CHAPTER -VIII

CONCLUSION

There is a great deal of interest in transition metal oxides from both fundamental and practical points of view especially in their nanosized form. Magnetic nanoparticles have received considerable interest owing to their potential applications in biomedical, magnetic storage and imaging fields. In the synthesis of nanoparticles, wherever possible use of IL offer a green route owing to their high thermal stability, non-toxic and environmentally benign in nature. In the present investigation one such ionic liquid, [Bmim][TfO] IL is tried. This judicious choice of the [Bmim][TfO] IL tried in the present investigation over the commonly used ionic liquids such as [Bmim][PF₆] and [Bmim][BF₄] is based on the fact that the anionico moiety, C-F bond is inert to hydrolysis and they do not produce any toxic compound. On contrary, BF₄⁻, PF₆⁻ anions tend to liberate HF acid (Bond energy C-F 485 kJ/mol, P-F 490 kJ/mol, B-F 613 kJ/mol). Further the imidazolium based cations are highly structured hydrogen bonded network and they have strong effect on reaction process that could lead to the anisotropy of the medium providing anisotropic micro-domains for the reaction system facilitating the anisotropic growth of metal oxide nanoparticles restricting the movement and polarization of ions paving way for the control over the nucleation and growth steps of the precipitation process. Thus the IL forms superamolecular structures that act as “entropic drivers” making possible the control of size and shape of nanostructured materials. The significant characteristic results obtained for the synthesized nanoparticles of Iron oxide, Nickel oxide, Manganese oxide and Cobalt oxide in the ionic liquid medium is summarized below.

The insignificant weight loss for the IL decomposition indicates that the purity of the Fe₃O₄ NPs where the wrap of IL on the surface of Fe₃O₄ nanoparticles was
scanty. The XRD analysis illustrate that the structural property of the synthesized Fe$_3$O$_4$ nanoparticles was consistent with the face-centered cubic standard pattern. As the base and IL concentration increases, the crystallite size was decreased. The crystallinity is improved with calcination. All the FTIR spectra of Fe$_3$O$_4$ nanoparticles exhibit the peaks for the Fe-O bond and the appearance of peaks at lower wavenumber could be correlated with the spinel structure of Fe$_3$O$_4$ nanoparticles. Spherical morphologies with homogeneously dispersed Fe$_3$O$_4$ nanoparticles with an average size of 22 ± 2 nm were obtained at higher base and IL concentration. Highly crystalline cubo-octahedral shape rather uniform Fe$_3$O$_4$ nanoparticle was observed through TEM and SAED patterns. The synthesized Fe$_3$O$_4$ nanoparticles exhibit superparamagnetic characteristic behaviour.

The formation of pure NiO nanoparticles was confirmed by the TGA analysis. The as-prepared samples at room temperature and 250°C calcined samples were indexed to β form of Ni(OH)$_2$. The 400°C calcined samples exhibit pure cubic phase of NiO. The grain size of NiO was reduced uniformly with increasing the [Bmim][TfO] IL concentration. The FT-IR spectral studies of 400°C calcined samples confirm the formation of NiO nanoparticles. The synthesized NiO particles are agglomerated sheet like morphology and polycrystalline cubic structure. A linear magnetization curve with the magnetic field was observed for all the synthesized NiO samples exhibiting superparamagnetism.

The presence of IL and PVP favours the formation of stable Mn$_3$O$_4$ nanoparticles. The formation of tetragonal structured hausmannite nanoparticles was ascertained by the X-ray diffraction, SAED and FTIR studies. The homogeneity of the Mn$_3$O$_4$ nanoparticle dispersion was increased linearly with increase in IL concentration and also with the addition of PVP. The control over the morphology
and structural characteristics of Mn₃O₄ (Husmannite) nanoparticles were more efficiently tailored by the IL and PVP. The synthesized Mn₃O₄ nanoparticles delivered a superparamagnetic behavior with no coercivity and remanence.

The TGA spectra reflect the stable formation of Co₃O₄ nanoparticles. On increasing [Bmim][TfO] IL concentration, reduced crystallite size was observed. The increased crystal growth was noted with the addition of H₂O₂. In addition to the metal oxide (Co-O) bond, appearance of peaks at lower wave number in FT-IR spectra confirms the spinel structure of Co₃O₄ nanoparticles. The FE-SEM micrographs show that the synthesized Co₃O₄ particles were nanoworm like morphology. The restructuring of Co₃O₄ nanoworms to irregular hexagonal plate like structure was noted with the addition of H₂O₂ at 80°C. The Co₃O₄ nanoparticles exhibit weak ferromagnetic character, which attracts a great interest in the energy storage, magnetic and electrochemical devices.

Among the synthesized magnetic nanoparticles, iron oxide and manganese oxide demonstrate that the homogeneously dispersed particles in the size range of 20 nm over the other two types. To conclude, the chosen IL has good control on the Fe₃O₄ and Mn₃O₄ nucleation and growth that led to the tiny particle formation. The Fe₃O₄, Mn₃O₄ and NiO nanoparticles exhibit potentially good magnetic property of superparamagnetism, they could be employed in MR imaging, targeted drug delivery, nanoscale encapsulation and so forth.