CHAPTER 1

INTRODUCTION TO MAGNETISM AND IRON OXIDE NANOPARTICLES

1.1 INTRODUCTION

Nanoparticles are made of inorganic or organic materials, which have many novel properties compared to the bulk materials [1]. Among these, iron oxide nanoparticles have unique magnetic properties such as superparamagnetism, high coercivity, low Curie temperature, high magnetic susceptibility, etc. In the last few decades, great efforts have been made on synthesis of iron oxide nanoparticles due to their broad range of applications like magnetic fluids, data storage, catalysis and bio-applications [2-7]. Currently, iron oxide nanoparticles are also used in important bio-applications such as magnetic bio-separation, detection of biological entities (cell, protein, nucleic acids, enzyme, bacteria, virus, etc.), magnetic resonance imaging (MRI), magnetic fluid hyperthermia (MFH) and targeted drug delivery. However, it is important to select the materials for the fabrication of nanostructure and devices with controllable physical and chemical properties. In the last few decades, much efforts have been made on investigations of several types of iron oxides nanoparticles including the Fe$_3$O$_4$ magnetite (ferrimagnetic, superparamagnetic when the size is less than 15 nm), α-Fe$_2$O$_3$ (hematite, weakly ferromagnetic or antiferromagnetic) and γ-Fe$_2$O$_3$ (maghemite, ferrimagnetic) among which magnetite and maghemite are the more promising and popular candidates.

Nevertheless, it is a challenge to control the phase, size, shape and stability of iron oxide nanoparticles. The magnetic property of the iron oxide nanoparticles depends upon shape and size of the particle. In one dimensional
(1D) iron oxide nanoparticles, ferro or ferrimagnetic properties arise due to their shape anisotropy. 1D iron oxide nanoparticles are routinely used for the storage of digital and analog signals in the area of advanced flexible media [8], whereas spherical iron oxide nanoparticles with small size have emerged as one of the primary nanomaterials for biomedical applications due to their superparamagnetic property. However, small size iron oxide nanoparticles tend to form agglomerates to reduce the energy associated with the high surface to volume ratio. Furthermore, iron oxide nanoparticles can be easily oxidized in air and resulting in the loss of magnetism. Therefore, the surface coating is essential to stabilize the magnetic iron oxide nanoparticles. These strategies comprise grafting or coating with organic molecules, including small organic molecules, polymers and biomolecules or coating with an inorganic layer (silica, metal and carbon). In many cases, the protecting shells not only stabilize the magnetic iron oxide nanoparticles but can also be used for further functionalization to use the biological applications.

1.2 INTRODUCTION TO MAGNETISM

Magnetism is a physical behavior of the magnetic materials which originates from electron orbital motion or intrinsic spin from the presence of unpaired electrons (Fig. 1.1). Iron and certain iron containing materials can have unpaired electrons necessary to show magnetic behavior. Due to the large number of electrons in materials, magnetic solids are more easily viewed as a collection of magnetic dipole moments. Generally, the magnitude of a magnetic dipole moment increases with the number of unpaired electrons and is given by

\[ \mu = -g_s \mu_0 \left( \frac{S}{\hbar} \right) \] ..........................(1.1)

\[ \mu_s = -g_s \mu_0 \sqrt{s(s+1)} \] ..........................(1.2)
where $s$ is the total spin quantum number from unpaired electrons, $g_s$ is the electron “g factor” predicted by quantum electrodynamics and $\mu_0$ and $\hbar$ are the Bohr magnetron and the Planck constant, respectively.

![Diagram](image)

**Figure 1.1** Demonstration of the magnetic moment associated with (a) an orbiting electron and (b) a spinning electron.

The potential energy $U$ and force $F$ on a magnetic dipole in a magnetic field are

\[
U = -\mu B 
\]

\[
F = -\nabla(-\mu B) = \nabla(\mu B)
\]

eq. 1.4 implies that magnetic dipoles are attracted to regions where the density of magnetic field lines is greater. In order to minimize energy according to eq. 1.3, dipoles close to each other will tend to line up in the same direction.
1.3 TYPES OF MAGNETISM

The magnetic behavior of materials depends on the structure of the material and particularly on its electron configuration. Magnetism in materials can be classified into several types viz. paramagnetism, ferromagnetism, superparamagnetism, antiferromagnetism and ferrimagnetism.

1.3.1 Paramagnetism

Paramagnetic materials possess a permanent magnetic moment due to unpaired electrons in partially filled orbital. In the absence of an external magnetic field, the orientations of these magnetic moments are random, such that a piece of material possesses no net magnetic moment. These magnetic moments are free to rotate and paramagnetism results when they preferentially align by rotation with an external field as shown in Fig. 1.2.

Figure 1.2 Atomic dipole configurations with and without an external magnetic field for a paramagnetic material.
Susceptibility for paramagnetic materials is found to be of the order of \( \sim 10^{-6} \text{ emu mol}^{-1} \text{ Oe}^{-1} \). The temperature dependence of the susceptibility for many paramagnetic materials follows the well known Curie law \( \chi = C/T \), where C is the Curie constant.

1.3.2 Ferro/Ferrimagnetisms

Ferromagnetic materials possess a permanent magnetic moment in the absence of an external field and manifest very large and permanent magnetizations. Permanent magnetic moments in ferromagnetic materials result from atomic magnetic moments due to the unpaired electrons in the atoms. Ferromagnetism is characterized by strong interactions between the magnetic moments of adjacent atoms, the interactions causing the alignment of the magnetic moments. These coupling interactions cause net spin magnetic moments of adjacent atoms to align with one another even in the absence of an external field (Fig. 1.3). These materials have large positive susceptibility (up to \( 10^6 \text{ emu mol}^{-1} \text{ Oe}^{-1} \)).

![Diagram of magnetic moments aligning without an external field](image)

**Figure 1.3** Schematic illustration of the mutual alignment of atomic dipoles for a ferromagnetic material, which will exist even in the absence of an external magnetic field.
Ferrimagnetic materials consist of antiparallel alignment of the magnetic moments, yet the material maintains a net magnetization. Ferrimagnets have high susceptibility (up to $10^6$ emu mol$^{-1}$ Oe$^{-1}$) and net magnetic moments even in the absence of an applied field, much the same as ferromagnets. At sufficiently high temperatures, both ferromagnetic and ferrimagnetic materials exhibit the paramagnetic behavior (at the Curie temperature).

1.3.3 Superparamagnetism

Ferromagnetic and ferrimagnetic materials can change their magnetic behavior if they are produced in a fine powder form, so that the grain size reaches a critical value. When the critical value is achieved, the thermal vibration energy of each particle has a magnitude comparable to the magnetic energy and thus even though magnetic moments are prone to line up in the field direction, the thermal vibration causes the magnetic moments to change its direction randomly. Therefore, there is no net magnetic response and the material behaves as though it is a paramagnetic material [9].

1.4 **THE BASIC PARAMETERS FOR MAGNETIC MEASUREMENTS**

If a magnetic material is placed in a magnetic field $H$, the individual atomic moments in the material contribute to induce the magnetic flux inside the materials

$$B = \mu_0(H + M)$$

where $\mu_0$ is the vacuum permeability ($12.566 \times 10^{-7}$ V s A$^{-1}$ m$^{-1}$) and the magnetization $M = m/V$ is the magnetic moment per unit volume, where $m$ is the magnetic moment on a volume $V$ of the material. In the regime, where the
magnetization scales linearly with $H$, it is useful to define the magnetic susceptibility ($\chi$) as,

$$M = \chi H \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots (1.6)$$

 Basically, there are two types of magnetic measurements for magnetic particles: i) magnetization as a function of applied field at a given temperature ($M-H$ loop) (ii) Magnetization as a function of temperature at a given applied magnetic field (zero-field-cooled and field-cooled magnetization curves).

Fig. 1.4 (a) shows hysteresis loop of magnetic material at constant temperature. Magnetic hysteresis refers to the irreversibility of the magnetization and demagnetization process. The saturated magnetization ($M_s$) is the magnetic moment per unit volume of the material obtained when a sufficiently large magnetic field is applied to remove all domain walls and aligns the magnetization of the sample with the field. Remanent magnetization ($M_r$) is the magnetization that remains after an applied field has been removed. Coercivity ($H_c$) is the applied magnetic field required for reduction of a saturated magnetic material to zero magnetization. The temperature dependent magnetization data measured from zero-field-cooled (ZFC) and field cooled (FC) procedures are usually used to obtain the blocking temperature ($T_b$) of the magnetic materials [Fig. 1.4 (b)]. The ZFC-FC magnetization measurement is carried out as follows.

For the ZFC curve, the sample is first cooled in a zero field from a high temperature well above $T_b$, where nanoparticles are in a superparamagnetic state, down to a low temperature well below $T_b$, where nanoparticles are in a ferromagnetic state. Then, a magnetic field is applied and the magnetization as a function of temperature is measured in the warming process to a temperature well above the blocking temperature.
Figure 1.4  (a) Hysteresis curve of a ferromagnetic material at constant temperature and (b) A typical ZFC-FC magnetization measurement of magnetic material.
The FC curve is obtained by measuring the magnetization, when cooling the sample to the low temperature in the same field. In the ZFC and FC measurements the, field must be weak enough in comparison with the anisotropy field to guarantee that the ZFC-FC curve reflects the intrinsic energy barrier distribution [10].

1.5 NANOSCALE IRON OXIDE

1.5.1 Magnetic Nanoparticles

In the past decades, the synthesis of magnetic nanoparticles has been intensively studied, not only for its fundamental scientific interest but for its potential in technological applications such as magnetic storage media, magnetic resonance imaging in medicine, drug delivery systems, hyperthermia, magnetic separation and magnetic inks for jet printing [11]. The control of the monodisperse size is very important because of both physical and a chemical property of the nanocrystals strongly depends on the dimension of the nanoparticles. A key reason for the change in the physical and chemical properties of small magnetic particles is due to their increase in the surface to volume ratio with decreasing the particle size [12].

1.5.2 Iron Oxide Nanoparticles

Iron oxides are a group of minerals widespread in nature and readily synthesized in laboratory. There are three major types of iron oxide: Hematite (α-Fe₂O₃), Maghemite (γ-Fe₂O₃) and Magnetite (Fe₃O₄). The α-Fe₂O₃ is a blood red iron oxide found widespread in rocks and soils. Crystal structure of α-Fe₂O₃ is corundum (Al₂O₃), which can be described as rhombohedral or hexagonal with the space group $D_{3d}^6$. The γ-Fe₂O₃ occurs naturally in soils as a weathering
product of Fe₃O₄, to which it is structurally related. Both γ-Fe₂O₃ and Fe₃O₄ exhibit a spinel crystal structure, wherein the oxygen atoms form a FCC closed packed orientation and the iron cations occupy the interstitial tetrahedral and octahedral. Bulk iron oxide consists of both Fe²⁺ and Fe³⁺ atoms and exhibits ferromagnetic behavior. Large ferrimagnetic crystals of Fe₃O₄ are comprised of multiple magnetic domains that exhibit magnetic moments and these are aligned within a domain, but between domains magnetic moments are oriented in random directions.

1.5.3 Magnetic Properties of Iron Oxide Nanoparticles

Magnetic property of iron oxide is significantly size-dependent and is intrinsically different from bulk magnetic particles [13]. In large sized magnetic particles, it is well known that there is a multi-domain structure, where regions of uniform magnetization are separated by domain walls. According to magnetic domain theory, the formation of a domain wall inside a magnetic particle is not thermodynamically favoured when the size decreases to a certain level called critical volume (Dₛ).

Under this condition, magnetic moments are aligned in the same direction within only one magnetic domain. The superparamagnetism can be understood by considering the behavior of a well-isolated single-domain particle. The magnetic anisotropy energy per particle which is responsible for holding the magnetic moments along a certain direction can be expressed as follows,

\[ E(\theta) = K_{\text{eff}} V \sin^2 \theta \] .................(1.7)

where \( \theta \) is the angle between the magnetization and the easy axis. The energy barrier \( K_{\text{eff}} V \) separates the two energetically equivalent easy directions of magnetization. Under certain temperature bulk, materials have magnetic
anisotropic energies much larger than the thermal energy $k_B T$ [Fig. 1.5 (a), green line].

Figure 1.5 Nanoscale transition of magnetic nanoparticles from ferromagnetism to superparamagnetism: (a) energy diagram of magnetic nanoparticles with different magnetic spin alignment, showing ferromagnetism in a large particle (top) and superparamagnetism in a small nanoparticle (bottom). (b) and (c) size dependent transition of iron oxide nanoparticles from superparamagnetism to ferromagnetism showing TEM images and hysteresis loops of (b) 55 nm and (c) 12 nm sized iron oxide nanoparticles.
The thermal energy of the nanoparticles is sufficient to invert the magnetic spin direction. For single domain nanoparticles, the thermal energy $k_B T$ exceeds the energy barrier $K_{eff} V$ and the magnetization is easily flipped [Fig. 1.5 (a), blue line]. For $k_B T > K_{eff} V$, the system behaves like a paramagnet. This system is named a superparamagnet. Superparamagnetic system has no hysteresis and the data of different temperatures superimpose onto a universal curve of $M$ versus $H/T$. For example, Fe$_3$O$_4$ nanoparticles of 55 nm exhibit ferromagnetic behavior with a coercivity of 52 Oe at 300 K, but smaller 12 nm sized Fe$_3$O$_4$ nanoparticles show superparamagnetism with no hysteresis behavior [Fig. 1.5 (b, c)].

The relaxation time of the moment of a particle ($\tau$) is given by the Néel-Brown expression [14].

$$\tau = \tau_0 \exp \left( \frac{KV}{k_B T} \right)$$

where $k_B$ is the Boltzmann’s constant and $\tau_0 = 10^{-9}$ s. If the particle magnetic moment reverse at times shorter than the experimental time scales, the system is in a superparamagnetic state, if not, it is in the so called blocked state. The temperature, which separates these two regimes, is called blocking temperature ($T_b$) and measured through zero-field cooled/field cooled set of measurement as mentioned above. In ZFC curve, the peak temperature is normally the blocking temperature $T_b$.

### 1.6 REVIEW OF LITERATURE

In the past year researchers are concentrating on shape and size dependent magnetic properties of iron oxide nanoparticles for biomedical applications. As a result of the dipolar interaction between the magnetic particles, they are intriguing building blocks for self-assembly into various
nanostructures. The assembly structures (1D, 2D and 3D) are important for fundamental studies and for the fabrication of magnetic-force triggered nanodevices. Recently, the self-assembly of magnetic nanoparticles into specific shapes were reported by many researchers [15]. The synthesis of discrete 1D nanostructured magnetic materials, such as iron oxide nanorods, ellipsoidal and wires through the oriented attachment of monodisperse spherical nanoparticles has been described. Cao et al., [16] reported synthesis of uniform α-Fe$_2$O$_3$ nanoparticles by surfactant mediated hydrothermal method. The synthesized products were α-Fe$_2$O$_3$ nanoellipsoids of 115-140 nm in long axis and 60-80 nm in short axis.

Mao et al., [17] have synthesized uniform hollow α-Fe$_2$O$_3$ spheres with diameter of about 600-700 nm and shell thickness lower than 100 nm were obtained by direct hydrothermal treatment of dilute FeCl$_3$ and tungstophosphoric acid (H$_3$PW$_{12}$O$_{40}$) solution at 180 °C. The hollow spheres were composed of robust shells with small nanoparticles standing out of the surface and present a high-surface area and a weak ferromagnetic behavior was obtained at room temperature. The effect of concentration of H$_3$PW$_{12}$O$_{40}$, reaction time and temperature for the formation of the hollow spheres were investigated in the series of experiments. The surfactant assisted hydrothermal method can induce the self alignment of the nanocrystals. Recently, hierarchical Fe$_3$O$_4$ nanostructure with coral-like morphology was synthesized by a simple glucose-assisted solvothermal method by Qin et al., [18].

The structure consists of tens of twigs with lengths about 1-2 μm. The root of the nanostructure was composed of random-aggregated particles with sizes of 10 nm, and the twigs were formed from the oriented-aggregation of nanoparticles. During the formation of the hierarchical structure, glucose played an important role. Its derivates coordinated with iron ions to control the nucleation and growth of Fe$_3$O$_4$ and also acted as a morphology-directing agent. Superparamagnetic nanoparticles do not show any residual
magnetization upon removal of external magnetic field, unless they cooled to below their blocking temperature [19]. Therefore, while easily manipulate by external magnetic field due to their large induced magnetization, they don’t undergo aggregation or coagulation in the absence of external magnetic field. Furthermore, superparamagnetic nanoparticles are very attractive than ferromagnetic nanoparticles due to their broad range of biomedical applications. Surfactants or polymers are often employed to passivate the surface of the nanoparticles during or after the synthesis to avoid agglomeration. In general, electrostatic repulsion or steric repulsion can be used to disperse nanoparticles and keep them in a stable colloidal state. The best known example for such systems is the ferrofluids which were invented by Papell in 1965 [20].

In the case of ferrofluids, the surface properties of the magnetic particles are the main factors determining colloidal stability. The major measures used to enhance the stability of ferrofluids are the control of surface charge [21] and the use of specific surfactants [22-24]. For instance, magnetite nanoparticles synthesized through the co-precipitation of Fe$^{2+}$ and Fe$^{3+}$ in ammonia or NaOH solution is usually negatively charged, resulting in agglomeration. To achieve stable colloids, the magnetite nanoparticle precipitate can be peptized (to disperse a precipitate to form a colloid by adding of surfactant) with aqueous tetramethylammonium hydroxide or with aqueous perchloric acid. The magnetite nanoparticles can be acidified with a solution of nitric acid and then further oxidized to maghemite by iron nitrate. After centrifugation and redispersion in water, a ferrofluid based on positively charged $\gamma$-Fe$_2$O$_3$ nanoparticles was obtained, since the surface hydroxy groups are protonated in the acidic medium [25]. Commercially, water or oil based ferrofluids are available. They are usually stable when the pH value is below 5 (acidic ferrofluid) or over 8 (alkaline ferrofluid). In general, surfactants or polymers can be chemically anchored or physically adsorbed on magnetic nanoparticles.
to form a single or double layer [26, 27], which creates repulsive (mainly as steric repulsion) forces to balance the magnetic and the van der Waals attractive forces acting on the nanoparticles. Thus, by steric repulsion, the magnetic particles are stabilized in suspension. Polymers containing functional groups, such as carboxylic acids, phosphates, and sulfates, can bind to the surface of magnetite. Suitable polymers for coating include poly(pyrrole), poly(aniline), poly(alkylcyanoacrylates), poly(methyldiene malonate), and polyesters, such as poly(lactic acid), poly(glycolic acid), poly(e-caprolactone), and their copolymers [28-31]. Surface-modified magnetic nanoparticles with certain biocompatible polymers are intensively studied for magnetic-field-directed drug targeting, and as contrast agents for magnetic resonance imaging [32, 33].

Chu et al., have reported a synthesis of polymer-coated magnetite nanoparticles by a single inverse microemulsion [34]. The magnetite particles were first synthesized in an inverse microemulsion, consisting of water/sodium bis(2-ethylhexyl)sulfosuccinate/toluene. Subsequently, water, monomers (methacrylic acid and hydroxyethyl methacrylate), crosslinker (N,N-methylenebis(acrylamide) and an initiator (2,2’-azobis(isobutryonitrile)) were added to the reaction mixture under nitrogen, and the polymerization reaction was conducted at 558 °C. After polymerization, the particles were recovered by precipitation in an excess of an acetone/methanol mixture (9:1 ratio). The polymer-coated nanoparticles have superparamagnetic properties and a narrow size distribution at a size of about 80 nm. However, the long-term stability of these polymer-coated nanoparticles was not addressed. Polyaniline can also be used to coat nanosized ferromagnetic Fe₃O₄ by oxidative polymerization in the presence of the oxidant ammonium peroxodisulfate [35]. Water soluble magnetic Fe₃O₄ nanoparticles were synthesized by combining the in situ synthesis and decomposition of a magnetic polymer hydrogel. Fe₃O₄ nanoparticles with an average diameter of 6.3-8.3 nm were synthesized in a
cross-linked polyacrylamide hydrogel by coprecipitating iron ions. The decomposition of the magnetic polymer hydrogel by an aqueous solution of sodium hydroxide led the transfer of Fe₃O₄ nanoparticles into the aqueous medium. The saturation magnetization of Fe₃O₄ nanoparticles were 44.6 and 54.7 emu g⁻¹ at 300 K and 5 K, respectively [36]. Bora et al., [37] have reported covalently binding of BSA molecules with stearic acid capped iron oxide nanoparticle. Magnetic property by was retained even after binding of BSA on Fe₃O₄ nanoparticles.

Folic acid (FA)-functionalized Fe₃O₄ nanoparticles were synthesized from iron (III) 3-allylacetylacetonate (IAA) through in situ hydrolysis and ligand modification. The γ-carboxylic acid of FA was successfully bound to the ligand of the Fe₃O₄ nanoparticles without the loss of the α-carboxylic acid group of folic acid, which has an affinity for folate receptors expressed on tumor cells. The diameter of the folic acid conjugated Fe₃O₄ nanoparticles is 8 nm, exhibited superparamagnetic behavior and a relatively high magnetization at room temperature. The SAR of the FA-Fe₃O₄ nanoparticles was 670 W g⁻¹ in a 230 kHz alternating magnetic field and 100 Oe. The chemo selective surface modification of magnetite particles with FA yielded a novel cancer-targeting system for use in hyperthermia treatment [38].

Although there have been many significant progresses in the synthesis of organic materials functionalized iron oxide nanoparticles, simultaneous control of their shape, stability biocompatibility, surface structure and magnetic properties is still a challenge. As an alternative, inorganic compound functionalized iron oxide nanoparticles can greatly enhance the antioxidation properties for naked iron oxide nanoparticles, and its corresponding scope of application has been greatly extended. Moreover, inorganic compounds functionalized iron oxide nanoparticles are very promising for application in catalysis, biolabeling and bioseparation. The applied coating inorganic
materials include silica, metal, nonmetal, metal oxides, and sulfides. Composite nanoparticles can roughly be divided into two major parts: preserved the magnetic property of iron oxides and preserved the other properties of inorganic materials. The structure of inorganic compound functionalized iron oxide nanoparticles (always core) can roughly be divided into five types: core-shell, mosaic, shell-core, shell-core-shell, and dumbbell. Many studies have shown that in the presence of core-shell structure composite Nanoparticles such as Ag@Fe and Fe$_2$O$_3$@Ag nanocomposites, its two-layer structure include magnetite core and silver shell in the outer layer. Generally, superparamagnetic colloid particles offer some attractive possibilities in bioseparation, biodetection and microbial activities. Less toxic Fe$_2$O$_3$@Ag core/shell nanocomposites were prepared via in situ chemical reduction of silver ions by maltose in the presence of particular magnetic phase and molecules of polyacrylate serving as a spacer among iron oxide and silver nanoparticles [39].

Zhang et al., [40] have reported synthesis of Fe$_3$O$_4$/Ag composite was synthesized by simple sonochemical method. These composites were obtained from sonication of Ag(NH$_3$)$_2^+$ and (3-aminopropyl)triethoxysilane coated Fe$_3$O$_4$ nanoparticles solution at room temperature in ambient air for 1 h. Fe$_3$O$_4$/Ag nanocomposite exhibits superparamagnetic characteristics at room temperature. Furthermore, these composites have good catalytic properties. In the recent years, great efforts have been made to incorporate the magnetic nanoparticles, luminescent quantum dots (CdSe, CdTe and carbon) and organic dyes to synthesis the synthesizing magnetic and fluorescent nanocomposites. In the recent years, great efforts have been made to incorporate the magnetic nanoparticles and luminescent particles on silica sphere to get both magnetic and fluorescent properties. Therefore, for further extended function of silica functionalized iron oxide nanoparticles, some quantum dots and other optical materials have been introduced.
The fluorescent CdTe quantum dots (QDs) were covalently linked and assembled around individual silica-coated superparamagnetic Fe₃O₄ nanoparticles. Active carboxylic groups were presented on the surface for easy bioconjugation with biomolecules. Fe₃O₄ nanoparticles were first functionalized with thiol groups, followed by chemical conjugation with multiple thioglycolic acid modified CdTe QDs to form water-soluble Fe₃O₄/CdTe magnetic/fluorescent nanocomposites. The nanocomposites exhibited magnetic and fluorescent properties favorable for their applications in magnetic separation and guiding as well as fluorescent imaging. Further, Fe₃O₄/CdTe nanocomposites conjugated with anti-CEACAM8 antibody were successfully employed for immuno-labeling and fluorescent imaging of HeLa cells [41].

1.7 OBJECTIVES OF THE THESIS

This work is highly interdisciplinary bridging the field of chemistry, physics and materials science. The main objectives of the research work are as follows.

- Synthesis and characterization of iron oxide nanoparticles with different morphologies.
- Surface coating of iron oxide nanoparticles by bovine serum albumin (BSA), polyethylenimine (PEI), Ag and study of their properties.
- Development of novel multifunctional nanocomposites: magnetic, fluorescent and silica nanospheres and study of their magnetic and luminescence properties.
1.8 ORGANIZATION OF THE THESIS

The thesis has been organized into 5 chapters. Chapter 2 provides a brief introduction about synthesis procedures; mainly chemical routes are reviewed and briefly discussed about analytical techniques used in the present work. Chapter 3 discusses the synthesis and characterization of two different phases (hematite and magnetite) of iron oxide nanostructures. Section 3.1 summarizes the synthesis of hematite nanorods by reverse micelles followed by heat treatment. The length and diameter of the nanorods were measured from TEM micrograph were 30-50 nm and 120-150 nm respectively. The weak ferromagnetism of nanorods was confirmed by VSM measurements. In Section 3.2 synthesis, structural, morphological, vibrational and magnetic properties of hematite nanoparticles are given. Section 3.3 deals with the synthesis of Fe$_3$O$_4$ nanorice by surfactant assisted hydrothermal method. Nanorice with an average diameter of 150 nm and length of 500 nm were obtained, as confirmed by electron microscopy. The morphology evolution studies regarding the formation mechanism of such nanoparticles are also reported. High resolution TEM image shows the single crystalline structure of the Fe$_3$O$_4$. Vibrational and magnetic properties of the nanorice also been discussed. Section 3.4 includes the synthesis of superparamanetic Fe$_3$O$_4$ nanoflowers through surfactant assisted hydrothermal method using TETA as a surfactant. The formation mechanism is also discussed. Magnetic property of nanoflowers is investigated by VSM measurement.

Chapter 4 gives the synthesis of surface coating/functionalization of superparamagnetic magnetite nanoparticles and its properties. Section 4.1 includes the synthesis and BSA coated magnetic fluid and heating characteristic measured for magnetic fluid hyperthermia applications. Section 4.2 deals with synthesis, structural, morphological and magnetic properties of PEI coated iron oxide nanoparticles. Further, adsorption properties of BSA with PEI coated Fe$_3$O$_4$ nanoparticles are studied.
Section 4.3 represents synthesis of Ag coated Fe₃O₄ nanoparticles with surface plasmon resonance and superparamagnetic properties. Section 4.4 details the synthesis of multifunctionalized nanocomposite: magnetic, fluorescent and silica. Both optical and magnetic properties of nanocomposites were discussed by PL and VSM measurements respectively. Chapter 5 gives about overall summary of the present work and scope of future work.