



Original Research Article

Facile preparation of $\text{NiFe}_2\text{O}_4/\text{NaCl}$ nanocomposites by wet chemical co-precipitationAliakbar Dehno Khalaji^{a,*}, Marketa Jarosova^b , Pavel Machek^b^a Department of Chemistry, Faculty of Science, Golestan University, Gorgan, Iran^b Institute of Physics of the Czech Academy of Sciences, Na Slovance 2, 182 21 Prague, Czech Republic

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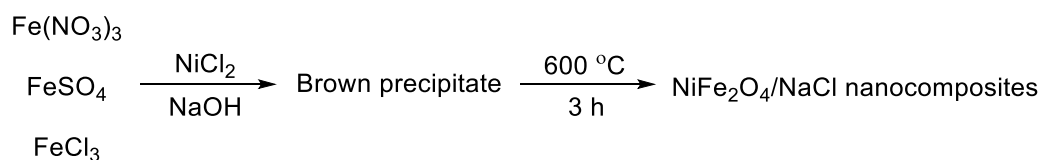
Cubic

ABSTRACT

In this work, we utilized various iron salts $\{\text{Fe}(\text{NO}_3)_3, \text{FeSO}_4 \text{ and } \text{FeCl}_3\}$ at the presence of aqueous solutions of NiCl_2 and NaOH , respectively, to prepare $\text{NiFe}_2\text{O}_4/\text{NaCl}$ nanocomposites using wet chemical co-precipitation. The dark-brown products were afterwards calcined for 3 h at 600 °C. All products were characterized using Fourier-transform infrared spectroscopy (FT-IR), X-ray diffraction (XRD) and transmission electron microscopy (TEM). XRD results confirmed formation of the $\text{NiFe}_2\text{O}_4/\text{NaCl}$ nanocomposites. The TEM results revealed that the particles are relatively similar in cube-like shape; however, the size was distinct.

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Graphical Abstract

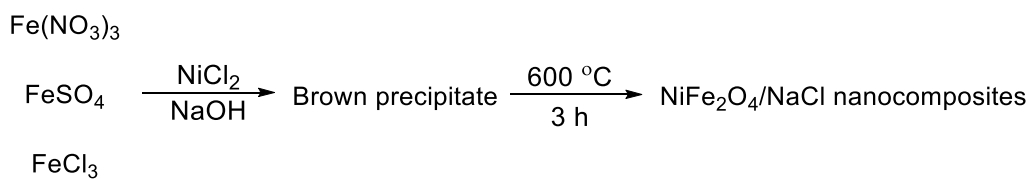


Introduction

In recent years, various nanoferrites have been prepared and characterized and their capability to adsorb different organic dyes and heavy metals ions has been studied [1–5]. Among them, NiFe₂O₄ is one of the best due to its interesting structures [6–13]. NiFe₂O₄ demonstrates super-paramagnetic properties and various applications such as catalysts, gas sensor, site specific drug delivery and magnetic fluids [14–17]. Until now, many techniques were developed to preparation of NiFe₂O₄ [6–13] such as wet chemical co-precipitation [11], sol-gel auto combustion [12] and flotation extraction [13]. Multiphase solid materials that one of the phases has dimensions of less than

100 nanometers (nm) named as nanocomposites [18–22]. Nowadays, various nanocomposite containing NiFe₂O₄ such as NiFe₂O₄/ZnO [23], NiFe₂O₄/NiO [24], NiFe₂O₄/Cu [25], NiFe₂O₄/Graphene [26], NiFe₂O₄/Co₃O₄ [27], BiOBr/NiFe₂O₄ [28] have been prepared by hydrothermal [18], calcination [19], deposition/precipitation [22] cop-recipitation/hydrothermal [29], sol-gel auto-combustion [30, 31] and mechanochemical [32].

In this research study, we prepared NiFe₂O₄/NaCl nanocomposites from various iron salt at the presence of NiCl₂ by wet chemical co-precipitation and calcination at 600 °C for 3 h (Scheme 1).



Scheme 1. Synthesis of NiFe₂O₄/NaCl nanocomposites

Experimental

Material and Methods

All the compounds used in this work were purchased from Merck and Sigma-Aldrich chemical company and were used as received without further purification. XRD patterns of the as-prepared compounds were obtained on Empyrean powder diffractometer of PANalytical in Bragg-Brentano configuration equipped with a flat sample holder and PIXcel3D detector (Cu K α radiation, $\lambda = 1.5418 \text{ \AA}$). TEM images of nanoparticles were recorded on transmission electron microscope Philips CM120 with a LaB₆ cathode operating at 120 kV and equipped with CCD camera Olympus Veleta.

Preparation

An appropriate of iron salts {Fe(NO₃)₃ or FeSO₄ or FeCl₃} (1 g) was dissolved in a 5 mL of de-ionized water and then a solution of nickel(II) chloride (1 g) in 5 mL de-ionized water was added drop by drop and the mixture was stirred for 10 min. After that, 10 mL of NaOH (1 g) dissolved in de-ionized water was added. The final dark-brown solution was magnetically stirred for 90 min at 50 °C followed by addition of 10 mL of de-ionized water to the solution. The dark-brown precipitates (**1**, **2** and **3**) as precursor were filtered off and dried at room temperature. Then the dark-brown powders were calcined in alumina crucible at 600 °C for 3 h, with a heating rate of 20 °C/min and the black precipitates (**4**, **5** and **6**) were collected. Finally, all precipitates were characterized by FT-IR, XRD and TEM.

Results and Discussion

FT-IR Spectroscopy

In the FT-IR spectra of $\text{NiFe}_2\text{O}_4/\text{NaCl}$ products (Figures 1–3) two broad peaks appeared at about 1600 and 3420 cm^{-1} can be assigned to the symmetric vibrations of O–H groups [12]. Also, a broad peak at 1080 cm^{-1} assigned to O–H bending vibration [33, 34]. The sharp peak at 1355 cm^{-1} in the FT-IR spectrum

of **1** represents N–O stretch due to the presence of nitrate group [35]. On the contrary, intensity of this peak in the FT-IR of **4** has been very low and broad, confirmed the elimination of nitrate group by calcination of **1**. There is a broad peak at about 563 cm^{-1} in the FT-IR spectrum of **4** representing tetrahedral and octahedral modes of NiFe_2O_4 [11, 12, 33, 36]. Similar peaks are also observed in the FT-IR spectra of the other as-prepared compounds.

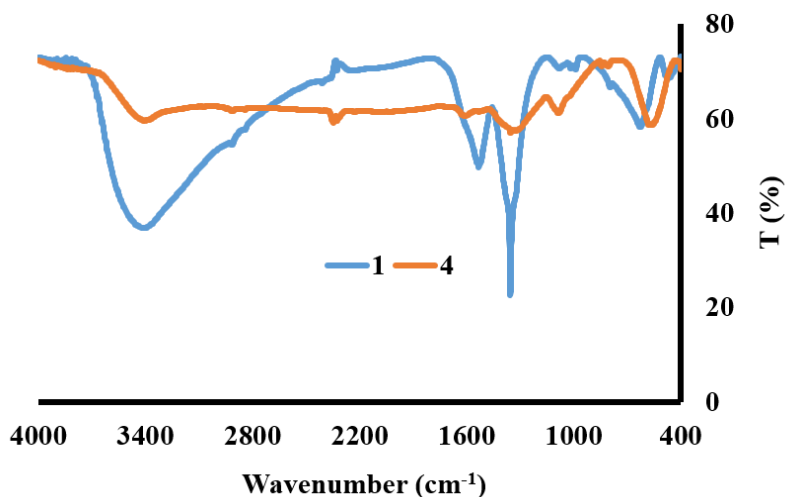


Figure 1. FT-IR spectra of precipitates **1** and **4**

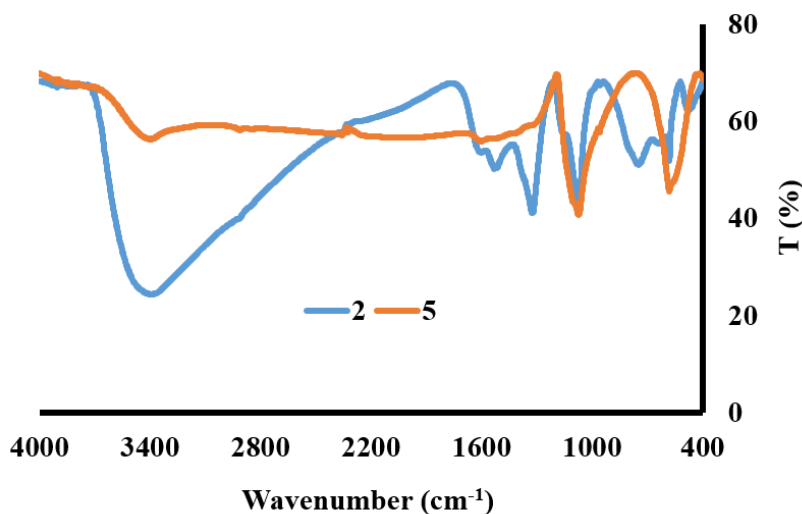


Figure 2. FT-IR spectra of precipitates **2** and **5**

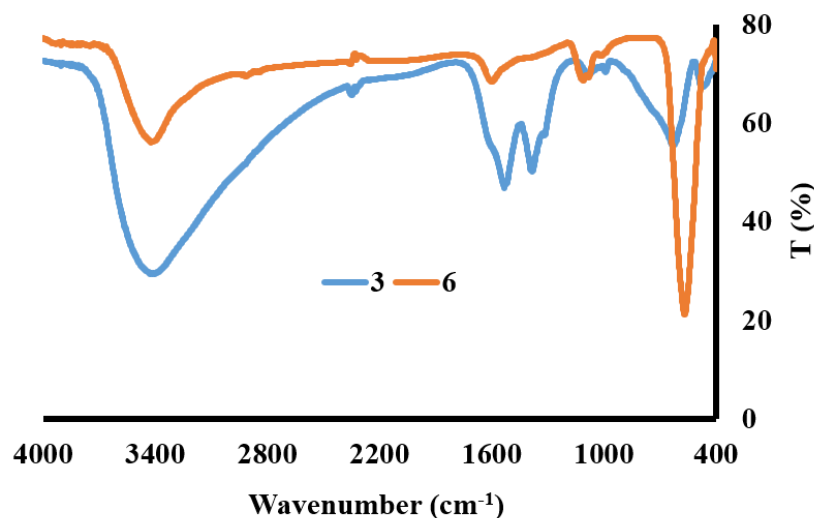


Figure 3. FT-IR spectra of precipitates 3 and 6

XRD analysis

Figures 4-6 reveals the XRD patterns of the precursors and the as-prepared $\text{NiFe}_2\text{O}_4/\text{NaCl}$ products. Diffraction patterns confirm the formation of $\text{NiFe}_2\text{O}_4/\text{NaCl}$ nanocomposites in all precipitates after calcinations. The diffraction peaks of the (111), (220), (311), (222), (400), (422), (511), (440), and (533) crystal planes, corresponding to the cubic phase structure of the NiFe_2O_4 (JCPDS No. 87-0722) [37, 38].

Also, the diffraction peaks at the (200), (220), (222) and (420) crystal planes, corresponding to the halite NaCl structure [39]. It can be also noticed that the intensity of diffraction peaks increased with change of iron precursor from $\text{Fe}(\text{NO}_3)_3$ to FeSO_4 and FeCl_3 , confirmed the higher crystallinity of the as-prepared NiFe_2O_4 nanoparticles. In Fig. 4 sharp and intense peaks of NaCl can be seen whereas only few weak and broad peaks of NiFe_2O_4 are visible. This suggests that either nickel ferrite particles are very small and don't reflect

sufficiently yet or that amount of nickel ferrite is lower compared to NaCl . In Figure 5, on the contrary, diffraction peaks of NaCl are negligible in comparison with peaks of NiFe_2O_4 , indicating higher proportion of nickel ferrite. Finally, in Figure 6 diffraction peaks of NaCl and NiFe_2O_4 are comparable, suggesting comparable amounts of both phases. Unfortunately, quality of measured data was insufficient to allow us to determine the proportions of both phases in the mixtures.

TEM images

Figures 7-9 show the morphology of prepared particles. It was not almost possible to see separated particles because all products form big clusters. However, it is possible to distinguish cube-like shape of most particles. The influence of used iron salt on particle size or morphology is not evident. The particles after calcination (precipitates 4, 5, 6) seem to be finer-grained compared to precipitates 1, 2 and 3.

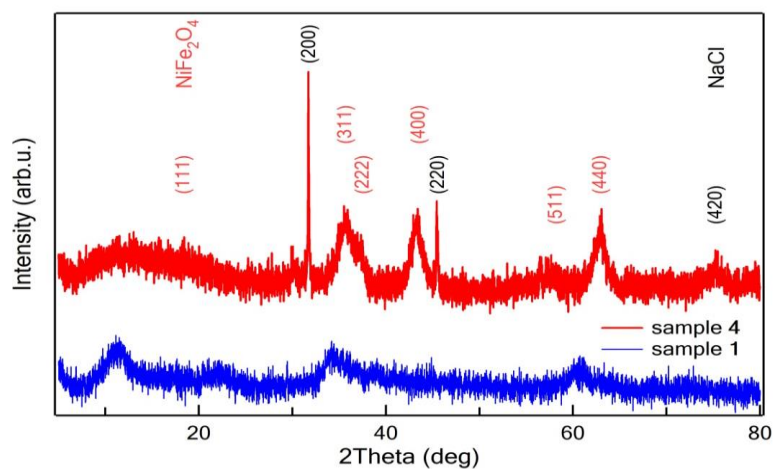


Figure 4. XRD patterns of **1** and **4** (NiFe_2O_4 peaks are in red, NaCl peaks are in black).

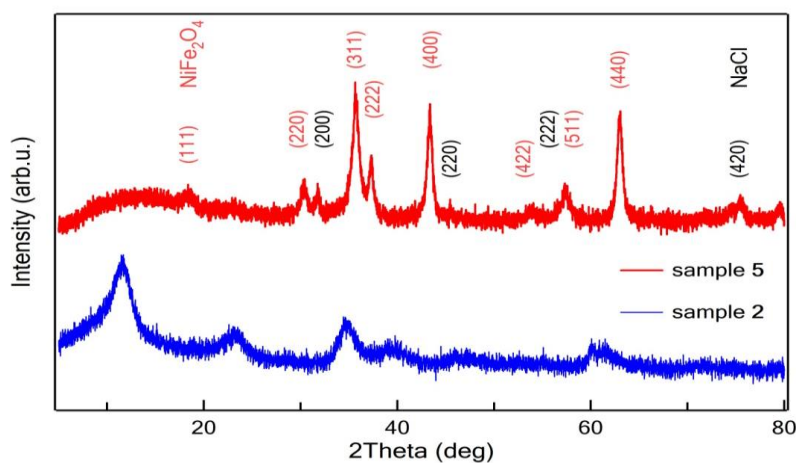


Figure 5. XRD patterns of **2** and **5** (NiFe_2O_4 peaks are in red, NaCl peaks are in black).

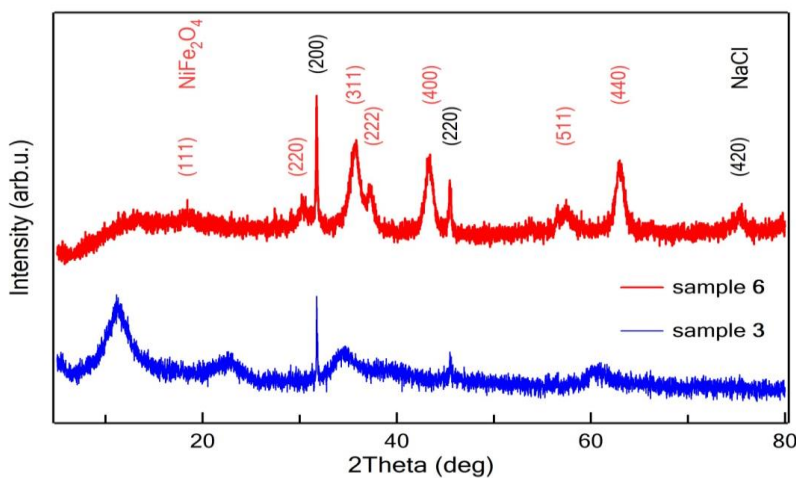


Figure 6. XRD patterns of **3** and **6** (NiFe_2O_4 peaks are in red, NaCl peaks are in black).

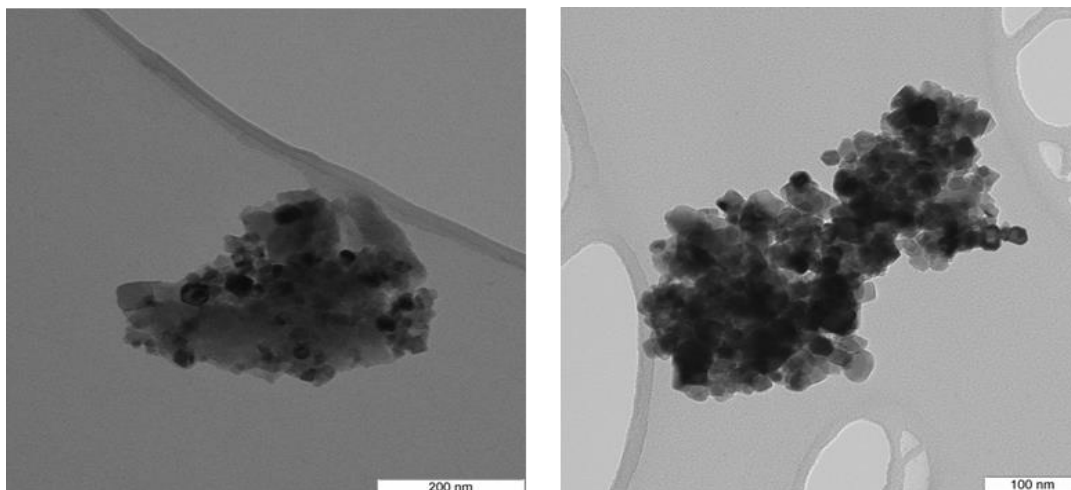


Figure 7. TEM images of **1** and **4**

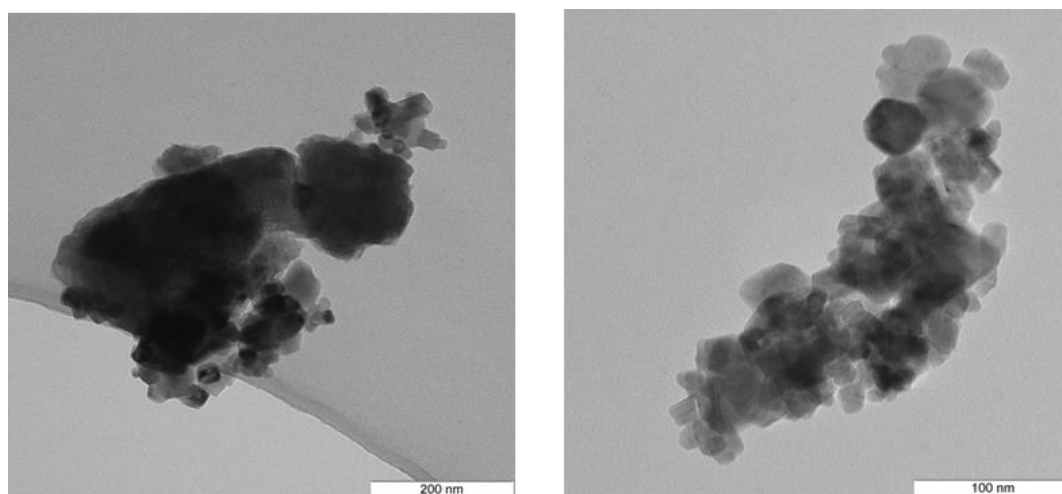


Figure 8. TEM images of **2** and **5**

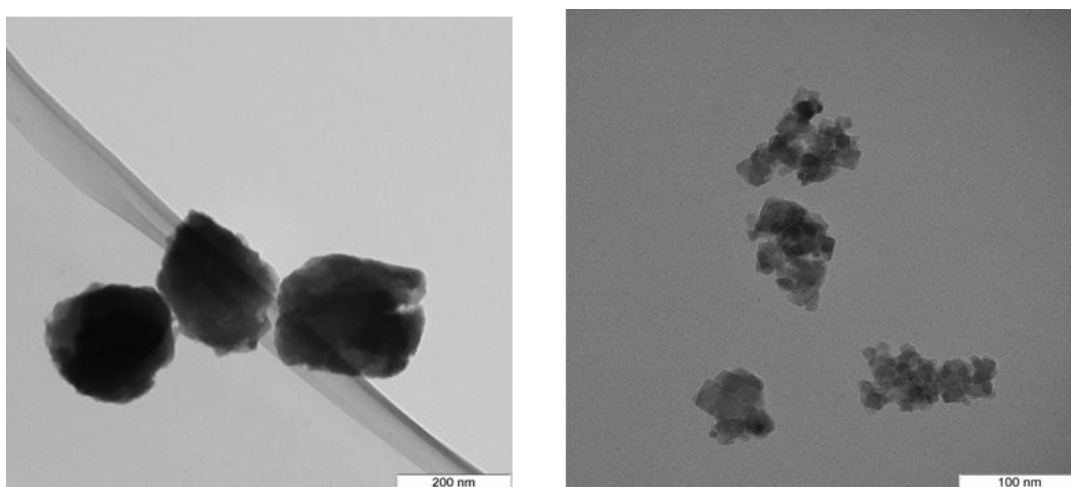


Figure 9. TEM images of **3** and **6**

Conclusions

In this paper, we report a facile, cheap, and eco-friendly for preparation of NiFe₂O₄/NaCl nanocomposites. FT-IR spectra show that the NiFe₂O₄/NaCl nanocomposites were prepared by calcination of the synthesized precursors at 600 °C. In XRD patterns of the products, in addition to the peaks of NiFe₂O₄ composition, peaks related to NaCl are also seen, then XRD patterns confirm the formation of NiFe₂O₄/NaCl nanocomposites in all precipitates after calcinations. The height as well as the width of the peaks are different from each other, which is a reason for their different size and crystallinity of the as-prepared NiFe₂O₄/NaCl nanocomposites. TEM images confirmed that all products form big clusters with a cube-like shape for most particles.

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Disclosure Statement

No potential conflict of interest was reported by the authors.

Orcid

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