

EXPERIMENTAL INVESTIGATION ON PROPERTIES OF NICKEL FERRITE WITH RESPECT TO MAGNETISM

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ABSTRACT

Nanocrystalline ferrites have piqued the interest of researchers in the realm of electrical technology due to their outstanding property of "ferrimagnetism." Ceramics made of ferrite have a wider temperature range of -40 to +225°C and higher specific resistivity and dielectric constant values than metals. According to a new study, nickel ferrite has magnetic and electrical conductivity (NiFe₂O₄). A technique for generating nickel ferrite nanoparticles in a system of direct sodium dodecyl sulphate micelles utilising ion flotation was proposed in this work. The size distribution of the produced nanoparticles has a maximum in the region of 4 to 6 nm, according to transmission electron microscopy data, and the ferrite is X-ray amorphous. The blocking temperature of the nanoparticles produced is 25 K. The ferrite is superparamagnetic, with a particular saturation magnetization.

Keywords: Magnet, Ion, Particles, Ferrite, Nano.

I. INTRODUCTION

Due to their excellent magnetic characteristics, magnetic nanoparticles of ferrite have gained prominence in the fields of medicine, the environment, magnetic recording media, sensors, and catalysts. Magnetic nanoparticles display superparamagnetic behaviour and quantum phenomena that can be used in a variety of applications. Because of their superior magnetic and electrical capabilities, spinel ferrites are particularly helpful in a variety of technical devices such as switching circuits, high density magnetic storage, microwave-based instruments, magnetic fluids, gas sensors, catalysts, water purification, and so on. A spinel ferrite is often defined as (MFe₂O₄), where M is a divalent metal ion such as Co, Ni, Zn, Mg, Cu Mn, and so on. Spinel ferrite has two interstitial sites: tetrahedral (A) and octahedral (B). Cations of varying valence and type can accept at these sites, resulting in a wide range of structural and magnetic characteristics. Spinel ferrites are characterised as normal, inverse, or random ferrites based on the distribution of cations at tetrahedral A and octahedral B sites.

The structural, electrical, and magnetic characteristics of ferrites are known to be very sensitive to

preparative conditions, composition quantity and type, magnetic interactions, and cation distribution at tetrahedral (A) and octahedral [B] sites. The cation distribution at tetrahedral and octahedral sites is affected by the ionic radii of the Fe/Co and M ions, the type of bonding, and the technique of synthesis. Nickel ferrite is a soft magnetic material with modest coercivity and saturation magnetization among the spinel ferrites. It has an inverted spinel structure. A number of scholars have explored nickel ferrite with various substitutions of divalent cations in the literature. Despite the fact that several publications on the magnetic, electric, and dielectric characteristics of nickel ferrites are available, work on the systematic analysis of structural and magnetic properties of nickel ferrites generated by sol-gel auto combustion technique is still absent. It is worth noting that the sol-gel auto-combustion approach has distinct benefits over other wet chemical processes. The process is simple, inexpensive, and yields uniform tiny particles of nanoscale diameters. As a result, in this paper, we sought to explore the structural and magnetic characteristics of nickel ferrite generated using the sol-gel auto combustion approach.

II. METHODS AND MATERIALS

The following approach was used to produce nickel ferrite in this study: From water and ethanol, sodium dodecyl sulphate (SDS) (Shostka Chemical Reagents Plant) was recrystallized. The absence of a minimum in the surface tension isotherm of their aqueous solutions around the critical micelle concentration (CMC), 8×10^{-3} M, determined the purity of amphiphiles. The Wilhelmy plate method was used to measure surface tension with an accuracy of ± 0.1 mN/m. Reagent grade iron(II) sulphate ($\text{FeSO}_4 \cdot 6\text{H}_2\text{O}$) and nickel chloride ($\text{NiCl}_2 \cdot 6\text{H}_2\text{O}$) were used to make nickel ferrite nanoparticles. The organic phase utilised in flotation extraction was a 4: 1 combination of toluene and isoamyl alcohol (Fluka reagents), and the flotation extraction procedure was carried out in a specially built 2L reactor. Its bottom section had a ceramic distributor for air distribution by rotameter with the aid of a compressor. The air distributor was fastened in such a way that it may be removed in order to purify the column. The air supplied into the column was broken down into bubbles, which transferred SDS and metal ions on their surfaces to an extractant, which was located on the surface of an aqueous solution in the form of a distinct phase and was discharged from the top of the column through an access hole. Rubber tubes linked the compressor to the column. To manage the air flow rate and the column discharge flow rate, glass stop cocks were utilised.

Model solutions were used to assess the production of nickel ferrite nanoparticles through flotation extraction of precursors with SDS. The concentrations of NiCl_2 and FeSO_4 in solution were 1×10^{-4} M. SDS was added to 1500 mL of solution to achieve a concentration of 4×10^{-4} M. To avoid the production of micelles, which would have hindered the manufacture of nickel and iron dodecyl sulphates, the SDS was first dissolved in 5 mL of ethanol and then added to an aqueous solution. For 10 minutes, the metal ions drifted. As a result, the solution became colourless and the organic phase became green, suggesting that nickel ions had been transported from one phase to another. The extractant and precursor were isolated from the aqueous solution using the flotation extraction method. To remove the toluene and isoamyl alcohol, the solvent was distilled out and the residue was vacuum dried. The resulting powder was diluted in distilled water to the critical micelle concentration of the salts produced and treated with sodium hydroxide until the iron and nickel hydroxides were completely

precipitated. Because of its amphoteric character, nickel hydroxide was partially reduced, resulting in the creation of a "magnetic colloid" composed of X-ray amorphous nonstoichiometric nickel ferrite and iron oxide.

The precipitate was filtered out, and the resulting powder was air dried for several days before being dried in a drying chamber at 50° for three hours. This powder manufacturing method, which did not include calcination, was employed to maintain the amphiphiles that created a protective adlayer on the powder nanoparticles, preventing them from adhering together.

X-ray diffraction (XRD) patterns of the produced powder were acquired using a copper target X-ray tube on a DRON3M diffractometer. In addition, an XRD pattern was computed using a crystallographic database for a cubic lattice parameter of Fe_3O_4 $a = 0.838$ nm, with the Fe ions on the A site of the spinel structure substituted by nickel ions. The computed XRD pattern of such an inverse spinel structure differs little from that of conventional, orthorhombic NiFe_2O_4 , with a difficult-to-calculate cation distribution.

Transmission electron microscopy (TEM) using a JEOL JEM1011 with a 100 kV accelerating voltage was used to determine the form and size of the nanoparticles. Sonication was used to disperse the nanopowder in ethanol prior to particle size measurement. The resulting dispersion was put on a copper grid that had been coated with Formvar and carbon successively. Elemental analysis was performed using a Philips SEMS 515 scanning electron microscope with an EDAX ECON IV microanalysis system. For the elements present, the detection limit and greatest error of determination were 0.2 and 2.0 per cent, respectively. The microanalysis scan area was $1.0 \times 1.0 \times 5.0$ m.

We used a versatile cryogenic Highfield measuring device in our magnetic experiments. We investigated the magnetic susceptibility of the material after cooling in zero field (ZFC) or a low magnetic field (LFC) in addition to typical magnetization versus magnetic field experiments (FC). The sample was cooled to 4 K without the use of a magnetic field in the ZFC method, and measurements were taken in a static magnetic field. The temperature was then gradually increased, and the magnetization was measured. The sole difference between the FC and ZFC measurements was that the sample was cooled

in a non-zero magnetic field. At high temperatures, the FC and ZFC curves of magnetically inhomogeneous magnetic materials often agree but diverge below their blocking temperature, T_b . Their ZFC curves peak at T_b , but their FC curves often grow monotonically down to very low temperatures.

III. RESULTS AND DISCUSSION

The experimental elemental analysis results for the produced powder (table) revealed higher iron and oxygen contents than stoichiometric nickel ferrite (NiFe_2O_4). The powder obtained in this investigation has the following composition: $\text{NiFe}_2\text{O}_4 \cdot y\text{Fe}_2\text{O}_3 \cdot z\text{H}_2\text{O}$ ($\text{NiFe}_{4.6}\text{O}_{14.8}$); hence, the process described above produced nonstoichiometric nickel ferrite. Because of its amphoteric character, nickel hydroxide was partially removed during the synthesis process, resulting in this composition. In addition to iron and

nickel hydroxides, the produced powder most likely comprised the iron compounds $(\text{C}_{12}\text{H}_{25}\text{SO}_4)_3\text{Fe}$, $(\text{C}_{12}\text{H}_{25}\text{SO}_4)_2\text{Fe}(\text{OH})$, and $(\text{C}_{12}\text{H}_{25}\text{SO}_4)\text{Fe}(\text{OH})_2$. They were difficult to separate by filtering the precipitate and most likely stayed in the combination of nickel and iron hydroxides as well as the powder produced. This is demonstrated by the material's relatively high carbon concentration, which can only be attributed to residual SDS. This form of the compound is known as "magnetic soap."

In contrast to diffraction patterns of flawless crystals, the XRD pattern of the produced powder (Fig. 1) contained no sharp peaks. The elemental analysis and XRD data for the powder show that it was made up of X-ray amorphous, nonstoichiometric nickel ferrite and a surfactant. Heat treatment at 600°C resulted in the appearance of diffraction peaks from macrocrystalline nickel ferrite.

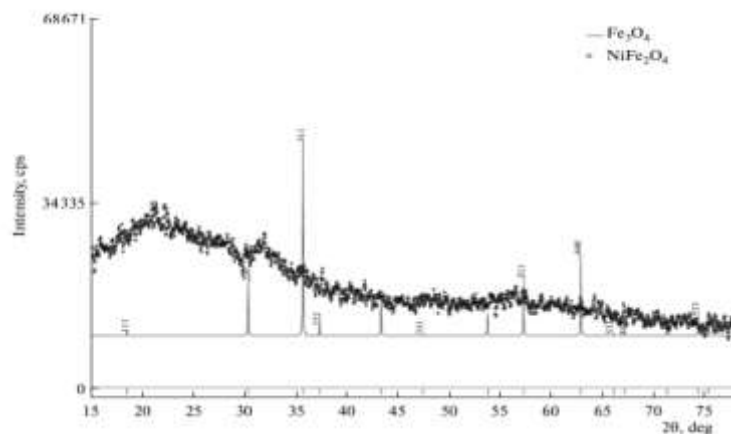


Figure 1. XRD pattern of the synthesized nickel ferrite in comparison with a calculated XRD pattern of Fe_3O_4 .

The inclusion of dodecyl sulphates and hydroxyl groups on the ferrite nanoparticles resulted in the creation of a strong adlayer. The nanoparticles of the nonstoichiometric

nickel ferrite produced varied in size from 2 to 6 nm (Fig. 2a). The particle size was enhanced after a 600°C heat treatment.(Fig. 2b)

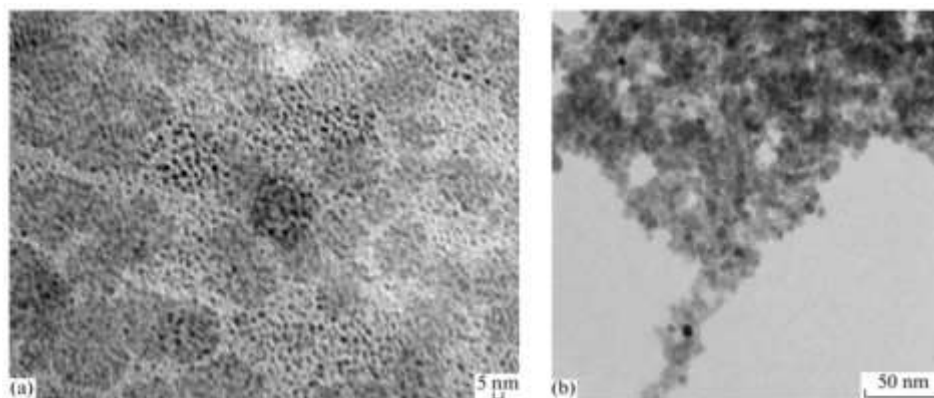


Figure 2: TEM micrographs of (a) as prepared and (b) heat-treated nickel ferrite nanoparticles.

Single domain nanoparticles with sizes ranging from 2 to 6 nm. The magnetic moments of all atoms in a domain's bulk are parallel to each other, whereas those on its surface are aligned at random. The information storage density will be 104 times greater if a bit of information is carried by a 60 μ m particle of synthetic nickel ferrite rather than a 60m domain in contemporary discs. To convey information, a nanoparticle's magnetization must be reversed by an applied positive or negative field (writing or erasing), and it must preserve its orientation in the absence of a

field (storage). Nanoparticles have such capabilities until they reach their blocking temperature. A critical field (coercive force) to be applied in the opposite direction to reverse the magnetization direction. The nanomaterials under consideration have a critical field as low as 0.1Tc (Fig. 3), which means that even modest magnetic fields surrounding us will lead such nanoparticles to "forget." The produced nickel ferrite nanoparticles have superparamagnetic characteristics at temperatures above $T_b = 25$ K (Fig. 3), which is typical of magnetic nanomaterials.

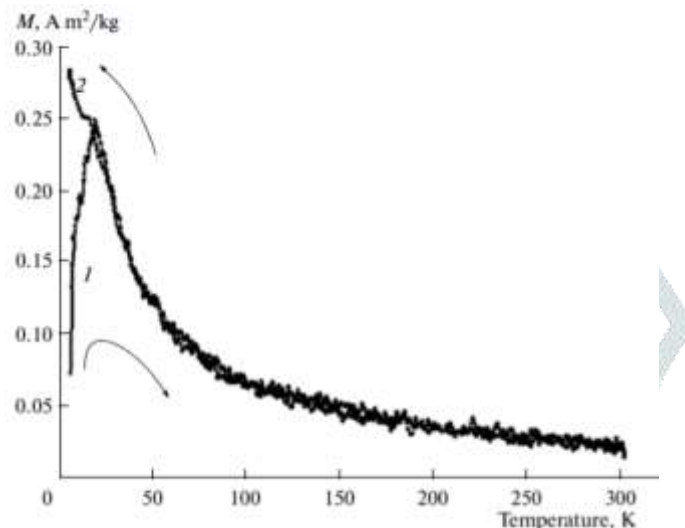


Figure 3. Temperature dependences of specific magnetization for the synthesized nickel ferrite in a magnetic field of 24 kA/m: (1) ZFC, (2) FC.

The blocking temperature of nanoparticles can be increased through self-organization. Magnetic dipole-dipole interactions in nanoparticles will therefore boost the system's internal energy. The blocking temperature will surpass that of an individual particle due to the rise in energy barrier height and will be provided by $T_b \sim (KV + Edip)/25kB$. It should be noted that this formula is for the T_b of a periodic colloidal structure on the substrate surface, not the T_b shown in Fig. 3. The particular saturation magnetization of a

magnetic nanomaterial is another essential property. At 5 K, the saturation magnetization of the produced ferrite nanoparticles is 15.0 A m²/kg (Fig. 4), which is somewhat lower than the saturation magnetization of bulk NiFe₂O₄ (17.6 A m²/kg). Magnetite shows similar differences: 50.3 or 60.1 A m²/kg against 92.0 A m²/kg; that is, the particular saturation magnetization of the nanoparticles in our instance is lower than that of bulk magnetic materials.

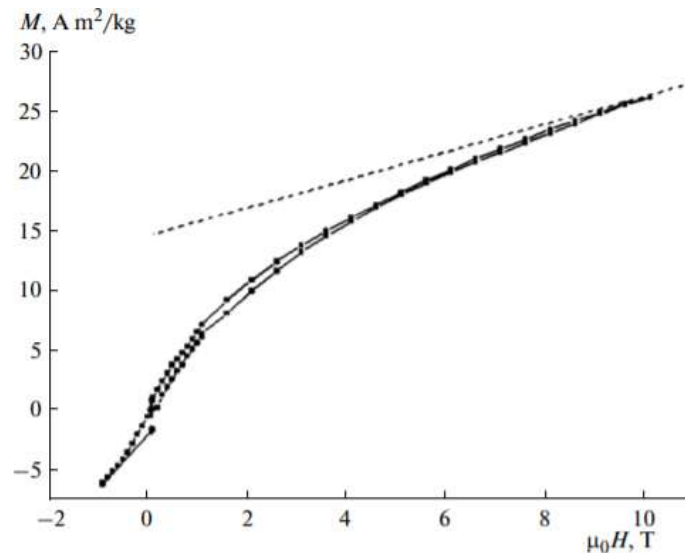


Figure 4. Magnetic field dependence of specific magnetization for the synthesized nickel ferrite at 5 K

IV. CONCLUSIONS

Ion flotation was used to create nanoparticles of nonstoichiometric nickel ferrite with the composition $x\text{NiFe}_2\text{O}_4 \cdot y\text{Fe}_2\text{O}_3 \cdot z\text{H}_2\text{O}$ in a system of direct sodium dodecyl sulphate micelles. The technique may be used to recycle old magnets. In contrast to diffraction patterns of flawless crystals, the XRD pattern of the material generated by this approach has no strong reflections, but heat treatment causes significant reflections, which are typical of bulk nickel ferrite. The size distribution of the produced nanoparticles is maximal in the 4 to 6 nm range, and they can form periodic island formations. The produced nanopowder has a blocking temperature of roughly 25 K and superparamagnetic characteristics, with a particular saturation magnetization of 15 A m²/kg at 5 K.

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