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# Chemical Separation Process for Highly Saline Water. 1. Parametric Experimental Investigation

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The separation of highly saline waters under various conditions is carried out using a nonconventional partial-desalting process. The method utilizes a series of chemical reactions involving the conversion of sodium chloride, the major constituent in saline waters, into sodium bicarbonate, which precipitates under the experimental conditions, and ammonium chloride, which can be separated by crystallization. Experiments of absorption of carbon dioxide in an ammoniated brine have demonstrated the efficiency of the method. Interpretations using conversion parameters such as temperature, initial concentrations of ammonia, sodium chloride, and the ratio of  $\text{NH}_3/\text{NaCl}$  are discussed.

## Introduction

The proposed separation method implies the absorption of  $\text{CO}_2$  gas into ammoniated brine solution as a vehicle for accomplishing separation for a wide range of applications: (a) desalting of highly saline water resources (sabhka); (b) product recovery and gas purification for a number of effluent gas streams, with subsequent conversion into useful products; (c) production of chemical products of economic potential values such as sodium bicarbonate, ammonium chloride, and magnesium chloride, using indigenous brine resources along with abundant  $\text{CO}_2$  and  $\text{NH}_3$  sources.

The unique features of such a process are the facts that separation and purification are carried out simultaneously in both the gas phase (separation of  $\text{CO}_2$  or  $\text{CO}_2/\text{NH}_3$  from a gas stream) and the liquid phase (separation of  $\text{NaCl}$  from brine solution).

As far as desalination is concerned, the current conventional desalination methods are based on physical separation techniques and normally utilize either sea or brackish water as a feed having a concentration of 4000–35 000 ppm or 0.068–0.598 mol/L. In contrast, the proposed separation method utilizes a series of chemical reactions for the conversion of sodium chloride ( $\text{NaCl}$ ) found in saturated brine solutions of 4–5 mol/L concentration into separable products, namely, soda ash ( $\text{Na}_2\text{CO}_3$ ) and ammonium chloride ( $\text{NH}_4\text{Cl}$ ). As a result, a partially desalted water is obtained. Its salinity is a function of parameters investigated in this work. The chemical reactions involved in this process are modifications of the well-known "Solvay" process described in the literature (Hou, 1942). The disadvantages of the classical Solvay process, however, are the low percentage utilization of  $\text{NaCl}$  and plugging problems of plate columns because of solid precipitation (Miyata, 1983). Nevertheless, no references characterized the Solvay process as a separation method capable of producing partially desalted water along with  $\text{Na}_2\text{CO}_3$  and  $\text{NH}_4\text{Cl}$  from highly saline waters.

The main objective of this work is apply the chemical separation method to desalt indigenous sabhka resources, which leads to the recovery of chemical products as well as partially desalted water. The products obtained are sodium bicarbonate ( $\text{NaHCO}_3$ ), soda ash ( $\text{Na}_2\text{CO}_3$ ), and water rich in fertile salts, namely, ammonium chloride ( $\text{NH}_4\text{Cl}$ ).

The chemical separation method is not to be considered as an alternative to the existing conventional

Table 1. Sabhka Water Analysis

species (ppm)	sample 1	sample 2
$\text{Na}^+$	56830	58410
$\text{K}^+$	2342	2396
$\text{Ca}^{2+}$	1407	1433
$\text{Mg}^{2+}$	4788	4810
$\text{Cl}^-$	101060	102130
$\text{SO}_4^{2-}$	6172	6188
T. alkalinity	52.1	56.4
TDS	181112 or 1.049 mol/L	182488 or 1.041 mol/L
pH	8.1	8.3

desalination processes. The proposed method uses saturated brines at least 5 times more concentrated than sea water and produces valuable chemical products in addition to partially desalted water. In this respect, it is regarded as a supplement to the physical multistage flash desalination processes. The economics of the proposed chemical process as compared to the existing ones cannot be easily evaluated accordingly.

Table 1 depicts the chemical composition of sabhka water, in the Eastern Province of Saudi Arabia, which represents an indigenous resource for raw material having a salinity of about 20% by weight or 3–5 mol/L.

## Approach to Separation

The proposed method utilizes the Solvay principles as a chemical separation technique (Figure 1). It uses saturated brine as a feed stock. The core of the system under study rests on the absorption of  $\text{CO}_2$  into an ammoniated brine solution. The enhanced absorption reaction of the gas in the liquid takes place in a contactor that will meet the requirements of the available reaction system. The primary reaction happens between the absorbed  $\text{CO}_2$  (Lewis acid) and the ammonia in the ammoniated brine that acts as a chemical base to form carbamic acid, which reacts with ammonia to form ammonium carbamate (Danckwerts, 1970; Pincet *et al.*, 1956):



The net result of these two reactions is the overall reaction:



which is called the *primary reaction*.

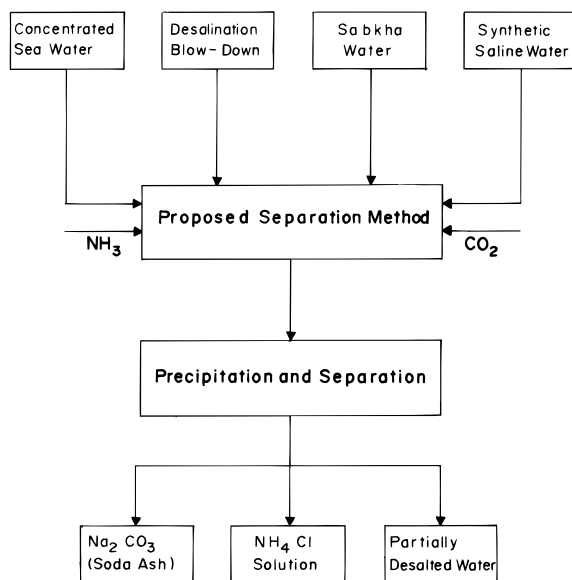
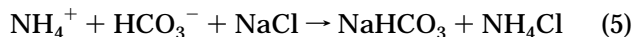


Figure 1. Outline of the proposed method.

The rapid reaction, leading to the formation of ammonium carbamate, accompanies the absorption process and determines its rate. In the bulk of the solution, the carbamate hydrolyzes comparatively slowly to bicarbonate.



This is called the secondary reaction. The bicarbonate ion is precipitated as sodium bicarbonate:



The system described above involves both mass transfer and chemical reaction; therefore, consideration of both factors is important in an actual design. The  $\text{NH}_3$  concentration and the purity of the  $\text{CO}_2$  can influence the regimes responsible for the overall absorption rate. Mass transfer of  $\text{CO}_2$  into highly carbonated ammoniated brine solution proceeds extremely slowly (Danckwerts and Sharma, 1966). The primary reaction that consumes the  $\text{CO}_2$  is fast, and the system is diffusion controlled at higher carbonation time. Therefore, to obtain a higher yield from this system, it is important to maximize the absorption of  $\text{CO}_2$  and increase the rate of the secondary reaction. A bubble-column contactor, where gas is dispersed through a relatively deep pool of liquid, is found to be a suitable choice for the aforementioned system.

Bubble-column absorber reactors recommended to carry out the proposed separation process normally possess the following design features:

(a) To carry out the primary reaction, the interfacial area of contact for mass transfer and the number of stages are relatively small. Theoretically speaking, a complete absorption in a single stage is possible only when the absorbed compound has a negligible vapor pressure over the liquid. This is exactly the case of absorption with an irreversible chemical reaction forming a nonvolatile species.

(b) To carry out the secondary reaction, a large volume of liquid is needed to provide the required residence time for the hydrolysis of the carbamate—formed in the reaction—to the carbonate.

### Absorption with Chemical Reaction

When the reaction occurs in the liquid phase and is rapid and irreversible, the rate of absorption may be

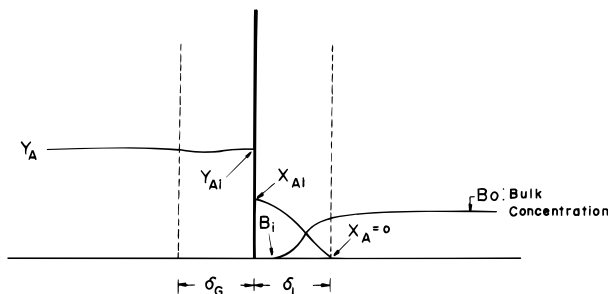


Figure 2. Schematic outline of the concentration profiles at the gas-liquid interface for a pure  $\text{CO}_2/\text{NH}_3/\text{brine}$  system.

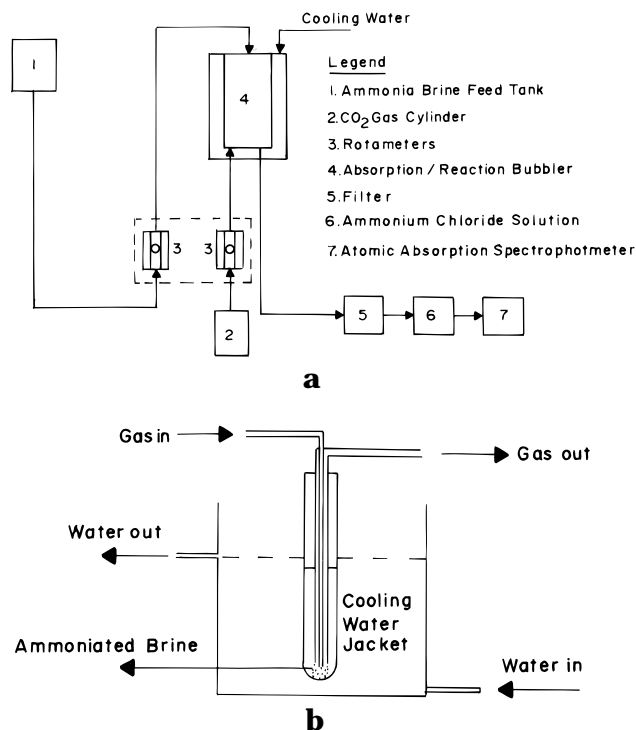
controlled primarily by the gas-phase resistance to mass transfer. Since in the experiments undertaken in this paper absorption of pure  $\text{CO}_2$  is taking place, the gas phase contributes no resistance to diffusion.

The effect of chemical reaction in the liquid phase, on the other hand, is generally to increase the liquid-film absorption coefficient over that which would be observed with simple physical absorption.

With very slow reactions, however (such as that between carbon dioxide and water), the dissolved molecules apparently migrate well into the body of the liquid before reaction occurs so that the overall mass-transfer rate is not appreciably increased by the occurrence of the chemical reaction. In this case, the liquid-film resistance is the controlling factor, the liquid at the interface can be assumed to be in equilibrium with the gas, and the rate of mass transfer is governed by the molecular  $\text{CO}_2$  concentration gradient between the interface and the body of the liquid.

At the other extreme are very rapid reactions such as the one we are dealing with, where the dissolved molecules migrate only a very short distance before reaction occurs. The location of the reaction zone and the value of absorption coefficient will depend primarily upon the diffusion rate of reactants and reaction products to and from the reaction zone, the concentration of solute at the interface, and the concentration of reactant in the body of the liquid. However, since the distance which the solute must diffuse into the liquid is extremely small compared to the distance which it would have to travel for simple physical absorption, a higher liquid-film coefficient is observed.

Despite the very fast reaction by which free  $\text{CO}_2$  is chemically reacted with  $\text{NH}_3$ , there is, nevertheless, a very appreciable resistance to the passage of carbon dioxide into the ammoniated brine solution. Most of the resistance is in the liquid phase. This could be explained by the fact that (a) salt solutions exhibit greater viscosity, which lowers the diffusion coefficient of  $\text{CO}_2$ , and (b) salt solutions reduce the solubility of free  $\text{CO}_2$  as compared to aqueous ammonia solutions. This implies that the reaction must take place at a finite rate in an appreciable volume of the liquid rather than on the interface itself. Unless diffusion of carbon dioxide away from the interface occurs, no volume of the liquid is available for the reaction; thus, two phenomena take place: (i) the faster the diffusion, the deeper is the reaction layer at the interface, while (ii) the faster the reaction, the thinner is the zone near the interface, which has to be supplied with carbon dioxide moles by diffusion. This explains the fact that both rates of diffusion and reaction have to go hand in hand to produce the right effect on the separation process. Figure 2 is a schematic illustration of the case involving the absorption of pure  $\text{CO}_2$  in ammoniated brine.

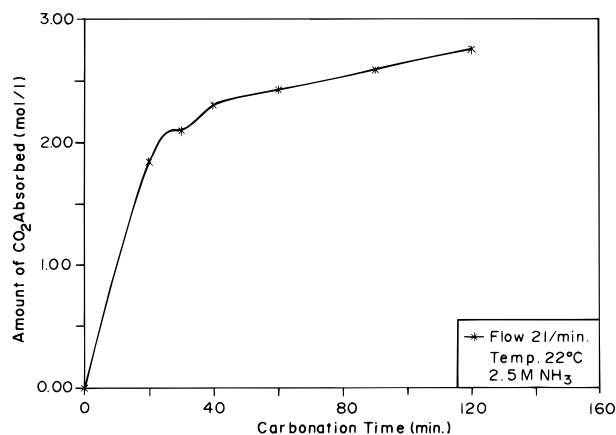


**Figure 3.** (a) Schematic diagram of experimental setup. (b) Sketch of the batch gas bubbler.

### Apparatus and Procedure

Bubble columns are frequently used as absorbers, strippers, and reactors. For gas-liquid systems, bubble columns have been widely used because of ease of construction, maintenance, and the absence of moving parts. In addition, solids can be handled without any erosion and plugging problems (Mashelkar, 1970; Mashelkar and Sharma, 1970). Moreover, slow reactions can be carried out due to high liquid residence time, high values of effective interfacial area, and high overall mass coefficients (Abraham and Sawant, 1990). The absorption of CO<sub>2</sub> in an ammoniated brine involves both mass transfer and chemical reaction. The primary reaction that consumes the CO<sub>2</sub> is a fast reaction, while the hydrolysis of the carbamate is a slow one. To provide sufficient residence time of liquid for slow hydrolysis of carbamate and to reduce the plugging problems due to solid precipitation, bubble columns are used in this investigation.

The schematic diagram of an experimental setup is given in Figure 3a. The batch gas bubbler comprises an absorption glass vessel of about 40 cm long and 5 cm i.d., fitted with a socket, inlet cone, and a long inlet tube which passes almost to the bottom of the absorption vessel as shown in Figure 3b. The unabsorbed gas is vented or circulated. The brine solution is prepared synthetically in the laboratory by adding pure NaCl salt in deionized water. Then, the brine solution is treated with NH<sub>3</sub> to give an ammoniated brine of known and fixed concentration of salt and ammonia. The resultant solution is placed in the batch gas bubbler. Then, carbonation of the ammoniated brine is carried out using a pure CO<sub>2</sub> gas. The gas passes through a gas meter for setting the required flow rate. The temperature of the system is regulated by using a cooling water jacket. The carbonation time is recorded during the operation. The carbonation of the solution is suspended after specified periods, and the solution is passed through a filter to separate the precipitate formed from the mother liquor (filtrate).



**Figure 4.** Absorption of CO<sub>2</sub> in an ammonia solution.

In the filtrate, analysis of carbonates, chloride, NaCl, and NH<sub>4</sub>Cl is accomplished, while the precipitate analysis of carbonates, chloride, and NaCl is carried out. The Na<sup>+</sup> was determined by an atomic absorption spectrophotometer Model 3100, Perkin Elmer).

In all experiments, a volume of 100 mL of ammoniated brine was subjected to carbonation. The parameters taken into account in this study include the initial NaCl content in the water, the initial concentration of NH<sub>3</sub>, and the effect of the extent of carbonation. The main purpose of the parametric investigation is to enhance the chemical conversion of NaCl into NaHCO<sub>3</sub> and NH<sub>4</sub>Cl. The NaHCO<sub>3</sub> is separated as a solid precipitate, while NH<sub>4</sub>Cl either separated by crystallization or was left in the solution to provide desalinated water rich in NH<sub>4</sub>Cl, which can be used as fertilizer. The progress of chemical conversion was monitored by determining the concentration of unreacted NaCl as well as NH<sub>4</sub> formed in the solution. The latter was determined by a gravimetric method. The accuracy and the precision in the determination of Na<sup>+</sup> was far greater than any other species present in the solution. Therefore, the efficiency of the modified Solvay process is manifested by the decrease of Na<sup>+</sup> in the ammoniated brine.

### Results and Discussion

The following is a discussion of the important results obtained during the course of the investigation.

**Temperature and pH Effects.** The absorption of CO<sub>2</sub> in an ammoniated brine is a diffusion-controlled process, where the resistance lies in the liquid phase as explained earlier. Figure 3 depicts the extent of CO<sub>2</sub> absorption in a NH<sub>3</sub> solution, when the gas flow rate is 2 L/min. The rate of CO<sub>2</sub> absorption increases appreciably during the initial bubbling period, due to a higher driving force, resulting from the absorption-enhanced reaction of CO<sub>2</sub> in a NH<sub>3</sub> solution. However, with a further increase in time, the rate of absorption of CO<sub>2</sub> diminishes as the establishment of equilibrium of ammonium carbonates is approached, as given by eq 3 and 4.

The results reported in Figure 4 were conducted without NaCl to avoid precipitation of sodium bicarbonate salts, which otherwise would plug the gas bubbler and adverse accordingly the performance of the bubble reactor. The rate of absorption of CO<sub>2</sub> in ammoniated brine is less pronounced; however, a trend similar to the one exhibited in Figure 4 is produced. This is based on the fact that reactions between NaCl and ammonium carbonates are fast ones.

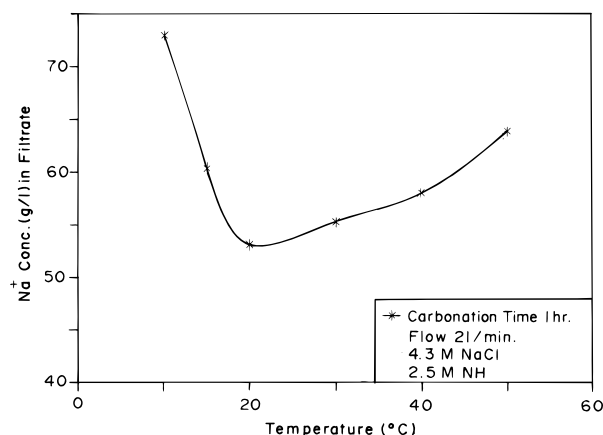
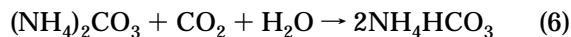


Figure 5. Effect of temperature on NaCl removal.

The optimum operating temperature for the separation of NaCl in the saline water was investigated. This was done by studying the effective removal of NaCl from a solution containing 2.5 M  $\text{NH}_3$  and 4.3 M NaCl, for 1 h of carbonation at different temperatures. The result, which is given in Figure 5, indicates that an operating temperature around 22 °C is the most favorable value. This is shown by the minimum  $\text{Na}^+$  concentration in the filtrate. At a lower temperature, separation is less efficient because of the poor reaction rates of  $\text{CO}_2$  and aqueous  $\text{NH}_3$ . Similarly, separation is suppressed at higher temperatures because the absorption of  $\text{CO}_2$  gas decreases with the increase in temperature.

The effects of the pH value on the formation of carbonate and bicarbonate and the equilibrium relationship between them were studied, using a 3.5 M  $\text{NH}_3$  solution, in both the presence and absence of NaCl salts. The  $\text{CO}_2$  gas was allowed to bubble until precipitates of  $(\text{NH}_4)_2\text{CO}_3$  were observed. Further carbonation of the solution shows the transformation of carbonates according to this equilibrium relationship:



The analysis of the results indicated as well that the total sum of  $(\text{NH}_4)_2\text{CO}_3$  and  $\text{NH}_4\text{HCO}_3$  remains unchanged, but transformation from one form to another takes place. In other words, initially, the concentration of  $(\text{NH}_4)_2\text{CO}_3$  is predominant; however, further carbonation leads to the formation of  $\text{NH}_4\text{HCO}_3$ , which increases with time as a result of a decrease of  $(\text{NH}_4)_2\text{CO}_3$ , as given by the above reaction. On the other hand, it was found that the pH value decreases appreciably from 12 to 9 with the increase of bubbling of  $\text{CO}_2$  which neutralizes the amine solution. However, further carbonation time brings a slight change in the pH value. The formation of bicarbonate is favored over the pH range of 8–10. Figure 6 shows the effect of carbonation time on the pH value of the solution.

**Carbamate Hydrolysis.** As mentioned earlier, the rapid reaction leading to the formation of ammonium carbamate accompanies the absorption process and determines its rate. In the bulk of the solution, the carbamate hydrolyzes comparatively slowly to bicarbonate as given by eq 4:



It is observed from Figures 3 and 6 that the carbonation rates of  $\text{CO}_2$  in ammoniated solutions are much

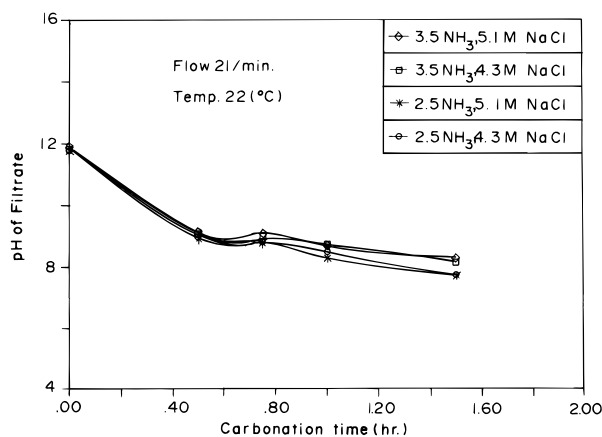


Figure 6. Effect of carbonation on pH.

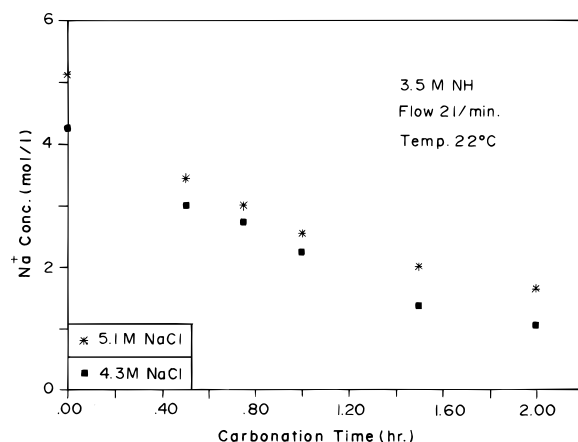


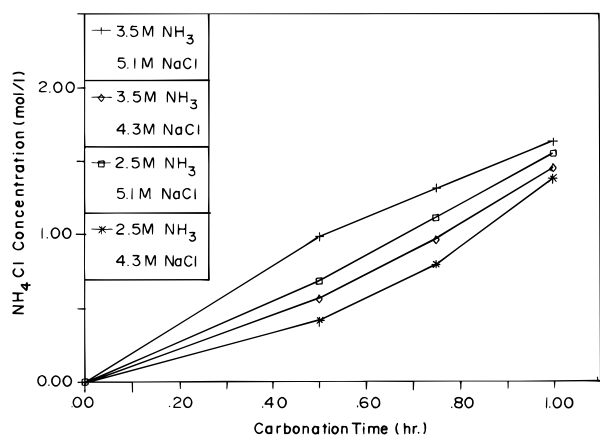
Figure 7. Effect of salt on  $\text{Na}^+$  removal.

greater than the rates of  $\text{Na}^+$  depletion. This comparison would indicate that the hydrolysis of carbamate is an important step in determining the rate of the overall process. Due to this fact, the reactor type selected for this separation process is the bubble column which provides a sufficient holding time, but it has a negligible effect on the absorption rate of  $\text{CO}_2$ .

**Separation of Synthetic Saline Water.** The percentage conversion of NaCl for various conditions was studied, using laboratory synthetic saline water as feed. The carbonation of the ammoniated brine is carried out at specified conditions. Figure 7 shows the result when a 3.5 M  $\text{NH}_3$  solution is carbonated at the rate of 2 L/min. The result indicates that the rate of  $\text{Na}^+$  removal increases with higher carbonation time and tends toward a constant value. The percentage removal of  $\text{Na}^+$  is found to be 75 when the initial salt in the water is 4.3 M; however, the percentage drops to 67 when the initial concentration of salt is 5.1 M. This is attributed to the decrease in the solubility of  $\text{CO}_2$  in ammoniated brine because of a high initial salt content.

Other experimental conditions exhibiting the effects of different initial concentrations of  $\text{NH}_3$  and NaCl in the feed have been investigated and reported (Ibrahim, 1993). The highest conversion of 82.2% was achieved when initial concentrations of  $\text{NH}_3$  and NaCl are 7.5 and 4.3 M, respectively. However, this does not represent the optimum conditions in utilizing the excessive amounts of  $\text{NH}_3$  as a reactant. In this regard, 82.2% of 7.5 M or 6.16 mol of  $\text{NH}_3$  is consumed, while 1.34 mol is not utilized in the reaction.

Along with the formation of sodium carbonates,  $\text{NH}_4\text{Cl}$  comes as an additional product in the filtrate as a



**Figure 8.** Rate of formation of ammonium chloride.

result of the reaction between ammonium and chloride ions. Figure 7 shows the rate of formation of  $\text{NH}_4\text{Cl}$  as a function of carbonation time. For the same initial concentration of  $\text{NH}_3$ , the higher the  $\text{NaCl}$  salinity, the higher is the  $\text{NH}_4\text{Cl}$  formation. With the same initial salinity, the formation of  $\text{NH}_4\text{Cl}$  is marginally increased with an increase of  $\text{NH}_3$  concentration.

**$\text{NaCl}$ ,  $\text{NH}_3$ , and  $\text{NH}_3/\text{NaCl}$  Concentration Effects.** The initial  $\text{NaCl}$  concentration has a considerable effect on the separation process. Figure 8 compares the effect of two different initial salinities in solutions of the same  $\text{NH}_3$  concentration under the given conditions. The rate of conversion of  $\text{Na}^+$  in the filtrate is higher when less saline feed source is used. In other words, the concentration of  $\text{Na}^+$  in the filtrate is greater when a higher saline source is used while maintaining the same conditions. A similar dependence of the conversion of a  $\text{NaCl}$  solution on the initial concentration of brine was shown, using a higher concentration of the  $\text{NH}_3$  solution, for example, 4.5 M (Ibrahim, 1993).

Experimentally, it was found that the solubility of  $\text{NaCl}$  is reduced at a high concentration of  $\text{NH}_3$ . On the other hand, if very high concentrations of  $\text{NH}_3$  are employed, then a large amount of  $\text{NH}_3$  is going to coprecipitate with  $\text{NaHCO}_3$  as  $(\text{NH}_4)_2\text{CO}_3$  exceeds its solubility product. This may be considered a loss it is not taking part in the conversion reaction of the salt.

Theoretically, it is required to have equimolar concentrations of  $\text{NH}_3$  and salinity ( $\text{NaCl}$ ), as the stoichiometry of the reaction indicates. However, it is necessary to use higher amounts of  $\text{NH}_3$  to compensate losses due to evaporation or reaction with impurities in the feed.

It is economically best to have a  $\text{NH}_3/\text{NaCl}$  ratio of 1.2 or slightly higher than 1 (1.9). If the ratio is too high, excessive amounts of  $(\text{NH}_4)_2\text{CO}_3$  would precipitate and leave without reacting with  $\text{NaCl}$  in the solution. This is definitely not economical in the usage of  $\text{NH}_3$ , which is a relatively expensive chemical. On the other hand, if the ratio is too low, then the decomposition of  $\text{NaCl}$  will drop in the reactor and most of the  $\text{NaCl}$  salt will remain in the brine solution unchanged. This means there is insufficient or depleted  $\text{NH}_4\text{HCO}_3$  to react with a saturated  $\text{NaCl}$  solution. Therefore, this ratio directly affects the rate of salt decomposition, or conversion of  $\text{NaCl}$  into the required products, and utilization of  $\text{NH}_3$ . Table 2 shows the effect of the initial concentration ratio of  $\text{NH}_3/\text{NaCl}$  on the separation of synthetic saline water for the given conditions. The conversion efficiency of  $\text{Na}^+$  is relatively low at lower values of the ratio—less than unity—and increases with its increase toward unity.

**Table 2.** Sabkha Water Analysis<sup>a</sup>

$\text{NH}_3$ (M)	$\text{NaCl}$ (M)	$\text{NH}_3/\text{NaCl}$	conversion (%)
3.5	5.1	0.7	67.8
3.5	4.3	0.8	75.4
4.5	4.3	1.1	76.0
12.0	3.3	3.6	72.0

<sup>a</sup> Feed water = synthetic saline, flow of  $\text{CO}_2 = 2$  L/min, temp = 22 °C.

**Application of Experimental Results.** In these experiments, the transfer of mass ( $\text{CO}_2$ ) accompanied by chemical reaction (between  $\text{CO}_2$  and  $\text{NH}_3$ ) has focused upon point conditions within a small-scale gas bubbler as a chemical reactor.

An understanding of the driving forces and the controlling resistances at a point permits the evaluation of the rate of transfer and, hence, the conversion rate of  $\text{NaCl}$  in the ammoniated brine at that point. Before the above "raw" experimental data could be applied for commercial applications, a need exists to scale up these results first and then go to a pilot-scale level. Once these data become available, the rate of transfer accompanied by chemical reaction at all points within the contactor could be related to the total transfer area required for the process equipment.

Specifically, it is desired to know how tall a contactor should be for a given separation.

## Conclusions

The following are the most important concluding remarks on both the parametric investigation of the process and its characteristic as a potential attractive method, to be applied in the future.

(1) Separation of high-salinity synthetic saline water was carried out in a batch gas bubbler using a modified Solvay process. The conversion of  $\text{NaCl}$  in an ammoniated brine into  $\text{NaHCO}_3$  and  $\text{NH}_4\text{Cl}$  was investigated. The highest percentage conversion of  $\text{NaCl}$  achieved was 82.2.

(2) The effect of temperature, initial concentrations of  $\text{NH}_3$  and  $\text{NaCl}$ , and the ratio of  $\text{NH}_3/\text{NaCl}$  were studied. An optimum separation temperature of 22 °C was found.

(3) It is concluded as well that the higher concentrations of  $\text{NH}_3$  enhance the separation, while the higher concentrations of  $\text{NaCl}$  reduce the conversion. A ratio of  $\text{NH}_3/\text{NaCl}$  of 1.2 was found to be the optimum. This proportion of the feed contributes to the successful utilization of these two principal raw materials and eventually brings better achievement in the separation of salt.

(4) The purity of the separated salts depends on the type of feed used in the separation process. For example,  $\text{Mg}$  salts could precipitate along with soda ash when sea water is used. However, the recovery of  $\text{Mg}$  salts separately is of potential value in this respect.

(5) Indigenous resources of raw materials (such as sabkha) are abundant, which can make the chemical separation approach attractive. For example, the sabkha waters are saline sources containing up to 4–5 mol/L of  $\text{NaCl}$ . When these resources are subjected to the proposed separation process, partially desalted water is obtained along with other valuable chemical products.

(6) The main products of this process are sodium bicarbonate ( $\text{NaHCO}_3$ ), soda ash ( $\text{Na}_2\text{CO}_3$ ), and water rich in fertile salts such as ammonium chloride ( $\text{NH}_4\text{Cl}$ ). Ammonium chloride is an extremely effective nitrogenous fertilizer, and its solution is well suited for

irrigation systems. As an alternative, instead of having the ammonium chloride in solution, it could be crystallized, i.e., the coproduction process of soda ash and ammonium chloride.

(7) The quantity of desalted water is estimated to be about 50% by weight of the intake saline feed water.

(8) The proposed method could be extended for pollution control applications. Brine solutions to be disposed of either from desalination plants or oil fields are excellent feed stocks for the purification of many industrial gas effluents that contain CO<sub>2</sub> and NH<sub>3</sub> gases.

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