Dysprosium substituted nickel cobalt ferrite nanomaterials and their composites with reduced graphene oxide for photocatalysis

Muhammad Shahid a, *, Ibrahim A. Alsafari a,b, Akmal Jamil a, Fekri A. Ahmed Ali c, Sajjad Haider c, Philips Agboola d, and Imran Shakir e

aDepartment of Chemistry, College of Science, University of Hafr Al Batin, Hafr Al Batin, Saudi Arabia; bDepartment of Biology, College of Science, University of Hafr Al Batin, Hafr Al Batin, Saudi Arabia; cDepartment of Chemical Engineering, College of Engineering, King Saud University, Riyadh, Saudi Arabia; dCollege of Engineering Al-Muzahmia Branch, King Saud University, Riyadh, Saudi Arabia; eSustainable Energy Technologies Center, College of Engineering, King Saud University, Riyadh, Saudi Arabia

Abstract

High-quality nanostructure of dysprosium substituted nickel–cobalt ferrite and their composite with reduced graphene oxide (rGO) were synthesized. The structural and photocatalytic properties of prepared samples were studied. The XRD and FTIR analysis confirmed the FCC spinel structure. The calculated average crystallite size of nanoparticles was around 7.8 nm. The bandgap energy of synthesized composition was found to be 2.27 eV. The uniform dispersion of nanoparticles on the graphene nanosheets were confirmed by the FESEM analysis. The photocatalytic efficiency of nanoparticles and their composites with rGO was studied for degradation of methylene blue (MB) under visible light irradiation. It was found that nanocomposite exhibited high photocatalytic activity (72.7%) as compared to Dy 3+ substituted Ni-Co ferrite nanoparticles (52.4%). This enhanced photocatalytic activity can be attributed to the presence of graphene, which enhances the stability of nanocomposite and reduces the recombination of charge carriers.

1. Introduction

Not only humans but plants and animals also need healthy and pure water. Water was available free of cost in this world until industrialization started impacting nature negatively. With the increment of standards in human life, microbes, and colours in the form of irremovable dyes, also get increased in water bodies. In addition to it, other substances from industries, commercial areas, agricultural land are released ferently into the environment. On the one hand, science has helped in making human lives easier, it has also impacted it negatively on the other hand. To overcome it, science also provides solutions to the pollution problem of the environment [1]. In third world countries, water pollution including chemical and microbial agents are major issues that are needed to be solved on a priority basis. So, chemistry helped in understanding the nature of the water pollutants and to combat them with minimum wastage of natural resources.

Sunlight is one of the most common and easiest elements of nature to approach and utilize for being less poisonous [2]. This is the right thing where chemistry found its major applications and uses sunlight for the degradation of artificial pollutants in water using chemical compounds named as photocatalysts in a process known as photocatalysis. Similarly, magnetic materials play a vital role in the removal of microbial agents. The first-ever photocatalyst used for water cleaning was titanium dioxide (TiO2) which is a semiconductor. Electron–hole pairs are generated within titanium dioxide surface when irradiated with light thus showing photocatalytic activity [3]. The recombination of electron–hole pairs on its surface was the major disadvantage of using titania as a photocatalyst for water purification [4]. To increase its efficiency as a photocatalyst many methods of modifications were introduced. Doping with non-metals for reducing its bandgap and to increase the number of active sites & surface area was carried out [5]. Moreover, titanium dioxide was prepared on the nonmetric scale which also increased its efficiency. The conversion of bulk titanium dioxide into nanobelts makes it more efficient for the removal of dyes during water cleaning. Titanium dioxide was also coated converted into magnetic material for easy recovery and to facilitate its repetitive usage as a photocatalyst [6]. Titanium dioxide was also coupled with ferrites. Ferrites have great applications in biomedical and
wastewater cleaning because it's easy to recover them from water by just collecting with magnets. For example, Zinc ferrites with certain modifications like coupling it with graphene nanosheets helped in remarkable electron–hole pair generation. Their visible and UV visible light-triggered photocatalytic activity is being enhanced on synthesizing composites with other metals. Magnetic nanoparticles are studied widely because of their significant properties. Cobalt ferrites CoFe₂O₄ have higher efficiency for being chemically stable and when composites of cobalt ferrites and graphene are prepared, they showed higher photocatalytic activity [7]. Treatment of wastewater using photocatalysts comprised of cobalt ferrite & graphene nanohybrid has increased the photocatalytic activity of it [8]. However, the major challenge lies in the separation or recovery of the photocatalysts from the water that's why ferrites are more significant as photocatalysts because they can be recovered from water easily through magnetic separations. Harmful compounds which used as plasticizers can be effectively removed by using cobalt ferrites. Li et al. prepared the cobalt ferrites and graphene nanocomposite by the facile hydrothermal method of synthesis with greater magnetic separation [9]. He also discussed that it has a higher adsorption affinity towards methyl orange thus providing more surface area for catalytic activity thus making it a beneficial compound for purification of water. Cobalt ferrites are already considered as very efficient catalysts under ultraviolet irradiation however are made more environmentally friendly when incorporated with graphene to make nanocomposite [7].

Nickel ferrites are also considered good catalysts for carrying out photocatalytic reactions in wastewater [10]. However, when coupled with the other semiconductors like titanium dioxide and substituted with other metal cations, their efficiency is improved [11]. Nickel ferrites were also used for the modification of titanium dioxide to reduce its bandgap and make it active in the visible region [12]. Due to the inverse spinal nature of NiFe₂O₄, it is always noticed that Ni²⁺ ions occupy B sites and Fe³⁺ ions with near about the same amount distributed in A and B sites. Substitution with other metal cations to NiFe₂O₄ resulted in tunable physical and chemical properties. Fu and coworkers prepared nickel ferrite composites with graphene and discussed it improves adsorption and photocatalytic activity [11]. The literature study revealed that many graphene-ferrites composites have been prepared to enhance the performance of ferrite semiconductors as photocatalyst [13,14]. The nickel ferrite and titanium dioxide photocatalysts without the loss of the magnetic property of ferrite was also prepared [15].

The rare earth elements are focusing more and more, due to their 4f electronic structure. The structural and photocatalytic properties of semiconductor-based material can be modified to a greater extent, when rare-earth ions are substituted into it. Among the lanthanides, the dysprosium substituted nanomaterials proved themselves as an efficient optical material due to its excellent light response performance [16]. The dysprosium ions can provide more excited states due to their f to f orbitals transition. So, such type of substitution can control the electrical and optical properties of a material.

In this paper, we reported magnetically separable Dy³⁺ substituted Ni-Co ferrite-graphene nanocomposites by a simple co-precipitation method. Photocatalytic of the synthesized nanocomposite was assessed by degradation of methylene blue (MB) used as a source of organic contaminant.

2. Experimental procedure

2.1. Synthesis of Dy³⁺ substituted Ni-Co ferrites nanoparticles

The aqueous solutions of all metal salts with molar ratios Ni²⁺ (0.1 M), Co²⁺ (0.1 M), Fe³⁺ (0.2 M) and Dy³⁺ (0.2 M) were prepared in separate beakers. All the prepared solutions of each metal were mixed in a beaker and stirred for half an hour to homogenize and then the pH of the solution was increased up to 9–10 using NaOH solution followed by stirring keeping temperature less than 50°C for 5 h. The sample solutions were kept overnight for the settling of prepared precipitates. After this, the synthesized precipitates were washed with deionized water to reduce the pH up to the neutral level. The precipitates were then dried in the oven at 100°C till the complete evaporation of the solvent. After drying of samples, grinding and annealing was done at 700°C in the furnace. This resulted in refine powders of nanoparticles [17].

2.2. Fabrication of rGO

Graphene oxide was prepared by modified Hummer’s method [18]. In a typical synthesis, 1 g of graphite powder was taken in a conical flask and 25 ml of concentrated H₂SO₄ was added slowly in it. After the addition of H₂SO₄, a black solution was formed. This solution was then placed on an ice bath and the mixture was added from the tip of a plastic pipette slowly in reaction mixture at the temperature of 0–5°C for about 30 min. The obtained mixture was then subjected to heating at 35°C for 1 h. Afterward, 50 ml of deionized water was added to the mixture and stirred for 30 min. About, 10 ml of 30% H₂O₂ was added slowly into the mixture followed by the addition of 120 ml of deionized water, a yellow suspension was formed. The resulting yellow colour suspension was then filtered and washed repeatedly with deionized water 10% HCl and ethanol.

For preparation of rGO, 20 ml of GO was dispersed into 400 ml of deionized water by ultrasonic irradiation
for 1 h. Then, 0.2 ml of hydrazine and 1.5 ml NH3 were added into the GO mixture. Afterward, the reaction mixture was subjected to stirring at 100°C. The resultant rGO was obtained after 2 h which was then dried and ground to get powderized rGO [19].

2.3. Synthesis of Dy3+ substituted Ni-Co ferrite/rGO nanocomposite

Dysprosium substituted nickel–cobalt ferrite/rGO nanocomposite were fabricated with the use of ultrasonication method. In a typical synthesis, 20 mg of Dy3+ substituted Ni-Co ferrite nanoparticles were mixed with a 100 ml solution containing 80 mg of rGO. The resultant solution was ultrasonicated for 1 h to get the homogeneous solution. The resultant solution was evaporated on a hot plate at 120°C. The obtained black colour powder was further dried in an oven at 100°C overnight. The obtained dried black colour nanocomposite was ground for further characterization [20].

3. Results and discussion

3.1. XRD analysis

The crystal structure of prepared nanoparticles was analysed by X-ray diffraction (XRD). The XRD diffraction pattern of synthesized nanoparticles is shown in Figure 2(a). The diffraction peaks at 2Theta values of 30.3°, 35.6°, 37.2°, 47.0°, 54.0°, 57.5°, 62.6° can be indexed to the (220), (311), (222), (331), (422), (511) and (440) crystal planes of nanoparticles [21]. The diffraction planes were matched well with standard data (ICDD #00-010-0325). The matching of data confirmed the synthesis of dysprosium substituted nickel–cobalt ferrites [22]. The value of lattice constant was also calculated using cell software and found to be 8.67 Å [23]. Lattice constant depends upon ionic radii of elements. Therefore, this higher value of lattice constant was attributed to the substitution of lower ionic radii Fe3+ with higher ionic radii Dy3+ [24]. The unit cell volume of the synthesized product was found to be 651.7 Å³.

The crystallite size of Dy3+ substituted Ni-Co ferrite nanoparticles was calculated by using the famous equation proposed by Deby-Scherer [25]. According to this equation, the average crystallite size of the synthesized product was 7.8 nm. Moreover, the Williamson-Hall plot method was also applied. According to this method, the crystallite size of nanoparticles was 5.2 nm. The average crystallite size calculated by both of these methods was nearly similar. However, the Scherer method has given larger crystallite size, which was might be due to another factor known as microstrain. The microstrain of synthesized product was also calculated and found to be -0.00547. The negative value of microstrain predicts that the unit cell is in the state of compression, which was due to the substitution of higher ionic radii Dy3+ with lower ionic radii Fe3+.

The other parameters such as, X-ray density (4.94) bulk density (2.52), and porosity (0.489) of Ni0.5Co0.5Dy0.03Fe1.97O4 were also calculated with the help of famous equations already reported in our previous publications [26].

The XRD pattern of GO and rGO are presented in Figure 2. It is evident from XRD signals that these samples have a crystalline structure. The GO shows a diffraction peak at 2θ = 25.64° in the XRD pattern. At this point, the d-spacing was 0.34 nm along the 002 orientation. In rGO, the value of 2θ is shifted at 25.07° from 12° which shows the reduction of GO through modified Hummer’s method. After reduction, the interplanar distance changed from 0.34 to 0.82 nm showing oxidation and exfoliation of GO. It confirms the formation of rGO from GO [27].

3.2. Fourier transforms infrared spectroscopic analysis

The FTIR spectra of ferrites always consist of two types of characteristics absorption peaks. These characteristic peaks have corresponded to stretching vibrations of metal–oxygen bonds at tetrahedral and octahedral sites of their crystal lattice denoted by ν1 and ν2 respectively. The FTIR spectra of Ni0.5Co0.5Dy0.03Fe1.97O4 nanoparticles were also recorded and shown in Figure 4. The peak observed around 530 cm⁻¹ has corresponded to metal–oxygen stretching vibrations at tetrahedral sites. On the other hand, the absorption band observed around 480 cm⁻¹ corresponded to metal–oxygen stretching vibrations at octahedral sites [28].

3.3. Optical analysis

UV-Visible spectroscopic analysis was also performed to study the optical nature of nanoparticles. The UV-Visible spectra of Ni0.5Co0.5Dy0.03Fe1.97O4 nanoparticles are shown in Figure 5(a). From this figure, it can be noticed that the synthesized product shows a sharp absorption band within 340–600 nm range, this might be observed due to both absorption and dispersion phenomenon. This broad absorption band was confirming the formation of metal oxide nanoparticles and may make it able to respond even in the visible region of light [29,30]. The optical bandgap energy of the synthesized product was also calculated by using the Tauc plot method as given in Equation (1).

$$\frac{1}{(a\nu)^{\frac{1}{2}}} = A(h\nu - E_g)$$  \hspace{1cm} (1)

where α = molar absorptivity, h = Plank’s constant, ν = frequency of light, A = absorbance, n is constant and equal to 1/2 for direct bandgap and Eg = Bandgap energy. Bandgap energy can be calculated by extrapolating the straight region of the graph plotted between
Figure 1. Schematic illustration of the preparation of $\text{Ni}_{0.5}\text{Co}_{0.5}\text{Dy}_{0.03}\text{Fe}_{1.97}\text{O}_4$ nanoparticles.

Figure 2. (a) XRD patterns of $\text{Ni}_{0.5}\text{Co}_{0.5}\text{Dy}_{0.03}\text{Fe}_{1.97}\text{O}_4$ nanoparticles and (b) W-H plot of $\text{Ni}_{0.5}\text{Co}_{0.5}\text{Dy}_{0.03}\text{Fe}_{1.97}\text{O}_4$ nanoparticles.

Figure 3. (a) UV-Visible spectra (b) XRD patterns of graphite powder and rGO.

"$h\nu$" (on the x-axis) and $(\alpha h\nu)^{1/2}$ (on the y-axis). From Figure 5(b), it can be noted that the direct bandgap of the synthesized product was 2.27 eV, which makes it a suitable candidate to respond even in the visible region of light.

The electronic absorption spectra of GO and rGO are shown in Figure 3(a). The absorption peaks for GO and rGO are observed at 231 and 253 nm respectively. The absorption peak of GO at 231 nm is attributed to the $\pi-\pi^*$ excitation of the $\text{C} = \text{C}$ bond in the aromatic
ring. Whereas rGO showed a redshift from 231 to 253 nm. The reason for this shift is n-π* excitation in the C=O bond embedded on layers of graphene after its reduction [31]. An extra peak at 303 nm in the GO spectrum shows unreacted graphite remained in the sample.

3.4. SEM analysis

The morphological studies of synthesized nanoparticles and their composites were investigated by Field emission scanning electron microscopy. Figure 6 shows the clear SEM images of Dy^{3+} substituted Ni-Co nanoparticles and their rGO based nanocomposite. SEM image of as-synthesized Dy^{3+} substituted Ni-Co nanoparticles revealed that these were in nano-flake like morphology. Furthermore, SEM images of Dy^{3+} substituted Ni-Co/rGO nanocomposite showed that Dy^{3+} substituted Ni-Co nanoparticles were fully embedded in graphene sheets that fully satisfied the formation of nanocomposite.

3.5. Application study of Dy^{3+} substituted Ni-Co ferrite and their composites

3.5.1. Photocatalysis

In this work, the degradation of methylene blue is reported by Dy^{3+} substituted Ni-Co ferrite and its rGO based nanocomposite. During this experiment, 5 mg of catalyst was taken in 50 cm³ of 5 ppm methylene blue dye. It was observed that with time characteristic absorption peak’s intensity of methylene blue decreased. The dye colour goes to become lighter and lighter within the 120 min of the time interval and in the end; it has almost become colourless as shown in Figure 7. The most noticeable observation was that little dye degradation takes place when Dy^{3+} substituted Ni-Co nanoparticles were used as compared to its rGO based nanocomposite, which is shown in Figure 8(a). This was observed due to the more conductive nature of graphene. It can be noticed from this figure that, there was zero or saying no degradation when methylene blue solution was kept in dark and light condition. Figure 8(b) shows that, Dy^{3+} substituted Ni-Co nanoparticles exhibited 52.4% while their rGO based nanocomposite showed 72.6% degradation within 120 min of visible light irradiation. Moreover, the kinetic data revealed that rGO based nanocomposite degraded methylene blue dye with a faster rate (0.0182 min⁻¹) as compared to bare nanoparticles (0.0091 min⁻¹).

3.5.2. Mechanism of photocatalysis

The detailed mechanism for the photocatalytic degradation of methylene blue dye by Dy^{3+} substituted Ni-Co – rGO nanocomposite material can be illustrated as follows.

Step 1: When light falls on the surface of nanoparticles, electrons hole pairs were generated. Electrons of conduction band get excited while in the valance band, holes were produced [32]:

![Figure 4. FTIR spectra of Ni_{0.5}Co_{0.5}Dy_{0.03}Fe_{1.97}O_{4} nanoparticles.](image-url)
Figure 6. (a) Typical SEM image for nanoparticles of Dy\(^{3+}\) substituted Ni-Co ferrites (b) SEM image for Ni\(_{0.5}\)Co\(_{0.5}\)Dy\(_{0.03}\)Fe\(_{1.97}\)O\(_4\)/rGO nanocomposite.

Figure 7. Absorption spectra of MB dye taken at different photocatalytic degradation time using (a) Ni\(_{0.5}\)Co\(_{0.5}\)Dy\(_{0.03}\)Fe\(_{1.97}\)O\(_4\) nanoparticles and (b) Ni\(_{0.5}\)Co\(_{0.5}\)Dy\(_{0.03}\)Fe\(_{1.97}\)O\(_4\)/rGO based composite.

Figure 8. (a) Kinetic plot of methylene blue degradation, (b) bar chart showing comparative percent degradation of MB with the photocatalysts under visible light illumination.
Figure 9. (a) -log (A/A₀) Vs Time graph, (b) rate constant for degradation of methylene blue dye.

Ni₀.₅Co₀.₅Dy₀.₀₃Fe₁.₉₇O₄ + hv → Ni₀.₂Co₀.₈Fe₂O₄ [hole (v.b) + electron (c.b)]

**Step 2:** Electron from the conduction band of ferrite gets combined with rGO. Due to its greater charge separation capability, the electron–hole pair recombination becomes difficult. This effect enhances the photocatalytic process.

Ni₀.₅Co₀.₅Dy₀.₀₃Fe₁.₉₇O₄ [electron (c.b)] + rGO → Ni₀.₂Co₀.₈Fe₂O₄ + rGO (electron)

**Step 3:** Electron of conduction band from rGO gets combined with oxygen and as a result of it, O₂⁻ is formed. Degradation of dye occurs due to these electrons.

rGO (electron) + O₂ → rGO + O₂⁻

**Step 4:** The hole of the valance band then combines with hydroxyl ion and get transformed into hydroxyl radical.

Ni₀.₅Co₀.₅Dy₀.₀₃Fe₁.₉₇O₄ [hole(v.b)] + OH⁻ → Ni₀.₂Co₀.₈Fe₂O₄ + OH⁻

**Step 5:** Finally, the active species such as O₂⁻ and OH radical drive the photodegradation of methylene blue.

Ni₀.₅Co₀.₅Dy₀.₀₃Fe₁.₉₇O₄ [hole(v.b)] + OH⁻ + O₂⁻ + M.B → CO₂ + H₂O + NH₄⁺ + NO₃⁻ + Cl⁻

Ni₀.₅Co₀.₅Dy₀.₀₃Fe₁.₉₇O₄/rGO nanocomposite play the significant role of an electron carrier. This photo process provides an attractive route to treat dye pollutants using a visible light source. The pictorial representation for the mechanism of photocatalysis is represented in Figure 10.

4. Conclusion

Dy³⁺ substituted Ni-Co ferrite nanoparticles and its rGO based nanocomposite was prepared by coprecipitation technique and characterized by XRD, FTIR, FESEM, and UV-Visible. From the XRD pattern, it is apparent that the compositions develop with the FCC spinel structure with 7.8 nm crystallite size. From FESEM analysis, it was noticed that Dy³⁺ substituted Ni-Co ferrite nanoparticles were uniformly distributed in rGO sheets. The bandgap energy of the synthesized product was around 2.27 eV which is evident for

Figure 10. Schematic illustration of the degradation of methylene blue by Ni₀.₅Co₀.₅Dy₀.₀₃Fe₁.₉₇O₄/rGO nanocomposite.
its visible light response. Dy$^{3+}$ substituted Ni-Co ferrite nanoparticles degraded 52.5% while rGO based its nanohybrid degraded 72.6% of methylene blue dye. Ni$_{0.5}$Co$_{0.5}$Dy$_{0.03}$Fe$_{1.97}$O$_{4}$/rGO nanocomposite proved as an efficient candidate for photocatalytic degradation of methylene blue dye rationalized with faster rate constant (0.0182 min$^{-1}$). Generally, it can be concluded that Ni$_{0.5}$Co$_{0.5}$Dy$_{0.03}$Fe$_{1.97}$O$_{4}$/rGO nanocomposite acts as an efficient photocatalyst for the removal of organic pollutants from contaminated water.

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No potential conflict of interest was reported by the author(s).

**ORCID**

Muhammad Shahid [http://orcid.org/0000-0003-3654-2277](http://orcid.org/0000-0003-3654-2277)  
Ibrahim A. Alsafari [http://orcid.org/0000-0003-2649-7265](http://orcid.org/0000-0003-2649-7265)  
Akmal Jamil [http://orcid.org/0000-0002-5589-4086](http://orcid.org/0000-0002-5589-4086)  
Fekri A. Ahmed Ali [http://orcid.org/0000-0003-1892-2057](http://orcid.org/0000-0003-1892-2057)  
Philips Agboola [http://orcid.org/0000-0001-6396-6986](http://orcid.org/0000-0001-6396-6986)  
Imran Shakir [http://orcid.org/0000-0003-3385-4363](http://orcid.org/0000-0003-3385-4363)

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