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Title: ABSORPTION OF CARBON DIOXIDE IN WATER USING A MULTIPLE STAGE CROSSCURRENT PACKED COLUMN

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Dr. Charles E. Wicks

A cascade crosscurrent column, packed with Pall rings, was employed for carbon dioxide absorption from a carbon dioxide-rich air phase by contacting with water. Absorption efficiency was presented as the percentage of CO_2 removed versus gas flow rates for constant liquid to gas ratios (L/G).

Curves were developed for an L/G molar flow ratio of 25 and 50. The curve for an L/G ratio of 25 exhibited a distinct mass transfer transition point which corresponded to the flow transition point, the point when cross flow first appears. The curve for L/G ratio of 50 did not exhibit the mass transfer transition point, however, the efficiency changes were small and the mass transfer transition point may well have been masked by experimental error even though a flow transition point was observed.

The mass transfer data was found comparable to carbon dioxide-

air-water data obtained by another investigator using a countercurrent column. This information coupled with the higher throughput and lower pressure drop attributed to the crosscurrent column make it an attractive alternative to conventional towers.

Absorption of Carbon Dioxide in Water Using a Multiple Stage Cross Current Packed Column

by

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I. INTRODUCTION

Mass transfer is a fundamental concept utilized in various industrial operations and research. The mass transfer operation in which a gas mixture is contacted with a liquid for the purpose of preferentially dissolving one or more of the gaseous components into the liquid is called gas absorption. Due to increased environmental concerns and energy costs much effort is focussed on improving gas absorption methods.

One of the most common pieces of absorption equipment is the packed column. The column, being filled with a packing material, promotes contact between the two phases, liquid and gas, therefore, increasing the mass transfer.

Two types of flow patterns are most commonly found in industry, countercurrent and cocurrent, with countercurrent being found most often. In countercurrent flow the liquid phase falls down through the column due to gravity while the gas phase rises through the column due to an imposed pressure gradient. This is the most efficient arrangement, requiring less packed height for a given mass transfer change and flow rates. However, the gas throughout is limited. In countercurrent flow there is a limit on upward gas velocity, above which the drag forces imposed on the liquid exceed gravity forces. When this occurs an unstable, undesirable condition known as flooding, is obtained. This

throughput limit can be overcome by using cocurrent flow. With cocurrent flow the liquid and gas both enter the top of the packed tower and flow downward due to the combination of gravity and imposed pressure gradient. There is no flooding present in cocurrent flow. However, the disadvantage of this type of flow arrangement is a much reduced concentration driving force and therefore, reduced efficiency. This means, that for the same mass transfer one would need a much larger cocurrent packed column than one operated countercurrently.

The alternative to countercurrent and cocurrent flow is the crosscurrent packed column. This flow pattern is essentially countercurrent in that the liquid enters at the top of the column, the gas entering at the bottom of the column. However, in a crosscurrent column, baffles are placed to divert the gas and liquid flows from side to side so that the two phases cross at right angles to each other. The concentration driving force profile of crosscurrent flow has been found to fall between that of countercurrent and crosscurrent towers. However, at high gas rates liquid accumulates on the baffles where the gas bubbles go through. This bubbling action coupled with the vigorous mixing in the packing add to the column efficiency. The cross flow columns enjoy higher throughput capacity than countercurrent, the low pressure drop of cocurrent and the high mass transfer efficiency of countercurrent. For these reasons, the crosscurrent column can be an attractive alternative for gas-liquid contacting.

The objective of this study is to add to the information presented in the literature survey by investigating the mass transfer of carbon dioxide (CO_2) in air to water, carbon dioxide being only slightly

soluble in pure water.

II. LITERATURE SURVEY

An extensive literature survey was given by Ghawamedin Bayan (1) covering the early development and up-to-date research concerned with the crosscurrent packed column.

Bayan collected extensive mass transfer data for the ammonia-air-water system in a cascade crosscurrent column. It was found that the system exhibited a distinct transition point in the mass transfer data illustrated by a sharp rise in the mass transfer efficiency versus gas rate curve. This transition point corresponded to the point where the gas flow rate was high enough, for a given liquid to gas ratio, for the column to begin to exhibit cross flow. Number and spacing of baffles as well as size of the packed core were varied, trends noted, and models proposed. The theoretical model proposed was successful in predicting column behavior.

The gas absorption of CO_2 in air by water in a crosscurrent column has received little attention. This system was studied utilizing a countercurrent column by Koch, Stutzman, Blum, and Hutchings (2). Their goal was to obtain mass transfer coefficients, however, data was presented from which mass transfer efficiencies could be calculated. With an inlet gas CO_2 concentration in the area of 13%, efficiencies calculated varied between 4% to 11% depending on flow rates.

The desorption of oxygen in a single stage crosscurrent column was studied by Pittaway and Thibodeaux (3). Oxygen, like CO_2, is relatively insoluble in water. Liquid phase mass transfer coefficients were measured and a correlating equation developed.

III. EXPERIMENTAL EQUIPMENT AND PROCEDURE

The main pieces of equipment employed in this work were the cascade crosscurrent packed column and the gas chromatograph. Detailed information concerning construction and makeup of the column and related equipment, as seen in Figure 1, can be obtained from Bayan (1) and Zuehlsdorff (4).

A. The Crosscurrent Packed Column

The crosscurrent packed column, as illustrated in Figure 2, is essentially a rectangular box constructed of plexiglass and metal. A central packed core was positioned in the column, held by screens from the sides and top and bottom. Baffles were positioned at regular intervals on opposite sides of the central packed section and attached to the side walls. These baffles were used to divert the gas phase into the packing affecting a crisscross gas-liquid flow pattern.

The column was designed to have the ability to change its internal arrangements. The number and placement of baffles as well as the size of the packed section were variables. In this experiment, six baffles were placed on alternating side walls at a vertical distance between baffles of 254 mm. The packing section configuration, to use Bayan's (1) terminology, was "expanded" with a packed cross sectional area of 127 mm by 152 mm.

The gas phase was air enriched with CO_2, the liquid phase was city water. The carbon dioxide was supplied as a liquid in a 110 kg capacity

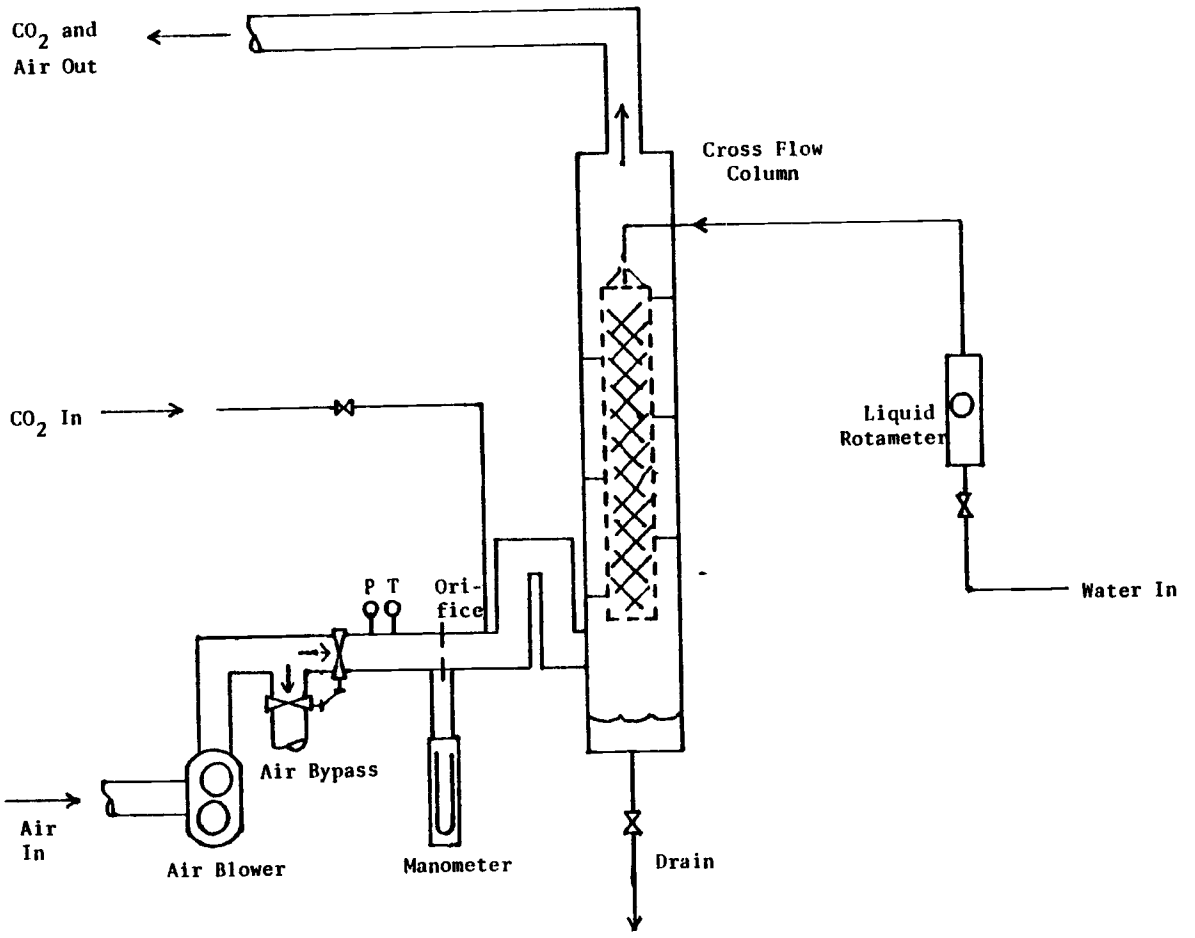

Figure 1. Schematic of Equipment Arrangement

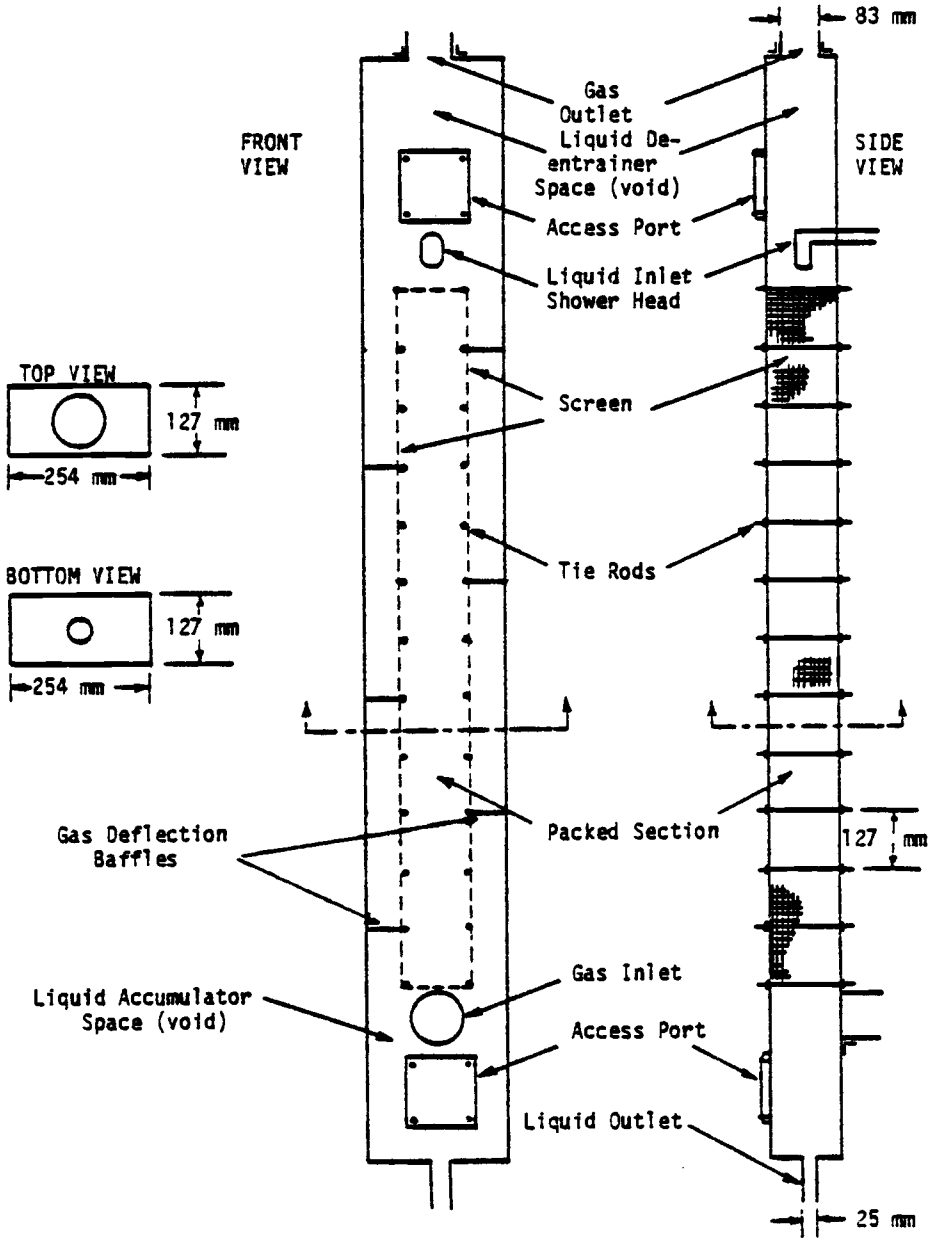

Figure 2. Multiple stage crosscurrent packed column.

cylinder as welding grade CO_2 and delivered through a regulator as gas. Flow rate was roughly set by the regulator and fine controlled by a needle valve. The CO_2 entered the air stream through a plastic diffuser extending the width of the air pipe and containing numerous holes on the upstream side.

This experiment did require a major modification of the existing column. In order to insure good mixing of CO_2 and air, the section of pipe immediately prior to the column was lengthened and baffles installed. The baffles were made of 6.35 mm plexiglass and placed in the airstream with silicone seal in the configuration shown in Figure 3. A sample port with septum and thermometer was placed after the mixing section and prior to the column. The CO_2 diffuser was also part of this modification.

Test Conditions

All of the tests for this study have been conducted within the following range of conditions.

Molar Liquid to Gas (L/G) Ratio = 25 or 50

Gas Flow Rate = 44 - 152 kg mole/$m^2 \cdot$ hr

Liquid Flow Rate - 1,171 - 5,060 kg mole/$m^2 \cdot$ hr

Inlet Molar CO_2 Concentration = 3.5% to 5.0% CO_2

B. Measurement of CO_2 Concentration with the Gas Chromatograph

The concentration of CO_2 was determined using a Basic Gas Chromatograph from Carle Instruments, Inc. The column was packed with Porapak Q, the carrier gas was helium, the operating temperature varied

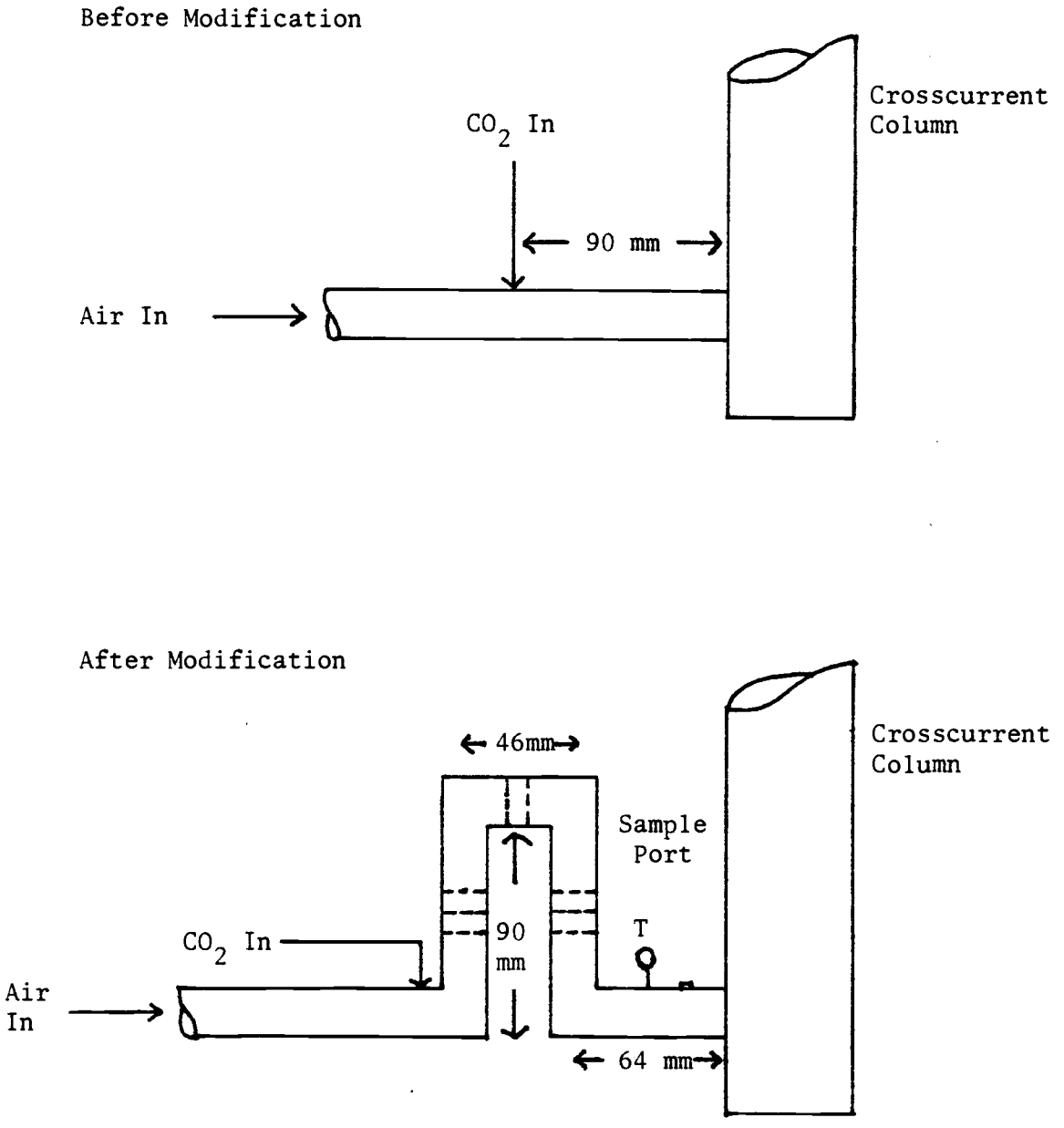

Figure 3. Schematic of Mixing Section Modification

slightly around 61°C. Inlet and outlet gas samples were taken with a Hamilton 250 µl syringe. The inlet sample was obtained at the sample port immediately prior to the column. The outlet sample was taken at the end of the exit air stream pipe.

The outlet sample volume was arbitrarily set at 200 µl. The inlet sample volume varied according to the sample point temperatures as follows:

$$\text{inlet sample volume } (\mu l) = 200\mu l \frac{T_{\text{inlet } (°K)}}{T_{\text{outlet}(°K)}} \qquad (1)$$

The chromatograph response was recorded on a Houston Instrument Omniscribe Recorder. The area under the carbon dioxide peak was obtained manually by assuming the peak to be a triangle. Carbon dioxide gas standards were used to compute percent CO_2 from peak areas.

C. Working Equations to Calculate Flow Rates

A theoretical development of the equations necessary to calculate the flow rates were presented by Bayan (1) and Zuehlsdorff (4). A summary is presented here.

1. Entering Air Flow Rate

$$\dot{m}_{air} = K_1 \left[1.0 - K_2 \frac{\Delta P}{P_1}\right] \sqrt{\frac{P_1 \, \Delta P}{\overline{M}, \, T_1}} \qquad (2)$$

where $\dot{m}$ = molar flow rate, g moles/sec

P_1 = upstream pressure, Pa

ΔP = orifice pressure drop, Pa

T_1 = orifice upstream temperature, °K

$\overline{M} = \frac{28.97 + 18.01\ (1.609)Z}{1.0 + 1.609Z}$, average molecular weight of gas stream, kg/kg mole

Z = humidity, kg H_2O/kg dry air

Table 1
The Constants K_1 and K_2 Used in Calculating $\dot{m}$ air

Diameter mm	K_1	K_2	Operating Range moles/sec
16.0	1.779×10^{-3}	0.29321	0.159-0.393
25.4	4.50×10^{-3}	0.29510	0.393-1.172
50.8	20.720×10^{-3}	0.32871	0.975-4.891

2. Exit Air Flow Rate

Since the amount of water stripped from the air is negligible, a good approximation is:

$$\dot{m}_{air,\ in} \cong \dot{m}_{air,\ out} \tag{3}$$

where $\dot{m}$ = gmoles/sec

3. Entering Water Flow Rate

$$\dot{m}_{H_2O,\ in} = F\ (\%\ \text{of scale}) \tag{4}$$

where F = 0.1574 for the small rotameter
F = 1.091 for the large rotameter
$\dot{m}$ = gmoles/sec

4. Exit Water Flow Rate

$$\dot{m}_{H_2O,\ out} \cong \dot{m}_{H_2O,\ in} \tag{5}$$

where $\dot{m}$ = gmoles/sec

D. Operating Procedures

Preliminary operating procedure including baffle placement and packing location have been previously explained. Carbon dioxide standards were prepared.

1. Start Up Procedure
   a) open liquid out valve
   b) open manometer valves 1, 4, and 5
   c) place appropriate orifice plate in the system
   d) turn on air blower, allow air inlet temperature to stabilize
   e) turn on gas chromatograph, allow to stabilize
   f) set approximate desired water flow rate
   g) set approximate desired air flow rate

At this point the exact air flow rate was calculated. Knowing the desired liquid to gas flow ratio (L/G), one could then set the liquid flow rate. Ten minutes was allowed for flow patterns and temperatures to stabilize. The CO_2 flow was then adjusted, samples taken and run on the gas chromatograph, until the inlet CO_2 concentration was approximately 4-4.5% CO_2. Inlet sample volume was determined using equation (1). Five to ten minutes were allowed for the column to reach equilibrium before actual CO_2 concentration measurements were made. Flow rates were checked during the run to make sure no major fluctuations had occured. If needed, adjustments were made.

For each run the following information was recorded.

P_{ATM} = atmospheric pressure
P_1 = orifice upstream pressure
ΔP = pressure drop across orifice
T_1 = orifice upstream air temperature
Tw, Td = wet and dry bulb temperatures of inlet gas stream
% of scale for water rotameter
Temperature at inlet gas sample port
Temperature at gas exit

During each run, repetitive samples were taken of the inlet and outlet gas as quickly as possible. The normal sampling order was two inlet samples followed by two outlet samples repeated three times. Sampling was extended if needed to obtain reproducible data.

2. Shut Down Procedures

a) close the CO_2 tank valve

b) turn off gas chromatograph

c) turn off main water supply

d) turn off air blower

d) close manometer valves

IV. RESULTS AND DISCUSSION

A. Modification of Column

The first major problem encountered in this investigation was the inability to obtain a material balance around the column. The inlet sample point was at the mouth of the inlet gas stream as it flowed into the column. The outlet sample point was at the mouth of the exiting gas stream pipe where it reentered the atmosphere. Different volumes of gas were taken at the inlet and exit sample points to compensate for temperature differences by using the ideal gas equation. With the above system the exiting CO_2 concentration was 7% higher than the incoming concentration when only gas streams entered and left the column.

Preliminary runs indicated that poor mixing of CO_2 and air prior to the column were causing inaccurate inlet concentration readings. This was found to be true when inlet samples were taken at various points within the inlet gas stream which showed up to a 10% variation in CO_2 concentration.

In order to promote better mixing the gas stream pipe just prior to the column was replaced by a longer U-shaped pipe as previously described (see Figure 3). A total of eight baffles were inserted into this mixing section. The baffles plus the longer pipe, which lengthened the available mixing time and provided turbulence due to the three additional 90° elbows used in constructing the U-shape, did promote good mixing. Inlet samples taken at different points within

the gas stream showed 0.9% difference in CO_2 concentration with no consistant pattern.

Operating the column with only gas streams entering and leaving, the inlet and outlet sample CO_2 concentrations now matched within 0.5% with no consistant pattern. To illustrate temperature effects, the same volume for inlet and outlet samples was used. Measurements showed 2% more CO_2 exiting than entering. This shows the importance of the mixing section as well as use of temperature compensation in determining sample volumes. The discovery of the above lent additional confidence to data obtained under these modified procedures.

B. Carbon Dioxide Removal Efficiency

Previous work studying the absorption of CO_2 by water used various liquid to gas ratios (L/G), ranging from about twenty to several hundred. An initial L/G of 50 was chosen with the idea of decreasing or increasing it depending on the results obtained.

The first step was to locate the flow transition point, the point where crisscross flow first develops, in order to define the area of gas flow rates that needed to be studied. The CO_2 removal efficiencies versus gas rate for an L/G of 50 are shown in Figure 4. The lack of any trend towards increasing efficiencies with increasing flow rates indicated that flow rates would have to be decreased to decrease initial efficiencies in order to see any changes in efficiency versus flowrate. This was accomplished with an L/G of 25. A plot of CO_2 removal efficiencies versus gas rate for an L/G of 25 is shown in Figure 5.

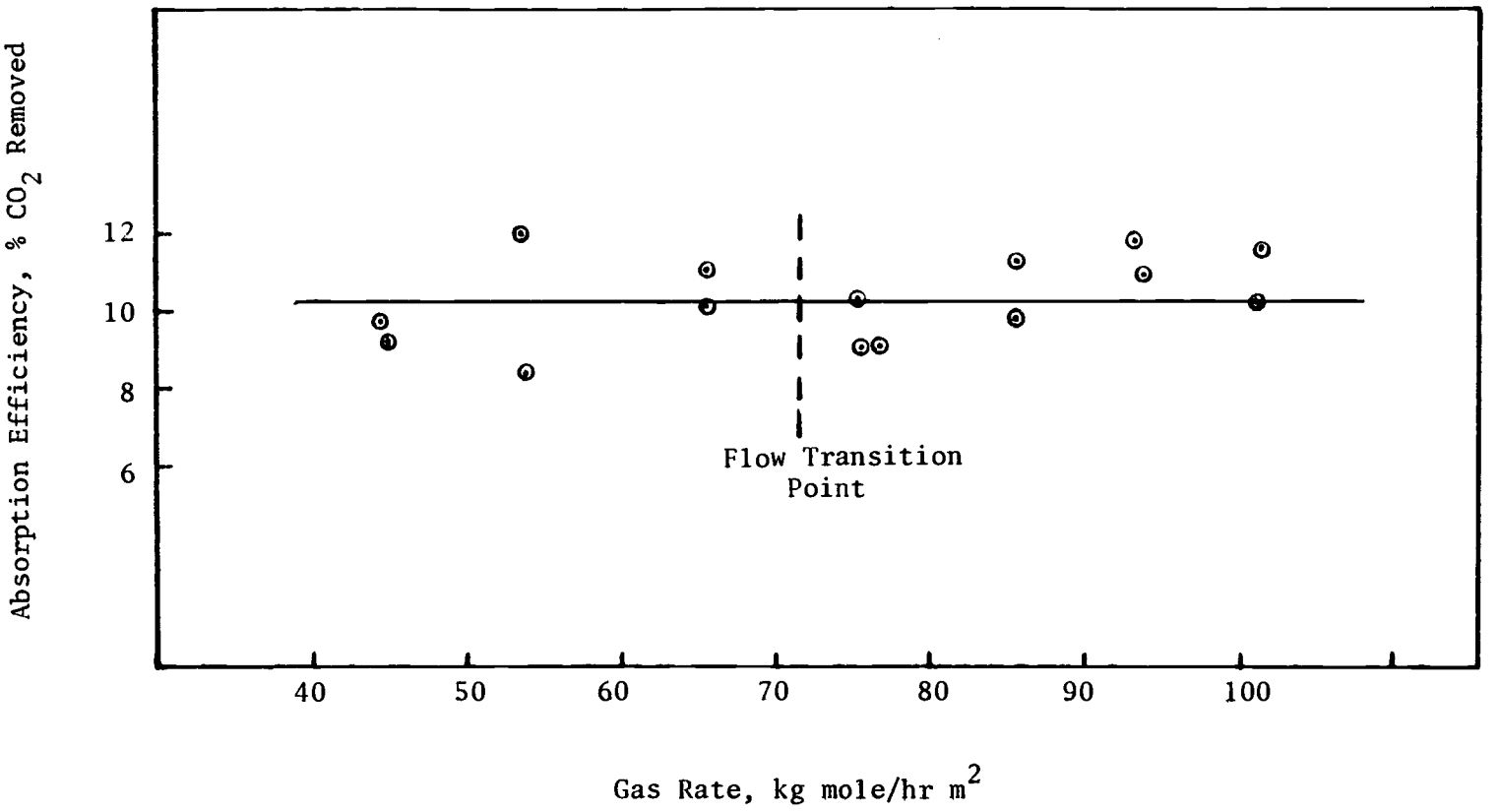

Figure 4. Absorption Efficiency versus Gas Rate for an L/G Ratio of 50

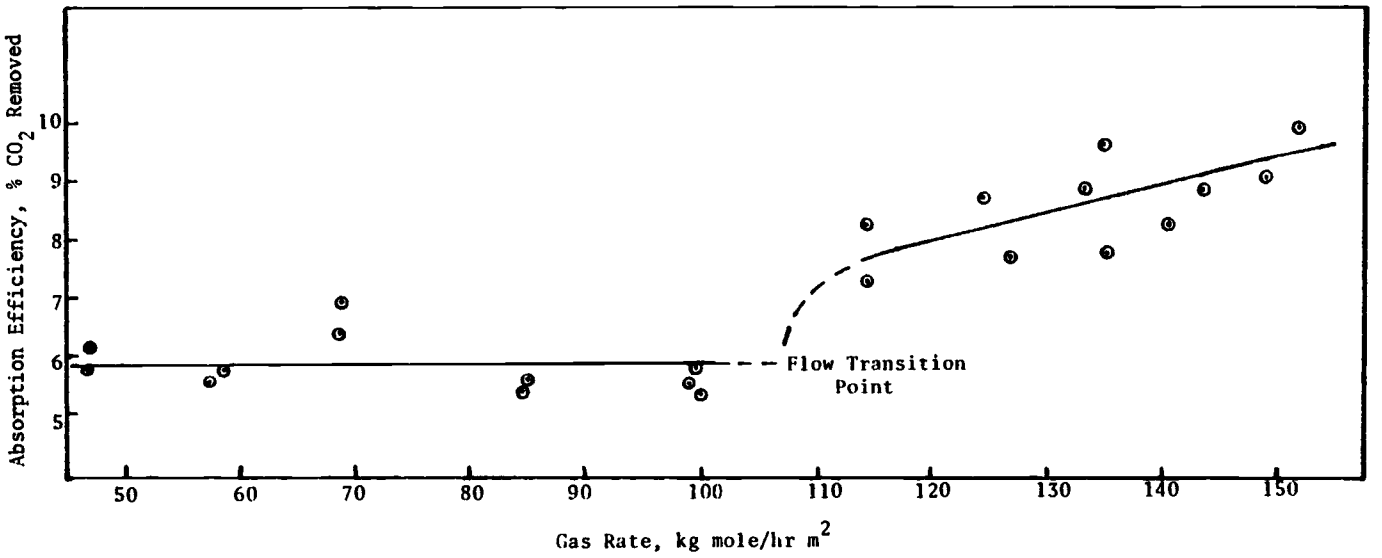

Figure 5. Absorption Efficiency versus Gas Rate for an L/G Ratio of 25

For an L/G of 50, the efficiency of CO_2 removal essentially did not change with changing gas rate. Efficiencies were randomly scattered between 8.4% and 11.8% with an average of 10.2%. The increased contacting between the gas and water phases experienced after the flow transition point, where cross flow first appeared, did not seem to be a factor in the absorption of CO_2 by water.

For an L/G of 25, the CO_2 removal efficiencies remained essentially constant at an average of 5.9% until the flow transition point was reached. After the transition point, the efficiencies increased markedly to about 8% and continued to rise with increasing gas rate until a leveling off at about 10% efficiency.

Previous work (2) which studied the CO_2 absorption in water from air for a countercurrent packed column presented data from which efficiencies could be calculated. Efficiencies varied from 4.1% to 11.4% depending of flow rates. This observation coupled with our data seems to show a practical maximum of water absorption of CO_2 from air in a packed bed in the area of 10-11%.

For an L/G of 50 the initial gas and water flow rates used resulted in sufficient contacting between the phases to achieve this maximum efficiency level. More than doubling the gas rate (and thereafter the liquid rate to maintain an L/G of 50) had no effect on CO_2 removal efficiencies. At the same initial gas rates for an L/G of 25, the lowered water rate resulted in less contacting and lowered efficiencies prior to the flow transition point. However, increased phase contact after the transition point showed a sharp rise in efficiencies which proceeded to level off around 10%. This corresponds

well with the 10.2% average efficiency obtained for an L/G of 50.

The basic shape of the efficiency curve for L/G ratio of 25 was proposed by Bayan (1). His data for an ammonia-air-water system in a crosscurrent column yielded relatively flat curves followed by a steeper curve at the flow transition point. It is supposed that for an L/G of less than 25 for the carbon dioxide-air-water system, the curves obtained would also follow the trend proposed by Bayan. It is also possible that the curve for L/G of 50 follows this trend but that the curves are so flat that the transition point is completely hidden by our experimental error.

VI. RECOMMENDATIONS FOR FUTURE WORK

This study began the investigation of the performance of cross flow columns in the absorption of relatively insoluble gases. Continuing the study of the carbon dioxide-air-water system would not be profitable without making several changes as listed below:

1) use of gas chromatograph system with automatic integration of peak areas,
2) expansion of the column water drain so that higher L/G ratio studies can be made,
3) at L/G ratios lower than 25 the gas rate required to reach the flow transition point is not attainable with the present air supply system.

The study of the oxygen-air-water system is also difficult because available city water is almost saturated with oxygen. It is recommended that another relatively insoluble gas be selected for future studies, the study being performed such that data can be compared with the work done by Bayan (1).

V. CONCLUSIONS

Mass transfer data for the carbon dioxide-air-water system were obtained for flow rates both before and after the flow transition point, the point where cross flow first occurs within the tower. For an L/G ratio of 25 the system exhibited a distinct mass transfer transition point which corresponded with the flow transition point. This curve supports the work performed by Bayan (1). For an L/G ratio of 50 the mass transfer transition point was not apparant but might have been masked by the experimental error present.

The mass transfer data obtained with the cascade crosscurrent column was comparable to countercurrent data (2) for the carbon dioxide-air-water system. Therefore, the higher throughput and lower pressure drop experienced by the cross flow packed column make it an attractive alternative to industry.

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APPENDICES

SAMPLE CALCULATIONS

The following data and calculations are from Run #71.

Run #71 Date:

Orifice Diameter - 2.0 inches L/G = 25

P_{ATM} = 29.87 in. Hg.

$$P_1 = [P_{ATM} + \frac{(25.08)-(11.61)}{13.6}]3386.4 \frac{P_a}{\text{in Hg}} = 104{,}506\ P_a$$

$$\Delta P = [(25.00)-(23.5)]249./\frac{P_a}{\text{in.}H_2O} = 373.65\ P_a$$

T_{AIR} = 41°C = 314°K

Air In Td = 95°F Tw = 72.5°F Z = 0.012

$$M_1 = \frac{28.97 + 28.98Z}{1.0 + 1.609Z} = 28.76 \ \frac{kg}{kg\ mole}$$

For orifice diameter of 2.0 inches: $K_1 = 20.721 \times 10^{-3}$

$K_2 = 0.32871$

$$\dot{m}_{air\ in} = 20.721 \times 10^{-3} [1.0 - (0.32871)\frac{373.65}{104{,}506}]\sqrt{\frac{(104{,}506)(373.65)}{(28.76)(314)}}$$

$$= 1.361 \ \frac{moles}{sec}$$

$$\dot{m}_{water\ in} = (L/G)(G) = (25)(1.361) = 34.02 \ \frac{moles}{sec}$$

Rotameter Setting = $\frac{34.02}{1.091}$ = 31.2% of large rotameter

Inlet Sample Size = 200 µl $\frac{(273 + 36.7)°K}{(273 + 16.4)°K}$ = 214 µl

At this point in a run, inlet and outlet samples are taken and analyzed by the gas chromatograph. The following method is used to determine the CO_2 peak area. All peak dimensions are relative to the recorder chartpaper. Peak areas are reported as "squares."

1. Determine the widths of the peaks at the baseline. Assume that peaks of the same general height have the same baseline width. Average the widths for all CO_2 inlet peaks and record. Average the widths of all the CO_2 outlet peaks and record.

   inlet CO_2 peak width = 3.03
   outlet CO_2 peak width = 2.93

2. Determine peak heights.

inlet samples	outlet samples
68.9	64.9
69.3	65.6
71.3	65.1
70.9	65.9
71.6	66.1
71.1	66.5
Average Heights 70.5	65.7

3. Assume peak areas are represented by a triangle and that Area = $\frac{1}{2}$(base)(height).

   Average Inlet Peak Area = $\frac{1}{2}(3.03)(70.5) = 106.8$ squares

   Average Outlet Peak Area = $\frac{1}{2}(2.93)(65.7) = 96.2$ squares

   $$\text{Efficiency} = \left(\frac{\text{Inlet-Outlet}}{\text{Inlet}}\right)100\% = \left(\frac{106.8-96.2}{106.8}\right)100\% = \underline{9.93\%}$$

4. Determine inlet and outlet concentrations.

The Standard Factor (SF) was determined as follows:

$$SF = \frac{\text{concentration of standard}}{\text{standard peak area (squares)}} = \frac{4.0\%}{101.0 \text{ squares}} = 0.0396\%/\text{square}$$

Inlet Concentration = (106.8 squares)(0.0396%/squares) = 4.23%

Outlet Concentration = (96.2 squares)(0.0396%/squares) = 3.81%

Table 2. Inlet Sample Size Data

Run Number	Temperature Inlet Sample (°F)	Temperature Outlet Sample (°F)	Size Outlet Sample (µl)	Size Inlet Sample (µl)
31	88	69	200	207
32	88	69	200	207
33	86	66	200	208
34	88	67	200	208
35	91	65	200	210
36	93	66	200	210
37	94	66	200	211
38	95	66	200	210
39	96	65	200	212
40	97	66	200	212
41	96	66	200	211
42	98	66	200	212
43	100	65	200	213
44	98	66	200	212
45	99	66	200	212
46	88	67	200	208
47	91	67	200	209
48	93	67	200	210
49	89	68	200	208
50	94	68	200	210
51	95	67	200	211
52	94	68	200	210
53	99	67	200	212
61	106	66	200	213
62	102	66	200	214
63	102	66	200	214
64	96	64	200	212
65	98	64	200	213
66	98	64	200	213
67	96	63	200	213
68	97	64	200	213
69	99	64	200	213
70	98	62	200	214
71	99	61	200	215
72	94	65	200	211
73	96	65	200	212
74	96	65	200	212

Table 3. Mass Transfer Data

Run Number	L/G	$\dot{m}_{air}$ mole/sec	$\dot{m}_{H_2O}$ mole/sec	CO_2 In %	CO_2 Out %	Efficiency % CO_2 Removed
31	50	0.407	20.33	5.10	4.62	9.26
32	50	0.397	19.84	4.27	3.86	9.70
33	50	0.482	24.09	3.94	3.61	8.39
34	50	0.480	24.01	4.28	3.76	12.00
35	50	0.587	29.34	4.14	3.72	10.17
36	50	0.588	29.41	4.18	3.72	11.1
37	50	0.675	33.76	4.15	3.72	10.35
38	50	0.684	34.74	4.27	3.88	9.14
39	50	0.678	34.52	4.35	3.95	9.02
40	50	0.767	39.01	4.21	3.80	9.83
41	50	0.766	38.31	4.12	3.66	11.29
42	50	0.840	41.99	4.28	3.81	10.96
43	50	0.907	45.34	4.27	3.77	11.59
44	50	0.897	44.85	4.09	3.67	10.24
45	50	0.835	41.76	4.01	3.53	11.85
46	50	0.420	10.49	3.95	3.72	5.82
47	25	0.524	13.10	3.87	3.65	5.78
48	25	0.615	15.39	4.18	3.91	6.43
49	25	0.421	10.54	4.03	3.78	6.18
50	25	0.514	12.86	4.45	4.21	5.59
51	25	0.616	15.40	4.34	4.04	6.95
52	25	0.761	19.05	4.29	4.05	5.63
53	25	0.888	22.21	4.44	4.19	5.55
61	25	1.136	28.40	4.28	3.95	7.70
62	25	1.027	25.67	3.72	3.45	7.30
63	25	1.027	25.67	4.01	3.68	8.26
64	25	1.116	27.90	4.91	4.48	8.71
65	25	1.208	30.19	4.83	4.37	9.63
66	25	1.192	29.79	4.25	3.88	8.91
67	25	1.286	32.15	3.92	3.57	8.88
68	25	1.260	31.50	4.21	3.86	8.29
69	25	1.335	33.37	4.23	3.85	9.08
70	25	1.212	30.29	4.43	4.09	7.78
71	25	1.361	34.02	4.23	3.81	9.93
72	25	0.896	22.40	3.81	3.61	5.35
73	25	0.893	22.32	4.44	4.18	5.81
74	25	0.757	18.92	4.38	4.15	5.40