

***VARIOUS FACTORS AFFECTING THE STRUCTURAL  
AND MAGNETIC PROPERTIES OF SOME FERRITE  
NANOSTRUCTURES***

**MUKESH KUMARI**



**DEPARTMENT OF PHYSICS  
INDIAN INSTITUTE OF TECHNOLOGY DELHI**

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NANOSTRUCTURES***

*by*

**MUKESH KUMARI**

**Department of Physics**

*Submitted*

*in fulfillment of the requirements of the degree of Doctor of Philosophy*

*to the*



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## *Dedication*

*I would like to dedicate my thesis*

*To my beloved grand Parents*

*Late MR. Hoshiyar Singh and Late Mrs. Sahabkaur  
and Parents Mr. Jai Singh and Mrs. Shanti Devi*

# *Certificate*

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This is to certify that the thesis entitled "*Various factors affecting the structural and magnetic properties of some ferrite nanostructures,*" being submitted by *Mukesh Kumari* to the Department of Physics, *Indian Institute of Technology Delhi*, is worthy of consideration for the award of the degree of Doctor of Philosophy and is a record of the original bonafide research work carried out by her under my guidance and supervision and that the results contained in it has not been submitted in part or full to any other university or institute for the award of any degree/ diploma.

**Professor Mukesh Chander Bhatnagar**

Department of Physics

Indian Institute of Technology Delhi

Hauz Khas, New Delhi – 110016

India.

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**MUKESH KUMARI**

# *Abstract*

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The properties of the ferrites depend upon the crystal structure, which plays a crucial role in their versatile applications and can be influenced by various factors. The present thesis focuses on changes in properties and morphology by varying synthesis parameters like surfactant, sintering temperature, time, synthesis temperature, pH, reducing agent, and doping of rare-earth ions in ferrite nanoparticles, synthesized by sol–gel auto combustion method and hydrothermal method.

Cobalt ferrite (CFO) has been synthesized in the form of nanoparticles (NPs) through sol–gel auto combustion method. The prepared NPs of CFO were sintered for four hours at various temperatures from 300°C to 900°C. The average crystallite size of the NPs found to be increased from 28 nm to 59 nm with sintering temperature. The Magnetic behavior of CFO NPs has been tailored by varying the sintering temperature and strongly enhanced the range of application in spintronic and memory devices. The magnetic studies revealed that the saturation magnetization ( $M_s$ ) increases 63 emu/gm to 88 emu/gm, while the coercivity ( $H_c$ ) of nanoparticles decreases with the increase of sintering temperature. The wide range of coercivity of CFO NPs has been achieved from 934 Oe to 2237 Oe. Also, the high  $M_s$  value along with high coercivity has been reported. The effect of the surfactant cetyltrimethylammonium bromide (CTAB) on cobalt ferrite ( $\text{CoFe}_2\text{O}_4$ ) nanoparticles (NPs) using sol–gel auto–combustion method taking a different weight percent ratio of CTAB, i.e., 0%, 1%, 2%, 3%, and 4% with respect to metal nitrates also studied. Saturation magnetization and crystallite size were both found to be lowest in the case of a sample containing 2% CTAB.

Nickel ferrite nanoparticles (NFO) has been synthesized by the hydrothermal method. The NFO nanoparticles have been synthesized using  $\text{NaBH}_4$  as a reducing agent by the synthesis temperature vary like 80°C, 100°C, 120°C, 140°C, 160°C, 170°C, 200°C, 220°C, and 240°C, and the respective samples were named as N15, N1, N2, N3, N4, N5, N6, N7 and N8 (N-series).

The study is further extended and we synthesized NiFe<sub>2</sub>O<sub>4</sub> nanoparticles using 3M NaOH solution as a reducing agent with the variation in reaction temperatures 140°C, 160°C, 180°C, 200°C and 220°C, these prepared samples named as T1, T2, T3, T4 and T5 (T-series) respectively. Magnetic properties were measured at room temperature using a physical property measurement system (PPMS). The crystallite size of the nanoparticles calculated using XRD data was found to be less in the case of the reducing agent NaBH<sub>4</sub> (12–22 nm) compared to the samples prepared using NaOH (35–40 nm), and in increasing order in size from 12 nm to 22 nm with an increase in synthesis temperature. A regular trend in crystallite size and coercivity was also observed for the samples prepared using NaBH<sub>4</sub> as a reducing agent with temperature. It was found that nanoparticle behavior tends to be superparamagnetic from ferromagnetic as we increase the synthesis temperature. But no regular magnetic behavior has been shown by the nanoparticles prepared using NaOH as a reducing agent. As different properties are achieved with temperature as above by using two different reducing agents, further synthesis of NFO nanoparticles is done by using both NaOH and NaBH<sub>4</sub> and studying the variation in structural properties with variation in synthesis time and pH of the solution using the hydrothermal method.

We further go to study the effect of doping on NFO behavior. We study the effect of Pr doping on structural, morphological and magnetic properties of NFO. Praseodymium rare-earth ion (Pr<sup>3+</sup>) doped nickel ferrite (NiFe<sub>2-x</sub>Pr<sub>x</sub>O<sub>4</sub>, 0 < x < 0.1) nanocrystals (NFPO) has been synthesized. The saturation magnetization (M<sub>S</sub>) was found to be decreased and coercivity (H<sub>C</sub>) increases by adding rare earth ions in nickel ferrite nanoparticles. All samples show the ferromagnetic behavior at room temperature. Further, in order to improve the solubility of rare-earth ions in NFO, we study the effect of reducing agents and pH variation on doping. The series NiFe<sub>2-x</sub>RE<sub>x</sub>O<sub>4</sub> (RE= Pr and Gd, x= 0.1, 0.15 and 0.20) of samples has been also synthesized by hydrothermal method using NaBH<sub>4</sub> as a reducing agent and focus on the factors affecting the solubility of RE ions. The enhancement in solubility noted i.e.x=0.15 for Pr ions for reducing

agent  $\text{NaBH}_4$  as compared to  $x=0.025$  for  $\text{NaOH}$  as a reducing agent. The solubility of Pr ions is found to be higher than Gd ions. We bring into notice in our study that not only ionic radii of RE earth ions and method of preparation of doped ferrite affects the solubility limit of RE ions in nickel ferrite, but there are other factors also like reducing agent. Then we further study the effect of pH on the solubility limit of Pr ions. It is found that as we increase the pH, solubility has been decreasing for both the reducing agents.

## सार

फैराइट्स के गुण क्रिस्टल संरचना पर निर्भर करते हैं, जो उनके बहुमुखी अनुप्रयोगों के लिए महत्वपूर्ण भूमिका निभाता है और विभिन्न कारकों से प्रभावित हो सकता है। वर्तमान थीसिस में, सोल-जेल ऑटो दहन विधि और हाइड्रोथर्मल विधि द्वारा हम सर्फेक्टेंट, सिल्टरिंग तापमान, समय, संश्लेषण तापमान, पीएच, रिडयूसिंग एजेंट और संश्लेषित फैराइट्स नैनोकणों में रेयर-अर्थ आयनों के डोपिंग जैसे विभिन्न संश्लेषण मापदंडों द्वारा गुणों और आकारिकी में परिवर्तन पर ध्यान केंद्रित किया है।

कोबाल्ट फैराइट्स (सीएफओ) को सोल-जेल ऑटो दहन विधि के माध्यम से नैनोकणों (NPs) के रूप में संश्लेषित किया गया है। सीएफओ के तैयार NPs को 300°C से 900°C तक विभिन्न तापमानों पर चार घंटे के लिए सिल्टर्ड किया गया था। NPs के औसत क्रिस्टलाइट साइज को 28 nm से बढ़कर 59 nm तक सिल्टरिंग तापमान के साथ बढ़ा पाया गया। सीएफओ NPs के चुंबकीय व्यवहार को सिल्टरिंग तापमान में बदलाव करके, स्पिंट्रोनिक्स और मेमोरी उपकरणों में आवेदन की सीमा को दृढ़ता से बढ़ाया गया है। जबकि चुंबकीय अध्ययनों से पता चला है कि सैचुरेशन मैग्नेटाइजेशन ( $M_s$ ) 63 emu/gm से 88 emu/gm तक बढ़ जाता है, जबकि नैनोकणों की कोएर्सिंविटी ( $H_c$ ) सिल्टरिंग तापमान में वृद्धि के साथ घट जाती है। सीएफओ NPs की  $H_c$  की विस्तृत रेंज 934 Oe से 2237 Oe तक हासिल की गई है। इसके अलावा, उच्च  $M_s$  मूल्य उच्च  $H_c$  के साथ सूचित किया गया है। कोबाल्ट फैराइट्स ( $CoFe_2O_4$ ) नैनोकणों (NPs) पर सर्फेक्टेंट सीटैल ट्राई मिथाइल अमोनियम ब्रोमाइड (CTAB) का प्रभाव, CTAB का एक अलग वजन प्रतिशत अनुपात धातु नाइट्रेट के संबंध में लेते हुए यानी 0%, 1%, 2%, 3% और 4% सोल-जेल ऑटो-दहन विधि का उपयोग करके अध्ययन किया गया। 2% CTAB वाले नमूने के मामले में  $M_s$  और क्रिस्टलीय आकार दोनों सबसे कम पाए गए।

निकेल फ़ैराइट्स नैनोपार्टिकल्स (NFO) को हाइड्रोथर्मल विधि द्वारा संश्लेषित किया गया है। एनएफओ नैनोकणों को 80°C, 100°C, 120°C, 140°C, 160°C, 170°C, 200°C, 220°C, 220°C, और 240°C जैसे संश्लेषण तापमान द्वारा रिड्यूसिंग एजेंट के रूप में NaBH<sub>4</sub> का उपयोग करके संश्लेषित किया गया है, और संबंधित नमूनों को N15, N1, N2, N3, N4, N5, N6, N7 और N8 (N-श्रृंखला) के रूप में नामित किया गया था। अध्ययन को आगे बढ़ाया गया है और हमने प्रतिक्रिया तापमान 140 ° C, 160 ° C, 180 ° C, 200 ° C और 220 ° C में भिन्नता के साथ रिड्यूसिंग एजेंट के रूप में 3M NaOH का उपयोग करके NiFe<sub>2</sub>O<sub>4</sub> नैनोकणों को संश्लेषित किया है, इन तैयार नमूनों को क्रमशः T1, T2, T3, T4 और T5 (T-श्रृंखला) नाम दिया गया है। फिजिकल प्रॉपर्टी मेज़रमेंट सिस्टम (PPMS) का उपयोग करके रूम टेम्परेचर पर चुंबकीय गुणों को मापा गया। XRD डेटा का उपयोग करके गणना किए गए नैनोकणों के क्रिस्टलीय आकार को रिड्यूसिंग एजेंट NaBH<sub>4</sub> (12 to 22 nm) के मामले में NaOH (35 to 40 nm), का उपयोग करके तैयार किए गए नमूनों की तुलना में कम पाया गया, और संश्लेषण तापमान में वृद्धि के साथ आकार में 12 nm से 22 nm तक बढ़ते क्रम में पाया गया। तापमान के साथ रिड्यूसिंग एजेंट NaBH<sub>4</sub> का उपयोग करके तैयार किए गए सैम्पल्स के लिए क्रिस्टलीय आकार और H<sub>c</sub> में एक नियमित प्रवृत्ति भी देखी गई। यह पाया गया कि नैनोकणों का व्यवहार फेरोमैग्नेटिक से सुपरपैरामैग्नेटिक की ओर जाता है जैसे ही हम संश्लेषण तापमान बढ़ाते हैं। लेकिन रिड्यूसिंग एजेंट के रूप में NaOH का उपयोग करके तैयार किए गए नैनोकणों द्वारा कोई नियमित चुंबकीय व्यवहार नहीं दिखाया गया है। जैसा कि ऊपर चर्चा की गई है, दो अलग-अलग रिड्यूसिंग एजेंटों का उपयोग करके तापमान के साथ गुणों की विभिन्न विविधताएं प्राप्त की जाती हैं, NFO नैनोकणों का आगे संश्लेषण NaOH और NaBH<sub>4</sub> दोनों का उपयोग करके और हाइड्रोथर्मल विधि में सिंथेसिस टाइम और पीएच में भिन्नता के साथ संरचनात्मक गुणों में भिन्नता का अध्ययन किया जाता है।

हम आगे NFO के व्यवहार पर डोपिंग के प्रभाव का अध्ययन करने के लिए जाते हैं। हम एनएफओ के संरचनात्मक, रूपात्मक और चुंबकीय गुणों पर Pr डोपिंग के प्रभाव का अध्ययन करते हैं। प्रेसियोडीमियम रेयर-अर्थ आयन ( $Pr^{3+}$ ) डोपड निकल फ़ैराइट्स ( $NiFe_{2-x}Pr_xO_4$ ,  $0 < x < 0.1$ ) नैनोकृस्टल (NFPO) को संश्लेषित किया गया है। निकल फ़ैराइट्स नैनोकणों में रेयर-अर्थ आयनों की डोपिंग से सैचुरेशन मैग्नेटाइजेशन ( $M_s$ ) में कमी पाई गई और कोएरसीविटी ( $H_c$ ) बढ़ गई। सभी सैम्पल्स रूम टेम्परेचर पर फेरोमैग्नेटिक व्यवहार दिखाते हैं। इसके अलावा, एनएफओ में रेयर-अर्थ आयनों की घुलनशीलता में सुधार के लिए, हम डोपिंग पर रिड्यूसिंग एजेंटों और पीएच भिन्नता के प्रभाव का अध्ययन करते हैं। श्रृंखला  $NiFe_{2-x}RE_xO_4$  ( $RE = Pr$  and  $Gd$ ,  $x = 0.1, 0.15$  and  $0.20$ ) नमूनों को भी हाइड्रोथर्मल विधि द्वारा रिड्यूसिंग एजेंट के रूप में  $NaBH_4$  का उपयोग करके संश्लेषित किया गया है और RE आयनों की घुलनशीलता को प्रभावित करने वाले कारकों पर ध्यान केंद्रित किया गया है। रिड्यूसिंग एजेंट के रूप में  $NaOH$  के लिए  $x=0.025$  की तुलना में कम करने वाले एजेंट  $NaBH_4$  के लिए Pr आयनों के लिए घुलनशीलता में वृद्धि यानी  $x = 0.15$  नोट किया गया। Pr आयनों की विलेयता Gd आयनों से अधिक पाई जाती है। हमने देखा है कि न केवल RE आयनों की आयनिक रेडियस और डोपड फ़ैराइट्स की तैयारी की विधि निकल फ़ैराइट्स में RE आयनों की घुलनशीलता सीमा को प्रभावित करती है, बल्कि अन्य कारक भी हैं जैसे रिड्यूसिंग एजेंट भी घुलनशीलता में महत्वपूर्ण भूमिका निभाते हैं। फिर हम आगे Pr आयनों की विलेयता सीमा पर pH के प्रभाव का अध्ययन करते हैं। यह पाया गया है कि जैसे-जैसे हम पीएच बढ़ाते हैं, दोनों रिड्यूसिंग एजेंटों के लिए घुलनशीलता कम होती जा रही है।

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