Abstract

Compounds enriched in $^{10}$B isotope of boron are used for neutron counting, reactor control and neutron capture therapy for the treatment of melanotic cancer etc. which dictate the requirements for enrichment of this isotope of boron from its natural abundance of approx. 20 %. In ion exchange chromatographic enrichment process for this isotope, the increase in acidity of boric acid helped in separation of these isotopes with increased value of isotopic exchange separation factor. The work presented in this thesis was undertaken to investigate the solution to various problems related to cost-effective separation of isotopes of boron by ion exchange chromatography.

After discussing about the various methods used for the separation of isotopes of boron and reviewing the literature with emphasis to ion exchange chromatography, the effect of concentration of four complexing reagents for boric acid viz., ethylene glycol (EG), propylene glycol (PG), dextrose and mannitol on pH and ionization constant of boric acid (0.1M) was studied by half neutralization method and by the material balance of various species present during the pH-metric titration of boric acid with standard NaOH so that any relationship between pK$_a$ and separation factor, if exists, could be established. From these determined parameters, quantitative relationships between pH and concentration, pK$_a$ and concentration of each of these complexing reagents were derived by least square polynomial curve fitting. Mannitol was found to be most effective in increasing the ionization of boric acid.

The above choice of the complexing reagent was also validated by conductimetric measurements. Using this electrochemical technique, the concentration of boric acid and the complexing reagent (mannitol) that could be used for plant operation was optimized. An attempt was also made to determine the rates of uptake of boric acid by anion exchange resin in hydroxyl form (in absence and presence of mannitol at different concentrations) and to estimate the concentration of polymeric species (polyborates) present in boric acid at different concentrations. The optimum concentration of mannitol required to be added to boric acid to study the effect of concentration of boric acid on the isotopic exchange separation factor by batch method was also determined.

Three numbers of type I and an equal number of type II indigenously
available candidate resins were analysed for their various characteristic properties viz., capacity, moisture content and degree of cross-linking, density, rates of ion exchange for the reactions of interest, swelling, pH titration curves and bead strength etc. The changes in volume of the resin during conversion from one ionic form to another, void volume of the ion exchange column, selectivity coefficients for $\text{Cl}^- \leftrightarrow \text{OH}^-$ exchange and borate $\leftrightarrow \text{Cl}^-$ exchange and distribution coefficients for $\text{OH}^- \leftrightarrow$ borate exchange were also estimated.

Since the operating capacity of the resin depends upon the regeneration of the resin from chloride to hydroxyl form, the various factors which affect the regeneration of the strong base anion exchange resins (of type I and type II) viz., regeneration level, flow rate, concentration and purity of the regenerant were investigated. The effect of concentration and regeneration level on selectivity coefficients for $\text{Cl}^- \leftrightarrow \text{OH}^-$ exchange and also that of flow rate and concentration of regenerant on height equivalent of a theoretical plate for $\text{Cl}^- \leftrightarrow \text{OH}^-$ exchange, removal of carbonate ions from regenerant NaOH by strong base anion exchange resin of type I and BaCO$_3$ precipitation and possibility of reuse of Ba(OH)$_2$ were studied.

The separation of isotopes depends upon the value of $c$ (deviation of separation factor from unity). The effects of concentration of boric acid and mannitol on isotopic exchange separation factors were determined by batch as well as column methods using type I and type II resins. From these values, a relationship was found to exist between the isotopic exchange separation factor and $pK_a$ of boric acid. Experiments were also carried out to find out the possibility of reaction between HCl and mannitol.

The various aspects of the suitability of the three electrochemical techniques viz., pH-metry, conductimetry and differential potentiometry to monitor the band movements at different stages during the operation of the plant have been discussed and the amount of mannitol required to be added to HCl (used for displacement of the borate band) has been established.