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Water and Waste Laboratories

Laboratory Worksheet C11 : **Ion Exchange Resin Column Test**. by *WS LAM*

Objective : To evaluate Ion Exchange operation cycle behavior.

Introduction

An ion exchange resin is a polymer (normally styrene) with electrically charged sites at which one ion may replace another. Natural soils contain solids with charged sites that exchange ions, and certain minerals called zeolites are quite good exchangers. Ion exchange also takes place in living materials because cell walls, cell membranes, and other structures have charges. In natural waters and in wastewaters, there are often undesirable ions and some of them may be worth recovering. For example, cadmium ion is dangerous to health but is not present usually at concentrations that would justify recovery. On the other hand, silver ion in photographic wastes is not a serious hazard, but its value is quite high. In either case, it makes sense to substitute an ion such as sodium for the ion in the wastewater.

Synthetic ion exchange resins are usually cast as porous beads with considerable external and pore surface where ions can attach. Whenever there is a great surface area, adsorption plays a role. If a substance is adsorbed to an ion exchange resin, no ion is liberated. Testing for ions in the effluent will distinguish between removal by adsorption and removal by ion exchange. Of course, both mechanisms may be significant in certain cases, and mass balances comparing moles removed with moles of ions liberated will quantify the amounts of adsorption and ion exchange.

Ion exchanger must have a balance of charges and interaction of ions. In addition, the selectivity of each ion is the basis for development of an exchanger. The Donnan Exclusion allows for acceptance of a predominating ion. This comes from the ability of the resin to exclude one ion over another. Also, there is a screening affect that takes place that rejects large ions or polymers. Moreover, the transport of a substance through a column is a reflection of the affinity of the ions present. While there are numerous functional groups that have charge, only a few are commonly used for man-made ion exchange resins. These are:

- -COOH which is weakly ionized to -COO^-
- -SO_3H which is strongly ionized to -SO_3^-
- -NH_2 that weakly attracts protons to form NH_3^+
- -secondary and tertiary amines that also attract protons weakly
- -NR_3^+ that has a strong, permanent charge. (R stands for some organic group)

Column tests are performed to study the shape of the breakthrough curve and also the regeneration pattern to provide the experimenter with the optimal parameters for eventually design of full scale treatment system.

Applications of Ion Exchange

Today, there are many different industrial applications of ion exchangers, mostly for purification. Some examples are:

Water and Waste Treatment

- Softening (removal of hardness), demineralizing, silica removal, and alkalinity reduction
- Removal of cations and anions from boiler feeds
- Deionizing water Treatment of trade effluents

Purification

- Recovery of organic and inorganic substances
- Separation of ion mixtures

Apparatus and Materials

- Spectrophotometer
- Volumetric flasks
- Measuring cylinders
- Breakers
- Ion Exchange Resin (Amberlite 1R 120, Cation Exchange Resin)
- Glass Column
- Influent Solution (10000 mg/L $CuSO_4$ soultion, max. Abs at 816nm)
- Regenerant (2N HCl)

Procedure

Resin Column Preconditioning

1. Weigh about 3g of ion exchange resin and pack it into the column.
2. Flush the resin with 150 mL regenerant in a flow of about 10mL per min.
3. Stop the flow to leave about 2mL of regenerant above the resin bed top.
4. Allow the regenerant to react with the column for one hour.
5. Fill the column with distilled water and flush the column until the effluent water is neutral, i.e. pH = 7.
6. The resin column is then ready for use.

Standard Curve calibration

1. Prepare a set of standard sorbate solutions of known concentrations.
   (250, 500, 1000, 5000, 10000 mg/L for $CuSO_4$)
2. Measure the absorbances of the standards using a spectrophotometer.
3. Plot absorbances against standard sorbate concentrations to obtain a calibration curve for unknown sorbate concentration measurements.

Column Test (breakthrough and regeneration)

1. Weigh about 3g of ion exchange resin and pack it into the column. Record the resin name, type and its expected sorptive capacity.

2. Fill the column with distilled water and flush the column until the effluent water is neutral, i.e. pH = 7.

3. Let the distilled water flow until the water level drops to 2mL above the top of the resin bed.

4. Carefully fill the column with influent sample and prepare to collect effluent sample.

5. Never allow the resin column to get dry. Keep the influent level well above the resin bed.

6. During the experiment, the effluent volume should be measured accurately.

7. Measure the absorbance of the influent sample.

8. Start and adjust the effluent flow to about 5 mL/min., take 5 mL of effluent and measure its absorbance.

9. At every bed volume of effluent, collect 5 mL of effluent and determine its absorbance at until exhaustion.

10. After exhaustion, fill the column with regenerant to begin column regeneration with a flow of 5 mL/min.

11. Continue collecting 5 mL of effluent and determine its absorbance every bed volume of effluent until the effluent drops no further.

12. Construct the column breakthrough and regeneration curve.

Discussions

1. Determine the sorptive capacity of the resin and estimate the regeneration cycle.

2. Ion exchange can be viewed as a kind of adsorption process. Does it necessary to perform the following two kind of tests for ion exchange resin ? Why ?

   a. Adsorption Isotherm.
   b. Adsorption Rate.