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Microwave Assisted Synthesis of $CoFe_2O_4$ Nanoparticles by Utilizing Organic Promoters and Evaluation of Its Properties †

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Abstract: Nano-sized spinel ferrites are highly regarded owing to their special optical, electrical, and magnetic properties. Cobalt ferrite ($CoFe_2O_4$) is a nominee of particular interest due to its high saturation magnetization, high coercivity, strong anisotropy, and excellent chemical stability. The synthesis of these materials with a pure crystalline phase is sometimes limited due to the required high temperatures for their calcination. In this work, we report a one-pot simple synthesis procedure of cobalt ferrite by the auto-combustion under microwave irradiation into a domestic microwave oven with a power of 900 W for 30 min. Glycine and ammonium nitrate were used as organic promoters and metal nitrates as precursors. The synthesized nanoparticles were characterized by Fourier transform infrared spectroscopy (FT-IR), X-ray diffraction (XRD), scanning electron microscopy (SEM), and energy dispersion of X-ray spectrometry (EDX) techniques. The electrochemical properties and capability of the prepared product as a pseudocapacitive material were evaluated using cyclic voltammetry (CV) tests in details.

Keywords: nanoparticles; magnetic ferrite; microwave; auto-combustion

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1. Introduction

Amongst various types of nanomaterials, magnetic nanoparticles (MNPs) have been widely studied for decades because of their special properties and their extensive set of applications from catalysis to biomedicine [1,2]. Spinel ferrites with a common formula of MFe_2O_4, (M: Fe, Mn, Co, Ni, Cu, Zn, etc.) are a large category of this family, which have attracted attentions due to their outstanding magnetic and electrical properties alongside their semiconducting properties [3–5]. Cobalt ferrite ($CoFe_2O_4$) nanoparticles as an interesting member of these materials have notable features such as excellent chemical stability, high coercivity, strong anisotropy, and great saturation magnetization [6]. Various methods such as co-precipitation, sol-gel, thermal decomposition, etc., have been reported for the synthesis of $CoFe_2O_4$ [5–7]. Generally, the synthesis of a pure phase of magnetic ferrites with a suitable crystallinity needs to use a post calcination treatment with high temperature, which can raise the production costs. With the purpose of acquiring proper results, we report the simple low-cost technique of microwave assisted auto-combustion. Glycine was used as a fuel and also driving agent in this process. This method enables us to decrease the reaction time and supply sufficient heating energy to form a pure phase of product by one-pot reaction. Furthermore, since this process is carried out in a solid state without using any organic solvent it can be classified as an eco-friendly method in green chemistry domain [8]. As the properties evaluation of the synthesized cobalt ferrite, we examined its electrochemical properties as a pseudocapacitive nanomaterial by performing cyclic voltammetry (CV) tests.

2. Experimental Features

2.1. Initial Materials

All initial reactants were provided from Merck Co. (Kenilworth, NJ, USA) and used without further purification.

2.2. Synthesis Procedure

Fe $(NO_3)_3 \cdot 9H_2O$ and $Co(NO_3)_2 \cdot 6H_2O$ with a proper stoichiometric ratio in the presence of Gly and ammonium nitrate as fuel/driving agents were mixed to each other in solid state. The mixture put into a domestic microwave oven with a power of 900 W for 30 min. The prepared magnetic product was collected, washed, dried overnight, and then characterized in details.

2.3. CV Tests

Cyclic voltammetry test has been performed to evaluate electrochemical characteristics of the prepared $CoFe_2O_4$ nanoparticles in H_2SO_4 aqueous solution (0.5 M) at the different scan rates of 10 to 100 mV s^{-1} with potential window of −0.6 to 0.8 V. A conventional three-electrode system on an electrochemical PG-State 204 workstation, Iran was used for this aim. The electrodes of Pt and saturated calomel were selected as the counter and reference electrodes, respectively.

The specific capacitance (C_{sp}) was estimated by integration of voltammetric charges divided by potential window as follow:

$$Csp = \frac{\int \mathrm{IdV}}{\nu \mathrm{mV}} \quad (1)$$

where I is the response current, V is the potential, v is the scan rate potential (Vs^{-1}), m (g) is the mass of active materials loaded onto the working electrode [9]. The working electrode was constructed and coated on a graphite piece substrate using the desired amounts of product, carbon, polytetrafluoroethylene, and absolute [10].

2.4. Characterizations

X-ray diffraction (XRD) pattern was recorded by a DRON-8 powder diffractometer (Mission Company, Russia) using Cu Kα radiation (λ = 1.54060 Å). Fourier transform infrared (FT−IR) spectrum was obtained by a Shimadzu-8400S spectrometer (Shimadzu company, Japan) in the range of 400–4000 cm^{-1} using KBr pellets. Scanning electron micros copy (SEM) images and energy-dispersive X-ray were taken on a VEGA TESCAN S360 (TESCAN company, Czech Republic) with gold coating.

3. Results and Discussion

Figure 1 shows the FT−IR spectrum of the as-synthesized product. Two vibration bands at around 400–600 cm^{-1} can reveal the formation of spinel cobalt ferrite structure. The observed strong broaden peak at 574 cm^{-1} can be related to vibration frequency of Fe^{3+}–O. The small peak at around 400 cm^{-1} can be assigned to the vibration frequency of Co^{2+}–O in ferrite structure. As a known, cobalt ferrite is a reverse spinel ferrite, whereas the Fe^{3+} and Co^{2+} ions occupy at tetrahedral and octahedral sites, respectively. The peaks at 3426 and 1650 cm^{-1} can be related to the respective stretching and bending vibrational frequencies of adsorbed H_2O on the metal oxide surface. The peak at 2363 cm^{-1} can be due to the CO_2 adsorbed on the surface of prepared FT-IR sample.

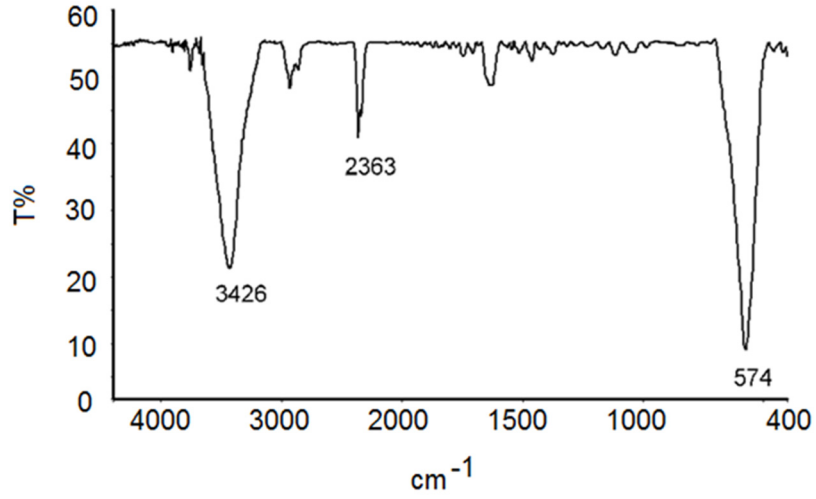

Figure 1. FT−IR spectrum of the prepared $CoFe_2O_4$.

XRD pattern as shown in Figure 2 reveals the successful formation of cubic phase of spinel cobalt ferrite structure, which confirmed FT-IR data. The diffraction peaks at 18.33°, 30.14°, 35.50°, 37.12°, 43.11°, 47.15°, 53.52°, 57.00°, 62.61°, 65.75°, 71.09°, 74.01°, 75.01°, and 79.02° are in a good agreement with the 111, 220, 311, 222, 400, 331, 422, 511, 440, 531, 620, 533, 622, and 444 crystal planes of cubic phase of $CoFe_2O_4$ with a space group of Fd3m (JCPDS card No. 22-1086) [11]. There are no other peaks related to the impurities or secondary phases in this pattern revealing the formation of pure phase of $CoFe_2O_4$ using the designed microwave assisted procedure.

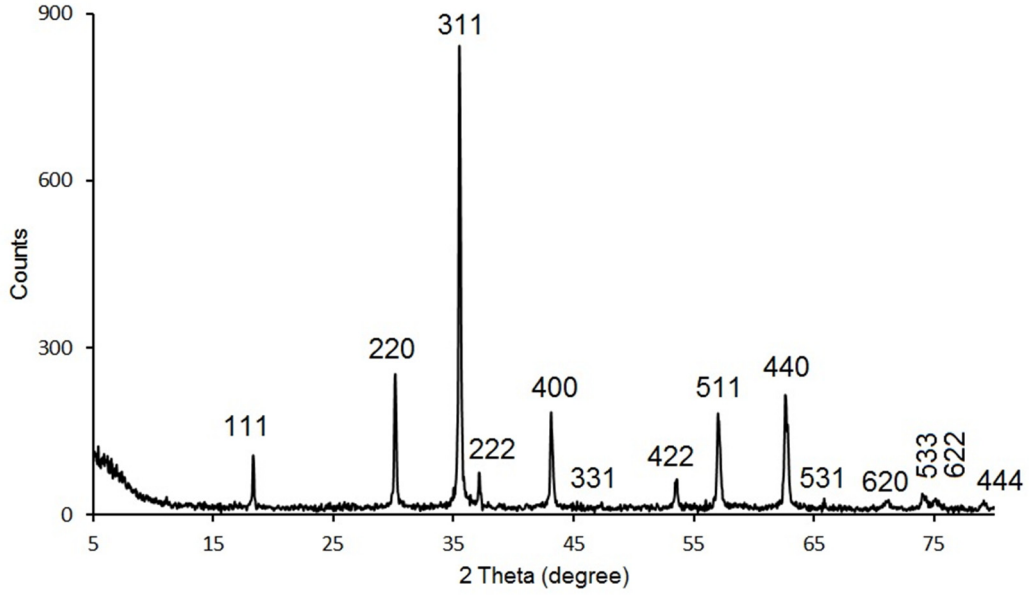

Figure 2. XRD pattern of $CoFe_2O_4$.

Elemental analysis by EDX (Figure 3) confirmed the presence of Co, Fe elements. These data show the concentration of Co and Fe elements in the resulting product in a close agreement with the expected stoichiometrical ratio of 2.

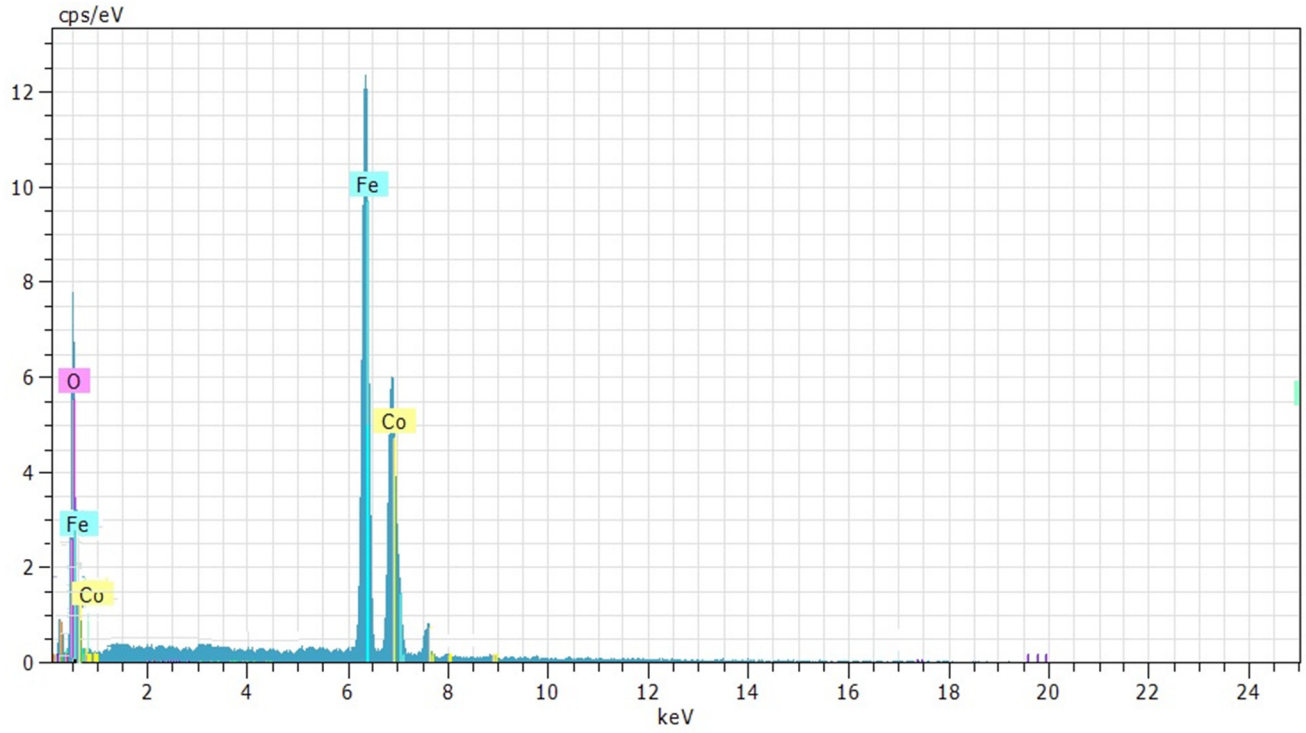

Figure 3. EDX analysis of the resulting cobalt ferrite.

Based on FESEM images, a morphology of porous sheets consisting of magnetic particles with an average size of 56 nm was identified for the synthesized cobalt ferrite (shown in Figure 4).

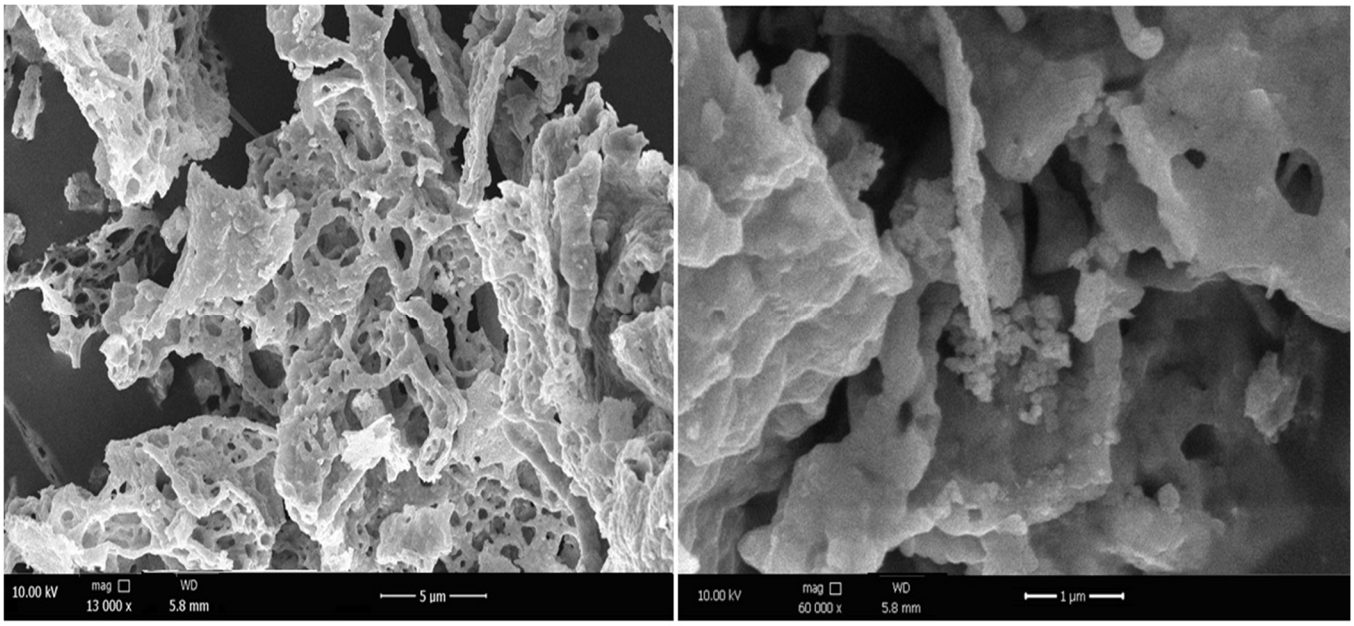

Figure 4. SEM images of the prepared product.

The electrochemical feature of the synthesized nanoparticles was evaluated using CV tests. The recorded voltammogram as shown in Figure 5 displayed a nearly rectangular curve with humps during cathodic/anodic scans at the potential scan rate of 10 mV^{-1}. It is supposed that the appeared peaks can be resulted from adsorption and desorption of alkali ions (Li^+, K^+, Na^+ or H_3O^+) at the interface of electrode and electrolyte on the metal

oxide surface. In addition, it can be originated from redox reaction due to the insertion and deinsertion of alkali ions to metal oxide.

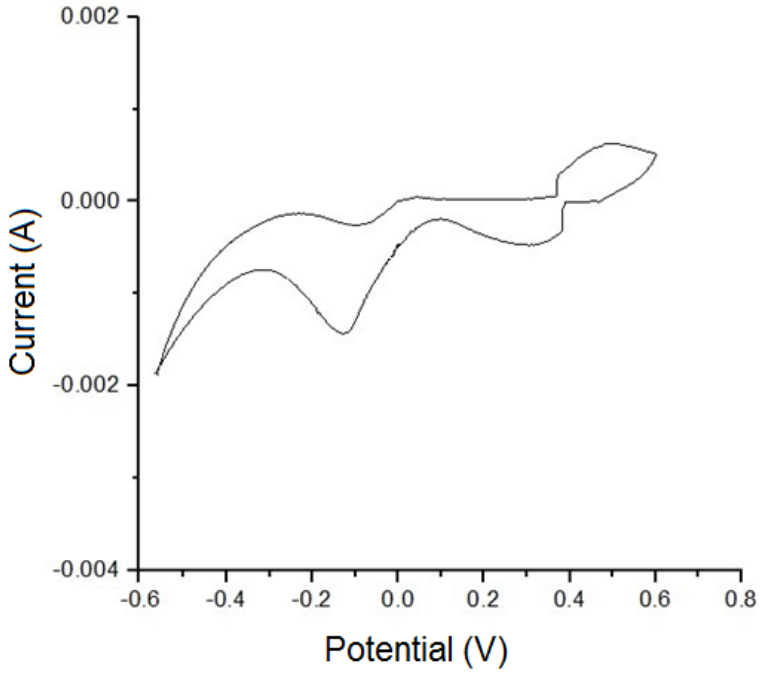

Figure 5. CV curves of the product at the potential scan rate of 10 mVs^{-1}.

Based on the CV curve, the specific capacitance (C_{sp}) of 254 Fg^{-1} was estimated at the potential scan rate of 10 mVs^{-1} for the resulting product, which is a comparable value with other electrode materials. Figure 6 represents a comparison of the specific capacitances at the different scan rates of 10–100 mVs^{-1}. Based on the results, it was observed that the specific capacitance reduces by the increase of scan rate value. It can be explained by the transfer failure of redox pairs due to the lack of enough time for the deep penetration of ions into pores and internal areas [12,13].

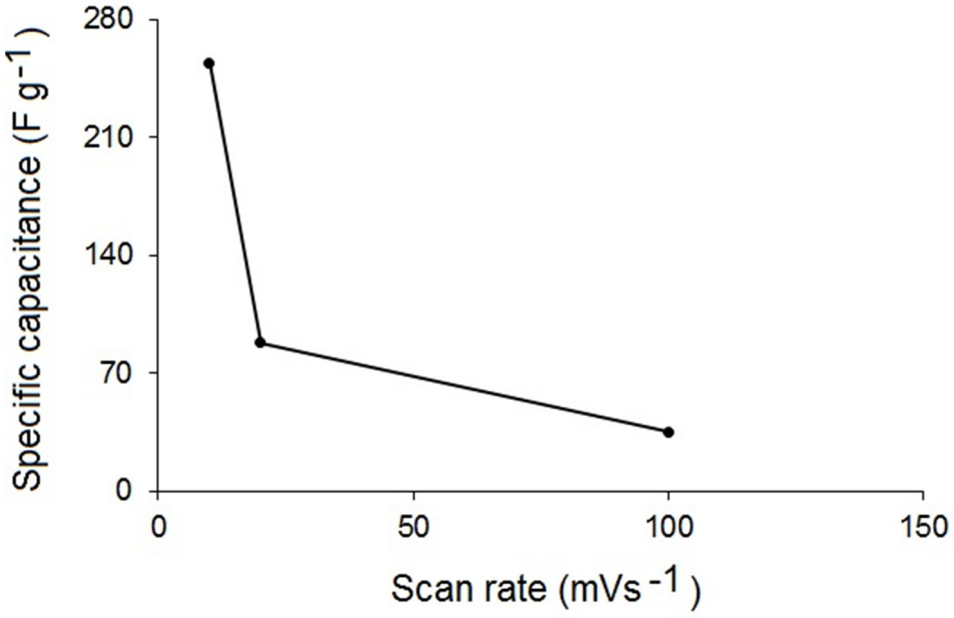

Figure 6. The relation of specific capacitance with change of potential scan rate.

4. Conclusions

The synthesis of a pure phase of the magnetic cobalt ferrite with a porous morphology of nanoparticles using a simple one-pot microwave assisted technique was presented. It is notable that the introduced procedure easily and rapidly caused to produce single phase of magnetic nanoparticles without using any additional post-reaction heat treatment. The examination of CV tests of the resulting product depicted a comparable high capacitance ability with the C_{sp} about 254 $F \cdot g^{-1}$. The obtained results revealed that this product can be suggested as an electrode active material for energy storage applications as well as other electrode materials

Institutional Review Board Statement: This study did not involve humans or animals.

Informed Consent Statement: Not applicable.

Data Availability Statement: The necessary data has been given and this study does not report more data.

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References

1. Kodama, R. Magnetic nanoparticles. *J. Magn. Magn. Mater.* **1999**, *200*, 359–372.
2. Prabhakaran, T.; Hemalatha, J. Combustion synthesis and characterization of cobalt ferrite nanoparticles. *Ceram. Int.* **2016**, *42*, 14113–14120.
3. Peredkov, S.; Neeb, M.; Eberhardt, W.; Meyer, J.D.; Tombers, M.; Kampschulte, H.; Niedner-Schatteburg, G. Spin and orbital magnetic moments of free nanoparticles. *Phys. Rev. Lett.* **2011**, *107*, 233401.
4. Salunkhe, A.; Khot, V.M.; Phadatare, M.R.; Pawar, S.H. Combustion synthesis of cobalt ferrite nanoparticles—Influence of fuel to oxidizer ratio. *J. Alloys Compd.* **2012**, *514*, 91–96.
5. Kefeni, K.K.; Msagati, T.A.; Mamba, B.B. Ferrite nanoparticles: Synthesis, characterisation and applications in electronic device. *Mater. Sci. Eng. B* **2017**, *215*, 37–55.
6. Maaz, K.; Mumtaza, A.; Hasanaina, S.K.; Ceylan, A. Synthesis and magnetic properties of cobalt ferrite ($CoFe_2O_4$) nanoparticles prepared by wet chemical route. *J. Magn. Magn. Mater.* **2007**, *308*, 289–295.
7. Deraz, N. Glycine-assisted fabrication of nanocrystalline cobalt ferrite system. *J. Anal. Appl. Pyrolysis* **2010**, *88*, 103–109.
8. Tadjarodi, A.; Shahrab, F. Synthesis of bismuth ferrite nanoparticles by Microwave irradiation. In Proceedings of the 20th International Electronic Conference on Synthetic Organic Chemistry, 1–31 November 2016.
9. Kennaz, H.; Harat, A.; Guellati, O.; Momodu, D.Y.; Barzegar, F.; Dangbegnon, J.K.; Manyala, N.; Guerioune, M. Synthesis and electrochemical investigation of spinel cobalt ferrite magnetic nanoparticles for supercapacitor application. *J. Solid State Electrochem.* **2018**, *22*, 835–847.
10. Imani, M.; Radmanesh, L.; Tadjarodi, A. Synthesis and study of the pseudocapacitive behavior of heterojunctional $NiTiO_3$-based chitosan nanocomposite. *J. Phys. Chem. Solids* **2020**, *139*, 109309.
11. Dom, R.; Subasri, R.; Radha, K.; Borse, P. Synthesis of solar active nanocrystalline ferrite, MFe_2O_4 (M: Ca, Zn, Mg) photocatalyst by microwave irradiation. *Solid State Commun.* **2011**, *151*, 470–473.
12. Bououdina, M.; Manoharan, C. Dependence of structure/morphology on electrical/magnetic properties of hydrothermally synthesised cobalt ferrite nanoparticles. *J. Magn. Magn. Mater.* **2020**, *493*, 165703.
13. Shanmugavani, A.; Kalpana, D.; Selvan, R.K. Electrochemical properties of $CoFe_2O_4$ nanoparticles as negative and $Co(OH)_2$ and $Co_2Fe(CN)_6$ as positive electrodes for supercapacitors. *Mater. Res. Bull.* **2015**, *71*, 133–141.