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Performance of Packed Columns VII. The Effect of Holdup on Gas-Phase Mass Transfer Rates

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To account for the differences in the gas-phase mass transfer coefficients obtained by the use of vaporization and absorption techniques, data were obtained for ring and saddle packings in a 12-in. diam. column employing water vaporization and methanol absorption. The ratio of the vaporization to absorption coefficient, corrected for differences in diffusivity, is found to be directly proportional to the ratio of the total holdup to the operating holdup in confirmation of the proposed model in which the effective interfacial area for each type of operation is proportional to the holdup of the liquid active for that operation.

Numerous investigations of mass transfer rates in packed columns have resulted in the accumulation of a large amount of data which is of value in estimating the effects of changes in flow rates and physical properties on the performance of packed columns. The methods proposed for the determination of the absolute values of the mass transfer coefficients, however, are of doubtful value because there are large discrepancies between the data published by different investigators. The discrepancies are particularly apparent when gas-phase coefficients are measured by two different methods for the same packing. Comprehensive reviews of the literature presented by Pigford and Colburn in Perry's "Handbook" (2) and by Sherwood and Pigford (5) reveal discrepancies of several hundred percent between gas-phase coefficients obtained by water vaporization and ammonia absorption methods. Explanations in terms of end effects and the possibility of a slow chemical reaction accompanying the solution of ammonia are inadequate to account for such large differences.

In the first paper of this series (*6*) a tentative explanation of these differences was offered in terms of static, h_s, and operating, h_o, holdups. It was observed that the total liquid holdup, h_t, in a packed column was made up of relatively stagnant pockets corresponding to the static holdup and liquid which flows rapidly over the packing surface corresponding to the operating holdup, that is, $h_t = h_o + h_s$. It was proposed that for vaporization work the surface area of both types of holdup is effective whereas for absorption the stagnant pockets can become saturated and ineffective. As a result vaporization coefficients would be expected to be larger than absorption coefficients. Employing the data for ring packings available in 1955 it was possible to show a simple relationship between the coefficients and holdups as follows:

$$\frac{(k_g a)_{vap}}{(k_g a)_{abs}} = \frac{a_{vap}}{a_{abs}} = 0.85\,\frac{h_t}{h_o} \qquad (1)$$

Unfortunately the data used to obtain Equation (1) were obtained from a variety of conflicting sources with no data available from any one source employing both vaporization and absorption techniques. In addition all the absorption data used came from work employing ammonia absorption in water thus introducing uncertainties as to the effect of a slow chemical reaction in the liquid phase.

So that the validity of the proposed model could be determined in terms of holdups an investigation was undertaken to obtain vaporization and absorption data in the same equipment with attempts to eliminate end effects and the possibility of a chemical reaction in the liquid phase. This was accomplished by employing adiabatic water vaporization and methanol absorption in water in a column designed to minimize end effects. Several ring and saddle packings were used to extend the generality of the conclusion.*

Fortunately Yoshida and Koyanagi (8) carried out an independent study employing the same systems at the same time this work was underway, thus providing additional data for comparison and checking of the proposed model.

Holdups and the relationship between holdups and effective interfacial areas may be useful for providing qualitative and quantitative treatments of problems associated with absorption and chemical reaction in packed columns as well as the transient behavior of packed columns. In the earlier paper (6) it was predicted that the presence of static holdup should make mass transfer coefficients for ammonia absorption in acid solutions a function of ammonia concentration in the gas because the relatively stagnant liquid pockets will become more nearly saturated at higher ammonia concentrations. This was confirmed by the experimental data of Secor (3) who absorbed ammonia in boric acid solutions and found that the mass transfer coefficients decreased rapidly as ammonia concentration in the gas increased.

APPARATUS

In order to obtain data for several packings it was necessary to use a 12-in. diam. column. The 1.5-in. rings gave a minimum column diameter to nominal packing size ratio of eight to one.

End effects are known to introduce serious errors in mass transfer work particularly in the case of vaporization data where very short columns must be used to avoid too close an approach to equilibrium. Many previous investigators have attempted to eliminate these errors by estimating and correcting for the end effects. For this work an attempt was made to construct a column which would minimize end effects to such an extent that it would be unnecessary to employ corrections of doubtful reliability.

Uniform liquid and gas distribution are also of importance especially in short columns.

Liquid Distributor

The essential features of the liquid distributor are indicated in Figure 1. A twelve-armed distributor has been constructed from 3/8 in. O.D. hard copper tubing. The distributor arms, the ends of which are sealed by brass caps, are joined to a machined brass coupling by means of a force fit. Hole distribution on the copper plate soldered to the bottom of the coupling is shown in the diagram.

Hole diameter and arrangement are critical. If the total hole cross section is too small for the maximum liquid flow rate, liquid will spray above the packing. A linear liquid velocity of approximately 2 ft./sec. for a flow rate of 1,000 lb./hr sq.ft. has arbitrarily been chosen as a design criterion. This velocity corresponds to a hole diameter of 1/16 in. Hole distribution, selected to provide uniform liquid coverage, also insures that the entire surface of the packing is wetted. Twenty-five percent of the column cross section area and 25% of the holes lie *within* a 3 in. radius of the column center. Similarly, 56% of the cross section area and 66% of the holes are within a 4½ in. radius.

Gas Distributor

The design of the gas distributor, as shown in Figures 2 and 3, should largely eliminate gas and liquid contact beneath the bed. A bundle of risers and downcomers rests upon a steel drum which also supports the entire test column. The inlet gas stream cannot contact the effluent liquid owing to the arrangement of the thirty downcomers and fifty-seven risers. In order to insure that liquid does not enter through the riser openings, brass caps are placed above each row of risers. These seven caps form the support plate for the column packing and are secured to the two risers at the end of a given row by means of brass machine screws.

Half-inch Berl saddles fill most of the space beneath the top of the risers and the steel plate which rests upon the drum flange. This packing provides liquid drainage while creating sufficient resistance so that the gas stream is directed up through the test bed.

The gas inlet stream is separated from the liquid seal, the level of which is indicated by the sight glass, by a steel plate. A 1/8 in. clearance between the edge of this plate and the drum wall has been included in the design to allow for drainage of any liquid which might seep into the risers. A test run, after which the equipment was disassembled, indicated that such seepage does not occur.

Column

The column is a 12-in. I.D. Pyrex cylinder of 30-in. length. Glass is advantageous since its use allows visible determination of any spray condition which might exist above the packing. The flange of the gas-distributor drum is securely fastened to a steel angle rack which supports the entire column.

Figure 4 indicates the construction of the upper part of the system. Six tie rods, equally spaced, connect the plate adjacent to the glass column with the gas-distributor drum, thereby making the entire structure rigid. The plate which seals the top of the column contains a packing gland. This allows the liquid distributor to be positioned at any desired height within the column. Flexible pressure hose connecting the distributor shaft and the water supply line does not restrict its movement.

The schematic of the entire system, which can be used for both absorption and vaporization studies, is shown in Figure 5.

Atmospheric air, circulated by means of a multistage centrifugal blower, is used for both vaporization and absorption operations. During absorption studies the humidification column is employed to saturate the gas stream with water vapor. This column, 12 in. in diam. and 5 ft. high, is filled to a height of approximately 4 ft. with 1-in. carbon rings. Approximately 1 ft. of these rings covers the liquid distributor in order to eliminate any entrainment. The inlet gas stream is metered by either of two calibrated rotameters, and samples for assay are withdrawn before the flow rate is determined. Temperature measurements, made with thermometers and thermocouples, are taken at points indicated in the schematic.

Methanol vapor is introduced into the inlet air stream from a boiler. An immersion heater, power regulated by a variable transformer, supplies energy to the boiler. Use of an ammeter enables an approximate prediction of methanol concentration in the gas inlet stream. Thorough mixing of the air and methanol vapor is insured by a 6-ft. length of pipe preceding the rotameter manifold.

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* Tabular material has been deposited as document 7552 with the American Documentation Institute, Photoduplication Service, Library of Congress, Washington 25, D. C., and may be obtained for $1.25 for photoprints or for 35-mm. microfilm.

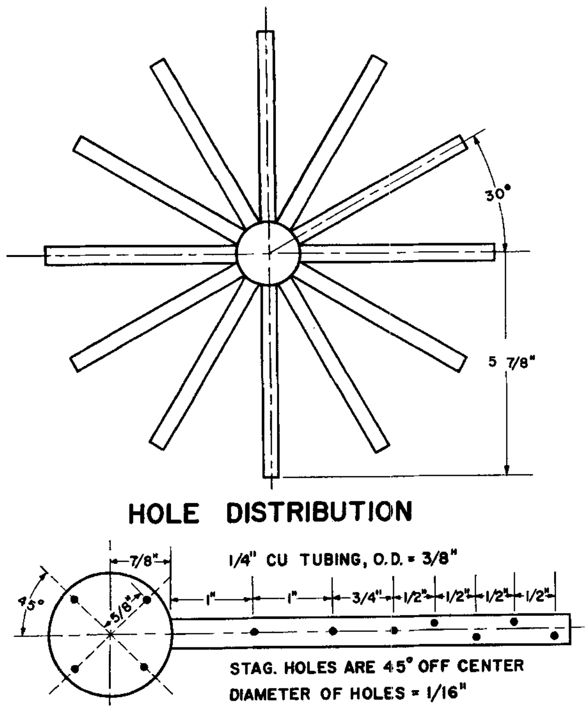

Fig. 1. Liquid distributor.

The water stream in the vaporization system is continually recirculated by a centrifugal pump so that its temperature will correspond to the adiabatic temperature of the inlet gas stream. A 55 gal. drum is used for a water reservoir and a line filter before the calibrated water rotameter removes any particles larger than 50 mesh.

An air preheater, equipped with automatic temperature controls, is included in the system and its use is optional.

Temperature Measurements

Temperatures of all streams, gas and liquid, are recorded during vaporization and absorption runs. Thermocouple measurement of stream temperatures serves as a check and also allows a correction to be calculated for heat transfer occurring in the gas-distributor drum. Thermopile construction is necessary to obtain the required sensitivity. Five chromel-constantan thermocouples, connected in series, measure the liquid-exit temperature at various points below the packed bed. These couples, inserted in the ½ in. Berl saddles beneath the gas risers, directly give an average temperature for the liquid effluent as it leaves the test bed. The inlet gas temperature is measured by four copper-constantan couples, in series, placed in different gas riser ports. Directly above the packing, and inserted through the pressure tap,. are four series-connected copper-constantan thermocouples. Positioned in the base of the liquid distributor are three chromel-constantan couples, which measure the temperature of the inlet water. All thermopiles are calibrated with respect to a cold junction temperature at 0°C.

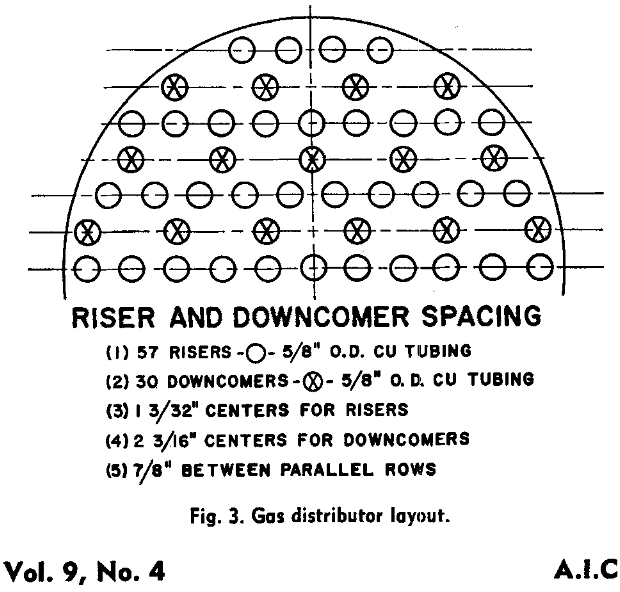

Fig. 3. Gas distributor layout.

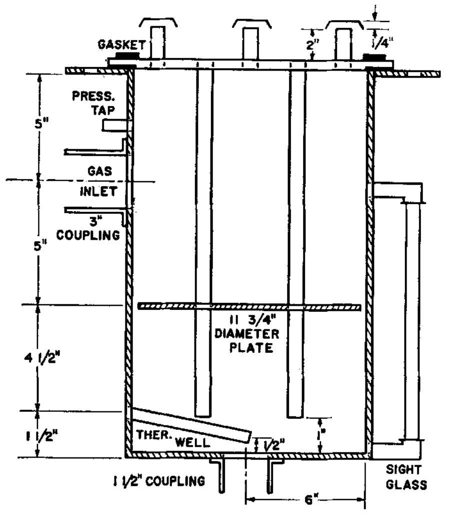

Fig. 2. Base of column. Gas distributor-schematic.

SELECTION OF SYSTEM

For absorption systems, one experimentally determines the overall volumetric mass transfer coefficient, K_ga (or K_La). Since the gas-phase coefficient is required, it is desirable to select a system for which the major resistance to the transfer process lies within the gas phase. Liquid-phase resistance can be approximated by the Sherwood and Hollaway (4) correlation; however, a great degree of dependence upon liquid-phase correction casts doubt upon the validity of the final result.

Data of Houston and Walker (*1*) indicate over 90% of the total transfer resistance lies within the gas phase for the methanol-water system. For this system, liquid-phase correction cannot possibly contribute an appreciable error to the calculated value of k_ga.

For all vaporization processes, the only resistance is that of the gas phase if the column is operated as an adiabatic saturator. Economic considerations dictate the use of water.

ANALYSES AND CALCULATIONS

Methanol Absorption

Volumetric mass transfer coefficients are obtained from the solution of

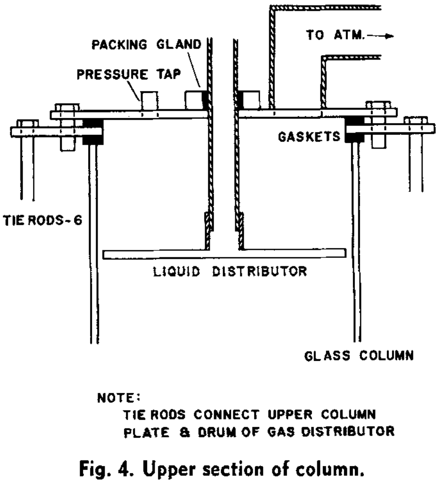

Fig. 4. Upper section of column.

TABLE 1. CONSTANTS C FOR EQUATION (7)

Packing	C
0.5 in. porcelain saddles	1.07
1.0 in. porcelain saddles	1.16
1.0 in. porcelain rings	1.01
1.5 in. porcelain rings	0.92
1.0 in. carbon rings (carbon holdups)	0.65
1.0 in. carbon rings (porcelain holdups)	0.95

$$Z = \int_{y_2}^{y_1} \left[\frac{G}{k_g a \, P M_M} \right] \left[\frac{dy}{(1-y)(y-y_i)} \right] \quad (2)$$

Low solute concentration results in an operation characterized by linear operating and equilibrium lines, and the use of a log mean overall driving force and an overall coefficient is justified. When the absorption process is restricted to dilute systems, the above equation reduces to

$$K_g a = \left[\frac{G}{Z P M_M} \right] \left[\frac{y_1 - y_2}{(\Delta y)_{\text{lm}}} \right] \quad (3)$$

For all of the methanol absorption runs the concentration of methanol in the liquid stream was so low that the methanol partial pressure could be assumed to be zero. As a result Equation (3) reduces to

$$K_g a = \left[\frac{G}{Z P M_M} \right] \left[\frac{y_1}{y_2} \right] \quad (4)$$

To obtain $k_g a$ from $K_g a$ a small correction for liquid-phase resistance was made by means of the Sherwood and Hollaway correlation (*4*).

Gas-phase analysis was accomplished with a gas chromatograph. Methanol peak heights were found by calibration to be proportional to concentration. This made it possible to employ peak height ratios in place of concentration ratios in Equation (4).

During the early runs the system was checked by material balances. Liquid streams were analyzed for methanol by means of a differential refractometer.

Water Vaporization

Vaporization work is characterized by an extremely quick approach to equilibrium within the test bed. Therefore, water-vapor analysis is extremely critical since small driving forces are encountered with only several inches of bed depth. Gravimetric procedures, although reliable, are lengthy and offer metering problems. Wet-dry bulb thermometry has the advantage of being extremely quick and convenient. If the vaporization system operates at the adiabatic saturation temperature of the inlet air, temperature measurements can be used directly for calulating the volumetric gas-phase mass transfer coefficient. In actual operation, the system did not obtain true adiabatic conditions, for the water temperature decreased, approximately 0.3°C., as it flowed through the column. Calculation of inlet and exit humidities was therefore necessary. The equation, obtained from a mass and energy balance (*7*), describing the process is

$$H = \left[\frac{P_w}{P - P_w} \right] \left[\frac{18}{29} \right] - \left[\frac{T_D - T_w}{\lambda_w} \right] \left[\frac{h_g}{k_H} \right] \quad (5)$$

The psychometric ratio, (h_g/k_H), has been experimentally evaluated by a number of investigators, and the recommended value (*7*) of 0.236 was used. When using wet-dry bulb thermometry, one should realize the physical operation for which the preceding relationship applies. A radiation correction has not been included since this is negligible if the gas stream passing the wet-bulb thermometer has a fairly high velocity and all temperatures measured are close to room temperature. Another condition to be satisfied is supplying water to the wet-bulb thermometer at the wet-bulb temperature. The apparatus used in this work contains a reservoir bottle which continuously feeds water to the wet-bulb cotton wick. This wick, extending horizontally for approximately 1 ft., is in the direct path of the exhaust gas stream. Sufficient wick length allows the water to attain the wet-bulb temperature before reaching the thermometer.

Two simple units allow simultaneous inlet and exit stream measurements. Basic components are 1-in. copper tees in which the thermometers are vertically mounted. The tees are inserted at the ends of 1-in. copper lines, horizontally, which are connected to the rotameter manifold and column exhaust system. All thermometers, including those measuring gas and liquid stream temperatures, were calibrated against a Bureau of Standards thermometer and cover the temperature range for 0° to 100°C. in increments of 0.1°C.

The mass transfer coefficient for vaporization was calculated from

$$k_g a = \left[\frac{G}{29 \, PZ} \right] \left[\frac{H_2 - H_1}{(\Delta H)_{\text{lm}}} \right] \quad (6)$$

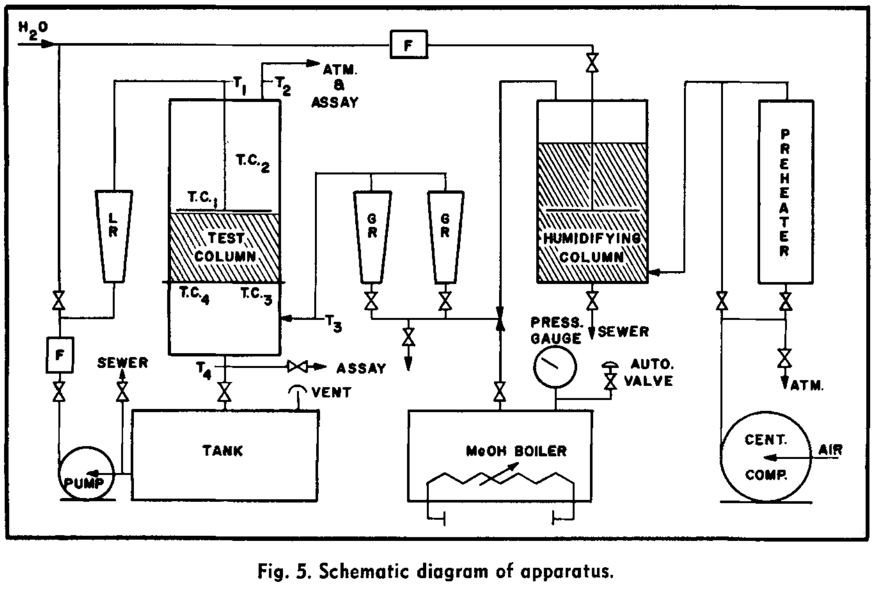

Fig. 5. Schematic diagram of apparatus.

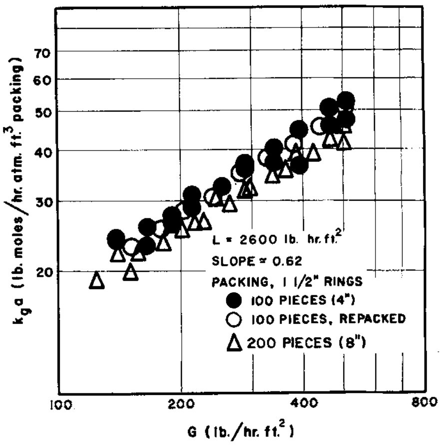

Fig. 6. Effect of repacking and packing height. Water vaporization data.

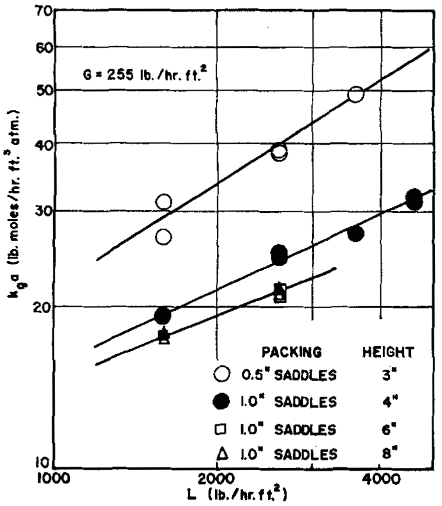

Fig. 7. Methanol absorption in porcelain Berl saddles.

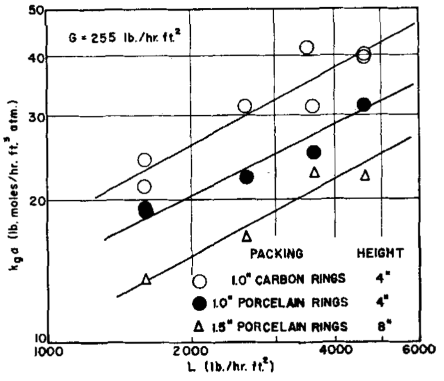

Fig. 8. Methanol absorption in carbon and porcelain Raschig rings.

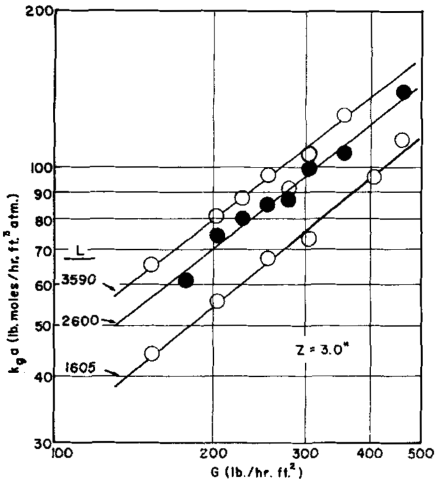

Fig. 9. Vaporization of water in 0.5-in. porcelain Berl saddles.

PROCEDURE

Vaporization

Packing in the humidification column is dried thoroughly. During vaporization runs, no water flows through this column. The blower is turned on 1 hr. before the start of a test run. Approximately 25 gal. of line water initially contained in the liquid effluent tank is continually recirculated by the centrifugal pump. Liquid flow rate, controlled by a valve in the line, is set at a high value. After a period of 1 hr., stream rates are set at the desired levels. Barometric pressure is recorded. Other measurements for a given run are stream thermometer and thermocouple temperatures, flow rates, and the wet and dry bulb temperatures of the gas streams.

Absorption

Water for the test column is continually drawn from the suppy line during absorption runs. Line water also flows through the humidifying column. A current of 7 amps from a 220 V A.C. power source provides sufficient methanol boilup. Barometric pressure, stream temperature, and flow rates are recorded as in vaporization runs. To insure that steady state conditions prevail, the run is monitored by gas chromatography. The run is terminated when gas stream analysis has been established.

Testing of Column

Figure 6 shows the results of some of the test runs made to determine the effect of repacking the column and the presence of end effects. From these tests it can be concluded that there is no effect due to repacking, and that end effects for extreme conditions (1.5-in. rings and short packing heights) represent no more than a 10% contribution to the mass transfer coefficients. Since vaporization and absorption runs are compared as ratios determined at the same flow rates and packing heights the end effects are effectively eliminated.

EXPERIMENTAL RESULTS AND DISCUSSION

To test the applicability of a relationship of the form given in Equation (1) a variety of packings was employed. Figures 7, 8, 9, 10, 11, 12, and 13 give water vaporization and methanol absorption data for 0.5-in. and 1-in. porcelain Berl saddles, 1-in. and 1.5-in. porcelain Raschig rings, and 1 in. carbon Raschig rings. To compare the desired ratios of the smoothed data the vaporization coefficients were corrected by use of the gas-phase diffusivity ratio taken to the 0.67 power. Then $(k_g a)_{vap}/(k_g a)_{abs}$ which is equal to a_{vap}/a_{abs} was plotted vs. the holdup ratio h_t/h_o in Figure 14. Holdups were obtained from the first paper in this series (6). The data can be approximated by straight lines for each packing of the form

$$\frac{a_{vap}}{a_{abs}} = C\frac{h_t}{h_o} \tag{7}$$

Table 1 shows the values of C for each packing.

The values for C are all close to 1.0 except for carbon rings. The earlier work on holdups (6) indicated that operating holdups for 1-in. porcelain rings and 1-in. carbon rings were of the same order of magnitude, but static holdups were four times greater for the carbon rings. This discrepancy was attributed to the porosity of the carbon rings. If this were true it should be possible to correlate the carbon-ring mass transfer data with porcelain-ring holdup ratios. These attempts are included in Figure 14 and Table 1 from which it can be seen the C value obtained, 0.95, is close to those obtained for the porcelain rings.

The data of Yoshida and Koyanagi (8) for the same systems employed in this work are also included in Figure 14. This was done by using 0.5-in. porcelain-ring holdups for the 15-mm. ring data and 1.0-in. porcelain-ring holdups for the 25-mm. ring data. The agreement varies from good for the 15-mm. rings to fair for the 25-mm. rings. Part of the disagreement may be accounted for by noting the use of a 12 cm. column with 25-mm. rings. For such

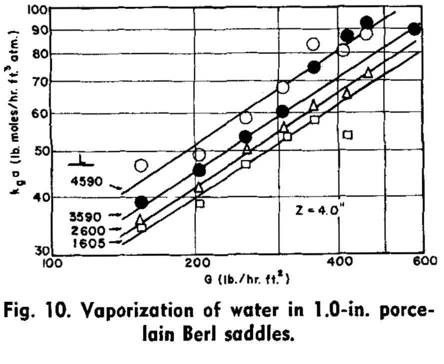

Fig. 10. Vaporization of water in 1.0-in. porcelain Berl saddles.

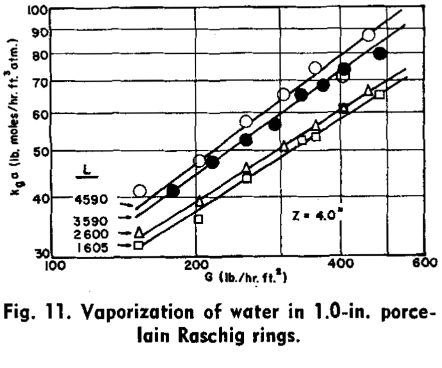

Fig. 11. Vaporization of water in 1.0-in. porcelain Raschig rings.

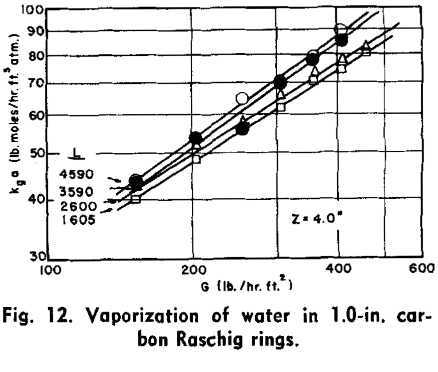

Fig. 12. Vaporization of water in 1.0-in. carbon Raschig rings.

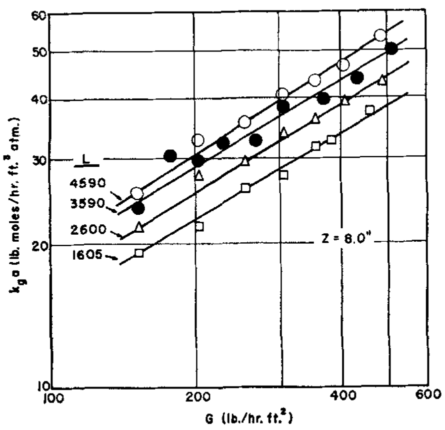

Fig. 13. Vaporization of water in 1.5-in. porcelain Raschig rings.

a low column diameter-to-ring ratio (4.8) the wetted area of the column wall can make an appreciable contribution to the apparent performance of the column and result in an a_{vap}/a_{abs} ratio closer to 1.0 than would be expected for packing alone. Since the disagreement lies in this direction the explanation may be valid.

For design purposes it can be seen that all of the data can be represented by a C value of 1 within ±15% provided porcelain holdups are used for carbon rings. The use of this value to give

$$\frac{(k_g a)_{vap}}{(k_g a)_{abs}} = \frac{a_{vap}}{a_{abs}} = \frac{h_t}{h_o} \tag{8}$$

provides a very simple design correlation and eliminates the uncertainties of up to several hundred percent which had been reported earlier (*2*, *5*).

Undue importance should not be attached to the direct proportionality between the ratios of Equation (8). The implication here is that the operating holdup represents pockets of fluid which are perfectly stagnant. Earlier observations (*6*) indicated that the pockets in 1.5-in. rings are washed out by the random splashing and flow of fresh liquid at least every 5 to 10 minutes. This rate of turnover may not be significant for physical absorption, but for absorption accompanied by chemical reaction the high capacity of the liquid may result in significantly higher rates of mass transfer approaching those predicted for vaporization.

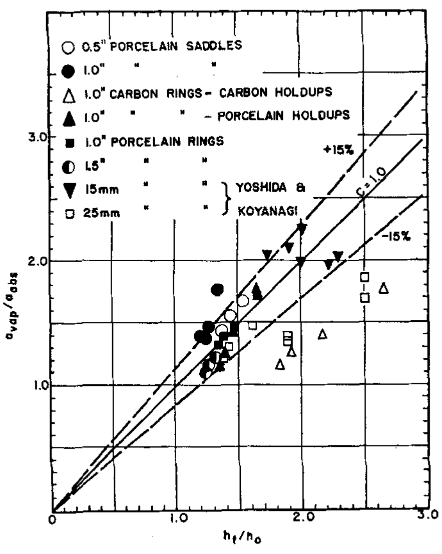

Fig. 14. Correlation of $(a_{vap})/(a_{abs})$ with h_t/h_o.

SUMMARY

An experimental investigation of absorption and vaporization in packings indicates that differences in mass transfer rates can be accounted for in terms of effective interfacial areas which are proportional to holdups as given by Equation (8).

ACKNOWLEDGMENT

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NOTATION

a = effective interfacial area, sq.ft./cu.ft.
C = proportional constant in Equation (7)
G = superficial gas rate, lb./(hr.)(sq.ft.)
H = humidity, lb. water/lb. of dry air
h_g = gas-phase heat transfer coefficient, B.t.u./(hr.)(sq.ft.)(°F.)
h_o = operating holdup, cu.ft./cu.ft.
h_s = static holdup, cu.ft./cu.ft.
h_t = total holdup, cu.ft./cu.ft.
k_g = gas-phase mass transfer coefficient, lb. moles/(hr.)(sq.ft.)(atm.)
K_g = overall gas-phase mass transfer coefficient, lb. moles/(hr.)(sq.ft.)(atm.)
k_H = gas-phase mass transfer coefficient, lb./(hr.)(sq.ft.)(ΔH)
L = superficial liquid rate, lb./(hr.)(sq.ft.)
M_M = mean molecular weight of gas, lb./lb. mole
P = total pressure, atm.
P_w = vapor pressure at wet bulb temperature, atm.
T_D = dry bulb temperature, °F.
T_w = wet bulb temperature, °F.
y = solute mole fraction
Z = packing height, ft.
λ_w = latent heat of vaporization at wet bulb temperature, B.t.u./lb.

Subscripts

abs = for physical absorption
i = at the interface
vap = for vaporization
1 = at bottom of column
2 = at top of column

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