Synthesis of Iron-nickel Particles by Co-precipitation Technique and Used as a Contrast Medium in an MRI Machine

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Abstract. In this study, Nickel Zinc Ferrite was prepared using the co-precipitation method according to the chemical formula Ni1-xZnxFe2O4, where the values of (x = 0,0.1,0.3,0.5,0.7,0.9) respectively, the structural and magnetic properties were studied through X-ray diffraction spectroscopy. FESEM showed the porosity and distribution of the homogeneous nanoparticles roughly also its particle size. The vibration sample Magnetometer (VSM)was used to study magnetic properties, and the Fourier transform infrared (FTIR) spectra were measured in the range of 400-4000 cm⁻¹. Magnetic nanoparticles effect on contrast factors in magnetic resonance imaging (MRI) images was studied, it showed Good and robust imaging technique for diagnosis and post-treatment evaluation for a wide range of diseases. MRI contrast can be enhanced by using negative or positive contrast agents, resulting in clearer (T1-weighted) or darker (T2-weighted) images, respectively, with super-magnetic nanoparticles yielding T2 contrast agents widely used in molecular imaging applications and cellular. As most of the work in this field focuses on the acquisition of biological species using nanoparticles, which in turn will improve the magnetic properties that lead to higher imaging.

Keywords: Co-precipitation method, MRI, Contrast.

1. Introduction

The magnetic properties of nanometer-sized particles have attracted much attention in recent years due to their unique properties. For the preparation of magnetic liquids, nanoparticles with a particle size of the order of 10 nm and a narrow size distribution were required [1]. Magnetic nanoparticles have aroused increasing interest among researchers in various fields due to their wide applications such as information storage system, medical diagnostics, ferrous fluid technology, etc. [2,3]. In this work, the prepared particles were used as a contrast material in MRI as a biological imaging tool with the fact that these particles or tissues have good magnetic sensitivity and when they interact with contrast agents, they provide high contrast images [4]. These prepared magnetic particles have the ability to produce dipoles and magnetic fields with strong spatial differences, which accelerate the spin relaxation of adjacent water molecules in soft tissues and thus determine the spatial resolution and sensitivity of the MRI. It can lead to longitudinal (T1) and transverse (T2) relaxation. [5].

Magnetic nanoparticles (MNP) can accelerate the relaxation of adjacent water molecules by creating local magnetic fields, inside the prepared sample molecules [6]. Magnetic spinel minerals are more efficient, and efficiencies are influenced by the size, composition, atomic structure, and surface characteristics [7]. One of the fundamental aspects of MRI research is the need for high sensitivity, which in turn will allow high-contrast MR images even at a low dose, which results in a lower risk of toxicity of the MRI itself [8]. The sensitivity of the contrast agent T2 increases as the magnetic values of the prepared particles increase. Increasing the size of the magnetic core can lead to the use of strong magnetizing materials and promoting the diffusion of water using low porosity nanostructures associated with the nanoscale size of magnetic particles [9].
2. Experimental Procedure

Ferrite nanoparticles were prepared with the following formula Ni$_{1-X}$Zn$_X$Fe$_2$O$_4$ by co-chemical precipitation method according to the proportions (X= 0, 0.1, 0.3, 0.5, 0.7, 0.9) to prepare Ni$_{1-X}$Zn$_X$Fe$_2$O$_4$ (NP) particles from FeCl$_3$ mixtures and ZnCl$_2$ solutions were used. NiCl$_2$, in the presence of 5% NaOH solvent in 100 ml of distilled water at pH = 12 with continuous stirring at 80 °C, field gradient. In this way the magnetic nanoparticles were prepared and the compatibility of biomagnetic magnetite was demonstrated. [10] The structural composition of all samples was studied. Using X-ray diffraction (6000 XRD, Cu-κα radiation, λ = 1.54. Shapes for all samples [11]. The particle size was calculated using Scherrer’s equation (1) The lattice constant (a) was worked out. Calculate it also using Equation (2) [12]. The magnetic properties that can be obtained with (VSM) were studied, after obtaining nano ferrite powders, and based on the structural and magnetic properties, the prepared materials are transformed into a magnetic liquid using deionized water with an organic solvent to ensure the dispersion process. The powders were dissolved using an ultrasound machine (Ultra-sonic) for a period of 5 hours where the nanomagnetic liquid shown in figure 1 was obtained, and it was found that the time factor has no effect on the prepared liquid, and (1) represents the prepared liquid. After one month, the magnetic fluid is thus used in the medical application by using it as a coloring tool in the magnetic resonance device. It was applied to the type of laboratory mice (albino) / pulpec that was purchased from the Center for Cancer Research and Medical Genetics.

\[ D = \frac{k\lambda}{\beta \sin \theta} \]  
(1)

where \( D \) is the average crystal size, \( K \) is the Scherrer coefficient (0. 9), \( \lambda \) is the x-ray wavelength, \( \theta \) is Bragg’s angle (2\( \theta \)) and \( \beta \) the full width at half-maximum (FWHM) in radians.

\[ a = \frac{n\lambda \sqrt{k^2+k^2+l^2}}{d \sin \theta} \]  
(2)

where \( a \) the lattice parameter, \( d \) is the interplanar distance of each plane and \( (hkl) \) are Miller indices, \( \theta \) is Bragg’s angle, \( \lambda \) is the x-ray wavelength, \( n = 1, 2, 3..... \)

![Figure 1](image_url). The stabilized Ferro fluid containing cysteine magnetite nanoparticles after 1 month (concentration: 2 mg Fe / ml)

3. Results and Discussion

The structural properties of the prepared particles have been studied using XRD data, and the average crystal size of the diameter of the crystals can be estimated according to Shearer equation (1). Table 1 shows the particle size, the particle retinal values, and the figure 2 showing the results of X-ray diffraction.

**Table 1.** XRD analysis of peak position (311) crystal size and Lattice constant.

| X  | Particle Size (nm) | Lattice Constant (Å) |
|----|--------------------|----------------------|
| 0  | 9.8                | 3.78                 |
| 0.1| 9.9                | 3.78                 |
| 0.3| 10.0               | 3.78                 |
| 0.5| 10.1               | 3.78                 |
| 0.7| 10.2               | 3.78                 |
| 0.9| 10.3               | 3.78                 |
The X-ray diffraction result of ferrite nanoparticles of nickel-zinc imaged with the chemical formula Ni$_{1-x}$Zn$_x$Fe$_2$O$_4$ (x = 0, 0.1, 0.3, 0.5, 0.7, 0.9) analyzed by comparing it with the standard JCDPS card. All observed peaks matched well with JCPDS card No. 52-0278 of nickel ferrite, confirming the single-phase cubic spinel Fd m of ferrite samples. From figure (2) it can be seen that the small shift of the peaks towards the short angle to increase the concentration of Zn$^{2+}$, the X-ray test also showed that there is no growth for stages other than the ferrite phase.

![Figure 2: XRD patterns for Nickel zinc Ferrite nanoparticles at 700 °C.](image)

Figure 3 show the electron microscopy of field emissions of ferrite nickel-zinc samples according to the chemical formula Ni$_{1-x}$Zn$_x$ Fe$_2$O$_4$ giving a clear appearance of the composition of the porous nature and nanoparticles and the nearly homogeneous distribution of the size of the nanoparticles. From these images, it can be seen that the micrograph of nanoparticles with spinel structure replacing nickel ferrite reveals that the fine structure of ferrite has been affected by substitution and appears stacked with homogeneous spherical and polyhedral particles, and thus the particle represents the sizes fixed on each image average grain sizes for each A group of closer particles in each sample, then the
clarified particles represent the maximum size, meaning that there is also another size range in another region. The grain size also changes according to the change in the X ratio due to the difference between the radii of zinc and nickel ions.

The formation of the porous structure of the ferrite samples may be due to the release of excess amounts of gases during the combustion method [13] In the FE-SEM images, some agglomerated areas appear. This is most likely due to the naturally occurring interaction between magnetic nanoparticles and the thermal process. It was observed in many cases the tendency for the nanoparticles to agglomerate in Nano crystalline spinel ferrites [14]. Infrared Fourier transforms spectra (FTIR) measured in a range of 400-4000 cm⁻¹ for prepared samples as shown in Figure (4) Spectra give a composition about the spinal structure and network vibration, while the ranges are around 3846-1417 cm⁻¹ attributed to the expansion vibration of the hydroxyl group OH, the ranges at

Figure 3: FE-SEM micrographs of Ni₁₋ₓZnxFe₂O₄ nanoparticles for (X=0.0,0.1,0.3,0.5,0.7,0.9) at 700 °C.
1384 cm\(^{-1}\) indicate the group of carboxyl’s (C=O) the vibration and absorption range is about 1037 cm\(^{-1}\). The nitrate group produces no stretch vibrations [15]. Also, two vibration bars between 600 cm\(^{-1}\) and 400 cm\(^{-1}\) related to the formation of the spinal structure can be observed according to Waldron's theory all these values noted in the table (2) [16].

![FTIR spectrum of nickel-zinc ferrite, Ni\(_{1-x}\)Zn\(_x\)Fe\(_2\)O\(_4\) (x = 0.0, 0.1, 0.3, 0.5, 0.7, 0.9).](image)

**Figure 4.** FTIR spectrum of nickel-zinc ferrite, Ni\(_{1-x}\)Zn\(_x\)Fe\(_2\)O\(_4\) (x = 0.0, 0.1, 0.3, 0.5, 0.7, 0.9).

**Table 2:** The FTIR absorption bands measured for Ni-Zn spinal ferrite samples.

| Sample | Molur ratio | Composition | \(\nu_1\) cm\(^{-1}\) | \(\nu_0\) cm\(^{-1}\) |
|--------|-------------|-------------|----------------|----------------|
| B1     | 0           | NiFe\(_2\)O\(_4\) | 583.6 | 421.7 |
| B2     | 0.1         | Ni\(_{0.9}\)Zn\(_{0.1}\)Fe\(_2\)O\(_4\) | 574.3 | 428.0 |
| B3     | 0.3         | Ni\(_{0.7}\)Zn\(_{0.3}\)Fe\(_2\)O\(_4\) | 586.8 | 421.7 |
| B4     | 0.5         | Ni\(_{0.5}\)Zn\(_{0.5}\)Fe\(_2\)O\(_4\) | 593.0 | 415.5 |
| B5     | 0.7         | Ni\(_{0.3}\)Zn\(_{0.7}\)Fe\(_2\)O\(_4\) | 580.6 | 428.0 |
| B6     | 0.9         | Ni\(_{0.1}\)Zn\(_{0.9}\)Fe\(_2\)O\(_4\) | 574.3 | 402.3 |

The magnetic properties of the prepared particles were analyzed using a magnetic scale (VSM) at room temperature. Figure 5 shows the M-H curves for the samples. The ring is narrow which means that prepare samples are soft magnetic material. The values of saturation magnetization (Ms) and coercive (HC) were extracted directly from these curves as in the figures and were included in the various Zn content in table 3. It can be seen from figure 5, where the saturation and Compulsive magnetization increases with zinc content and then decreases to (x= 0.5). The behavior of the saturation magnetization may be explained by the distribution of actions and by the superior exchange
interactions, and also because of the different ratios of magnetic moments in every magnetic domains according to the changing of \((x)\) ratio for every sample.

**Figure 5.** Magnetic hysteresis loops of \(\text{Ni}_{1-x}\text{Zn}_x\text{Fe}_2\text{O}_4\) \((x=0, 0.1, 0.3, 0.5, 0.7\) and \(0.9)\).

**Table 3.** The values of saturation magnetization, remanence, connectivity, squareness ratio \(M_r/M_s\), anisotropy constant, and permeability.

| Sample | Molur ratio | Composition       | \(M_s\) (emu/g) | \(M_r\) (emu/g) | \(H_C\) (Oe) | \(S=M_r/M_s\) |
|--------|-------------|-------------------|-----------------|----------------|-------------|---------------|
| B1     | 0           | \(\text{NiFe}_2\text{O}_4\) | 0.54            | 0.004          | 21.27       | 0.007         |
| B2     | 0.1         | \(\text{Ni}_{0.9}\text{Zn}_{0.1}\text{Fe}_2\text{O}_4\) | 0.691           | 0.03           | 80.54       | 0.04          |
| B3     | 0.3         | \(\text{Ni}_{0.7}\text{Zn}_{0.3}\text{Fe}_2\text{O}_4\) | 15.2            | 4.627          | 27.21       | 0.29          |
| B4     | 0.5         | \(\text{Ni}_{0.5}\text{Zn}_{0.5}\text{Fe}_2\text{O}_4\) | 9.68            | 4.728          | 154.4       | 0.48          |
| B5     | 0.7         | \(\text{Ni}_{0.3}\text{Zn}_{0.7}\text{Fe}_2\text{O}_4\) | 1.177           | 0.08           | 89.42       | 0.06          |
| B6     | 0.9         | \(\text{Ni}_{0.1}\text{Zn}_{0.9}\text{Fe}_2\text{O}_4\) | 4.218           | 0.301          | 229.4       | 0.07          |

**4. Medical Application**

For the MRI study, samples with nanoparticles and magnetic properties present in the prepared particles were selected within the B3B4 samples. Laboratory rats were used for an MRI scan, and the mice were anesthetized and then the stable nanoparticles were introduced through aqueous ferric fluid prepared at a dose of 2.5 mg (Fe) / kg bw. An MRI scan was performed half an hour after injection [17]. We observe the accumulation of macromolecules and aggregate mainly in tissues such as liver and spleen, however, smaller (28 nm) will penetrate the tissue. From the injected mice, the mice were
anesthetized laterally in the groin area in an MRI chamber, and the side of the mouse was placed for a sagittal plane, then T1 and T2 MRI was performed before the nanomaterial was injected and then compared with those injected in figure 6. The magnetic Nanofluid was injected with Ferrite Nickel Zinc, sample B3 with a concentration of \( X = 0.3 \) Nanoscale (14.3nm). The examination with the MRI the device of this sample revealed pictures of the T1 promoter shown in figure 7, which shows a highly sensitive appearance of the material and shows the penetration of the material to the tissue at Likely T2 shown in figure 8.

Figure 6. shows the resulting images before using the magnetic nanoparticles.

Figure 7. T1 shows the Images from the B3 sample MRI that show clarity of the images produced.
Figure 8. T2 shows the Images from the B3 sample MRI that show clarity of the images produced Weighted MRI scan. B4: X = 0.5. An injection in the right revealed a hypersensitive area also in both T1 and T2 MRI images, however, the fluid appeared to penetrate deeper than the previous material (figure 9 to 10). It was injected, showing good resolution images [18].

Figure 9: A ferromagnetic fluid present at the intestine (*) was shown when the T1.

Figure 10: Shows that the fluid had a greater penetration than the previous material. There were no statistically significant differences between the previous liquid and ferromagneti
5. Conclusions

Stable ferromagnetic nickel particles were fabricated with average nano scale size using co-chemical precipitation method. The nano scale liquids were prepared using an Ultra-sonic machine. The prepared liquids were marked by an iron nanometer stability. This was used as a staining agent in studying images in the MRI machine, and it has been observed to enhance good contrast in post-injection MRI.

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