A facile green synthesis of nickel ferrite nanoparticles using *Tamarindus Indica* seeds for magnetic and photocatalytic studies

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**Abstract**

Magnetic NiFe\(_2\)O\(_4\) nanoparticle (NFNPs) were synthesized by the microwave-assisted method using *Tamarindus indica* seeds as a green reducing agent for better readability. The identification of NFNPs was carried out by X-ray powder diffraction (XRD), Fourier transform infrared spectroscopy (FTIR), scanning electron microscope (SEM) with energy dispersive X-ray analysis (EDX), transmission electron microscopy (TEM), UV–Visible spectroscopy and luminescence techniques. Spinel structure of NFNPs with a single phase has been confirmed by XRD with an average crystallite size of 21 nm. FTIR confirms the presence of tetrahedral and octahedral sites of prepared NFNPs. SEM with EDAX and TEM gives the information of agglomeration of magnetic nanoparticles with elemental composition present in the NFNPs and narrow size distribution of nanoparticles with a particle size of 28 nm. The prepared NFNPs are visible active with band gap of 1.92 eV. NFNPs show effective photocatalytic activity under visible light irradiation towards the degradation of anionic Alizarin red S dye and cationic Methylene Blue dye. NFNPs show good magnetic property in VSM studies. The prepared NFNPs can be useful for wastewater treatment.

**Keywords** NiFe\(_2\)O\(_4\) · *Tamarindus Indica* seeds · Microwave method · VSM analysis · Anionic Alizarin red S dye · Methylene blue dye

**Introduction**

In industrial wastewaters, degradation of dyes has generated a great attention due to low decoloration, slow biodegradation, high toxicity and high volume of production. Every year dyes are produced in large scale and are naturally resistant towards sunlight, water, detergents and other basic chemicals which make them basically difficult to treat [1, 2]. These dyes are applied to various types of industries including paper, textile, leather, cosmetic, nutrition and pharmaceutical industries. Annually over 700,000 tons of dyes will be produced off which nearly 100,000 different type of dyes are commercially available, during dying process 15% of which are discharged into the water sources. Azo dyes constitute more than 50% of these globally produced dyes. These complex aromatic dyes cause severe impact on the human health and aquatic life. These concerns lead to develop new technologies for waste water treatment [2, 3]. Recently, due to their optical, magnetic and photocatalytic applications, nickel ferrite (NiFe\(_2\)O\(_4\)) nanoparticles have attracted very much interest in scientific society. NiFe\(_2\)O\(_4\)
is a soft magnetic material with saturation magnetization ($M_s = 40.8$ emu/g), coercive field ($H_c = 98$ Oe), remanence magnetization and low electrical resistivity [4]. Generally, in nanoparticles crystallinity, morphology and band gap play an important role in magnetic studies and the photocatalytic applications. Hence, it is a suitable material for magnetic, gas and humidity sensing, catalytic and magneto-optical applications [5]. For the synthesis of NiFe$_2$O$_4$ nanoparticles, several methods have been used which are co-precipitation, sol–gel, combustion, microwave, hydrothermal, and sol–gel auto-combustion methods [6, 7]. Among these methods, microwave-assisted method has extended much attention because it is environmentally benign, less reaction time, simple process, and relatively lowers the processing time and temperature which permits to prepare the nanocrystalline ferrite nanoparticles [8]. Table 1 gives the comparative study of green/biosynthesis of NiFe$_2$O$_4$ NPs.

From the literature, it can be observed that NiFe$_2$O$_4$ played a vital role in the field of magnetic and photocatalytic applications. Few authors reported the magnetic and photocatalytic studies of NiFe$_2$O$_4$ NPs. Hirthna et al. have described in the work that NiFe$_2$O$_4$ NPs synthesized by chemical co-precipitation method for photocatalytic degradation of methyl orange dye (~72.66% within 5 h) [13]. Andris suta et al. have prepared the anatase-NiFe$_2$O$_4$ heterostructures (co-precipitation method) for photocatalytic activity (methyl orange: 96% degradation after 3 h) [14]. D. Li et al. have synthesized spinel NiFe$_2$O$_4$ ferrite by sol–gel method for magnetic studies with saturation magnetization ($M_s = 66.6$ emu/g), remanence magnetization ($M_r = 15.9$ emu/g) and coercivity ($H_c = 161.6$) [15].

In the present study, using Tamarindus indica seed powder as a reducing agent, green synthesis of NiFe$_2$O$_4$ NPs has been carried out by microwave-assisted method. Tamarind has sugar, tartaric acid, and vitamins and it is used as the main ingredient in traditional food and medicinal methods. Tartaric acid present in seed extract acts as a reducing agent for preparation of nanoparticles. Silver nanoparticles were prepared using Tamarindus indica fruit extract [16].

The as prepared microwave-assisted NiFe$_2$O$_4$ NPs were characterized by different methods such as XRD, SEM with EDAX, FTIR, TEM, UV–Visible and luminescence spectroscopy for photocatalytic (anionic Alizarin red S dye and cationic Methylene blue dye) and magnetic studies.

### Experimental

#### Materials

The Tamarindus indica seeds were obtained from a local market of Shivamogga, Karnataka, India. The precursors used were Fe(NO$_3$)$_3$·9H$_2$O and Ni(NO$_3$)$_2$·6H$_2$O were obtained from HIMEDIA, India which are used without additional purification and of analytical grade. Throughout the experiment, deionized water was used.

#### Preparation of Tamarind seeds powder

The collection of dried seeds of Tamarindus indica were washed in tap water to remove the dust particles and then followed by the doubled distilled water for two–three times. After washing, seeds were shaded and dried at room temperature for 10–15 days. After air-dried, seeds were crushed using electric mixture. After crushing, the seeds powder was stored in refrigerator for further analysis.

#### Synthesis of NiFe$_2$O$_4$ NPs

NiFe$_2$O$_4$ NPs were prepared by taking Fe(NO$_3$)$_3$·0.9H$_2$O and Ni(NO$_3$)$_2$·6H$_2$O with the ratio of 2:1 (Fe:Ni) in extracted Tamarindus indica seed powders with metal nitrates to extract 1:1 ratio in 50 ml deionized water. Stoichiometric molar ratio of 2:1 (Fe:Ni) of metal nitrates was taken. Metal

| Method of synthesis          | Green materials       | Calcination temperature (°C) | Morphology               | Ref      |
|-----------------------------|-----------------------|------------------------------|--------------------------|----------|
| Hydrothermal                | Urtica extract        | -                            | Rod like platelet        | [9]      |
| Sol–gel combustion          | Honey-mediated        | 1100                         | Octahedron               |          |
| Modified sol–gel            | Aloe vera extract     | 900                          | Agglomerated             | [10]     |
| Simple sol–gel auto-combustion | Onion extract      | 750                          | Agglomerated             | [11]     |
| Reduction                   | Desmodium gangeticum (Linn) DC roots | - | - | [12]     |
| Hydrothermal                | Aloe vera extract     | 600                          | Spherical                | Present work |

The bold value indicates the present work compared with previously published works

Italic is given to the reducing agent name which is the Scientific Name of the Seed/Plant

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nitrate to seed extract ratio is 1:1 weight ratio. pH of the reaction mixture is nearly 8 before microwave treatment. Control test without the seed powder was carried out and it was difficult to obtain the product. Tartaric acid present in seed extract acts as a reducing agent for the preparation of NiFe$_2$O$_4$ nanoparticles. Over other conventional methods, microwave-assisted method found to be an attractive choice for nanoparticle synthesis due to effective heating energy. Furthermore, the method shows acceleration in reaction rate, short reaction time, improvement in yield, small and narrow particle size distribution. To get the homogeneous solution the reaction mixture was stirred for about 1 h and a domestic microwave oven (frequency of 2.45 GHz at 900 W output power) was used. The solution of reaction mixture was irradiated for 15 min. After the synthesis, particles were washed with ethanol followed by distilled water, the product was dried at 100°C for 60 min in an oven and calcined at 600°C for 5 h.

The particles were formed after 15 min microwave and the particles formed without 600°C treatment. Further, calcination of 600°C will be maintained to increase the crystallinity of the particles. Microwave-assisted synthesis of nanoparticles is associated with advantages such as uniform heating, fast reaction under controlled environment, product of high purity, and high yield [17].

Characterization techniques:

Rigaku Smart Lab XRD (Tokyo, Japan) with Cu-Kα radiation source ($\lambda = 1.5406$ Å) was used for X-ray diffraction (XRD) measurements. To study the two major bond frequencies of nickel ferrite NPs like tetrahedral and octahedral sites was analyzed in the range 4000–350 cm$^{-1}$ by using Fourier transform infrared spectrum (FTIR) (Bruker alpha-P infrared spectrophotometer). Zeiss electron microscope was used to observe the surface morphologies of the samples like scanning electron microscopy (SEM) and elemental analysis. For shape and morphological observation Field Emission Gun-Transmission Electron Microscope 300 kV was used. Shimadzu UV–Visible spectrophotometry was used for optical studies. During degradation, Agilent technology Cary-60 spectrophotometer was used for the absorbance of the organic dye. Agilent technology Cary Eclipse Spectrofluorometer was used for luminescence studies.

Photocatalytic degradation procedure

In a photoreactor, a medium pressure tungsten vapor lamp (300 W, capable of emitting complete spectrum from 400–800 nm) is placed at the central axis of the system, and it is refrigerated by means of a cooling jacket and also fans have been fixed at four corners to cool the reaction. Cold water circulation was maintained continuously through cooling jacket to control the heat generated from the lamp. Around the lamp a set of quartz tubes of 100 mL capacity (37 cm length, 2.3 cm i.d., and 2.7 cm o.d.) will be filled with organic pollutant were placed. The distance between quartz tube and tungsten lamp was 6 cm.

Photodegradation of anionic Alizarin red S (ARS) dye and cationic Methylene blue (MB) dye under visible light irradiation in the presence of microwave-assisted NiFe$_2$O$_4$ NPs was evaluated. In a photoreactor, 50 mg of NiFe2O4 photocatalyst was added to a quartz tube containing 100 mL of 20 ppm anionic ARS dye and cationic MB dye aqueous solution. In dark condition to attain the adsorption/desorption equilibrium the reaction mixture was stirred in dark for 30 min. At room temperature, the solution (reaction mixture) was irradiated under visible light. The degradation residual was collected every 30 min and was tested by UV–Vis absorption spectra at the absorbance of 525 nm (ARS) and 663 nm (MB). By the following relation the photocatalytic degradation percentage of dyes was measured,

$$\text{percentage of degradation} = \frac{C_o - C_t}{C_o} \times 100$$ (1)

where $C_o$ is the initial absorbance of aliquot and $C_t$ is the absorbance of anionic ARS dye aliquot after time t in min, respectively.
Results and discussion

XRD analysis

XRD pattern of microwave-assisted NiFe$_2$O$_4$ nanoparticles is shown in Fig. 1, and the spinel structure with single phase material was confirmed with JCPDS No. 86–2267. The diffraction peaks (2θ) at 18.44, 30.32, 35.63, 37.44, 43.44, 53.95, 57.47, 63.12 and 74.77 correspond to the planes (1 1 1), (2 2 0), (3 1 1), (2 2 2), (4 0 0), (4 2 2), (5 1 1), (4 4 0) and (5 3 3), respectively, and it confirms the formation of the spinel structure with single phase material of NiFe$_2$O$_4$ NPs. All the major lines were selected and average was taken for application of Scherrer formula (Eq. 2). Average crystalline size of NiFe$_2$O$_4$ nanoparticles was found to be 21 nm.

\[
D = \frac{0.9\lambda}{\beta\cos\theta}
\]

where \(\lambda\) is wavelength of radiation source (1.5406 Å) and \(\beta\) is the full width half maximum.

By the following formula it was calculated that dislocation density of NiFe$_2$O$_4$ NPs was found to be $2.2675 \times 10^{15}$ lines/m$^2$,

\[
\delta = 1/D^2
\]

Microstrain of green synthesized NiFe$_2$O$_4$ NPs was 4.96 $\times 10^{-2}$ and was found by following relation,

\[
\mu = \frac{\beta\cos\theta}{4}
\]

Morphological analysis

Figure 2a shows the SEM micrograph of microwave-assisted NiFe$_2$O$_4$ NPs where the agglomerated with uniform distribution of NPs and also narrow size distribution of NPs observed. During microwave combustion process the high energy generated leads to the agglomeration of NiFe$_2$O$_4$ NPs. TEM micrograph (Fig. 2b) showed the uniform formation NiFe$_2$O$_4$ nanoparticles. Figure 2d represents the SBL (Statistical Bin Limits) nanosizer histogram of magnetic nanoparticles that revealed the mean particle size is 28 nm in diameter. From these results, the NiFe$_2$O$_4$ nanoparticles indicate that they provide homogeneously dispersive and narrow size distribution. The chemical elemental compositions of prepared NiFe$_2$O$_4$ NPs was observed in EDX spectrum. The characteristic peaks of elements Ni, Fe and O are present in Fig. 2c and also it shows that there are no impurities present in the prepared NiFe$_2$O$_4$ NPs.

FTIR analysis

FT-IR spectrum of microwave-assisted NiFe$_2$O$_4$ NPs (Fig. 3) recorded in the range between 4000 and 350 cm$^{-1}$ in transmission mode. Spinel ferrites have two main frequencies of absorption bands in those two the absorption band at ~598 cm$^{-1}$ assigned between Ni–O in tetrahedral sites which occurs due to intrinsic stretching vibration of bonds. The weakest absorption peak observed at ~399 cm$^{-1}$ assigned to the stretching vibration of bonds between Fe–O in octahedral sites. The weak band at 2928 and 1398 cm$^{-1}$ corresponds to stretching vibration of C-H group. The band at 3384 and 1631 cm$^{-1}$ is attributed to the stretching vibration of H–O–H groups of water molecules adsorbed. The corresponding absorbed carbon dioxide on the surface of NiFe$_2$O$_4$ NPs was observed at 1105 cm$^{-1}$ [18]. Based on the overhead results, the ferrite structure expected can be concluded.

Optical studies

Figure 4 shows the optical absorption spectrum of NiFe$_2$O$_4$ NPs. The absorption edge of the NiFe$_2$O$_4$ NPs was observed at 460 nm. The fundamental absorption calculations were used for the optical energy band gap calculation and were calculated by the relation [19]

\[
(\alpha h\nu)^{1/n} = A(h\nu - E_g)
\]

where \(\alpha\) is energy-dependent absorption coefficient, A is a constant, \(E_g\) is the band gap of the material and the exponential n depends on the transition type. For direct allowed transition the parameter n with value 2 can be used and for indirect allowed transition \(1/2\) can be used. In the present study, for indirect allowed transition \(1/2\) has been considered for calculating band gap. Tauc plot between \((\alpha h\nu)^{1/2}\) and energy \((h\nu)\) are plotted and the linear straight line which meets the energy axis was extrapolated to obtain the energy bandgap (Fig. 4). The optical energy band gap of NiFe$_2$O$_4$ NPs was found to be 1.92 eV. The obtained result matched nearly with the literature and the materials is visible active [20].

The luminescence spectrum of the microwave-assisted NiFe$_2$O$_4$ NPs can be observed in Fig. 5 at an excitation wavelength of 230 nm. In the visible region it exhibits violet, blue, green and orange emission at 449, 489, 532 and 604 nm. The sharp peaks in the spectra are assigned to the transfer of charges at octahedral sites between Fe$^{3+}$ and surrounded O$^{2-}$ ions. The peak at 532 nm attributed to the recombination of photo-generated holes from nickel interstitials and oxygen vacancies which are originated from the near band gap emission with ionized charged species. The blue green emission of NiFe$_2$O$_4$ NPs can be confirmed by electron transition from deep donor level oxygen vacancies to the valence band [21, 22].
Magnetic studies

The magnetic properties NiFe₂O₄ NPs were characterized using a vibrating sample magnetometer (VSM) with an applied magnetic field − 15,000 of + 15,000 Oe at room temperature. The microwave-assisted NiFe₂O₄ NPs confirmed as ferromagnetic (soft magnetic) from Fig. 6 which shows the plot of magnetization versus applied field (M–H) loop. The remanent magnetization ($M_r$), saturation magnetization ($M_s$), and coercivity ($H_c$) of NiFe₂O₄ nanoparticles are 0.1347 emu/g, 0.3713 emu/g, 1380.7 Oe, respectively [23, 24]. These results confirmed that the prepared NPs will become a renewable photocatalyst because by using an external magnetic field the photocatalyst can be removed effectively from the aqueous solution of reaction mixture.
Photocatalytic activity

The photodegradation of anionic ARS dye ($\lambda = 525$ nm) and cationic MB dye ($\lambda = 663$ nm) in the presence of microwave-assisted NiFe$_2$O$_4$ NPs has been assessed under visible light (Fig. 7a). The absorbance of anionic ARS dye solution was decreased with increasing visible light irradiation time. The absorbance of anionic ARS dye cationic MB dye has drastically decreased and gives 83 and 82% of degradation in 120 min.

The photocatalytic degradation of microwave-assisted NiFe$_2$O$_4$ nanoparticles mainly depends on crystalline nature, charge separation and on the distinctive surface morphology (SEM and TEM). The degradation of anionic ARS dye and cationic MB dye follows the pseudo-first-order kinetic from the kinetic studies which was confirmed by the relation,

$$C_t = C_0 e^{-Kt}$$

Alternatively

$$\ln \left( \frac{C_0}{C_t} \right) = Kt$$

where $C_0$ is the initial concentration, $C_t$ is the concentration of aqueous solution of dye at respective time interval t and K is the rate constant (min$^{-1}$). The obtained first-order rate constant of NiFe$_2$O$_4$ photocatalyst for ARS and MB dyes were $6.02 \times 10^{-3}$ min$^{-1}$ and $5.55 \times 10^{-3}$ min$^{-1}$, respectively (Fig. 8). R$^2$ value obtained from the rate constant plot for ARS and MB dyes are 0.9906 and 0.9916, respectively.

Photocatalytic degradation mechanism

Following equations shows the degradation mechanism of anionic ARS dye cationic MB dye solution in the presence of NiFe$_2$O$_4$ photocatalyst,

$$NiFe_2O_4 + hv(Vis) \rightarrow NiFe_2O_4(e_{CB}^- + h_{VB}^+)$$

$$NiFe_2O_4(e_{CB}^-) + O_2 \rightarrow 'O_2^-$$

$$H_2O \rightarrow H^+ + HO^-$$

$$HO^- + h^+ \rightarrow HO'$$
In the photodegradation process of the anionic ARS dye cationic MB dye *OH and *O₂⁻ are the notable reactive species (Fig. 9). Photocatalytic degradation efficiency of anionic ARS dye of microwave-assisted NiFe₂O₄ NPs compared with some other metal oxide NPs given in Table 2.

**Conclusion**

Microwave-assisted green method was used to synthesis the NiFe₂O₄ NPs using Tamarindus indica seed powder as reducing agent. XRD confirmed the spinel structure with single phase of NiFe₂O₄ nanoparticles with an average crystallite size of 21 nm. The presence of tetrahedral (598 cm⁻¹) and octahedral (399 cm⁻¹) sites in ferrite NPs was observed.
in the prepared sample by FTIR analysis. The agglomeration of magnetic nanoparticles and the presence of elemental compositions were observed in SEM images and EDAX spectrum. TEM image showed the uniform formation of NiFe$_2$O$_4$ nanoparticles with a mean particle size of 28 nm in diameter. It was confirmed that the sample was visible active because the optical band gap was 1.92 eV and green emission around 532 nm was observed in luminescence spectrum. Microwave-assisted NiFe$_2$O$_4$ nanoparticles showed significant photocatalytic (anionic ARS dye and cationic MB dye) and magnetic studies. Biosynthesized NiFe$_2$O$_4$ NPs are
the appropriate material for optical studies, wastewater treatment and magnetic studies.

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Declarations

Conflict of interest The authors declare that there are no conflicts of interest.

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