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Cyclic Voltammetry Characteristics of Hybridized Carbon Electrode Incorporated in Carbon Nanotube Matrix in Aqueous Electrolytes

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Abstract

In this work, a design of a hybridized carbon electrode combining the super microporosity and high electrical conductivity is proposed by incorporating carbon nanotubes (CNTs) at different percentage weights in a porous carbon matrix with <2nm pores by sol-gel method. Results showed a semi-ideal capacitive response during the cyclic voltammetry tests. The proposed design safe operation and low cost using aqueous electrolytes, as a promising solution for intensive energy storage applications.

Keywords: Microporosity; Activated carbon; Carbon nanotubes; Energy storage
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1. Introduction

When talking about the electrode materials in aqueous EDLC supercapacitors, the porous carbon materials are the first option due to their abundance, light weight, chemical stability, and good electrical conductivity. However, not all porous carbon materials can be used for this purpose [1-3]. The literatures reveal that the pore smaller than 2 nm (micropores) actually contribute to increase the electrical capacitance when an aqueous electrolyte (such as H_2SO_4 or KOH) is used because its ions show small hydrodynamic sizes (mostly smaller than 1 nm). This enables them to enter inside the micropores and form double dens layer [4-6]. Here, the problem is double, the excess dependence on the micropores hinders the motion of ions due to the high diffusion resistance inside narrow channels, particularly at high charging/discharging rates, which reduces the actual power density [7-10]. The carbon material of high micro-porosity often lack enough overall electrical conductivity due to the availability of holes and structural defects. Here, the carbon nanotubes (CNTs) to play the role of the carbon dioxide (CO_2) [11-13]. CNTs show exceptional electrical conductivity (approaching to that of metals) with single-wall or multiwall cylindrical structures, which provide fast conducting pathways to pass electrons through he electrode, in addition to good specific surface area [14-18]. Though, addition of CNTs only is not sufficient as the synergistic tuning is required to combine the high electrical conductivity of CNTs with the super microporous area of another carbon source [19-23]. This structure is known as the hybridized carbon compound.

Accordingly, the aim of this work is the design of hybrid carbon electrode with controlled microporosity (>80% with pore size <2nm) and incorporated with 5-15 wt.% CNTs by sol-gel method. The relationship between the nanostructure (porous distribution and CNTs alignment) and electrochemical behavior (ionic diffusion resistance, capacitance at different discharging rates, and long-term cyclic stability) will be studied.

2. Experimental Part

The multi-walled carbon nanotubes (MWCNTs) of 10-20 nm outer diameter and 10-30 nm length were firstly purified by boiling in HNO_3 acid at 120°C for 4 hours to remove the metallic impurities and insert carboxyl functional groups to increase the dispersion. Then, 50 mg of purified MWCNTs was dissolved in 40 mL methanol using ultrasonic waves for 30 minutes. After that, 1.17 g of hexahydrate zinc nitrates ($Zn(NO_3)_2.6H_2O$) with magnetic stirring. In a separate container, 2.6 g of 2-methyl imidazole was dissolved in 40 mL methanol. The two solutions were quickly mixed and strongly stirred at room temperature for 30 minutes. The mixture was left static for 24 hours to react. The product (ZIF-8/CNT) was collected by filtering, washing three times in methanol, and dried for 12 hours at 80°C.

To produce the hybridized carbon, the resulted powder was placed in alumina boat and entered in a tube furnace as argon gas was flowing at 100 mL/min rate. The temperature was increased by 5 °C/min to reach 900°C to completely carbonize the powder for 2 hours and then the furnace was cooled down to room temperature. It was observed that the MWCNTs formed a conductive network capping the carbon particles derived from ZIF-8, which enhances the electrical contact between the particles. A pure carbon sample was prepared without CNTs to represent the reference samples, while another sample was prepared from oxidized MWCNTs alone. The hybridized sample showed a weight fraction of about 12% MWCNTs according to the TGA analysis in air. Figure (1) shows schematically the proposed design of the hybridized carbon electrode for EDLC cell.

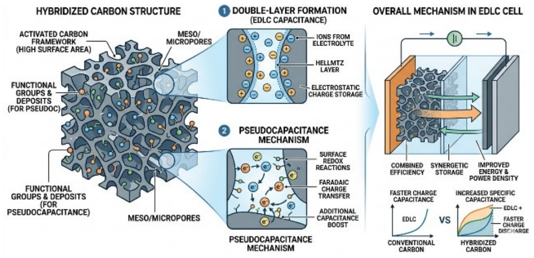

Fig. (1) Scheme of the principle of the proposed design of the hybridized carbon electrode for EDLC cell

The cell was constructed from two electrodes separated by a Celgard 3501 polypropylene film and an opposite electrode made from high-capacitive carbon. The performance was measured by a Bio-Logic SP-300 electrochemical station at room temperature. The measurements included cyclic voltammetry (CV) with span velocities of 5-200 mV/s within potential range of 0-1.6 V, Galvano-static charging and discharging (GCD) using current densities from 0.2 to 20 A/g, and electrochemical impedance spectroscopy (EIS) within frequency range of 100 kHz – 10 MHz with signal amplitude of 10 mV.

3. Results and Discussion

Figure (2) shows the cyclic voltammetry curves of the hybridized carbon electrode prepared in this work throughout the variation of current density with applied voltage for different scanning velocities (50, 100, and 200 mV/s). The electrode exhibits approximately rectangular loop particularly at high scanning velocity of 200 mV/s, which reveals to semi-ideal mechanism of capacitive charge storage depending on the formation of the dual electric layer without irreversible Faradaic reactions [24]. The good symmetry of the forward and reverse scanning curve confirms the a high efficiency in the charging/discharging process with small energy loss, whereas the wide curve and higher current density refer to fast response of the porous structure incorporated to CNTs network, which facilitates the ion diffusion within the hybridized structure. Despite the slight deviation of the 200 mV/s curve from the rectangular shape, which is attributed to the ion diffusion resistance in deep pores, the overall behavior reveals a successful integration of microporosity and enhanced conductivity [25].

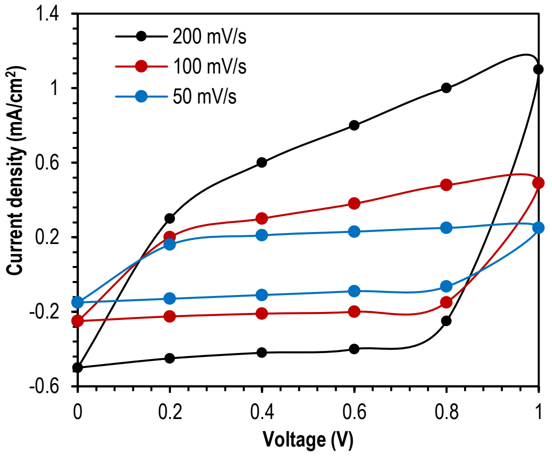

Fig. (2) Variation of current density with applied voltage for different scanning velocities (50, 100, and 200 mV/s) (cyclic voltammetry curves) of the hybridized carbon electrode prepared in this work throughout the

Figure (3) shows the Bode plot of the phase angle as a function of frequency for the hybridized carbon electrode in the EDLC cell. The variation of phase angle in the frequency range of 0.01-10 Hz confirms the dominant ideal capacitive behavior with the formation of effective dual electrical layer and very low equivalent series resistance. The behavior at very low frequencies is ascribed to the presence of deep micropores that increase the capacitance with some resistance to ion diffusion [26].

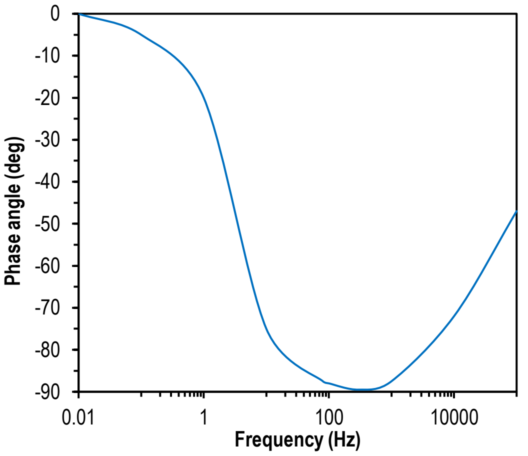

Fig. (3) Bode plot of the phase angle as a function of frequency for the hybridized carbon electrode in the EDLC cell

At very high frequencies (>1000 Hz), the phase angle decreases to about -50° due to the Ohmic resistance of the electronic network. Maintaining phase angle at about -90° within the frequency range of 100-1000 Hz confirms the synergistic effect of microporosity and CNTs network, as the latter enhances the high-frequency conductivity while the former provides capacitive storage at low frequencies. This behavior makes the proposed electrode ideal for energy storage applications from the moderate to high power ranges.

4. Conclusion

The results obtained from this work reveal that the incorporation of CNTs at different percentage weights in porous carbon matrix can achieve the balance between maintaining the high porosity and enhancing the electrical conductivity. The proposed hybridized carbon electrode showed excellent cyclic stability as the capacity retention was maintained approximately constant after large number of charging/discharging cycles. The cyclic voltammetry and Bode plots confirmed the semi-ideal capacitive behavior with phase angle close to -90° within a wide frequency range (100-1000 Hz).

References

[1] Y. Duan et al., “Advances in wearable textile-based micro energy storage devices: structuring, application and perspective”, *Nanoscale Adv.*, 3(22) (2021) 6271-6293.

[2] R. Bahadur et al., “Advanced porous borocarbonitride nanoarchitectonics: Their structural designs and applications”, *Carbon*, 190 (2022) 142-169.

[3] Z. Zhu et al., “Versatile carbon-based materials from biomass for advanced electrochemical energy storage systems”, *eScience*, 4(5) (2024) 100249.

[4] Y. Bai and H. Zhang, “Freeze-enabled synthesis of functional materials: fundamental, progress, and applications”, *Prog. Mater. Sci.*, 155 (2026) 101523.

[5] G. Dorji et al., “Binary transition metal oxides vs. binary metal oxides for electrochemical supercapacitors: Performance, challenges, and future prospects”, *J. Energy Stor.*, 147 (2026) 120116.

[6] Y. Yuan et al., “Research advances of magnesium and magnesium alloys globally in 2024”, J. Magnesium Alloys, 13(10) (2025) 4689-4732.

[7] V. Singh and H.R. Byon, “Advances in electrochemical energy storage with covalent organic frameworks”, *Mater. Adv.*, 2(10) (2021) 3188-3212.

[8] D.-L. Yang et al., “Micro-sized nanoaggregates: Spray-drying-assisted fabrication and applications”, *Particuology*, 85 (2024) 22-48.

[9] M. Gowtham et al., “Progress and challenges in carbon nanomaterial-based supercapacitors: Towards sustainable high-energy storage”, *Renew. Sustain. Energy Rev.*, 235 (2026) 116930.

[10] Z. Zhai et al., “A review of carbon materials for supercapacitors”, *Mater. Design*, 221 (2022) 111017.

[11] H. Lin et al., “Poly(ionic liquid)s with engineered nanopores for energy and environmental applications”, Polymer, 202 (2020) 122640.

[12] N. Desai et al., “Electrospun PAN/TEMPO nanofiber electrode: Dual charge storage mechanism for supercapacitor applications”, *Compos. B: Eng.*, 313 (2026) 113405.

[13] S.M. Parsa et al., “Advances on multi-heteroatom doped carbon catalysts: Synthesis, mechanistic insight, synergies, and application in microbial electrochemical energy, fuel and chemical production”, *Carbon*, 251 (2026) 121315.

[14] M. Gopalakrishnan et al., “Recent advances in oxygen electrocatalysts based on tunable structural polymers”, *Mater. Today Chem.*, 23 (2022) 100632.

[15] P. Siddu N.K., S.M. Jeong, and C.S. Rout, “MXene–carbon based hybrid materials for supercapacitor applications”, *Energy Adv.*, 3(2) (2024) 341-365.

[16] V. Pavlenko et al., “A comprehensive review of template-assisted porous carbons: Modern preparation methods and advanced applications”, *Mater. Sci. Eng. R: Rep.*, 149 (2022) 100682.

[17] Y. Chen et al., “Exploring the potential of low-dimensional materials from cigarette butts for energy applications: A comprehensive review”, *Adv. Powder Mater.*, 4(3) (2025) 100295.

[18] M.S. Islam et al., “Carbon gel materials: synthesis, structural design, and emerging applications in energy and environmental technologies”, *Mater. Adv.*, 6(20) (2025) 7153-7206.

[19] G. Wei et al., “Biomass vs inorganic and plastic-based aerogels: Structural design, functional tailoring, resource-efficient applications and sustainability analysis”, *Prog. Mater. Sci.*, 125 (2022) 100915.

[20] A.C.M. Loy et al., “Elucidation of single atom catalysts for energy and sustainable chemical production: Synthesis, characterization and frontier science”, *Prog. Energy Combus. Sci.*, 96 (2023) 101074.

[21] Y. Sun et al., “Non-noble metal single atom-based catalysts for electrochemical reduction of CO_2: Synthesis approaches and performance evaluation”, *DeCarbon*, 2 (2023) 100018.

[22] M. Zhang et al., “From wood to thin porous carbon membrane: Ancient materials for modern ultrafast electrochemical capacitors in alternating current line filtering”, *Energy Stor. Mater.*, 35 (2021) 327-333.

[23] S. Yousaf et al., “Engineering strategies in the rational design of Cu-based catalysts for electrochemical CO_2 reduction: from doping of elements to defect creation”, Mater. Adv., 5(20) (2024) 7891-7978.

[24] X. Zheng et al., “Chlorination and oxygenation of carbon electrodes for covalent attachment of thiol-terminated molecules”, *Carbon Trends*, 20 (2025) 100539.

[25] E.A. Alabdullkarem, J. Khan, and H. Alrobei, “Integration of multiwalled carbon nanotubes with $MoS_2/BiFeO_3$: a hybrid matrix for high-energy asymmetric supercapacitors”, *Energy Adv.*, 5(1) (2025) 71-82.

[26] O.B. Abdillah et al., “Recent advances of macroporous architectures for multidisciplinary applications”, *Adv. Powder Technol.*, 37(6) (2026) 105265.