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Synthesis of rGO/$CoFe_2O_4$ Nanocomposite For Supercapacitor Application

P. SHARMA[1], R. KUMAR[1], DIKSHA[1] and R. K. SINGH[1, †]

[1]*Department of Physics and Astronomical Science, Central University of Himachal Pradesh, Kangra, H.P, India 176215*

†*E mail: rksinghbhu@gmail.com*

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Abstract. In this study, we have synthesized the composite of Reduced Graphene Oxide (rGO) with cobalt ferrites due to the presence of multiple oxidation states of cobalt ions that enhance the properties. The presence of rGO in cobalt ferrite also enhances the surface area and porous structure leading to enhanced electro- chemical properties for supercapacitor application. These properties include high specific capacitance (CS) and long cyclic stability. Here, the composite of rGO/cobalt ferrite ($rGO/CoFe_2O_4$) was prepared by microwave synthesis method and characterization techniques like X-ray Diffraction (XRD), Scanning Electron Microscopy (SEM) and Fourier Transformed Infrared Spectroscopy (FTIR) gave us the information of structural/ morphological and chemical compositions of rGO/$CoFe_2O_4$. The XRD peaks confirmed the formation of a single phase of $CoFe_2O_4$ and we have calculated the crystallite size which was found to be 11nm. The nanorods like structure appeared in the Scanning Electron Micrograph. Furthermore; we have done the electrochemical property measurement which gives us the specific capacitance of about 16.434 Fg^{-1} by performing cyclic voltammetry at scan rate $5mV\ s^{-1}$ and for other scan rates we got different values of specific capacitance.

Keywords: Reduced graphene oxide, cobalt ferrite, supercapacitor, electrochemical properties

1. Introduction

Energy plays very important role in social and economic development. Over last two centuries there has been radical improvement in standard of living because of the development of new technologies. So, the demand of the energy increases day by day because of increasing population and industrial change and

development. [**1-3**] Ferrites are the form of ceramic material which consists of Iron oxide (Fe_2O_4) mixed with other metal oxides like nickel, zinc, or manganese etc. Due to their distinctive electrical and magnetic properties, which include high electrical resistance and poor conductivity, they are utilized in a variety of electronic components, including inductors, transformers, and ferrite beads. Ferrites are also often utilized as electrode materials for supercapacitors due to their special characteristics that make them ideal for energy storage application. The increased surface area of ferrites in supercapacitor electrodes, enables more effective charge storage, and is one of their key benefits. Additionally, ferrites have a high electrical conductivity, which speeds up the transfer of charge carriers between the electrodes and produces a high-power density. Addition to that, ferrites are good magnetic materials that may be used for various purposes, such as magnetic storage and sensors. Overall, ferrites are an appealing material for application in a variety of electrochemical devices, including supercapacitors, due to the distinct mix of features they display. [**4-8**] Cobalt ferrite ($CoFe_2O_4$) is a particular kind of ferrite that has attracted a lot of attention. Cobalt ferrite has a number of advantageous characteristics, including high specific capacitance, strong magnetic anisotropy, mechanical hardness, chemical stability, and high corrosive force, which make it appropriate for storing energy in supercapacitor electrodes [**9**]. The high specific capacitance of cobalt ferrite, which is used in supercapacitors, is one of its main benefits. Cobalt ferrite also possesses outstanding electrical conductivity, which is essential for effective charge transfer inside the electrode material. Cobalt ferrite also exhibits strong cycling stability and durability over time, which means that its performance may be maintained through a number of charge-discharge cycles without noticeably degrading. Cobalt is a relatively rare and expensive element compared to other elements, which may have an impact on the price and scalability of cobalt ferrite-based supercapacitors. To address these issues, however, ongoing research and development projects attempt to improve synthesis techniques and investigate alternate materials. Continued research in this area holds the potential to enhance the performance and commercial viability of cobalt ferrite-based supercapacitors. Nanocomposites, which blend two or more different

materials at the nanoscale to provide synergistic features, are one such potential class of materials. In this situation, a strong nanocomposite system for supercapacitor applications has been attempted by combining reduced graphene oxide (rGO) with cobalt ferrite ($CoFe_2O_4$). A derivative of graphene called reduced graphene oxide is ideally suited for effective charge storage due to its high surface area and strong electrical conductivity. Over using the two elements separately, rGO and cobalt ferrite combined into a nanocomposite structure offer a number of benefits. First off, adding cobalt ferrite nanoparticles enhances the surface area of the rGO matrix, allowing for more active sites for charge absorption and improving specific capacitance. Additionally, the cobalt ferrite's electrochemical performance and rGO's high electrical conductivity work in concert to improve charge transfer kinetics and lower internal resistance, which increases power density. Additionally, the structural stability and mechanical strength of the nanocomposite architecture may prevent nanoparticle aggregation and retain the electrode's integrity after numerous charge-discharge cycles. This characteristic is essential for attaining long-term cycle stability and making sure supercapacitors are durable [**10**]. In conclusion, this study has discussed the synthesis, characterization, and use of rGO/cobalt ferrite nanocomposite as electrodes in supercapacitors. The performance and energy storage capacity are investigated when rGO and cobalt ferrite were combined. Excellent electrochemical characteristics of the nanocomposite make it an attractive option for application in high-performance supercapacitors. To optimize the synthesis procedure and enhance the performance of the nanocomposite in real-world applications, more study is necessary.

2. Experiment

2.1 Materials used

Graphite powder, Sulfuric acid (H_2SO_4) 98wt. %, Potassium permanganate ($K\ MnO_4$), Dilute HCl, 30% Hydrogen peroxide (H_2O_2) aqueous, and Ferric chloride hexahydrate, ($FeCl_3 6H_2O$), Cobalt chloride hexahydrate of ($CoCl_2 6H_2O$). All chemicals were purchased from Merck India.

2.2 Preparation of GO nanosheets

GO was prepared by various methods, and one of them was the modified Hummers method, which was used in this study to prepare GO nanosheets. In a conical flask, 120ml of H_2SO_4 was taken, and graphite powder was added to it. The conical flask was put on the magnetic stirrer while being kept on an ice bath. $KMnO_4$ was gradually added into the flask until the solution turned green. After overnight stirring of the solution, 100ml of deionized water was added gradually. After that, the flask was placed in a container for a hot bath for an hour, and then 10ml of hydrogen peroxide (30%) was added until the solution appeared a bit yellowish. After that, the solution was stirred, and 90ml of distilled water was added. Furthermore, 15ml of hydrochloric acid was poured into the solution, causing it to turn yellow. Then, distilled water was added. The solution was left overnight at room temperature. Finally, the resulting material was filtered, washed several times with deionized water until neutrality was obtained, and the color of the material appeared brownish. The sample was then dried in an oven for 24 hours at a temperature of 80 degrees Celsius [**11**].

2.3 Preparation of $CoFe_2O_4$

Dissolve $CoCl_24H_2O$ and $FeCl_36H_2O$ in deionized water and stir the solution for 2hours at different temperatures (20 ,40 ,60 and 80.), NaOH is used as a base; add dropwise until the mixture is solidified. Precipitates were filtered and thoroughly washed with deionized water. Dried the mixture at 70^0 C and the ultrafine powder was obtained.

2.4 Preparation of $CoFe_2O_4$/rGO

GO was dispersed in 100ml of deionized water with sonication for 1h. Mix 0.1 M of $CoCl_26H_2O$ and 0.2M of $FeCl_36H_2O$ in deionized water. The above two solutions were mixed together under the stirring condition with rise in temperature up to 80 C and the solution color turns into brownish to yellowish. Dry the solution in hot air oven. After drying the solution, it can put into the microwave oven for microwave

irradiation for 10 minutes. Grind the sample and brownish color powder was obtained.

3. Results and Discussion

3.1 XRD analysis:

The XRD technique was used to assess the crystal structure and phase purity of the synthesized samples. The peak positions regarding different planes provide valuable information about the crystal lattice and its arrangement. By comparing the obtained 2Θ values with known standard reference data, the crystallographic phases present in the samples can be identified. Figure 1 revealed five distinct peaks of various planes: (220), (311), (400), (511), and (440), corresponding to 2Θ values of 30.3°, 35.7°, 43.3°, 57.3°, and 62.7°, respectively for $CoFe_2O_4$ JCPDS Card No. 22-1086. The (311) plane, characterized by the peak at 35.7°, exhibited the highest intensity among all the observed peaks. This suggests that the crystallographic orientation of the material in this plane is predominant. It is important to note that the presence of an additional peak at 65.7°, not corresponding $CoFe_2O_4$(JCPDS Card no. 22-1086), may be attributed to impurities.

To determine the crystallite size, the Scherer's formula was employed.

$$D = \frac{K.\lambda}{\beta \cos\theta} \qquad (1)$$

This formula utilizes the wavelength of X-rays emitted from the target material (λ), Scherer's constant (K), the Bragg's angle (θ), and the greatest intensity at half its entire breadth (β). By applying the formula, the crystallite size was calculated for the maximum intensity peak, which was found to be 10.37 nm for the (311) plane. The crystallite size represents the average dimensions of the crystalline domains within the material, providing insights into its microstructure.

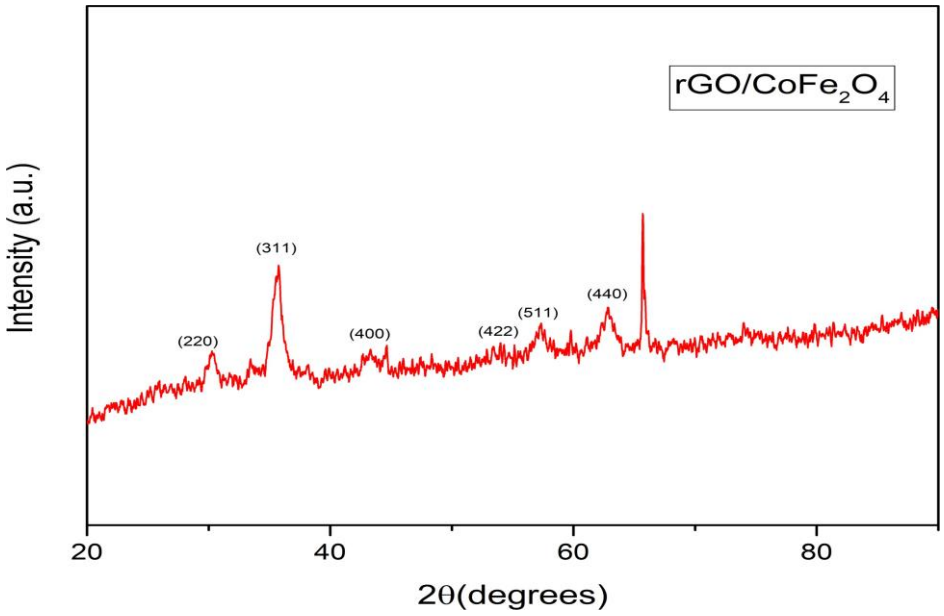

Fig. 1: XRD Patterns of rGo/$CoFe_2O_4$

3.1 Field Emission Scanning Electron Microscope (FE-SEM):

All of the synthesized composite's microstructure and morphology were examined using a field emission scanning electron microscope (FE-SEM). The scanning electron microscope (SEM) images of the $CoFe_2O_4$/rGO nanocomposite displays nanorods indicate the presence of a specific structural morphology at the nanoscale as shown in figure 2. Nanorods are elongated structures with a high aspect ratio, wherein their length greatly exceeds their width. In the context of the $CoFe_2O_4$/rGO nanocomposite, the observed nanorods in the SEM images suggest that the composite material comprises elongated particles or clusters arranged in a rod-like fashion. The morphology of nanorods holds significant implications for the properties and potential applications of the material. Nanorods often exhibit enhanced mechanical, electrical, and optical characteristics compared to other morphologies due to their elongated shape and increased surface area. Additionally, the alignment of nanorods may result in anisotropic properties, wherein the material displays varying characteristics along different directions.

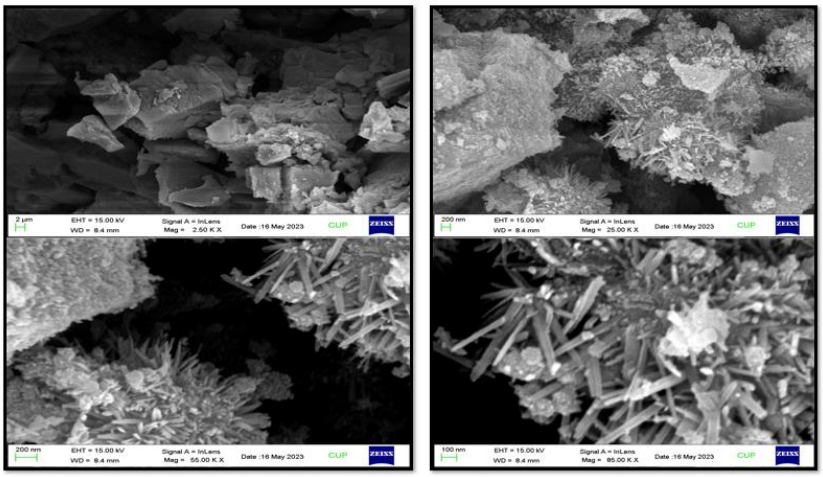

Fig. 2: SEM images shows nanorods like structure of rGO/$CoFe_2O_4$ nanocomposite

3.2 Electrochemical Analysis:

The Cyclic voltammetry measurements offer insights into the electrochemical behavior and performance of the nanocomposite as an electrode material. The electrochemical performance of active material for the supercapacitor application, cyclic voltammetry study (CV) fig **3** and galvanostatic charging-discharging (GCD) measurements was performed in a three-electrode system. In this three electrodes system, one is working electrode which consists of active material and another two are the reference electrode and the auxiliary electrode /counter electrode which consists of Ag-AgCl and platinum respectively. NaOH has been used as an electrolyte. From this study we calculated the specific capacitance near about 16.434$F\ g^{-1}$ at scan rate 5$mV\ s^{-1}$ and we got the different values of specific capacitance for different scan rates as shown in table 1. also, rGO/$CoFe_2O_4$ demonstrates pseudocapacitance, an electrochemical phenomenon associated with Faradaic processes that involve fast surface redox reactions, leading to additional energy storage beyond the electrical double layer capacitance. Firstly, rGO provides a highly conductive pathway, enabling efficient charge transport throughout the composite material. This conductivity is advantageous for applications such as energy storage and electronic devices. Additionally, the special surface structure of rGO, with its high surface area, facilitates the deposition of nanometer-sized $CoFe_2O_4$

particles. The deposition of $CoFe_2O_4$ onto the rGO surface helps maintain the mechanical strength of the composite material, preventing agglomeration or loss of structural integrity.

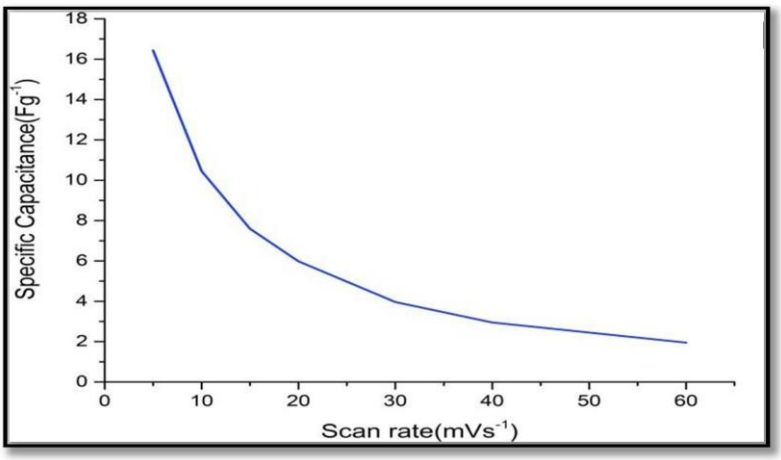

Fig. 3: Graphical representation of CV curves of $CoFe_2O_4$

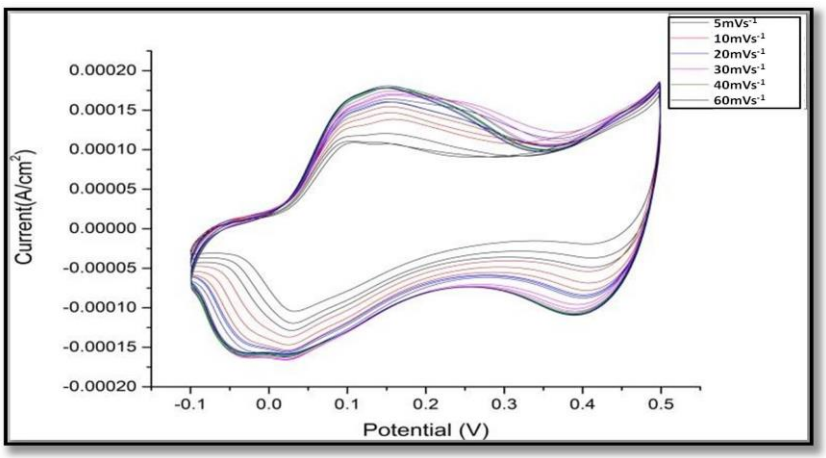

Fig. 4: Specific Capacitance curve for different Scan rates

Table 1: Different values of specific capacitance for different Scan rate

Scan Rate (mV s^{-1})	Specific Capacitance ($F\,g^{-1}$)
5	16.43420463
10	10.44924774
15	7.595402041
20	5.979665987
30	3.963354401
40	2.949529321
60	1.949476976
100	5.973392816

4. Conclusion

In this study the composite of rGO/$CoFe_2O_4$ was synthesized in which the inclusion of reduced graphene oxide (rGO) serves multiple purposes. The XRD results indicate the crystal structure and phase composition of the nanocomposite. In the case of the rGO/$CoFe_2O_4$ nanocomposite, the XRD pattern reveals the presence of characteristic diffraction peaks corresponding to both reduced graphene oxide (rGO) and cobalt ferrite ($CoFe_2O_4$). The SEM images provide valuable information about the morphology and surface characteristics of the rGO/$CoFe_2O_4$ nanocomposite. From the SEM analysis, it can be observed that the nanocomposite exhibits a well-defined structure with a homogeneous distribution of $CoFe_2O_4$ nanorods on the surface of the rGO sheets. The SEM images also reveal the presence of interconnected pores and a high surface area, which are beneficial for various applications. The cyclic voltammogram obtained for the rGO/$CoFe_2O_4$ nanocomposite exhibits characteristic oxidation and reduction peaks, indicating the occurrence of redox reactions during the electrochemical cycling process. The observed redox peaks suggest that the nanocomposite possesses excellent electrochemical activity and charge storage capabilities. Also, the specific capacitance of the material was calculated from the CV curve and a maximum specific capacitance of $16.434F\ g^{-1}$ at scan rate $5mV\ s^{-1}$ was found. In summary, the XRD analysis confirms the presence of RGO and $CoFe_2O_4$ phases in the

nanocomposite, while the SEM images reveal a well-defined morphology with good distribution of $CoFe_2O_4$ nanoparticles on RGO sheets. The cyclic voltammetry results demonstrate the nanocomposite's electrochemical activity and potential for energy storage applications. These findings collectively suggest that the rGO/$CoFe_2O_4$ nanocomposite holds promise as a versatile material with applications in various fields, such as energy storage, catalysis, and sensing.

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