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PRODUCTION OF MAGNETIC NANOPARTICLE-BASED FERRITES AND COMPREHENSIVE CHARACTERIZATION OF THEIR ELECTRICAL AND MAGNETIC PROPERTIES IN COLLOIDAL SYSTEMS

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This study investigates the synthesis, structural characterization, and physicochemical properties of magnetic nanoparticles derived from iron group metals (Fe, Co, Ni) and their corresponding ferrite dispersions. Magnetic nanoparticles were synthesized via chemical condensation, and their morphology and structure were analyzed using transmission electron microscopy (TEM) and X-ray diffraction (XRD). The synthesized magnetic nanoparticles, comprising Fe_3O_4, $CoFe_2O_4$, and $NiFe_2O_4$ ferrites, exhibited nano-scale dimensions ranging from 10 to 50 nm. The precise correlation between TEM and XRD measurements validated the structural and dimensional characteristics of the synthesized nanoparticles. Aqueous-based magnetic liquids with varying nanoparticle concentrations were prepared, enabling systematic investigation of their electrical conductivity and magnetic susceptibility at ambient temperature. The experimental findings provide critical insights into the fundamental properties of magnetic nanoparticle-based colloidal systems, potentially facilitating advanced applications in materials science, magnetic device engineering, and emerging technological domains. The methodological approach and results presented herein contribute to the expanding understanding of magnetic fluid behavior and performance.
Keywords: *Magnetic nanoparticles; Ferrite dispersions; Colloidal systems; Magnetic fluid; Nanostructured materials; Electrical conductivity; Magnetic susceptibility*
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INTRODUCTION

Magnetic fluids, a class of smart materials, have attracted significant attention in modern materials science and technology due to their ability to alter their physical properties under an external magnetic field [1,2]. These fluids primarily consist of magnetic nanoparticles dispersed in a liquid medium, forming a stable colloidal suspension. The unique responsiveness of magnetic fluids to external magnetic fields enables dynamic changes in their viscosity, electrical conductivity, magnetic susceptibility, and other key physical properties, thereby expanding their applicability across various scientific and industrial domains [3]. Typically, the magnetic nanoparticles in these fluids range in size from 10 to 50 nm, and their ability to undergo controlled modifications under an external magnetic field distinguishes them from conventional industrial fluids [4]. The exceptional tunability of their electrical and rheological properties makes magnetic fluids highly valuable for applications in automotive engineering, aerospace technology, biomedicine, sensors, and advanced cooling systems. The ability to manipulate their viscosity and conductivity through external magnetic stimuli is particularly advantageous in the design of next-generation functional materials [5]. The synthesis and characterization of magnetic fluids are crucial to advancing nanotechnology and materials science. Their adaptability to external stimuli and their integration into novel smart material systems pave the way for groundbreaking technological innovations. The physicochemical properties of magnetic fluids, including nanoparticle size, shape, dispersion, and interactions within the fluid medium, fundamentally influence their performance [6]. Therefore, comprehensive studies focusing on the electrical conductivity and magnetic behavior of these materials are essential for both scientific exploration and practical applications.

This study investigates the synthesis of magnetic fluids with varying nanoparticle concentrations and systematically analyzes their electrical and magnetic properties. Key factors such as nanoparticle classification, dispersion stability, and carrier fluid viscosity are considered, as each plays a significant role in determining the overall behavior of magnetic fluids. The interaction between magnetic fluid components and the correlation between their electronic and magnetic characteristics remains of substantial scientific interest, with implications for both fundamental research and technological innovation [7]. Despite extensive research in this field, a unified theoretical framework for understanding the structural, electrical, and magnetic properties of magnetic fluids, particularly those based on spinel ferrite nanoparticles such as Fe_3O_4, $CoFe_2O_4$, and $NiFe_2O_4$, has yet to be established [8]. Furthermore, the variations in their magneto-electronic properties remain insufficiently explored. In this study, we synthesized magnetic nanoparticles based on 3d transition metals (Fe, Co, Ni) and investigated their size, chemical composition, crystal structure, and the electrical conductivity and magnetic susceptibility of their corresponding magnetic fluids at room temperature. By systematically analyzing these properties, we aim to contribute to the understanding of their fundamental behavior and potential technological applications.

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METHODOLOGY

The preparation of magnetic fluids involved a two-step process: (I) the synthesis of colloidal magnetic nanoparticles and (II) their stabilization with a surfactant layer, followed by dispersion in water to obtain a homogeneous magnetic fluid. A low-concentration magnetic fluid containing magnetic nanoparticles was synthesized to ensure colloidal stability and optimal dispersion [9]. The chemical co-precipitation method was employed, as it is one of the most widely used and straightforward techniques for the synthesis of magnetic materials [10]. This method allows simultaneous precipitation of multiple metal ions, resulting in magnetic nanoparticles with uniform chemical composition.

Synthesis of Fe_3O_4, $CoFe_2O_4$, and $NiFe_2O_4$ Magnetic Nanoparticles

For the synthesis of Fe_3O_4, ferric chloride hexahydrate ($FeCl_3 \cdot 6H_2O$), ferrous sulfate heptahydrate ($FeSO_4 \cdot 7H_2O$), and sodium hydroxide (NaOH) were used, and the procedure was carried out as follows:

1. Preparation of precursor solution: A 1 M solution of the$FeSO_4 \cdot 7H_2O$ and a 2 M solution of the$FeCl_3 \cdot 6H_2O$ were prepared separately and dissolved in 200 mL of distilled water.
2. Heating and mixing: The solution was heated to 90°C and stirred vigorously to ensure uniform distribution.
3. Precipitation reaction: A 100 mL solution of 1 M NaOH was added dropwise to the mixture while stirring continuously using a magnetic stirrer. The pH of the solution was maintained at 9–10 to facilitate controlled precipitation.
4. Ageing process: The reaction mixture was further heated at 80 °C for 2 hours to enhance nanoparticle crystallization.
5. Magnetic separation: A small portion of the reaction mixture was extracted, diluted to twice its volume, and placed in a 0.2 T permanent magnetic field to separate the precipitated magnetic nanoparticles.
6. Drying process: The obtained precipitate was washed thoroughly, dried at room temperature for 48 hours, and collected for further characterization.

The synthesis of $NiFe_2O_4$ (Nickel ferrite) and $CoFe_2O_4$ (Cobalt ferrite) followed the same procedure, using appropriate metal salts as precursors. The chemical reaction equations for the synthesis are presented below:

a) $$3\,FeX^{2+} + 6\,FeX^{3+} + 24\,OHX^{-} \rightarrow 3\,Fe(OH)X_2 + 6\,Fe(OH)X_3 \overset{80X^{\circ}C}{\rightarrow} 4\,FeX_3OX_4 + 12\,HX_2O$$

b) $$CoX^{2+} + 2\,FeX^{3+} + 8\,OHX^{-} \rightarrow Co(OH)X_2 + 2\,Fe(OH)X_3 \overset{80X^{\circ}C}{\rightarrow} CoFeX_2OX_4 + 4\,HX_2O$$

c) $$NiX^{2+} + 2\,FeX^{3+} + 8\,OHX^{-} \rightarrow Ni(OH)X_2 + 2\,Fe(OH)X_3 \overset{80X^{\circ}C}{\rightarrow} NiFeX_2OX_4 + 4\,HX_2O$$

The synthesized magnetic nanoparticles were mixed with oleic acid ($C_{18}H_{34}O_2$) as a surfactant and distilled water as the liquid medium, followed by heating at 90°C for 1 hour to ensure uniform dispersion. Magnetic fluids with varying volumetric concentrations were prepared using Fe_3O_4, $CoFe_2O_4$, and $NiFe_2O_4$ magnetic nanoparticles. The volumetric concentrations of the samples were determined using the following formula:

$$\varphi = \frac{m_1/\rho_1}{(m_1/\rho_1) + (m_2/\rho_2) + (m_3/\rho_3)} \cdot 100\%$$

RESULTS AND DISCUSSION

We investigated the crystal structure of Fe_3O_4, $CoFe_2O_4$, and $NiFe_2O_4$ magnetic nanoparticles using X-ray diffraction (XRD) analysis. The synthesized nanoparticles were dried, finely powdered, and placed in a special sample holder. Monochromatic Cu Kα X-rays (λ = 1.5406 Å) were directed onto the sample surface at incident angles ranging from 10° to 80°.The diffracted rays were analyzed at angles satisfying the Wulf-Bragg condition, allowing us to determine the crystallographic structure of the nanoparticles. The relationship between the intensity of the diffracted X-rays and the angle of incidence on the nanoparticle surface is presented in Figure 1.

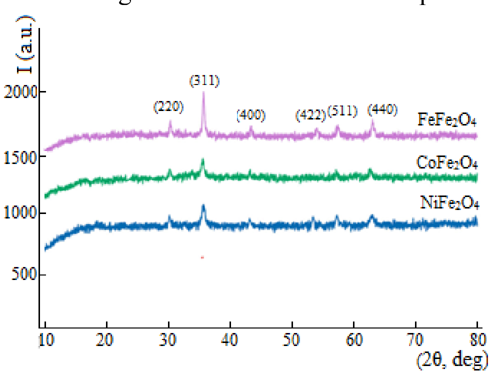

Figure 1. X-ray spectrum of magnetic nanoparticles MFe_2O_4 (M=Fe,Co, Ni)

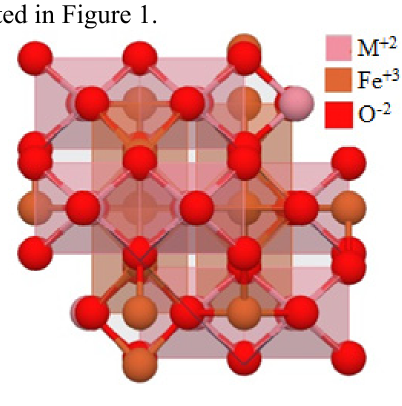

Figure 2. Crystal structure of (M=Fe,Co, Ni) MFe_2O_4 magnetic nanoparticles

The crystal structure of the synthesised magnetic nanoparticles was analysed based on the relationship between the X-ray diffraction (XRD) intensity and the angle of incidence. As shown in Figure 1, the intensity peaks in the XRD spectrum of the obtained Fe_3O_4, $CoFe_2O_4$, and $NiFe_2O_4$ nanoparticles correspond to the (220), (311), (400), (422), (511), and (400) planes. The diffracted X-ray spectrum confirms that all observed diffraction peaks are characteristic of a single-phase inverted cubic spinel structure, which [11]. Spinel ferrites follow the general chemical formula M Fe_2O_4, where M = Fe, Co, or Ni represents the divalent metal ion, while Fe^{3+} exists in the trivalent state. These materials exhibit remarkable physicochemical properties, making them highly relevant for various applications. As illustrated in Figure 2, MFe_2O_4 spinel ferrites comprise 64 tetrahedral and 32 octahedral crystal lattice sites within a single magnetic nanoparticle. The Fe^{3+} ions predominantly occupy octahedral sites, while the M^{2+} (Fe^{2+}, Co^{2+}, or Ni^{2+}) ions are positioned within tetrahedral sites. In these spinel structures (Figure 2), half of the Fe^{3+} ions are also located in tetrahedral sites, whereas the remaining half ones occupy octahedral sites [12]. The magnetic properties of these nanoparticles are primarily governed by the M^{2+} (Fe^{2+}, Co^{2+}, Ni^{2+}) ions. The average crystallite size of Fe_3O_4, $CoFe_2O_4$, and $NiFe_2O_4$ nanoparticles was estimated using the Debye-Scherrer equation $D = \frac{K\lambda}{\beta\cos(\theta)}$, based on the most intense (311) diffraction peak. The results of transmission electron microscopy (TEM)are presented in Figure 3.

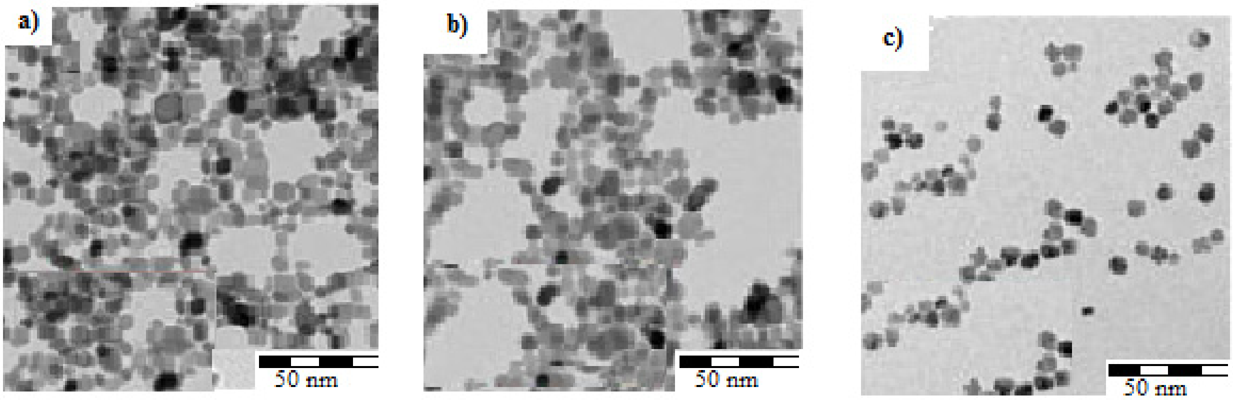

Figure 3. TEM images of magnetic nanoparticles a) Fe_3O_4,b) $CoFe_2O_4$ and c) $NiFe_2O_4$

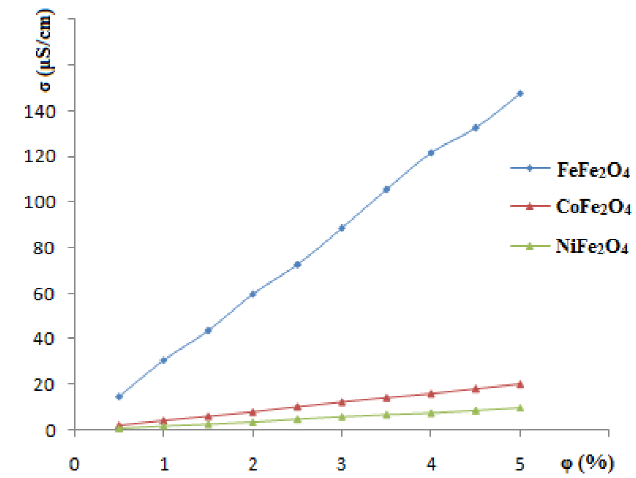

Figure 4. Concentration dependence of the electrical conductivity of magnetic fluids at room temperature

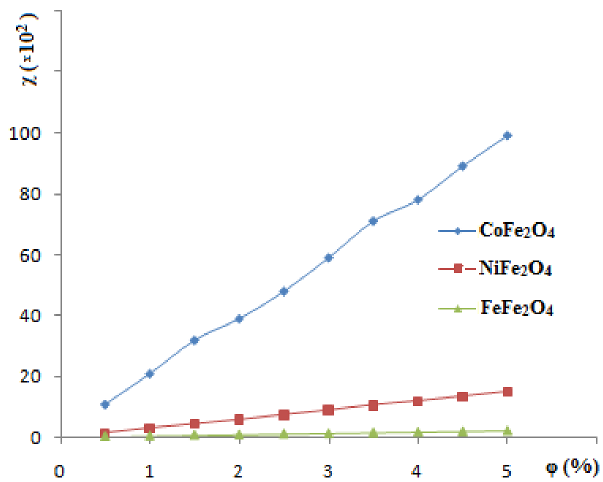

Figure 5. Concentration dependence of the magnetic susceptibility of magnetic liquids at room temperature

The electrical conductivity and magnetic susceptibility of magnetic fluids based on Fe_3O_4, $CoFe_2O_4$, and $NiFe_2O_4$ nanoparticles were measured at room temperature (t = 25 °C) across various concentrations. The results are presented in Figures 4 and 5. The electrical conductivity of magnetic fluids is highly complex and depends on several factors, including their internal structure, the properties of the surfactant, the liquid base (distilled water), and the concentration of magnetic nanoparticles within the fluid.

Ferrites such as $CoFe_2O_4$, $NiFe_2O_4$, and Fe_3O_4 (Magnetite) exhibit intrinsically low electrical conductivity, as they are primarily dielectric materials. However, as the concentration of magnetic nanoparticles in these magnetic fluids increases, their electrical conductivity rises significantly. As shown in Figure 4, the electrical conductivity of the synthesized magnetic fluids at room temperature was as follows:

- For the Fe_3O_4-based magnetic fluid, the electrical conductivity increased linearly from 15.6 μS/cm to 147.8 μS/cm as the magnetic nanoparticle concentration increased from 0.5 % to 5 %.
- For the Co Fe_2O_4-based magnetic fluid, the electrical conductivity increased linearly from 2.1 μS/cm to 20.2 μS/cm within the same concentration range (0.5 % to 5 %).
- For the $NiFe_2O_4$-based magnetic fluid, the electrical conductivity increased linearly from 0.23 μS/cm to 10.4 μS/cm, also within the same concentration range (0.5 % to 5 %).

Ferrites such as $CoFe_2O_4$, $NiFe_2O_4$ and Fe_3O_4 are mainly ferrimagnetic materials in their properties. However, magnetic fluids based on magnetic nanoparticles are mainly paramagnetic. As the concentration of magnetic nanoparticles in these magnetic fluids increases, their magnetic susceptibility increases significantly. As can be seen from Figure 5, the magnetic susceptibility of the magnetic fluids at room temperature was as follows: for the $FeFe_2O_4$-based magnetic fluid, the magnetic susceptibility increased linearly from $0.2 \cdot 10^2$ to $1.9 \cdot 10^2$ with a magnetic nanoparticle concentration of 0.5 % to 5 %; For the $CoFe_2O_4$-based magnetic fluid, the magnetic susceptibility increased linearly from $11.1 \cdot 10^2$ to $98.7 \cdot 10^2$ with a magnetic nanoparticle concentration ranging from 0.5 % to 5 %; for the $NiFe_2O_4$-based magnetic fluid, the magnetic susceptibility increased linearly from $1.5 \cdot 10^2$ to $14.9 \cdot 10^2$ with a magnetic nanoparticle concentration ranging from 0.5 % to 5 %.

Table 1. General parameters of synthesized magnetic fluids

Magnetic fluid nanoparticle	Temperature (°C)	Concentration (%)	Density (g/cm^3)	Electrical conductivity (μS/cm)	Magnetic susceptibility (χ 10^2)
Fe_3O_4	25	0.5	1.09	15.6	0.2
		5	1.24	147.8	1.9
$CoFe_2O_4$	25	0.5	1.11	2.1	11.1
		5	1.27	20.2	98.7
$NiFe_2O_4$	25	0.5	1.15	0.23	1.5
		5	1.32	10.4	14.9

CONCLUSIONS

Magnetic nanoparticles with the general formula (M = Fe, Co, Ni) M Fe_2O_4 were successfully synthesized using the chemical precipitation method. X-ray diffraction (XRD) analysis showed that the dominant diffraction peak appeared at the (311) plane, confirming the formation of single-phase cubic spinel structures in all (M = Fe, Co, Ni) M Fe_2O_4 samples. These results agree well with previously reported findings in the literature. The particle sizes of the (M = Fe, Co, Ni) M Fe_2O_4 nanoparticles were determined using transmission electron microscopy (TEM) and calculated using the Debye–Scherrer equation $D = \frac{K\lambda}{\beta \cos(\theta)}$. The estimated diameters ranged from approximately 20-40 nm for Fe_3O_4, 12–27 nm for $CoFe_2O_4$, and 8–20 nm for $NiFe_2O_4$.The lowest electrical conductivity measured at room temperature was 0.23 μS/cm at a concentration of 0.5 % for the $NiFe_2O_4$-based magnetic fluid, whereas the highest value, 147.8 μS/cm, was recorded at a concentration of 5 % for the Fe_3O_4-based magnetic fluid. Likewise, the lowest magnetic susceptibility was 0.2×10^2 at 0.5 % concentration for the Fe_3O_4-based fluid, while the highest value, 98.7×10^2, was obtained at 5 % concentration for the $CoFe_2O_4$-based fluid. These results demonstrate that both electrical conductivity and magnetic susceptibility vary significantly with increasing concentration, which is an important observation for the design of smart materials and highlights their potential use in advanced manufacturing technologies.

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ВИРОБНИЦТВО ФЕРИТІВ НА ОСНОВІ МАГНІТНИХ НАНОЧАСТИНОК ТА КОМПЛЕКСНА ХАРАКТЕРИЗАЦІЯ ЇХ ЕЛЕКТРИЧНИХ ТА МАГНІТНИХ ВЛАСТИВОСТЕЙ У КОЛОЇДНИХ СИСТЕМАХ

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У цьому дослідженні досліджується синтез, структурна характеристика та фізико-хімічні властивості магнітних наночастинок, отриманих з металів групи заліза (Fe, Co, Ni) та їхніх відповідних феритових дисперсій. Магнітні наночастинки були синтезовані методом хімічної конденсації, а їх морфологія та структура були проаналізовані за допомогою просвічувальної електронної мікроскопії (TEM) та рентгенівської дифракції (XRD). Синтезовані магнітні наночастинки, що містять ферити Fe_3O_4, $CoFe_2O_4$ та $NiFe_2O_4$, демонстрували нанорозміри в діапазоні від 10 до 50 нм. Точна кореляція між вимірюваннями TEM та XRD підтвердила структурні та розмірні характеристики синтезованих наночастинок. Були підготовлені магнітні рідини на водній основі з різною концентрацією наночастинок, що дозволило систематично досліджувати їхню електропровідність та магнітну сприйнятливість за кімнатної температури. Експериментальні результати дають критичне розуміння фундаментальних властивостей колоїдних систем на основі магнітних наночастинок, що потенційно сприяє передовим застосуванням у матеріалознавстві, магнітній приладобудуванні та нових технологічних галузях. Методологічний підхід та результати, представлені тут, сприяють розширенню розуміння поведінки та характеристик магнітної рідини.

Ключові слова: *магнітні наночастинки; феритові дисперсії; колоїдні системи; магнітна рідина; наноструктуровані матеріали; електропровідність; магнітна сприйнятливість*