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Studies in the Reaction–Separation Method for the Preparation of Barium Chloride from Barite Using Ion Exchange

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ABSTRACT

We report the application of an ion-exchange process as a reaction–separation strategy for the preparation of barium chloride from barite ore and sodium chloride. Experimental studies were carried out to evaluate the process efficiency and purity/yield of barium chloride using a strong acid cation-exchange resin, Tulsion T-42. The effects of various process parameters such as concentration of barium sulfide and concentration of sodium chloride were investigated, and optimization of the experimental variables was attempted. The results indicate the developed strategy to be attractive and an alternative route to existing processes. The methodology developed has large potential for the inorganic chemical process industry in general.

Key Words. Ion exchange; Barite; Barium chloride; Sodium sulfide; Reaction–separation

INTRODUCTION

Barium chloride has been extensively used in boiler feedwater treatment and also in the brine purification section of the chlor-alkali industry for the removal of sulfate ions. It also finds use in the making of barium-based colors. Barium chloride is mainly produced by the reaction of hydrochloric acid and

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barium carbonate or barium sulfide. If barium sulfide is used, highly toxic hydrogen sulfide gas is evolved, which requires further treatment. The use of hydrochloric acid also poses corrosion problems. Barium carbonate or barium sulfide is prepared by the carbon reduction of barite mineral (barium sulfate) using a high temperature rotary kiln operation known as the black ash process (1–4). Barium sulfide is soluble in water. It is double decomposed with sodium carbonate to obtain sodium sulfide and barium carbonate. Although the chemical method is capable of efficiently converting barium salts to barium chloride, the process involves the use of costly chemicals, problems of handling toxic materials, and corrosion. The by-products and gases generated in this process create effluent treatment problems (5, 6). The reported use of chemical methods for the preparation of high grade barium chloride (7–13) is mostly in patented form. Some studies on the recovery of the chemicals involved have been reported (14–16). The use of natural and synthetic zeolite for the preparation of barium chloride from barium sulfide and sodium chloride has been reported (17). Synthetic ion-exchange resins have been used for the preparation of barium hydroxide or nitrate using barium sulfide (18–22). Preparation of some other salts of barium has also been reported (23, 24).

Ion-exchange processes are most commonly employed for separations and in the area of catalysis. However, their application in the preparation of inorganic chemicals in general and of barium chloride in particular has not been investigated in detail. Ion exchange could be useful from the industrial applications point of view due to 1) the simplicity of the process, 2) the use of moderate operating conditions along with ease of handling, and 3) the possibility of cyclic operation. In the present work our objective is to report the preparation of two inorganic chemicals, barium chloride and sodium sulfide, by using a strong acid cation-exchange resin, Tulsion T-42. Equilibrium and column studies were carried out to investigate the effect of different parameters on the purity and yield of the two chemicals.

Ion-exchange resins belong to a larger class of reactive polymers. Although the functional groups on the resin matrix are most commonly considered to be responsible for the separation or catalytic effect, the effect of the polymer matrix can also be important (25, 26). In a conventional sense, reactive separation implies separation of components by preferentially reacting one component and thereby facilitating their separation by redefining the separation problem. The reaction–separation strategy, on the other hand, involves carrying out a reaction in such a manner that in-situ separation of the products is most convenient. In this context it is instructive to study the ion-exchange method as a reaction–separation strategy for the preparation of barium chloride and sodium sulfide. Further, this method has the advantage of being pollution-free.

PROCESS DESCRIPTION

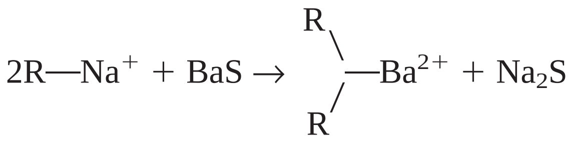
In this section we illustrate the preparation of barium chloride using the ion-exchange process. The following steps can be performed sequentially:

1. Resin in the H^+ form is converted to the Na^+ form by using a 1 M NaOH solution. The reaction is nothing but a neutralization reaction and proceeds to completion.

$$R{-}H^+ + Na^+\,OH^- \rightarrow R{-}Na^+ + H_2O \qquad (1)$$

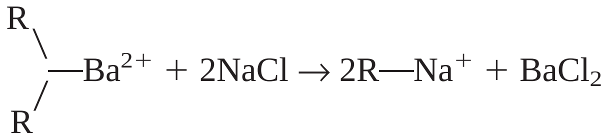
   where R represents a negatively charged resin group attached to the polymer matrix.

2. After the resin is completely converted to the Na^+ form, the resin bed is washed with demineralized water followed by the addition of BaS solution to convert the resin to the Ba^{2+} form. A high BaS concentration and a large excess of solution is used to ensure complete conversion of the resin to the Ba^{2+} form.



$$2R{-}Na^+ + BaS \rightarrow R_2{-}Ba^{2+} + Na_2S \qquad (2)$$

3. Sodium chloride solution of known concentration is then passed through the column to elute barium chloride solution as the resin in the column again converts itself from the Ba^{2+} to the Na^+ form.



$$R_2{-}Ba^{2+} + 2NaCl \rightarrow 2R{-}Na^+ + BaCl_2 \qquad (3)$$

Steps 2 and 3 can be repeated to get cyclic operation.

The process scheme can be thought of as consisting of two operations: reaction and separation. In the reaction part, the reactivity of the resin is important for obtaining high rates of exchange and maximum resin capacity utilization. In the separation part, high purity barium chloride and/or sodium sulfide may be obtained by manipulating the different process parameters in situ.

Reaction Steps 2 and 3 are most crucial for the yield and purity of barium chloride and sodium sulfide. The column behavior, i.e., the form of the breakthrough curve, will be dictated by the reactivity of the resin phase, the equilibria, and the column hydrodynamics. Since the reaction–separation strategy involves cyclic operation of Steps 2 and 3, high reactivity of the resin in both steps is most desirable. The preference by the resin polymer matrix for one ion over the other should not be very strong. In other words, the curvature of the equilibrium plot should be less here compared to conventional ion-exchange separation where maximum departure from the diagonal is preferred.

EXPERIMENTAL STUDIES

A strong acid cation-exchange resin, Tulsion T-42 (polystyrene matrix, obtained from Thermax Ltd., Pune, India), in its H^+ form was used in the present study. Pretreatment of the resin was carried out using the standard procedure of washing the resin with deionized water and then treating it with alkali and acid to remove chemical impurities (27). The resin characteristics were obtained by using the procedure given by Helfferich (27). The resin capacity was found to be 4.6 meq/g on the basis of dry weight of resin, and the water content of the fully swollen resin beads was found to be 1.33 cm^3/g of dry weight of resin. The average particle size of resin used for column experiments was in the 3×10^{-4} to 1.2×10^{-3} m range.

Equilibrium Studies

All equilibrium and column studies were carried out at room temperature, 28 ± 1°C. One-half gram of the pretreated ion-exchange resin in its Ba^{2+} form was added to flasks containing 100 cm^3 of sodium chloride solution. The samples were analyzed for their sodium and barium concentrations. The content of barium in the samples was estimated by the standard EDTA method using Erichrome Black-T indicator (28). The approach to equilibrium was checked at regular intervals of time. Concentration measurement of the samples after 24 and 48 hours showed no difference, indicating that equilibrium is essentially attained after 24 hours. The results of equilibrium experiments for the exchange of hydrogen ions with sodium ions and of barium ions with sodium ions are shown in Fig. 1.

Column Studies

The ion-exchange column tests were conducted in a glass column of 0.15 m height and 0.01 m diameter. The conversion of the resin to the Na^+ form was carried out by using a 1 M sodium hydroxide solution. During the column test studies, an aqueous solution of barium sulfide (prepared indigenously) was used for the preparation of barium chloride and sodium sulfide. Sodium chloride (GR grade) was used for elution. A specific volume of the prepared solution was passed through the column for each experimental run. Samples were collected at regular time intervals and analyzed for sodium sulfide or barium chloride (28). The column bed was continuously operated in the cyclic mode for the preparation of sodium sulfide and barium chloride during the column test experiments.

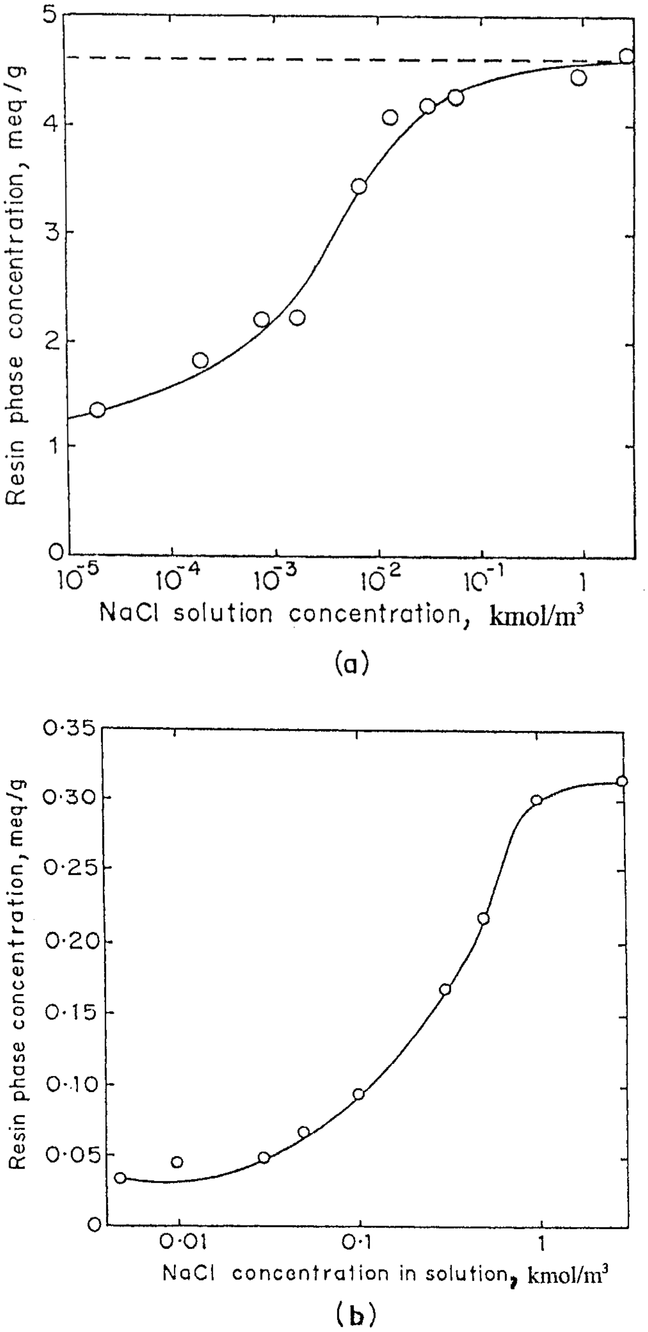


FIG. 1 Exchange equilibria on Tulsion T-42. (a) H^+ to Na^+ exchange. (b) Ba^{2+} to Na^+ exchange.

RESULTS AND DISCUSSION

Ion-Exchange Equilibria

It is evident from Fig. 1 that the conversion of resin from the Ba^{2+} form to the Na^{+} form (Reaction Step 3) is highly unfavorable. However, the negative isotherm is unavoidable in this case since divalent ions are generally preferred over monovalent ions because they simultaneously neutralize two resin sites. As a rule, an ion exchanger prefers the counterion of higher valence. The electrostatic preference for the higher valence ion is reflected in the separation factor and is generally referred to as "electroselectivity" (27). It may be possible to reduce the negative curvature in the isotherm by changing the resin type/polymer matrix. However, the negative isotherm in the Ba^{2+} to Na^{+} conversion indicates ease of operation in the other cycle (Reaction Step 2), wherein the resin is converted back to Ba^{2+} from Na^{+} when the BaS cycle begins.

Column Dynamics

In order to obtain the maximum yield of barium chloride and have effective ion-exchange column utilization, it is instructive to explore the effects of various parameters such as the barium ion concentration and its effect on sodium sulfide formation, the effect of sodium chloride concentration, the effect of pH of sodium chloride solution, etc. In the following text these effects are discussed in detail.

Effect of Barium Sulfide Concentration of Formation of Sodium Sulfide

Figure 2 gives experimentally observed column profiles for different BaS concentrations for a column diameter of 0.01 m and a bed height of 0.15 m. The solid lines in this figure indicate the sodium sulfide concentration trends while the dashed lines indicate the barium sulfide concentration profile. The barium sulfide concentration variation study was carried out at a flow rate of 3×10^{-5} m^3/h by utilizing the cation-exchange resin in its sodium form. The pH of the barium sulfide solution was 10.5 in these studies. The barium sulfide concentration was varied from 3 to 10% w/v. It was observed that the peak elution volume decreases with an increase in the barium sulfide concentration. The purity and yield of sodium sulfide increases along with an increase in the barium sulfide concentration. A nearly 75% change (lowering) was observed in the elution volume from a 3% barium sulfide concentration to the 10% level. Consequently, the yield of sodium sulfide increased from 1.2 to 4% w/v. It is also important to note that the solubility of barium sulfide in water at room temperature is low and a maximum 10% w/v concentration

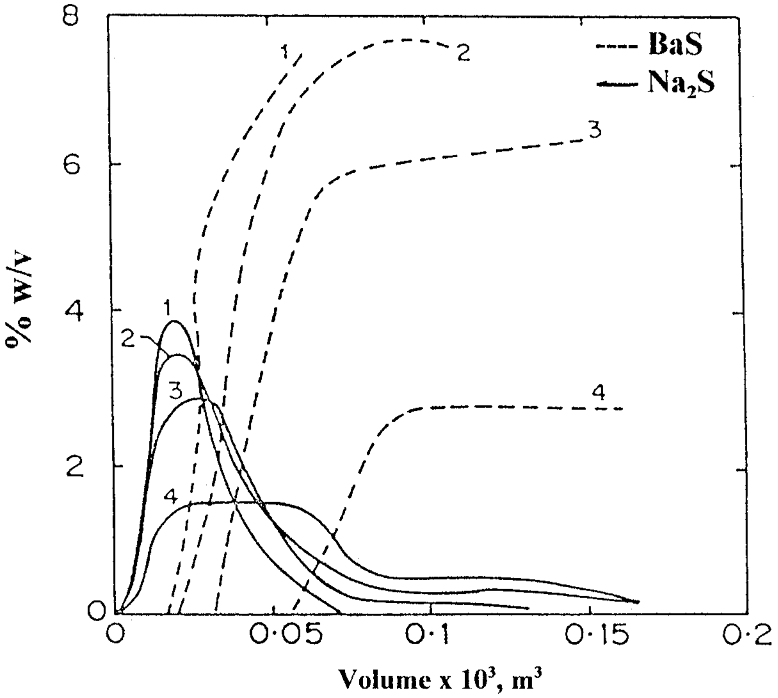


FIG. 2 Experimental data on variation of BaS (- - -) concentration and corresponding effect on Na_2S (——) formation. (1) 10% BaS; (2) 8% BaS; (3) 6.76% BaS; (4) 3% BaS.

could be obtained. Even a 10% concentration level was found to give problems because of crystallization. Further, the tailing effect was observed in all cases. It was also observed that the oxidation of barium sulfide to barium sulfate, which is a slow process, occurs at higher concentrations of BaS even at room temperature. Further studies were carried out using a moderate barium sulfide concentration of 6.76% w/v.

Effect of Sodium Chloride Concentration on Formation of Barium Chloride

It was believed that there would be a significant role for the sodium chloride concentration in the aqueous solution on the preparation of barium chloride. To ascertain this, the sodium chloride concentration was varied from 2 to 30% w/v. Figure 3 gives the experimentally observed column profiles for barium chloride (solid lines) and sodium chloride (dashed lines). The experimental conditions (flow rate, pH of the solution, and column diameter) were the same as those listed in the preceding section. During column operation for converting the resin from barium to sodium, the peak elution volume decreases with an increase in the sodium chloride concentration. A nearly 40% lowering in the volume occurs over the 2 to 30% w/v range. The concentra-

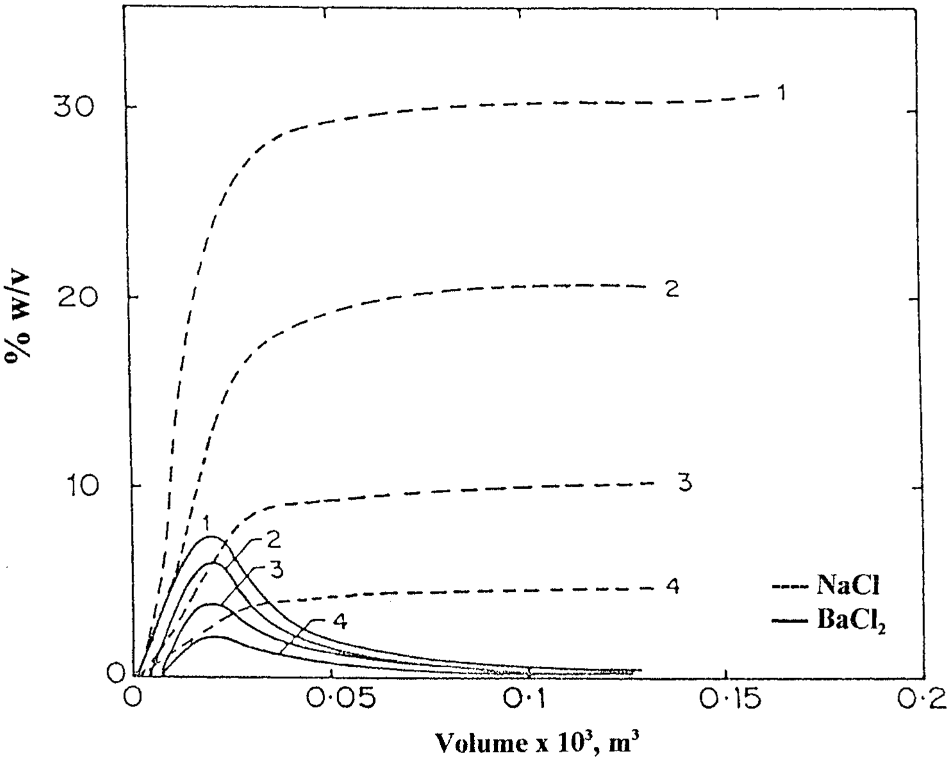


FIG. 3 Experimental data on variation of NaCl (——) concentration and corresponding effect on $BaCl_2$ (- - -) formation. (1) 30% NaCl; (2) 20% NaCl; (3) 10% NaCl; (4) 5% NaCl.

tion of barium chloride increases with an increase in the sodium chloride concentration, the maximum being 7% w/v for 30% sodium chloride concentration. Thus, NaCl concentration has a substantial effect on the overall yield of barium chloride. As before, the concentration decreases slowly after reaching the peak value, i.e., a tailing effect is observed, which can lower the effective yield of barium chloride.

Effect of pH of Sodium Chloride Solution on Reaction Rate and Product Yield

With the objective of enhancing both the separation and the reaction rate, the effect of pH of the sodium chloride solution was studied in the 6.5 to 1.0 range and also in the 0.2 to 0.5 M hydrochloric acid concentration range. Figure 4 shows the concentration profiles at different pH values for barium chloride (solid lines) and sodium chloride (dashed lines). A flow rate of 3×10^{-5} m^3/h was maintained in these studies. The column diameter and the bed height were the same as mentioned earlier. A sodium chloride concentration of 5% w/v was used in these studies. Peak elution volumes observed were similar in

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the pH range of 6.5 to 1.0. The barium chloride concentration in the elute increased from 3.8 to 4.4% w/v when the pH was changed from 6.5 to 1, even at constant NaCl concentration. This constitutes a nearly 16% change in barium chloride concentration for the same NaCl concentration level, and it can be expected to be different for different NaCl concentrations. The peak elution volume decreased with an increase in the hydrochloric acid concentration in sodium chloride in the 0.1 to 0.5 M hydrochloric acid concentration range. However, here the effect of HCl concentration on barium chloride in the elute was significant, and an approximately 24% increase in the barium chloride concentration was observed. To evaluate the effect of pH on the purity and yield of barium chloride, it is necessary to analyze the outgoing stream for sodium chloride and barium chloride. This is presented in Fig. 5. The increase in the purity and yield of barium chloride with an increase in hydrogen ion concentration can be attributed to the increase in the total cation concentration and activity. An interesting feature of this is the modification in the reaction rate due to adjustment in the pH. This is shown graphically in Fig. 6 where the initial rate is plotted against the H^+ ion concentration in solution. It is evident that the increase in the rate is very high as the pH of the solution goes below 2.

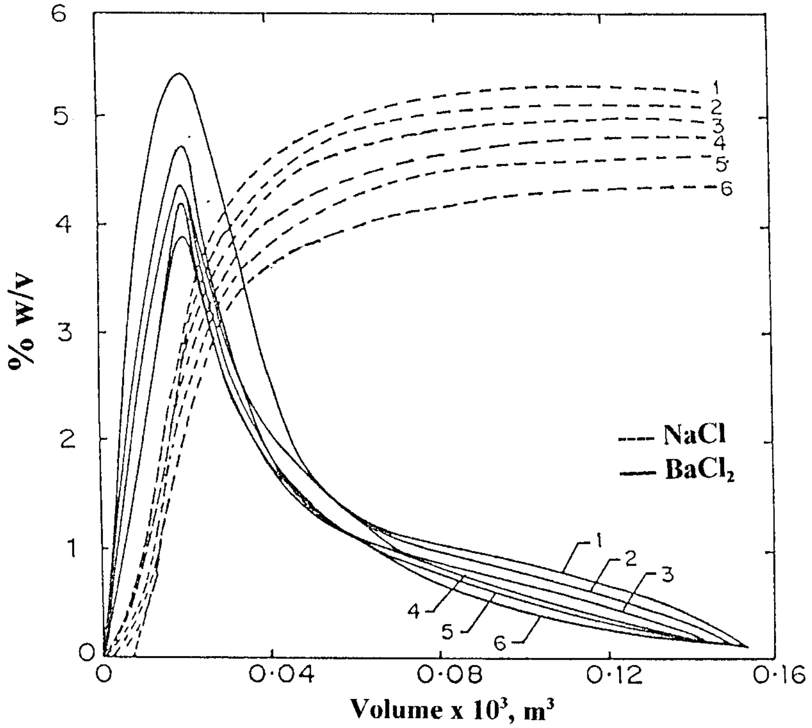

FIG. 4 Experimental data on $BaCl_2$ (——) concentration profile corresponding to changes in pH of NaCl (- - -) solution. (1) pH 6.5; (2) pH 4.65; (3) pH 2.88; (4) 0.1 M HCl; (5) 0.2 M HCl; (6) 0.5 M HCl.

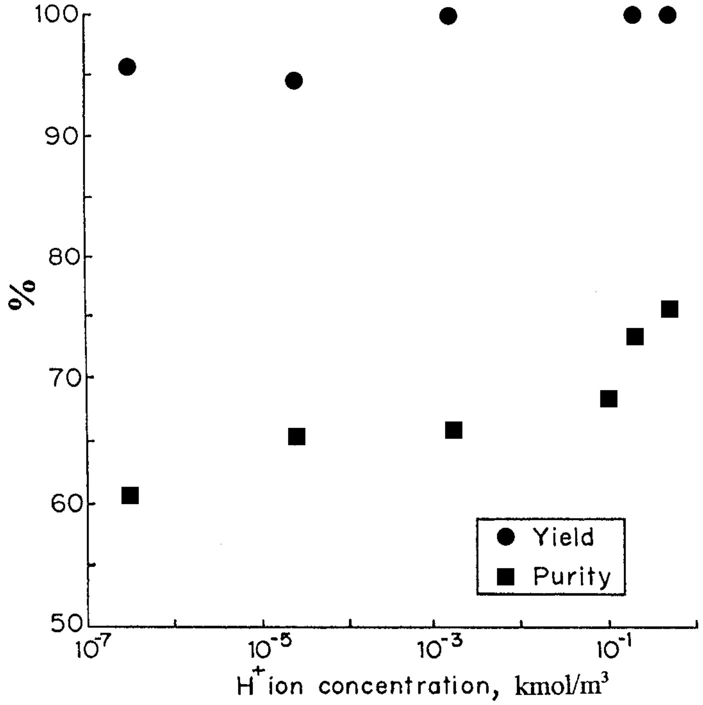

FIG. 5 Variation of $BaCl_2$ yield/purity with pH of NaCl solution.

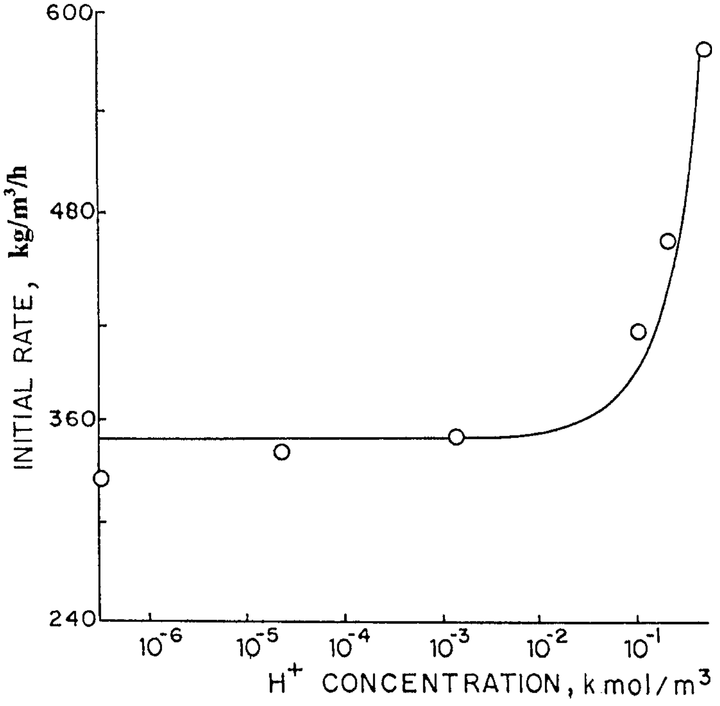

FIG. 6 Initial rate vs pH of NaCl solution. (——): Fit of Eq. (4).

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The initial rate can be well correlated with the hydrogen ion concentration in solution by using the following relation:

$$\text{Initial rate} = 5.82 \exp\{1.09\,[\mathrm{H}^+]\} \tag{4}$$

where the term in brackets indicates hydrogen ion concentration. The solid line in Fig. 6 gives the fit of the above correlation to the experimental data. Thus, the preparation of barium chloride can be carried out in an efficient manner from sodium chloride solution by using the optimum conditions. However, corrosion problems and cost factors should be taken into account when developing the process.

The effect of flow rate on separation was not significant in the 3×10^{-5} to 1.2×10^{-4} m^3/h range. Thus, the approach to equilibrium in the column is sufficiently rapid and the flow rate has little impact on the rate of distribution of the species in the resin phase.

Concentration Profiles in the Column

The nature of the breakthrough curve which identifies the concentration profile inside the column at any time is important for predicting the extent of separation of products from reactants. The concentration front inside the column and that represented by the concentration profile at the outlet of the column can be of the dispersive/spreading type or of the compressive/constant pattern type. An understanding of these profiles is therefore important from the point of view of column utilization and process efficiency. Figures 7(a) and 7(b) show the forms of breakthrough curves for different resin bed heights (0.15, 0.25, 0.3, and 0.55 m) and for different column diameters (0.006, 0.01, and 0.025 m) for the Reaction Step 3, i.e., barium to sodium exchange. The concentration profiles were of the constant pattern type for the preparation of sodium sulfide by passing a solution of barium sulfide, since here a strongly retained species replaces a weakly retained one. The concentration profiles are more or less of the dispersive type for replacing barium by sodium ions. The profiles for different bed heights and for different bed diameters confirm this observation. Thus, one would expect lower capacity utilization in the NaCl cycle, which in turn will dictate the economics of the process.

Economic Considerations

Barium carbonate and hydrochloric acid are the raw materials used in the preparation of barium chloride by the conventional route. In the process developed above, cheaper raw materials (viz., black ash containing water-soluble barium sulfide and sodium chloride) are used. Considering the stoichiometry of the conventional process and the cost of basic raw materials (29), the production cost works out to be approximately $0.75 per kg barium chloride,

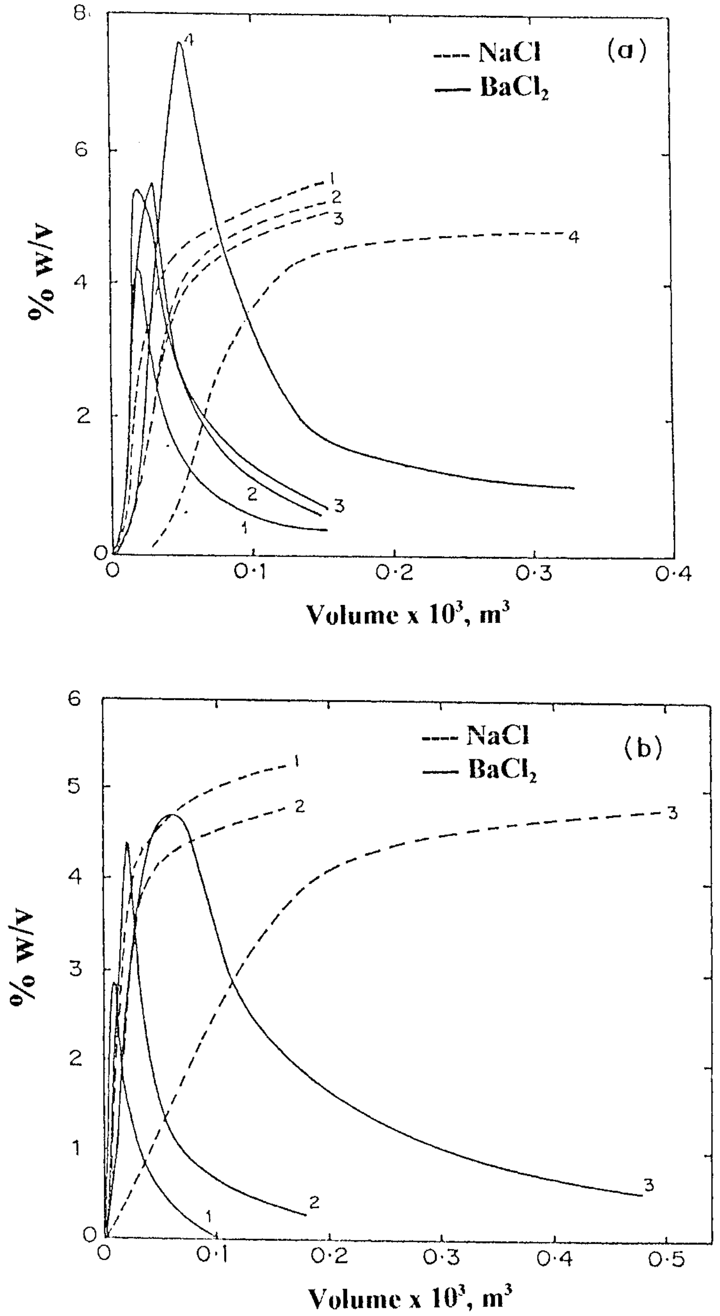

FIG. 7 Experimental data on concentration profile pattern (NaCl cycle). (a) Bed height: (1) 0.15 m; (2) 0.25 m; (3) 0.3 m; (4) 0.55 m. (b) Bed diameter: (1) 0.006 m; (2) 0.01 m; (3) 0.025 m.

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whereas the process developed here has a production cost of approximately \$0.38 per kg $BaCl_2$ based on raw material consumption. Therefore, even after accounting for the lower efficiency of the process, the developed route can be considered to be cost effective as compared to the conventional route. Further, credit can also be obtained for the by-product sodium sulfide. In terms of the present market price of barium chloride, which is approximately \$0.97 per kg $BaCl_2$, the developed process can be very attractive.

CONCLUSIONS

A new application of ion exchange as a reaction–separation strategy for the preparation of barium chloride from barium sulfide and sodium chloride has been discussed. Such parameters as the concentrations of barium sulfide and sodium chloride and the pH substantially affect column efficiency and the yield of pure barium chloride. Employing a higher barium sulfide concentration and proper adjustment of the pH are the two critical factors in obtaining high yields of the products. In view of the limited solubility of barium sulfide in water at ambient temperature, process performance can be improved by carrying out the ion-exchange operation at higher temperatures. However, further detailed investigations are required to improve both the equilibria and column dynamics. The present study can form the basis for the development of a simple and environmentally friendly alternative to the existing industrial processes for the preparation of inorganic chemicals.

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