PREFACE

Ferrites are an important class of magnetic materials that have many technological applications in magnetic, electronic and microwave devices. It can even be said that ferrites find more applications than the ferromagnetic materials. Among ferrites nickel zinc ferrite is a very important material which has many applications in both low and high frequency devices. They are characterized by high resistivity, low dielectric loss, mechanical hardness, relatively high Curie temperature and chemical stability. They find many applications as microwave devices like isolators, circulators, power transformer cores in electronic instruments, antenna elements, read-write heads in magnetic storage devices, etc.

Recently there has been a lot of interest in preparing these materials in nanoform using soft chemical approaches since they exhibit unusual properties in nanoform. The properties of these materials in nanoform make them suitable for new and improved device applications. The study of these materials also gives new insights into the physical properties of these materials in nanoform.

The present thesis describes the results obtained in the studies on \(\text{Ni}_x\text{Zn}_{1-x}\text{Fe}_2\text{O}_4\) \((x = 0.3, 0.4, 0.5)\) nanoferrites prepared by co-precipitation method. The ferrite compositions were annealed at three different temperatures 200°C, 500°C and 800°C in order to increase the particle size. Studies were carried out using X-ray diffraction, scanning electron microscopy (SEM), field emission scanning electron microscopy (FE-SEM) for structural characterization. Further structural characterization was carried out using FTIR spectroscopy in the range of 200 cm\(^{-1}\) to 400 cm\(^{-1}\). Vibrating sample magnetometer was used for magnetization measurements. Parameters corresponding to saturation magnetization \((\overline{M_r})\), coercive field \((\overline{H_c})\) and remanent magnetization \((\overline{M_r})\) were obtained. Field cooling (FC) and zero field
cooling (ZFC) experiments were carried out in order to obtain the blocking temperature \((T_B)\). Mössbauer studies were carried out in order to obtain the hyperfine field at \(\text{Fe}^{3+}\) ions in tetrahedral (A) and octahedral (B) sites. Superparamagnetic behaviour of the nanoparticles was studied as a function of composition and annealing temperature (grain size).

The thesis is divided into seven chapters.

Chapter I gives brief introduction to magnetism and the physical properties of ferrites. This chapter also gives an objective of the present studies.

Chapter II gives brief introduction to the experimental techniques like the method preparation of the ferrite samples and other experimental techniques used to study them. The materials were prepared by using co-precipitation technique using a pH value of 11 for NaOH which is the precipitating agent. The other experimental details employed in the present studies are described in this chapter.

Chapter III describes the results obtained in the X-ray diffraction studies. This chapter describes the estimation of average crystallite size, average lattice strain, the radii of tetrahedral and octahedral sites, bond lengths of tetrahedral and octahedral sites. The oxygen positional parameters were also calculated. The theoretical lattice parameters were obtained and compared with experimental lattice parameters. The densities of the ferrite compositions were measured and these were compared with X-ray densities to obtain the porosity present in the present systems.
The SEM and FE-SEM micrographs recorded in the present studies are presented in this chapter and their results are discussed. The particle sizes calculated using FE-SEM are compared with those obtained from X-ray diffraction data.

Chapter IV describes the magnetization studies carried out using vibrating sample magnetometer at room temperature. The values of saturation magnetization, remanent magnetization and coercive field obtained are presented. These results are compared with those reported by earlier workers on nickel zinc ferrites prepared in nanoform using different methods including co-precipitation technique. The blocking temperature obtained by employing zero field cooling and field cooling techniques are also presented. The ferrite compositions exhibited superparamagnetic behaviour. An attempt was made to interpret the results using the concepts of random anisotropy model and coercivity theory of fine particles.

Chapter V describes the Mössbauer spectroscopic studies of the ferrites samples. Mössbauer parameters like isomer shift, hyperfine field, quadrupolar splitting etc., are presented. The isomer shift was used to identify the Fe$^{3+}$ ions in A and B sites. The quadrupolar doublet indicates the existence of superparamagnetism. The variation of these parameters as a function of composition and annealing temperature is discussed. The Mössbauer spectroscopic studies are complementary to FC and ZFC studies in understanding the superparamagnetic behaviour of these samples. The variation of hyperfine field parameters at A and B sites as a function of composition and annealing temperature are discussed. The observed isomer shifts rule out the presence of Fe$^{2+}$ in the present samples.

Chapter VI discusses the results obtained in FTIR spectroscopic studies. The four frequency bonds $\nu_1, \nu_2, \nu_3$ and $\nu_4$ corresponding to cubic spinel structure were observed. The $\nu_1$ and $\nu_2$ frequency bands corresponding to
Fe$^{3+}$ - O$^2-$ stretching in tetrahedral and octahedral sites are used to estimate the force constants. The variation of these frequency bands as a function of the ferrite composition and annealing temperature is used to obtain information regarding the change in Fe$^{3+}$ - O$^2-$ bond length in tetrahedral and octahedral sites as ferrite composition and annealing temperature are varied. The bands corresponding to hydroxyl group vibrational frequencies were observed and their significance was discussed.

Chapter VII gives the conclusions arrived in the present studies. The results obtained in the present studies are different from those reported so far in literature for nickel zinc ferrites prepared in nanoform by co-precipitation techniques or other wet chemical methods. This is attributed to the pH and other methods employed in the present studies. The present studies also indicate that the critical particle size below which single domain nature exists varies with the composition of the ferrites system. The ferrite systems in the present study exhibiting superparamagnetic behaviour are seems to be independent of anisotropy constant, but depends on the crystallite size irrespective of the composition.