A detailed investigation on the adsorption of Ni(II), Co(II) and Cr(VI) using treated *Terminalia catappa* nut shell (TTCNS), treated *Azaridachta indica* nut shell (TAINS), modified *Terminalia catappa* nut shell (MTCNS), modified *Azaridachta indica* nut shell (MAINS) and Nanocomposited *Azaridachta indica* nut shell composite (NAINSC) has been dealt in this thesis. The adsorbents were categorized into different mesh sizes using scientific test molecular sieves and their particle sizes were thereby confirmed under occular microscopy.. The adsorbent materials were collected and subjected to three different treatment methods viz., Acid-base treated, modified using disodium hydrogen phosphate and composited with nano hydroxyapatite. Analyses of BET, BJH surface area were performed and percentage of elements (C, H, N, S) of the treated adsorbents were investigated. The amounts of acidic surface groups present on both TTCNS and TAINS were determined using Boehm titration method. Physical and chemical characteristics of the adsorbents were experimentally verified. Scanning electron micrographs (SEM) of treated adsorbents were taken to understand the surface morphology of the adsorbents before and after adsorption. The adsorption of metal ions by the adsorbents was confirmed by EDAX analysis. FT-IR spectral analysis was carried out to explore the participation of functional groups present on the surface of the adsorbents with metal ion during adsorption. The different parameters pertaining to the adsorption at equilibrium were determined in order to establish the behaviour of the reactions of the six systems [Ni(II)-TTCNS, Ni(II)-TAINS, Co(II)-TTCNS, Co(II)-TAINS, Cr(VI)-TTCNS and Cr(VI)-TAINS]. The six adsorption systems were studied by batch equilibration method.

Batch equilibration method was performed to study the effect of the variables viz., particle size and dosage of the adsorbents, initial concentration of the adsorbate solutions, contact time between the sorbates and sorbent materials, pH of the solution medium, influence of cations, anions, co-ions and temperature on the adsorption rate of the systems

was determined. Exploratory desorption and regeneration experiments were carried out for metal laden adsorbents, as the repeated reusability of the same is essential.

Establishment of the adsorption characteristics were studied using, Langmuir, Freundlich, Tempkin and Dubinin-Kaganer-Radushkevich isotherm models. To understand the dynamics of the adsorption reaction and to find out the rate of the adsorption process, Pseudo-first-order kinetic, Pseudo-second-order kinetic, Elovich and intraparticle diffusion were investigated. Efficiency of treated, modified and nano-composited adsorbents on the removal of Ni(II) and Cr(VI) has been checked with effluent samples collected from chemical and electroplating industries.

From the observed results, a judicious comparison of the sorptive behaviour of the employed adsorbents and a preferential order of adsorption of the metal ions were discussed. Based on the experimental results and observations the following conclusions are arrived at.

- Analysis of BET and BJH plots support the enhanced surface area of both the treated adsorbents.
- Comparison of SEM pictures of adsorbents and metal loaded adsorbents revealed that the surface of Ni(II) loaded TTCNS underwent a significant change compared to other systems. The FT-IR spectra of metal loaded adsorbents indicated the involvement of carboxylic acid, hydroxyl and phenolic groups in adsorption.
- Maximum adsorption occurred with the smallest particle size and this is attributed to the larger surface area of the material.
- Increase of contact time and adsorbent dosage enhanced the percentage of adsorption of all the metal ions employed.
- The pH of the medium is the controlling master parameter of the adsorption process. The removal efficiency of the adsorbents raised with the decrease in pH for Cr(VI) and increased till optimum for Ni(II) and Co(II).

- Adsorption of Ni(II) and Co(II) suffered an inhibition in the presence of sodium, potassium, calcium and magnesium cations of which potassium ions registered marked influence. Significant inhibition was observed under the influence of chloride ions for the above mentioned metal ions rather than sulphate and nitrate ions.
- Adsorption of Cr(VI) was not affected by the presence of cations, since it was adsorbed as $HCrO_4^-$. Presence of anions compete for the adsorption sites and decreased the percentage removal of Cr(VI).
- Mutual inhibitive characteristics was observed when Ni(II) and Co(II) were employed as co-ions among themselves. Both the metal ions had least influence on the removal of Cr(VI) and vice versa.
- The results of desorption and regeneration studies carried out for both the materials exhibited least influence on further studies.
- Adsorption equilibrium data are in good agreement with Langmuir, Freundlich and DKR isotherm models. This indicated the monolayer adsorption and heterogeneous surface condition of the adsorbent employed. The equilibrium parameter 'R_L' values from Langmuir and 'n' values from Freundlich isotherms indicate that the sorption process is favourable for all the systems. The mean free energy of sorption value obtained from DKR isotherm indicated chemical ion exchange mechanism in Ni(II), Co(II) and Cr(VI) adsorption. The adsorbate-adsorbent interactions were corroborated by Tempkin isotherm model.
- The kinetics was well described by pseudo-second-order model for all the systems. Intraparticle diffusion plot revealed that the sorption process of metal ions occurred in two phases. First phase was due to film diffusion and the second phase due to particle diffusion.

- The adsorbed amount of metal ions increased with the solution temperature. The ΔG^0 and ΔH^0 values indicated spontaneous and endothermic nature of adsorption. The positive values of ΔS^0 showed increased randomness at the solid-solution interface.
- Utility of the adsorbents has been tested using industrial effluents. All the treated, modified and nanocomposited adsorbents are reasonably effective in the removal of Cr(VI) ions from industrial effluents.
- Comparison of the treated and modified adsorbents revealed that the performance of TTCNS, MTCNS is better than TAINS, MAINS.
- The order of preferential adsorption of metal ions by TTCNS and TAINS was Ni(II) > Co(II) > Cr(VI). This is further confirmed by hydrated ionic radius, covalent index, atomic number, ionization energy and solvation property of these metal ions.
- Nanocomposited adsorbent is found to possess enhanced performance in the removal of Ni(II) ion from aqueous solutions and industrial effluents as compared to Cr(VI)

From the above reports, it is concluded that the low-cost TTCNS and TAINS are effective, easily available, non-toxic, indigenous adsorbents for the removal of Ni(II), Co(II) and Cr(VI) from aqueous solutions and industrial effluents. The results of the present investigations would be useful for the fabrication and designing of wastewater treatment plants, utilizing TTCNS, MTCNS, TAINS and MAINS in a large scale.

Our future study aims at the establishment of performance oriented approach of MTCNS and MAINS for other toxic heavy metal ions. This can be accomplished by developing suitable modification procedures in the sorbents viz. encapsulation and immobilization, thereby ensuring the safe disposal of the metal loaded sorbents, which in turn does not pose a great threat to the environment. Also the further work accomplishes to synthesize novel nanocomposites utilizing various agricultural waste materials to enhance and upgrade the sorption quality of these low cost sorbents.