Bimetallic nickel-ferrite nanorod particles: greener synthesis using rosemary and its biomedical efficiency

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ABSTRACT

Nickel-ferrite (NiFe2O4) nanorods particles (NRP) was biosynthesised for the first time by the Rosemary Extract. The NRP was fully characterised, including the type, nanostructure and physicochemical properties of using XRD, HRTEM, FeSEM, XPS, FTIR and VSM. TEM confirmed rod-shaped nano-sized particles with average sizes ranging from 10 nm to 28 nm. The EDAX Analysis showed the presence of iron, nickel, oxygen, and carbon. XRD analysis confirmed the synthesis of NiFe2O4 crystals. XPS curves showed photoelectron for iron, oxygen and nickel. EDS showed the atomic, weight percentages ratios of Ni(12%): Fe(24%) and: O(48) are close to the theoretical value (Ni: Fe: O = 1:2:4), of bimetallic magnetic NiFe2O4 NRP. NiFe2O4 NRP had cytotoxicity effect on MCF-7 cells survival which suggests that NiFe2O4 NRP can be used as a new class of anticancer agent in design novel cancer therapy research.

GRAPHICAL ABSTRACT

Abbreviations: Fig.: Figure; mL: Millilitre; nm: Nanometre; FeCl3.6H2O: Ferric chloride hexahydrate; NiCl2.6H2O: Nickel(II) chloride hexahydrate; Na2CO3: Sodium carbonate; XRD: X-ray diffraction; NiFe2O4: Nickel ferrite; HRTEM: High-resolution Transmission electron microscope; FESEM: Field emission scanning electron microscopy; VSM: vibrating-sample magnetometer; FTIR: Fourier-transform infra-red spectroscopy; XPS: X-ray photoelectron spectroscopy; DMEM: Dulbecco’s Modified Eagle Medium

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Introduction

The past decade has been a huge surge in the applications of the metallic based nanoparticles (NP) ranging from biomedical applications to other industries such as catalytic water treatment or electronic. Nowadays, extensive research into the production of metal nanostructures and study the application of them is widely developed [1–4]. Among the various types of nanostructures produced, NP of iron, cobalt and nickel are considered for magnetic reasons [5].

Magnetic NPs have attracted a lot of attention due to their unique properties, especially magnetic and electromagnetic ones, and have been used in various applications [6–10]. Magnetic materials based on spinel ferrite with the general formula MFe2O4 (M: divalent metal ion, e.g. Mn, Mg, Zn, Ni, Co, Cu, etc) [11] due to their unique properties, have excellent potential for application in electronics [12], catalyst [13], battery [14], magnetic storage devices and various medical fields such as antibacterial agents [15], immunosassys
One of the most important and attractive nanostructures of spinel ferrite is the nickel-ferrite (NiFe\textsubscript{2}O\textsubscript{4}). This attraction is because of their saturation magnetisation and paramagnetic behaviour, super-paramagnetism and unique magnetic structure. These properties are strongly dependent on the shape, chemical composition and of size NP that can be determined by the synthesis process. In order to produce NP with the desired physico-chemical properties, research and development of various methods for the synthesis of NP is being carried out using various methods. Different synthesis methods have been reported for the production of NiFe\textsubscript{2}O\textsubscript{4} bimetallic nanostructures. A lot of efforts have been put to build NiFe\textsubscript{2}O\textsubscript{4} nanostructures using co-precipitation, sol-gel, electrospinning, combustion and microwave methods. Sivakumar et al. synthesised NiFe\textsubscript{2}O\textsubscript{4} bimetallic NP using ethyl acetate solution with co-precipitation method. Ethyl acetate is flammable and toxic, and this chemical can damage internal organs if it is exposed repeatedly or prolonged. Ethyl acetate can also cause irritation to the eyes or skin.

Nejati and Zabihi were synthesised NiFe\textsubscript{2}O\textsubscript{4} bimetallic NP by the hydrothermal method using Hydrazine hydrate. Hydrazine hydrate is toxic to humans. Current mentioned chemical methods usually have disadvantages such as dependence on expensive equipment, high energy consumption and dependence on chemical compounds toxic to the environment and humans. Therefore, finding simple, economical, inexpensive, non-toxic and environmentally friendly methods are very important and necessary for the synthesis of these types of NP. A method that can eliminate the need to use any harmful chemicals is shown in Nejati and Zabihi. The methods that recently attracted the attention of scientists are synthesis of nanostructures using natural resources such as herbal, bacterial, fungal or their derivative (green methods). Compared with chemical synthesis methods, green methods have various important advantages, including facility, low external energy consumption, affordable, rapid synthesis process, non-toxicity, convenient one-step process and eco-friendly.

The aim of this study was the development of a novel technique of synthesising of bimetallic NiFe\textsubscript{2}O\textsubscript{4} NRP using natural resources. For the first time, NiFe\textsubscript{2}O\textsubscript{4} NRP was synthesised using rosemary. The use of pharmaceutical grade rosemary extract provides an alternative method for the rapid, one-step process and low external energy consumption for the production of NiFe\textsubscript{2}O\textsubscript{4} NRP.

**Materials and methods**

**Synthesis of NiFe\textsubscript{2}O\textsubscript{4} nanorod particles**

Initially rosemary young leaves were washed in distilled water containing Sodium hypochlorite (Merck, Germany) and then placed at room temperature to remove the surface moisture. 200 g of rosemary healthy leaves were added to 1000 mL of deionised water and heated at 80 \(^\circ\)C for period of one hour, then filtered with Whatman filter paper with the size No. 40.

NiFe\textsubscript{2}O\textsubscript{4} NRP were synthesised by green and sustainable process using natural plant extract. For synthesis of NiFe\textsubscript{2}O\textsubscript{4} nanostructure, 0.5 g of FeCl\textsubscript{3}.6H\textsubscript{2}O (98%) and 0.1 g of NiCl\textsubscript{2}.6H\textsubscript{2}O (98%, Merck) were dissolved in 30 mL of rosemary extract at 70 \(^\circ\)C under vigorous stirring. Then, Na\textsubscript{2}CO\textsubscript{3} (99.5%) 1 M solution used to bring pH to 7.5 and stirred at 25 \(^\circ\)C for 3 h. Finally, NRP were washed with ethanol followed by deionised water three times each and then dried in oven (at 60 \(^\circ\)C for 12 h).

**Characterisation of NiFe\textsubscript{2}O\textsubscript{4} nanorod particles**

High-resolution Transmission electron microscope (HRTEM, HRTEM, FEI, Tecnai 20) was used to study the structure and morphology of metallic NP. Field emission scanning electron microscopy (FESEM, MIRA3, TE-SCAN, Czech Republic) were used to study the morphology and size of resulting NiFe\textsubscript{2}O\textsubscript{4} NRP. The chemical composition of the NRP was determined using Energy Dispersive X-Ray Spectroscopy (EDS). Strength of magnetic behaviour of NP was investigated using a vibrating-sample magnetometer (VSM, LBKF model-Meghnatis Daghigh Kavir Company). The X-ray powder diffraction (XRD) was used to analyse the crystal structure of synthesised NRP using X’PertPro, Panalytical (Made in Netherland) with Cu K\textalpha radiation (\(\lambda = 1.54 \text{ Å}\)). Fourier-transform infra-red spectroscopy (FTIR) spectra of the rosemary extract alone and greener synthesis NiFe\textsubscript{2}O\textsubscript{4} NRP was done to evaluate possible functional groups involved in the synthesis process. This analysis obtained using an FTIR spectrometer (TENSOR II, Bruker, Germany) in the range of 400–4000 cm\(^{-1}\). The X-ray photoelectron spectroscopy (XPS) was done for studying the surface chemistry of a NRP by Thermofisher Scientific K-Alpha (Thermofisher Scientific K-Alpha).

**Cytotoxicity investigation of NiFe\textsubscript{2}O\textsubscript{4} nanorod particles**

The human breast cancer (MCF-7) cell lines were cultured in Dulbecco’s Modified Eagle Medium (DMEM, Sigma-Aldrich) supplemented with 10\% foetal bovine serum (Sigma-Aldrich), 100 IU/mL of penicillin (Invitrogen, UK) and 100 \(\mu\)g/mL of streptomycin (Sigma-Aldrich), 10% foetal bovine serum (Sigma-Aldrich) and 100 IU/mL of penicillin (Invitrogen, UK).

10\(^4\) cells/well of MCF-7 cell lines were seeded on the plates (96 well plate) and kept at 37 \(^\circ\)C with 5% CO\textsubscript{2}. After 24 h, the medium was aspirated and 100 \(\mu\)L NiFe\textsubscript{2}O\textsubscript{4} nano-rod particles with concentrations of 2, 4, 8, 16, 32, 64, 128, 256 and 512 \(\mu\)g/mL were added to each well, separately. Then the 96 well plates were incubated for 24 h at 37 \(^\circ\)C with 5% CO\textsubscript{2}. The WST-1 (10 \(\mu\)L) was added to the wells, and then plates incubated for 4 h. Finally the optical density (OD) was measured at 420–580 nm using ELISA (BioTeks E1x800).

The survival rate (%) was calculated using following Equation (1).

\[
\text{Survival rate} \% = \left( \frac{\text{OD in treatment group}}{\text{OD in control group}} \right) \times 100
\]

(1)
Results

The X-ray diffraction pattern of NiFe₂O₄ NRP is shown in Figure 1. XRD spectrum showed peaks in 2θ around 30.02°, 35.67°, 38.19°, 54.01°, 57.98° and 62.92° that are corresponded to the 220, 311, 222, 422, 511 and 440 reflection planes of the NiFe₂O₄, respectively [50]. The three peaks are 311, 222 and 440 planes of the spinel NiFe₂O₄ structure [51,52]. The phenolic compounds of the rosemary extract and extensive of the XRD pattern expresses the size of very fine particles and the background of amorphous [53,54]. The results are close of the previously published reported of NiFe₂O₄ NRP XRD pattern [32,55–58].

Figure 1. X-ray diffraction pattern of biogenic NiFe₂O₄ nanorod particle.

Figure 2. FTIR spectrum of rosemary extract alone (1, blue) and greener synthesised of NiFe₂O₄ nanorod particle (2, red).
FTIR analysis

Figure 2 shows the functional groups involved in the synthesis process. The broad absorption band at 3448.92 cm\(^{-1}\), 400–700 cm\(^{-1}\) and the peak around 1637.72 cm\(^{-1}\) were related to O–H functional group, bending out of plane C–H and stretching vibrations of conjugated C–C of benzenoid ring, respectively, in rosemary extract [59]. The intensity of these bands decreases significantly in NiFe\(_2\)O\(_4\) NRP curve. The FTIR absorption band at \(\approx 600\) cm\(^{-1}\) \((v_1)\), corresponds to intrinsic stretching vibrations of the metal ion at the tetrahedral site, \((M - O)\) and absorption band at around \(\approx 450\) cm\(^{-1}\) \((v_2)\) is assigned to octahedral metal stretching vibration \((M - O; Fe^{3+} \leftrightarrow O \text{ and } Ni^{2+} \leftrightarrow O)\) [60].

The force constant of octahedral \(K_{octa}\) and the tetrahedral \(K_{tetra}\) site have been estimated for \(v_1\) and \(v_2\) by using the

Figure 3. FESEM and EDS of bimetallic magnetic NiFe\(_2\)O\(_4\) nanorods particles synthesis using Rosemary.
Waldron [61] suggested Equation (2):

\[ K = 4\pi^2 \times C^2 \times M \times v^2 \]  

(2)

“\( K \)” is the force constant, “\( v \)” is the corresponding wave number, “\( M \)” is the atomic mass, “\( C \)” is speed of light (free space).

In FTIR spectra of greener NiFe\(_2\)O\(_4\) NRP the band at 3385.52 cm\(^{-1}\) are attributed to the H–OH bending vibration of the free hydroxyl groups of phenolic compounds of can be due to the free or absorbed water on the surface of ferrite nanoparticles [62]. The band at 1604.41 cm\(^{-1}\) are attributed to the H–OH bending stretching of the carboxylic acid [54,55]. The broad absorption band at higher ~600 cm\(^{-1}\) corresponds to intrinsic stretching vibrations of the metal at the tetrahedral complexes (Fe \(\leftrightarrow\) O) and the absorption band

Figure 4. HRTEM images at different scale (a), EDS results (b) and SAED patterns (c) of bimetallic magnetic NiFe\(_2\)O\(_4\) nanorods particles synthesis using Rosemary.
lower at $\sim 400 \text{ cm}^{-1}$ corresponds to the vibrations of octahedral complexes (Ni $\leftrightarrow$ O) [52]. These two absorption bands show very small particles of spinel ferrites [63,64].

FESEM and EDS was used to observe the morphology and chemical composition of bimetallic magnetic NiFe$_2$O$_4$ NRP. The results (Figure 3(a)) show the synthesis of rod-shaped particles with the particle size of 40–200 nm and a diameter of about 5 nm. The EDS analysis confirmed the presence of Ni, Fe and O elements in the NiFe$_2$O$_4$ NRP sample (Figure 3(b)).

EDS spectra (Figure 3) of the bimetallic magnetic NiFe$_2$O$_4$ NRP showed the atomic, weight percentages ratios of Ni(12%): Fe(24%) and O(48) are close to the theoretical value (Ni: Fe: O = 1:2:4), suggesting purity of green synthesised bimetallic magnetic NiFe$_2$O$_4$ NRP. The content of C element is due to the residual organic matters (plant extract).

In Figure 4 are shown the results obtained from HRTEM and SAED analyses. HRTEM micrograph identified the of NiFe$_2$O$_4$ rod-like nanostructure (Figure 4(a)). The EDS chemical composition analysis confirmed the presence of Ni, Fe and O elements in the NiFe$_2$O$_4$ NRP sample (Figure 4(b)). The selected area electron diffraction (SAED) pattern shows diffuse rings matches well with (222), (311) and (440) NiFe$_2$O$_4$.
planes. Also, the overlapping of individual rings indicates the presence of amorphous nanorod [55,65].

The Figure 5 shows the composition of the surface chemistry of NiFe₂O₄ NRP samples using XPS. The peak over binding energy curves showed photoelectron for iron (Fe 2p₃/2, at 711 eV and Fe 2p₄/5, 725 eV), oxygen (Fe 2p₄/5 at 725 eV), and nickel, Ni 2p showed two split-orbit peaks in the range of Ni 2p₃/2 at 874 eV) and Ni 2p₄/5 at 856 eV). In addition the presence of two peaks 874 and 862 is shows presence ions Ni²⁺/Ni³⁺ on the of the surface of the NiFe₂O₄ NRP. This has leads significant increase in electro-catalytic activity in the synthesis of NiFe₂O₄ NRP.

The magnetic properties of bimetallic magnetic NiFe₂O₄ NRP were investigated by VSM. Greener synthesised NiFe₂O₄ NRP showed a magnetic saturation value of 0.54 emu g⁻¹ at room temperature (Figure 6). The saturation values for bulk of Fe, Ni and NiFe₂O₄ and are 221.7, 54.4 and 56 emu/g, respectively [31,66]. Reduction of the magnetisation for greener NiFe₂O₄ NRP can be due to formation thin oxide layer, the surface spin disorder and the size confinement [66].

Figure 7 shows the toxicity of the NiFe₂O₄ NRP does rate. The increase in NiFe₂O₄ NRP over 4 μg/mL significantly effect of MCF-7 cancer cells studied survival, at the concentration of the 500 μg/mL, there was over 55% cells died.

Discussion

The results of this study revealed a successful self-assembly NiFe₂O₄ NRP using a green, simple, non-toxic, low-cost
synthesis methodologies using rosemary extracts. XRD spectrum of NiFe\textsubscript{2}O\textsubscript{4} NRP showed peaks in 2\(\theta\) around 30.02\(^\circ\), 35.67\(^\circ\), 38.19\(^\circ\), 54.01\(^\circ\), 57.98\(^\circ\) and 62.92\(^\circ\) that corresponded to the 220, 311, 222, 422, 511 and 440 reflection planes of the NiFe\textsubscript{2}O\textsubscript{4} respectively. The three peaks are 311, 222 and 440 planes of the spinel NiFe\textsubscript{2}O\textsubscript{4} structure. HRTEM micrograph identified the of NiFe\textsubscript{2}O\textsubscript{4} rod-like nanosctructure.

Greener synthesised NiFe\textsubscript{2}O\textsubscript{4} NRP showed a magnetic saturation value of 0.54 emu g\(^{-1}\) at room temperature. NiFe\textsubscript{2}O\textsubscript{4} nano-sized particles are of great attention due to their applications in environment, medicine and energy [67]. The proposed method of the synthesis provides less risk to the human health and environment and due to the low cost higher applications. In our knowledge, this is the first time rosemary extract has been used to synthesis NiFe\textsubscript{2}O\textsubscript{4} with nanorod shape structures. EDS spectra of the bimetallic magnetic NiFe\textsubscript{2}O\textsubscript{4} NRP showed the atomic, weight percentages ratios of Ni(12%): Fe(24%) and: O(48) are close to the theoretical value (Ni: Fe:O = 1:2:4), suggesting purity of green synthesised bimetallic magnetic NiFe\textsubscript{2}O\textsubscript{4} NRP. The content of C element is due to the residual organic matters (plant extract).

We have a serious lack of investigation regarding the application of NiFe\textsubscript{2}O\textsubscript{4} NP in medicine. Our result showed that NiFe\textsubscript{2}O\textsubscript{4} nanorod particles with cytotoxicity activity can be used as a new class of anticancer agent in design novel cancer therapy research.

The use of pharmaceutical rosemary provides an alternative method for the one-step rapid process and low external energy consumption and non-toxicity synthesis of NiFe\textsubscript{2}O\textsubscript{4} nanorod particles. The green synthesis of NiFe\textsubscript{2}O\textsubscript{4} NP using rosemary avoids the need for harmful reducing or capping agents.

There are few publications attempted to develop NiFe\textsubscript{2}O\textsubscript{4} NRP, using verity of the methods, but the synthesis technique either has been complex or not high-quality end products. Examples are the synthesis of NiFe\textsubscript{2}O\textsubscript{4} nanorod shape particles using Urtica plant extract [62], but they had to use a hydrothermal process by employing Teflon autoclave. Their TEM results showed that their NPs are agglomerated with irregular morphology. However cannot recognise a rod-shaped NP in their SEM or TEM results. Other attempted to synthesis NiFe\textsubscript{2}O\textsubscript{4}/Fe\textsubscript{2}O\textsubscript{3}/CeO\textsubscript{2} nanocomposite using a walnut green hull extract with calcinated at 600 °C [68]. Their results showed that their NPs are highly agglomerated with irregular morphology. Our technique of production of the NiFe\textsubscript{2}O\textsubscript{4} NRP is simple and cost effective by rosemary plants without using any external device, calcination or chemicals. We have fully characterised the product and showed that regular rod morphology of NiFe\textsubscript{2}O\textsubscript{4} nano-sized particles without agglomeration. In term of the cytotoxicity, our results are similar to others. Figure 8 showed the schematic of the green and sustainable process for NiFe\textsubscript{2}O\textsubscript{4} nano-rod particles.

**Conclusion**

We have developed a NiFe\textsubscript{2}O\textsubscript{4} nanorods particles using greener and cost-effective synthesised methodologies using for first time Rosemary extract. The application of metallic nanoparticles in biomedicine and environmental are a multibillion-dollar. This technique helps the industries application faster to the end products. The type, structure and physicochemical properties of nanostructures produced were studied using HRTEM, XRD, FeSEM, XPS, VSM and FTIR. Moreover, NiFe\textsubscript{2}O\textsubscript{4} NP had cytotoxicity effect on MCF-7 cells survival which suggests that NiFe\textsubscript{2}O\textsubscript{4} NP can be used as a new class of anticancer agent in design novel cancer therapy research. NiFe\textsubscript{2}O\textsubscript{4} nanorod particles can be used to increase the level of public health. Bimetallic magnetic NiFe\textsubscript{2}O\textsubscript{4} NP synthesis using Rosemary should be used in design for biomedical applications such as medical senor or antibacterial application.

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**Disclosure statement**

No potential conflict of interest was reported by the authors.
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