Experimental Methods for Measuring Static Liquid Holdup in Packed Columns

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Introduction

The observed influence of hydrodynamics on mass transfer operations in packed columns has led to numerous efforts directed at understanding nonideal trickle flow over solid packing. Shulman et al. (1955) first pointed out that the total liquid holdup in a packed column could be divided into static and dynamic portions. Total liquid holdup could be determined by the difference between the dry and irrigated column weight. Dynamic holdup could be found by collecting the liquid draining from the column after interruption of input liquid; static holdup was then determined by the difference between total and dynamic holdups.

This framework for treating nonideal flow in packed columns has been used to describe the influence of hydrodynamics on mass transfer. Shulman et al. found that the ratio of evaporation to physical absorption volumetric mass transfer coefficients, different by up to 300%, could be correlated to within 15% by the relative proportions of total and dynamic holdup:

$$\frac{(k_G a)_{\text{vap}}}{(k_G a)_{\text{phys}}} = 0.85 \frac{h_t}{h_d} \tag{1}$$

They concluded that the static holdup is relatively ineffective for physical absorption and fast chemical absorption because it quickly becomes saturated. However, these same stagnant regions are fully effective for evaporation and moderately paced chemical absorption. Baldi and Sicardi (1975) have shown that a similar relationship holds for the data of Joosten and Dankwerts (1973):

$$\frac{(k_L a)_{\text{chem}}}{(k_L a)_{\text{phys}}} = \frac{h_t}{h_d} \tag{2}$$

Here, the ratio of volumetric mass transfer coefficients for moderately paced chemical absorption to physical absorption follows the ratio of the total to the dynamic holdup.

The use of this relationship between holdups to correlate otherwise vastly different mass transfer coefficients illustrates the importance of knowing the fraction of total liquid holdup (and, hence, interfacial area) that is useful for mass transfer. Recognizing this, Puranik and Vogelpohl (1974) employed literature physical and chemical absorption data to develop general correlations for static and dynamic interfacial areas. In addition, Patwardhan (1978) has proposed an extended crossflow model to describe the relationship that exists between concentrations in the static and dynamic holdups for physical absorption and different types of chemical absorption.

The importance of quantifying the fraction of the total liquid holdup that is dynamic or static has led to a number of efforts to measure these parameters. One of the more interesting approaches suggested is that of Bennett and Goodridge (1970), who described the use of step decreases in tracer concentration for determining holdups. This method involves applying a step decrease in tracer concentration to the column inlet and measuring the outlet response. Total and static holdups can be obtained by integration of the appropriate portions of the response curve. The slope of the linear tail of the response curve can be used to determine the rate of interchange between the static and dynamic regions (Patwardhan and Shrotri, 1981). Unlike methods based on column draining, the input liquid is not stopped, so that column hydrodynamics are not disturbed.

In addition to the step decrease in tracer method of Bennett and Goodridge, several methods of estimating the amount of total and static holdups under irrigated conditions from impulse tracer data have been proposed. Van Swaaij et al. (1969) have shown that total holdups determined by impulse tracer and by weighing are equivalent. This implies that all the liquid holdup is accessible to the tracer. However, when static holdups are required, it can be argued that impulse tracer methods are not as versatile as the step decrease in tracer approach. For instance,

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the impulse tracer method of Hoogendoorn and Lips (1965) requires the assumption that the dynamic holdup is in plug flow, while the method of Ruszkay (1963) requires data accurate enough to calculate the first three moments of the response curve.

The concepts of static and dynamic holdup have been widely used for assessing and modeling packed column performance. Yet, although both draining methods and tracer techniques have been suggested for static and dynamic holdup determination, no critical comparison of these methods has been made. Shulman et al. assumed that the amount of liquid remaining in the column after draining is equal to the static holdup under irrigated conditions. If this is the case, tracer techniques should yield equivalent amounts of static holdup.

Experimental Work

The step decrease in tracer method of Bennett and Goodridge was used to determine the amounts of total, dynamic, and static holdups in a 0.05 m column packed with 0.0065 m ceramic Intalox saddles (Norton Chemical Co.). This small packing size was chosen because it was expected to have relatively large static holdups, Figure 1. The step decrease in tracer method was used for several reasons. Unlike methods in which draining is used, there is no upset in column hydrodynamics due to interruption in liquid flow. Also, this method can be used to assess the rate of interchange between the static and dynamic holdups.

Step decrease in salt tracer experiments were performed using the apparatus shown in Figure 2. A Beckman RA6X conductivity meter was used to monitor salt concentrations at the column outlet. This meter, along with two flow-through conductivity cells, was used to monitor conductivity changes over five orders of magnitude. The capability to monitor continuously the decay in outlet liquid conductivity throughout the course of the experiment avoided the need to extrapolate the data to determine holdups.

Step decrease experiments were done as follows. The column was filled with salt solution and then irrigated for at least 30 min at a liquid flow rate of about 10 $kg/m^2 \cdot s$. The liquid rate was then reduced to the desired rate. After at least one hour at this rate, the salt solution was replaced with deionized water by means of a three-way valve. The outlet water temperature and conductivity were sampled every 5 s by a data acquisition system built around an Apple II$^+$ microcomputer. Conductivities were corrected for any temperature changes that occurred during the run. The response curve (in normalized concentration units) for a representative run is shown in Figure 3.

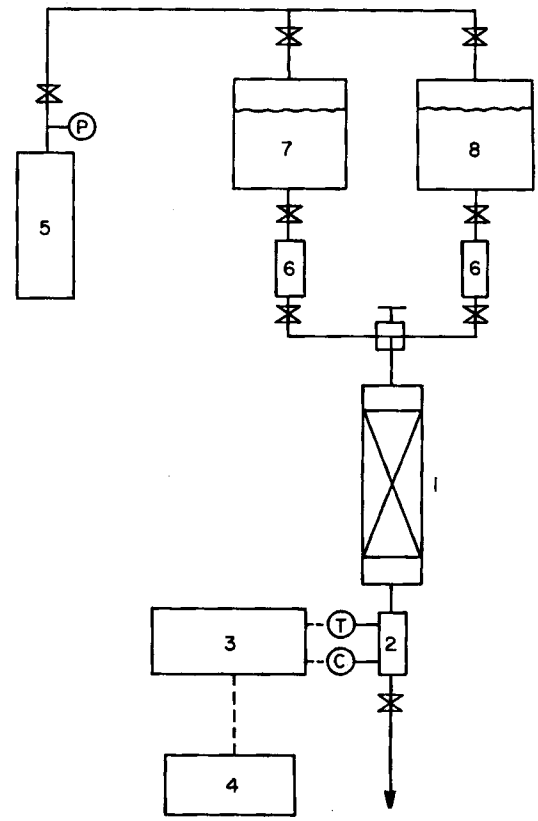

Figure 2. Diagram of experimental apparatus.

1. Column packed with 0.0065 m ceramic Intalox saddles
2. Temperature and conductivity transducers
3. Multiplexer
4. Microcomputer
5. Nitrogen bottle
6. Rotameter
7. Water tank
8. Water and KCl tank

Liquid delivered to column by pressurizing holding tanks with nitrogen.

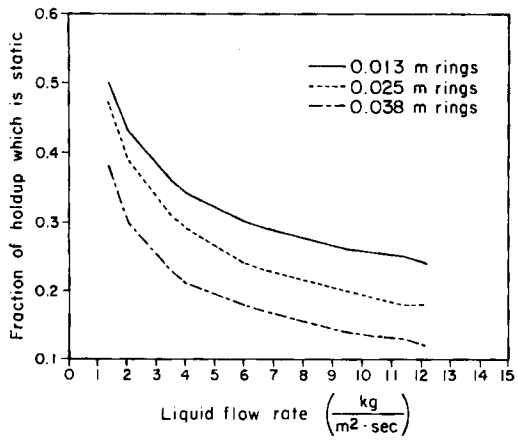

Figure 1. Static fraction of total holdup.

Data of Shulman et al. (1955). Ceramic Berl saddles show a similar trend.

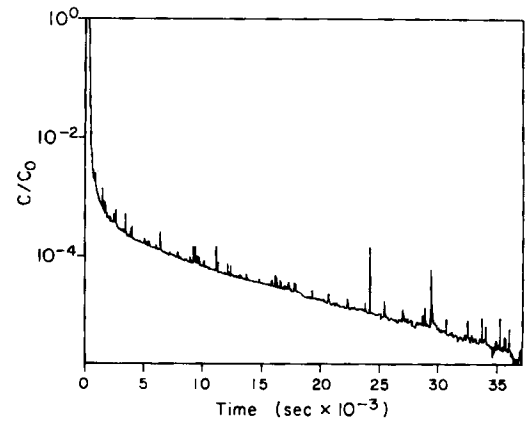

Figure 3. Sample response of packed column to step decrease in tracer.

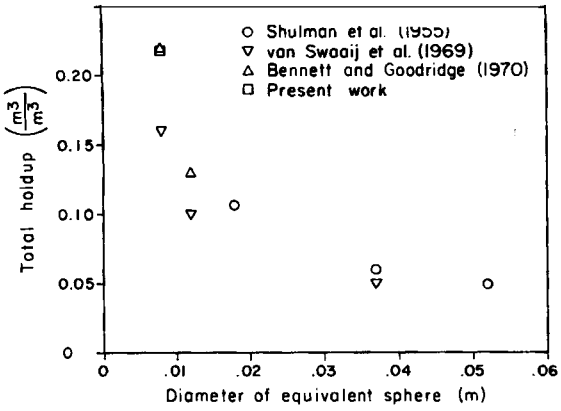

Figure 4. Total liquid holdup vs. packing size.
Liquid flow rate = 6 kg/m² · s.
Dia. of equivalent sphere = dia. of sphere with surface area equal to that of a piece of packing.

In order to determine if residual salt was left in the column after the experiment, the column was filled with water and the conductivity of the drainage determined. No significant amount of additional salt was found.

Experimental runs to determine total liquid holdup were done by both step decrease and impulse tracer methods. The results from both approaches always agreed to within 3% and the total holdups were directly proportional to column height for several packing levels. Also, results for total holdup were consistent with other workers, Figure 4.

Figure 5 shows total and dynamic holdups for this packing for a wide range of liquid flow rates as determined from step-decrease experiments. The difference between these two holdups for a particular liquid flow rate is the static holdup. The amount of static holdup is much smaller than expected given the earlier results of Shulman et al. with other packings using the draining method, Figure 1. This was pursued further through draining experiments. Dynamic holdups that were determined by draining were subtracted from total holdups determined by impulse and step decrease tracer. The resulting static holdups were significantly larger than those determined by step decreases.

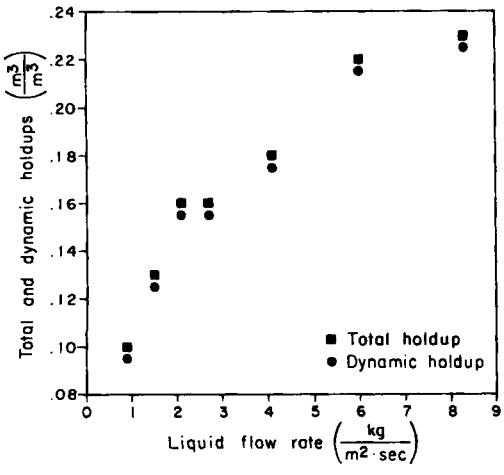

Figure 5. Total and dynamic liquid holdups vs. liquid flow rate for 0.0065 m Intalox saddles.

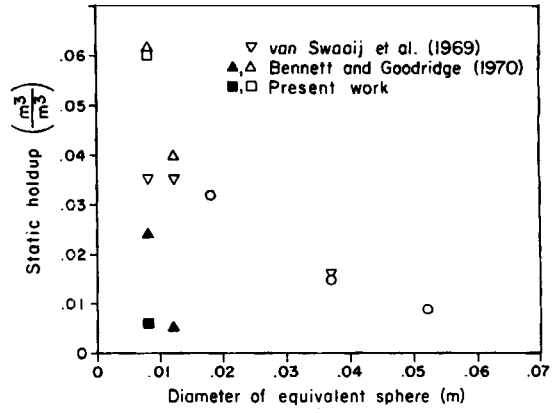

Figure 6. Static holdups determined by draining and step-decrease methods.
Open symbols: draining method data.
Filled symbols: step decrease tracer method data.
Holdups are independent of liquid flow rate.

Discussion

Figure 6 shows reported static holdups, measured by draining and step decrease methods, for a variety of packings. Our data and the data of Bennett and Goodridge indicate that, in general, there is substantial disagreement between static holdups determined by draining and step decrease in tracer methods. Given that no significant amount of tracer remained in the column, and that van Swaaij et al. have shown that all the liquid is accessible to the tracer, we conclude that a large fraction of the liquid retained in the packing after draining is actually well irrigated during operation. The data of Bennett and Goodridge support this conclusion. The difference between static holdups determined by draining and tracer methods is probably the result of liquid sweeping through these regions. It is doubtful that the amount of static holdup observed under irrigated conditions is large enough to account completely for the considerable differences in mass transfer coefficients mentioned above.

There is no doubt that the use of dynamic and static holdups for describing differences in packed column operations provides a framework for addressing the complex interaction of mass transfer and hydrodynamics. The results presented here suggest, however, that a more sophisticated approach will be necessary if a mechanistically correct description of this relationship is to be established. We intend to further explore the nature of these differences through tracer and absorption experiments.

Acknowledgment

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Notation

a = gas-liquid interfacial area per unit bed volume
h = liquid holdup per unit bed volume
k = local mass transfer coefficient

Subscripts

chem = chemical absorption
d = dynamic holdup
G = gas phase
L = liquid phase
phys = physical absorption

t = total holdup
vap = vaporization

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