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Enhancing Electrochemical Performance of ZnO Nanocomposite by Integrating Carbon Black for Supercapacitor Electrode

Shubham Mural[1], Shweta Tanwar[1], Anjali[1], Nirbhay Singh[2], Ramesh Kumar[3], Anurag Gaur[1]*

[1]Department of Physics, Netaji Subhas University of Technology, New Delhi, India
[2]Department of Applied Physics, School of Engineering and Technology, Amity University Gwalior, Madhya Pradesh, India
[3]Department of Physics, Meerut Institute of Engineering and Technology, Meerut, India

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Corresponding author: anuragdph@gmail.com

Abstract

Driven by the rising energy consumption throughout the world, there is an urgent demand for highly efficient energy storage devices. Zinc Oxide, due to its cost-effective and environmentally friendly nature, shows a promising future in this direction. However, its practical application is hindered by its low electrical conductivity, leading to poor electrochemical performance. To increase the energy storage capacity of ZnO, this study designs a Zinc Oxide/ Carbon Black (Zn/CB) nanocomposite electrode. The Zn/CB nanocomposite electrode exhibited a remarkable increase in the specific capacitance (S_c) as compared to the pristine ZnO. It increased from 128 F g^{-1} (ZnO) to 375 F g^{-1} (Zn/CB) @ 10 mV s^{-1} scan rate. GCD analysis confirms this, showing an increase from 15 F g^{-1} (ZnO) to 183 F g^{-1} (Zn/CB) @ 4 A g^{-1}. Crucially, the composite displays exceptional cyclic stability by showing capacitance retention of 145% and coulombic retention of 94% after 3000 cycles @ 15 A g^{-1}. This finding demonstrates that the integration of carbon black with the Zinc Oxide substantially enhances its electrochemical performance, positioning the ZnO/CB nanocomposite electrode as a cost-effective, high-performance, nature-friendly electrode for energy storage systems.

Keywords: Carbon; Zinc Oxide; co-precipitation method; Hybrid supercapacitor.

1. Introduction

Rapid increase in the energy demands combined with the need for eco-friendly development has driven advances in the sustainable energy technologies [1], [2]. Energy resources are a vital and critical aspect of modern society, as they have been used for powering homes and businesses, fueling transportation, and industries. Traditionally, non-renewable resources like coal, fossil fuels, and oil have fulfilled the energy demands. As the need to save the planet has increased, there is a total shift in the energy sector. Sustainable energy resources are the necessity of the hour [3]. Energy storage systems play a critical role in making the sustainable energy resources feasible in day-to-day life [4], [5]. Electrochemical capacitors and batteries have become the leading option for energy storage applications [6]. Limitations of traditional carbon-based materials in the electrochemical performances led a path for the transition metal oxide (TMOs) materials to become highly desired candidates for energy storage applications [4], [7], [8].

Zinc Oxide (ZnO) stands out among TMOs as a highly capable energy storage material. It demonstrates excellent electrochemical activity, eco-friendliness, and ease of production [9], [10]. Despite these advantageous properties, ZnO supercapacitors face several significant challenges. Various composites of ZnO have been synthesized to overcome these shortcomings [7]. Recent advances in ZnO/Carbon composites have demonstrated that the carbon critically governs conductivity, ion transport, and cyclic stability in supercapacitor electrodes [11]. In ZnO/graphene systems, reduced graphene oxide (rGO) provides a high surface area and conductive network that enhances the charge collection. Microwave-assisted graphene-ZnO nanocomposites have reported capacitances of 146 F g^{-1}, while rGO/ZnO hybrids achieved ~260 F g^{-1} [12]. ZnO/CNT composites offer high-conductivity networks that facilitate rapid electron transport and mechanical robustness in flexible devices. More elaborate architectures, such as ZnO quantum dots/carbon/CNT, reach 185 F g^{-1} at 0.5 A g^{-1} current density and an all-solid-state asymmetric device delivering 23.6 Wh kg^{-1} energy density [13]. ZnO/Activated Carbon (ZnO/AC) composites present a cost-effective route with accessible porosity and good electrochemical reversibility. A ZnO/AC nanocomposite electrode showed 160 F g^{-1} specific capacitance for a 1:1 composition, with stable capacitance up to 5000 cycles [14]. Although ACs are economical and readily available, they often require higher mass loadings, which can limit rate capability [15]. Given this landscape, carbon black is used over CNTs or graphene because, while graphene and CNTs offer high performance, they are significantly more expensive and require complex processing. Carbon black is highly cost-effective and scalable. It is compatible with standard slurry mixing, achieving effective percolation, whereas the CNTs and graphene are 3 to 8 times more expensive and require specialized dispersion. Carbon black's percolation behaviour and conductivity support the formation of an efficient conductive network in composites at moderate loadings [16], [17].

This study reports the synthesis of pure ZnO and its composite with carbon black (Zn/CB) via the co-precipitation method. A systematic investigation of their structural, morphological, and electrochemical characteristics was carried out.

2. Experimental Section

2.1. Chemicals Utilized

Sodium hydroxide $(NaOH)$, Zinc Nitrate hexahydrate $(Zn(NO_3)_2.6H_2O)$ and carbon black (CB), all procured from Loba chemicals. Double-deionized water (DI) is used as a solvent for sample preparation.

2.2. Synthesis of the ZnO and ZnO/CB composite

Figure 1. Schematic depicting preparation of (a) ZnO, and (b) ZnO/CB

Zinc Oxide nanoparticles were fabricated via the co-precipitation route as shown in **Figure 1a**. Initially, 14.88 g of $(Zn(NO_3)_2.6H_2O)$ was dissolved in 50 mL of distilled water under non-stop magnetic stirring at 600 rpm for 6 hours. The solution's pH was then adjusted to 8-10 by mixing 2-4 $(NaOH)$ Pallets. After 6 hours of ageing, white precipitates were formed. Further washing and drying of the formed precipitates was done to collect the fine powder. To remove remaining moisture, the ZnO nanoparticles were dried at 60 °C for 24 hours in a vacuum oven. ZnO/CB nanocomposite were made by merging equal amounts (1:1) of ZnO and Commercial Carbon Black as depicted in **Figure 1b**.

2.3. Instruments used:

Powder X-ray diffraction (XRD) was performed to observe the crystallographic structure and phase purity of the prepared samples. The analysis utilized a D-8 X-ray diffractometer (Bruker, USA) with Cu Kα radiation, having a wavelength of approximately 1.5406 Å. The samples' morphological features were studied using a field-emission scanning electron microscope (FESEM) on a Zeiss Gemini SEM 500 (Carl Zeiss, Germany), operated at an accelerating voltage of 10 kV. Electrochemical properties of the electrode materials were assessed through Cyclic Voltammetry (CV) and Galvanostatic Charge-Discharge (GCD) using a SP-240 potentiostat (BioLogic Science). All electrochemical tests were directed in ambient conditions using a conventional three-electrode setup.

2.4. Electrode fabrication for electrochemistry

The working electrode for electrochemical analysis was fabricated using a standardized composition of 8:1:1 by weight. Initially, the active material, either ZnO or ZnO/CB nanocomposite, was weighed to constitute 8 wt.%, while polyvinylidene fluoride (PVDF) binder and conductive carbon black were incorporated at 1 wt.% each. These three components were homogenized using a mortar and pestle with N-methyl-2-pyrrolidone (NMP), thus forming a uniform slurry [18]. This slurry was evenly applied onto a 1×1 cm^2 area of nickel foam substrate (measuring 2×1 cm^2) functioning as the current collector. After coating, the electrodes were dried at 60°C for 2 hours to eliminate any remaining solvent.

3. Results and discussion

3.1. Crystal Structure investigation

The XRD patterns of pristine ZnO and ZnO/CB composite are depicted in **Figure 2**. The grey curve corresponds to the ZnO nanoparticle whereas the red curves correspond to the Zn/CB nanocomposite.

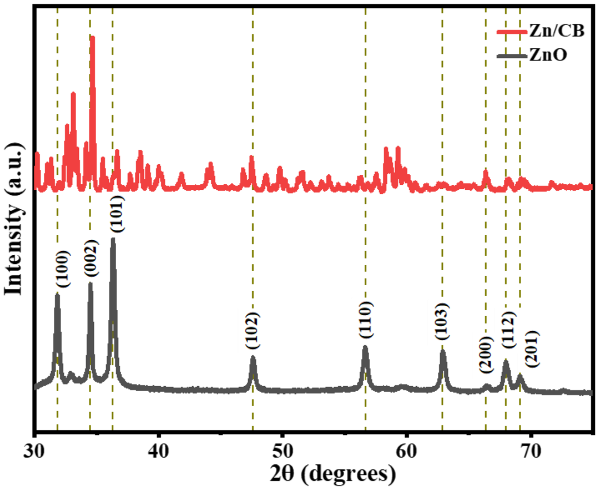

Figure 2. XRD curves of ZnO (Grey) and ZnO/CB (Red) sample.

The pristine ZnO sample exhibits distinct sharp and intense peaks at 2θ angles near 31.8°, 34.4°, 36.3°, 47.6°, 56.6°, 62.9°, 66.4°, 68°, and 69.1°, connects to (100), (002), (101), (102), (110), (103), (200), (112), and (201) crystal planes. From (JCPDS- 36-1451), it is confirmed that these are the peaks of the hexagonal ZnO structure [19]. Using the Debye-Scherrer equation applied of the prominent (101) peak, the average crystallite size of the pristine ZnO nanoparticles was calculated to be around 47nm. The ZnO/CB composite showed a noticeable decrease in the intensity of ZnO peaks along with slight broadening of the peaks. This broadening and decrease in peak intensity is due to the amorphous property of the Carbon Black component.

3.2. Morphology study using FESEM

The FESEM image of ZnO nanoparticles **(Figure 3a)** reveals a highly agglomerated microstructure. The material exhibits densely packed grains that form large, cauliflower like aggregates [20]. Whereas the FESEM image of ZnO/CB nanocomposite **(Figure 3b)** exhibits a heterogeneous and highly textured architecture. The ZnO/CB nanocomposite's structure appears more interconnected with increased surface roughness, suggesting a successful integration of the CB particles.

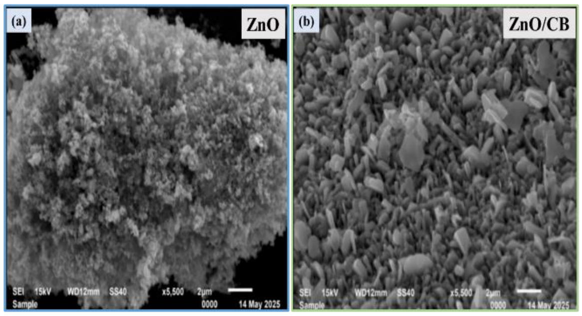

Figure 3. FESEM images (a) pure ZnO, and (b) ZnO/CB composite at two-micrometer scale.

Such an increase in granular morphology and textural complexity is generally favorable for electrochemical applications. It can facilitate improved electrolyte interaction and support enhanced charge storage performance as observed in the following electrochemical evaluation [21], [22].

3.3. Electrochemical behaviors study

Cyclic Voltammetry (CV) analysis revealed distinct redox peaks, attributable to oxidation and reduction processes, which align with the reports from previous studies on ZnO-based electrodes [23], [24]. In this setup, ZnO material was deposited onto the working electrode, while the Ag/AgCl (reference electrode) and Platinum (counter electrode) were used as other electrodes in a three-electrode configuration. 1M KOH was used as electrolyte and 0 V to 0.55 V was optimized as working potential window. CV curves at several scan rates (10 to 100 mV s^{-1}) for both ZnO and ZnO/CB samples are depicted in **Figure 4(a-b)**. ZnO electrode materials showed two distinct peaks at 0.31 and 0.5V. These oxidation and Redox peaks depicts that the charge storage mechanism is pseudocapacitance. These strong redox peaks are due to the K^+ ions intercalation/de-intercalation into the ZnO composite [25]. Upon incorporation of CB into ZnO, significant enhancements in the electrochemical behavior were observed in CV analysis. Graphically the area of the I-V curve increased significantly. The integration of Carbon Black changed the shape of the curve to quasi-rectangular with oxidation and reduction peaks around 0.35V and 0.5V. This indicates that the ZnO/CB composite comprises of both pseudocapacitance and electrochemical double-layer capacitance as the charge storage mechanism. As depicted in **Figure 4c**, when both materials were evaluated at a scan rate of 10 mV s^{-1}, the S_c of ZnO/CB almost reached 375 F g^{-1}, nearly three times more than the S_c of pristine ZnO (128 F g^{-1}), under same conditions. The S_c of the sample was estimated for different scan rates as shown in **Figure 4d**. Formula applied for calculating specific capacitance (S_c) [3] from the CV curves is:

$$S_c = \frac{\int i.dv}{m \times v \times \Delta V} \quad (F\,g^{-1}) \qquad (1)$$

Where area under CV curve is $\int i.dv$ (mA V), active mass loading (m) is 1 mg, scan rate is v (mV s^{-1}), and potential window ΔV (V).

GCD was performed to further describe the mechanism involved in charge accumulation and evaluate the capacitive performance of ZnO and ZnO/CB electrodes (**Figure 5**). Similar trends of the CV analysis is verified through the GCD curves also (**Figure 5a**). The discharging curve of the ZnO composite is nonlinear, which shows the pseudocapacitance as the charge storage mechanism. But in the ZnO/CB composite, after 0.35V, the discharge curve tends to be linear, showing the Electrochemical Double Layer capacitance component of charge storage. This clearly indicates the dual charge mechanism of the ZnO/CB (**Figure 5b**).

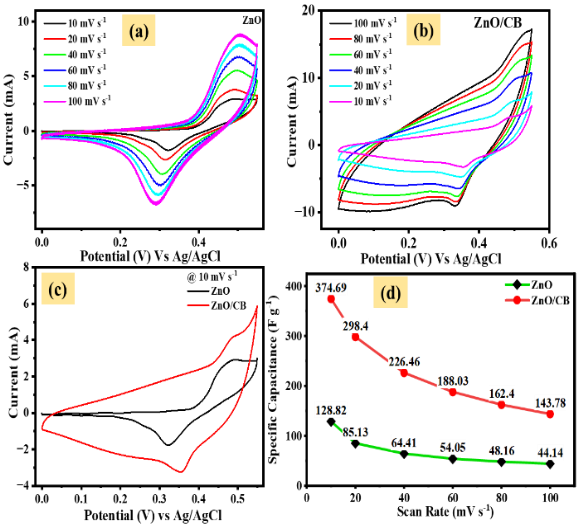

Figure 4. Cyclic voltammetry curves (a) ZnO, (b) Zn/CB, (c) evaluation at 10 mV s^{-1}, and (d) calculated S_c against scan rate for both prepared samples.

Figure 5c presents the comparative GCD profiles for the pristine ZnO, pure CB, and ZnO/CB nanocomposite electrodes, recorded at a current density of 4 A g^{-1}. The ZnO/CB nanocomposite exhibited the maximum discharge time, followed by pristine ZnO and finally the pure CB electrode, proving a genuine synergistic effect. The substantial prolongation of the discharge time confirms that the enhanced charge storage capacity arises from a cooperative interaction between the ZnO and the conductive CB network.

Figure 5(a-c) depicts the GCD curves for Zn/CB display noticeably prolonged charge time and discharge time at all tested current densities as compared to pristine ZnO, further implying a substantial improvement in charge storage capacity. The direct comparison at current density 4 A g^{-1}, highlights a dramatic improvement. In GCD analysis, the specific capacitance (S_c) [26] was determined by:

$$S_c = \frac{\Delta t \ \times I}{\Delta V \ \times m}\ (F\ g^{-1}) \tag{2}$$

Here, discharge time is Δt (s), Current density is I (A g^{-1}), potential window is ΔV (V), and active mass loading is m. The maximum specific capacitance shown by the Zn/CB sample is 183 F g^{-1}, while ZnO shows 15 F g^{-1} @ 4 A g^{-1}.

The disparity between CV and GCD derived specific capacitance values can be attributed to the fundamentally different operational mechanisms of the two techniques. At low scan rate of 10 mV s^{-1}, there is ample time for electrolyte ions to diffuse deeply and bulk Faradaic reactions to occur, yielding higher capacitance. Whereas the lower GCD-derived values are due to the high current density of 4 A g^{-1}, the charge-discharge process does not have enough time for proper diffusion of the electrolyte ions, and only the outermost surface of the electrode participates in charge storage.

Long-term cycling **(Figure 5e)** was evaluated for ZnO/CB nanocomposite over 3,000 cycles at a current density of 15 A g^{-1}. Remarkably, the composite exhibited an excellent durability, with the specific capacitance actually increasing over every cycle, making the capacitance retention increase to 145%. It depicts that this electrode actually gets better as it operates. This increase is caused by the activation of the electrode. Initially, the electrolyte only interacts with outer surface of the electrode. However, the continuous charging and discharging acts as a driving force, slowly pushing the liquid electrolyte deeper into the electrode. This exposes previously inaccessible interior surface area and activates deeply embedded ZnO redox sites. The continuous integration of these fresh active sites counteracts any structural degradation and demonstrates exceptional capacity retention [27]. The coulombic efficiency improved from 87% to 94%, confirming the high reversibility and structural stability of the ZnO/CB electrode. Electrochemical Impedance Spectroscopy (EIS) was also performed on the ZnO/CB nanocomposite electrode. The resulting

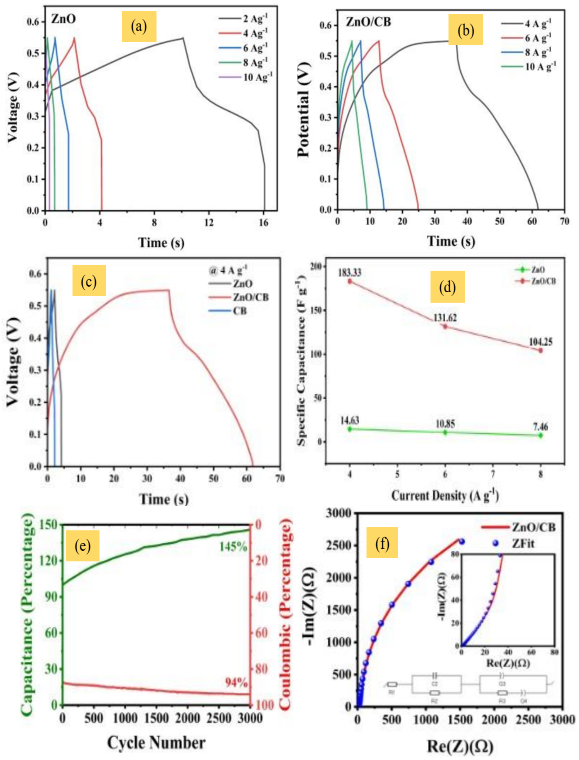

Figure 5. GCD curves: (a) ZnO, (b) ZnO/CB composite, (c) GCD of both ZnO and ZnO/CB 4 A g^{-1}, and (d) calculated S_c against current density for both prepared samples. (e) Capacitance retention and Coulombic retention over 3,000 cycles at 15 A g^{-1} of ZnO/CB composite. (f) Nyquist plot of ZnO/CB composite with equivalent fitted circuit.

Nyquist plot **(Figure 5f)** was fitted to an equivalent circuit. This composite demonstrates a notably low equivalent series resistance (R_s) of 1.1 Ω. Furthermore, the charge transfer resistance (R_{ct}) is restricted to a low value of 47 Ω. These low impedance values confirm that the integration of CB establishes a robust conductive network and facilitates rapid electron transport.

4. Conclusion

This study highlights a significant improvement in electrochemical performance achieved by incorporating carbon black into zinc oxide to form a Zn/CB nanocomposite. Pristine ZnO, while cost-effective and environmentally benign, suffers from low electrical conductivity, which limits its application as an efficient electrode material. The strategic inclusion of carbon black addresses this drawback establishing a highly conductive network within the composite, that significantly enhances the electron transportation. Electrochemical testing showed dual charge storing mechanism which increased S_c of 128 F g^{-1} for pristine ZnO to 375 F g^{-1} for ZnO/CB @ 10 mV s^{-1} scan rate and from 15 F g^{-1} for ZnO to 128 F g^{-1} for Zn/CB @ 4 A g^{-1} current density. This notable increase underscores the role of conductive additives in improving the overall charge storage performance of metal oxide-based materials. Furthermore, the simplicity and scalability of the synthesis method, coupled with the low cost of the components, position the ZnO/CB nanocomposite as a promising candidate for practical applications in energy storage. Overall, this work offers valuable insights into the design of efficient, low-cost, and environmentally sustainable electrode materials.

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