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Chemical Separation Process for Highly Saline Water. 2. System Analysis and Modeling

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The reactions and equilibrium relationships underlying the absorption process of CO_2 gas in ammoniated brine solutions as proposed in part 1 are described first. A numerical solution of a set of nonlinear equations developed in the form of an algorithm, to calculate the concentration of unreacted ammonia, is obtained next. Calculated values are compared with experimental data for the system under investigation. The instantaneous absorption rate is evaluated by using the approximate approach (film model) and the exact solution (penetration theory).

Introduction

A separation process was proposed by the authors-in part 1-which utilizes a series of chemical reaction sequences in order to separate NaCl from highly-saline water resources (sodium chloride-rich brines). The process involves bubbling of CO2 gas into ammoniated brines, thus converting Na⁺ and Cl⁻ into NaHCO₃ and NH₄Cl, respectively. Bubble columns were used as chemical reactors. Detailed experimental findings are reported by Ibrahim (1993). More than 80% NaCl conversion was achieved for saturated brines, leading to the production of desalted water along with valuable chemical products, namely, NaHCO₃ and NH₄Cl. The theoretical design of the proposed process depends primarily on the absorption of carbon dioxide accompanied by chemical reaction in the ammoniated brine solution. The reaction between NaCl and the resultant product "ammonium bicarbonate" is a fast ionic reaction.

Reactions are represented by the following equations:

$$CO_2 + 2NH_3 \rightarrow NH_2COO^- + NH_4^+$$
 (1)

$$NH_2COO^- + H_2O \rightarrow NH_3 + HCO_3^-$$
 (2)

$$NH_4^+ + HCO_3 + NaCl \rightarrow NH_4Cl + NaHCO_3^-$$
 (3)

Although the absorption of CO_2 into NH_3 solutions is a well-known process, most of the references cited in the open literature were published before 1970, especially the work by Danckwerts and co-workers (1966, 1970). A recent publication by Pelkie and co-workers (1992) reports on the product distribution for CO_2 – NH_3 – H_2O systems using liquid conductivity measurements. However, no other published research work relevant to our investigation could be cited.

The gas—liquid reaction involved in this process as given in eq 1 is described as an instantaneous diffusion-controlled reaction with the rate given by:

$$rate = K_{NH_3}C_{NH_3}C_{CO_2}$$
 (4)

as reported by Pincet et al. (1956) and Danckwerts and Sharma (1966). It can safely be mentioned that the controlling stage in this proposed separation method is the absorption of CO_2 by an ammonia solution accompanied by chemical reaction leading to the formation

of NH_4HCO_3 . The work presented in this paper provides a systematic approach for the analysis of the CO_2/NH_3 solution system underlying the separation process described above.

Modeling and Assumptions

The types of concentration profiles near the gas—liquid interface in a chemically reacting liquid, bimolecular system represented by:

$$C_{\rm A} + C_{\rm B} = C_{\rm C} \tag{5}$$

have been well explained by Sherwood, Pigford, and Wilke (1975). The reaction rate in many such examples can be represented by:

$$R = KC_{\Delta}C_{R} \tag{6}$$

This system could represent very well our separation process in which the absorption of carbon dioxide in an ammonia solution (in the presence of NaCl) to form ammonium carbonates takes place. The process involves simultaneous diffusion and chemical reaction near an interface. To account for the influence of a chemical reaction on the concentration of a diffusing species in the important region near an interface between phases, one must include a reaction rate expression in the steady-state diffusion equation. For the second-order reaction, the mass balances using the film theory are:

$$0 = D_{A} \frac{d^{2}C_{A}}{dy^{2}} - KC_{A}C_{B}$$

$$0 = D_{B} \frac{d^{2}C_{B}}{dy^{2}} - KC_{A}C_{B}$$

$$(7)$$

The solution of these equations with their appropriate boundary conditions is somewhat difficult because of the nonlinear reaction term. To avoid this, special limiting cases are considered. Cussler (1984) reported the limiting case of a very fast irreversible reaction in which the reaction rate K is so large that A and B react immediately and completely upon contact in the solution. Then the product $C_{\rm A}C_{\rm B}$ must be zero everywhere

in the liquid. The result could be simulated as two film theories slapped one on top of the other. The rate of mass transfer could be described accordingly by the well-known relationship:

$$\frac{k}{k^0} = 1 + \left(\frac{C_{\rm B}}{zC_{\rm Ai}}\right)\left(\frac{D_{\rm B}}{D_{\rm A}}\right) \tag{8}$$

where k is the mass-transfer coefficient with chemical reaction and k^0 is the mass-transfer coefficient in the absence of chemical reaction (physical absorption) defined by:

$$k^0 = D_{\rm A}/\delta \tag{9}$$

The ratio of k/k^0 is known as the enhancement factor, E_i . The rate of mass transfer of CO_2 in an ammoniated brine solution, N_A , is given by:

$$N_{\rm A} = k^0 C_{\rm Ai} E_{\rm i} \tag{10}$$

Other variables are defined as follows: $D_A = diffusivity$ of CO_2 gas in the ammonia solution; $D_B = diffusivity$ of ammonia; $C_{Ai} = solubility$ of CO_2 gas in the ammonia solution; $C_B = concentration$ of ammonia in bulk; $z = stoichiometric coefficient of the reaction; <math>\delta = film$ thickness.

Now, to approximate the kinetics involved in this process, the following are stated:

- (1) The reaction between CO₂ and aqueous NH₃ is an acid—base reaction, which could be described as fast *instantaneous*, second-order reactions.
- (2) Reactions can take place in both the liquid film and in the main body of the liquid in general. However, for this reaction, it is assumed that it takes place at a plane within the liquid film (narrow zone).
 - (3) Liquid film resistance is dominant.
- (4) Despite the very fast reactions by which free CO_2 is destroyed chemically, there is, nevertheless, a very appreciable resistance to its passage into solution. Most of the resistance is attributed to the liquid phase. (Other details were given in part 1).
- (5) Molecules of the solute (CO_2) react with molecules of the reactant (NH_3 (aq)) whenever both are present at the same point in the liquid. Chemical equilibrium, therefore, exists everywhere in the liquid phase, and a further increase in the reaction rate would have no effect. This situation is designated as the "instantaneous reaction regime", in which the rate of mass transfer is independent of chemical kinetics and dependent only on factors affecting the physical transfer of reactants and reaction products.

Having proposed the instantaneous reaction model to describe our system, we turn next to verify this regime. In the course of providing this proof, the concentration of unreacted ammonia in the bulk (C_B) is to be found. The formulation of an algorithm to solve for C_B is to be carried out first as illustrated in the next section. Before this is done, a summary of the main tasks involved in the modeling process is given as follows:

- (1) Presentation of the main diffusion equation accompanied by chemical reaction for carbon dioxide in aqueous ammonia solution. Two different domains in the gas absorption process are identified: (i) interface region; (ii) liquid bulk region.
- (2) Formulation of the relationships that describe the interactions between different species participating in

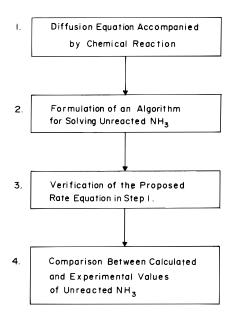


Figure 1. Solution steps for modeling.

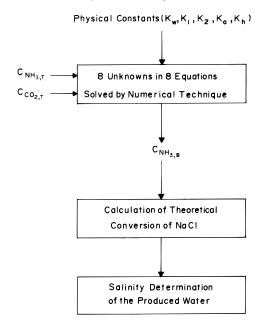


Figure 2. Schematic determination of unreacted NH₃.

this reaction. This constitutes a set of nonlinear equations which provides an algorithm to be solved for the dependent parameter, that is, unreacted ammonia $C_{\rm B}$ for a given set of the independent parameters: CO_2 absorption and the total NH_3 used in a given run. Formulation of this algorithm is applied to the liquid bulk region, that is, $C_{\rm B}$.

- (3) Verification of the instantaneous regime, where a graphical plot is carried out between the absorption rate of CO_2 versus the value of C_B .
- (4) Finally, values of unreacted ammonia \mathcal{C}_B determined experimentally are to be compared with the theoretical values obtained in step 2. This comparison is valid provided that the experimental conditions allow for enough absorption of carbon dioxide to form the ammonium bicarbonate regime. This modeling will establish a system that provides a method to calculate the conversion efficiency of the separation process. These steps are proposed in the form of a block diagram as shown in Figures 1 and 2.

Table 1. Jacobian Matrix^a

-[OH ⁻]	-[H ⁺]	0	0	0	0	0	0	-f1
$-[HCO_3^-]$	0	$-[H^{+}]$	$10^{-6.382}$	0	0	0	0	-f2
$-[CO_3^{2-}]$	0	$10^{-10.377}$	0	$-[H^{+}]$	0	0	0	-f3
$-[NH_3]$	0	0	0	0	$-[H^{+}]$	2.2^{-10}	0	-f4
0	0	$-[NH_3]$	0	0	$-[HCO_3^-]$	0	0.285	-f5
0	0	0	0	0	-1	-1	-1	-f6
0	0	-1	00	-1	0	0	-1	-f7
1	-1	-1	0	-2	0	+1	-1	-f8

 $[CO_2^{2-}] + [H_2NCOO^-]$. f8 = $[NH_4^+] + [H^+] - \{[OH^-] + [HCO_3^-] + 2[CO_3^{2-}] + [NH_2COO^-]\}$.

Table 2. Equilibrium Composition of an Aqueous Carbonated Ammonia Solution (6.2 M NH₃, Flow 2 L/min, Temp 22 °C)

time (min)	[CO ₂] (mol/L)	[H ⁺] (mol/L)	[OH ⁻] (mol/L)	[HCO ₃ ⁻] (mol/L)	[H ₂ CO ₃] (mol/L)	[CO ₃ ⁻] (mol/L)	[NH ₃] (mol/L)	[NH ₄ ⁺] (mol/L)	[H ₂ NCOO ⁻] (mol/L)
1	0.054 66	4.0394×10^{-12}	0.0025	$5.2588 imes 10^{-7}$	5.1192×10^{-12}	0.0546	6.0882	0.1118	1.1234×10^{-5}
4	0.13292	$9.8922 imes 10^{-12}$	0.01	$3.1308 imes 10^{-6}$	7.4638×10^{-11}	0.1329	5.9332	0.2668	$6.5178 imes 10^{-5}$
5	$0.155\ 66$	1.1661×10^{-11}	$8.5755 imes 10^{-4}$	$4.3217 imes 10^{-6}$	1.2145×10^{-10}	0.1556	5.8878	0.3121	$8.9282 imes 10^{-5}$
6	0.17654	1.3311×10^{-11}	$7.5128 imes 10^{-4}$	$5.5943 imes 10^{-6}$	1.7945×10^{-10}	0.1764	5.8462	0.3537	$1.1476 imes 10^{-4}$
7	0.21654	1.6539×10^{-11}	$6.0463 imes 10^{-4}$	$8.5247 imes 10^{-6}$	$3.3978 imes 10^{-10}$	0.2164	5.7663	0.4335	$1.7248 imes 10^{-4}$
8	0.231 83	1.7798×10^{-11}	$5.6187 imes 10^{-4}$	$9.8208 imes 10^{-6}$	$4.2123 imes 10^{-10}$	0.2316	5.7358	0.4640	$1.9765 imes 10^{-4}$
10	0.30804	2.4276×10^{-11}	$4.1192 imes 10^{-4}$	$1.7794 imes 10^{-5}$	1.0410×10^{-9}	0.3077	5.5835	0.6161	$3.4861 imes 10^{-4}$
20	$0.558\ 33$	4.8286×10^{-11}	$2.0710 imes 10^{-4}$	$6.4087 imes 10^{-5}$	$7.4574 imes 10^{-9}$	0.5571	5.0832	1.1157	0.0011
40	0.95625	$9.7953 imes 10^{-11}$	$1.0209 imes 10^{-4}$	$2.2231 imes 10^{-4}$	$5.2479 imes 10^{-8}$	0.9527	4.2876	1.9090	0.0033
60	1.372 91	1.7439×10^{-10}	$5.7343 imes 10^{-5}$	$5.6728 imes 10^{-4}$	2.3841×10^{-7}	1.3655	3.4547	2.7384	0.0069
80	1.758 33	2.8718×10^{-10}	$3.4821 imes 10^{-5}$	0.0012	8.2666×10^{-7}	1.7459	2.6845	3.5042	0.0113
100	2.062 08	4.3479×10^{-10}	$2.3000 imes 10^{-5}$	0.0021	$2.2189 imes 10^{-6}$	2.0445	2.0779	4.1066	0.0154
120	2.313 75	$6.4278 imes 10^{-10}$	$1.5557~E^{-5}$	0.0035	$5.4340 imes 10^{-6}$	2.2908	1.5760	4.6046	0.0194.

Formulation of an Algorithm To Solve for C_B

In this step, the setup of the main equilibrium relationships involved in the formation of ammonium bicarbonate and the formulation of a system of nonlinear equations is considered. A numerical solution is then carried out using the Newton/Raphson technique to compute the equilibrium compositions of all components involved in the system, mainly the unreacted ammonia concentration as a function of the rate of carbon dioxide absorption and the initial ammonia concentration. The main equilibrium relationships are:

$$H_2O \rightarrow OH^- + H^+ \tag{11}$$

$$H_2CO_3 \to HCO_3^- + H^+$$
 (12)

$$HCO_3^- \to CO_3^{2-} + H^+$$
 (13)

$$\mathrm{NH_4}^+ \! \rightarrow \mathrm{NH_3} + \mathrm{H}^+ \tag{14}$$

$$NH_2COO^- + H_2O \rightarrow NH_3 + HCO_3^-$$
 (15)

Comments on These Reactions. Equation 11 represents the equilibrium dissociation reaction of water, while eqs 12 and 13 are the first and second equilibrium dissociation reactions of carbonic acid, respectively. Equation 14 is the equilibrium dissociation reaction of ammonia in an aqueous solution, while eq 15 represents the hydrolysis of ammonium carbamate. This reaction is assumed to be at equilibrium in the liquid bulk. Short contact time in the interface region prevents the hydrolysis of the carbamate to take place as NH₃ being depleted; besides, reaction times of interest in the bulk of the liquid are long enough to allow equilibrium conditions. Reference is made to part 1 in this respect.

A system of eight nonlinear equations is then constructed as follows:

$$K_{\mathbf{W}} = [\mathbf{H}^+][\mathbf{O}\mathbf{H}^-] \tag{16}$$

$$K_{1} = \frac{[H^{+}][HCO_{3}^{-}]}{[H_{2}CO_{3}]}$$
 (17)

$$K_2 = \frac{[H^+][CO_3^{\ 2^-}]}{[HCO_3^{\ -}]}$$
 (18)

$$K_{\rm a} = \frac{[{\rm H}^+][{\rm NH_3}]}{[{\rm NH_4}^+]} \tag{19}$$

$$K_{\rm h} = \frac{[{\rm NH_3}][{\rm HCO_3}^-]}{[{\rm NH_2COO}^-]}$$
 (20)

$$[NH_3]_T = [NH_3] + [NH_4^+] + [NH_2COO^-]$$
 (21)

$$[CO_2]_T = [HCO_3^-] + [CO_3^{2-}] + [NH_2COO^-]$$
 (22)

$$[NH_4^+] + [H^+] = [OH^-] + [HCO_3^-] + 2[CO_3^{2-}] + [NH_2COO^-]$$
 (23)

The chemical equilibria represented by eqs 16-20 describe the situation of the bulk phase after time t of absorbing CO2 at a specified rate and under the assumption of instantaneous chemical equilibrium.

This system of eight nonlinear equations (16-23) has eight unknowns. Equations 21 and 22 are the total material balance for ammonia and carbon dioxide, respectively, while eq 23 is the electroneutrality or charge balance of all species present in the system. Table 1 shows the Jacobian matrix of the above system of equations. The solution of these equations provides the concentration of unreacted (free) ammonia, in addition to other equilibrium concentrations. Table 2

Figure 3. Verification of the instantaneous regime.

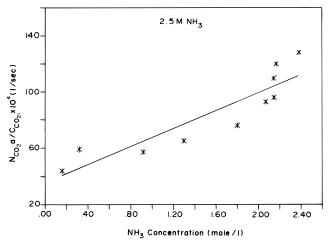


Figure 4. Verification of the instantaneous regime.

provides data for equilibrium concentrations for the case of a 6.2 M ammonia solution, obtained by the proposed numerical solution.

Verification of the Proposed Rate Equation

(a) Using the Approximate Approach (Film Model). Now verification of the existence of an instantaneous regime can be carried out, as the rate of absorption of carbon dioxide was measured experimentally. As proposed earlier, the absorption rate of CO_2 is given by:

$$N_{\rm A}a = k^0 a C_{\rm Ai} E_{\rm i} \tag{24}$$

where E_i , the enhancement factor for instantaneous reaction, is defined by eq 8.

Substitution of the value of E_i (given by eq 8) into eq 24 with some rearrangement gives the following result:

$$\frac{N_{\rm A}a}{C_{\rm Ai}} = k^0 a \left\{ 1 + \left(\frac{D_{\rm B}}{D_{\rm A}ZC_{\rm Ai}} \right) C_{\rm B} \right\} \tag{25}$$

If the instantaneous regime prevails, then the plot of the left-hand side of the equation versus \mathcal{C}_B should give a straight line. For this verification, it is important to know the unreacted ammonia concentration in the bulk. This ammonia concentration was obtained from the solution of the nonlinear equation considered above. Figures 3–5 show the existing instantaneous regime, for different cases of initial ammonia concentration used in the separation process.

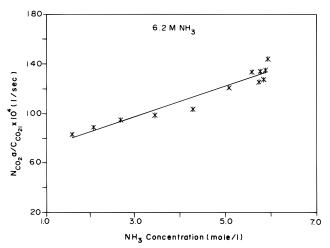


Figure 5. Verification of the instantaneous regime.

A further check utilizing these correlations appears to be relevant. The ratio, \mathcal{R} , between the ordinate read in Figures 3–5 at $\mathcal{C}_B=0$ and the slope of the straight lines is readily found to be:

$$R = (zC_{Ai})\frac{D_A}{D_B}$$
 (26)

If it is assumed, for the time being, that $D_A/D_B \cong 1$ and taking z=2 (as given by reaction 1), the following results arise for C_{Ai} :

initial concentration of NH $_3$ (number of moles): 1 2.5 6.2 calculated values of R: 0.40 1.1 5.10 calculated values of $C_{\rm Ai}$ [mol/L]: 0.20 0.55 2.55

The conditions implied by setting $\mathcal{C}_B=0$ may lead to the understanding that a comparison could be made with the case of absorption of CO_2 with *pure water* at saturation conditions of a pressure of 1 atm and 20 °C, where \mathcal{C}_{Ai} is reported to be 0.04 mol/L (Danckwerts, 1970). This value is not consistent with the ones calculated above for the experient under investigation. It is true that calculations took place by setting $\mathcal{C}_B=0$ for the ammonia in solution, but it is also true that other active chemical species are produced as a result of the chemical reaction. Their presence in the solution will contribute to increasing the absorption rate of CO_2 , over and above the case of pure water.

The following facts are in support of this conclusion:

- (1) Although NH_3 is depleted from a solution (i.e., C_B = 0), other active species are present in the solution. Most important are the OH^- , CO_3^{2-} , HCO_3^- , and NH_2COO^- .
- (2) OH^- has a strong influence on the absorption of CO_2 . Data published on the absorption coefficients for CO_2 in various solvents show that the value of (K_a) using NaOH is 40 times that of water (Sherwood et al., 1975).
- (3) Ibrahim (1993) reported that the pH values of the ammoniated solutions were in the range of 11-12. The reaction of CO_2 with OH^- is significant at such extremely basic conditions (Cussler, 1984). This enhances the absorption rate of CO_2 by such a solution.
- (4) Ibrahim (1993) referred to the fact that CO_2 could be consumed due to the reversion of carbamate as given by:

$$CO_2 + NH_2COO^- + 2H_2O \rightarrow NH_4 + 2HCO_3$$

To make that point more convincing, it is suggested to relate the value of C_{Ai} for the absorption of CO_2 in pure water—call it C_{Ai} —to other values calculated

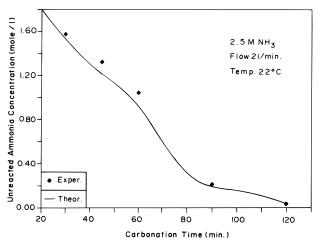


Figure 6. Comparison between calculated and experimental unreacted NH₃.

above for different concentrations of NH₃. The following form is proposed:

$$C_{\rm Ai} = C_{\rm Ai}^{\circ} (1 + 4N^{1.5}) \tag{27}$$

where C_{Ai} is the concentration of CO_2 for a given initial ammonia concentration of N moles of NH₃ and C_{Ai} is the concentration of CO₂ at standard conditions in pure water = 0.04 mol/L.

The proposed form (eq 27) could be checked for the conditions reported as follows:

for an initial concentration of 0 1 2.5 6.2 NH
$$_3$$
, N modes: calculated values of C_{Ai} : 0.04 0.2 0.67 2.51

Thus, the validity of eq 27 sounds reasonable. In support of this interpretation, a distinction has to be made between an aqueous system of pure water and CO_2 (that is, N=0 and $C_B=0$) and an ammonia solution/CO₂ system with different N concentrations and $C_{\rm B}=0$ as well.

Once the regime of reaction is established, a comparison can be carried out between the values of unreacted ammonia obtained from the experimental results as reported by Ibrahim (1993) and the corresponding values obtained using the numerical solution presented above. The theoretical calculation of unreacted ammonia requires feeding parameters such as the total amount of carbon dioxide absorbed for the specified time of carbonation and the total amount of ammonia fed to the column. The values of unreacted ammonia are plotted in Figures 6 and 7. These curves show a close agreement between experimental and theoretical val-

The experimental data reported in these figures were taken using the same experimental setup and under the same operating conditions as the ones involved in constructing the instantaneous regime.

(b) Using the Exact Solution (Penetration **Theory).** Another way of verifying that the system behaves as a diffusion-controlled mass-transfer system with an instantaneous reaction regime is to study the rate of absorption of carbon dioxide both experimentally and theoretically. This was made possible by using the total carbon analyzer (TOC) machine that measures the experimental absorption rates of CO₂ at different initial ammonia concentrations. The same experimental setup, that is, bubble column, is used in this type of experiment. The experimental values can be compared to theoretical values of absorption rates of carbon dioxide.

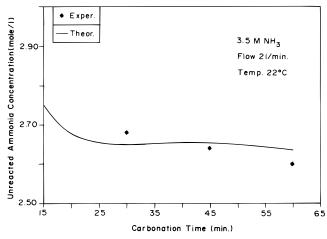


Figure 7. Comparison between calculated and experimental unreacted NH₂

In this case, the instantaneous enhancement factor, E_i , may be expressed, according to Danckwerts (1970), as:

$$E_{\rm i} = 1/{\rm erf}(\sigma/\sqrt{D_{\rm A}}) \tag{28}$$

where σ is defined by

$$e^{\sigma^2/D_B} \operatorname{erfc}\left(\frac{\sigma}{\sqrt{D_B}}\right) = \frac{C_{B^0}}{zC_{Ai}} \sqrt{D_B/D_A} e^{\sigma^2/D_A} \operatorname{erfc}\left(\frac{\sigma}{\sqrt{D_A}}\right) \quad (29)$$

Equations 28 and 29 are solved numerically, and the theoretical absorption rates of carbon dioxide are computed using eq 30:

$$N_{\rm A}a = 2\sqrt{\frac{D_{\rm A}}{\pi t}}aC_{\rm Ai}E_{\rm i} \tag{30}$$

Computational steps are illustrated by Figure 8. The results are shown graphically as a plot of the absorption rate of CO₂ against *carbonation time*. Figures 9–11 give the comparison between experimental and theoretical absorption rates of CO₂. The good agreement between the corresponding experimental and theoretical values again confirms the reaction regime underlying the separation process of the presented system.

A list of the values of parameters used in this investigation is given as follows:

NH₃ concentration (M): 0.5 - 6.2flow rate of CO₂ gas (L/min): 2 - 3absorption (carbonation) time (s): 100-8000

Discussions and Conclusions

The absorption of CO₂ in ammoniated brine offers a dual-action scheme for purification and separation in both the gas and liquid phases. The scheme as such can be recommended for the purification of industrial gases containing either a CO₂ or CO₂/NH₃ mixture. At the same time, chemical conversion takes place in the liquid phase, leading to the separation of NaCl or the desalting of the brine solutions. The main findings reported in this paper could be summarized as follows:

- (1) The CO₂ absorption accompanied by chemical reaction underlying the separation process was shown to be liquid diffusion-controlled. Such a regime holds at long enough contact times; that is why the characteristics of bubble columns are significant in reaching these results.
- (2) The numerical-solution procedure provides a method for the determination of unreacted ammonia for a given condition of total ammonia fed to the reactor and the amount of CO₂ absorbed. Values are compared

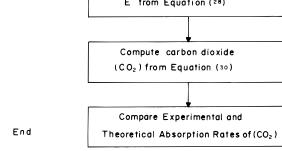


Figure 8. Verification of the absorption regime.

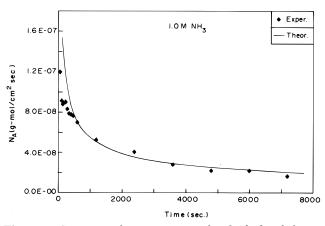


Figure 9. Comparison between measured and calculated absorption rates of CO_2 .

with experimental data for some runs. A close agreement is reported. The theoretical absorption rate of CO_2 was computed, and comparison was made with the experimental data.

(3) In presenting the exact solution, it was observed that a good matching between theory and experiments is achieved for long contact time particularly over 1000 s. Predicted values, on the other hand, are consistently greater at shorter contact times. This could be most significant because short times will be involved in agitated systems of practical interests.

(4) The above conclusions, although they are applicable for a CO₂/NH₃ solution system, can be extended for a CO₂/ammoniated brine system. The presence of NaCl in such a system leads to the precipitation of NaHCO₃ (soda ash), which hinders data-taking for

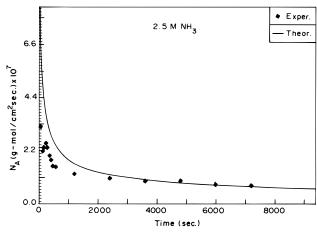


Figure 10. Comparison between measured and calculated absorption rates of CO_2 .

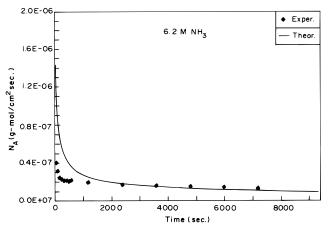


Figure 11. Comparison between measured and calculated absorption rates of CO_2 .

proving the instantaneous regime. Despite this observation, runs were made with brine as well.

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