CONCLUSIONS

The first requirement of any novel study of nanoparticulated oxides is the synthesis of the material. The development of systematic studies for the synthesis of oxide nanoparticles is a current challenge. Iron oxide nanoparticles are iron oxide particles with superparamagnetic properties. The two main forms are magnetite and oxidized form maghemite. Magnetite has inverse spinal structure with oxygen forming face centered cubic crystal system. Maghemite has cubic unit cell. A number of specific methods have been developed among which those broadly in use are: Co-precipitation methods, Sol-gel processing, Microemulsion technique, photo CVD methodologies and crystalline growth. In Solvothermal methods metal complexes are decomposed thermically either by boiling in an inert atmosphere or using an autoclave with the help of pressure. Gas-solid transformation methods with broad use in the context of ultrafine oxide powder synthesis are restricted to chemical vapor deposition (CVD) and pulsed laser deposition (PLD). Magnetic nanoparticles are of great interest for researchers from a broad range of disciplines including data storage, catalysis and magnetic fluids. Currently magnetic nanoparticles are also used in important bio applications like clinic diagnosis and MRI therapy. However, it is technological challenge to control size, dispersibility, stability and shape. Iron oxide has applications in paints, shoe polish, rubber, concrete, leather, medicines, lipstick, nutrients and feeds, body and face cream, Talcam powder, ATM cards, magnet, electronic parts, audio and video cassette tape. Transition metal oxides have many applications as catalyst, sensors, superconductors and adsorbents. Among transition metals oxides, copper oxide nanoparticles are of special interest because of their efficiency as nanofluids in heat transfer applications. It has been reported that 4% addition of CuO improves the thermal conductivity of water by 20%. CuO is a semiconducting compound with a narrow band gap and used for photoconductive and photothermal applications. Recently, Nanotechnology has emerged as the forefront of science and technologies. The intersecting fields of study that creates this domain of advancement of nanotechnology. Nanotechnology is forecasted as the second industrial revolution in the world. The novel properties have attracted global interst across disciplines. ZnO nanoparticles exhibit bright stable photoluminisence in colloidal dispersion. ZnO is a versatile semiconductor material. ZnO has band gap energy of 3.37 eV and it has very large excitation binding energy (60 meV) at room temperature. It is wurtzite type semiconductor.
Spinel ferrites are magnetic materials and have wide applications in magnetic devices and switching devices. Zinc ferrite (ZnFe$_2$O$_4$) is of interest as it has wide applications in not only to basic research in magnetism, but also has great potential in technological application, such as magnetic materials, gas sensors, catalysts, photocatalysts and absorbent materials. Nanosized nickel ferrite possesses attractive properties for the application as soft magnets, core materials in power transformers and low loss materials at high frequencies. Copper ferrite (CuFe$_2$O$_4$) is one of the important spinel because ferrites copper ferrite (CuFe$_2$O$_4$) have paramount advantages over other type of magnetic materials due to its unmatched flexibility in magnetic and mechanical parameters, high stability, high quality, low cost and low eddy current losses over a wide range of frequency from 10 KHz to 50 KHz. Copper ferrite (CuFe$_2$O$_4$) is an important material to be used in different applications such as catalytic, synthesis of temperature sensitive magnetic fluid, new pigments, sensors, Li-ion batteries, magnetic refrigeration and electrochemical cells.

Nickel-zinc ferrite is a soft magnetic material having low magnetic coercivity, high resistivity and little eddy current loss in high frequency operations (10–500 MHz) values. The high electrical resistivity and excellent magnetic properties make this ferrite an automatic choice as a core material for power transformers in electronic and telecommunication applications in megahertz frequency regions. The properties of ferrite materials are strongly influenced by the materials composition and microstructure, which are sensitive to the preparation methodology used in their synthesis. Cu$_{0.5}$Zn$_{0.5}$Fe$_2$O$_4$ is a spinel ferrite. The general formula of the spinel ferrite is MeFe$_2$O$_4$ where Me usually represents one or, in mixed ferrites more than one of the divalent transition metals Mn, Fe, Cu, Ni, Zn, Ca or Mg and Cd. Other combinations of equivalent valency are possible and it is also possible to replace some or all the trivalent iron ions with other trivalent metal ions. Ni-Cu ferrites are magnetic materials with improved magnetic, high resistance and high frequency response. Several researchers have focused their research on Ni-Cu ferrite because copper containing ferrites because copper constraining ferrites have interesting electrical and magnetic properties.

Several simulated experiments and theoretical evidences have led us to show that properties of the particles depend on the size and size of the particles depends on the procedure of synthesis. Properties of the particles also depend on the chemicals used in the synthesis. So an
attempt has been made on the synthesis of some nanosized metal/mixed metal oxides taking different starting materials and ammonia as precipitating agents.

Results of the above studies have been reported in the form of five chapters as follows.

Introduction deals with the topic “metal oxides, their synthesis procedures and literature survey on applications of nanosized metal oxides in various fields like catalyst, adsorbent, superconductors, sensors etc”.

Experimental methodology and instrumentation have been discussed. Synthesis of nanosized metal/mixed metal oxides, their characterization and methods of chemical analyses has been studied. The metal/mixed metal oxides have been synthesized by precipitation method using ammonia as precipitating agent and characterized using magnetic susceptibility measurements (VSM), TGA/DTA, X-ray diffraction patterns, Transmission electron microscopy (TEM) and Scanning electron microscopy (SEM). Experimental conditions and techniques used for the characterization of metal oxides/mixed metal oxides have been given.

Result of studies on the synthesis of nanosized iron oxide, nanosized nickel oxide, nanosized copper oxide, nanosized zinc oxide has been discussed. $\alpha$-Fe$_2$O$_3$ nanoparticles with corundum structure are synthesized successfully by aqueous precipitation method. From TEM study, it is found that particles are with average size of 15-49 nm. Magnetic measurements shows that Fe$_2$O$_3$ has five unpaired electron and hence paramagnetic in nature. XRD studies show that iron oxide was formed as $\alpha$-Fe$_2$O$_3$ instead of the commonly formed magnetite nanoparticles Fe$_3$O$_4$ or a mixture of magnetite and maghemite. Average crystallite size calculated is 35 nm.

Surface area of the metal oxide was 27 m$^2$/g. The magnetic moment for iron oxide was carried out at room temperature and was observed as 5.68 B.M. This value of magnetic moment supports the fact that the synthesized iron oxide is in the form of Fe$_2$O$_3$ with actual magnetic moment 5.92 B.M. This indicates the presence of 5 unpaired electrons in Fe$_2$O$_3$. Magnetic measurements were also carried out at temperatures ranging from 300K to 100K to determine the temperature of Morin transition. VSM studies were carried out at 300K to show hysteresis behavior of nanosized particles and it has been observed that Fe$_2$O$_3$ show ferromagnetic behavior in nanocrystalline form. Ms value being 0.17 emu/g. NiO nanoparticles with rhombohedral structure were synthesized successfully. From TEM study, it is found that particles have average size of 28-50 nm. Magnetic measurements showed that NiO has two unpaired electrons and hence paramagnetic in nature. Nanosized metal oxide namely copper oxide has been synthesized
by precipitation method and characterized. XRD studies show that copper oxide was formed as CuO and it has monoclinic structure. Magnetic measurements showed copper oxide has one unpaired electron and is paramagnetic in nature. The particle size of the synthesized copper oxide was determined by TEM. TEM images show that the size of particles of CuO varied from 12nm to 35nm which is in good agreement of the theoretically predicted size of nanomaterials. Zinc oxide has been synthesized by using simple and efficient precipitation method from zinc nitrate and liquid ammonia with calcinations step at high temperature at 500°C temperature for 5 hours. TEM images showed that the size of the particle of the zinc oxide varied from 21-40 nm which is in good agreement with the theoretically predicted size of nanomaterials. The results indicates that synthesized nanosized zinc oxide nanoparticles are having hexagonal wurtzite crystal structure. TEM images showed that the size of the particle of the zinc oxide varied from 21-40 nm which is in good agreement with the theoretically predicted size of nanomaterials.

Results of studies on synthesis of nanosized NiFe$_2$O$_4$ nanoparticles, ZnFe$_2$O$_4$ nanoparticles and CuFe$_2$O$_4$ have been discussed. NiFe$_2$O$_4$ nanoparticles with cubic structure are synthesized. TEM study shows very fine nanoparticles of diameter 10-28nm with average size of 20nm. VSM studies show ferromagnetic behavior of synthesized nanoparticles. It showed Ms value (15 emu/g). The average crystallite size $D_{\text{XRD}}$, $D_{\text{TEM}}$, and the lattice constant (a) of the sample obtained have been calculated and summarized. ZnFe$_2$O$_4$ nanoparticles with spinel structure are synthesized and characterized. X-ray diffraction pattern of ZnFe$_2$O$_4$ pure indicated that zinc ferrite in the form of ZnFe$_2$O$_4$. From TEM study it is found that particles are having average size of 10-30 nm. Magnetic measurements show that ZnFe$_2$O$_4$ is super paramagnetic in nature having saturation magnetization (Ms) value 50 emu/g. Monodispered CuFe$_2$O$_4$ nanoparticles were synthesized by a facile precipitation method using Ferric Nitrate, Zinc Nitrate and aqueous ammonia. Pure CuFe$_2$O$_4$ without CuO or Fe$_2$O$_3$ as by product was obtained by this method due to homogenous mixing of metal cations in initial solutions. XRD pattern of the sample prepared by precipitation method shows nano crystalline nature of the sample. It has been observed that the material prepared is having lattice constant of 8.398Å. The average particle size resulting synthesized were observed to the 20 nm which is well supported by SEM/TEM micrographs. Magnetic measurements show that CuFe$_2$O$_4$ is super paramagnetic in nature having saturation magnetization (Ms) value 50 emu/g.
Results of studies on synthesis of nanosized Ni$_{0.5}$Zn$_{0.5}$Fe$_2$O$_4$, Ni$_{0.5}$Cu$_{0.5}$Fe$_2$O$_4$ and Cu$_{0.5}$Zn$_{0.5}$Fe$_2$O$_4$ have been discussed. Ni$_{0.5}$Zn$_{0.5}$Fe$_2$O$_4$ nanoparticles with cubic spinel structure are synthesized successfully by aqueous precipitation method. From TEM study it is found that particles are having size of 20-40nm. Magnetic measurements show that Ni$_{0.5}$Zn$_{0.5}$Fe$_2$O$_4$ is super paramagnetic in nature having saturation magnetization (Ms) value 29 emu/g. Ni$_{0.5}$Cu$_{0.5}$Fe$_2$O$_4$ have been synthesized and characterized. The X-ray diffraction patterns confirm the formation of single phase cubic spinel structure. SEM (Scanning electron microscopy) was used to characterize the micro structure of the ferrite samples. A homogenous and fine grain structure was observed. By using TEM (Transmission electron microscopy) particle size was calculated. The particle size of synthesized Ni$_{0.5}$Cu$_{0.5}$Fe$_2$O$_4$ varied from 8nm to 41nm which is in good agreement of the theoretically predicted size of nano materials. Magnetic measurements show that Ni$_{0.5}$Cu$_{0.5}$Fe$_2$O$_4$ is super paramagnetic in nature at room temperature and hence used in magnetic devices. Nanosized Cu$_{0.5}$Zn$_{0.5}$Fe$_2$O$_4$ nanoparticles with cubic spinel structure are synthesized successfully by aqueous precipitation method. A homogeneous and fine grain microstructure was observed. Magnetic measurements shows that Cu$_{0.5}$Zn$_{0.5}$Fe$_2$O$_4$ is super paramagnetic in nature at room temperature and hence used in magnetic device. The particle size of synthesized Cu$_{0.5}$Zn$_{0.5}$Fe$_2$O$_4$ varied from 8nm to 39nm which is good agreement of the theoretically predicted size of nanomaterials. This method is advantageous over existing methods of synthesis of nanoparticles because other methods require specialized instrumentation, highly skilled labour, expensive materials and methods. Therefore, the proposed precipitation method is very promising, easy and cheap and may have extensive applications.