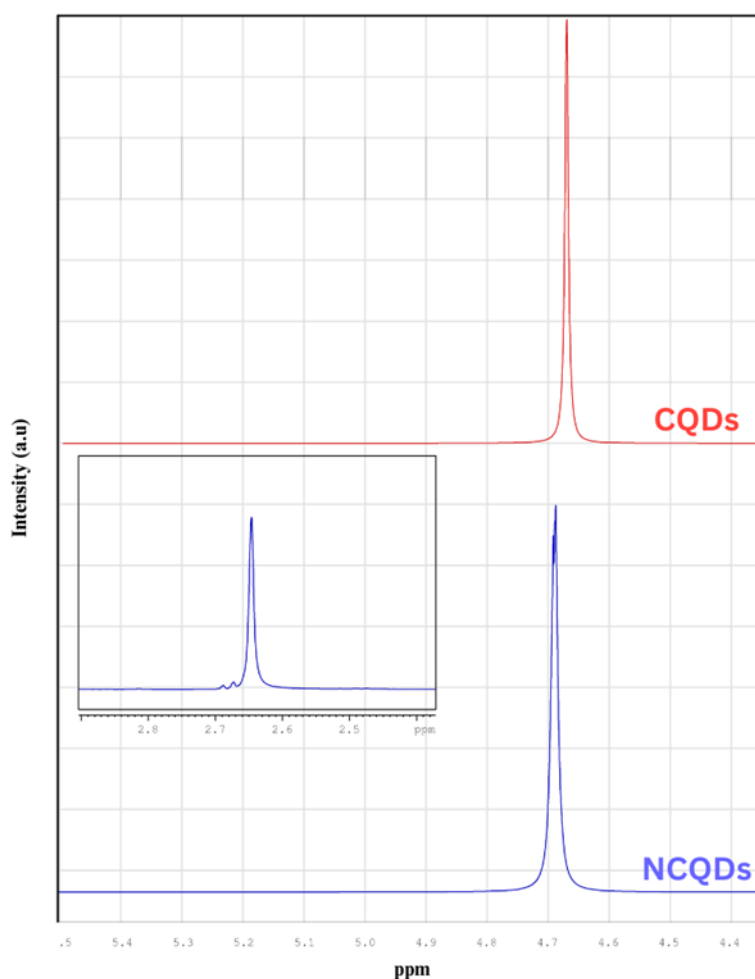


Fig. 3. HNMR spectra of CQDs and NCQDs

The UV–Vis spectra (Figure 4) reveal the optical properties and particle size variations of CQDs and NCQDs. Pristine CQDs exhibit absorption peaks at 282 nm and 364 nm, characteristic of π – π^* and n – π^* transitions, respectively. Following nitrogen doping via the coating method, these peaks shifted to 272 nm and 338 nm, while the in-situ nitrogen modification further blue-shifted them to 269 nm and 318 nm, signifying smaller particle sizes and stronger surface passivation effects. The pronounced blue-shift in the in-situ NCQDs indicates enhanced quantum confinement and improved surface states, resulting in a brighter photoluminescence emission [12]. Furthermore, this blue-shift correlates with a higher surface-area-to-volume ratio, a desirable property for supercapacitor electrodes due to improved ion accessibility and charge accumulation.

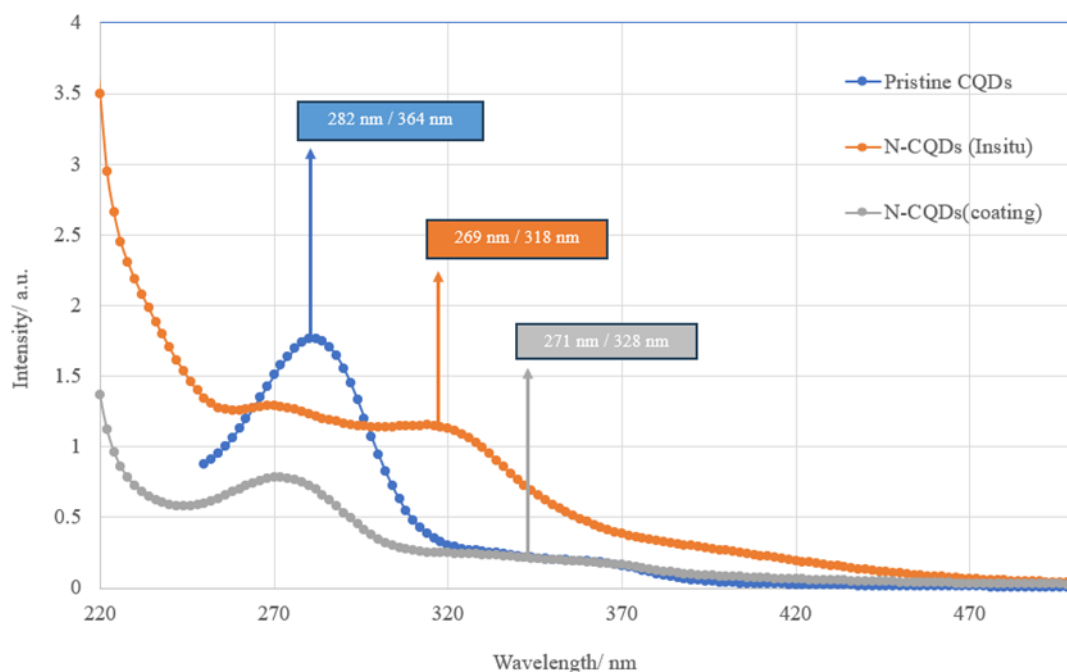


Fig. 4. Comparison of UV-Vis result between pristine CQDs, N-CQDs (coating) and N-CQDs (in-situ)

Fluorescence (FL) spectroscopy provides further insights into the optical efficiency of the synthesized quantum dots. As shown in Figure 5, the N-doped CQDs display a stronger emission peak intensity than undoped CQDs, reflecting improved quantum efficiency. This enhancement is attributed to the effective surface passivation conferred by nitrogen incorporation, which mitigates non-radiative recombination pathways and prevents particle agglomeration [9]. The homogeneous dispersion of NCQDs contributes to stable photoluminescence behavior, demonstrating their potential for consistent electron–hole pair dynamics. In the context of supercapacitors, this improved fluorescence behavior implies superior electron transport and long-term structural stability, which are critical for achieving reliable energy storage performance.

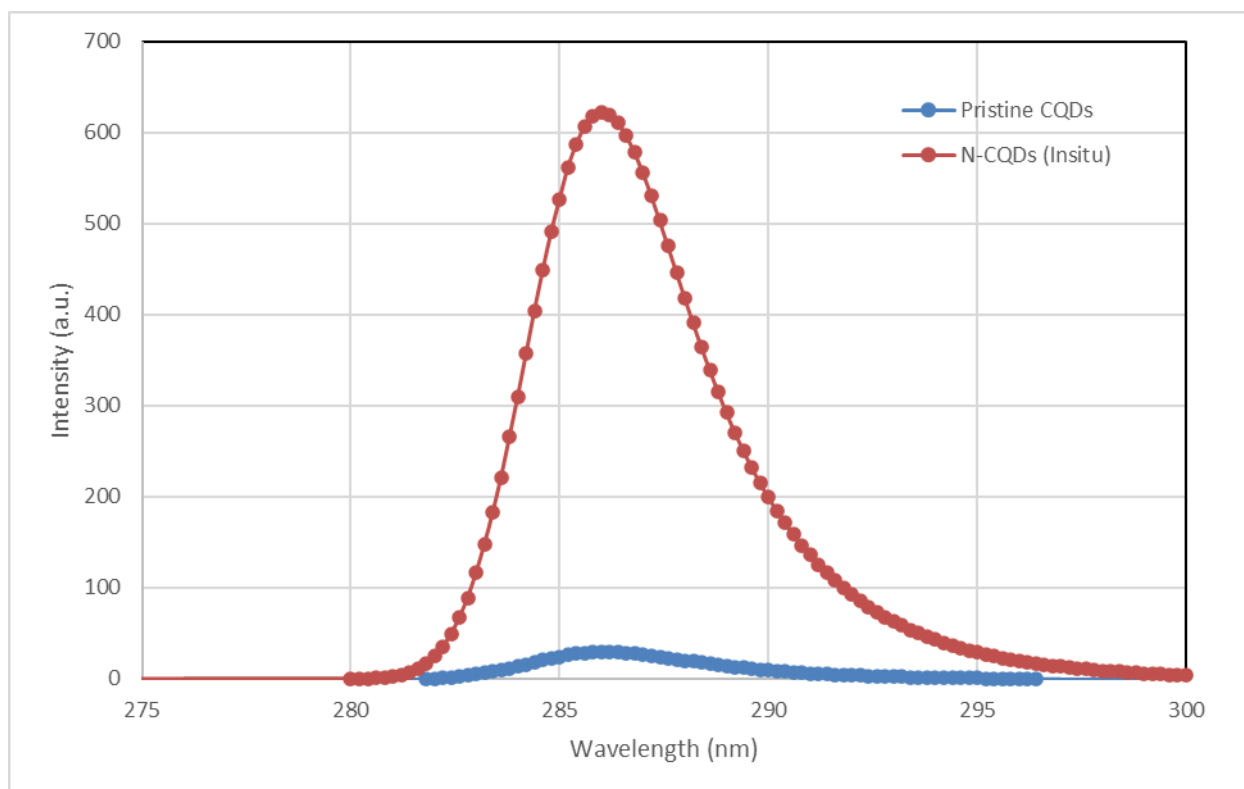


Fig. 5. Fluorescence Spectrophotometers (FL) result of (a) Pristine CQDs and (b) N-CQDs (In-situ)

Zeta potential analysis provides valuable insight into the colloidal stability of CQDs and NCQDs. Over time, CQDs in suspension tend to aggregate, leading to reduced quantum efficiency. As listed in Table 1, the zeta potential values for CQDs and NCQDs are -15.2 mV and -35.5 mV, respectively, with the higher negative potential indicating enhanced electrostatic repulsion and improved colloidal stability for NCQDs. The in-situ synthesized NCQDs exhibit the highest stability and the most homogeneous particle size distribution, as confirmed by the Nano Particle Size Analyzer (NPSA). The enhanced electron affinity of the amine group further improves electrical conductivity, increasing from 0.312 mS cm^{-1} to 1.7 mS cm^{-1} . This dual improvement in stability and conductivity is crucial for achieving high-performance electrodes in energy storage systems [13].

Table 1

Zeta potential, conductivity and particle size analysis

Sample	Zeta Potential (mV)	Conductivity (mS/cm)
CQDs	-15.2	0.312
NCQDs (coating)	-21.3	0.320
NCQDs (in-situ)	-35.5	1.70

The Brunauer–Emmett–Teller (BET) analysis (Figure 6) reveals the surface area changes of activated carbon (AC) after CQD and NCQD incorporation. The surface area decreased from 684.8 m² g⁻¹ (AC) to 607.9 m² g⁻¹ (AC/CQDs) and 553.4 m² g⁻¹ (AC/NCQDs), representing reductions of 11.86% and 19.18%, respectively. These reductions are consistent with nanomaterial deposition partially blocking micropores [14]. While this decrease may suggest reduced ion accessibility, the formation of an intimate interface between AC and NCQDs enhances the electrode's electrochemical response. Thus, despite a moderate reduction in porosity, the surface modification improves active site accessibility and charge transfer efficiency [15,16].

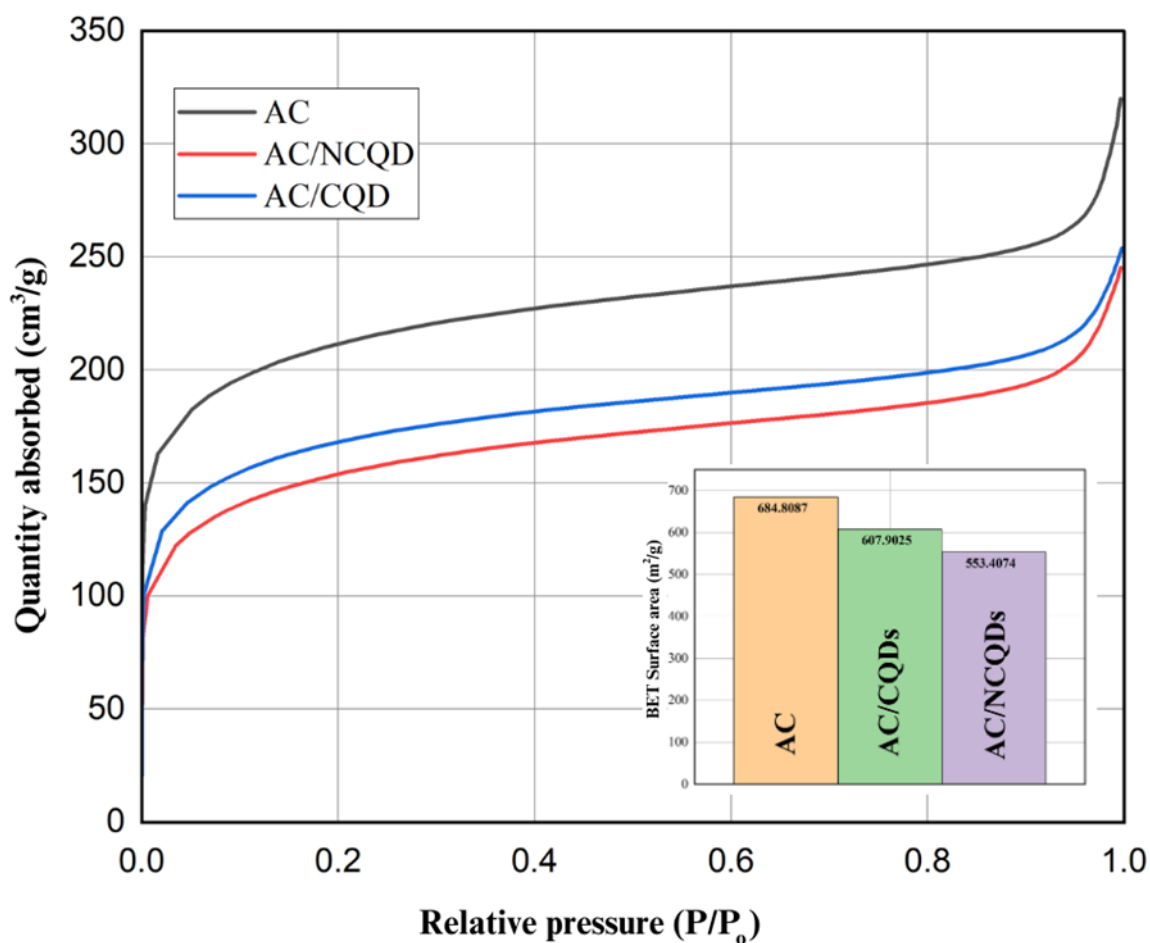


Fig. 6. Nitrogen absorption isotherm data compared between AC, AC/CQDs, and AC/NCQDs

3.2 Electrochemical Analysis

3.2.1 Cyclic voltammetry

The cyclic voltammetry (CV) results, shown in Figure 7, compare the electrochemical behavior of AC, AC/CQDs, and AC/NCQDs electrodes. By referring to the Figure 7, the specific capacitance value was calculated by Eq. (4):

$$C_p = \frac{I \Delta t}{m \Delta V} \quad (1)$$

$$C_p = \frac{I}{m \Delta \left(\frac{V}{t} \right)} \quad (2)$$

$$I = C_p \times m \times \frac{V}{t} \quad (3)$$

$$\int_{v_1}^{v_2} I = \int_{v_1}^{v_2} \left(C_p \times m \times \frac{v}{t} \right) dv = \text{area of CV graph}$$

$$C_p = \frac{A}{(V_2 - V_1) \times m \times \frac{V}{t}} \quad (4)$$

where C_p represents specific capacitance, m is the weight of the active electrode material, time is denoted by t , V is the voltage input, and A stands for the area of the CV graph region. The specific capacitance values demonstrate a 16.2% improvement for AC/CQDs and a 154.06% increase for AC/NCQDs relative to pristine AC. The significant enhancement summarized in Table 2 underscores the role of nitrogen doping in improving electron mobility and ionic interaction at the electrode–electrolyte interface. The in-situ NCQD route yields the most efficient charge propagation, confirming that nitrogen incorporation optimizes conductivity and double-layer formation, representing lower electronic resistivity.

Table 2

Comparison of specific capacitance of AC, AC/CQDs, AC/NCQDs electrode at 10mV/s

Electrode	Scan rate (mV/S)	Potential window (V)	Mass (g)	Specific capacitance (F/g)
AC	10	1	0.01	43.26
AC/CQDs	10	1	0.01	50.27
AC/NCQDs	10	1	0.01	109.92

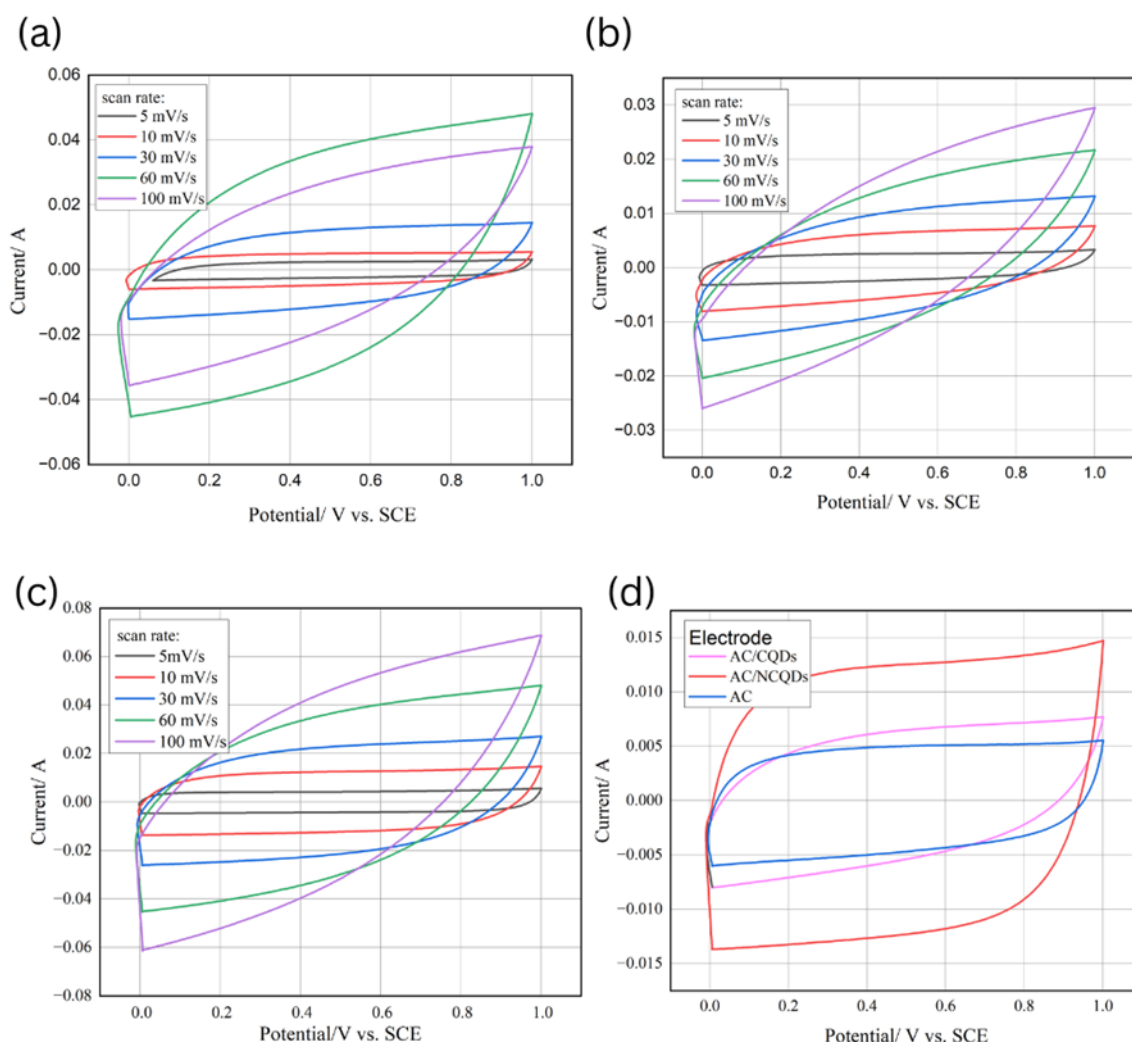


Fig. 7. (a-c) Results of cyclic voltammetry at scan rate of 5, 10, 30, 60, 100 mV/s for (a) pristine AC (b) AC/CQDs, (c) AC/NCQDs, (d) Comparison of cyclic voltammetry between AC, AC/CQDs and AC/NCQDs at the scan rate of 10 mV/s

3.2.2 Galvanostatic charge discharge

The GCD curves in Figure 8 show a symmetrical triangular curve, confirming the ideal capacitive behavior characteristic of carbon-based electrodes. As observed in Figure 6, the equivalent series resistance (ESR) value of the cells increases with the addition of CQDs and NCQDs. The ESR value can be calculated using equation (5).

$$ESR = \frac{iR_{drop}}{2i} \quad (5)$$

The calculated ESR values for AC, AC/CQDs, and AC/NCQDs are 105.9, 116.10, and 106.8 mΩ, respectively. Although the inclusion of CQDs slightly increases resistance due to reduced carbon black content, the NCQDs maintain near-identical resistivity to pristine AC, indicating that nitrogen doping effectively compensates for conductivity loss by improving charge transfer [22–24]. This demonstrates that NCQDs can preserve low internal resistance while enhancing capacitance, an advantageous trade-off for energy storage optimization.

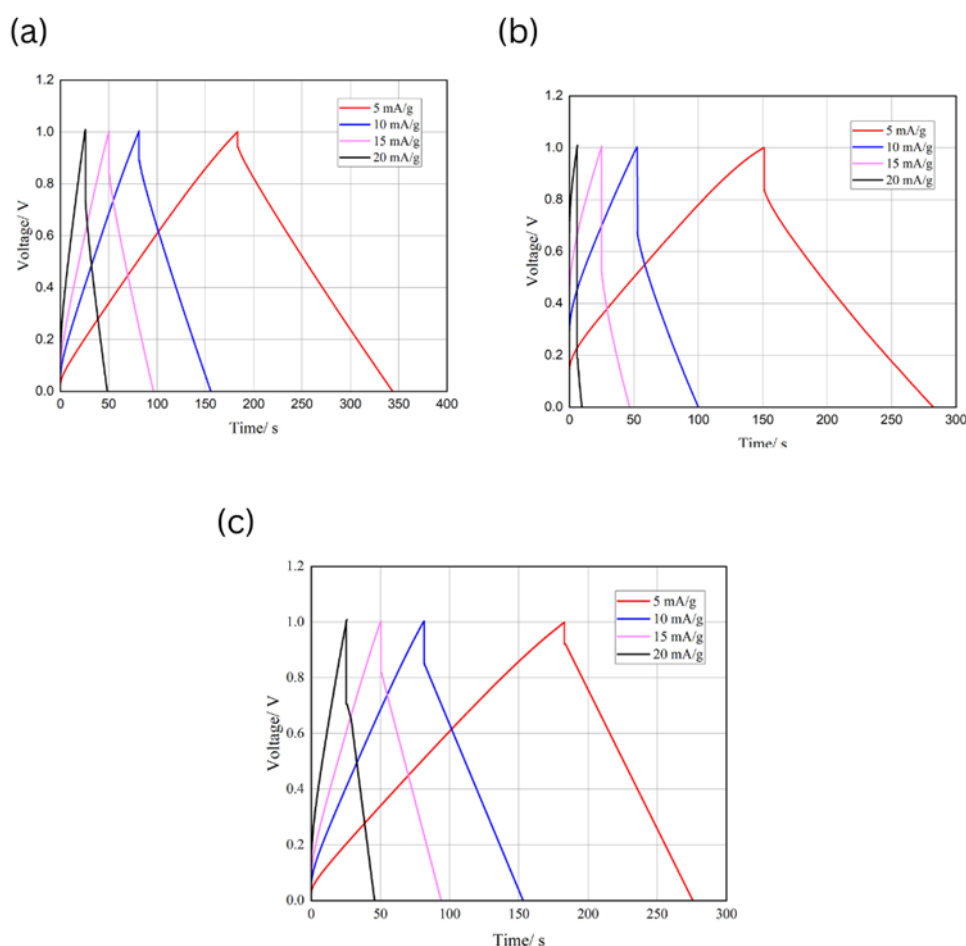


Fig. 8. (a-c) Results of Galvanostatic Charge Discharge at different current densities for AC, AC/CQDs, AC/NCQDs respectively

3.2.3 Electrochemical impedance spectroscopy

Nyquist plots (Figure 9) further clarify the impedance behavior of the electrodes. The semi-circle diameter in the high-frequency region represents charge transfer resistance, while the Warburg tail at low frequencies indicates ion diffusion [25]. The AC/CQDs electrode exhibits a larger semi-circle, signifying higher resistance, whereas the AC/NCQDs electrode presents the smallest semicircle, confirming superior charge transfer kinetics and faster ion transport [26]. The nearly vertical Warburg region of AC/NCQDs further demonstrates efficient ion diffusion across the porous structure, indicative of highly interconnected electrode–electrolyte interfaces [27].

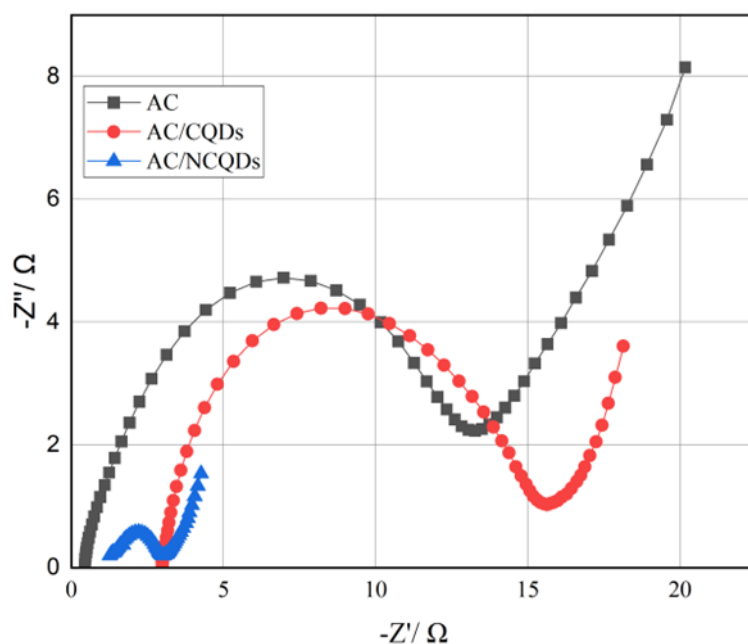


Fig. 9. Results of Electrochemical impedance spectroscopy of AC, AC/CQDs, and AC/NCQDs plotted in Nyquist

As illustrated in Figure 10, the enhanced capacitance of NCQDs can be attributed to two synergistic effects: (i) the π -conjugated structure of NCQDs enables greater charge delocalization, resulting in stronger Coulombic attraction of counter-ions, and (ii) the nitrogen dopant enhances electron density, enabling higher charge accumulation within the electrode pores [17–21]. This unique electronic configuration allows NCQDs to overcome the traditional trade-off between surface area loss and capacitance gain.

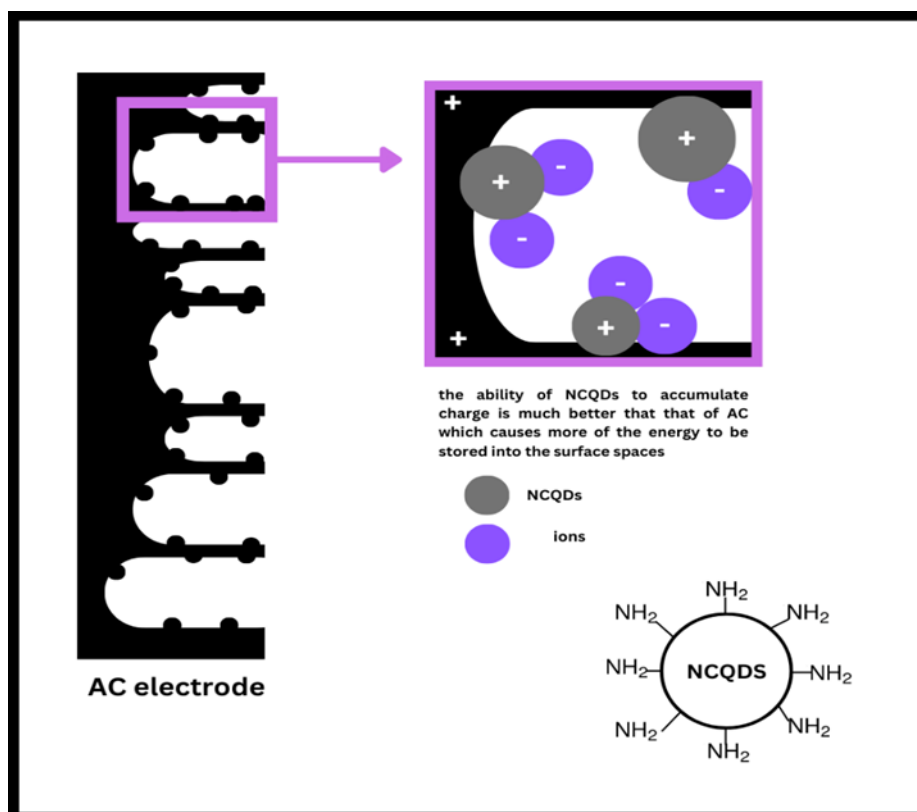


Fig. 10. The possible mechanism for capacitance enhancement in AC/NCQDs compared to AC pristine and AC/CQDs. (a) The relatively high-charge density of the CQDs enables the accumulation of a higher concentration of counter-ions within the remaining pores of the charged composite electrodes. (b) The NCQDs nanostructure illustration

4. Conclusions

This study establishes a sustainable strategy for converting banana peel waste into nitrogen-engineered carbon quantum dots, demonstrating how controlled heteroatom doping can transform low-conductivity biomass carbons into efficient electrochemical materials. The comparison of synthesis routes revealed that the N₂-doping plays a decisive role in determining charge transport and interfacial stability. These findings highlight the potential of in-situ N₂-doping to tailor surface chemistry and enhance electrochemical behavior while promoting sustainable biomass valorization. Minor experimental uncertainties may arise from instrument calibration (particularly during EIS measurements) and electrode mass uniformity during coating; however, all measurements were repeated three times to ensure reproducibility. Overall, this work provides both mechanistic insight and broader sustainability significance, positioning in-situ N-doped CQDs as a viable route toward next-generation, eco-friendly supercapacitor electrodes.

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