Ion Exchange

CBEMS 140B

UCI Department of Chemical Engineering and Materials Science

GENERAL OVERVIEW

Ion exchange is a natural process in which ions held on the surface of a solid displace other ions, of similar and equivalent electrical charge, from a solution in contact with the solid. The displaced ions become attached (i.e. held by electrostatic attraction) to the surface, while those originally on the surface go into solution. This process of exchange continues until the relative concentration of the two types of ions, on the surface and in solution, reach equilibrium. The process is reversible, the direction of the exchange depending upon these relative concentrations.

The simplest example of practical ion exchange is in the softening of water, when Ca²⁺ ions in the water (causing hardness) are exchanged for Na⁺ ions on the exchange material. When equilibrium is reached, i.e. when the exchange capacity of the material is exhausted, it can be regenerated by applying a concentrated solution of a sodium salt, usually sodium chloride, to restore Na⁺ ions on the surface.

By the use of suitable ion exchange materials in two or more stages it is possible to remove all dissolved salts from solution - the process of demineralization.

The ion exchange apparatus described in this manual enables both softening and demineralization to be studied. Besides these uses in the treatment of water supplies, ion exchange processes are also widely employed in industry.

HISTORY

The phenomenon of ion exchange was discovered in the middle of the nineteenth century, when H.S.M. Thompson observed that ammonium sulphate fertilizer applied to soil emerged as a solution of calcium sulphate. In 1850-54 the process was closely studied by J.T. Way, Consulting Chemist to the Royal Agricultural Society in England. He found that many forms of ion exchange occurred in various soils, and that the materials involved were complex hydrated aluminosilicates, known as zeolites. Way was able to prepare artificial aluminosilicates with ion exchange properties.

The first practical use of ion exchange in water treatment was by the German R. Gans in 1905, when he used a synthetic material to soften water, regenerating it with sodium chloride.

Since then, the range of synthetic materials has been greatly extended, with the introduction of sulphonated coals by Liebknicht in 1934, phenol-formaldehyde resins by Adams and Holmes in 1935 and polymerization resins based on styrene, by d'Aielio in 1944. The properties sought in the newer synthetic materials include physical and chemical stability as well as greatly increased exchange capacities. They can be made as either cation- or anion-exchangers and by using both types in series, demineralization is

possible. The first commercial ion-exchange demineralization plant was installed in 1937 at a brewery in Guildford, England.

EQUIPMENT DIAGRAM

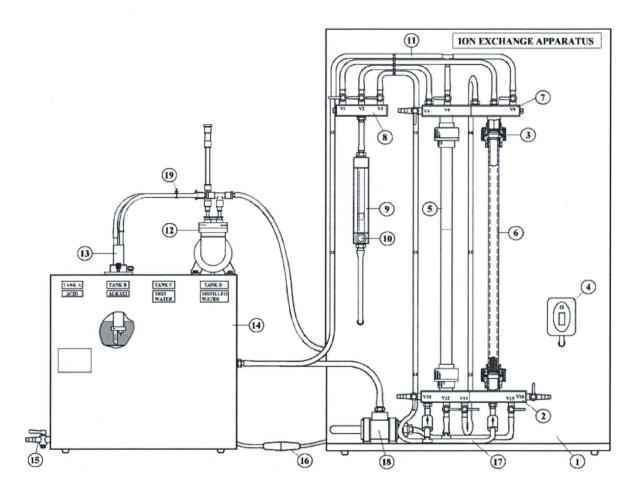


Figure 1: Ion Exchange Apparatus

DESCRIPTION

Where necessary, refer to the drawings in the Equipment Diagram section.

OVERVIEW

All numerical references relate to Figure 1. Also refer to the schematic diagram showing the valve positions in Data Sheet III (below).

The apparatus, which is designed for experiments on both water softening and demineralization, is supplied split into two major components; a backboard (1) incorporating the main process components and a sump tank arrangement (14) for storing and pumping the associated liquids.

Ion exchange takes place inside two vertical transparent columns (5 & 6), of approximately 16mm internal diameter, mounted on the backboard via manifolds at the top (7) and bottom (2). In use the left hand column (5) contains Cation exchange resin (golden colored granules) and the right hand column (6) contains Anion exchange resin (white colored granules).

The manifolds at the top (7) and bottom (2) of the columns are fitted with lever operated isolating valves which allow the flow to be directed through one or both columns, in either direction, to suit the process requirements. Screwed connectors fitted with 'O' ring seals (3) allow the columns to be removed for cleaning or changing the type of exchange resin. The liquids to be passed through the exchange columns are stored in the sump tank arrangement (14) to the left of the apparatus and supplied via a pump (12) and flowmeter (9). The liquids are selected by lifting and traversing the sliding tube arrangement (13) at the front of the sump tank. The pump is operated using the electrical switch (4) at the right hand side of the process backboard and is connected via an in-line electrical connector (16). The flexible tube from the pump outlet to the selector assembly returns excess liquid to the feed tank for reuse. An adjustable pinch valve (19) on the bypass tube is adjusted to give the correct flow conditions. This valve must not be fully closed to avoid excess pressure in the system.

A flow control valve (10) at the base of the flowmeter allows the flow of water, regenerating solution etc. to be adjusted as required. A distribution manifold (8) above the flowmeter allows the pumped liquid to be supplied to the top of the Anion column, to the top of the Cation column or to the base of both columns as required by the process, simply by opening the appropriate lever operated valve.

After passing through the columns the treated water, exhausted regenerating solution or wash water is fed to an effluent storage tank at the rear of the sump tank arrangement via flexible tube (11) from the top of the columns or flexible tube (17) from the bottom of the columns. This tank incorporates a lever operated valve (15) to facilitate draining. Lever operated valves (V4, V10 or V16) allow samples of water to be collected for analysis if required.

The equipment includes a battery operated digital conductivity meter (19) that is connected to an inline sensor (18) in the return line (17) to the effluent tank. This allows the quality of the water emerging from the ion exchange column(s) to be monitored.

The various processes involved in the experiments are as follows:

- a. Water to be softened, which will pass downwards through the Cation exchanger only.
- b. Water to be demineralized, which will pass downwards through the Cation exchanger and then upwards through the Anion exchanger.
- c. Regeneration solutions (followed by distilled or demineralized water for flushing), which are stored in separate tanks, and will pass downwards through either the Cation or the Anion exchange column.
- d. Water (preferably distilled or demineralized) which will pass upwards through either column to flush out any sediment and to release any air trapped in the resin.

Four separate experiments, (Experiment A, B, C and D) described in detail in separate sections below, will be carried out during this lab exercise.

OPERATION

Operating the Equipment

Please refer to the Laboratory Teaching Exercises for details on operating the equipment.

Operation of the Conductivity Meter

The conductivity meter is supplied separately and is designed to sit on the bench top alongside the equipment. The conductivity meter is powered by an internal 9 volt PP3 Alkaline battery.

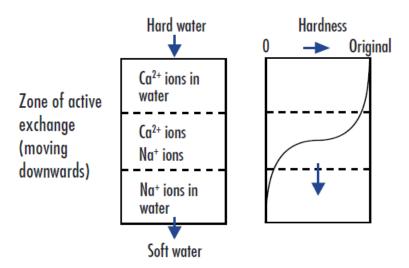
An inline conductivity sensor is installed at the outlet from the bottom manifold so that it can monitor the conductivity of the water following the ion exchange process. The lead from the inline sensor is connected to the socket marked INPUT at the top of the conductivity meter. To operate the conductivity meter, press the POWER button and adjust the position of the range switch until the meter indicates the Conductivity and Temperature. Dashes in the display indicate that the range switch is in the wrong position and should be adjusted to suit. The meter gives a direct reading of conductivity, corrected for temperature, in units of micro Siemens (μ S) or milli Siemens (μ S) depending on the position of the range switch. For further information on the conductivity meter refer to the TA or instructor.

Water Softening Theory

The most usual ion exchange material employed in water softening is a sulphonated styrene-based resin, supplied by the makers in the sodium form. This resin has a strong affinity for calcium and magnesium ions, and will also remove ferrous ions after the more or less complete removal of calcium and magnesium.

Softening can be carried out as a batch process by stirring a suspension of the resin in the water for a period until equilibrium, or an acceptable level of hardness, is reached.

However, it is more convenient to operate a continuous flow process by passing the water slowly downwards through a column of resin beads. The exchange reaction takes place rapidly enough for the upper layers of the bed to approach exhaustion before the lower layers being able to exchange ions. There is thus, a zone of active exchange which moves down the column until the resin at all depths becomes exhausted. The position at an intermediate stage can be illustrated as shown below.



When the zone of active exchange reaches the bottom of the column, the emerging water begins to show an increasing hardness. This is the breakthrough point, when it becomes necessary to regenerate the resin with a strong sodium chloride solution.

Regeneration Theory

Theoretically, for every millequivalent (meq) of hardness as CaCO₃ removed from the water under treatment, one millequivalent of NaCl is required for regeneration, i.e. 1 g of hardness as CaCO₃ removed requires 1.17 g NaCl for regeneration (equivalent weights: CaCO₃ 50.0; NaCl 58.5). In practice it is not possible to achieve complete regeneration with this quantity of NaCl, since this would require an unacceptable long

contact period. Larger quantities of NaCl are therefore used, generally twice or more the theoretical amount. The regeneration efficiency is thus around 50%.

A high level of regeneration gives a resin with a high exchange capacity approaching its theoretical, but it is uneconomic to operate at such a rate that this capacity is fully used in softening. In other words, a high regeneration efficiency is associated with a low degree of column utilization, and vice versa. The practical operation of an ion exchange bed is therefore a compromise in which the regeneration efficiency and the column utilization are both in the region of 50%.

After regeneration, distilled or demineralized water is passed through the bed to wash out any remaining regenerant. Water to be treated by ion exchange must be free of suspended solids which would block the passage-ways, reduce flow rates and interfere with the exchange process. To remove fine solids which may get into the bed, and to release any air pockets, the column is backwashed periodically by an upward flow of water which fluidizes the bed and agitates the resin beads.

The rate of flow of water through the bed in softening is usually not more than 40ml/min per cm² of surface area of bed. Regeneration rates are about one tenth of this.

Demineralization Theory

The removal of all dissolved salts from water can be achieved by using a two-stage ion exchange process. The water is first passed through a strong cation exchanger working on the hydrogen ion cycle, when cations in the water are replaced by H⁺ ions, giving a solution of acids. This is then passed through an anion exchanger in the hydroxyl ion form, when the acid ions are replaced by OH⁻ ions, which with the H⁺ ions, produce water.

It is often sufficient to use a weakly basic anion exchanger, which will remove all anions except HCO^{3-} (due to dissolved carbon dioxide) and H_3SiO_4 (due to dissolved silica). For a higher quality product water, a strongly basic anion exchanger must be used as the final stage, but it is generally more economical to precede this with a weakly basic anion exchanger of high exchange capacity to remove the bulk of the anions, and a degassing tower to release CO_2 from solution. The strongly basic resin is then required only to remove silica and any residuals of other anions which may still be present. This process can reduce total dissolved solids to below 1 mg/L.

Demineralization can also be performed in a single stage by using a mixed bed of strong cation and anion exchangers. The water repeatedly comes in contact with the two resins alternately, and is ultimately of very high purity. To enable the two resins to be regenerated with sulphuric acid and sodium hydroxide respectively, they are first stratified with an upward flow of water, the anion resin being of lower density and

therefore carried to the top. After regeneration, the two resins are re-mixed by compressed air.

Resin Volume and Density

When dry ion-exchange resins are immersed in water the beads swell as a result of hydration of the fixed and counter-ions (i.e. the attraction of water molecules to the ions) and the repulsion between the fixed ions.

A distinction must therefore be made between the dry and wet volumes and densities of a resin. It is also important to wet a resin thoroughly, before placing in a test column, in order to avoid damage as a result of the swelling.

The density of a resin can also be given as the true density, i.e. mass per unit volume of the beads alone, or as the apparent density, i.e. mass per unit volume of the bed, including the voids. For typical resins the true wet density is usually between 1.1 and 1.3 g/cm³, and the apparent wet density between 0.7 and 0.8 g/cm³.

The inside diameter of the cation exchange column is 16.16 mm and the inside diameter for the anion exchange column is 16.18 mm measured using a high accuracy caliper on Dec 2013. For the calculations in this lab exercise it is accurate enough to assume that both diameters are equal and use the value 16.2 mm.

Exchange Capacity

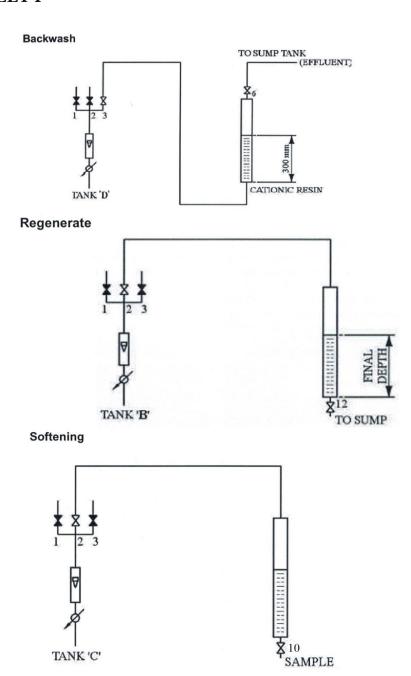
The exchange capacity of a resin is a measure of the quantity of ions which can be exchanged per unit mass of volume of the resin. The theoretical exchange capacity may be defined as the number of exchangeable ions which it contains per unit mass or volume. In practice it is not feasible to provide a long enough contact period for complete equilibrium to be attained, nor is it economic to regenerate fully. The practical exchange capacity is therefore rather less than the theoretical. It is expressed in various units, of which the most useful are probably millequivalents (meq) of exchanged ions per gram of dry resin, and meq/L of wet resin bed. In the softening of water it is also common practice to express the exchange capacity in terms of mass of CaCO₃ rather than millequivalents, the usual unit being kg CaCO₃/m³ of wet resin bed.

Data sheets for ion exchanger operation and experimental procedures

The following graphics are used to depict different valves in the figures below (Datasheet I, II and III).

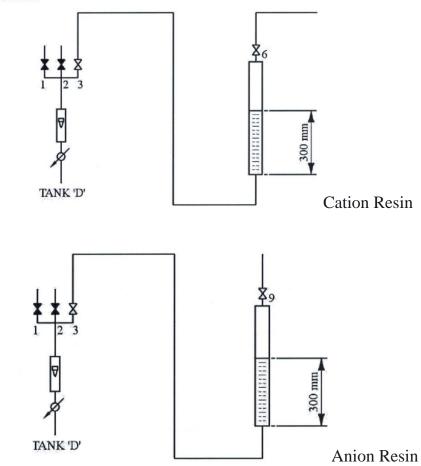


DATA SHEET I

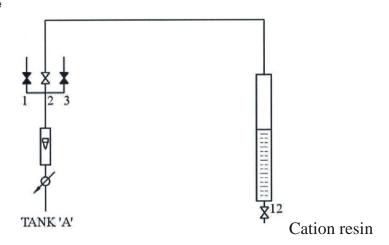


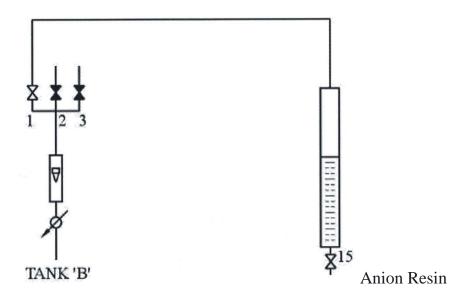
DATA SHEET II

Backwash

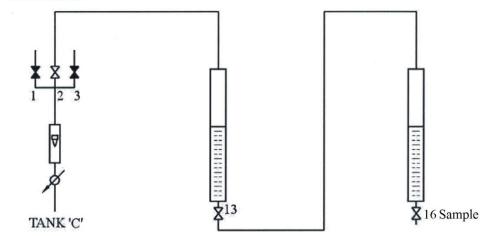


Regenerate

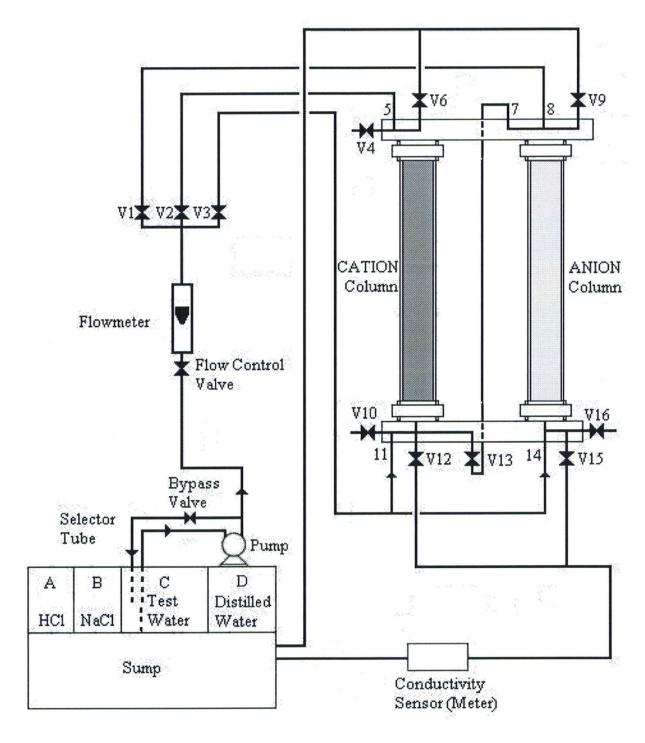




Demineralise



DATA SHEET III



Schematic Diagram of W9 showing valve positions

DATA SHEET IV

To determine the hardness of a sample of water by EDTA titration

The most common positively charged metal ions in natural waters are Ca^{2+} and Mg^{2+} . For this lab, you will assume that these are the only metal ions present in your water. Mg^{2+} and Ca^{2+} ion concentration can be found by titrating with 5 mM EDTA (ethylenediamine tetraacetic acid), a chelating agent.

Pipet 12 ml of water sample into an Erlenmyer flask. To the sample, add 5ml of 0.2M glycine and 3ml of 0.2M NaOH to buffer the solution at pH 10. Add 3 drops of Eriochrome black T (EBT) indicator which was prepared freshly by adding 0.2g EBT in 20ml methanol. Rinse and fill a clean automatic buret bottle with EDTA solution and record the initial volume after filling the buret. Titrate your water sample until the color changes from wine red to blue. Perform a quick titration to determine the approximate volume needed to reach the endpoint. In order to accurately identify the endpoint, where the last trace of red has been removed from the solution, you may want to add small volumes of city water to your titrated sample and practice reaching the endpoint. Once you have confidently identified the endpoint, save this sample as a reference for subsequent titrations.

Each EDTA molecule chelates one divalent ion:

Hardness of water = $mg CaCO_3 per L$ (Assume a Mg^{2+} as a Ca^{2+} ion in hard water)

$$= \frac{\text{Volume of 0.005M EDTA used (ml)} \times 0.005 \frac{\text{mol}}{\text{L}}}{1000 \frac{ml}{L}} \times 100 \frac{\text{g}}{\text{mol L}} \times 1000 \frac{mg}{g}$$

Increase water sample size if water is very soft.

EXERSICE A

Objective

To determine the exchange capacity of a cationic resin in the softening of water.

Procedure

Fill the test water reservoir with city water. Determine its hardness and compare result with data provided by the Irvine Water District (IRWD.com) annual water quality report. Make up 2 liters of 2% w/v NaCl solution by dissolving 40g NaCl in DI water. Place this solution in regenerant tank B. Fill tank D with deionized water. One of the taps in the lab supplies deionized water.

Backwashing

See Data Sheet I (Upward flow of water through the bed).

Backwashing removes any sediment from the bed, ensures that the resin beads are fully wetted and swollen and removes any air pockets which would interfere with the ion exchange process. The left hand cation exchanger column has been filled with cationic resin (golden colored granules) to a depth of ~150mm. If the level of resin appears to be significantly lower DO NOT attempt to add resin but consult with the TA or instructor. Select tank D, open valves V3 and V6 (close all the other valves), and backwash for five minutes at a flow sufficient to expand the bed by not more than 100%. Gradually turn off the flow and measure the final depth of the resin. Do not drain the bed as this would allow air to enter.

Regenerate

See Data Sheet I (Downward flow of salt solution through the bed).

Select tank B, open valves V2, V12 (and V10 if a sample is required and close all the other valves). Set flowmeter to not more than 50 ml/min. Regenerate column with two column volumes (CV) of NaCl solution (1 CV \approx 100ml for a column fixture height of 50 cm), turn off pump and switch to tank D (distilled water), continue the flow through the bed until the sodium chloride concentration in the effluent is equal to that of the distilled water, or at least very low or until 10 CV of DI water has passed through the column. Monitor the salt concentration by its conductivity.

Softening

See Data Sheet I (Downward flow of hard water through the bed).

Select tank C, open valves V2 and V10 (close all the other valves). Set flowmeter to between 50 and 70ml/min. After flow of 1 column volume (≈ 100 ml), start collecting

samples of 50ml at every 3 column volumes of flow. Continue the softening until the hardness of the effluent rises above 100mg/L as CaCO₃.

Results and Calculations

Plot the hardness readings against the volume of water treated and note the breakthrough point at which the increase in hardness starts. Calculate the milligrams of hardness as CaCO₃ removed from the water up to the breakthrough point. Graphically, this is given by the area between the curve plotted and the horizontal line, representing the original hardness of the water. Knowing the wet volume of the resin bed, calculate the exchange capacity of the resin as meq/mL of wet volume and in units of kg CaCO₃/m³.

Wet volume of resin bed (m³) =
$$\frac{\pi \times (16.2 \times 10^{-3})^2}{4} \times \text{Final Depth of the resin (m)}$$

Exchange capacity (kg
$$CaCO_3/m^3$$
) = $\frac{mass\ of\ CaCO_3}{Wet\ volume\ of\ resin\ bed}$

EXERSICE B

Objective

To determine the regeneration efficiency of an ion-exchange softening system.

Procedure

After completion of the experiment 'SOFTENING', i.e. when the hardness exchange capacity of the resin has been used up to just beyond the breakthrough point, carry out regeneration with 500ml of 2% w/v salt solution (NaCl). Collect the effluent via valve no. V10 in 50 ml subsequent aliquots.

Determine the sodium ion concentration in the collected regenerant by measuring Na⁺ (after dilution to working concentration) by flame photometry. Knowing the volume of solution collected calculate the meq of NaCl which has passed through the bed. Hence by subtraction from the original quantity of NaCl applied (10 g), determine the meq of NaCl actually used in regeneration.

Compare this with the theoretical quantity of NaCl equivalent to the amount of hardness removed in the experiment 'SOFTENING' and hence, calculate the efficiency of regeneration as a percentage.

The efficiency so calculated is based on the NaCl actually used in regeneration. However, in operation it is not possible to apply this quantity precisely, and an excess has to be applied. Further experiments could be carried out using different quantities of NaCl

for regeneration in solution from 5 to 10% in strength, in order to determine practical regeneration efficiencies. However, due to time constraints, these experiments will not be conducted during this lab exercise.

Results and Calculations

Calculate the regeneration efficiency using the equations below. Make sure you are consistent with units (for example, do not mix meq or g/L).

$$Theoretical\ exchange\ capacity = \frac{\text{theoretical\ quantity\ of\ NaCl\ equivalent\ to\ hardness\ removed}}{\textit{Wet\ volume\ of\ resin\ bed}}$$

Actual exchange capacity

 $= \frac{\text{quantity of NaCl passed through resin bed during regeneration - amount of NaCl collected}}{\textit{Wet volume of resin bed}}$

Regeneration efficiency = $\frac{Actual\ exchange\ capacity}{Theoretical\ exchange\ capacity}$

EXERCISE C

Objective

To study the demineralization of water and to determine the exchange capacities of a hydrogen ion cation exchanger and an anion exchanger.

Procedure

Backwashing

See Data Sheet II (Upward flow of water through the bed).

Each column should be separately backwashed in the manner described in experiment 'SOFTENING'. In each case, the rate of backwashing should be controlled to give not more than 50% expansion of the bed. Measure the final depths of the two beds.

Regeneration (CATION)

The left hand cation exchange column will need to be treated to exchange sodium ions with hydrogen ions to ensure that the cation exchanger resin is in the hydrogen ion form. This can be done by filling tank A with 2 L of 2% HCl solution and let 5 column volumes of this HCl solution pass through the cation exchange column as in the Regeneration step (Data sheet II). Use a flow rate of no more than 50 mL/min. Collect all

effluent via valve no. V10. Use the time while the cation exchange column is changing from sodium to hydrogen form to prepare all other solutions.

The anion column has been filled to a depth of ~100mm with anion exchange resin (white colored granules) in the hydroxyl form. If the level of resin appears to be significantly lower DO NOT attempt to add resin but consult with the TA or instructor.

Empty tank B and fill with 2 liters of a 1% sodium hydroxide solution. Fill tank C with 10 liters of city water. Fill tank D with deionized water.

Compare the conductivity of city water to standard solutions and calculate the total strength of city water in meq/liter. This will be used in calculating the exchange capacities of the two resins.

Additional equipment required:

- pH Meter
- Stop Clock

Regeneration (CATION) continues

See Data Sheet II (Downward flow of acid through the cation bed).

Select tank D, open valves V2 and V10 (close all the other valves) and follow the acid with deionized water to flush out any surplus acid. Check pH of effluent and continue flushing until pH has returned to above 5.0 or until 10 CV of DI water has passed through the column. Record pH value of your effluent.

Regeneration (ANION)

See Data Sheet II (Downward flow of sodium hydroxide through the anion bed).

Select tank B, open valves V1 and V16 (close all the other valves, collect all effluent and use it to neutralize part of your acidic waste). Set flowmeter to not more than 50 ml/min. Regenerate column with two column volumes (CV) of NaOH solution (1 CV \approx 100ml for a column fixture height of 50 cm), turn off pump and switch to tank D (distilled water), continue the flow through the bed until pH of the effluent has returned to below 9.0 or until 10 CV of DI water has passed through the column. Record pH value of your effluent.

Demineralization

See Data Sheet II (Downward flow of test water through both columns in series).

Select tank C, open valves V2, V13 and V16 (close all the other valves). Set flow rate to between 50 and 70ml/min. Monitor conductivity and pH value of your effluent. After flow of 1 column volume (≈ 100 ml), start recording total flow volume and record conductivity and pH readings at 5 minute intervals. At 20 minute intervals draw off samples from valve V10 and measure the pH value.

Note the total flow volume when the conductivity of the demineralized water begins to rise, i.e. the breakthrough point at which one of the resins has become exhausted. As soon as possible after this point, take another small sample from valve no. V16 and measure its pH. If this pH is higher than the values previously recorded, it indicates that the cation exchanger has become exhausted. It is advisable to confirm this by drawing one or two further samples for pH determination.

The experiment should be stopped at this point, and the exchange capacity of the cation exchanger can be calculated. The exchange capacity of the anion exchanger will not be determined in this experiment.

If, on the other hand, the pH of the cation exchanger effluent continues at a low value, the rising conductivity of the final effluent indicates that the anion exchanger is exhausted, and its capacity can be calculated.

In the latter event, the exchange capacity of the cation exchanger can be determined by continuing the flow of water through the first column only, collecting the water which passes through it and measuring pH values until the breakthrough point, when the pH begins to rise. However, if the anionic column is exhausted first, the exchange capacity of the cation exchanger will not be determined in this lab.

Results and Calculations

In the demineralization experiment, the breakthrough point is detected by readings of pH (for the cation exchanger) or conductivity (for the anion exchanger) instead of by direct measurement of concentrations as in the softening experiment. In order to calculate the exchange capacities in terms of millequivalents, it is necessary to convert pH or conductivity readings to meq/liter.

a. To convert pH values to meq/L

If the pH reading is x the hydrogen ion concentration is approximately 10^{-x} moles/liter. This is equivalent to a hydrogen ion concentration of 10^{3-x} meq/L.

b. To convert conductivity values to meg/L

For water with a given content of salts, the electrical conductivity is closely proportional to the concentration of total dissolved solids. Although these solids consist of several

salts of varying electrolytic properties, it is sufficiently accurate to assume that electrical conductivity is also proportional to the total concentration in terms of meq/liter.

The constant of proportionality was established by determination of the electrical conductivity and the strength of meq/L of the raw water. Hence the electrical conductivity of the demineralized water can be converted to meq/L. In any event these figures should be very low.

Exchange capacities can now be calculated according to previous equations with the knowledge of the final depths of the cation and anion exchange resins (for calculation of the wet volume).

EXERCISE D

Objective

To determine the regeneration efficiency of a cation resin or an anion resin.

Procedure

CATION RESIN (if cationic exchanger is depleted in Experiment C)

Tank A should have ~1.5 L of 2% hydrochloric acid. If not, fill tank A with 500ml of a 2% hydrochloric acid.

Select tank A and open valves V2 and V10 (close all the other valves). Carry out regeneration with 500ml of 2% hydrochloric acid. Collect the effluent in 50ml subsequent aliquots and analyze for hydrogen ion concentration by pH measurement. Note the final depth of the resin bed.

ANION RESIN (if anionic exchanger is depleted in Experiment C)

Note: Since the exchange capacities of cation resins are generally greater than those of anion resins, it is expected that the anion resin will be the first to be exhausted.

Tank B should have over 1L of 1% sodium hydroxide solution left from experiment C. If not, fill tank B with 500 ml of a 1% sodium hydroxide solution.

Select tank B and open valves V1 and V16 (close all the other valves). Collect the effluent in 50ml subsequent aliquots and analyze for hydroxyl ion concentration by pH measurement. Note the final depth of the resin bed.

Results and Calculations

The theoretical and actual exchange capacities as well as the regeneration efficiency can be calculated using the same theories as in EXCERCISE B. The concentration of hydrogen ions and hydroxyl ions in solution can be calculated from pH measurements.

CLEAN UP

Backwashing

See Data Sheet II (Upward flow of water through the bed).

Each column should be separately backwashed in the manner described in experiment 'SOFTENING'. In each case, the rate of backwashing should be controlled to give not more than 100% expansion of the bed.

Close all valves

Empty Tanks

Pump out tank A and tank B completely using the hand pump and a 25 ml pipet at the end and neutralize all pH waste. Rinse and empty these two tanks with city water twice followed by DI water once. Empty tank C (city water) and tank D (DI water).