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PREPARATION, CHARACTERIZATION AND APPLICATION OF SPINEL $MgFe_2O_4$ NANOPARTICLE

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Abstract

The present research deals with a study on the preparation, characterization and application of spinel $MgFe_2O_4$ nanoparticles. The spinel $MgFe_2O_4$ nanoparticle was synthesized by sol-gel method using oxalic acid. Magnesium nitrate and ferric nitrate were used as starting materials. The elemental composition of the prepared nanoparticle was characterized by Energy Dispersive X-ray Fluorescence (EDXRF). The crystallite size, phase ID and lattice parameters of the prepared nanoparticle were characterized by X-ray powder diffraction (XRD) spectroscopy. The type of chemical bonding in the prepared nanoparticle was characterized by Fourier Transform Infrared (FT IR) spectroscopy, and porosity and morphology of the prepared nanoparticle were characterized by Scanning Electron Microscopy (SEM) spectroscopy. The energy band gap of the prepared nanoparticle was determined by the Tauc Plot method. The electrochemical property of the prepared nanoparticle was determined by the cyclic voltammetry method. The specific capacitance of the prepared nanoparticle was determined from the cyclic voltammetry graph.

Keywords: spinel nanoparticles, sol-gel method, XRD, EDXRF, cyclic voltammetry and specific capacitance

Introduction

The environment has become contaminated in, i.e. air, water, and land, due to excess use of pesticides, fertilizers, global industrialization, and dumping of chemicals and pollutants from industries. An attempt to solve diverse problem is now being sorted out by the use of nanotechnology (Paramita, 2018). Environmental engineering science is witnessing drastic changes. In a similar manner, nanotechnology is moving from one visionary paradigm toward another. Nanotechnology is the frontier of science and engineering today for solving such problems. (Sukanchan, 2018).

Nanotechnology has emerged as a rapidly growing field with multifaced application for new materials at the nanoscale level. Nanoparticles are of great interest due to their unique physicochemical, magnetic, and optoelectronic properties that are governed by their size, shape, and size distribution and they have a small size and large surface area to volume ratio that lead to the significant differences in properties in their bulk form. Due to unique physicochemical and optoelectronic properties, nanoparticles are interest for a number of applications like catalysts, chemical sensors, electronic components, medical diagnostic imaging, pharmaceutical products and medical treatment proposal (Babita, 2018).

Today, various methods such as the sol-gel method (solution method), vapor phase compression method, mechanical alloying method or collision with high-energy pellets, plasma method, and electrochemical methods are used for the production and synthesis of nanoparticles. Although all the mentioned methods have the ability to produce large volumes of nanomaterial, the sol-gel method has a higher popularity and industrial application than other existing methods [Feinle, Haruta, 2016]. Due to its unique properties and characteristics, this method is capable of producing high quality nanoparticles of the same size on an industrial scale [Tian, 2007].

The sol-gel process is a bottom-up synthesis method. In this process, the final products are formed by performing a number of irreversible chemical reactions [Sarkar, 2012]. The rate of reaction of salts depends on various factors such as pH, concentration, type of solvent, and temperature. The main advantages of the sol-gel process are the high purity of the product, the

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narrow particle size distribution, and the achievement of uniform nanostructure at low temperatures. This method is commonly used to synthesize metal nanooxides.

In this research, $MgFe_2O_4$ nanoparticle was synthesized from magnesium nitrate, ferric nitrate, oxalic acid and ethylene glycol by so-gel method. $MgFe_2O_4$ is an important multifunctional material, which has been widely applied in catalysts and lithium-ion batteries because of their interesting catalytic and electrochemical properties [Hench, 1990]. Recently, $MgFe_2O_4$ nanoparticle has been successfully used as electrode material for pseudocapacitors due to the synergistic effects from iron and magnesium [Verma, et al 2015].

Materials and Methods

All chemicals were analytical grade. Magnesium nitrate ($Mg(NO_3)_2.6H_2O$), ferric nitrate ($Fe(NO_3)_3.\ 9H_2O$) and oxalic acid ($C_2H_2O_4$) were Merck product with a purity of 99.99 %. Ethylene glycol was product from Applichem, Germany. All solutions were prepared using distilled water during preparation procedures. Various conventional and modern instrumental techniques were used throughout the experimental procedure. These include Energy Dispersive X-ray Fluorescence (EDXRF), X-ray Diffraction (XRD), Fourier transform infrared (FT IR) spectroscopy, Scanning electron microscopy, cyclic voltammetry and UV-Visible spectroscopy.

Preparation of spinel $MgFe_2O_4$ nanoparticle using oxalic acid

In a typical experiment, 50 mL of 0.8 M $Mg(NO_3)_2.6H_2O$ was dissolved in 100 mL distilled water as well as 50 mL of 0.8 M $Fe(NO_3)_3.9H_2O$ was dissolved in 100 mL distilled water and they were mixed to obtain the first solution. The second solution was prepared by dissolving 50 mL of 0.8 M oxalic acid in 100 mL of distilled water and the first solution was mixed with the second solution. 10 mL ethylene glycol was added into the above mixture solution. The solution was heated and stirred at 80 °C for about 4 h on a magnetic stirrer to get gel material. The gel was dried at 100 °C in a hot air oven for about 2 h. The dried material was obtained. The dried material was crushed to fine powder with a mortar and pestle. The fine powder was transferred into a crucible and calcinated at 500 °C for about 4 h in a muffle furnace. Then the required $MgFe_2O_4$ nanoparticle was obtained.

Characterization of prepared spinel $MgFe_2O_4$ nanoparticle

Crystal structure and phase analysis were performed by X-ray diffraction (XRD) using Rigaku, D-Max 2200, Japan in the Department of Chemistry, Yangon University. The elemental composition of the prepared $MgFe_2O_4$ NP was confirmed by using the EDXRF 700 spectrometer in the Department of Chemistry, Monywa University. The chemical bonding in the prepared nanoparticle was characterized by (FT IR) in the Department of Chemistry, Monywa University. Porosity and morphology of prepared nanoparticles were determined by (SEM) in the Department of Chemistry Magway University.

Determination of crystallite size and interatomic spacing of prepared spinel $MgFe_2O_4$ nanoparticle

The crystallite sizes of spinel $MgFe_2O_4$ NP can be calculated by using Debye-Scherrer's formula,

$$D = \frac{0.9\lambda}{\beta \cos\theta} \text{ , and}$$

interatomic spacing (d) was calculated by Bragg's equation

$$d = \frac{\lambda}{2\sin\theta}$$

Where, λ = the wave length of X-rays (λ =1.540560Å)

θ = the diffraction angle

β = full width at half maximum in radian

D = average crystallite size

d = interatomic spacing

Determination of energy band gap of prepared spinel $MgFe_2O_4$ nanoparticle

The energy band gap of the prepared nanoparticle was measured by UV-Vis spectroscopy in the Department of Chemistry, University of Mandalay.

0.01g of $MgFe_2O_4$ NP was dissolved in 5 mL of distilled water and was settled for 12 h. 2 mL of the upper layer of the solution was taken and its absorbance was measured at 200 -750 nm range. To measure energy, the band gap of the prepared $MgFe_2O_4$ NP, the wavelength and related absorbance data were used in scientific 2018 origin software.

Cyclic voltammetry of prepared spinel $MgFe_2O_4$ nanoparticles

The electrochemical properties of the prepared $MgFe_2O_4$ nanoparticles were determined by cyclic voltammetry technique. 0.05 g of prepared $MgFe_2O_4$ NP was used. Electrochemical properties of the prepared nanoparticle were determined using three electrode systems (Ag/AgCl as a reference electrode, platinum as a counter electrode and FTO coated with $MgFe_2O_4$ NP as a working electrode) in 1 M KOH as a supporting electrolyte with scan rates of 50 mVs^{-1}.

Fabrication of prepared spinel $MgFe_2O_4$ nanoparticles

0.05 g of $MgFe_2O_4$ nanoparticles and 5 mL of ethylene glycol were mixed. The solution was heated in the water bath for 2 h. The $MgFe_2O_4$ nanoparticle gel solution was obtained. And then fluorine doped tin oxide (FTO) was coated with $MgFe_2O_4$ nanoparticle gel solution and dried at 80°C. $MgFe_2O_4$ coating film was obtained.

Determination of electrochemical behaviors of prepared spinel $MgFe_2O_4$ nanoparticle from cyclic voltammograms

To determine the electrochemical properties of prepared $MgFe_2O_4$ nanoparticles, the data of the CV graph were obtained by the cyclic voltammetry technique at a scan rate of 50 mVs^{-1} and 1M KOH electrolyte solution. The CV graph was plotted using origin 2018 software based on the obtained CV data. The electrochemical properties of the prepared nanoparticles were analyzed by interpreting the current and potential values.

Determination of specific capacitance of prepared spinel $MgFe_2O_4$ nanoparticle

The specific capacitance of prepared $MgFe_2O_4$ nanoparticles was determined using cyclic voltammetry in Department of Chemistry, at the University of Mandalay.

The cyclic area of the CV graph was measured from CV graph in origin software. The specific capacitance of the prepared nanoparticle was calculated using cyclic area and CV data according to the following equation.

$$C_p = \frac{A}{2mk\Delta V}$$

Where: C_p = specific capacitance

A = cyclic area

m = mass of active material

k = scan rate

ΔV = potential window

Results and Discussion

In this section, it contains characterization of the prepared spinel $MgFe_2O_4$ nanoparticles by EDXRF, XRD, FT IR, and SEM spectroscopic techniques. Furthermore, determination of energy band gap, and specific capacitance of the prepared spinel $MgFe_2O_4$ nanoparticle were discussed.

Yield percent of prepared spinel $MgFe_2O_4$ nanoparticle

The prepared powder was calcinated at 500˚C for 4h in a muffle furnace. The yield % of spinel $MgFe_2O_4$ NP was calculated from the mass of metals in initial nitrates. The value of this sample was shown in Table 1.

Table 1 Result of Yield Percent of Prepared Spinel $MgFe_2O_4$ Nanoparticle

Sample	Yield percent of sample (%)
$MgFe_2O_4$	88

According to the Table 1, it is found that yield percent of calcinated prepared spinel $MgFe_2O_4$ NP is 88%.

EDXRF analysis of prepared spinel $MgFe_2O_4$ nanoparticle

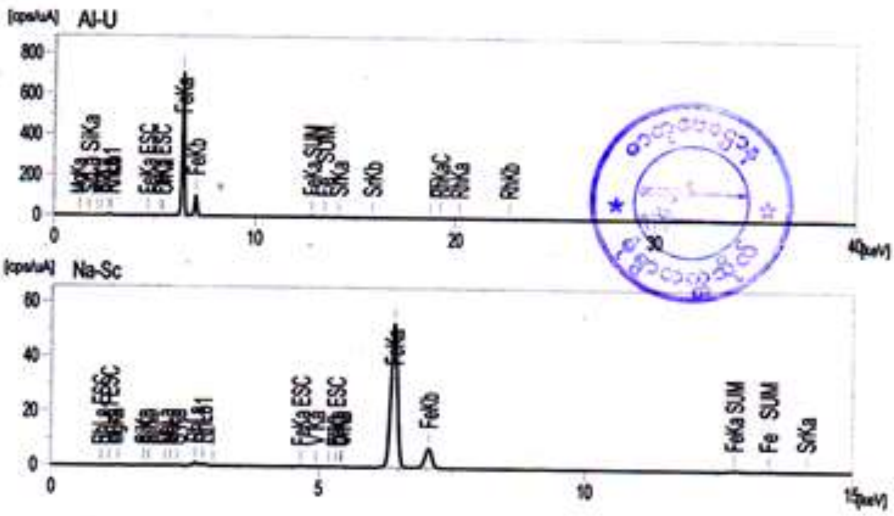

Figure 1 EDXRF spectrum of prepared spinel $MgFe_2O_4$ NP

Table 2 Elemental Composition of Prepared Spinel $MgFe_2O_4$ NP by EDXRF

No	Analytes	Results (%)
1	Fe	59.342
2	Mg	38.976
3	Si	1.174
4	S	0.327
5	Sr	0.106
6	Cr	0.075

From the EDXRF data, amount of Fe, Mg, Si, S, Sr and Cr in prepared spinel $MgFe_2O_4$ NP were found to be 59.342 %**,** 38.976 %, 1.174 % , 0.327%, 0.106 % and 0.075 % respectively.

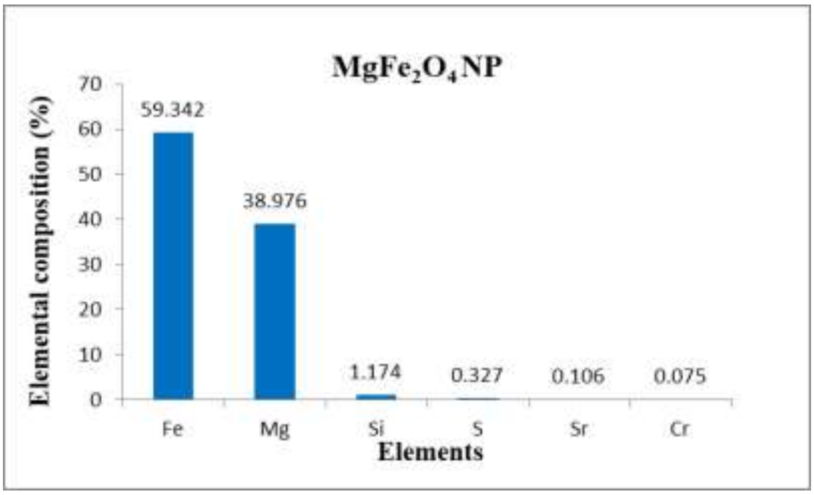

Figure 2 Bar graph showing elemental composition of prepared spinel $MgFe_2O_4$ NP

XRD analysis of prepared spinel $MgFe_2O_4$ nanoparticle

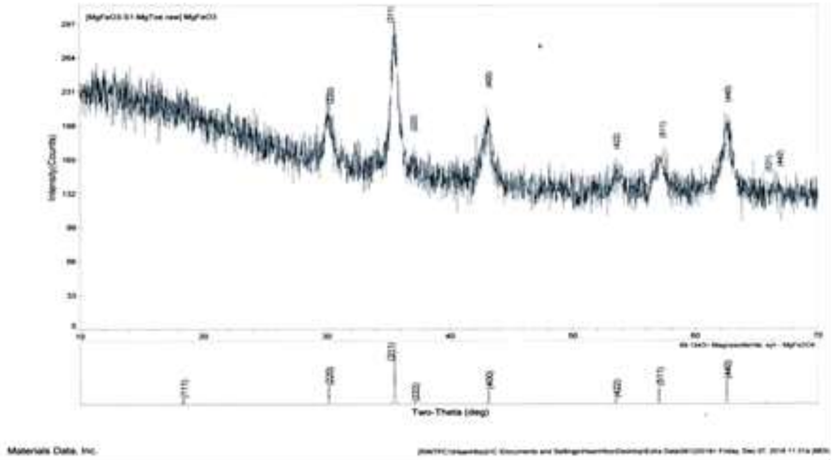

Figure 3 XRD Diffractogram of prepared spinel $MgFe_2O_4$ NP

Table 3 The Crystallite Size and Phase ID of Prepared Spinel $MgFe_2O_4$ NP

No	Bragg angle (2θ)	Miller indices (h k l)	β (radian)	Inter planar spacing d(nm)	Crystallite size D (nm)	Phase ID
1	30.261	2 2 0	0.01195	0.29511	13.85	$MgFe_2O_4$
2	35.483	3 1 1	0.01059	0.25278	29.42	$MgFe_2O_4$
3	37.028	2 2 2	0.00508	0.24258	28.89	$MgFe_2O_4$
4	43.113	4 0 0	0.00723	0.20965	20.69	$MgFe_2O_4$
5	53.490	4 2 2	0.01227	0.17116	12.72	$MgFe_2O_4$
6	57.251	5 1 1	0.01166	0.16078	13.59	$MgFe_2O_4$
7	62.614	4 4 0	0.01185	0.14824	13.73	$MgFe_2O_4$
8	65.910	5 3 1	0.00795	0.14160	20.69	$MgFe_2O_4$
9	66.859	4 4 2	0.00212	0.13982	81.56	$MgFe_2O_4$

The XRD spectrum of $MgFe_2O_4$ nanoparticles was indicated in Figure 3. The diffracted peaks are (220), (311), (222), (400), (422), (511), (440), (531) and (442). All peaks were well matched with those of the standard JCPDF (Joint Committee on Powder Diffraction standards) library file of magnesium ferrite ($MgFe_2O_4$). The most dominant peak at (222) plane was found at 37.028 deg. XRD patterns indicate that the crystal structures of $MgFe_2O_4$ were cubic structures. The crystallite sizes of $MgFe_2O_4$ were tabulated in Table 3. The average crystallize size was 26.13 nm.

Table 4 Lattice Parameters for Prepared Spinel $MgFe_2O_4$ Nanoparticle

Crystal Structure	a-Axis (nm)	b-Axis (nm)	c-Axis (nm)	α, °	β, °	γ, °
Cubic	0.83469	0.83469	0.83469	90.00	90.00	90.00
Cubic	0.83838	0.83838	0.83838	90.00	90.00	90.00
Cubic	0.84031	0.84031	0.84031	90.00	90.00	90.00
Cubic	0.83859	0.83859	0.83859	90.00	90.00	90.00
Cubic	0.83853	0.83853	0.83853	90.00	90.00	90.00
Cubic	0.83545	0.83545	0.83545	90.00	90.00	90.00

The average lattice constants calculated from the XRD pattern for spinel $MgFe_2O_4$ nanoparticles are a= b = c = 0.838 nm and $\alpha = \beta = \gamma = 90^\circ$. The crystal structures are cubic structures.

FT IR Analysis of Prepared Spinel $MgFe_2O_4$ Nanoparticle

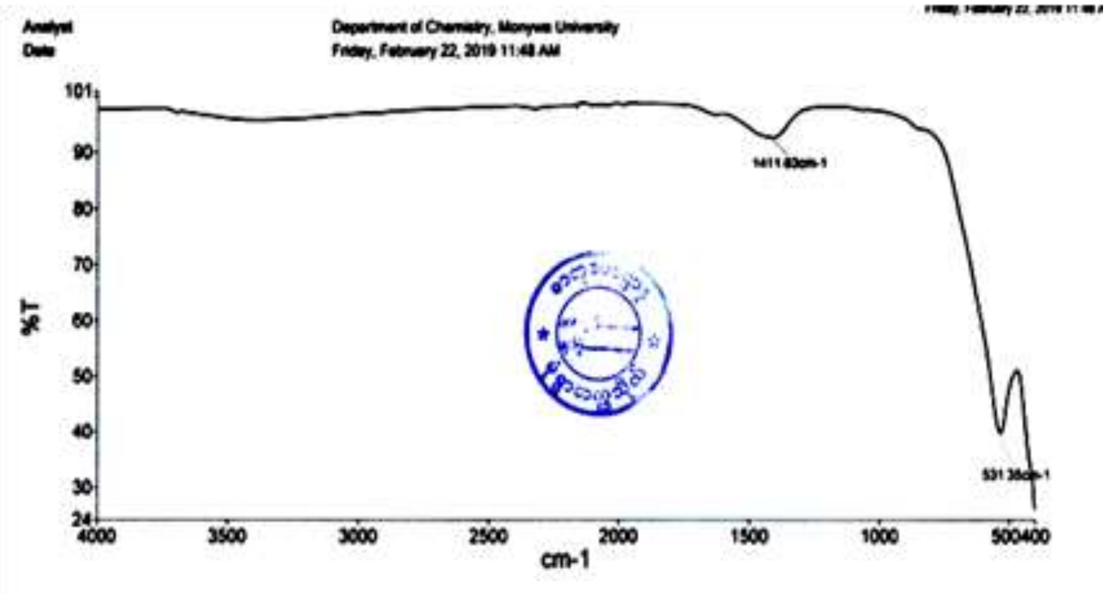

Figure 4. FT IR spectrum of prepared spinel $MgFe_2O_4$ NP

Table 5 FT IR Spectra Data for Prepared Spinel $MgFe_2O_4$ NP

Sample	Wave number (cm^{-1})	Remark	*Literature values
$MgFe_2O_4$	531.35	Stretching vibration of Mg-O and Fe-O bonds	650-430cm^{-1}

* Waldron R D (1955), Infrared Spectroscopy of Ferrites Phy Rev.

According to the Table 5, in FT IR spectrum of $MgFe_2O_4$ NP, stretching vibration of Mg-O and Fe-O bonds occurred at 531.35 cm^{-1} of wavenumber.

SEM analysis of prepared spinel $MgFe_2O_4$ nanoparticle

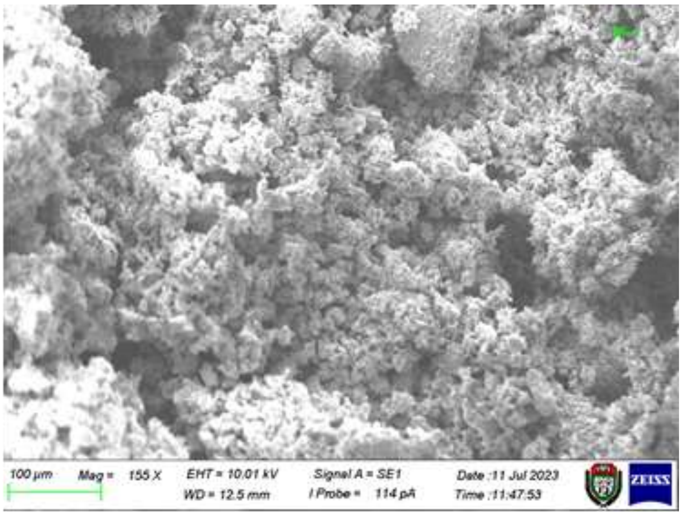

Figure 5 SEM spectrum of prepared spinel $MgFe_2O_4$ NP

The morphology of the spinel $MgFe_2O_4$ nanoparticles indicates irregular cubic shapes of various sizes that are agglomerated.

Determination of energy band gap of prepared spinel $MgFe_2O_4$ NP

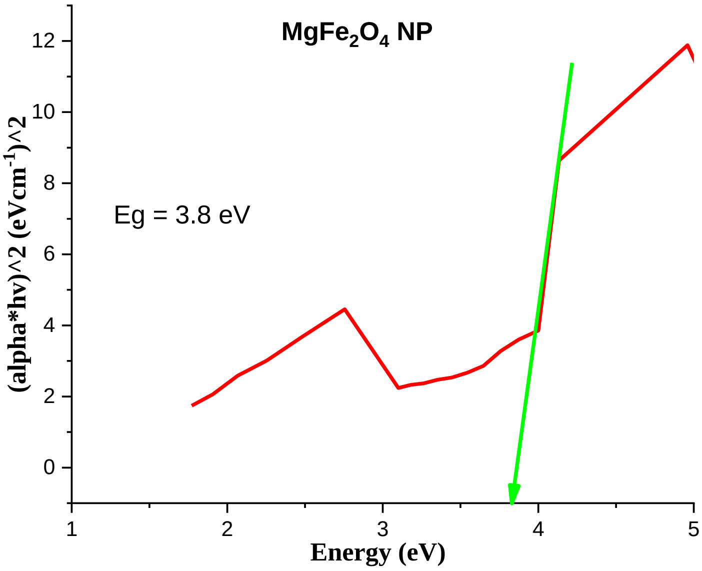

Figure 6 Tauc plot graph of prepared spinel $MgFe_2O_4$ NP

The energy band gap of prepared $MgFe_2O_4$ NP was found to be 3.8 eV. According to the Tauc plot method, $MgFe_2O_4$ NP is a type of little semi-conductor.

Determination of electrochemical property of prepared spinel $MgFe_2O_4$ NP

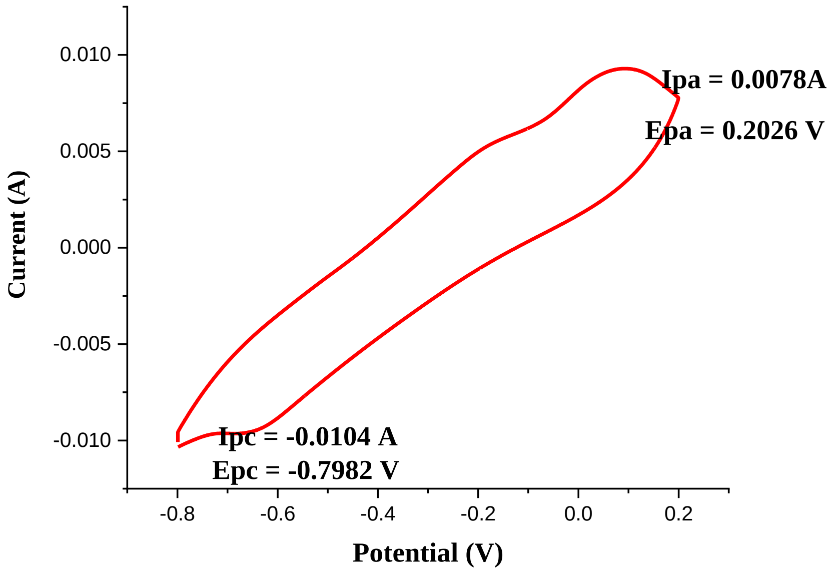

Figure 7. Cyclic voltammetry graph of $MgFe_2O_4$ NP with scan rate of 50 mVs^{-1} in 1M KOH solution

Table 6 The Value of Anode and Cathode Peak Current Densities of 0.05 g of $MgFe_2O_4$ NP in 1 M of KOH Electrolyte Solution

Scan rate (mV/s)	Electrolytes (1M)	Samples	Ipc(A)	Epc(V)	Ipa(A)	Epa(V)
50	KOH	$MgFe_2O_4$	-0.0104	-0.7982	0.0078	0.2026

According to this table, cathode and anode peaks potentials of $MgFe_2O_4$ were found at -0.7982V and 0.2026 V, respectively.

Determination of specific capacitance of prepared spinel $MgFe_2O_4$ NP

Parameters of $MgFe_2O_4$NP Data were as follows.

Scan rate (K) = 50 mV/s

Mass of $MgFe_2O_4$ nanoparticle (m) = 0.05g

Potential window (V_2-V_1) ΔV = 0.2- (-0.8) = 1.00 V

Area inside the CV curve (A) = 0.0051 AV

$$C_p = \frac{A}{2mK\Delta V} = \frac{0.00506}{2\times 0.05\times 50\times 1.00\times 10^{-3}}$$

$$= 1.012 \text{ F/g}$$

Calculated Specific Capacitance Cp of $MgFe_2O_4$ NP = 1.01 F/g

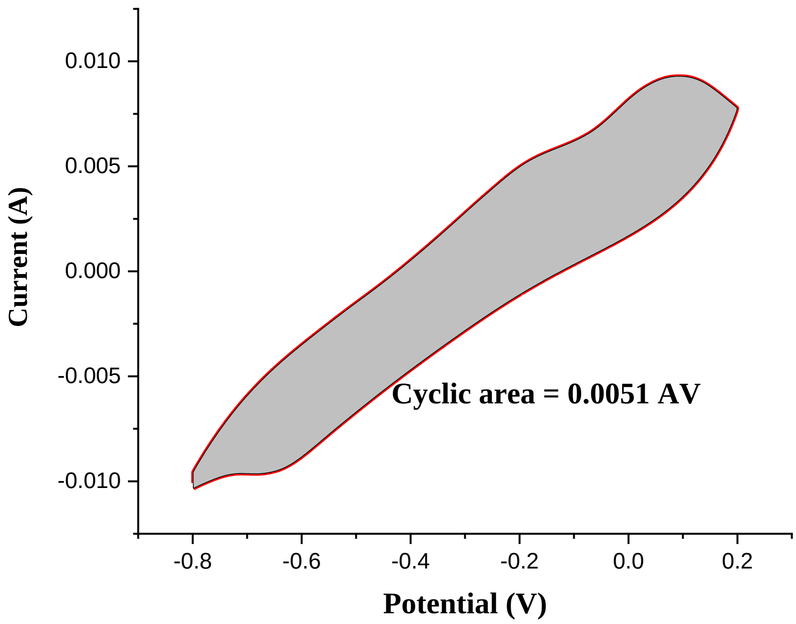

Figure 8 Cyclic area of prepared spinel $MgFe_2O_4$ NP

Conclusion

In this research, spinel $MgFe_2O_4$ nanoparticle was synthesized from magnesium nitrate and cobalt nitrate by sol gel method using oxalic acid. The prepared spinel nanoparticle was characterized by modern sophisticated methods such as EDXRF, XRD, FT IR and SEM spectroscopic techniques. From the characterization of modern techniques, the prepared $MgFe_2O_4$ particle is a nanoparticle since its crystallite size is in the range of nanoscale (1 – 100 nm). The crystal structure of the prepared spinel $MgFe_2O_4$ nanoparticle is cubic.

According to the determination of the energy band gap of prepared $MgFe_2O_4$ NP by the Tauc Plot method, it is a type of semiconductor because of the 3.8 eV energy band gap value. Therefore it can be used in electronic devices such as solar cells. According to the determination of electrochemical property by cyclic voltammetry, it is found that $MgFe_2O_4$ NP performs as an electrode in 1M KOH electrolyte solution. According to the determination of specific capacitance of prepared $MgFe_2O_4$ NP, its capacitance value is 1 F/g. So this nanoparticle can also be used in charging batteries since its capacitance value is a significant amount.

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Here are some suggested questions based on the provided research summary:

1. Questions on Synthesis and Characterization:

- What is the role of oxalic acid in the sol-gel synthesis of $MgFe_2O_4$ nanoparticles?
- Why were magnesium nitrate and cobalt nitrate chosen as precursor materials?
- What evidence from XRD confirms that the prepared $MgFe_2O_4$ nanoparticle has a cubic spinel structure?
- How do EDXRF, XRD, FT-IR, and SEM contribute to the characterization of $MgFe_2O_4$ nanoparticles?
- What is the observed crystallite size of the synthesized $MgFe_2O_4$ nanoparticles, and how was it determined?

2. Questions on Optical and Semiconductor Properties:

- How was the energy band gap of $MgFe_2O_4$ determined using the Tauc Plot method?
- What is the significance of the 3.8 eV band gap in classifying $MgFe_2O_4$ as a semiconductor?
- How does the band gap value affect the potential application of $MgFe_2O_4$ in solar cells?

3. Questions on Electrochemical Properties and Applications:

- How was cyclic voltammetry used to determine the electrochemical performance of $MgFe_2O_4$ nanoparticles?
- What does the specific capacitance value of 1 F/g indicate about the potential use of $MgFe_2O_4$ in energy storage devices?
- Why was 1M KOH chosen as the electrolyte in electrochemical studies?
- How does the electrochemical behavior of $MgFe_2O_4$ compare to other common electrode materials used in supercapacitors or batteries?

4. Questions on Potential Applications:

- What are the advantages of using $MgFe_2O_4$ nanoparticles in electronic devices such as solar cells?
- How does the specific capacitance value of $MgFe_2O_4$ compare to conventional capacitor materials?
- What are the possible improvements or modifications to enhance the performance of $MgFe_2O_4$ in energy storage applications?

These questions can help guide further discussions and exploration of the research findings. Let me know if you need more specific questions!