Summary

The transition metal oxides of Fe, Co, Ni and their mixed oxides are long been of interest potential applications in diverse fields due to open d shell. Magnetic iron oxide nanoparticles and their dispersions in various media have long been of scientific and technological interest. The cubic spinel structural MFe$_2$O$_4$ or MO-Fe$_2$O$_3$ represents a well known and important class of iron oxide materials where oxygen forms an fcc close packing and M$^{2+}$ and Fe$^{3+}$ occupy either tetrahedral or octahedral interstitial sites.

These materials are potential candidates for many important technological applications ranging from information storage and electronic devices to medical diagnostics and drug delivery. Depending on the chemical identity of M$^{2+}$, the densely packed nanocrystalline MFe$_2$O$_4$ based materials can have either high magnetic permeability and electrical resistivity and may be a potential candidate for future high performance electromagnetic devices. For example Fe-Co alloys, as traditional soft magnetic material, have the highest magnetic permeability and very good stability. Nano sized particles of alloys exhibit properties, which drastically differ from the bulk (118). As a possible solution of the problem, the preparation of superfine particles with homogeneous morphology and size of a great interest in the technological and theoretical fields.

For past two decades the Nanomaterials are gaining much more interest due to their size dependent physicochemical properties such as optical, structural, thermal, magnetic, catalytic and so on. Due to the increase in surface to volume ratio the surface area increases to large extent and the magnetic and catalytic properties are expected to change.

With this objectives, the mono-and bimetallic oxides i.e. ferrites of Fe, Co and Ni have been prepared by simple electrochemical process using sacrificial anodes and Pt as cathode. The advantages of this method is that by simply adjusting the current density for a fixed duration of electrolysis particle size can be controlled. The parameters such as duration of electrolysis, concentration of capping agent were optimized. Quaternary alkyl ammonium salts i.e. tetra propyl / butyl / octyl ammonium bromide was used as supporting electrolyte and capping agent for control of particle size. The vacuum dried samples after preparation were analyzed.
by UV-Vis, FTIR, and XPS spectroscopy. Structural confirmation was done by X-Ray diffractometry and TEM. The quantitative analysis was done by E-DAX.

The following observations were obtained.

Optical spectra were analyzed as a function of current density, nature of ligand and nature of solvent.

The increase in intensity of peaks in UV region is an indication of enhanced charge transfer process and red shift with increase in current indicates $\pi - \pi^*$ transitions.

IR analysis was carried out in order to understand the binding of various functional groups to the oxide. Initial appearance of strong $-\text{OH}$ group indicated hydrated form of as prepared oxides. After thorough washing, drying and heating upto 800°C, disappearance of these peaks and intensification of oxide peak confirmed the removal of ligand.

XRD and TEM analysis confirmed that with increase in current density, particle size decreases. The interplanar spacings were in perfect agreement with ASTM data. When the comparison with different ligands was made it was observed that with increase in alkyl chain length, the bulky nature of the group prevents the aggregation and controls the size. So it was proved in this work that ligands play a definite role in controlling the particle size.

X-ray photoelectron-spectroscopy confirmed that Fe is in +3, Ni in +2, Co in +2 and +3 state. In cobalt ferrite Co was present in +2 state.

Thus Fe$_2$O$_3$, NiO, Co$_3$O$_4$, NiFe$_2$O$_4$ and CoFe$_2$O$_4$ were prepared successfully. These materials are potentially useful to study the magnetic properties further.