RJC

RASĀYAN *J. Chem.*
Vol. 18 | No. 1 |124-131| January - March | 2025
ISSN: 0974-1496 | e-ISSN: 0976-0083 | CODEN: RJCABP
http://www.rasayanjournal.com
http://www.rasayanjournal.co.in

SYNTHESIS AND ELECTROCHEMICAL PERFORMANCE OF $ZnFe_2O_4$/GO-BASED ELECTRODE MATERIAL

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ABSTRACT

The present work demonstrates a low-cost synthesis and electrochemical potential of $ZnFe_2O_4$/GO-based material for supercapacitors. The synthesized electrode material has been characterized via different analytical methods. The electrochemical investigation has been conducted using a three-electrode system for evaluating maximum specific capacitance. The maximum capacitance was found to be 622.2 F/g at the current density of 2 A/g and a retention of 95.3% after 5000 GCD cycles. The specific capacitance was calculated to be 113.3 F/g at a current density of 2 A/g in a two-electrode system. It has been found that the synthesized material could potentially be utilized in device systems with a power density of 879.2 W/kg and an energy density of 12.7 Wh/kg.

Keywords: $ZnFe_2O_4$/GO, Synthesis, Characterization, Electrochemical Potential, Supercapacitors.
RASĀYAN *J. Chem.*, Vol. 18, No.1, 2025

INTRODUCTION

Supercapacitors (SCs) are very prominent energy storage devices because of their exceptional power capacity and cycle life, making them perfect for renewable energy generation and transportation.[1,2] Ferrite-based materials have been used in microwave, magnetic, and electrical devices. Ferrites exhibit diverse oxidation states and surface redox reactions, making them ideal electrode materials. The uses of spinel ferrite magnetic nanoparticles include water purification, sensing, biology, and catalysis.[3-10] Zinc ferrite ($ZnFe_2O_4$) has significant economic importance in sensors, magnetism, catalysis, water purification, and energy storage. The low-cost and controlled synthetic processes make $ZnFe_2O_4$ a more reliable material.[6] $ZnFe_2O_4$ has demonstrated great potential for SC applications because of its eco-friendliness, strong electrochemical activity, and abundance of resources.[7] High stability, large surface area, outstanding electrical and thermal conductivities, lightweight, and excellent mechanical properties are some of the advanced features of graphene. Therefore, graphene-based materials are effectively used in energy storage, environmental protection, electronics, and other areas due to their advanced features.[11] The $ZnFe_2O_4$-based hybrid materials such as activated carbon/multiwalled carbon nanotubes/$ZnFe_2O_4$, $ZnFe_2O_4$/CNTs, $ZnFe_2O_4$/polypyrrole, $ZnFe_2O_4$/polyaniline, and $ZnFe_2O_4$/C-based nanocomposites were effectively adopted as electrode materials for SCs in the recent past.[7,12-15] The present study demonstrates a cost-effective synthetic approach for $ZnFe_2O_4$ and graphene oxide-based electrode material for SCs. The electrochemical study was conducted using three and two-electrode systems. Various analytical techniques were also used for the characterization of $ZnFe_2O_4$/GO.

EXPERIMENTAL

Materials Used

Zinc chloride ($ZnCl_2$), ferric chloride ($FeCl_3$), sodium hydroxide (NaOH), sodium nitrate ($NaNO_3$), sulphuric acid (H_2SO_4), graphite, N-Methyl-2-pyrrolidone (NMP), carbon black, potassium hydroxide (KOH), polyvinylidene difluoride (PVDF), and double distilled water (DDW) were used in the present work. All of these chemicals were AR-grade.

Synthesis and Characterisation of $ZnFe_2O_4$/GO

1 g of $ZnCl_2$ and 2 g of $FeCl_3$ were mixed and dissolved in 100 mL of distilled water. The mixture was agitated for 60 min, and then a few drops of 0.1 M NaOH were added. This mixture was again agitated for 60 minutes at 60 °C. The precipitation of $ZnFe_2O_4$ was separated via centrifugation washed three times with

http://doi.org/10.31788/RJC.2025.1819065

DDW and then dried in a hot air oven. The dry powder of $ZnFe_2O_4$ was calcined at 650 °C for 3.5 hrs. Graphene oxide (GO) was prepared via Hummer's modified method. In this method, a conical flask is placed over an ice bath containing 0.5 g of graphite powder, 0.5 g of NaNO3, and 23.5 mL of concentrated H_2SO_4. Next, a steady addition of 3 g of $KMnO_4$ was carried out. After adjusting the water bath to 35°C, the solution continues to stir for 1 hr. This solution was incorporated with 50 mL of DDW and then agitated at 90 °C for 30 min. To this mixture, add 100 mL of DDW and 3 mL of 30% H_2O_2. The solution changed from dark brown to brilliant yellow in color. DDW was used to wash the residue after the solution's centrifugation. Once the solution had reached total neutrality, this process was repeated; the residue (GO) that was left behind was then fully dried in a vacuum oven. $ZnFe_2O_4$ and GO have been mixed with 100 mL of DDW in a 1:1 ratio. This mixture was stirred on a magnetic stirrer for 1 hr and then sonicated for 3 hrs. In a hot air oven, the $ZnFe_2O_4$/GO solution was then thoroughly dried. Different analytical techniques such as Fourier transform infrared (FTIR), X-ray diffraction (XRD), scanning electron microscopy (SEM), and energy-dispersive X-ray spectroscopy (EDX) have been used to characterize $ZnFe_2O_4$/GO.

Electrode Preparation and Electrochemical Study

The electrode slurry was obtained by incorporating a few drops of NMP into a mixture of $ZnFe_2O_4$/GO, carbon black, and PVDF (8:1:1). This slurry was coated onto Ni-foam and then dried in a hot air oven for 6 hrs. The electrochemical investigation of $ZnFe_2O_4$/GO has been carried out using different electrochemical parameters under three and two-electrode systems. The 1 M KOH solution has been used as an electrolyte for this investigation. Under the three-electrode system, $ZnFe_2O_4$/GO, Ag/Ag^+, and Pt-wire were considered as working, standard, and counter electrodes. The following mathematical equation was used to evaluate the specific capacitance from galvanostatic charge-discharge (GCD) curves:

$$C\ (F/g) = I\ \Delta t/m\Delta V \quad (1)$$

In the above equation, the current, mass of active material, discharge time, and voltage are presented by I, m, t, and V, respectively. The power density (P) and energy density (E) of the active material were calculated via the following equations:

$$E\ (Wh/kg) = 0.5\ CV^2/3.6 \quad (2)$$

$$P\ (W/kg) = E \times 3600/t \quad (3)$$

RESULTS AND DISCUSSION

Characterisation of $ZnFe_2O_4$/GO

Figure-1 (a) presents the FTIR spectra of $ZnFe_2O_4$ and $ZnFe_2O_4$/GO. The FTIR peaks of $ZnFe_2O_4$ were found to be 3464, 2885, 1633, and 625 cm^{-1} for $ZnFe_2O_4$. These peaks are associated with the presence of O-H, C-H (Stretching; introduced from citric acid), C-O (Stretching), and Zn-Fe bonds on the surface of $ZnFe_2O_4$.[16-18] Similarly, the FTIR peaks such as 3484, 2852, 1620, and 615 cm^{-1} were observed for $ZnFe_2O_4$/GO. These peaks are indicating the presence of O-H, C-H (symmetric CH_2), C=C, and Zn-Fe bonds.[17-19] Figure-1 (b) presents the XRD patterns of $ZnFe_2O_4$ and $ZnFe_2O_4$/GO. The XRD patterns of $ZnFe_2O_4$ and $ZnFe_2O_4$/GO are almost the same because the GO is a carbon-based material. The main XRD peaks are obtained at $2\theta = 35.5^0$ (311), 43.1^0 (400), 54.2^0 (422), 56.6^0 (511), 62.5^0 (440), and 75.4^0 (533), respectively. These peaks are related to JPCDS 22-1012.[20-23]

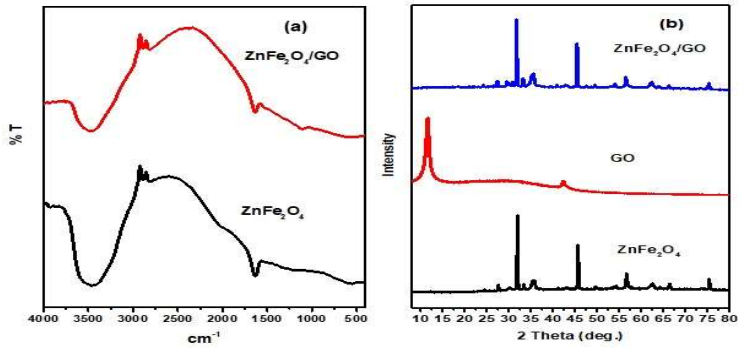

Fig.-1: (A) FTIR Spectra and (B) XRD Patterns of $ZnFe_2O_4$ and $ZnFe_2O_4$/GO

Figure-2 depicts SEM images of $ZnFe_2O_4$, GO, and $ZnFe_2O_4$/GO. Sphere-shaped particles of $ZnFe_2O_4$ appear in the SEM images of 2 (a) and (b). GO sheets are visible in Fig.-2 (c) and (d). After the combination of $ZnFe_2O_4$ and GO, the sphere-shaped particles of $ZnFe_2O_4$ have been embedded in the sheets of GO (Fig.-2 e & f). The EDX spectrum and EDX composition of $ZnFe_2O_4$/GO are presented in Fig.-3 and Table-1.

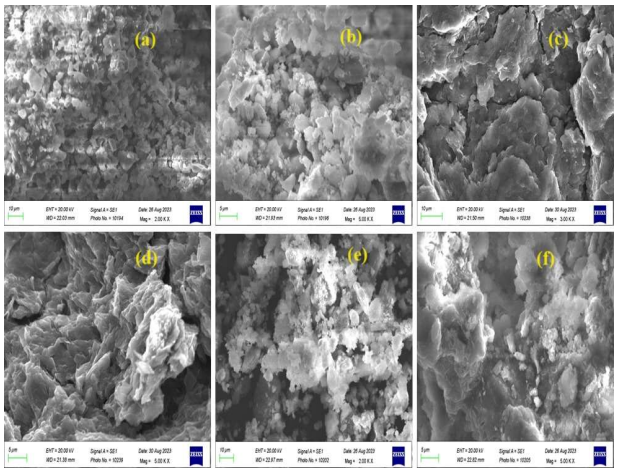

Fig.-2: SEM Images of (a) & (b) $ZnFe_2O_4$, (c) & (d) GO, and (e) & (f) $ZnFe_2O_4$/GO

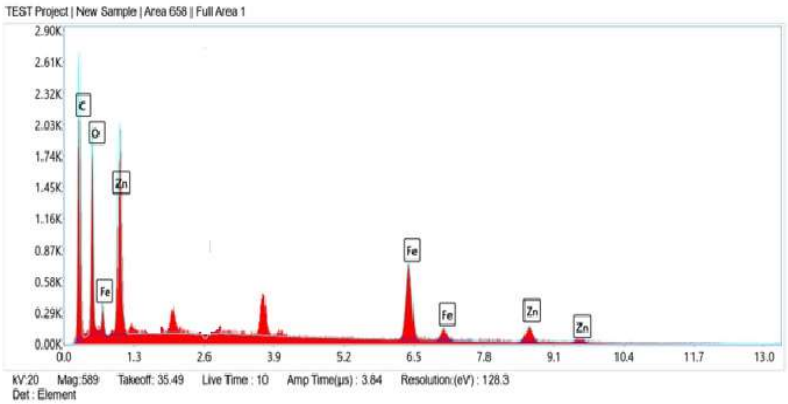

Fig.-3- EDX Spectrum of $ZnFe_2O_4$/GO

Table-1: EDX Composition of $ZnFe_2O_4$/GO

Elements	Weight %	Atomic %
C K	33.8	50.0
O K	23.1	26.3
Fe K	27.8	13.9
Zn K	15.3	9.8

Electrochemical Study of $ZnFe_2O_4$/GO

Cyclic voltammetry (CV) is an extensively used essential method for the electrochemical analysis of electrode materials used in energy storage devices. CV curves primarily reveal the oxidation-reduction potential, material feasibility, and electron transport kinetics.[24] Catalytic reaction studies may additionally profit from this method.[25] The CV curves (under three and two electrode systems) of $ZnFe_2O_4$ and $ZnFe_2O_4$/GO are shown in Fig.-4. These curves have been obtained at different scan rates, i.e., 10, 20, 30, 50, 75, and 100 mV/s, respectively. The CV curves of the selected materials show their effectiveness for energy storage systems because of their rectangular shape.[26,27] In general, redox peaks broaden and distort with increasing scan rates. It suggests more potent polarisations at higher scan rates.[28] There are no redox peaks observable in these CV curves. It suggests there is ion accumulation rather than ions undergoing intercalation or deintercalation.[29] GCD, or galvanostatic charge discharge, is a fundamental method for determining a specific capacitance under controlled current conditions.[30] The assessment of energy storage materials and technologies frequently utilizes the use of GCD techniques. In GCD, a system is charged and

discharged within a specific potential limit through the use of continuous positive and negative currents. This process has to be repeated for several cycles. Capacitance, performance, power, and energy are assessed using GCD profiles.[31-33] Figure-5 presents the GCD curves of $ZnFe_2O_4$ and $ZnFe_2O_4$/GO under three and two electrode systems. The specific capacitance of $ZnFe_2O_4$ and $ZnFe_2O_4$/GO has decreased with the rise in current density (Fig.-6). It is precisely the electrochemical diffusion process that takes place during the storage phase.[33] At current densities of 2, 4, 6, 8, and 10 A/g, $ZnFe_2O_4$ exhibits specific capacitances of 301.2, 133.3, 106.6, 62.2, and 33.3 F/g in a three-electrode system. $ZnFe_2O_4$/GO shows a specific capacitance of 622.2, 320, 256, 248.8, and 187.7 F/g at the same current densities under a three-electrode system (Fig.-6 a). The specific capacitance of 113.5, 80, 60, 53.3, and 43.3 F/g for $ZnFe_2O_4$/GO was evaluated at current densities of 2, 4, 6, 8, and 10 F/g under a two-electrode system (Fig.-6 b). These results suggest that $ZnFe_2O_4$/GO is a promising material for high-efficiency SCs. The comparison of $ZnFe_2O_4$/GO with other electrode materials is shown in Table-2.

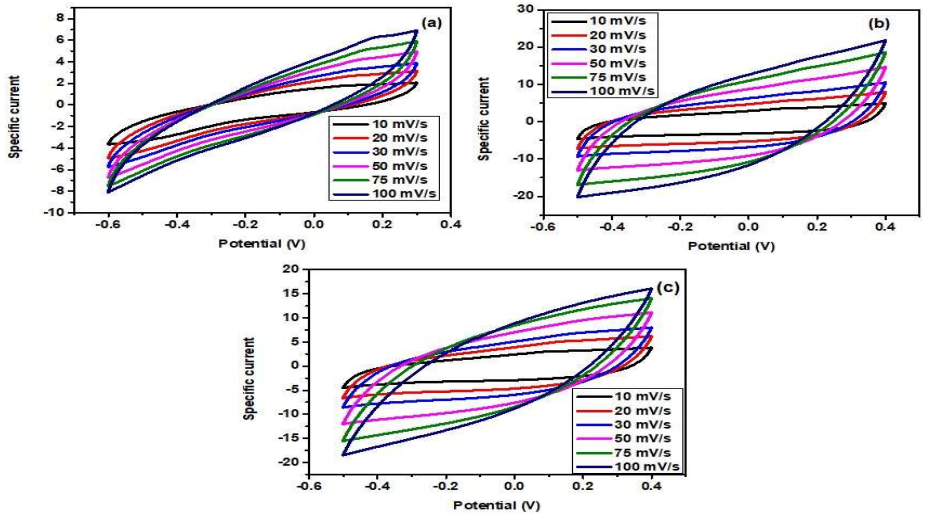

Fig.-4: CV Curves of (a) $ZnFe_2O_4$ (three electrodes), (b) $ZnFe_2O_4$/GO (three electrodes), and (c) $ZnFe_2O_4$/GO (two-electrode)

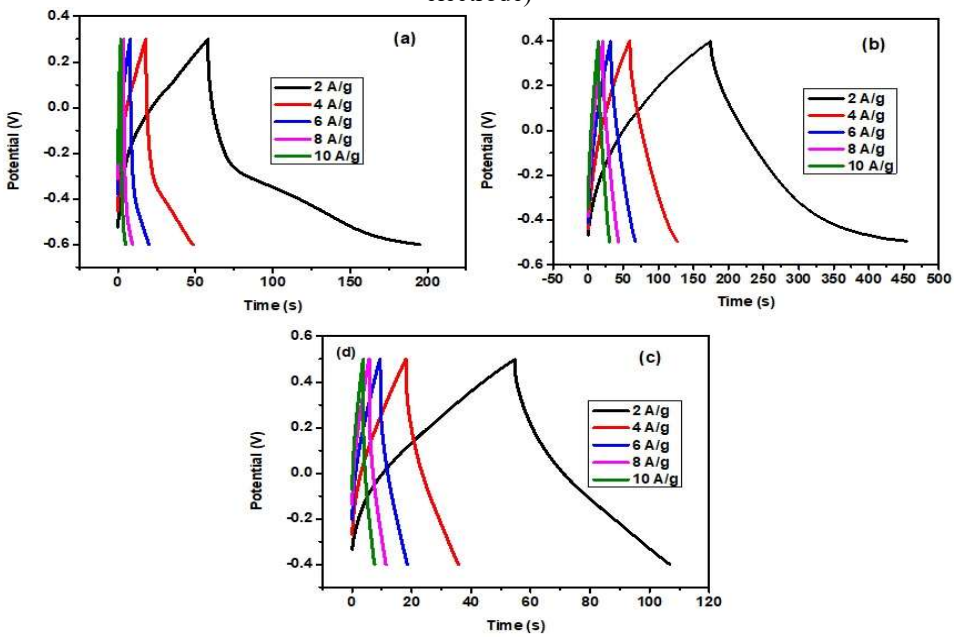

Fig.-5: GCD Curves of (a) $ZnFe_2O_4$ (three electrodes), (b) $ZnFe_2O_4$/GO (three electrodes), and (c) $ZnFe_2O_4$/GO (two-electrode)

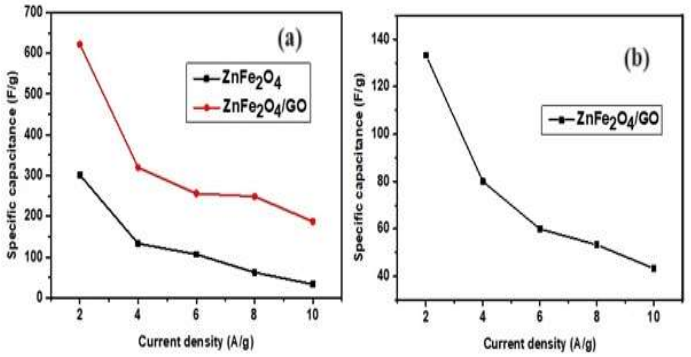

Fig.-6: Current Density vs Specific Capacitance (a) $ZnFe_2O_4$ and $ZnFe_2O_4$/GO (three electrode), and (b) $ZnFe_2O_4$/GO (two-electrode)

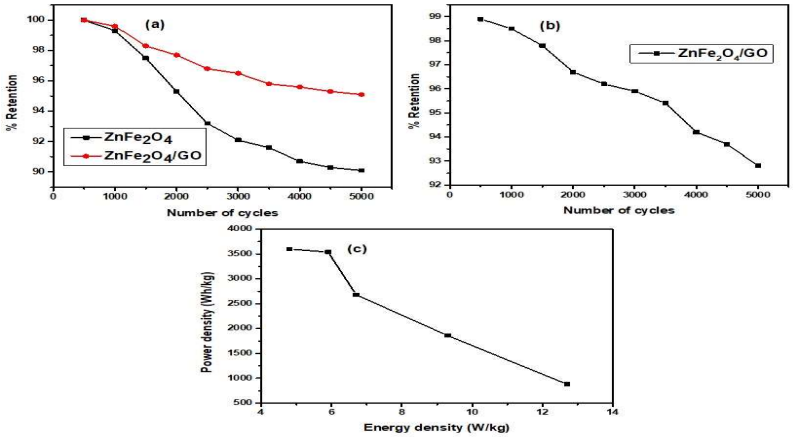

Fig.-7: Cyclic Stability of (a) $ZnFe_2O_4$ and $ZnFe_2O_4$/GO (three electrodes), (b) $ZnFe_2O_4$/GO (two electrodes), and (c) Energy Density vs Power Density

Table-2: Comparison of $ZnFe_2O_4$/GO with other Electrode Materials

Electrode materials	Scan rate (mV/s) or current density (A/g)	Electrolyte	Specific capacitance (F/g)	References
rGO/Ppy/Fe_2O_3	1 A/g	1 M KCl	125.7	[34]
rGO/Ppy/$CoFe_2O_4$	1 mV/s	1 M $LiNO_3$	107	[35]
$NiCoSe_2$	1 A/g	6 M KOH	535	[36]
$Ni_{0.5}Zn_{0.5}Fe_2O_4$	1 A/g	3M KOH	504.4	[37]
SnO_2/r-GO	1 A/g	1 M KOH	267.8	[33]
ZnO/CoS_2/NF	1 A/g	3 M KOH	400.2	[38]
$ZnFe_2O_4$/GO	2 A/g	1 M KOH	622.2	Present study

The $ZnFe_2O_4$/GO has been checked for cyclic stability under three and two-electrode systems. This experiment was completed for up to 5000 GCD cycles. Under the three-electrode system, 100% retention was observed after 500 GCD cycles, which decreased to 99.6% after 1000 cycles. As 2000 cycles were completed, a retention rate of 98.3 % was recorded. Retention after 3000 GCD cycles was observed to be 96.5%. Retention after 4500 GCD cycles was found to be 95.3%, which is 95.3 % recorded after 5000 GCD cycles (Fig.-7 a). On the other hand, in the two-electrode system, the retention of $ZnFe_2O_4$/GO was observed to be 98.9% after 500 GCD cycles, and it was observed to be 97.8% after 1500 cycles. After 3000 cycles, retention was recorded at 95.9%, which was evaluated at 94.2% after 4000 cycles; 92.8 % retention was evaluated after 5000 cycles (Fig.-7 b). The electrochemical activity of electrode materials is greatly affected by their energy density and power density.[39] In recognition of electrochemical energy storage devices, energy density is usually considered the primary objective. Batteries typically possess a higher energy density. The amount of electricity one can generate at a specific volume is known as the power density. SCs can provide energy substantially more quickly than batteries; they have a higher power density than

batteries.[40] The energy densities of $ZnFe_2O_4$/GO were found to be 12.7, 9.3, 6.7, 5.9, and 4.8 wh/kg at power densities of 879.2, 1860.1, 2680, 3540, and 3600 W/kg, respectively (Fig.-7 c).

CONCLUSION

$ZnFe_2O_4$/GO-based nanocomposite has been synthesized using an efficient and low-cost process and characterized using different available characterization techniques. $ZnFe_2O_4$/GO exhibited a high specific capacitance of 622.2 F/g at a current density of 2 A/g under a three-electrode system. This material was also found feasible for device systems, and its specific capacitance was found to be 113.3 F/g at a current density of 2 A/g under a two-electrode system. The energy densities of $ZnFe_2O_4$/GO have been evaluated as 12.7, 9.3, 6.7, 5.9, and 4.8 Wh/kg, respectively. Only 4.7% of $ZnFe_2O_4$/GO was lost after 5000 GCD cycles. All of these findings indicate that $ZnFe_2O_4$/GO was found to be a high-performance electrode material for SCs.

ACKNOWLEDGMENTS

We are very thankful to the Division of Research and Innovation, Uttaranchal University, Dehradun (India), for providing the necessary facilities during the experimental work.

CONFLICT OF INTERESTS

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.

AUTHOR CONTRIBUTIONS

All the authors contributed significantly to this manuscript, participated in reviewing/editing, and approved the final draft for publication. The research profile of the authors can be verified from their ORCID ids, given below:

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[RJC-9065/2025]