CHAPTER 7

SUMMARY AND SUGGESTIONS FOR FUTURE WORK

The application of nanomaterials in electronics, health and medicine, energy and environment is the main focus of current research and magnetic nanomaterials play a vital role in the above said fields. Nanometer sized magnetic iron oxide particles show promising response for most of these applications. However, the utility of pure iron oxide nanoparticles are limited because of their chemically unstable nature. In order to protect the particles from oxidation and to prevent them from forming aggregates, it is necessary to coat the particles with another inert layer thereby forming composites or core/shell structure. The core/shell structure is capable of maintaining favorable magnetic properties of metal iron and prevents the nanoparticles from oxidation.

Bearing in mind this scenario, the present study is devoted to the synthesis of the most versatile magnetic iron oxide nanoparticles of hematite, maghemite and magnetite nanoparticles. There have been several surface coating materials used for stabilizing iron oxide nanoparticles. In this study, polymer poly ethylene glycol was used to prevent the particles from forming aggregates. Efforts have been taken to coat the nanoparticles with noble metals silver and gold. There are several methods of preparing the iron oxide nanoparticles using both top down and bottom up approaches. But these approaches are either expensive or involve toxic materials during the preparation of the sample. The present study underlines the commonly used approaches such as hydrothermal, co-precipitation and sonication methods, which are known for simplicity, high yield and better quality of final products along with cost effectiveness.
Iron oxide nanoparticles of hematite, maghemite and magnetite of diameter less than 20 nm were successfully synthesized by the hydrothermal method. Using the same precursors and same experimental conditions with the sample treated at different temperatures, the three distinct phases of iron oxides were achieved. The morphology was studied by FESEM and TEM and the magnetic properties were studied by VSM. The nanoparticles in all the three phases were monodisperse and mesoporous. The average crystallite size is calculated as 52 nm for $\alpha$-Fe$_2$O$_3$, 13.2 nm for $\gamma$-Fe$_2$O$_3$ and 14.3 nm for Fe$_3$O$_4$ nanoparticles from the XRD results. TEM results confirmed that $\gamma$-Fe$_2$O$_3$ and Fe$_3$O$_4$ have average particle size of 11 and 16 nm respectively. FESEM and TEM results depicted clearly the porous nature of $\alpha$-Fe$_2$O$_3$ nanoparticles. The BET studies further confirmed that all were of mesoporous nature. Out of the three nanoparticles, the $\gamma$-Fe$_2$O$_3$ nanoparticles have high surface area and mesoporosity and thus can be used in various biomedical applications. The $M_s$ value of $\gamma$-Fe$_2$O$_3$ and Fe$_3$O$_4$ nanoparticles were 58.56 and 40.3 emu/g respectively at room temperature. The VSM results confirmed the weakly ferromagnetic nature of the $\alpha$-Fe$_2$O$_3$. These $\alpha$-Fe$_2$O$_3$ nanoparticles have potential applications in gas sensors.

A cost effect of functionalization of the synthesized magnetite nanoparticles with the polymer PEG 20,000 of various concentrations was presented. The morphology was studied by XRD, FT-IR, FESEM and TEM. Their magnetic properties were also studied. It was found that there was a gradual decrease in the size of the particles when the concentration of the PEG 20,000 was increased. Fourier transform infrared spectroscopy studies explained the vibration modes of the synthesized Fe$_3$O$_4$ NPs and confirmed the presence of PEG 20,000 in the synthesized magnetite (Fe$_3$O$_4$) nanoparticles. FESEM studies revealed the structure and surface morphology of the synthesized magnetite nanoparticles. TEM images also confirmed that the particles size is below 20 nm and the PEG coated Fe$_3$O$_4$ NPS are well
dispersed than the bare ones. Vibrating sample magnetometer studies ascertained the magnetic properties of the synthesized Fe$_3$O$_4$ nanoparticles. The magnetization value was found to increase slightly for the PEG 20,000 coated nanoparticles when compared to the pure magnetite nanoparticles but it was less than that of the bulk nanoparticles. Thus, the surface functionalization of the as-synthesized magnetite nanoparticles with PEG 20,000 effectively controls the particle size and showed slightly enhanced magnetization values. The present study demonstrates the surface modification of bare Fe$_3$O$_4$ by PEG molecules which not only reduces the possibility of oxidation but also enhances the magnetic nature of the NPs significantly.

An efficient co-precipitation method followed by solvothermal approach was successfully developed for coating one of the noble metal (Ag) over Fe$_3$O$_4$ nanoparticles in two different ratios. These particles presented a (Fe$_3$O$_4$)$_{core}$/(Ag)$_{shell}$ structures. In this study, non-toxic and bio-friendly reducing agent glucose was used to coat the Fe$_3$O$_4$ nanoparticles. XRD and TEM results authenticated the nanosize of the particles and the formation of Fe$_3$O$_4$/Ag nanocomposites. The SEM and FESEM pictures along with the EDX spectrum confirmed the surface morphology and elemental composition of the as-prepared nanocomposites. The VSM studies further revealed that there is a significant change in the magnetic property for the Ag coated particles in comparison to that of the parent Fe$_3$O$_4$ nanoparticles. This combination of good magnetic properties with optical characteristic might be useful to be applied as a recyclable magnetic carrier with antimicrobial and catalytic properties.

Au/Fe$_3$O$_4$ nanocomposites were successfully synthesized using the simple and cost-effective co-precipitation followed by solvothermal method without using any additives or capping ligands. The XRD diffractogram indicated the presence of both Au and Fe$_3$O$_4$ nanoparticles in the
nanocomposites. The UV-Visible absorption study confirmed the red shift in the Au coated Fe$_3$O$_4$ nanocomposites and the mechanism for such shift has been explained. The SEM pictures illustrated the surface morphology and EDX spectrum confirmed the percentage of the composition of iron and gold in the nanocomposites. The TEM analysis confirmed the presence of gold nanoparticles of size around 5 nm and iron oxide nanoparticles of size around 8-15 nm in the nanocomposites. The magnetic properties of the Au coated Fe$_3$O$_4$ were measured and compared with the bare Fe$_3$O$_4$ and also on the basis of gold content in the composites. The saturation magnetization values for the Au/Fe$_3$O$_4$ nanocomposites prepared in two different ratios at room temperature were found to be 12.77 emu/g and 14.3673 emu/g respectively.

In the present work, the magnetite nanoparticles were coated by the noble metals silver and gold and it was found that Au coated IONPs have better optical property than Ag coated NPs. However, in this study only two samples from each noble metal were prepared. In future, a more systematic work can be carried out by making more variations in the concentrations of Au and Ag in the nanocomposites and investigate the modifications in the optical and magnetic properties of the nanocomposites as a function noble metal concentration. In this work only the Au and Ag noble metals were used, in future, efforts can be taken to coat IONPs with other noble metals such as platinum and palladium. The iron oxide nanoparticles studied in this thesis may be multifunctionalized for various biomedical applications, especially for targeted cancer imaging and therapeutics, gas sensing and water treatment. Core-shell nanocomposites have recently received considerable attention owning to their physical and chemical properties. Hence, efforts can be taken to form more perfect core-shell nanocomposites. Attempts can also be taken to study the application of these nanoparticles in the above said fields. Novel functional building blocks can be generated for applications in fields ranging from optoelectronics to information technology and also to healthcare.
Characterization techniques such as PXRD, FT-IR, BET, UV-Vis, SEM, EDX, FESEM, TEM and VSM have been used in the present investigation. In future, advanced techniques such as High Resolution Transmission Electron Microscopy (HRTEM), X-ray Photoelectron Spectroscopy (XPS), Mösbaur spectroscopy, Atomic Force Microscopy (AFM), Scanning Tunneling Microscopy (STM) and Wide-angle X-ray diffraction (WAXD) can be used to investigate the properties of the nanocomposites. The Particles Size Distribution (PSD) can be measured using particle size analyzer. Dielectric and thermal studies can also be done. Photo electric and conductive properties can also be investigated.