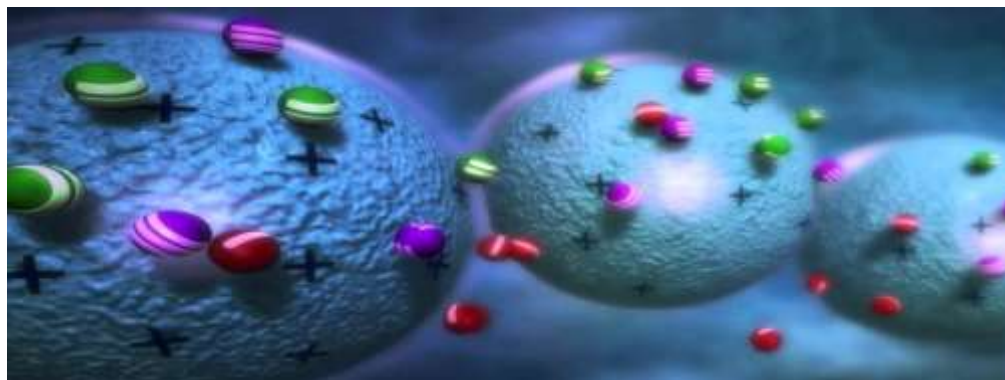


ION EXCHANGE CHROMATOGRAPHY



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Introduction

The process by which a mixture of similar charged ions can be separated by using an ion exchange resin

- Ion exchange resin exchanges ions according to their relative affinities.
- There is a reversible exchange of similar charged ions

Mostly similar charged ions like cations or anions can be separated by this technique

Principle

- Reversible exchange of ions b/n the ions present in the solution and those present in the ion exchange resin.

- **Cation exchange:**

Separation of cations

- $\text{Solid-H}^+ + \text{M}^+ \text{ (solution)} \rightleftharpoons \text{Solid-M}^+ + \text{H}^+ \text{ (solution)}$

The cations retained by the solid matrix of ion exchange resin can be eluted by using buffers of different strength and hence separation of cations can be effected.

Anion exchange

Separation of anions using

- Anion exchange resin
- $\text{Solid-OH}^- + \text{A}'^- \rightarrow \text{Solid-A}^- + \text{OH}^-$
(solution) (solution)
- The anions retained by the solid matrix of ion exchange resin can be eluted by using buffers of different strength

Classification of resins

- According to the chemical nature

1. Strong cation exchange resin
2. Weak cation exchange resin
3. Strong anion exchange resin
4. Weak anion exchange resin

- According to the source

- Natural: cation⁺ Zeolites, clay etc

anion -> Dolomite

- Synthetic: inorganic & organic resins Organic resins are the most widely used

Org ion exchange resins are polymeric resin matrix containing exchange sites.

The resin is composed of polystyrene & Divinyl benzene, polystyrene contains sites for exchangeable functional groups

Divinyl benzene acts as a cross linking agent & offers adequate strength i. mechanical stability

Functional groups present in different ion exchange resins

- Strong cation exchange resin SO_3H

Weak cation exchange resin \rightarrow COOH , OH , SH , PO^-H ,

- Strong anion exchange resin \rightarrow N^+R_3 , NR ,
- Weak anion exchange resin \rightarrow NHR , NH ,

Common ion exchange resins

Class or resin	Nature	pH range	Applications
Anion strong	Quaternary ammonium polystyrene	0-12	Fractionation of anions Alkaloids, vitamins, fattyacids
Anion-weak	Polyamine polystyrene or phenol H-CHO	0-9	Fractionation of anionic complexes, anions of diff valency vitamins, aminoacids



Common ion exchange resins

Class of resin	Nature	pH range	applications
Cation-strong	Sulfonated polystyrene	1-14	Fractionation of cations, inorganic separations, peptides, aminoacids, B vits
Cation weak	Carboxylic methacrylate	5-14	Fractionation of cations, biochemical separations, org bases, antibiotics

Structural types of ion exchange resins

- i. Pellicular type with ion exchange film
- 2. porous resin coated with exchange beads
- 3. macroreticular resin bead
- 4. surface sulfonated and bonded electrostatically with anion exchanger

resins

- i. Pellicular type with ion exchange film:
- The particles have a size of 30-40P with i-2p film thickness.
- These have very low exchange capacity
- Ion exchange efficiency: 0.01 - 0.1 meq/g of ion exchange resin.
- 2. Porous resin coated with exchanger beads: size 5-iop
- They are totally porous & highly efficient Exchange capacities 0.5-2 meq/g or ion exchange resin

Structural types of ion exchange resins

- 3. macroreticular resin bead: A reticular network of the resin is seen superficially on the resin beads
- They are not highly efficient & have very low exchange capacities
- 4. surface sulfonated and bonded electrostatically with anion exchanger:
- The particles are sulfonated, & they are bonded electrostatically with anion exchanger resin.
- They are less efficient & have low exchange capacity
- Exchange capacity is 0.02meq/g of exchange resin.

Physical properties of resins

When less cross linking agent is present, they are less rigid, but swell more

- Separation will not be efficient as exchange of functional groups does not take place due to wide pore
- Hence an optimum quantity of cross linking agent should be added to the polymeric ion exchange resin for the separation to be effective.

Practical requirements

- 1. column material & dimensions:
- Glass, stainless steel or polymers which are resistant to strong acids & alkalies
- Length: diameter ratio 20:1 to 100:1

Selection of ion exchange resin

- Depends upon
- 1. type of the ions to be separated - cations or anions 2. nature of the ions to be separated- strong or weak
- 3. efficiency of the resin: measured by ion exchange capacity
Ion exchange capacity is the total ion exchange capacity in terms of the exchangeable functional groups expressed as meq/g of the ion exchange resin
- $\text{m.eq/g} = 1000/\text{eq.wt}$
- 4. particle size of the resin: 30-100 mesh or 100-200
- 5. structural type of the resin: porous, pellicular etc
- 6. Amount of cross linking agent present: which decides swelling of the resin.

Packing of the column

- *Wet packing*
- The resin is mixed with the mobile phase & packed in the column uniformly
- The sample to be separated is dissolved in the mobile phase and introduced all at once into the column.

Mobile phase

- Organic solvents are less useful & they are not used at all.
Only diff strengths of **acids, alkalies & buffers** are used as eluting solvents
- Eg: 0.1N HCl, 1N NaOH, phosphate buffer acetate buffer, borate buffer, phthalate buffer .etc.,

Development of the chromatogram & elution

- i. isocratic elution technique
- 2. gradient elution technique

Analysis of the elute

- Spectrophotometric method
 - Polarographic method
 - Conductometric method
 - Amperometric method
 - Flame photometric method
- Radiochemical methods (GM counter, ionization chamber method)

Regeneration of the ion exchange resin

The ion exchange resin after separation may not be useful for next separation as exchangeable functional groups are lost

But due to cost of the ion exchange resins they cannot be disposed off

Hence **reactivation, regeneration**

- Regeneration makes the used ion exchange resin to be as efficient as a virgin resin.

Regeneration: replacement of the exchangeable cations or anions present in the original resin

Hence regeneration of the cation exchange resin is done by the charging the column with strong acid like HCl

- Vice versa

Factors affecting ion exchange separations

- i. Nature & properties of ion exchange resins:- Cross linking & swelling
- 2. Nature of exchanging ions:
- **A) Valency of ions:** at low cone & ordinary temp , extent of exchange increases with increase in valency
$$\text{Na}^+ < \text{Ca}^{2+} < \text{Al}^{3+} < \text{Th}^{4+}$$

Applications

Softening of water

Demineralisation or deionisation of water

Purification of some solutions to be free from ionic impurities

Separation of inorganic ions

Organic separations: mixture of pharmaceutical compounds can be separated

Biochemical separations like isolation of drugs or metabolites from blood, urine etc

Concentration of ionic solutions