ABSTRACT

Magnetic iron oxide nanoparticles have drawn tremendous attraction from both fundamental aspect as well as applications in biomedicine such as magnetic bio-separation, detection of biological entities, magnetic resonance imaging, magnetic fluid hyperthermia and targeted drug delivery due to their fascinating magnetic properties. These properties of iron oxide nanoparticles can be tuned by modifying their phase, size, shape and surface coating of the nanoparticles. In the present thesis, synthesis of iron oxide nanoparticles with different morphology and their surface coating/functionalization were carried out. The structural, morphological, vibrational, magnetic and optical properties of these synthesized products were characterized by using X-ray diffraction (XRD), field emission scanning electron microscopy (FESEM), high-resolution transmission electron microscopy (HRTEM), microRaman spectroscopy, Fourier transform infrared spectroscopy (FTIR), vibrating sample magnetometer (VSM), ultraviolet-visible spectroscopy (UV-Vis) and photoluminescence spectroscopy (PL).

In the first part of the thesis deals with synthesis and characterization of iron oxide (hematite and magnetite) nanoparticles with different morphology. Hematite nanorods were synthesized by reverse micelles method using cetyltrimethyl ammonium bromide (CTAB) as a surfactant and calcined at 300 °C. Nanorods have hexagonal crystal structure with diameter of 30-50 nm and length 120-150 nm. A weak ferromagnetic behavior was observed with saturation magnetization ($M_s$) of 0.6 emu g$^{-1}$ and coercive force ($H_c$) of 25 Oe. Ferromagnetic $\alpha$-Fe$_2$O$_3$ nanoparticles were synthesized by gel evaporation method in air at 300 °C. The average size of the as synthesized $\alpha$-Fe$_2$O$_3$ nanoparticle was estimated to be 30 nm and the particles were of good crystalline nature. The ferromagnetic
properties of the nanoparticles are due to the existence of $\gamma$-Fe$_2$O$_3$ phase along with $\alpha$-Fe$_2$O$_3$. Hydrothermal method is an effective method to control the shape and crystalline quality of the nanoparticles than other methods. In this work, ferromagnetic single-crystalline rice shaped Fe$_3$O$_4$ nanoparticles were synthesized by a one-pot surfactant-assisted hydrothermal method using dodecyltrimethyl ammonium bromide (DTAB) as a surfactant. The as synthesized products have a regular rice-like shape with an average diameter of 150 nm and length of 500 nm and are monodisperse. Shape evolution from cauliflower-to rice-like morphology has been controlled by varying the DTAB concentration. The possible formation mechanism of nanorice is the selective adsorption of surfactant on crystallographic facet. Further, superparamagnetic Fe$_3$O$_4$ nanoflowers were synthesized by surfactant assisted hydrothermal method using tetraethylene tetramine (TETA) as a surfactant. The evolution of the morphology from polyhedron to flower was obtained by varying the amount of TETA.

Second part of the thesis focuses on surface coating and functionalization of iron oxide nanoparticles. In order to enhance the heating characteristic and stability of the Fe$_3$O$_4$ nanoparticles, bovine serum albumin (BSA) was coated by co-precipitation technique. The size of nanoparticles was 10 nm with good crystalline quality. The magnetic nanoparticles show the superparamagnetism with saturation magnetization of 33.7 emu g$^{-1}$. The maximum SAR of 96.5 W g$^{-1}$ was obtained at magnetic field amplitude of 3.5 kA m$^{-1}$ and frequency of 300 kHz. Polyethyleneimine (PEI) capped Fe$_3$O$_4$ nanoparticles were synthesized by precipitation of Fe$^{2+}$ and Fe$^{3+}$ in the presence of PEI. Nanoparticles show room temperature superparamagnetism with saturation magnetization of 34 emu g$^{-1}$ and negligible remanence or coercivity. The adsorption of the BSA with PEI capped Fe$_3$O$_4$ nanoparticles were investigated. The electrostatic interaction was found to
take place between negatively charged BSA and positively charged PEI capped Fe₃O₄ nanoparticles.

The self-assembled Fe₃O₄/Ag core/shell (Fe₃O₄@Ag) nanocomposites were synthesized using microemulsion method. The XRD pattern shows only cubic phase of Ag for Fe₃O₄@Ag nanocomposites. TEM results show the average size of nanoparticles to be 10-15 nm. These nanoparticles were self-assembled with a branch like morphology. The self-assembly process was formed due to the oriented aggregation of nanoparticles. The optical properties of Fe₃O₄ nanoparticles were modified by surface plasmon resonance (SPR) of silver shell. The saturation magnetization of superparamagnetic Fe₃O₄@Ag nanoparticles was 40 emu g⁻¹. Magnetic and fluorescence silica nanospheres were synthesized by attachment of Fe₃O₄ nanoparticles and fluorescent carbon nanodots onto a silica shell through electrostatic interaction. The obtained nanocomposites exhibit superparamagnetic and fluorescence behavior, which can be used for targeting and fluorescent imaging applications.