Radioisotope Technique for the Determination of Flow Characteristics in Liquid-liquid Extraction Columns

S. E. MARKAS and R. B. BECKMANN
Carnegie Institute of Technology, Pittsburgh, Pennsylvania

A radioisotope technique has been utilized to study the point holdup of the dispersed phase and some operational flow characteristics in a packed countercurrent liquid-liquid extraction tower. The system used was toluene-water and the packing was Raschig rings. The toluene dispersed phase was tagged with gamma-radiating iodine-131.

The study revealed that holdup experiences a hysteresis cycle with variations of the continuous-phase flow rate. Correlating equations are presented for "total" and permanent holdup, below loading, to account for this hysteresis. Entrance effects and flow maldistribution effects are readily determined by the method utilized. Displacement studies demonstrated that no simple "operational" dispersed-phase flow holdup exists, but rather a complex dispersed-phase movement, which involves all the "nonhysteresis" permanent holdup.

During recent years the widespread use of packed dispersed-solvent–extraction columns as a means of separating the components of solutions has shown the need for fundamental information on the extraction mechanism of the column. This investigation was undertaken to elucidate the phenomena of dispersed-liquid-phase holdup* within a randomly packed extraction column in a partial attempt to determine the interfacial area of contact of the two phases. Holdup information, along with drop-size information, for a given system and packing provides a means of calculating the interfacial-contact area.

The effect of (1) flow rates and (2) packing size upon the point holdup of a toluene dispersed phase flowing countercurrent to a continuous water phase in a packed liquid-liquid extraction column was studied by means of a radioisotope technique which entailed the measurement of gamma radiation emitted from the "tagged" toluene droplets containing iodine-131.

In general, the available holdup data in the literature can be classified into two principal categories, depending upon the manner in which the data were obtained: (1) holdup data in which the volume of holdup was measured by draining the column and separating the desired phase and (2) holdup data obtained by simultaneously closing all streams to and from the column and then measuring the volume of continuous phase needed to restore the altered two-phase interface to its initial position.

Row, Koffolt, and Withrow (*6*) obtained holdup data during their study of the rate of mass transfer of benzoic acid from water to toluene in an 8.75-in. I.D. packed column. Their holdup data on ½-in. Berl saddles, ½-in. Raschig rings, and knitted-copper-cloth packing indicate a sharp increase in the liquid holdup as flooding was approached. Allerton, Strom, and Treybal (*1*) measured holdup in a 3.56-in. I.D. column packed with ½-in. carbon rings with benzoic acid as the solute in the kerosene-water system. Their results indicated an increase in holdup with increasing rates of both phases. Gier and Hougen (*5*) reported similar results in their holdup measurements made in a 6.0-in. I.D. column packed with ½- and ¾-in. Raschig rings. Their holdup data, which were obtained by determining the volume alteration in the lowering of the interfacial level, were found to be a linear function of the dispersed-phase rate when the continuous-phase rate was constant.

Gayler and coworkers (*3* and *4*) conducted an extensive study of dispersed-phase holdup using 3-, 6-, and 12-in. I.D. columns with a packed height of 10 ft. The packing used were ¼-, ⅜-, ½-, ¾-, 1- and 1½-in. ceramic Raschig rings and ¼-, ½-, and 1-in. ceramic Berl saddles. The continuous phase in each of the systems studied was water; methyl isobutyl ketone, butyl acetate, dibutyl carbide, benzene, and isooctane were each used as the dispersed phase. The normal holdup data used by the authors in developing their conclusions were obtained by determining the volume alteration in the lowering of the interfacial level. During the course of the investiga-

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S. E. Markas is with California Research Corporation, Richmond, California.

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*In liquid-extraction columns the term *holdup* will be used to designate the fractional holdup of the dispersed phase in the portion of the total column volume which may be occupied by liquid.

tion it was found that an additional volume of the dispersed phase was also present in the column, presumably in the form of droplets trapped in the packing. The quantity of permanent holdup present was determined by draining the column after removing the normal holdup from the column. The authors concluded that only the normal holdup was involved in the mass transfer operation; consequently, the normal holdup data were correlated by means of the following equation:

$$V_D + \frac{X}{1 - X}(V_C) = F\bar{v}_0 X(1 - X) \qquad (1)$$

Where V_D and V_C are the superficial velocity of the dispersed phase and the continuous phase respectively. X is the fractional holdup, F is the fractional voidage of packing, and $\bar{v}_0$ is a velocity characteristic of a given packing.

Wicks and Beckmann (*7*) conducted a study of dispersed-phase holdup using three extraction columns and six different sized packings. The columns were 3-, 4-, and 6-in. I.D., and the packings consisted of ¼-, ⅜-, ½-, ⅝-, ¾-, and 1-in. unglazed porcelain Raschig rings. One liquid-liquid system was used, with toluene as the dispersed and water as the continuous phase, the experimental technique being the same as that utilized by Gayler and Pratt (*3*). The normal holdup, denoted as *free* holdup, was obtained by returning the interface to its initial position; permanent holdup was measured by drainage of the column. An empirical correlation was presented for total holdup (i.e., the summation of free and permanent holdup):

$$X_T = A_1(V_D)^r + B_1(V_D)(V_C)^s \qquad (2)$$

where X_T is the total holdup, V_D and V_C are the dispersed- and continuous-phase flow rates, respectively, and the other terms represent constants characteristic of a given packing size and column diameter.

APPARATUS

A schematic flow diagram of the equipment is shown in Figure 1. Basically the equipment utilized was the same as reported earlier by Wicks (*7*) with the addition of the counting equipment to measure the gamma radiation from the tagged toluene dispersed phase.

Scintillation-counting Circuit

A diagram of the scintillation-counting circuit is illustrated in Figure 2. The scintillation counter *A* and the rate meter and high-voltage supply *B* were Nuclear-Chicago models. The recorder *C* was manufactured by the Esterline-Angus Company.

The a.c. input *F* to the rate meter was standard, 110 to 115 volts, 60 cycle. The rate meter *B* was capable of producing voltages up to 1,500 volts and registering counting rates to 50,000 counts/min. Cable *D* supplied the high voltage to the scintillation counter. The cathode follower input cable *E* transmits the necessary low voltages and also receives the negative ionization pulses produced by the counter. Cables *H* represent the chart drive to the recorder and carry alternating current (110 to 115 volts, 60 cycle). The final component of the circuit *G* denotes cables, through which the counting rate is transmitted to the recorder by means of a d.c. milliamperage signal.

The scintillation counter is shown in detail in Figure 3. The detector consisted of a sodium iodide thallium-activated crystal *B*, a Lucite light pipe *D*, a DuMont 5819 photomultiplier tube *H*, and a preamplifier *I*, for effective coupling of the signal to the count-rate meter. The gamma radiation from iodine-131, entered the detector and caused scintillation within the crystal. These gamma photons are channeled by the light pipe, and the signal is in turn amplified by the multiplier tube.

As shown in Figure 3, an appreciable quantity of lead was utilized for shielding and collimation. The sensitive scintillation counter thus shielded is capable of detecting gamma activities in samples as low as 10^{-10} curie. The collimating shield permitted sharp discrimination against activities more than 15 deg. off the axis of the probe. Scattered and extraneous gamma radiation were filtered from the counter by means of a 1/32-in. lead absorber, and consequently only the 0.364-mev. primaries of iodine-131 were predominately measured.

The scintillation counter and lead shield rested on a mounting, which in turn rested on a platform consisting of a 5-ft.-square section of ¾-in. plywood, with a 1-ft.-diam. hole in the center, permitting the column to be positioned in the center. The platform was attached to the angle-iron support structures by means of elevating screws, which enabled the platform to be raised or lowered and thus permitted a scanning of the column at various vertical positions. In addition, rollers attached to the underside of the counter mounting allowed it to be revolved for a circumferential scan of the extraction column.

Chemical Form of Iodine-131

For this investigation iodine-131 was required to be in a chemical form which was soluble in toluene and completely insoluble in water, to permit "tagging" of the toluene droplets. After a survey of the available forms of the isotope, it was decided that a radioiodinated triolein supplied by the Abbott Laboratories of North Chicago, Illinois, was satisfactory. This compound was prepared from molecularly distilled 99.5% triolein and had iodine-131 attached to the double-bonded carbon groups.

Experiments with a DuNoiiy Tensiometer revealed that iodinated triolein and its triolein carrier had no effect on the interfacial tension of the toluene-water system.

Column Packings

Four different sizes of nonporous unglazed-porcelain Raschig rings were used: ½, ⅝, ¾, and 1 in. The packing was carefully sorted to remove all broken or irregular pieces and was initially cleaned by washing in a 1-1 hydrochloric acid–water solution. The column was packed by slowly dropping several rings at a time into the column, which was filled with water. The method proposed by Wicks (*7*), that of passing an air stream up through the column at a rate high enough to flood the column was utilized to settle the packing; this is the method generally accepted for a packed absorption column.

EXPERIMENTAL PROCEDURE

The holdup of the dispersed-solvent phase, toluene, passing countercurrent to the continuous-solvent phase, water, was investigated. Before operation was started, the liquids in the feed drums *A* (Figure 1)

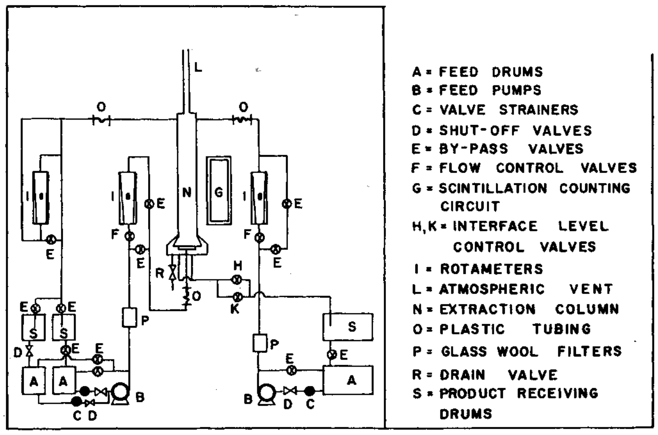


Fig. 1. Schematic flow diagram of experimental equipment.

were mutually saturated with the opposite solvent, and the radioiodinated triolein containing iodine-131 (about 6 mcurie.) was placed in the toluene feed drum *A*. The liquids were circulated through the column for a period of about 6 hr. to insure that all of the isotope was uniformly distributed throughout the solvent phase.

For a run the by-pass line valves *E* were partially closed and the shut-off valve *D* in the water feed line at the entrance to the tower was opened. The flow rate of the aqueous phase was set by adjusting the flow control valve, *F*. This value was indicated by reading the rotameter *I* in the water feed line. The shut-off valve *D* in the toluene feed line was opened, and the flow rate of the toluene phase was set by adjusting the toluene flow-control valve *F*. Toluene entered the column via the dispersed-phase distributor and rose through the packing as drops. Valve *K* was adjusted until the two-phase interface was located approximately 3 in. above the top of the packing. By means of the finer control valve *H*, the interface was located at the exact mark, 3 in. above the packing. Operation was held constant and the flow rates were observed on the rotameters *I*.

The scintillation counter was oriented so that the front end was approximately 6¼ in. from the outer edge of the column. It was so positioned that the counter and the cross section of the column were symmetrical; i.e., the axis of the probe passed through the center of the cross section of the column. The platform was elevated to a position where the scintillation counter viewed the middle section of the column at a point equidistant from the entrance and exit sections.

When a count was taken, the rotameter (Figure 2) was turned on and the high voltage adjusted. After a period of approximately 20 min. both the toluene-water system and the rate meter came to equilibrium. At this point the Esterline-Angus recorder was turned on to give a permanent record of the counting rate.

At a given vertical position the counter was revolved around the column to four different positions 90 deg. apart, and a separate counting record was obtained for each. For the larger packing, i.e., ¾- and 1-in. rings, two and sometimes three different vertical positions were also investigated. This produced eight and even twelve separate counting rates for averaging purposes at a given dispersed- and continuous-phase flow rate. These horizontal planes were 3 in. apart.

To calibrate these radiation rates in terms of holdup, i.e., the fractional part of the total void volume within the column that is occupied by the dispersed-phase droplets, the column was filled only with a stationary column of toluene. Correspondingly, all the void volume in the column was occupied by the normally dispersed "tagged" phase. The radiation emitted under these conditions was recorded as outlined above. The ratio of the two radiation intensities (with suitable corrections) yielded directly the fractional holdup within the column under operating conditions.

In the investigations to determine whether all or a fraction of the total holdup had a net movement through the column, both a tagged and an "isotope-free" toluene phase were employed.

Initially equilibrium holdup was generated in the column by the method previously outlined, only the water and tagged toluene phases being used. When equilibrium conditions were reached, the flow of the tagged toluene was stopped and replaced by the flow of an isotope-free toluene phase. This was accomplished by closing valves *D* and *E* (Figure 1) corresponding to the tagged toluene feed and receiving tanks *S* and *A* and opening valves *D* and *E* corresponding to the isotope-free toluene-phase feed and receiving tanks *S* and *A*. Consequently the radiation rates detected by the scintillation-counting circuit *G* were indicative of the rate at which the dispersed phase was being displaced and of the quantity of the total holdup that had a net movement through the column, resulting from collisions with succeeding toluene droplets.

The quantity of radioiodinated triolein used was generally 6 mcurie. when it was initially placed in the system. Because of decay, fresh batches of isotope had to be introduced into the system every 3 or 4 weeks.

TYPES OF DISPERSED-PHASE HOLDUP

Gaylor and Pratt (*3*) and Wicks (*7*) have presented data and correlations for the dispersed-phase holdup within a packed extraction column. The total dispersed-phase holdup was defined as the sum of the free holdup and the permanent holdup. The free holdup was measured by determining the number of dispersed-phase droplets that would rise to the interface and coalesce when all entry and exit streams to and from the column were stopped. The permanent holdup was then determined by draining and washing the column free of all the remaining dispersed phased trapped within the packing.

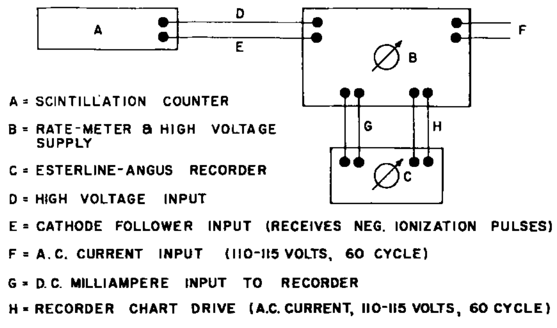


Fig. 2. Diagram of scintillation-counting circuit.

Gaylor and Pratt (*3*) presumed that the permanent holdup was permanently locked in the packing and that, becoming saturated, it has no influence on mass transfer. Wicks (*7*) however, presumed that some of this permanent holdup had a net movement through the column, resulting from collisions with succeeding droplets, and consequently tentatively defined an "operational" holdup. This holdup consisted of the free holdup plus a fraction of the permanent holdup presumably having a net movement through the column. Wicks endeavored, to free these temporarily held droplets by means of pulsations of the continuous phase, although he realized that this procedure only approximated the actual droplet behavior.

By using a tagged toluene phase, as well as an isotope-free toluene phase, these studies revealed that besides the free holdup all the permanent holdup had a net movement through the column. Although all these droplets comprising the permanent holdup have an over-all movement, this movement is not uniform. A range of different retention times in the packing exists for these permanent holdup droplets.

In addition, this investigation revealed a new type of holdup, which is termed *hysteresis* holdup. As the name implies, it was noted that total holdup experienced a hysteresis cycle when the continuous-phase velocity was varied from a given value to a maximum terminal value and back again to the original value at a given dispersed-phase velocity. This hysteresis did not exist for variations of the dispersed-phase velocity at a given continuous-phase velocity in the region below loading. Although the free holdup is the same for the given V_D and V_C, this hysteresis is actually experienced by the permanent holdup and consequently influences the total holdup of the dispersed phase. As a result, four types of holdup were studied:

1. *Permanent holdup*—the volume of dispersed phase which is trapped in the packing and does not rise freely to the interface because of difference in density.
2. *Total holdup*—the total volume of dispersed phase within the effective packing volume at any time, i.e., free holdup plus permenent holdup.
3. *Hysteresis holdup*—the volume of

dispersed phase which is permanently trapped in the packing as a consequence of the prior history of the column with respect to variations of the continuous phase. At a given dispersed-phase velocity the same curve does not define the variation of permanent holdup with increasing and decreasing continuous-phase velocity. The difference between these two curves at a given V_C represents the hysteresis holdup.

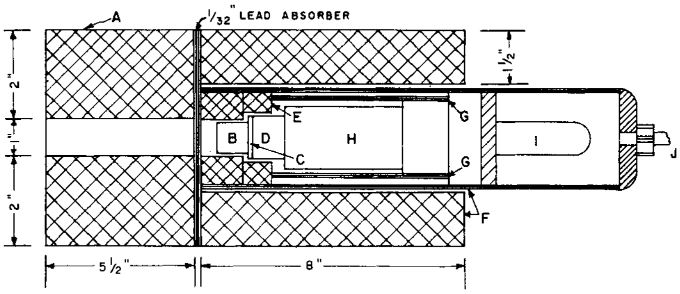
4. *Operational holdup*—the volume of dispersed phase which Wicks (7) considered to be the active portion of the dispersed phase taking part in mass transfer.

EXPERIMENTAL HOLDUP RESULTS

Values of permanent and total holdup, reported as volume percentage of the total void, were obtained and grouped in series to illustrate the hysteresis effect. The range of data covered a 6-in. I.D. column packed with ½-, ⅝-, ¾-, and 1-in. Raschig rings and an unpacked spray column. Table 1 lists typical experimental results obtained for ⅝-in. rings in the 6-in. I.D. column.

The total-holdup data obtained were checked for consistency by plotting the percentage of total holdup vs. the dispersed-phase velocity at an constant continuous-phase velocity. A typical illustration of the consistency obtained is presented in Figure 4.

The total-holdup results obtained for a specific packed bed were reproducible within 8 to 9% on a relative basis. The results for the runs with the ½- and ⅝-in. packings were reproducible within 5 to 6%, owing to the larger magnitude of the holdup values. The holdup data for the ¾- and 1-in. rings, as well as for the spray column, however, were reproducible within 9 to 10%. Similar trends in reproducibility existed for the permanent holdup, although, because of the smaller range of magnitudes involved, the percentages tend to be a little higher.



A = COLLIMATING SHIELD (LEAD) (TAPERED SLIT, 5.5" x 1")
B = NaI (Tl) CRYSTAL (0.75" DIAMETER x 0.75" LONG)
C = SILICONE OPTICAL COUPLING
D = LUCITE LIGHT PIPE
E = INTERNAL LEAD SHIELDING
F = HOUSING & EXTERNAL SHIELD
G = MUMETAL MAGNETIC SHIELD
H = PHOTOTUBE
I = PREAMPLIFIER
J = ELECTRIC CABLE

Fig. 3. Scintillation-counter specifications.

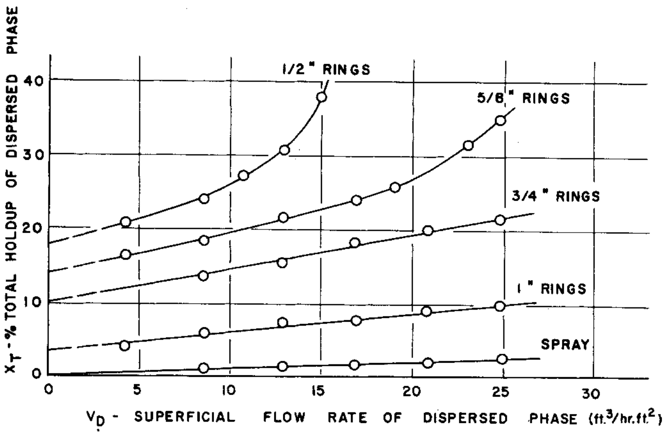


Fig. 4. Percentage of total holdup at zero continuous-phase flow.

Channeling of the Dispersed Phase

As mentioned in the description of the experimental procedure, in order to obtain an average holdup at a given vertical position in the packing, the scintillation counter was revolved around the column. A typical illustration of the variation encountered is presented in Figure 5. For the 1-in. rings the variation is quite pronounced, with the maximum value deviating 33% from the minimum value. This deviation is about 25% for the ¾-in. rings and only 9% for the ⅝- and ½-in. rings. It would thus appear that the 25 and 33% deviations represent channeling or maldistribution of the dispersed-phase droplets. The 1- and ¾-in. rings in the 6-in. I.D. column represent a column diameter to packing size ratio of 6.0 and 8.0 respectively, and in the other two cases the ratio is 9.6 and 12.0. The criterion, therefore, to eliminate channeling in a packed extraction column would be that the ratio of column diameter to packing size should be maintained larger than 8.0 and preferably about 10.0.

TABLE 1. COEFFICIENTS FOR CORRELATION OF HOLDUP DATA

Packings size, in.	A_1	B_1	C_1	D_1
½	17.5	0.402	0.768	0.0914
⅝	13.5	0.080	0.658	0.00682
¾	10.5	0.038	0.480	~0.001
1	3.8	~0.0	0.201	~0.0008
Spray	0.0	0.0	0.099	~0.0

The tabulated values of total holdup shown on Figure 5 represent the average of the four values obtained through circumferentially scanning the column. Various other methods of averaging were analyzed, including ones postulated by radiation-counting theory. It was found, however, that the circumferential deviation was not of sufficient magnitude to permit other averaging methods to yield results which were different from the arithmetic average; consequently the experimental results of holdup represent arithmetic averages.

Holdup Hysteresis

The dispersed-phase–holdup results were obtained by setting V_D and increasing V_C from zero in intervals as outlined by the data to a terminal V_C. When the variation was reversed, i.e., V_C decreased from its maximum value back to zero again, the data did not fall on the original curve. In other words there was a hys-

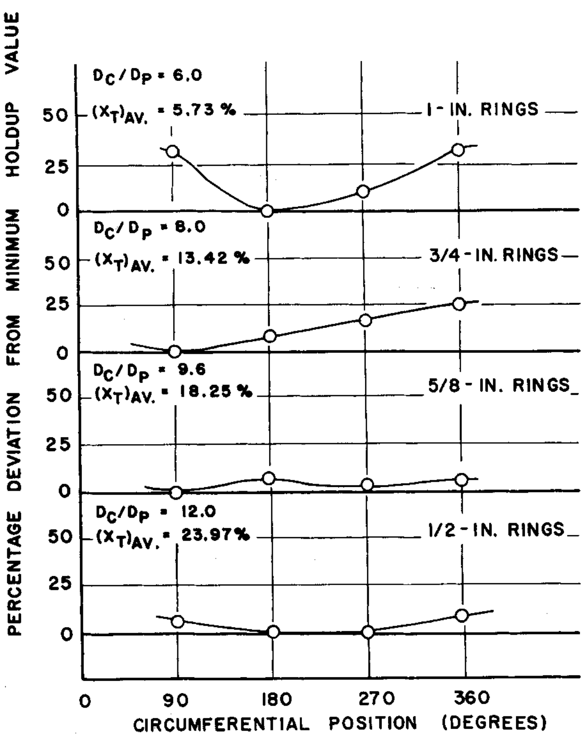


Fig. 5. Circumferential variation of holdup at midpoint of packed height; V_D = 8.51 cu. ft./(hr.)(sq. ft.), V_C = 0.

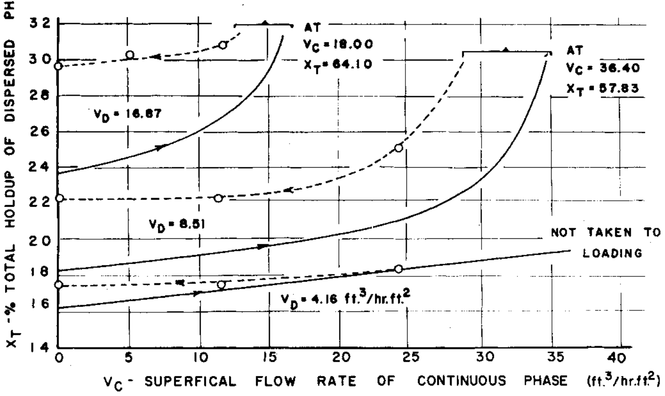


Fig. 6. Percentage of total holdup for 5/8-in. rings with hysteresis of holdup.

teresis effect. To validate these results, runs were performed which lasted 3 to 4 hours, revealing that hysteresis actually was present; the effect was not due to unsteady state conditions. Figure 6 represents an illustration of this effect for the ⅝-in. Raschig rings. The lower curve with V_D = 4.16 as a parameter corresponds to data taken in the region below loading; the hysteresis holdup (difference between upper and lower positions of curve) is rather small. For V_D = 8.51 and V_D = 16.87, however, the hysteresis holdup is quite large, because these data consist of runs taken to the region of loading and back again. Experimentally it was found that the free holdup remains the same for both the upper and lower portions of the hysteresis curve; it is actually the permanent holdup that undergoes hysteresis and produces this effect in the total holdup results. Below the region of loading, however, no hysteresis existed for variations of the dispersed-phase velocity at a constant continuous-phase flow rate.

Operational Holdup Conclusions

In order to discover which type of holdup constituted the active area of mass transfer and to obtain an insight into Wicks's operational holdup, a series of runs was performed on both a tagged and an isotope-free toluene phase. Initially, the tagged toluene phase was taken to loading and then returned to a continuous-phase flow rate of zero; i.e., a hysteresis cycle was executed. The flow of the tagged toluene was stopped, and the flow of the isotope-free toluene was simultaneously started.

Such an operating procedure disclosed the quantity of toluene droplets which had a net movement through the packing. Figure 7 illustrates the results of such an operation performed with ⅝-in. Raschig rings. The "normal" 100% mark on the ordinate axis denotes the total holdup of the dispersed phase corresponding to the lower portion, or base line, of the hysteresis curve. The other horizontal line designates the total holdup corresponding to the upper portions of the hysteresis curve.

The results reveal that during operation on the lower portion of the total-holdup hysteresis curve all the total holdup has a net movement through the packing. On the upper portion, however, only the holdup corresponding to the lower portion of the curve will be displaced. The hysteresis holdup, i.e., the difference between the upper and lower portions of the curve, is permanently trapped in the packing.

Entrance Effects

During the course of this investigation the column was completely scanned vertically as well as horizontally, and thus were permitted the determination of vertical variations of the holdup of the dispersed phase (Figure 8). As this run was performed at a rather large continuous-phase flow rate, entrance effects were quite pronounced.

In the region of the top of the packing there is an appreciable increase in holdup. This increase is due to back-circulating toluene droplets. In Figure 1 it will be noted that the water phase enters the column via two water-inlet pipes located about 3 in. from the top of the packing. At higher water flow rates the downward thrust of the incoming phase causes many of the rising toluene droplets to back circulate into the packing. For this column height and for the given flow rates, Figure 8 reveals that there is over a 100% increase in holdup in a region constituting about 15% of the packed height. For larger packed heights this region will, of course, represent a smaller percentage of the packed height of the column.

CORRELATION OF HOLDUP DATA

The primary criteria used in developing equations suitable for correlating the various holdup effects were simplicity of ultimate use and the interrelation of any coefficients for the three types of holdup reported. Accordingly, the following empirical equations were used to correlate the data. It should be kept in mind that the equations developed are for the specific packings used, for flow conditions below loading, for the system toluene-water, and for the specific mode of tower operation and packing herein reported.

Permanent Holdup

The permanent holdup in the region below loading can be represented by

$$X_P = A_1 + B_1(V_C) \qquad (3)$$

where X_P = permanent holdup and V_C = continuous-phase flow rate, cu. ft./(hr.)(sq. ft.)

Total Holdup

The experimental data on total holdup have been correlated by

$$X_{T0} = A_1 + C_1(V_D) \qquad (4)$$

where X_{T_0} = the total holdup at zero continuous-phase flow rate and

$$X_T = A_1 + B_1(V_C) + C_1(V_D) \qquad (5)$$

where X_T = the total holdup of the dispersed phase. Equation (5) was found to represent about 95% of the total holdup within the column, and an additional term was required to account for the remaining 5%. Accordingly, an *additional* term was added to Equation (5) to account for all the total holdup:

$$X_T = A_1 + B_1(V_C) + C_1(V_D) + D_1(V_C)(V_D) \qquad (6)$$

The free holdup present within the packing is obtained as the difference between Equations (6) and (3):

$$X_F = C_1(V_D) + D_1(V_C)(V_D) \qquad (7)$$

where X_F = the free holdup.

Holdup with Hysteresis

The equations for correlating the hysteresis holdup were obtained in an analogous manner on the assumption that the permanent holdup of the dispersed phase was defined by the maximum continuous-phase velocity employed during the continuous operation of the packed column. The equations obtained were

$$X_P{}^* = A_1 + B_1(V_{Cm}) \qquad (8)$$

where $X_P{}^*$ = the permanent holdup corresponding to the upper portion of the hysteresis curve and V_{Cm} = the maximum continuous-phase flow rate that has been employed in the continuous operational history of the column.

The total holdup, with hysteresis, is given by

$$X_T{}^* = X_P{}^* + X_F \qquad (9)$$

Substitution of Equations (7) and (8) into (9) yields

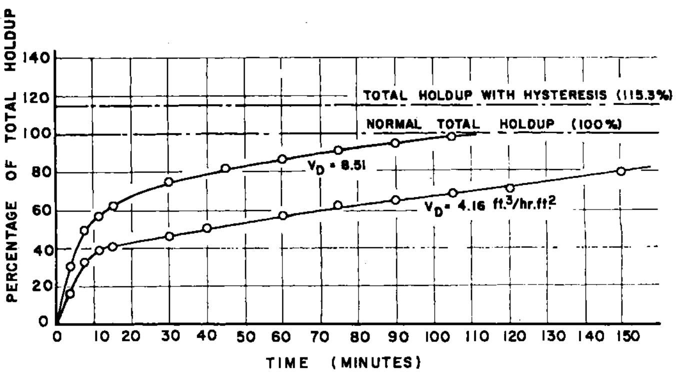


Fig. 7. Relative displacement of dispersed-phase holdup for 5/8-in. rings at zero continuous-phase flow.

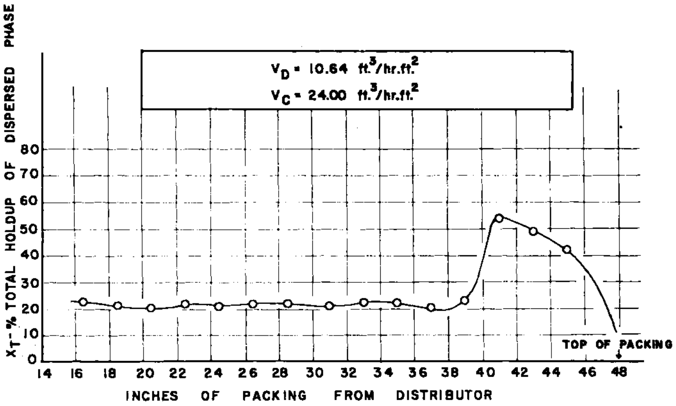


Fig. 8. Vertical variation of dispersed-phase holdup for 5/8-in. Raschig rings: V_D = 10.64 cu. ft./(hr.)(sq. ft.), V_C = 24.00 cu. ft./(hr.)(sq. ft.).

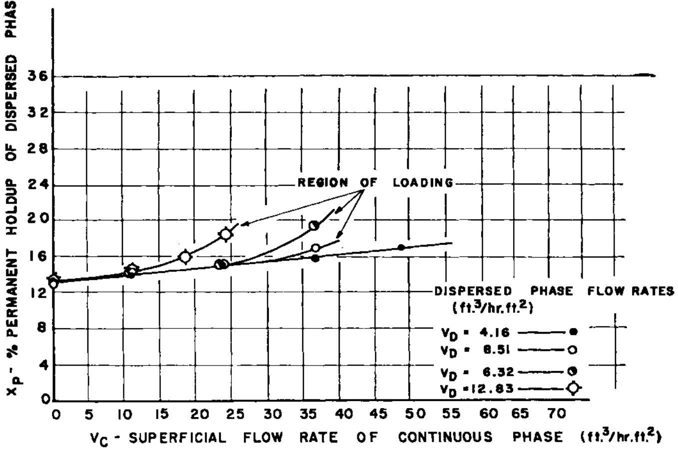


Fig. 9. Percentage of permanent holdup for 5/8-in. Raschig rings.

$$X_T^* = A_1 + B_1(V_{C_m}) + C_1(V_D) + D_1(V_C)(V_D) \qquad (10)$$

Table 2 presents the values of the coefficients A_1, B_1, C_1, and D_1 obtained from the experimental data. It was generally found that the equations presented permitted the calculation of the holdup to within 8% of the observed data with the exception of the data in the vicinity of loading.

SUMMARY

This investigation involved the study of the effect of flow rates and packing size upon the holdup of a toluene-dispersed phase flowing countercurrent to a continuous water phase in packed columns. Four different packings were used: ½-, ⅝-, ¾-, and 1-in. nonporous, unglazed Raschig rings, in an experimental column of 6 in. I.D. The results of the investigation are as follows:

Consistent data were obtained for three different types of holdup: permanent, total, and hysteresis. The permanent holdup includes the dispersed phase droplets, which do not rise freely to the water face. The total holdup is the total volume of dispersed phase within the effective packing volume at any time. The hysteresis holdup is the volume of dispersed phase permanently trapped in the packing as a consequence of executing a hysteresis cycle with variations of the continuous-phase velocity. This holdup represents the difference between the upper and lower portions of a total-holdup hysteresis curve.

The holdup data below the loading point have been correlated by the equations

$$X_P = A_1 + B_1(V_C)$$

$$X_T = A_1 + B_1(V_C) + C_1(V_D) + D_1(V_C)(V_D)$$

$$X_P^* = A_1 + B_1(V_{C_m})$$

Tabulated values for the coefficients are given.

When operating procedure is defined by the lower portion of the hysteresis curve, all the dispersed-phase droplets, free and permanent, have a net movement through the column. All the permanent holdup droplets are normally freed during the course of operation by impact from succeeding droplets. Hysteresis holdup, however, represents permanently trapped solvent droplets that have not net movement through the column.

Channeling, i.e., maldistribution of the dispersed phase occurs when the ratio of column diameters to packing size is 8.0 or less.

The effect of packing size on holdup is in agreement with the findings of previous investigators (*3*, *4*, *7*). The holdup of the dispersed phase increases as the packing size decreases.

Two distinct zones of flow were found to occur. The increase in holdup with increasing continuous- and dispersed-phase velocities was different in each zone. In the zone below loading, the holdup was found to increase linearly with the dispersed-phase flow at a constant continuous-phase flow rate. In addition, the increase of holdup with continuous-phase flow was approximately linear at a constant dispersed-phase flow rate in the region below loading.

Table 2

6 in. I.D.
Packed height: 48 in.

⅝-in. rings
Void fraction: 0.680

Run	Flow rates, cu. ft./(hr.)(sq. ft.) Toluene in	Flow rates, cu. ft./(hr.)(sq. ft.) Water in	Stream temperature °F. Toluene inlet	Stream temperature °F. Water inlet	Holdup data, vol. % of total voids Permanent	Holdup data, vol. % of total voids Total
2–1	4.16	0.0	91	87	14.83	16.17
–2	4.16	11.50	92	90	13.88	17.18
–3	4.16	24.00	92	94	14.92	18.26
–4	4.16	36.40	93	97	15.75	19.35
–5	4.16	48.00	92	98	16.79	21.75
–6	4.16	24.00	93	99	16.12	18.01
–7	4.16	11.50	94	100	—	17.33
–8	4.16	0.0	96	95	16.08	17.67
–9	6.32	0.0	91	88	12.65	16.05
–10	6.32	11.50	91	91	13.98	17.52
–11	6.32	24.00	93	94	14.66	19.58
–12	6.32	36.40	94	97	16.88	22.40
–13*	6.32	48.00	94	98	18.32	29.56
–14	6.32	24.00	95	100	16.49	22.38
–15	6.32	11.50	93	100	—	21.86
–16	6.32	0.0	95	95	17.70	20.75
–17	8.51	0.0	85	87	13.28	18.25
–18	8.51	11.50	86	87	14.38	19.33
–19	8.51	24.00	86	90	15.01	21.09
–20*	8.51	36.40	91	91	19.38	57.83
–21	8.51	24.00	90	93	18.71	25.13
–22	8.51	11.50	92	95	18.06	22.53
–23	8.51	0.0	92	92	17.06	22.30
–24	12.83	0.0	89	87	13.33	21.41
–25	12.83	4.90	89	88	13.49	22.02
–26	12.83	11.50	91	92	14.84	22.73
–27	12.83	18.00	93	94	15.80	24.34
–28	12.83	24.00	93	95	18.32	27.46
–29*	12.83	30.25	94	97	16.93	58.44
–30	12.83	24.00	94	99	18.44	29.46
–31	12.83	11.50	94	100	19.30	26.91
–32	12.83	0.0	94	95	16.71	24.50

*Flow rates in vicinity or in excess of loading-point velocities.

NOTATION

A_1 = coefficient in holdup correlation
B_1 = coefficient in holdup correlation
C_1 = coefficient in holdup correlation
D_1 = coefficient in holdup correlation
V = superficial liquid velocity, cu. ft./(hr.)(sq. ft. of column cross section)
X = fractional holdup of the dispersed phase in that portion of the column volume which may be occupied by liquid

Subscripts

C = continuous phase
D = dispersed phase
F = free
P = permanent
T = total

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