Chapter X

Conclusion

Adsorption studies had been investigated to define the sorption capacities of the chosen biosorbents viz., *Prosopis juliflora* Bark (PJB), *Tamarindus indica* Hull (TIH) and Goat Hoofs (GH) for Pb(II), Cd(II) and Ni(II) ions. The selected litter materials were procured from specific localities in Coimbatore, were chemically treated using 0.1 N HCl/ 0.2 N NaOH, labeled as TPJB, TTIH, TGH and categorized into varied mesh sizes using Scientific Test Molecular Sieves. The sorted sorbents' particle sizes were confirmed by optical microscopic studies.

Physio– chemical characteristics, surface acidic groups, elemental analysis, surface area/ pore volume were determined using Standard methods, Boehm titration, CHNS analyzer and BET/ BJH plots. The surface morphologies of unloaded and metal loaded sorbents', elemental constituents and changes in peak shifts of functional groups were recorded using SEM, EDAX, FTIR techniques.

Operating factors viz., particle size, dosage, agitation time, initial metal ion concentration, pH, cations, anions, co-ions and temperature of the following systems: Pb(II)- TPJB, Cd(II)-TPJB, Ni(II)-TPJB, Pb(II)-TTIH, Cd(II)-TTIH, Ni(II)-TTIH, Pb(II)-TGH, Cd(II)-TGH and Ni(II)-TGH, pertaining to the sorption rate and equilibrium conditions were experimented by Batch equilibration method. Isothermal models (Langmuir, Freundlich, Tempkin and DKR), Kinetics (Pseudo-First-Order, Pseudo-Second-Order, Elovich and Intraparticle Diffusion), Thermodynamic parameters were studied to establish the sorption, kinetic and equilibrium characteristics of the systems. The reusability of the metal laden materials were evaluated through sorption/ desorption experiments and compatibility of the chosen adsorbents in treating industrial leachate / synthetic solutions were tested.

0.18 mm particle size, 100 mg/L initial metal ion concentration for all the systems, except Pb(II)- TGH system (250 mg/L), pH 5/ 7 for Pb(II)/ Cd(II), Ni(II) ions, dosages as 200 mg (TGH), 300 mg (TPJB, TTIH), 400 mg (Ni(II) – TTIH), along with preset time frames were optimized under equilibrium conditions. Pb(II) – TGH system exhibited a maximum sorption capacity of 56.89 mg/g. K^+ and Cl^- exhibited greater ionic inhibition and Zn^{2+} and Cr^{6+} ions revealed lesser co-ionic inhibitions for all the systems.

Langmuir model fitted linearly well with the equilibrium data supporting monolayer adsorption as evident from adsorption parameters viz., 'q_m', 'R_L' and 'n'. The mean free energy values derived from DKR isotherm specified the physisorption process. Kinetic studies revealed the best linear fit for Pseudo-second-order kinetic model. Difference in free energy change, enthalpy and entropy data suggested the systems to be feasible, endothermic (TPJB, TTIH) / exothermic (TGH) and spontaneous in nature. Extension of batch studies to Pb(II) samples collected from paint industry and synthetic Cd(II) and Ni(II) solutions employing the three sorbents resulted in appreciable removal of lead ions at appropriate conditions.

The data obtained from batch factors were verified as input variables by applying SPSS 20 Software leading to the generation of output variables with 95% confidence level. The sorptive performances of the three adsorbents were judiciously compared, on the basis of parametric and isothermal constant values, wherein the order was found to be TGH > TPJB > TTIH. The preferential metal ion sorption order was observed to be Pb(II) > Cd(II) >Ni(II).

Sorption probabilities of TPJB and TGH were enhanced through the synthesis of their nano counterparts (NTPJB/ NTGH), followed by preparation of magnetic nanocomposites of TGH, adopting ex-situ/ one-pot synthesis. Subsequently, nano materials/ MNCs were characterized using AFM, TG-DTA/ SEM, EDAX, FTIR, XRD, VSM, Zeta- Potential and Particle Size Analyzer techniques, which affirmed the requisitive properties. Batch verification concluded that one half dose of nano sized material and one fourth dose of magnetized nanocomposite were sufficient to trap the metal ions against their treated sorbents.

Quantification of batch studies were manipulated through the continuous column running of Pb(II) aqueous / industrial effluents with TGH sorbent, by adopting short-term and long-term analyses, which evidenced maximum removal. The reusability and economic viability of the loaded materials were explored by desorption and regeneration studies. Complete TGH exhaustion had occurred after collecting 44 litres of aqueous Pb(II) and 17 litres of Pb(II) effluent at the end of fifth and third regeneration cycles. The exhausted TGH were utilized as manures for growing pulses, which ensured a

remarkable shoot growth against their controls, obviously facilitating the non-dumbing of Pb(II) laden TGH as a solid waste, otherwise posing disposal problems. This was confirmed by non-bioaccumulation of Pb(II) in the varied modules of the grown plants, tested through digestion method.

Scaling up of laboratory observations to field level was carried out through installation of prototype Fibre Reinforced Polymer columns, after their successful pilot studies under pre-scaling setup. The outcome of the prototype device implementation was reported as 1.62 mg / L Pb(II) residual concentration against 90.47 mg / L initial concentration of raw effluent at Visaka Paints and Chemicals, Coimbatore.

Our future study aims at setting up and involvement of identified sorbents and novel materials to treat industrial effluents containing many heavy metals under field conditions, which in turn should not pose an inordinate risk to the environs.