Chapter 5

Conclusions and Suggestions for Future Work
5.1. Conclusions

Zirconium(IV) phosphate precipitate modified by incorporation of poly-o-toluidine (a conducting polymer), was prepared in this study as a novel fibrous type ‘polymeric-inorganic’ composite cation-exchange material, has better ion-exchange capacity and is highly selective for mercury. The polymer selected is poly-o-toluidine after careful scrutiny of the literature survey and their chemical synthesis by standard oxidative polymerization as found in literature was successfully applied.

The preparation methodology of this composite (poly-o-toluidine Zr(IV) phosphate) cation-exchanger is a successful modification of that such materials mentioned in the literature, providing a new class of “organic-inorganic” hybrid ion-exchangers possess fairly good yield, better mechanical and granulometric properties, good ion-exchange behavior, higher chemical, mechanical as well as thermal stabilities, reproducibility behavior as is evident from the fact that these materials obtained from various batches did not show any appreciable deviation in their percentage of yield and ion-exchange capacities and selectivity for some specific heavy metals indicating its useful environmental applications.

The composite material was characterized by means of some instrumental techniques such as SEM, FT-IR, TGA-DTA, X-ray, elemental analysis, AAS.
UV/VIS spectrophotometry etc. Scanning electron microscope (SEM) photographs of poly-o-toluidine, Zr(IV) phosphate and poly-o-toluidine Zr(IV) phosphate obtained at different magnifications, indicating the binding of inorganic ion-exchange material with organic polymer, i.e. poly-o-toluidine. The SEM pictures showed the difference in surface morphology of organic polymer, inorganic precipitate and composite material. It has been revealed that after binding of poly-o-toluidine with Zr(IV) phosphate, the morphology has been changed. The X-ray powder diffraction pattern of poly-o-toluidine Zr(IV) phosphate composite cation-exchanger exhibited no peak in the spectrum that suggesting an amorphous nature of the composite material. The chemical composition, tentative formula and structure of the composites were determined on the basis of FT-IR, TGA-DTA, elemental analysis, AAS, UV/VIS spectrophotometric studies.

This material possessed a better Na\(^+\) exchange capacity (1.71 meq g\(^{-1}\)) as compared to Zr(IV) phosphate (1.46 meq g\(^{-1}\)). The solubility experiment showed that the material has reasonable good chemical stability as the results indicated that the material was resistant to different acids, bases, organic solvents etc. The chemical stability may be due to the presence of binding polymer, which can prevent the dissolution of heteropolyacids sols or leaching of any constituent element into the solution.

The distribution coefficient (K\(_d\)) values obtaining from sorption (selectivity) studies indicate that the composite cation-exchange material was found to be highly selective for Hg\(^{2+}\) ions. This adsorption behavior of this cation-exchanger is
promising in the field of pollution chemistry where an effective separation method is needed for Hg(II) from other pollutants.

The selectivity of the composites was tested by achieving some important binary separations, like separation of different synthetic metal mixtures involving Hg(II), for example: Hg$^{2+}$-Zn$^{2+}$, Hg$^{2+}$-Cd$^{2+}$, Hg$^{2+}$-Cu$^{2+}$, Hg$^{2+}$-Ni$^{2+}$, Hg$^{2+}$-Mg$^{2+}$ and Hg$^{2+}$-Al$^{3+}$, on their columns, successfully.

Electrochemical sensors represent an important subclass of chemical sensors in which an electrode is used as the transduction element. According to such devices hold a leading position among sensors presently available, having reached the commercial stage, and have found a vast range of important applications in the field of clinical, industrial, environmental and agricultural analyses fermentation control, pharmaceutical analysis, environmental monitoring, sewage treatment, food industries, etc. Since use of ion-selective electrodes offer several advantages over other analytical techniques, such as rapid, accurate and low cost analysis, hence the development of ion-exchange membrane electrodes has also been carried out in the present studies. The chemical, thermal and mechanical strength of the prepared composite materials can be utilized to make its ion-selective membrane electrode for the selective determination of Hg$^{2+}$ in the solutions. The ion-exchange membrane electrode was prepared through incorporation of the composite cation-exchanger (as electroactive material phase) in an inert matrix PVC. So the efforts have been made to develop the ion-exchange membrane electrodes having quick response and enough lifetime in the determination of toxic heavy metal ions in
synthetic samples as well as real samples like effluents, wastewaters, etc. The heterogeneous precipitate Hg(II) ion-selective membrane electrode obtained from poly-o-toluidine Zr(IV) phosphate cation-exchanger material was utilize with satisfactory detection limits in the given range of $1 \times 10^{-1}$ to $1 \times 10^{-6}$ molar with a slope of 26.97 mV per decade change in Hg(II) ion concentration, the slope value is close to Nerstian value, 29.6 mV per concentration decade for divalent cations, quick response 35 seconds, high working pH range of 2.0-8.0, and enough life time 2 months for the selective determination of heavy toxic meta ions \textit{i.e.} Hg$^{2+}$.

The proposed composite cation-exchanger membrane electrode assembly was also employed for the titration of Hg(NO$_3$)$_2$ solution against EDTA solution that also established their practical and analytical utility. Finally, we hope that the various physiochemical properties and analytical applications studied on this composite material will be helpful and encouraging resource for the material as well as environmental scientists to do further research work in this field.

5.2. Possible Applications

- Water softening
- Separation of toxic and hazardous ionic species
- Nuclear separations and nuclear medicine
- Catalysis
- Electrodialysis
5.3. Suggestions Future Work

The research work done in this dissertation will be continued for the development of the materials. The research is going on-

- To develop simple selective and economical “organic-inorganic” composite ion-exchange materials having selectivity towards heavy toxic metal ions and radioactive elements to decrease the pollution load of the environment.

- To study in detail, the SEM (Scanning Electron Microscopy) analysis of the inorganic, organic as well as composite cation-exchanger in terms of surface morphology, pore size, etc.

- To study about the ion-exchange kinetics and adsorption of pesticides (adsorption thermodynamics) on these materials.

- To more studies of electrical behavior of electrically conducting “organic-inorganic” composite ion-exchange materials.

- Furthermore, to study in detail, the electrically conducting composite ion-
exchange materials used as electrochemically switchable ion-exchangers for water treatment; especially for water softening.

- Efforts will be made to use these composites as adsorbents for air (i.e. gas separations) by controlling their electrical conductivity.

- A detailed study of composite ion-exchangers used as catalysts for reaction of gases and of liquids or solutes.

- Chromatographic and thin layer, separation, identification and determination of pharmaceuticals and related drugs in drugs formulations and biological samples.

- To develop ion-selective electrodes for a number of heavy toxic elements with detection limits down to parts-per-million ($10^{-10}$ M), which is possible by studying the underlying chemical principles and modifying the nature of electroactive materials used in the membrane electrodes.