

# Structural and Antibacterial studies of Cr substituted Nickel Ferrite nanoparticles

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**Abstract** — Ferrites are known for their significant properties in various fields. In this work, Chromium substituted nickel ferrites with general composition  $\text{NiCr}_x\text{Fe}_{2-x}\text{O}_4$  ( $x= 0, 0.25, 0.5, 0.75, 1$ ) were prepared using sol gel auto combustion method. The structural characterization of the samples was carried out using X-ray diffraction (XRD) which confirmed the presence of single-phasic spinel structure and energy dispersive X-ray (EDX) spectroscopy analysis confirmed the presence of all ions as per the stoichiometric ratios. Surface morphology of the samples has been investigated using Field Emission Scanning Electron Microscope (FESEM). The bandgap of the prepared samples was measured using UV-Visible-Diffuse Reflectance Spectroscopy (UV-Vis-DRS Spectroscopy). The antibacterial properties of the prepared compounds were tested against gram-positive and gram-negative bacterium and found that antibacterial property decreased with increase in concentration of Chromium for gram-positive bacteria.

**Keywords** - Chromium; nickel ferrite; antibacterial; sol-gel autocombustion; gram-positive.

## I. INTRODUCTION

Ferrites are materials having significant promise due to their appealing properties. Their rich physics and chemistry have attracted both scientists and engineers that made them extensively researched in diversified applications including drug delivery, microfluidics, magnetic resonance imaging, sensors, magnetic cooling and so on [1]. Among the ferrite nanoparticles, nickel ferrite is unique owing to their applications in catalysis, ferrofluidics, microwave and energy storage device, magnetic materials and biomedical fields [2].

Ferrites have been synthesized by various techniques such as sol-gel, hydrothermal, ball milling, chemical vapour synthesis, hydrothermal and combustion [2]. Among these sol-gel autocombustion is a suitable method for preparing nanoferrites in bulk scale. It is a low cost, single step technique

which require only simple equipment and low synthesis temperature. Here we synthesized Chromium substituted nickel ferrites ( $\text{NiCr}_x\text{Fe}_{2-x}\text{O}_4$  ( $x= 0, 0.25, 0.5, 0.75, 1$ ) using sol-gel autocombustion method. The structural, morphological, optical and antibacterial studies of the selected samples were carried out.

## II. EXPERIMENTAL

### A. Synthesis of chromium doped nickel ferrites

Chromium doped nickel ferrites ( $\text{NiCr}_x\text{Fe}_{2-x}\text{O}_4$ ,  $x= 0, 0.25, 0.5, 0.75$  and  $1$ ) were synthesized by sol-gel autocombustion method with precursors Iron (III) Nitrate nona hydrate ( $\text{Fe}(\text{NO}_3)_3 \cdot 9\text{H}_2\text{O}$ ), Nickel (II) Nitrate hexahydrate ( $\text{Ni}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$ ), Chromium Nitrate nona hydrate ( $\text{Cr}(\text{NO}_3)_3 \cdot 9\text{H}_2\text{O}$ ) and ethylene glycol. The metal nitrates were weighed appropriately and dissolved in ethylene glycol. The dissolved liquid like substance was sonicated in a sonicator at room temperature for the formation of gel. This homogeneous solution is then vaporized at  $150^\circ\text{C}$  on a hot plate till dry gel is formed. This dry gel is grounded using a mortar and pestle and is then sintered at  $800^\circ\text{C}$  for 3 hours to obtain the required samples.

## III. RESULTS AND DISCUSSIONS

### A. Structural analysis using XRD

The X-ray diffraction patterns of selected three samples with doping concentration ( $x= 0, 0.5, 1$ ) is shown in fig.1. The XRD patterns show high intensity peak at (311) proves the formation of pure single phasic spinel structure on comparison with standard ICDD. The XRD patterns are well indexed at the plane of reflections (220), (311), (811), (400), (422), (511) and (440). No substantial phase shift was produced by the partial substitution of the little amount of  $\text{Cr}^{3+}$  ion in the location of  $\text{Fe}^{3+}$  ion.

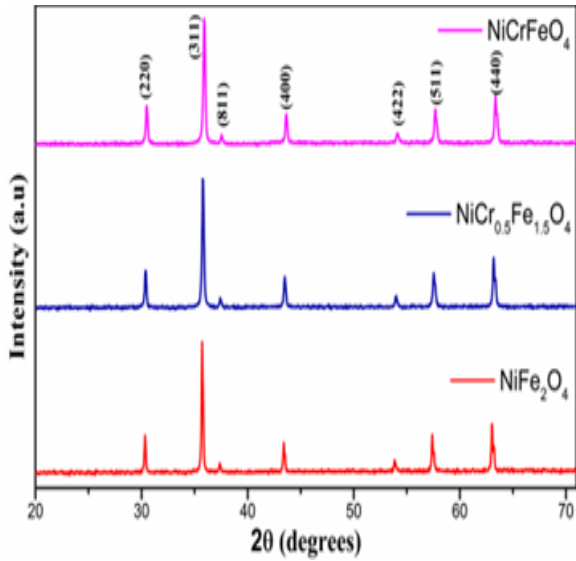


Fig.1. X-ray diffraction patterns of  $\text{NiCr}_x\text{Fe}_{2-x}\text{O}_4$  ( $x=0,0.5,1$ )

When chromium (Cr) is doped into the nickel ferrite lattice, it substitutes some of the Fe ions. The full width at half maximum is calculated from the XRD pattern and the crystallite size of the synthesized particles is found out using the Debye Scherrer equation [3]. The calculated average crystallite size of the samples ( $x=0, 0.5, 1$ ) is tabulated in table 1.

TABLE 1. CRYSTALLITE SIZE OF  $\text{NiCr}_x\text{Fe}_{2-x}\text{O}_4$  ( $x = 0, 0.5, 1$ )

Sample	Composition	Crystallite Size (nm)
1	$\text{NiCr}_{0.5}\text{Fe}_{1.5}\text{O}_4$	58.76
2	$\text{NiCrFe}_2\text{O}_4$	54.99
3	$\text{NiFe}_2\text{O}_4$	68.72

### B. Morphological studies using FESEM

The physical structure and morphology of selected samples were studied using FESEM. The images are shown below in figure 2 and it is clear that the particles are spherical in shape and agglomerated. The SEM results show that the grains are clearly visible in both samples, uniformly distributed and are indeed nanoparticles. Pores and voids may be due to the release of gases during synthesis of nanoparticles [4].

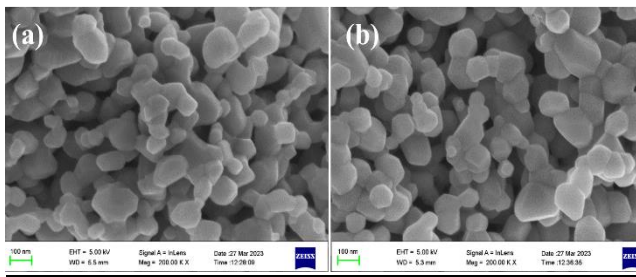


Fig. 2. FESEM image of a)  $\text{NiFe}_2\text{O}_4$  b)  $\text{NiCr}_{0.25}\text{Fe}_{1.75}\text{O}_4$

### C. Composition Analysis using EDX

The EDX curves for the two compounds ( $x= 0, 0.25$ ) are given below. The elemental mass composition found out

from the EDX analysis are tabulated for the given compounds at  $x=0$  and  $x=0.25$  in figures 3 and 4 respectively.

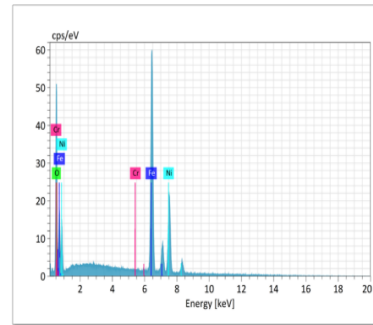


Fig.3. EDX curve and elemental composition of  $\text{NiFe}_2\text{O}_4$

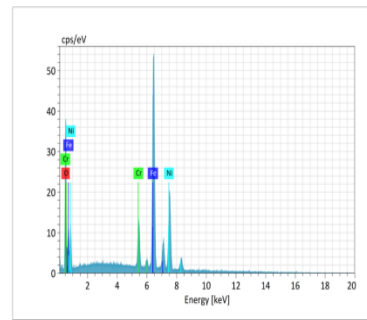


Fig.4. EDX curve and elemental composition of  $\text{NiCr}_{0.25}\text{Fe}_{1.75}\text{O}_4$

The spectra confirm the composition of constituent elements in their stoichiometric ratio. The mass composition observed in is good agreement with the stoichiometric ratio proving that the sample synthesized is indeed of good nature.

### D. Optical Studies

The optical characteristics of all five compounds were observed using UV-Visible- DRS spectroscopy. The optical bandgap of the compounds was calculated with the help of the reflectance spectroscopy by measuring the absorption coefficient  $\alpha$  using Kubelka Munk formula and plotting Tauc plot of all the compounds as shown in figure 5. The band gap of the compounds is found and it seems to be decreasing on increasing doping concentrations.

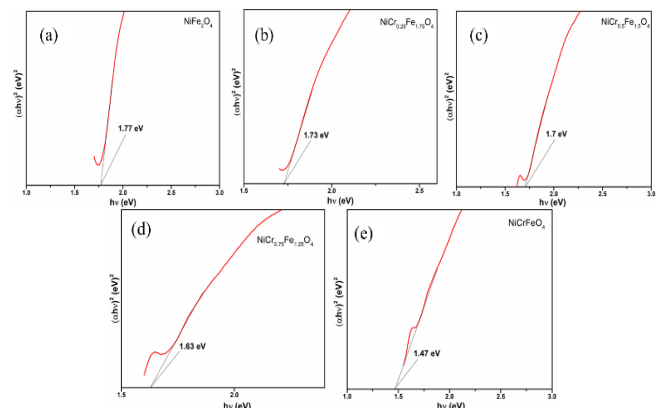


Fig.5. Tauc plot of  $\text{NiCr}_x\text{Fe}_{2-x}\text{O}_4$  a)  $x=0$  b)  $x=0.25$  c)  $x=0.5$  d)  $x=0.75$  e)  $x=1$

The band gap found using this method is tabulated in table 2. The table suggest that as concentration of Cr doping increases the value of band gap decreases.

TABLE 2. BANDGAP OF  $\text{NiCr}_x\text{Fe}_{2-x}\text{O}_4$  ( $x = 0, 0.25, 0.5, 0.75, 1$ )

Value of x	Compound	Band Gap (eV)
0	$\text{NiFe}_2\text{O}_4$	1.77
0.25	$\text{NiCr}_{0.25}\text{Fe}_{1.75}\text{O}_4$	1.73
0.5	$\text{NiCr}_{0.5}\text{Fe}_{1.5}\text{O}_4$	1.7
0.75	$\text{NiCr}_{0.75}\text{Fe}_{1.25}\text{O}_4$	1.63
1	$\text{NiCrFeO}_4$	1.47

### E. Antibacterial activity of chromium doped nickel ferrites

Nickel ferrites ( $\text{NiFe}_2\text{O}_4$ ) are known to exhibit significant antibacterial properties against a wide range of bacteria, making them potential candidates for various biomedical applications [5]. The antibacterial properties of nickel ferrites are mainly attributed to their unique physical and chemical properties, such as increased surface area, high reactivity, and magnetic properties. The synthesized nanoparticles (selected samples) were tested for antibacterial activity using disk diffusion method [6]. Bacterial culture of *Staphylococcus aureus* (gram positive) and *Escherichia coli* (gram negative) was used for the analysis. The tested bacteria were cultured in a sterile nutrient broth and allowed to grow in an incubator at 37°C. The inoculums were then used for antibacterial study. The synthesized nanoparticles suspended in chemicals were introduced to study their activity on both the gram positive and gram-negative bacteria.

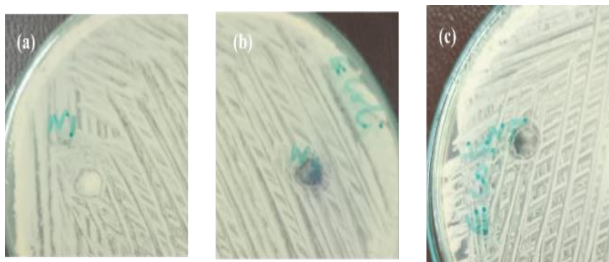


Fig.6. Activity of a)  $\text{NiFe}_2\text{O}_4$  b)  $\text{NiCr}_{0.5}\text{Fe}_{1.5}\text{O}_4$  and c)  $\text{NiCrFeO}_4$  against *Escherichia Coli*



Fig.7. Activity of a)  $\text{NiFe}_2\text{O}_4$  b)  $\text{NiCr}_{0.5}\text{Fe}_{1.5}\text{O}_4$  and c)  $\text{NiCrFeO}_4$  against *S-aureus*

Figures 6 and 7 show the activities of selected samples ( $x = 0, 0.5, 1$ ) against gram-positive and gram-negative bacteria. We also used a standard antibiotic along with the sample for comparison. On observations wells were formed

of different diameters for different doping concentrations, the results of the doping and its effects in diameter for the different bacteria is tabulated in table 3.

TABLE 3. ANTIBACTERIAL ACTIVITY RESULTS OF  $\text{NiCr}_x\text{Fe}_{2-x}\text{O}_4$  ( $x = 0, 0.5, 1$ )

Compound used	Activity on <i>Escherichia coli</i>	Activity on <i>S. aureus</i>
	Diameter of the well (In Millimeters)	Diameter of the well (In Millimeters)
$\text{NiFe}_2\text{O}_4$	6	30
$\text{NiCr}_{0.5}\text{Fe}_{1.5}\text{O}_4$	6	20
$\text{NiCrFeO}_4$	6	12
Standard Antibiotic (for comparison)	32	45

The results show that chromium doped nickel ferrites have noticeable antibacterial properties against *S-aureus* [7]. In gram negative bacteria (*E-coli*) the diameter remains same in all doping concentrations that is the antibiotic property of the ferrite is unaffected by the doping concentration. Even though it still has certain antibacterial activity. But in the case of gram-positive bacteria (*S-aureus*) the antibacterial activity decreases as doping concentration of chromium is increased. This suggests that the primary cause for antibacterial activity is the presence of ferrites, as the amount of iron decreases so does antibacterial property against gram positive bacteria. Hence doping of chromium in nickel ferrites can be used to control the antibacterial activity in gram positive bacteria.

### IV. CONCLUSION

Chromium doped nickel ferrites ( $\text{NiCr}_x\text{Fe}_{2-x}\text{O}_4$ ,  $x = 0, 0.25, 0.5, 0.75$  and 1) with different doping concentrations were prepared using sol gel auto combustion method. Characterization of the prepared particles were done using XRD, UV-Vis-DRS Spectroscopy and FESEM. The structural characterization using XRD revealed that the synthesized particles have a single phasic spinel structure. The crystallite size of the nanoparticles decreased as the doping concentration is increased. The SEM micrograph indicated that the grains in the sample are homogeneous, agglomerated and spherical in size. The energy dispersive X-ray (EDX) spectroscopy confirms the compositions of the elements in the prepared sample as per the stoichiometry ratio. The tauc plot shows a decrease in energy bandgap due to increase in doping concentration. The synthesized compounds were tested against both gram positive and gram negative bacteria. The compounds showed antibacterial properties in both the medium. The effect of doping didn't affect the gram negative bacteria but showed a decrease in activity in gram positive bacteria as doping concentration was increased. This is due to the decrease in Fe ions in the compounds. Thus, suitable modification of this work may bring remarkable advancements in the biomedical field.

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