CHAPTER-VI

SUMMARY AND SUGGESTIONS FOR FUTURE WORK

The work reported in this thesis addresses a systematic investigation of the nanostructured antiferromagnetic materials of nickel and cobalt oxides and their magnetic properties at room and low temperatures. The synthesized samples were characterized by various techniques such as TGDTA, XRD, FTIR, UV, PL, Raman, SEM, TEM, XPS and VSM. The finite size and surface plays vital role in improving the magnetic performance of these metal oxides due to uncompensated spins.

In the course of our investigations of magnetic properties of NiO nanostructures, we found out that the surfactant CTAB was more helpful to produce nanorod like structures with reduced dimensions due to the controlled growth under the direction of CTAB micelles which acts as a template in the solution resulted to enhance the performance of magnetic nature in NiO material.

In the present work, we have also investigated and compared the synthesis of CTAB assisted NiO nanostructures by microwave and hydrothermal methods. Significantly, microwave heating helps quick chemical reactions and facilitates the diffusion of the mass, and favours the stable growth of flower-like Ni(OH)\textsubscript{2} nanosheets when compared to hydrothermal heating process. Even though slightly better morphological nature has been found in hydrothermal method, uniform growth in microwave treated sample shows similar flower like nature with almost same magnetization value of 0.40–0.45 emu/g at room temperature magnetic measurements.

A comparative study of microwave and hydrothermal methods further triggered the optimization of microwave reaction method. In this way, the role of
various microwave powers (900 W, 600 W, 450 W and 300 W) was investigated for the controlled growth of nanostructured NiO samples with distinct functional, structural, optical, morphological and magnetic properties. Flake-like structure with high uniformity is obtained for 900 and 600 wattage synthesized NiO samples than samples synthesized at lower power with average crystallite size of all the samples were in the range of below 10 nm. This work concluded that higher power synthesized samples exhibit superparamagnetic behaviour at ambient and low temperature magnetic measurements.

For the study of the influence of transition metal ions (Co, Fe, Mg and Zn) on the structural, optical and magnetic properties of NiO nanostructures various techniques were employed including X-ray diffraction, photoluminescence and vibrating sample magnetometer. Dopants can segregate on the nanostructured metal oxide surfaces or they can be incorporated into the lattice, where the dopants can be substitutional or interstitial or both. The substitution of parts of Ni lattice of NiO by $M^{2+}$ (Co, Fe, Mg and Zn) ions was confirmed by XRD with reduction of crystallite size than undoped NiO. The ferromagnetic behavior observed in this work is an intrinsic characteristic of the sample and has been explained on the basis of bound magnetic polaron mechanism, where the ferromagnetic ordering arises due to indirect interaction between localized electrons of dopant metal ions mediated by defect like oxygen vacancy.

In general, doping of a rare earth metal into metal oxide semiconductors temporarily generates charge carriers. Among different rare earth metals, cerium has gained considerable interest in research due to its remarkable properties such as observation of cerium oxide shift between CeO$_2$ and Ce$_2$O$_3$ due to the formation of redox couple Ce$^{3+}$/Ce$^{4+}$ based on oxidizing and reducing conditions.
and it could easily form oxygen vacancies with relatively high mobility of bulk oxygen species. The incorporation of Ce into NiO led to the existence of defects in the lattice sites and the formation of new energy levels in the band gap. The presence of new emissions is related to the effect of Ce$^{3+}$ ions and metal interstitials. The decreasing size of the ferromagnetic loops is observed in this work is due to the effect of cerium ion impacts on size and surface of NiO and thus improves super paramagnetic nature of the samples.

In this thesis, the magnetic behaviour of spinel cobalt oxides was also investigated with different synthesis parameters such as surfactants, calcination temperatures, comparison of microwave and hydrothermal methods, transition metal (TM) and rare earth metal (RE) doped effects. The results obtained in this particular study shows improved magnetic performance of the material. Cubic phase of Co$_3$O$_4$ nanostructures was observed for all the samples from XRD pattern which revealed the formation of nanosized Co$_3$O$_4$ without altering the crystalline nature.

SEM analysis revealed the existence of hexagonal stacked plate for CTAB assisted and metal (Ni, Mg, Zn, Fe and Ce) incorporated Co$_3$O$_4$ and spherical like morphologies for citric acid added Co$_3$O$_4$. The agglomeration of morphological structures was controlled by the addition of dopants into Co$_3$O$_4$ lattice sites. FTIR and Raman analyses confirmed the vibration modes of Co$_3$O$_4$ nanostructures. It is concluded from the results that the addition of surfactants or incorporation of TM and RE metals is the reason for the size / surface modifications which is found to accountable for the enhancement of magnetic properties at room temperature.
FUTURE SCOPE

In the present work, nanostructured nickel and cobalt oxides have been prepared using the microwave reaction method and the effect of surfactants, microwave powers, transition and rare earth metals doping on structural, morphological, functional, optical and magnetic properties of the samples have been studied. In the same way there is a future scope by performing magnetic measurements of the bimetallic samples (AB₂O₄) as a function of synthesis parameters in order to observe changes in size and surface for the understanding of magnetic phenomena.

1. The spinel structure of AB₂O₄ with face centered cubic arrangement of oxygen ions with metal ions occupying half of the octahedral and one-eighth of the tetrahedral interstitial sites within the anion sublattice can be synthesized from microwave and hydrothermal reaction methods.

2. Microwave reaction can be used to prepare Perovskite magnetic material (R₁₋ₓAₓMnO₃), where R is a tri-valent rare earth cation and A is a divalent alkaline earth cation. It is not only known for their unique properties such as colossal magnetoresistance (CMR), phase separation, charge, and orbital ordering states but also for Magnetocaloric effect.

3. A wide variation of synthesis parameters can be adopted to prepare nanostructured magnetic material with various shapes and their physical properties of size, structure and morphology can be characterized from XRD, FTIR, Raman, PL, SEM and TEM. The magnetic investigations can be carried out using VSM and SQUID instruments for energy storage device fabrication.