5. SUMMARY, CONCLUSIONS AND RECOMMENDATIONS

5.1 SUMMARY OF RESULTS

The primary objective of this study was to determine the relative efficiency of static mixers as gas/liquid contactors. This was accomplished through an experimental investigation of interfacial areas and mass transfer coefficients obtained per dissipated power for gas/liquid co-current upflow.

Experiments were performed on three different types of static mixers, namely:

1) Kenics;

2) Ross LLPD;

3) Koch CY with spacers;

placed in a 1 inch diameter pipe. The experimental apparatus was set up for evaluation of liquid holdup and total pressure drop as well as for the parameters necessary to evaluate interfacial areas and mass transfer coefficients.

The interfacial areas and mass transfer coefficients were determined with a chemical method and the use of Danckwerts' plots for a chemical system of carbon dioxide absorption into carbonate-bicarbonate buffer solution with arsenite catalyst. All experiments were conducted at atmospheric pressure and room temperature.

The operating variables (i.e. V_L and V_G) were chosen so that the operating flow conditions were in the bubble flow

regime. The experimental fluid velocities cover the range: V_G = 0.073 to 0.324 m/s and V_L = 0.193 to 0.630 m/s.

The liquid holdup, $1-\epsilon$, ranged from 0.97 to 0.70 and varied with gas and liquid flowrates. Each mixer type differed in how the holdup varied with these parameters. Neither the homogeneous model nor the Lockhart-Martinelli correlation accuratly predicted the holdup values.

The Koch and Kenics mixers had holdup configurations similar to those found by Yung-Hsu for gas-lift reactors. The following correlation form was suggested:

$$\frac{(1-\epsilon)}{\epsilon} = D \left[\frac{V_L}{V_G}\right]^m \tag{34}$$

where $D = f(V_L, \rho_L, \mu_L, \sigma...)$

The same form could be used for the Ross mixer, except the leading term, D, would be a constant.

The Kenics mixer in a horizontal configuration gave liquid holdups smaller than in a vertical position at all flow conditions.

The Koch CY mixer produced the largest kinetic pressure drop per unit length of the three mixers tested. It's pressure drop ranged from 10 to 65 kPa/m which was 5 times greater than the Kenics and Ross mixers. The pressure drop, like the holdup was a function of the gas and liquid flow-rates. Increased gas flowrate increased the pressure drop.

Table 4 presents the results of the absorption exper-iments. These results showed that k_L was not a function of flowrates and averaged out at a value of 1.84×10^{-4} m/s.

Table 4

Summary of Experimental Results

Mixer	V_L	V_G	$1-\epsilon$	a	k_L	$k_L a$	P_w
	m/s	m/s		m^{-1}	m/s $\times 10^4$	s^{-1}	W/m^3 $\times 10^{-3}$
Kenics Vert.	0.630	0.146	0.93	1340	1.68	0.225	9.43
	0.450	0.146	0.90	754	1.81	0.136	3.90
	0.193	0.146	0.88	394	1.92	0.076	0.42
	0.450	0.219	0.87	1030	1.70	0.175	4.36
	0.450	0.073	0.95	484	2.13	0.103	3.44
Kenics Hor.	0.450	0.146	0.82	1140	1.34	0.152	5.11
Ross LLPD	0.630	0.146	0.93	776	2.28	0.177	8.84
	0.450	0.146	0.91	481	1.85	0.089	4.22
	0.193	0.146	0.85	285	1.83	0.052	0.52
Koch CY with spacers	0.193	0.324	0.70	4180	1.84	0.769	4.64
	0.193	0.217	0.79	2150	1.84	0.395	3.47
	0.193	0.103	0.88	1620	1.84	0.298	2.42
	0.450	0.217	0.89	4600	1.84	0.847	19.8
	0.579	0.217	0.83	8150	1.84	1.50	43.9
	0.579	0.217	0.91	6140	1.84	1.13	36.1
	0.579	0.103	0.96	2690	1.84	0.494	30.6

The amount of interfacial area per unit liquid volume varied with liquid and gas flowrates according to the following relationships:

Kenics $\qquad a \propto V_L^{1.0} \; V_G^{0.68}$ $\hspace{3cm}$ (45)

Ross LLPD $\qquad a \propto V_L^{0.85} \; V_G^{0.68}$ $\hspace{3cm}$ (46)

Koch CY $\qquad a \propto V_L^{0.67} \; V_G^{0.89}$ $\hspace{3cm}$ (47)

The Koch mixer produced 4 times more interfacial area at any given flow conditions than the others, while the Kenics mixer was twice as productive as the Ross mixer.

The comparative efficiencies of the static mixers are reflected in the following correlations:

Kenics $\qquad \dfrac{A}{V_R} = 77 \left[\dfrac{E}{V_R}\right]^{0.42} (V_G)^{0.59}$ $\hspace{2cm}$ (49)

Ross LLPD $\qquad \dfrac{A}{V_R} = 34 \left[\dfrac{E}{V_R}\right]^{0.48} (V_G)^{0.59}$ $\hspace{2cm}$ (50)

Koch CY $\qquad \dfrac{A}{V_R} = 344 \left[\dfrac{E}{V_R}\right]^{0.37} (V_G)^{0.76}$ $\hspace{2cm}$ (51)

The Koch CY mixer with spacers was the most efficient of the static mixers tested as gas/liquid contactors. When compared with other gas/liquid contactors, the Koch mixer was competitive with a packed bubble column reactor.

5.2 CONCLUSIONS

The main conclusion of this study is that the Koch CY mixer with spacers is a more efficient gas/liquid contactor than a Kenics or Ross LLPD static mixer.

The other conclusions of this study are as follows:

1) The Koch CY mixer with spacers provides five times more interfacial area per power dissipated than the Kenics mixer and 10 times more than the Ross mixer.

2) As a general rule for static mixers:

$$\frac{A}{V_R} \propto \left(\frac{E}{V_R}\right)^{0.4}$$

which is the same dependence as that found for packed bubble columns, bubble columns and empty pipes.

3) The Koch mixer is competitive with the packed column as a co-current gas/liquid contactor.

4) The mass transfer coefficient, k_L, is not a function of the liquid or gas velocities for gas bubble diameters less than 3.0×10^{-3}m in the static mixers.

5) The commonly used homogeneous model and Lockhart-Martinelli correlation do not adequately predict holdup and pressure drops for co-current gas/liquid upflow in static mixers.

5.3 RECOMMENDATIONS

The following are recommendations directed toward industry:

1) If the unique charateristics that a static mixer gives are prefered, (i.e. plug flow characteristics, low maintainance costs, horizontal configuration, radial thermal homogenization ...) then pack the tubular reactor with Koch CY mixers with element sized spacers.

2) If mixing is currently done in a pipe with no mixers, then better mixing efficiency can be accomplished by using static mixers.

The following recommendations are directed toward further experimentation:

3) Studies concerning the optimal spacing of the Koch CY mixers should be done.

4) Better correlations need to be devised to accurately predict pressure drop and holdup in vertical and horizontal two phase flow in static mixers.

7. ACKNOWLEDGMENTS

7.   APPENDICES

APPENDIX 7.1

Derivation of Rate of Absorption for Single Irreversible (Pseudo) First Order Reaction

The unsteady mass balance for gas A around a one dimensional element of liquid B with a pseudo-first order irreversible reaction can be written as:

$$\frac{\partial C_A}{\partial t} = D_A \frac{\partial^2 C_A}{\partial y^2} - rC_A \qquad (54)$$

with the following boundary conditions:

$$y = 0 \qquad t > 0 \qquad C_A = C_{A_i} \qquad (55)$$

$$y = \infty \qquad t > 0 \qquad C_A = 0 \qquad (56)$$

and initial condition:

$$t = 0 \qquad y > 0 \qquad C_A = 0 \qquad (57)$$

This partial differential equation can be solved by the method of Laplace Transforms to give:

$$\overline{C}_A = \frac{C_{A_i}}{s} \exp \left[-\sqrt{\frac{s+r}{D_A}} \, y \right] \qquad (58)$$

The molar flux of A at any time, t, across the gas/liquid interface by definition is given by the following equation:

$$N_A(t) = -D_A \frac{\partial C_A}{\partial y} \bigg|_{y = 0} \qquad (59)$$

Danckwerts' surface renewal theory provides the following equation for the average molar flux:

$$N_A = \int_0^\infty N_A(t)\, se^{-st}\, d\,t \tag{60}$$

Replacing Equation (59) into Equation (60), reversing the differentiation and integration and using the definition of Laplace Transforms the following equation is obtained:

$$N_A = -D_A s \left. \frac{\partial \overline{C}_A}{\partial y} \right|_{y\,=\,0} \tag{61}$$

The molar flux can now be solved by substituting Equation (58) into the above and performing the differentiation;

$$N_A = C_{A_i} \sqrt{D_A(s+r)} \tag{62}$$

Now a relationship for s can be obtained by letting $r \to 0$ and substituting for N_A with the expression for the flux due to physical absorption in terms of the mass transfer coefficient: This gives:

$$s = \frac{k_L^2}{D_A} \tag{63}$$

By substituting equation (63) into equation (62), dividing through by k_L^2 and using the definition of the Hatta number as expressed in equation (17), the molar flux can be written as:

$$N_A = k_L a \ C_{A_i} \ \sqrt{1 + Ha^2} \tag{22}$$

This equation provides the definition of the enhancement factor for absorption of gas A into a liquid B with an irreversible pseudo first order reaction using Danckwerts' surface renewal theory and can be expressed as:

$$E_L = \sqrt{1 + Ha^2} \tag{21}$$

According to Linek (15), for this derivation to hold true, two conditions must be satisfied:

$$1) \quad E_{i/2} \gg H_a \tag{53}$$

$$2) \quad H_a \gg 3 \tag{54}$$

$$\text{where} \quad E_i = 1 + \frac{D_B}{D_A} \cdot \frac{C_{B_o}}{C_{A_i}} \tag{18}$$

$$\text{and} \quad H_a = \frac{\sqrt{D_A \ r}}{k_L} \tag{17}$$

These requirements will be discussed in Appendix 7.8.

APPENDIX 7.2

Holdup, Total Pressure Drop and Absorption Procedures

7.2.1 Hold Up Procedure

1) Disconnect the two pressure tap lines, sealing the top one and attaching a shut-off valve on the bottom tap. Also disconnect the gas line from the bottom of the reactor and attach another shut-off valve.

2) Fill column with water, including the separator.

3) Measure the volume of liquid that drains out of the bottom pressure tap. Record this volume as u1.

4) Measure the volume of the remaining liquid that drains out of the gas inlet to the reactor. Record this volume as u2.

5) Reconnect the gas line to the bottom of the reactor.

6) With the gas and liquid streams flowing at the desired rates and the liquid level in the separator stable and below the wire screen, quickly and simultaneously shut off the master switch (which controls both the gas solenoid valve and the pump in this case) and the liquid on-off valve, v2.

7) Allow liquid to settle in the reactor.

8) Measure the volume of liquid that drains from the bottom pressure tap. Record this volume as u3.

9) Calculate and record the liquid holdup:

$$\epsilon = (u3 - u2) / (u1 - u2) \tag{64}$$

7.2.2 Total Pressure Drop Procedure

1) Measure the distance between the two pressure taps on the reactor. Record this distance as L_2.

2) Using water as the process fluid, circulate the water throughout the system allowing water to fill the pressure tap lines. For water, the purge stream apparatus is not needed.

3) Turn on gas flow and adjust to the desired rate. Maintain steady conditions.

4) Measure the difference in height of the two mano- meters. Record this difference as L_1.

5) Calculate and record the total pressure drop:

$$\Delta P_T \quad = \quad (L_1 + L_2) \; g \; \rho_{H_2O} \tag{29}$$

7.2.3 Mass Transfer Procedure

The following sections provides a detailed procedure for one run.

7.2.3.1 Initial Solution Preparation

1) Fill container with 8 liters of water.

2) Accurately weigh out 508.8 grams of Na_2CO_3 and 134.6 grams of $NaHCO_3$ in separate weighing containers in order to make an 8 liter buffer solution of 0.6 M carbonate and 0.2 M bicarbonate.

3) Add the solids to the 8 liters and mix completely with electic stirrer.

4) With the nitrogen slightly flowing, turn on pump and circulate the solution throughout the system for a few minutes.

5) Pipet 10 ml of solution and accurately titrate with 1 N HCl for total base concentrations (TBC). See TBC procedure on page 107. Record the intital and final buret readings. Their difference will equal $\Delta 1$.

6) Calculate and record the true volume of the solution:

$$V_{b \ (true)} = 8 \ (l) \ \frac{14 \ (ml)}{\Delta 1 \ (ml)} \qquad (65)$$

7) Add 500 grams (1 bottle) of $NaAsO_2$ to solution and mix completely.

8) Again with the nitrogen barely flowing, activate the pump and circulate the solution throughout the system for a few minutes.

9) Pipet 10 ml of activated solution and accurately titrate with 1 N HCl for TBC. Record initial and final buret readings. Their difference will equal $\Delta 2$.

10) Calculate and record true concentration of arsenite catalyst, [Ars]:

$$[Ars1] = \frac{\Delta 2 \ (ml) - \Delta 1 \ (ml)}{10 \ (ml)} \ x \ 1 \ N \qquad (66)$$

11) Calculate and record the grams of NaCl needed to maintain the ionic strenghth of the solution at 2.5.

$$\text{Grams}_{NaCl} = (0.5 - [Ars])(V_{b(true)})(58.44) \qquad (67)$$

12) Accurately weigh and add desired amount of NaCl to the solution. Again circulate solution throughout system to assure homogeneity.

7.2.3.2 Reaction Procedure

13) With the nitrogen flowing slowly, set liquid rate using pump motor variac to the desired flowrate refering to the liquid flowmeter. (It may be necessary to use the liquid control valve to help adjust the liquid flowrate especially at low levels).

14) Adjust the pinch valve on the separator outlet tubing in order to achieve and maintain the proper liquid level in the separator. The liquid level should be just above the wire screen.

15) Turn the master power switch off. This turns off the gas solenoid, but leaves the pump on. The pump should not be connected to the main power source.

16) Shut the nitrogen flow valve off, v_4, and open the CO_2 valve, v_3. Make sure that the CO_2 preheater is on.

17) Reset the timer which is connected to the main power source.

18) Collect a 50 to 75 ml sample of the initial solution from the outlet of the separator tube. Cap and label the flask "BEFORE".

19) Turn the master switch on and quickly adjust the CO_2 gas flowrate with the control valve to the desired flowrate refering to the gas inlet rotameter.

20) As the run proceeds, adjust and maintain the gas and liquid flowrates as well as the liquid level in the separator.

21) At the desired time, turn the master power switch off and quickly shut off the inlet gas control valve to assure that no CO_2 is inputed.

22) Open the separator outlet pinch valve and allow the solution to continue to circulate for a few minutes in order to assure a homogenous concentration.

23) Fill and label a second erlenmeyer flask with 50 to 75 ml of the final solution. Cap and label the flask "AFTER". Set the flask aside with "BEFORE" sample.

7.2.3.3 Dilution Procedure

24) Pipet out 10 ml of the batch solution and titrate the concentration of bicarbonate using the procedure on page 108. Record the initial and final buret readings. Their difference will equal $\Delta 3$.

25) Calculate and record the concentration of bicarbonate, $[HCO_3^-]$:

$$[HCO_3^-] = \frac{5 - (\Delta 3 \ (ml) - 10 \times [Ars])}{10} \quad (68)$$

26) Calculate the volume of solution to be removed and replaced by the same volume of water in order to obtain a bicarbonate concentration of 0.2 M by the following equation:

$$V_{b(replace)} = V_{b(true)} \times \left[1 - \frac{0.2}{[HCO_3^-]} \right] \qquad (69)$$

27) Remove a volume of solution equal to the volume calculated above less that volume that has previously been removed for samples and titrations. Add a volume of water equal to the volume calculated in step 26) to the remaining solution and circulate the solution throughout the apparatus.

28) Pipet out 10 ml of the diluted solution and titrate for the TBC. Record both the initial and final buret readings. Their difference will be $\Delta 4$.

29) Calculate and record actual dilution ratio, D_R, in order to determine the new concentrations of all the ionic species.

$$D_R = \Delta 4 \text{ (ml)} / \Delta 2 \text{ (ml)} \qquad (70a)$$

30) Record the new concentration of asenite, $[Ars2]$:
$$[Ars2] = [Ars1] \times (D_R) \qquad (70b)$$

31) Calculate the quantities of carbonate and bicarbonate needed to add in order to boost their concentrations to 0.6 M and 0.2 M, respectively (based on $V_{b(true)}$).

32) Add the carbonate and bicarbonate to the solution, mix and homogenize. Again pipet out 10 ml of new concentrated solution and titrate for the TBC. Record both initial and final readings. Their difference will be Δ5.

33) Calculate and record the actual volume by the following equation:

$$V'_{b_{true}} = \left[\left(\Delta 4 (V_{b_{(true)}}) + \left(\frac{2 \cdot Grams_{Na_2CO_3}}{106} \right) + \left(\frac{Grams_{NaHCO_3}}{84} \right) \right) \right] \Big/ \Delta 5 \qquad (71)$$

34) Calculate and add the amount of NaCl needed to maintain the ionic strength of the system equal to 2.5. This can be determined by the following equation:

$$Grams_{NaCl} = 58.44 \times V'_{b_{(true)}} \times (0.5 \times (1 - D_R)) \qquad (72)$$

35) Repeat steps 14) to 34) until [Ars] < 0.1 M.

7.2.3.5 Determination of CO_2 Absorption

1) Titrate a "BEFORE" sample for bicarbonate concentration to a color before the endpoint. (light rose to orange, see section 7.2.4.2).

2) Titrate two more "BEFORE" samples and three "AFTER" samples to the same color as the first titration. Record the initial and final buret readings for all titrations.

7.2.4 Chemical Analysis Procedure

7.2.4.1 Determination of Total Base Concentration

1) Pipet 10 ml of solution into a 250 ml erylenmeyer flask. Add two drops of bromothymol blue indicator.

2) Titrate solution with 1.0 N HCl from a 50 ml buret to a blue to yellow end point. Record the initial and final buret readings. Their difference will be Δ6.

3) Calculate the total base concentration as follows:

$$[Base] = (\ \Delta6(ml)\ /10\ (ml))\ \ x\ \ 1.0\ N \tag{73}$$

7.2.4.2 Determination of Bicarbonate Concentration

1) Pipet 10 ml of solution into a 250 ml erylemeyer flask.   Then pipet 5 ml of 1.0 N NaOH into the same flask and swirl.

2) Add 18 ml of 20% $BaCl_2$ to form the white $BaCO_3$ pre- cipitate.   Then add two drops of cresol red - thymol blue mixed indicator and swirl.

3) Immediately titrate the mixture with 1.0 N HCl from a 10 ml accurate buret to a pale orange color similar to a standard color of a previously titrated sample of known bi- carbonate concentration. (The mixed indicator color changes from purple to rose to white to yellow.   The end point is when the solution is white.   Since the samples contain arsenite the end point is very broad and difficult to detect as well as being time consuming.   The color matching is easier and more accurate.) Record the initial and final buret readings. Their difference will be Δ7.

4) The concentration of bicarbonate   is determined by the following formula:

$$[HCO_3^-] = \frac{5.0 - (\ \Delta7 - 10\ x\ [Ars]\ )}{10} \tag{74}$$

5) The titration of samples with no arsenite in solution has a different end point than the solutions with arsenite. The end point is very distinct; a color change from purple to pink. No color matching is needed.

APPENDIX 7.3

Rotameter Calibrations

This appendix provides the calibrations for the two gas rotameters and one liquid rotameter used in this study. They are provided for the benefit of future experimenters using the apparatus.
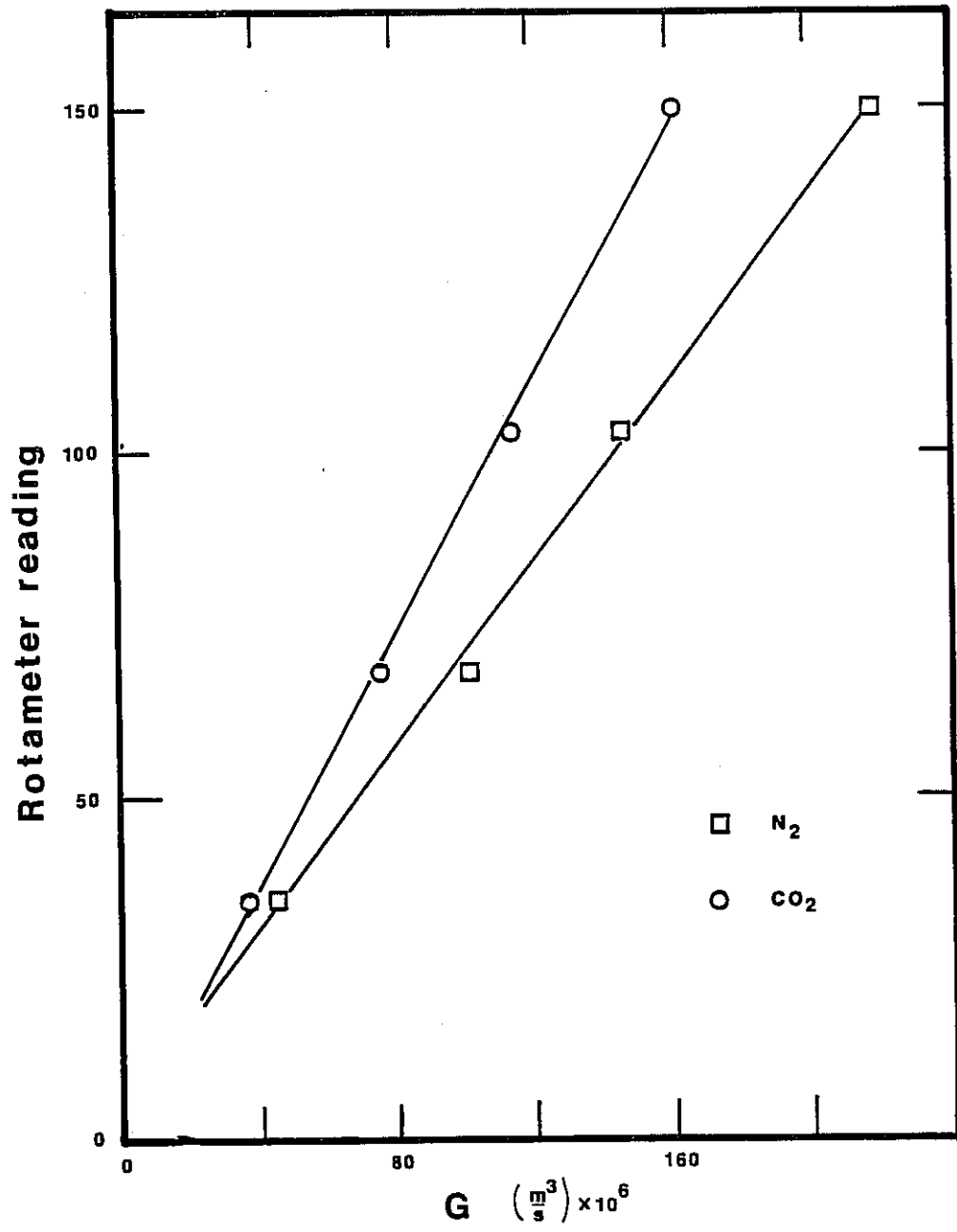
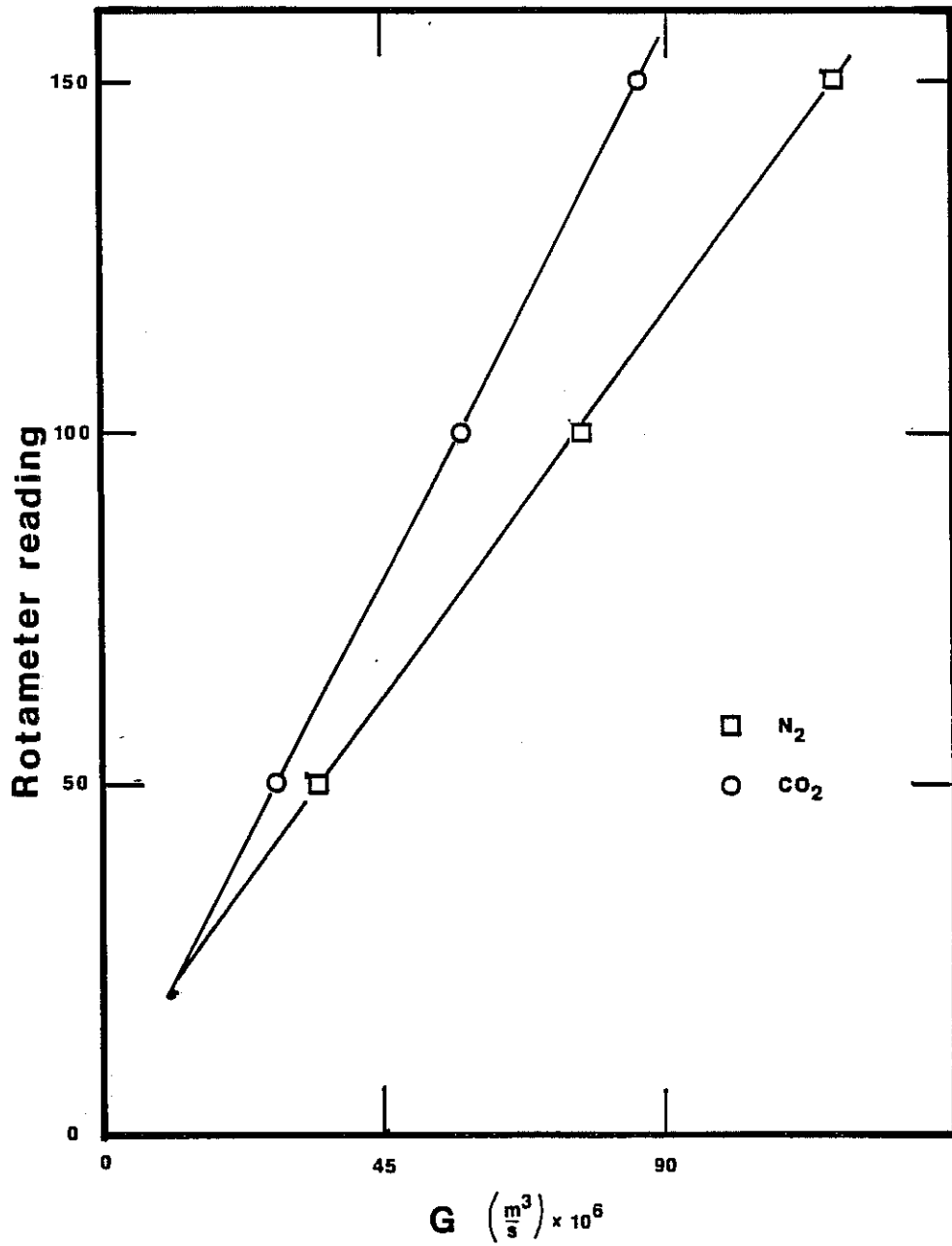Figure 7.3.1   Inlet Gas Rotameter Calibration Curve

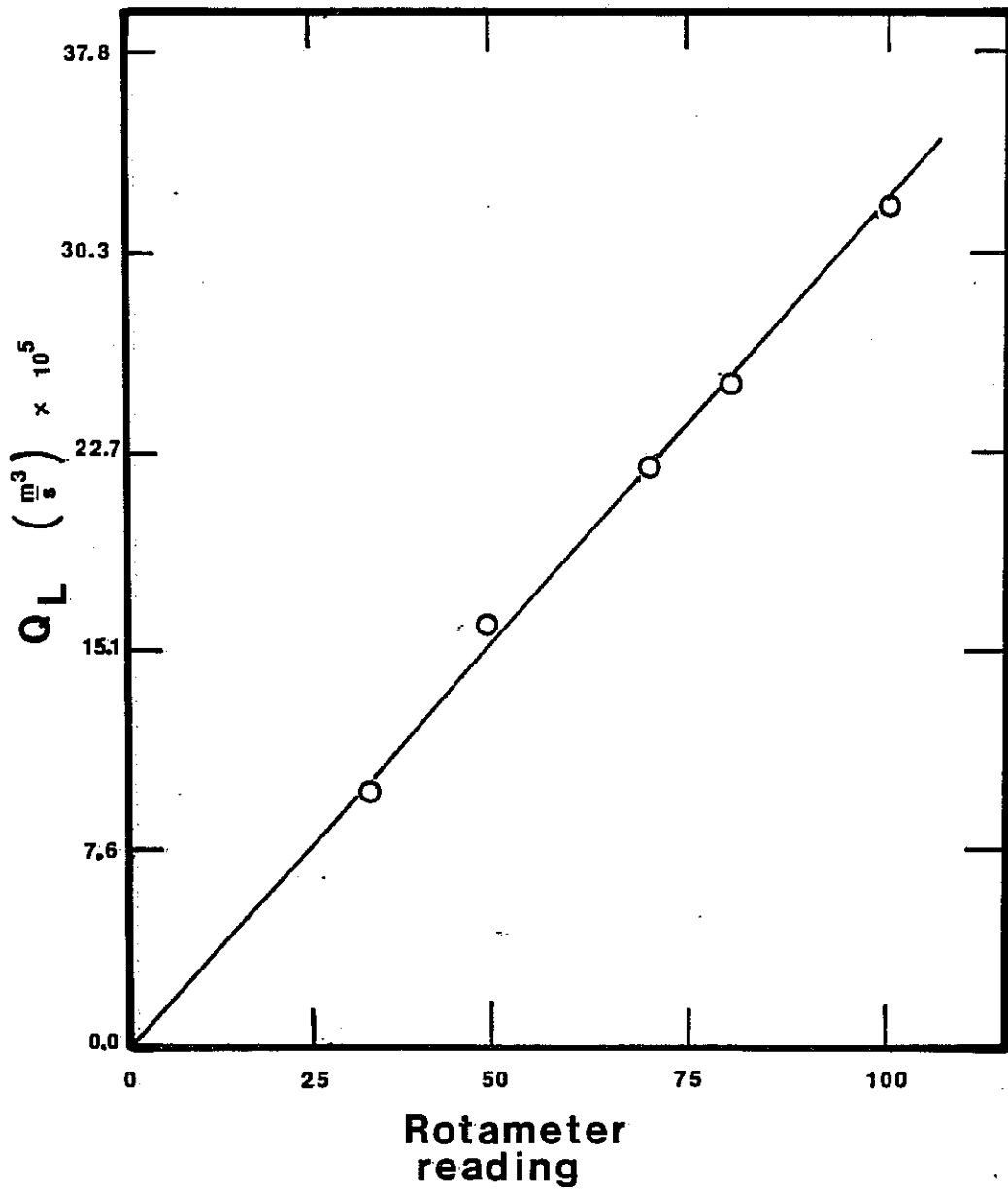Figure 7.3.2   Outlet Gas Rotameter Calibration Curve

Figure 7.3.3   Liquid Rotameter Calibration Curve

APPENDIX 7.4

Holdup Analysis

This analysis has the main purpose of determining the reason why the Koch and Kenics mixer have a family of lines on the plot of $(1-\epsilon)/\epsilon$ versus V_L/V_G and the Ross mixer does not.

Assume that for the static mixers the following experimentally found relationship holds:

$$\frac{1-\epsilon}{\epsilon} = D \left(\frac{V_L}{V_G}\right)^n \tag{34}$$

where $n \approx 1$.

Starting from the definition of slip velocity (21):

$$\Delta V = \frac{V_G}{\epsilon} - \frac{V_L}{1-\epsilon} \tag{32}$$

and solving for the holdup ratio one gets:

$$\frac{1-\epsilon}{\epsilon} = \frac{1-\epsilon}{V_G} \Delta V + \frac{V_L}{V_G} \tag{75}$$

Replacing Equation (34) into Equation (75) and dividing through by V_L/V_G allows D to be determined as:

$$D = \frac{1-\epsilon}{V_L} \Delta V + 1 \tag{76}$$

For the Ross mixer, since there is only one line, D is a constant for all liquid and gas velocities. Then, the consideration of two different liquid velocities provides the following equation:

$$\frac{(1-\epsilon)_1}{V_{L_1}} \Delta V_1 + 1 = \frac{(1-\epsilon)_2}{V_{L_2}} \Delta V_2 + 1 \tag{77}$$

or by rearranging:

$$\frac{\Delta V_2}{\Delta V_1} = \frac{(1-\epsilon)_1}{(1-\epsilon)_2} \frac{V_{L_2}}{V_{L_1}} \tag{78}$$

Supposing that :

$$\frac{V_{L_2}}{V_{L_1}} > 1 \tag{79}$$

then

$$\frac{(1-\epsilon)_1}{(1-\epsilon)_2} < 1 \tag{80}$$

However, since the change in liquid holdup is not large as the change in the liquid velocities, the following is true:

$$\frac{V_{L_2}}{V_{L_1}} \frac{(1-\epsilon)_1}{(1-\epsilon)_2} > 1 \tag{81}$$

and therefore

$$\frac{\Delta V_2}{\Delta V_1} > 1 \tag{82}$$

The confirmation of the last inequality comes from a case study from the Ross mixer data.

$$V_{L_1} = 0.19 \text{ m/s} \qquad (1-\epsilon)_1 = 0.76$$

$$V_{L_2} = 0.45 \text{ m/s} \qquad (1-\epsilon)_2 = 0.84$$

$$V_{L_2} / V_{L_1} = 2.13$$

This shows, in the Ross mixer case, that the slip velocity increases as the liquid velocity increases.

Now for the Kenics and Koch mixer:

$$\frac{D_2}{D_1} < 1 \qquad \text{when} \qquad \frac{V_{L_2}}{V_{L_1}} > 1 \qquad (83)$$

This condition provides the following inequality:

$$\frac{\Delta V_2}{\Delta V_1} < \frac{(1-\epsilon)_1}{(1-\epsilon)_2} \frac{V_{L_2}}{V_{L_1}} \qquad (84)$$

Unfortunately this inequality gives no definitive statement about the slip velocity relationship except that the slip velocity must change less than in the Ross mixer case.

Considering a case study for the Kenics mixer for $V_G = 0.142$ m/s:

$$V_{L_1} = 0.19 \text{ m/s} \qquad\qquad (1-\epsilon)_1 = 0.840$$

$$V_{L_2} = 0.52 \text{ m/s} \qquad\qquad (1-\epsilon)_2 = 0.863$$

$$D_1 = 3.9 \qquad\qquad\qquad D_2 = 1.67$$

then

$$\frac{\Delta V_2}{\Delta V_1} = 0.615$$

For the Kenics and Koch mixer, the slip velocity is decreased as the liquid velocity is increased.

It also can be shown directly that the slip velocity is increased as the gas velocity is increased for all mixers.

APPENDIX 7.5

Separator/Sparger Correction

If the total volume of the apparatus is broken up into two parts, the reactor volume and the separator and sparger volume, the following equation holds for an overall mass balance:

$$(N_A a)_T \ V_T \ (1-\epsilon)_T = (N_A a)_S \ V_S \ (1-\epsilon)_S$$

$$+ \ (N_A a)_R \ V_R \ (1-\epsilon)_R \tag{85}$$

where the subscripts mean the following:

T = Total

S = Separator/Sparger

R = Reactor

Assuming that all the liquid holdups equal the measured average liquid holdup and solving for the desired rate of absorption in equation (85) one gets:

$$(N_A a)_R = \frac{(N_A a)_T \ V_T - (N_A a)_S \ V_S}{V_R} \tag{86}$$

Since all the volumes are known and $(N_A a)_T$ is the experimentally determined value, the value of $(N_A a)_S$ needs to be evaluated. Previous to this analysis $(N_A a)_S \ V_S$ was

assumed to be negligible. Unfortunately, this assumption
was incorrect.

A few runs were performed to determine values for
$(N_A a)_S$ and their variation with catalyst concentration and
gas flowrate. The gas was injected through the top
pressure tap in the same manner as in all the runs. The
liquid flowrate was set at 70% of the maximum. Everything
else was performed in the same manner as when dealing with
the injection in front of the mixer. Table 7.5.1 gives the
results of these experiments.

It was assumed that for a constant liquid flowrate,
the following empirical power law relationship holds

$$(N_A a)_S = D \ r^b \ G^c \tag{87}$$

where $G \equiv$ outlet gas volumetric flowrate $(\frac{cm^3}{s})$ and a,
b and D are empirical constants. From the four data
points D, b, and c were evaluated and averaged. The final
correlation became for one liquid flowrate:

$$(N_A a)_S = 2.30 \times 10^{-8} \ r^{0.41} \ G^{0.70} \tag{88}$$

For any outlet gas flowrate and catalyst concentration,
the rate of absorption can now be estimated at a liquid
rate of 70% of the maximum. For regular runs, the gas
flowrate out of the separator is unknown, but can be

Table 7.5.1

Separator/Sparger Absorption Results

r	G_{in}	$1-\epsilon$	$(N_A a)_S$	G_{out}
s^{-1}	cm^3/s		$gmole/cm^3 s$	cm^3/s
			$x\ 10^6$	
2.92	34	0.90	3.4	32
2.92	110	0.81	8.7	105
135	34	0.90	17.6	23
135	110	0.81	38.1	88

calculated by the overall mass balance:

$$G_{out} = G_{in} - \frac{(N_A a)_T \; V_T \; (1-\epsilon)_T}{(\rho_{CO_2} / M_{\omega \; CO_2})} \tag{89}$$

Finally $(N_A a)_R$ can be calculated from the equation (86) where $(N_A a)_S$ is calculated from equations (88) and (89).

Although the correction scheme seems logical, it is very cumbersome and does include some assumptions. This correlation is only good for one specific liquid flowrate. More experiments would have to be done at more liquid flowrates which would be very costly.

From the visual observation of the bubbles in the separator, a simpler correction scheme was devised. The bubbles from the reactor traveled straight up without any radial movement. Also the bubbles did not seem to coalesce as they traveled to the surface. Therefore, the reactor volume can be increased by an imaginary volume that extends from the liquid surface in the separator to the end of the physical reactor length through the core of the separator and having a diameter of 1 inch, the same as the tubular reactor. This volume corresponded to a value of 81 cm^3.

Table 7.5.2 gives a comparison of the two proposed correction schemes for three different initial gas rates at one liquid rate (70% of the maximum). Both correction schemes correlate well together, with an error of 10%.

The second scheme's simplicity, physical reality and consistency for all liquid rates made it the better choice of the two schemes.

Table 7.5.2

Comparison of Results from Two Separator/Sparger
Correction Schemes

r	$(N_A a)_T$	G_{in}	G_{out}	eq. (88) $(N_A a)_S$	#1 eq. (86) $(N_A a)_R$	#2 $(N_A a)_R$
s^{-1}	gmole/m^3s	cm^3/s			gmole/m^3s	
112.4	3.55	76	24.1	2.2	4.5	5.1
111.0	3.97	76	18.0	1.9	5.5	5.7
90.5	3.20	76	29.2	2.06	4.1	4.6
52.7	2.50	76	32.0	1.77	3.9	4.3
42.4	2.51	76	39.3	1.37	3.4	3.6
34.9	2.17	76	44.3	1.25	2.9	3.1
2.9	2.05	76	46.0	0.22	3.4	3.0
138	4.67	115	49.0	3.41	5.60	6.72
104	4.97	115	44.8	2.70	6.70	7.15
79.6	4.26	115	54.8	2.18	4.42	4.98
60.9	3.46	115	66.1	2.18	4.42	4.98
31.7	3.62	115	63.9	1.36	5.30	5.21
2.92	2.41	115	81.0	0.28	3.99	3.47
137.5	1.40	38	16.4	2.17	0.82	2.01
104.1	2.00	38	7.1	1.27	2.54	2.88
79.0	1.79	38	10.4	1.22	2.21	2.57
49.2	1.99	38	7.3	0.76	2.91	2.86
32.2	1.60	38	13.3	0.72	2.25	2.30
2.93	1.31	38	17.8	0.15	2.17	1.88

APPENDIX 7.6

Gas Depletion Correction

A gas depletion correction scheme is necessary to correct an experimental value for $N_A\bar{a}$, which is an average value for the whole column, to a true value of $N_A a_o$, which is a point value at the beginning of the reactor when the gas molar flowrate is F_{go}.

The differential mass balance on the gas (assuming plug flow) can be stated as:

$$- \frac{d F_g}{dV} - N_A a = 0 \qquad (90)$$

with the boundary condition:

$$F_g = F_{go} \text{ at } V_R = 0$$

Since, the point value of the interfacial area, a is always changing in the column an average area $\bar{a}$, must be defined and can be written as:

$$\bar{a} = \frac{1}{V_R} \int_0^{V_R} a \, dV \qquad (91)$$

The experimenter measures the value $N_A\bar{a}$ not $N_A a$, so from the overall mass balance on the gas one gets:

$$F_{go} - F_{ge} = N_A \bar{a} \, V_R \tag{92}$$

or

$$\frac{F_{ge}}{F_{go}} = 1 - \frac{N_A \bar{a} \, V_R}{F_{go}} \tag{93}$$

An assumption must be made concerning the change of a with respect to F_g. This assumption is justified "a posteriori" by our findings.

$$a = D \, F_g^n \tag{94}$$

and

$$a_o = D \, F_{go}^n \tag{95}$$

By replacing equation (94) into equation (90) and integrating one gets:

$$-\int_{F_{go}}^{F_{ge}} \frac{dF_g}{F_g^n} = N_A \cdot D \int_0^{V_R} dV \tag{96}$$

for $n \neq 1$

$$\left(1 - \frac{F_{ge}}{F_{go}}\right)^{(1-n)} = N_A a_o \, (1-n) \, \frac{V_R}{F_{go}} \tag{97}$$

From equation (93) and (97) one obtains:

$$N_A a_o = \frac{F_{go}}{V_R (1-n)} \left[1 - (1 - \frac{N_A \bar{a} V_R}{F_{go}})^{(1-n)} \right] \tag{98}$$

For known values of V_R and n and experimental values of $N_A \bar{a}$ and F_{go}, $N_A a_o$ can be found.

There are two problems with this solution. The first is that there are no reliable values for n. It is not an experimentally determinable exponent. It is expected to be around 0.7, as for packed and bubble columns, but it is not known for sure.

The second problem is with the sensitivity of the solution. When $\frac{N_A \bar{a} V_R}{F_{go}}$ approaches a value of one, any significant error in the value of $N_A \bar{a}$ or F_{go} will cause the calculated value of $N_A a_o$ to vary tremendously.

In order to handle the first problem, an assumption concerning the average area, $\bar{a}$ is made and later shown to be experimentally justified:

$$\bar{a} = D' \left(\frac{F_{go} + F_{ge}}{2} \right)^m \tag{99}$$

$$a_o = D' F_{go}^m \tag{100}$$

From the following equation

$$N_A = C_{A_i} \sqrt{D_A (s + r)} \tag{101}$$

N_A is a constant for all liquid and gas rates at a
particular catalyst concentration and hence:

$$N_A \bar{a} = N_A \ D' \left(\frac{F_{go} + F_{ge}}{2} \right)^m \qquad (102)$$

Replacing for F_{ge} in equation (102) from equation (93)
one gets:

$$N_A \bar{a} = N_A \ D' \ F_{go}^m \left(1 - \frac{N_A \bar{a} \ V_R}{2 \ F_{go}} \right)^m \qquad (103)$$

With the use of equation (100), equation (103) becomes:

$$N_A \bar{a} = N_A a_o \left(1 - \frac{N_A \bar{a} \ V_R}{2 \ F_{go}} \right)^{-m} \qquad (104)$$

Now m is an experimentally determined value. Figures
7.6.1 and 7.6.2 show plots for the determination of m for the
Kenics and Koch mixer. The Kenics mixer shows that m is
a function of r or catalyst concentration. This
relationship is shown in Figure 7.6.3.

Equation (104) is preferred to equation (98) for
computation of $N_A a_o$ because the exponent m can be
obtained more readily.

Nevertheless equation (98) is the more realistic
solution while equation (104) is only an approximation,
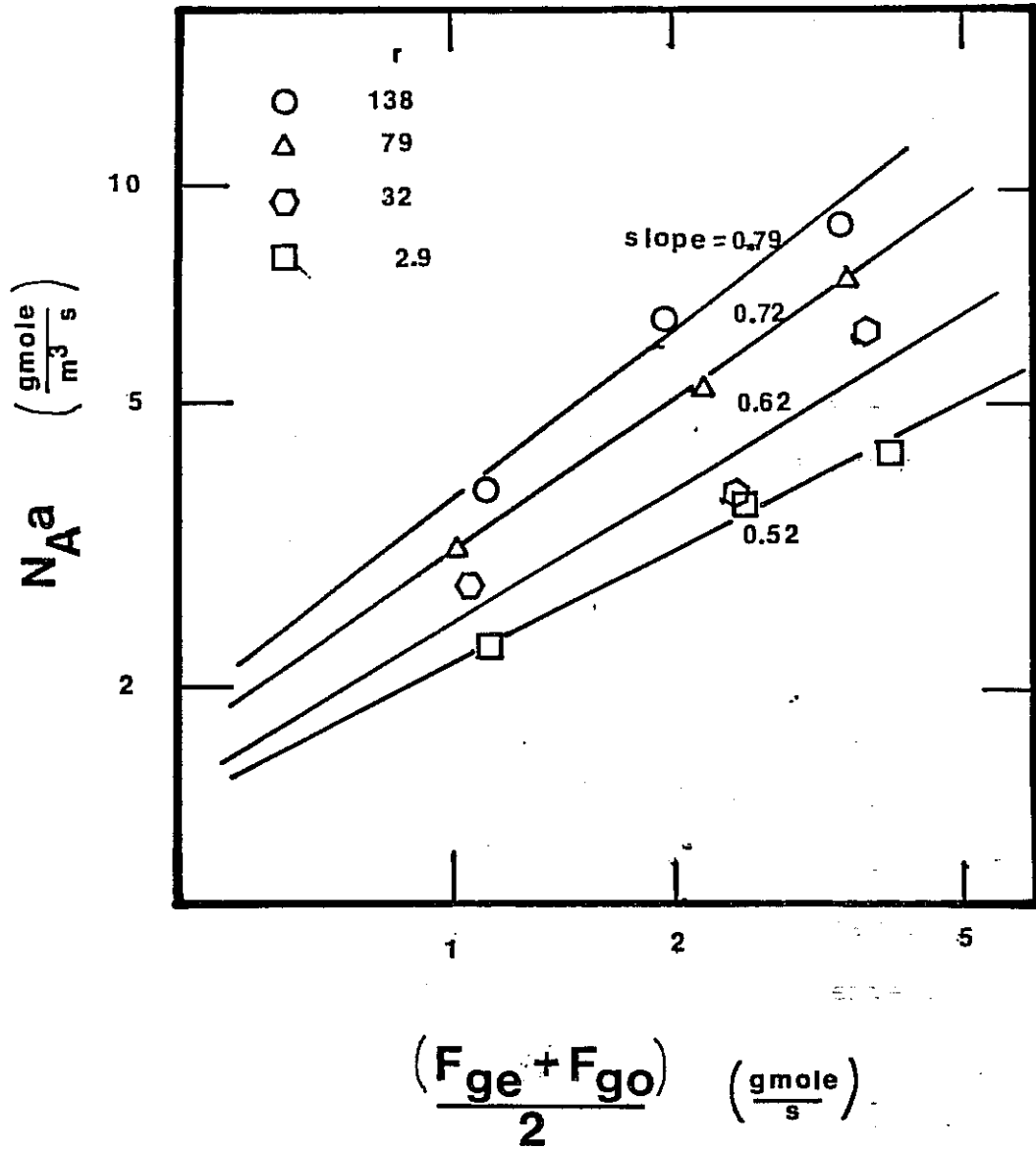
Figure 7.6.1   Relationship Between Rate of Absorption and
                Average Gas Flowrate in the Kenics Mixer
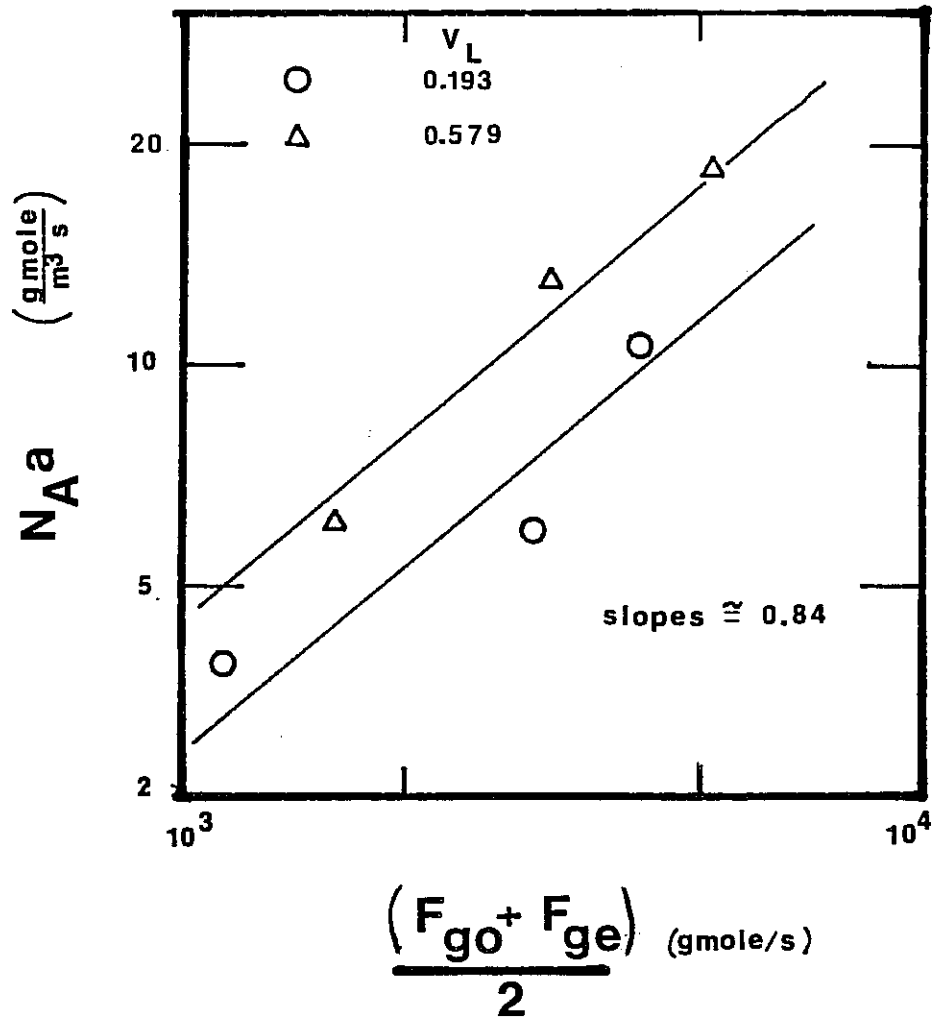
Figure 7.6.2   Relationship Between Rate of Absorption and Average Gas Flowrate in the Koch CY Mixer
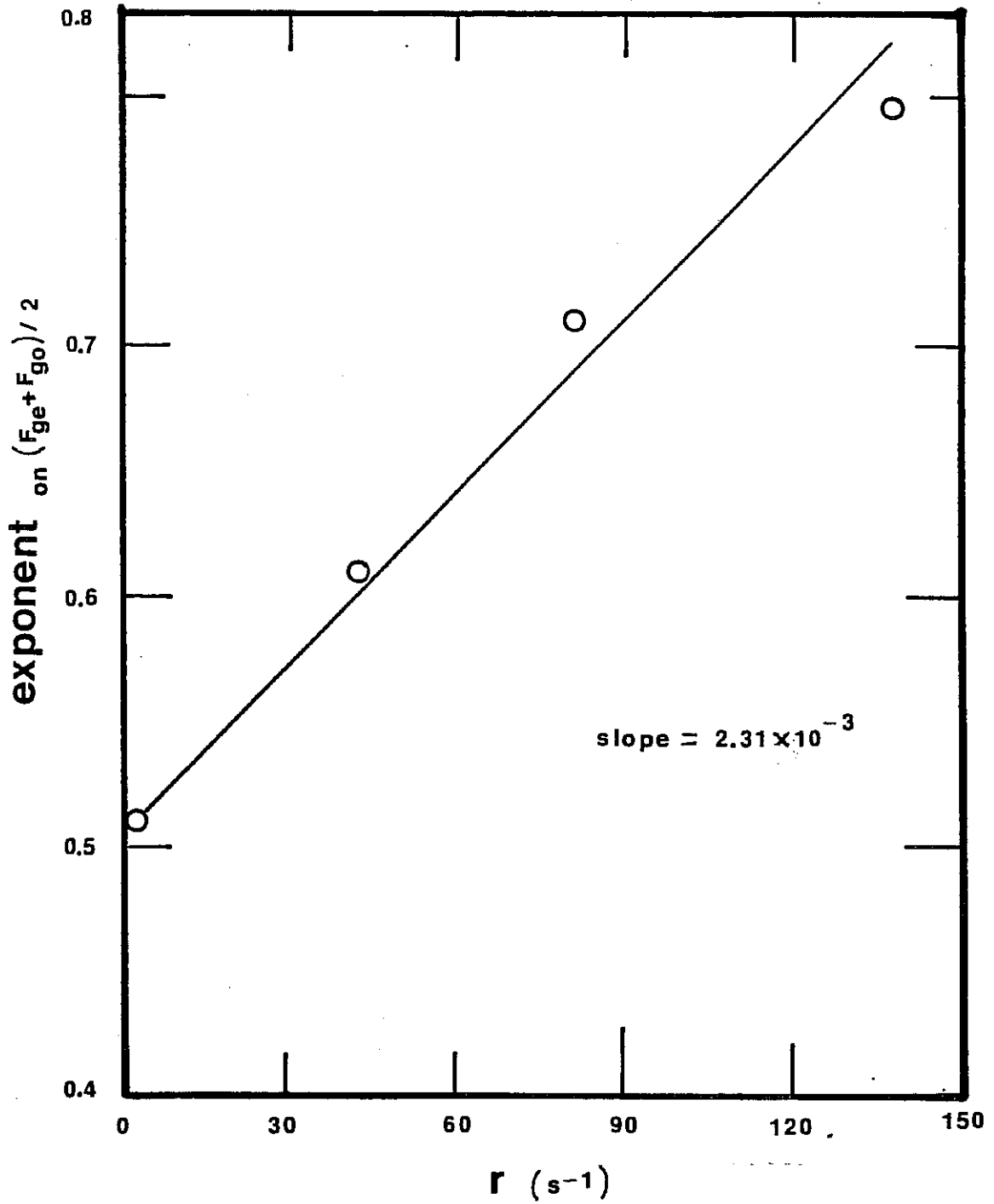
Figure 7.6.3   Varience of the Exponent on the Average Gas
                Flowrate as a Result of Changing Catalyst
                Concentrations

an approximation that does fail as $\dfrac{N_A \bar{a} \, V_R}{F_{go}}$ approaches unity. The smaller the gas uptake in the system, the better

the assumption. Since the Koch mixer caused such a

large reduction of gas at moderate catalyst concentrations,

the accuracy of the correction was questioned. However

zero catalyst concentration runs in the mixer only took

up 50% of the gas so the approximation could be used for

these points only.

After the evaluation was completed as shown in

Chapter 4, an approximate value for n was determined.

Table 7.6.1 shows the values for $(N_A a)^2$ evaluated from

equation (108) and equation (94). Equation (108)

consistently gave lower values, so that the final a values

given in the body of the report are on the conservative

side. Even so, the values are not that far apart and well

within the titration errors.

Table 7.6.1

Comparison of Results from Two Gas Depletion Correction Schemes

Run	r	original $(N_A a)_T$	eq. (98) $(N_A a)_R$	eq. (104) $(N_A a)_R$
#	s^{-1}	gmole/cm^{3}s x 10^6	gmole/cm^{3}s x 10^6	gmole/cm^{3}s x 10^6
1	139.8	1.89	3.53	3.60
1	112.4	2.61	5.92	5.43
2	111.0	3.26	9.08	7.55
1	90.5	2.12	4.21	3.85
2	69.2	1.87	3.48	3.14
1	52.7	1.30	2.10	1.92
2	42.4	1.30	2.10	1.90
1	34.9	0.97	1.44	1.32
2	2.9	0.87	1.26	1.12

APPENDIX 7.7

Error Analysis Supplement

This appendix provides the data and figure from which an error analysis was done and explained in Section 4.5.4.

The error data was taken from a set of runs done early in the study and had maximal titration errors. These points along with their associated titration error bars were plotted on Figure 7.7.1 and three lines were drawn through them. From the slopes and intercepts of these lines, the respective values and deviations in interfacial area, a and mass transfer coefficients, k_L were calculated and presented in Table 7.7.1.
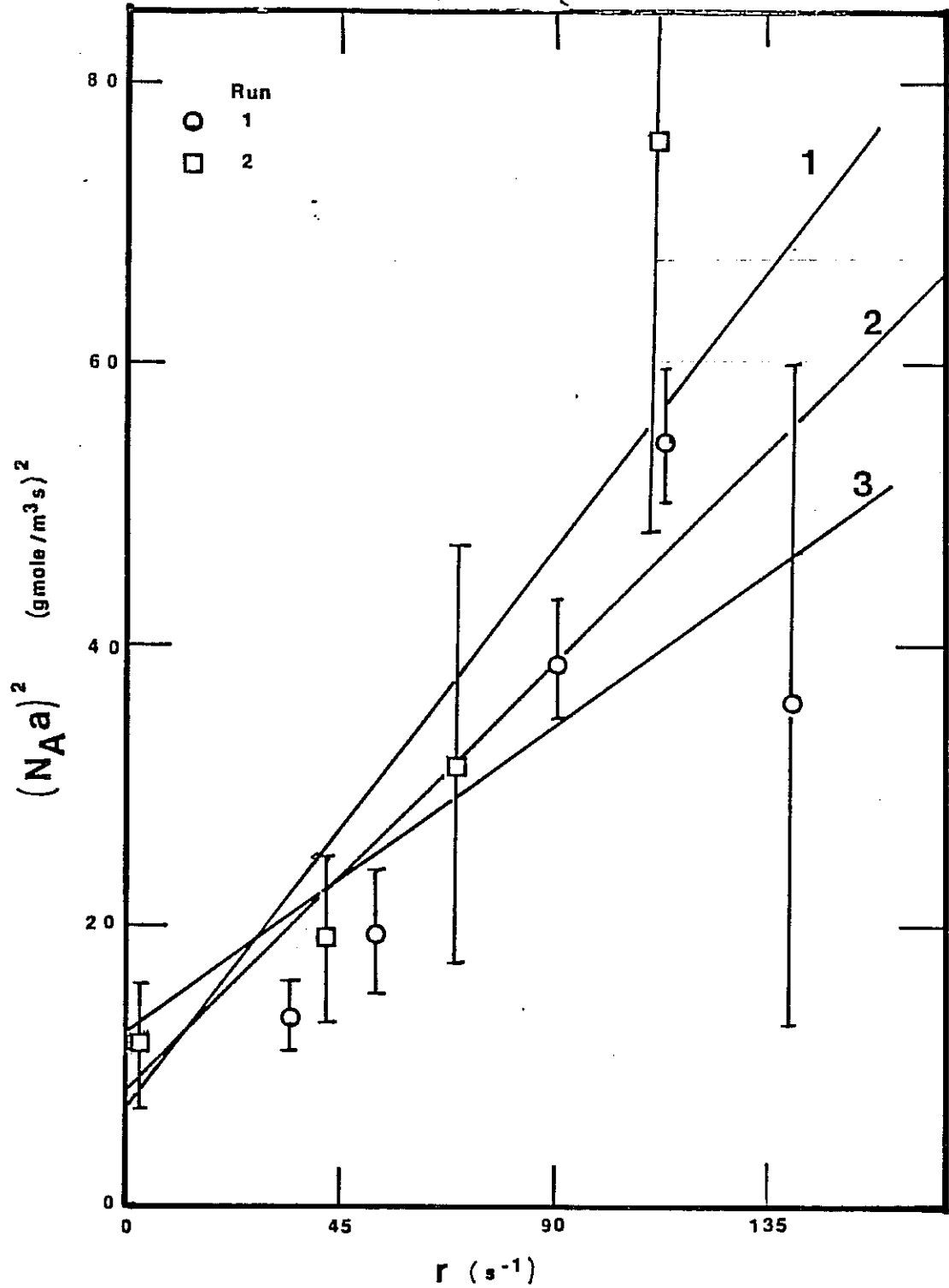
Figure 7.7.1   Figure Used for Evaluation of Errors in
the Values of the Mass Transfer Coefficients
and Interfacial Areas

Table 7.7.1

Maximal   Magnitudes   of Experimental Errors   and   Subsequent
Errors   in the Values of the Mass Transfer Coefficients   and
Interfacial Areas

r	$(N_A a) \times 10^6$	% titration error
s^{-1}	$gmole/cm^3 s$	
139.77	5.27	40
112.4	6.20	5
111.0	6.93	19
90.5	5.59	5
69.2	5.25	30
52.7	4.37	10
42.4	4.38	16
34.9	3.78	5
2.92	3.57	16

Line	Intercept	Slope	a	k_L
	$gmole^2/cm^6 s^2$	$gmole^2/cm^6 s$	m^{-1}	m/s
	$\times 10^6$	$\times 10^7$		$\times 10^4$
1	7.0	4.42	863	1.48
2	8.0	3.37	754	1.81
3	12.5	2.41	638	2.68
Average			751	1.99
% error			15%	31%

APPENDIX 7.8

Investigation of Appropriate Hatta

Number Magnitude

In Appendix 7.1, the second boundary condition for the problem of unsteady state absorption and first order reaction stated that the concentration of gas A in the bulk of the liquid B was equal to zero. In order to assure that this boundary condition represents reality well, the Hatta number, which relates the relative speed of reaction and diffusion through the film, must be large. Linek (15) and many others suggest that Ha > > 3.

This appendix addresses the question whether such large values of the Hatta number are necessary in order for the form of the enhancement factor derived in Appendix 7.1 to hold.

Consider the absorption and subsequent irreversible first order reaction of gas A into liquid B across a liquid film into a bulk that is modeled by a continuously stirred tank. From Film Theory, the mass balance on A in the film gives:

$$D_A \frac{d^2 C_A}{d y^2} - r C_A = 0 \tag{105a}$$

with boundary conditions:

$$y = 0 \qquad C_A = C_{A_i} \tag{105b}$$

$$y = \delta_L \qquad C_A = C_{A_b} \tag{105c}$$

A mass balance on A in the bulk is:

$$Q_L C_{A_b} + r \, C_{A_b} \, V_b + D_A \left. \frac{d \, C_A}{dy} \right|_{\delta_L} a \, V_b = 0 \tag{106}$$

The solution of equation (105)

$$C_A = C_{A_i} \exp\left(- \sqrt{\frac{r}{D_A}} \, y\right) +$$

$$\left[\frac{C_{A_b} - C_{A_i} \exp\left(-\sqrt{\frac{r}{D_A}} \, \delta_L\right)}{\exp\left(\sqrt{\frac{r}{D_A}} \, \delta_L\right) - \exp\left(-\sqrt{\frac{r}{D_A}} \, \delta_L\right)}\right] \quad X$$

$$\left[\exp\left(\sqrt{\frac{r}{D_A}} \, y\right) - \exp\left(-\sqrt{\frac{r}{D_A}} \, y\right)\right] \tag{107}$$

The derivative of C_A with respect to y at the film width, δ_L, can now be determined from the above equation and by using the definition of the Hatta number:

$$Ha = \frac{\sqrt{r \cdot D_A}}{k_L} \tag{17}$$

and film width:

$$\delta_L = \frac{D_A}{k_L} \tag{12}$$

can be written as follows:

$$\frac{d\,C_A}{dy}\bigg|_{\delta_L} = \sqrt{\frac{r}{D_A}}\,(C_{A_b} \cdot \coth Ha - C_{A_i} \cdot (1 + \coth Ha)) \tag{108}$$

The bulk concentration of gas A, C_{A_b}, can now be solved

by replacing equation (108) into equation (107) and is

given as follows:

$$C_{A_b} = \frac{C_{A_i} \cdot k_L a \cdot Ha \cdot (\tanh Ha + 1)}{(1/\tau + r) \cdot \tanh Ha + k_L aH_a} \tag{109}$$

where $\tau =$ CSTR residence time, (s).

The molar flux across the interface is:

$$N_A = -D_A \cdot \frac{d\,C_A}{dy}\bigg|_{y=0} \tag{110}$$

Again using equation (107) by taking the derivative with respect to y and then setting y equal to zero, the molar flux can be written as follows:

$$N_A = \frac{\sqrt{r \cdot D_A} \cdot C_{A_i}}{\tanh Ha} \left[1 - \frac{C_{A_b}}{C_{A_i}} \frac{1}{\cosh Ha} \right] \tag{111}$$

The true enhancement factor is then defined by:

$$E_{L_{true}} = \frac{N_A}{k_L \cdot C_{A_i}} = \frac{Ha}{\tanh Ha} \left[1 - \frac{C_{A_b}}{C_{A_i}} \cdot \frac{1}{\cosh Ha} \right] \tag{112}$$

The approximate enhancement factor for this situation assume $C_{A_b} = 0$ as described in Appendix 7.1 and is given by Film Theory as:

$$E_L = \frac{Ha}{\tanh Ha} \tag{113}$$

The error resulting from the use of the approximate enhancement factor can then be written from equation (112) and (113) as:

$$1 - \frac{E_{L_{true}}}{E_L} = \frac{C_{A_b}}{C_{A_i}} \cdot \frac{1}{\cosh Ha} \tag{114}$$

which with the aid of equation (109) becomes:

$$1 - \frac{E_{L_{true}}}{E_L} = \frac{\tanh Ha + 1}{(\alpha \cdot \tanh Ha + 1) \; \cosh Ha} \tag{115}$$

$$\text{where } \alpha = \frac{1/\tau + r}{k_L a \cdot Ha} \tag{115b}$$

The errors associated with using the approximate enhancement factor with Ha numbers less than 3 can be determined for the conditions of our experiments by using the following quantities:

$1/\tau = 0$ for semi-batch operation

$2.2 < r < 130 \qquad (s^{-1})$

$k_L = 1.84 \times 10^{-4} \, (m/s)$

$c_{A_i} \sqrt{D_A} = 7.7 \times 10^{-4} \left(\frac{mole}{m^2 \; s^{\frac{1}{2}}} \right)$

$D_A = 1.38 \times 10^{-9} \left(\frac{m^2}{s} \right)$

$300 < a < 1100 \; \left(m^{-1} \right)$

Table 7.8.1 presents some errors at select values of r and a. Most of the points on a Danckwerts' plot values

Table 7.8.1

Errors Associated with Low Hatta Numbers

r	Ha	a	α	$1 - \epsilon$
s^{-1}		m^{-1}		
130	2.3	700	440	0.0009
70	1.7	700	320	0.002
20	0.90	700	172	0.010
2.2	0.30	700	57	0.070
2.2	0.30	1100	36	0.110
20	0.90	1100	36	0.015
2.2	0.30	300	133	0.030

for Ha greater than 1. Therefore, from the results of this analysis, most of the points on any Danckwerts' plot used in this study have an error of 1% or less, even though the Ha numbers are less than 3.

The end points on any Dankwerts' plot in this study have a maximum error of 10% and are still within titration errors.

APPENDIX 7.9

Holdup Correlation

In Section 4.1, it was mentioned that the family of lines that resulted from a plot of the holdup ratios versus the superficial velocity ratios on log-log paper for the Koch and Kenics mixers could be correlated into one line. The type of correlation that was suggested was one similar to that proposed by Yung Hsu (24) for gas-lift reactors which was specifically presented as:

$$\frac{1-\epsilon}{\epsilon} = 10.14 \; \frac{V_L}{V_G}^{0.73} (Fr_{TP})^{0.26} (Re_{TP})^{0.0044} (We_{TP})^{0.42} \tag{116}$$

where (Fr_{TP}) = Froude No. = $M^2/d \, g \, \rho_H^2$; $\qquad$ (117)

$\qquad (We_{TP})$ = Weber No. = $d \, M^2/g \, \rho_H \, \sigma$; $\qquad$ (118)

$\qquad (Re_{TP})$ = Reynolds No. = $d \, M/\mu$ $\qquad$; $\qquad$ (119)

$\qquad$ M $\quad$ = Total mass flow = $V_G \rho_G + V_L \rho_L$; $\qquad$ (120)

and, $\qquad \rho_H$ = Homogeneous density = $\left(\frac{\epsilon}{\rho_G} + \frac{\epsilon}{\rho_L}\right)^{-1}$ $\qquad$ (121)

From equation (116), it is apparent that Reynolds number is not very important and that the Froude number and the Weber number contain nearly all the same parameters. Accordingly, without any data for systems with different surface tensions, and analysis containing both the Froude and Weber number would produce an infinite number of correlations. Therefore, the subsequent correlations contain only the Weber number, which is most often used for holdup correlations in the literature.

From this study's holdup data two correlations, one for the Kenics mixer and one for the Koch mixer, were developed and are written as follows:

Kenics $\quad\quad \dfrac{1 - \epsilon}{\epsilon} = 30.8 \ (V_L/V_G)(We_{TP})^{-0.23}$ $\quad\quad$ (122)

Koch $\quad\quad\quad \dfrac{1 - \epsilon}{\epsilon} = 24.6 \ (V_L/V_G)(We_{TP})^{-0.20}$ $\quad\quad$ (123)

Figures 7.9.1 and 7.9.2 compare the experimental and predicted values of the gas holdup for the Kenics and Koch mixers, respectively. Both figures show a very good correlation between the experimental and predicted values.
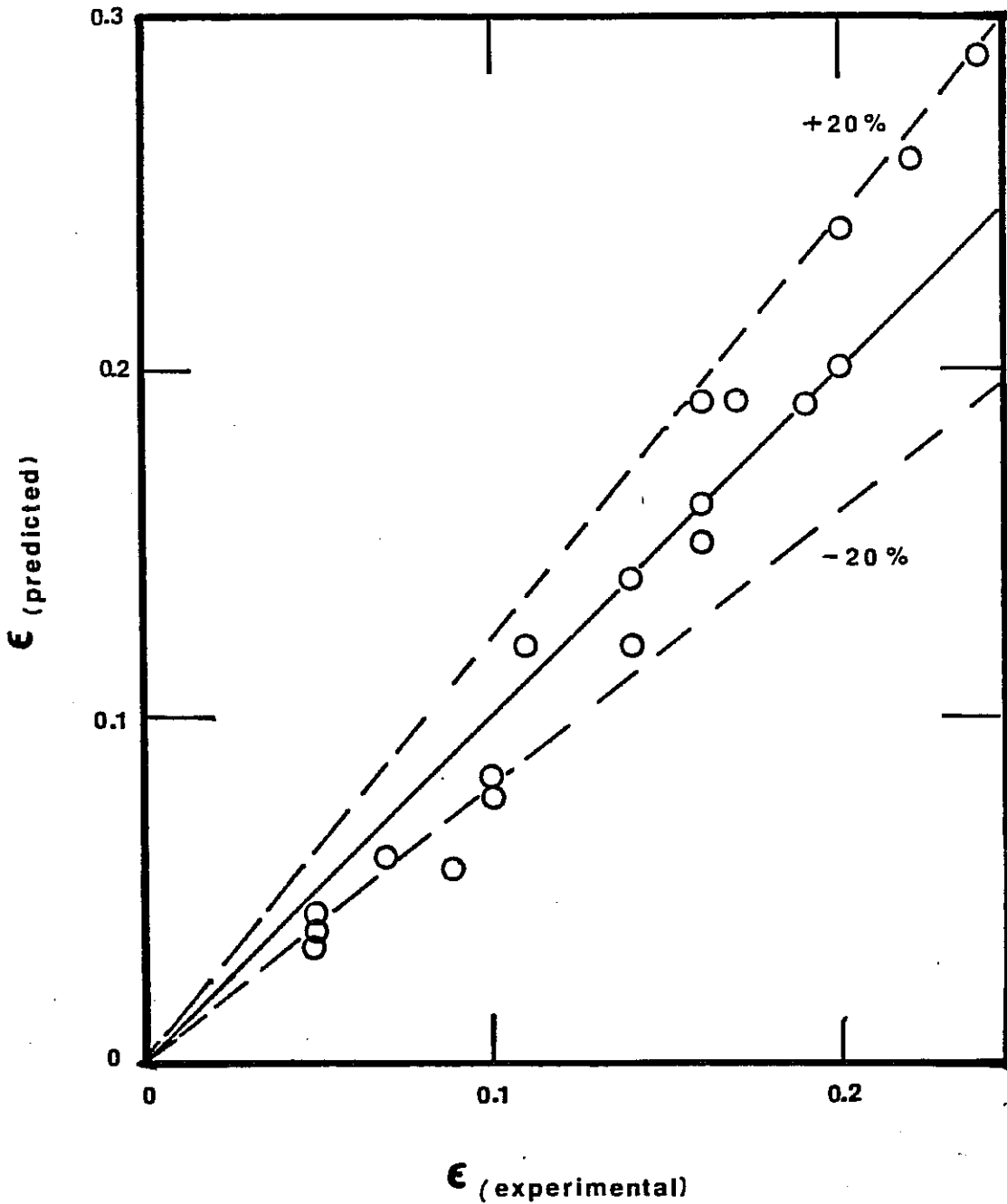
Figure 7.9.1   Experimental Gas Holdup Values Versus
Predicted Gas Holdup Values from Equation
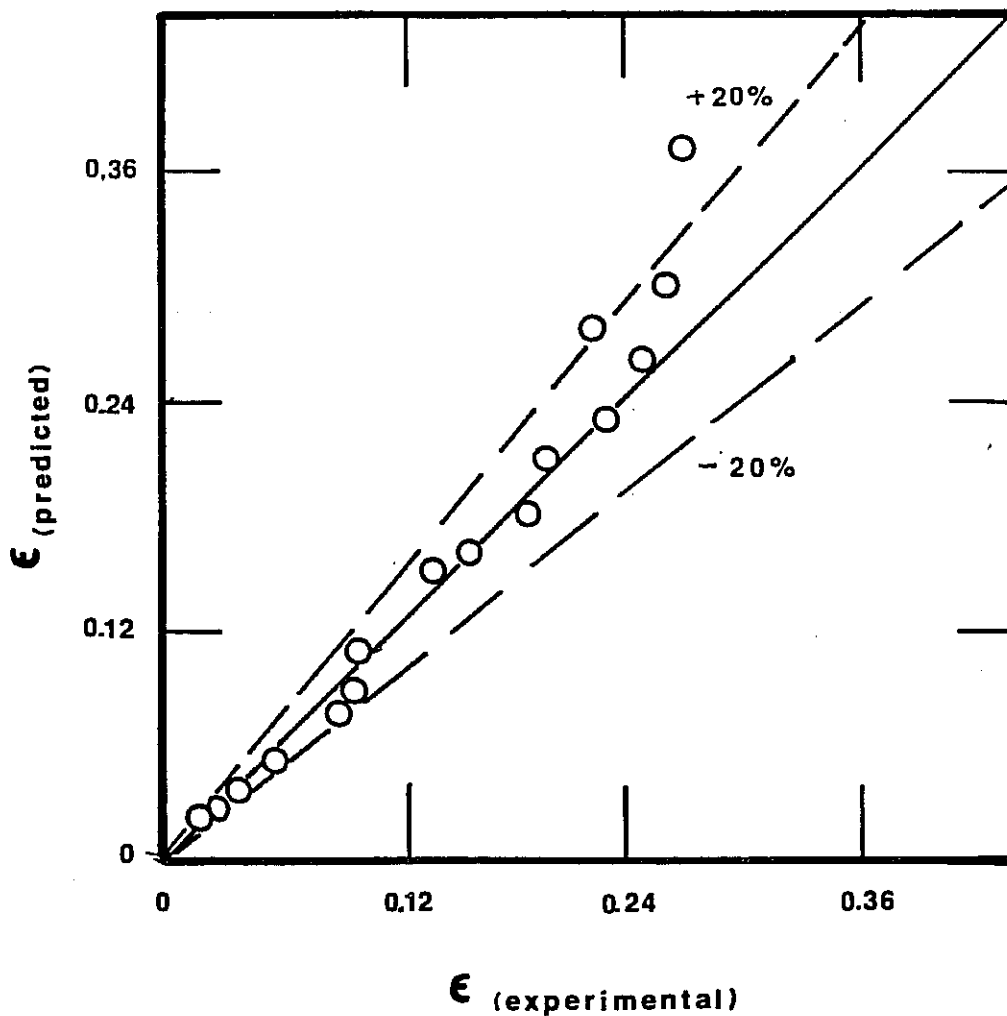(122) for the Kenics Mixer

Figure 7.9.2   Experimental Gas Holdup Values Versus
Predicted Gas Holdup Values from Equation
(123) for the Koch CY Mixer

APPENDIX 7.10

Nomenclature

English

a = Interfacial area between gas and liquid per unit liquid volume, m^2/m^3, cm^2/cm^3.

A = Total interfacial area between the gas and liquid in the reactor, m^2, cm^2.

C_A = Concentration of gas A, $gmole/m^3$, $gmole/cm^3$.

C_{A_b} = Concentration of gas A in the liquid bulk, $gmole/m^3$, $gmole/cm^3$.

C_{A_i} = Concentration of gas A at the gas/liquid interface, $gmole/m^3$, $gmole/cm^3$.

C_B = Concentration of B in liquid, $gmole/m^3$.

C_{B_b} = Concentration of reactive substance B in the liquid bulk, $gmole/m^3$, $gmole/cm^3$.

d = Inside pipe diameter, m.

d_B = Sauter mean drop size, m.

d_h = Mixing element hydraulic diameter, m, inch.

D_A = Diffusivity of the gas A in the liquid phase, m^2/s, cm^2/s.

D_B = Diffusivity of reactive species B in the liquid phase, m^2/s, cm^2/s.

E = Power dissipation per unit reactor voidage, W/m^3.

E_i = Instantaneous enhancement factor, Equation (18), dimensionless.

E_L = Overall enhancement factor, dimensionless.

f_H = Homogeneous friction factor for pipe flow, dimensionless.

F = Molar flowrate of gas, gmole/s.

F_{ge} = Molar flowrate of gas at outlet, gmole/s.

F_{go} = Molar flowrate of gas at inlet, gmole/s.

Fr_{TP} = Two phase Froude number, equation (117), dimensionless.

g = Acceleration of gravity, m/s .

G_{in} = Volumetric flowrate of gas at inlet, m^3/s, cm^3/s.

G_{out} = Volumetric flowrate of gas at outlet, m^3/s, cm^3/s.

Ha = Hatta number, equation (17), dimensionless.

k_L = Liquid side mass transfer coefficient, m/s.

k_G = Gas side mass transfer coefficient, m/s.

L = Length along a reactor, m.

L_1 = Length between the two liquid manometer water levels, m.

L_2 = Distance between the two pressure taps, m.

M = Total mass flow, equation (120), kg/ m^2s.

M_w = Molecular weight, g/gmole.

N_A = Rate of absorption of A per unit interfacial surface area, gmole/m^3s, gmole/cm^3s.

P_w = Energy dissipation per unit volume of liquid in reactor, W/m^3.

Q_L = Volumetric flowrate of liquid, m^3/s, cm^3/s.

r' = Pseudo-first order reaction rate constant, s^{-1}.

r = Second order reaction rate constant, m^3/gmole s.

R_A = Rate of appearance of gas A from a reaction, gmole/m^{3}s.

Re = Liquid Reynolds number, dimensionless.

Re_{TP}= Two phase Reynolds number, equation (119), dimensionless.

s = Surface renewal frequency, s^{-1}.

t = Time, s or minutes.

V_b = Total volume of semi-batch liquid, m^3.

V_G = Superficial gas velocity in the reactor, m/s, cm/s.

V_H = Superficial velocity of a two phase mixture, m/s.

V_L = Superficial liquid velocity in the reactor, m/s, cm/s.

V_R = Volume of reactor voidage, m^3 or cm^3 .

V_S = Volume of separator and sparger, m^3 or cm^3.

V_T = Volume of the total reactor apparatus, m^3 or cm^3.

We = Liquid Weber number, dimensionless.

We_{TP}= Two phase Weber number, equation (118), dimensionless.

y = Distance away from gas/liquid interface, m.

z = Stoichiometric coefficient on reactant B, gmoles.

Z = Volume of liquid per volume of interface in a reactor, equation (19), dimensionless.

Greek

α = Assigned parameter, Equation (15b), dimensionless.

δ = Film thickness, m.

Δ = Titration volume difference, (ml).

ΔP_K = Pressure drop associated with loss in kinetic energy due to friction, N/m^2.

ΔP_S = Pressure drop associated with a static head or height difference, N/m^2.

ΔP_T = Total pressure drop, N/m^2.

ΔV = Slip velocity between two phase, equation (32), m/s.

ϵ = Fractional gas holdup in two phase flow, dimensionless.

Θ = Exposure time of a fluid element at the interface, s.

μ_L = Liquid viscosity, kg/s m.

ρ_G = Gas density, kg/m^3.

ρ_H = Homogeneous density of a gas/liquid mixture, kg/m^3.

ρ_L = Liquid density, kg/m^3.

σ = Surface tension , N/m.

τ = Residence time in reactor, s.

Other

[ ] = Concentration of species inside brackets, gmole/m^3, gmole/l, gmole/cm^3.

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9. VITA

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