Environmental protection in chemical technology

Faculty of chemistry

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Exercise no. 4

Ion exchange processes in water treatment

Ion exchangers by common definition are insoluble solid materials which carry exchangeable cations or anions. These ions can be exchanged in stoichiometrically equivalent amount of other ions of the same sign when the ion exchanger is in contact with an electrolite solution.

Ion exchange resins (called ion exchangers too) are insoluble polyelectrolytes having the ion exchange functional groups incorporated to a crosslinked polymer matrix. Ion exchange resins are classified as cation exchangers, which have positively charged mobile ions available for exchange, and anion exchangers, whose exchangeable ions are negatively charged. Resins can be classified as strong or weak acid cation exchangers or strong or weak base anion exchangers. About 90 % of all ion exchange resins are based on a crosslinked polystyrene matrix.

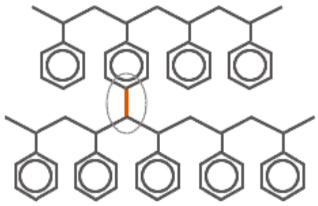


Fig.1 Cross-linked polystyrene

Most ion exchange resins are obtained by suspension polymerization in which spherical beads polymers are obtained. The beads can be gel or porous, see Fig.2. Next functional groups (strong base, weak base, strong acid or weak acid) are incorporated into its matrixes which are responsible for ion exchange process. Resins are characterized by surface areas, pore size and volume distribution, water regain (wettability) and functional groups capacity.

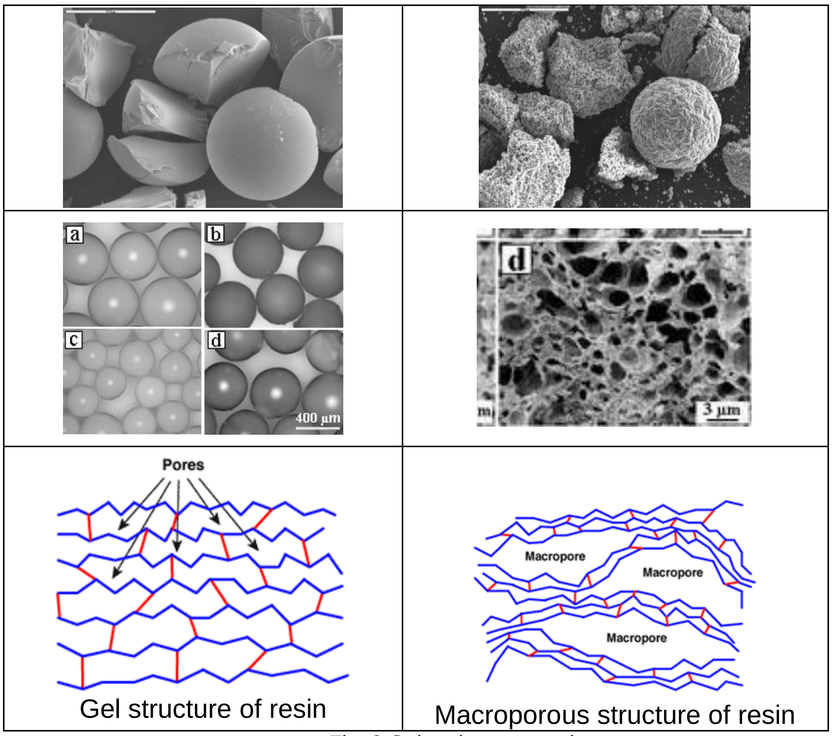
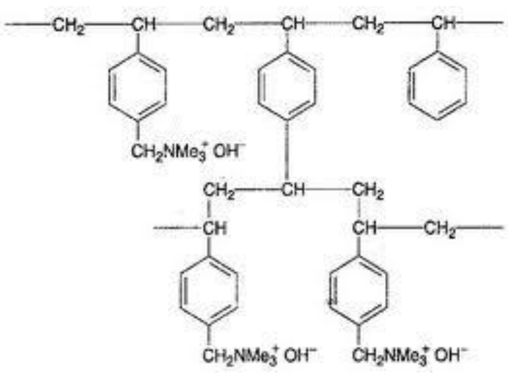
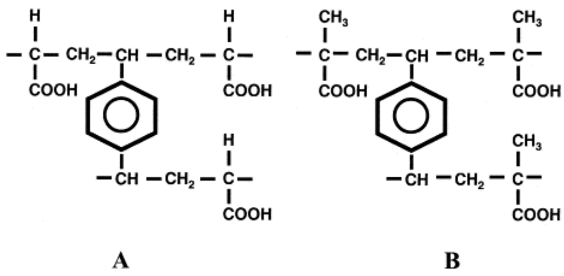


Fig. 2 Gel and porous resins



Strong base ion exchanger (SB)



Weak acid ion exchanger (WA)

Fig. 3 Strong base and weak acid ion exchangers

Ion exchange resins are used in a variety of specialized applications such as chemical processing, pharmaceuticals, mining, chemical processing – catalysis, waste treatment, pharmaceuticals and food beverage processing.

Ion exchange processes are used among other in:

- water treatment processes: for drinking water treatment, desalination of water and the softener, the water treatment process such as power plants,
- in hydrometallurgical processes for the recovery of metals such as precious metals and their concentration.

During the process of ion exchange an anion exchange resin bind an anion:

$$\text{R-NH}_3^+\text{OH}^- + \text{Na}^+\text{Cl}^- \Rightarrow \text{R-NH}_3\text{Cl} + \text{Na}^+\text{OH}^-$$

But cation are exchanged on cation exchange resin and is in the form of corresponding acid salt:

$$\text{R-COO}^-\text{H}^+ + \text{Na}^+\text{OH}^- \Rightarrow \text{R-COO}^-\text{Na}^+ + \text{H}_2\text{O}$$

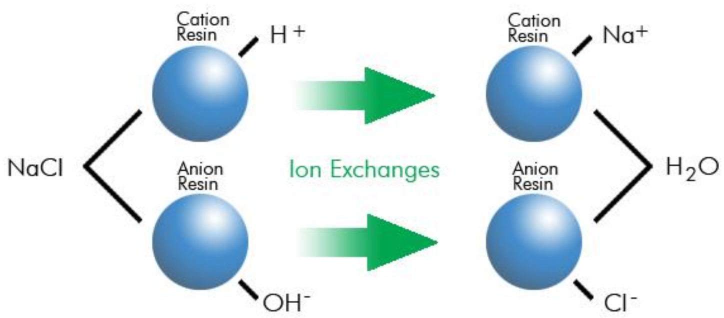


Fig. 4 Scheme of NaCl loading on ion exchange resins

Cation exchange resin conducted in the form of a salt can be completely regenerated with an acid and anion exchange resin with a hydroxide. The ion exchange process is a process of ion exchange equilibrium, and the phenomena occurring during the exchange takes place at the interface between solid and aqueous phase ion.

The purpose of this exercise is to evaluate the ability of ion exchange in a cascade columns system of two commercial anion and cation ion exchange resins from an aqueous solution of NaCl.

Manual instruction

1. Prepare a solution of 0.1 N NaCl in distilled water.
2. Assemble the system of two ion-exchange columns in a cascade: an effluent from one column is a feed stream for the second column according to Fig. 5.
3. Put swollen ion exchangers in different swelling water tube (a glass vial with a fritted glass bottom), balance them, and centrifuge for 5 min at 3000 rpm.
4. Weigh swollen amount of ion exchangers after centrifugation i.e. (Resin 1 - 11,2g, Resin 2 - 4,2 g), respectively.
5. Resin 1 should be placed in a column 1 and Resin 2 should be placed in a column 2.
6. Mark the columns, mark the hight of resin beads.
7. Next wash separately the columns with about 100 ml of distilled water.
8. Check the conductivity and pH of water effluents from columns, note in table 2.
9. Pour a feed solution, ie 50 ml solution of 0.1 N NaCl in water to the column 1, and pass through a bed of ion exchanger with a flow rate of 1 drop per second.
10. Collect the effluent into a measuring cylinder of volume 50 ml.
11. Mix the contents of the cylinder with a rod.
12. Measure the conductivity and pH of each new batches of leakage from the columns, note them in table. 3.
13. Remember - Effluent from the column 1 is the feed solution to the column 2.
14. Effluent from the column 2 is removed.
15. Do not wash the measuring cylinder.
16. The process should be complete when the value of conductance and pH reache a close values.
17. Complete table 1.
18. Next perform regeneration of ion exchangers.

Regeneration of ion exchange resins

The first operation to be performed is the regeneration of ion exchange resins. They work in the hydrogen - hydroxide cycles. Regeneration of the cation exchanger is carried out with a 5% solution of hydrochloric acid, and the regenaration of anion exchange resins is carried out with a 5% solution of sodium hydroxide. Regeneration consists of passing the solutions through a bed of suitable ion exchange resins in columns.

During this exercise flow eluents are regulated in an appropriate setting of the tap an ion exchange columns. The eluate from the column leakage is controlled by conductivity. The appearance in the eluate from the column 1 strong electrolyte solutions, which are evidence of the regeneration process of regeneration. Conductivity allows an accurate assessment of the condition and stop the operation when the conductivity of the effluent from the column 2 reaches about 5 mikroS / cm.

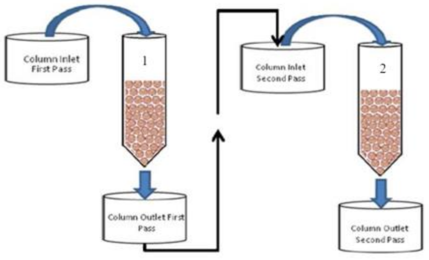


Fig. 5. Scheme of cascade columns used in the experiment

Additional information:

A. **Calculation of the dry weight of ion exchangers**

B. The weight of the wet swollen resin sample should be multiplied by the percentage of polymer in the swollen gel.

C. **Calculate the maximum capacity of ion exchange** resins (maximum capacity of ion exchangers in the conditions of the process) using the volume of solution sodium chloride passed over.

D. The results should be given in mmol/g wet and dry resin bed, in mmol/ml of resin swollen

E. Draw graphs pH =f(V NaCl), conductivity =f(V NaCl)

F. Write reaction equations *for the* reactions involved.

G. Students write the report in gropus.

The report sholud contain:

1. The results of measurements (pH and conductivity) in the table 1, depending on the volume of NaCl, figures pH and conductivity as a function of (V NaCl, ml).
2. The calculated ion exchange capacity of used ion exchange resins (the quantity of NaCl in grams (or mmol) divided by the mass of dry or wet ion exchanger.
3. Short description of the process.
4. Conclusions and comments.

Scope of knowledge required to pass before student's begin the exercise

1. The definition of ion exchange resins, ion exchange process, type of ion exchangers
2. The principle of ion exchange
3. Classification of ion exchangers: types, structure, properties
4. Characteristics of the ion exchange process
5. The use of ion exchange resins
6. The global resin manufacturers
7. Commercial ion exchangers

Literature

1. D. Muraviev, V. Gorshkov, A. Warshawsky, ION EXCHANGE, Marcel Dekker, New York Basel, 2000

2,. Any book on the ion exchange processess

Tab.1. CHARACTERISTICS OF ION EXCHANGERS

JONIT	Resin bed depth	Resin bed volume	ION-EXCHANGE CAPACITY, from SHEET		**WEIGHT**		**WATER REGAIN**	Moisture Retention	Content of ion exchange groups	ION-EXCHANGE CAPACITY respect to NaCl		
					WET	**DRY**				wet	dry	bed
	cm	ml	mmol/ml	mmol/g	g		g/g	%	mmol	mmol/g	mmol/g	mmol/ml
RESIN 1 PUT THE NAME			1,3				0,92	48-54				
RESIN 2 PUT THE NAME			4,2				1,00	45-55				

Water regain determine from the relation wet dry%

$$W = \frac{m_{wet} - m_{dry}}{m_{dry}} = \frac{m_{wet}}{m_{drya}} - 1, \left[\frac{gH_2O}{g}\right]$$

W – water regain [g H_2O/g dry resin]
m_{wet} – mass of swollen resin after centrifugation [g]
m_{dry} – mass of dry resin [g]

The content of the polymer in the swollen gel $\% = \frac{m_{dry}}{m_{wet}} \times 100\%$

Table: 2

	Conductivity UNITS	pH
H_2O		
NaCl, 0,1M		
HCl, 0,1M		

Table: 3

NO	sample	EFFLUENT 1 (**RESIN 1** PUT THE NAME)		EFFLUENT 2 **(RESIN 2** PUT THE NAME**)**	
		Conductivity UNITS	pH	Conductivity Units	pH
Water after washing the column					
VOLUME (summary) 0,1M NaCl, ml					
1	50				
2	100				
3	150				
4	200				
5	250				
6	300				
7	350				
8	400				
9	450				
10	500				