

Liquid Holdup in Packed Beds at Low Mass Flux

Arno de Klerk

Fischer-Tropsch Refinery Catalysis, Sasol Technology Research and Development, Sasolburg 1947, South Africa

Liquid holdup is an important hydrodynamic parameter in the characterization of packed beds. Although it has enjoyed considerable attention in the literature, data at low mass flux ($< 0.5 \text{ kg} \cdot \text{m}^{-2} \cdot \text{s}^{-1}$) are less common. This region, although not relevant on a commercial scale, is of importance in down-scaling commercial processes for laboratory evaluations.

The existence of multiple hydrodynamic states at low mass flux has already been described by Kan and Greenfield (1978) and later also by Levec et al. (1986, 1988) and Christensen et al. (1986). This not only complicates the prediction of liquid holdup in the low mass flux region, but also the choice of a suitable liquid holdup correlation. Kan and Greenfield (1979) evaluated a number of correlations from the literature and found the correlation of Specchia and Baldi (1977) to be the most suitable for the low mass flux region. However, this evaluation predated most of the more recent work, like that of Sáez and Carbonell (1985), Ellman et al. (1990), Wammes et al. (1991a), and Holub et al. (1992, 1993), as well as that more concerned with high-pressure hydrodynamic evaluations, such as studies by Larachi et al. (1991), Wammes et al. (1991b), Al-Dahhan and Dudukovic (1994), and Attou et al. (1999).

Hydrodynamic data from the literature spanning four decades has been collated by Iluita et al. (1998) and was used to improve the predictive capabilities of Holub's phenomenological slit-model. More recently, Larachi et al. (2000) used this database to evaluate a number of models and empirical correlations from the literature. They concluded that the models performed almost equally well on liquid holdup prediction, with average errors of estimation ranging between 12 to 25%. No specific mention was made of performance in the low mass flux region.

Liquid holdup

Liquid holdup and liquid saturation are both used in the description of liquid retention in packed beds. Liquid holdup refers to the liquid volume per total volume, while liquid saturation refers to the liquid volume per void volume. In a packed bed, the liquid holdup divided by the bed porosity would therefore yield the liquid saturation.

The description of liquid holdup is further refined by the following terms:

(1) Internal liquid holdup is the liquid holdup contained inside a porous particle in the packed bed. For nonporous particles there will be no internal liquid holdup.

(2) External liquid holdup is the liquid holdup not contained in particles in the packed bed. For nonporous particles the external liquid holdup will be the same as the total liquid holdup.

(3) Residual liquid holdup is the part of the external liquid holdup that remains in the packed bed after the packed bed was completely wetted and then drained.

(4) Dynamic (or free-draining or operative) liquid holdup is the part of the external liquid holdup that collects at the bottom of the column after a sudden shutoff of the liquid feed.

(5) Static liquid holdup is the internal liquid holdup plus the residual liquid holdup. For a bed of nonporous particles, static holdup and residual holdup are the same.

Al-Dahhan et al. (1997) list five limiting cases to describe the effect of gas flow rate and pressure on liquid holdup. This gives a thorough description of the forces affecting liquid holdup, but for modeling purposes only three categories are used to describe dynamic liquid holdup: no gas flow (free-trickling liquid), low gas-liquid interaction regime, and high gas-liquid interaction regime. Residual liquid holdup is a function of the liquid phase and packed-bed properties only.

Experimental

Dynamic liquid holdup

Apparatus. The packed bed was contained in a vertical column provided with shutoff valves at the inlet and outlet, as well as valves for draining purposes. Gas and liquid flow were mixed prior to being introduced on top of the column. The liquid flow rate was controlled by a precalibrated positive-displacement pump (Spectrachrom P240). The gas flow rate was controlled by a metering needle valve at constant pressure drop and the gas flow rate was measured with a dry-gas flow meter (Elster special range $0.01\text{--}10 \text{ m}^3 \cdot \text{h}^{-1}$).

Packed Beds. All experiments were conducted with glass beads 3 mm in diameter ($2.98 \text{ mm} \pm 0.02 \text{ mm}$). Two different internal column diameters were used for the packed beds: 49.3 mm (bed voidage of 0.36) and 26.6 mm (bed voidage of 0.37).

Fluids. All experiments were done with tap water (conductivity $0.2 \text{ mS} \cdot \text{cm}^{-1}$) and nitrogen (99.99% pure).

Procedure. The experiments were preceded by a leak test at an elevated pressure. The packed bed was prewetted by filling the column with water and then draining it. The fol-

Table 1. Experimental Data on Dynamic Liquid Holdup

Set	Liquid-Phase Mass Flux (kg/m ² ·s)	Gas-Phase Mass Flux (kg/m ² ·s)	Column Dia. (mm)	Dynamic Liquid Holdup	
				<i>x</i>	<i>s</i>
A	0.10	0.03	49.3	0.029	0.003
	0.10	0.04		0.018	0.002
	0.10	0.08		0.014	0.003
	0.10	0.17		0.019	0.002
B	0.17	0.03	49.3	0.040	0.001
	0.17	0.04		0.027	0.002
	0.17	0.07		0.022	0.002
	0.17	0.17		0.027	0.003
C	0.23	0.03	49.3	0.046	0.001
	0.23	0.04		0.031	0.001
	0.23	0.08		0.029	0.001
	0.23	0.19		0.034	0.003
D	0.29	0.03	49.3	0.054	0.004
	0.29	0.04		0.037	0.001
	0.29	0.08		0.034	0.002
	0.29	0.17		0.035	0.002
E	0.35	0.03	49.3	0.063	0.002
	0.35	0.04		0.041	0.001
	0.35	0.08		0.037	0.001
	0.35	0.21		0.035	0.004
F	0.04	0.04	49.3	0.011	0.000
	0.09	0.08		0.015	0.001
	0.13	0.13		0.023	0.001
G	0.15	0.14	26.6	0.027	0.002
	0.30	0.29		0.037	0.002
	0.45	0.44		0.046	0.002

lowing steps were repeated for every experiment: (a) the liquid and gas flow rates were set and time was allowed for the system to stabilize; (b) the shutoff valves on the inlet and outlet of the column were closed simultaneously; (c) the liquid in the column was drained through the drain valves at the bottom of the column (5 min were allowed for draining) and its weight noted. Every experiment was repeated four times, but not in a clustered fashion. All experiments were done at ambient conditions (25°C ± 5°C and 85 kPa ± 1 kPa).

Residual liquid holdup

Apparatus. Glass columns of different diameters were used, and each was fitted with a wire mesh at the bottom to retain the packed bed.

Procedure. The columns were packed to a height of 500 mm with the same glass beads that were used for the dynamic liquid holdup experiments. The columns were filled with water and drained. The mass of the water used to fill the columns and the mass of water that was drained were both noted. Experiments were done in duplicate.

Results

Dynamic liquid holdup

Seven sets of experiments were done. In the first five sets, the gas-to-liquid mass flux ratio was changed, while in the last two sets the gas-to-liquid mass flux ratio was kept constant at a 1:1 ratio (see Table 1). Although the sample stan-

Table 2. Experimental Data on Residual Liquid Holdup

Column Dia. (mm)	<i>D/d</i>	Bed Porosity	Residual Liquid Holdup
21.9	7.3	0.40	0.053
27.7	9.3	0.37	0.040
33.7	11.3	0.37	0.039
39.7	13.3	0.36	0.036
44.5	14.9	0.36	0.036
53.1	17.8	0.36	0.032
57.8	19.4	0.36	0.029

dard deviations of most repeat experiments were below 10%, the data sets still had a fair amount of scatter.

Residual liquid holdup

The residual liquid holdup was determined for a range of column diameters that overlapped the column diameters used during the dynamic liquid holdup experiments (see Table 2). The values of repeat experiments did not differ by more than 10%.

Discussion

Dynamic liquid holdup

The experimental work focused on the low mass flux region ($< 0.5 \text{ kg} \cdot \text{m}^{-2} \cdot \text{s}^{-1}$). At low mass flux the cocurrent downflow of a gas-liquid mixture in a packed bed is gener-

ally in the trickle-flow (gas continuous or low interaction) regime. However, this qualitative assessment was verified against the flow-regime transition data of various authors presented by Gianetto et al. (1978) and the three criteria for trickle flow presented by Holub et al. (1992, 1993). In all instances, it was concluded that the experiments were done in the trickle-flow regime.

The average bed porosity was used as an indication of the presence of wall effects, and from Table 2 it can be seen that the average bed porosity remains constant when $D/d > 13$. Wall effects therefore can be neglected, except for data set G in Table 1.

The correlations and models presented in the literature for liquid holdup in the low interaction regime with nonfoaming liquids were evaluated against the present experimental data (see Table 3). The correlations of Charpentier and Favier (1975), Midoux et al. (1976), and Ellman et al. (1990) were also tested, but were either outside their range of validity, or gave very high estimation errors.

It was interesting to note that the recommendation by Kan and Greenfield (1979) to use the Specchia and Baldi (1977) correlation for the low mass flux region, still holds true. A similar performance was found with the model of Sáez and Carbonell (1985). The correlations of Larkins et al. (1961) and Wammes et al. (1991a) also fared well, except in the very low liquid mass flux region (sets A and F). However, since the model of Sáez and Carbonell has a sound theoretical basis, it is considered to be the method of choice.

Since the model equations of Sáez et al. (1985) and Holub et al. (1992) are similar in form (see Eq. 1), the relatively poor performance of the latter was surprising and could not be explained otherwise than by the choice of correlating constants.

$$P^Q \cdot f_L - \frac{\rho_G}{\rho_L} \cdot \left(1 - \frac{\epsilon_L}{\epsilon}\right)^R \cdot f_G - 1 + S \cdot \frac{\rho_G}{\rho_L} = 0 \quad (1)$$

with

$$f_\lambda = \left[E_1 \cdot \frac{Re_\lambda}{Ga_\lambda} + E_2 \cdot \frac{Re_\lambda^2}{Ga_\lambda} \right]$$

	Sáez et al. (1985)	Holub et al. (1992)
P	$(\epsilon_L - \epsilon_{L,r})/(\epsilon - \epsilon_{L,r})$	ϵ_L/ϵ
Q	2.43	3
R	4.8	3
S	0	1

Residual liquid holdup

An extensive investigation into residual liquid saturation was presented by Dombrowski and Brownell (1954). Residual liquid saturation was correlated in terms of bed permeability and liquid properties (density, surface tension, and contact angle). It was noted that residual liquid saturation has a limiting value of 0.075 (or 0.05 when expressed as liquid holdup). The modified Charpentier-type correlation for liquid holdup presented by Sáez and Carbonell (1985) makes use of the Eötvös number as a correlating parameter and has the same limiting value.

As in the present study, Wammes et al. (1991a) used 3-mm glass spheres in a water–nitrogen system. They obtained residual liquid saturation values on the order of 0.035–0.050 for a packed bed with a porosity of 0.39. For a similar system with a bed porosity of 0.375–0.385, Levec et al. (1986) reported a residual liquid holdup of 0.022. This low value was explained in terms of the possible poor wetting properties of the glass used (Wammes et al., 1991a).

No specific precautions were taken to ensure that the glass beads used in the present study had good wettability. The values in Table 2 nevertheless compare well with those presented by Wammes et al., although the liquid holdup at lower bed porosity tended to be somewhat lower. However, despite proper prior wetting of the bed, irrigation under dynamic conditions was probably uneven (Sie, 1991).

Conclusions

The present work was aimed at laboratory test work and cannot necessarily be extrapolated to commercial scale. Evaluation of the present dynamic liquid holdup experimental work against models and correlations from the literature, indicated that those of Specchia and Baldi (1977) and Sáez and Carbonell (1985) perform the best in the low mass flux region. The comparatively poor performance of the model by Holub et al. (1992), which is of a similar form to that of Sáez

Table 3. Liquid Holdup Estimation Errors with Present Experimental Data (%)

	Larkins et al. (1961)	Turpin and Huntington (1967)		Specchia and Baldi (1977)	Sáez and Carbonell (1985)	Wammes et al. (1991a)	Holub et al. (1992)
		Turpin data	Larkins data				
A	47	190	74	21	27	50	125
B	26	122	50	21	16	28	79
C	21	79	41	23	15	19	62
D	16	66	31	24	15	17	53
E	15	56	25	22	16	19	51
F	62	208	82	7	28	67	156
G	38	30	23	24	20	12	56
Average	32	107	47	20	19	30	83

et al. (1985), could not be explained otherwise than by the choice of correlating constants.

Acknowledgments

The author gratefully acknowledges Sasol Technology Research and Development for the use of their facilities, René Kriel and Daniella Vassiloudis for their tireless effort in the laboratory and Ntombikayise Motloung for the residual holdup data.

Notation

D = column diameter, m
 d = particle diameter, m
 E_1, E_2 = constants in the Ergun (1952) pressure drop equation
 g = gravitational acceleration, $\text{m} \cdot \text{s}^{-2}$
 Ga = modified Galileo number $[\rho^2 g d^3 \epsilon^3] / [\mu^2 (1 - \epsilon)^3]$
 Re = modified Reynolds number $[\rho d u] / [\mu (1 - \epsilon)]$
 s = sample standard deviation
 u = velocity $\text{m} \cdot \text{s}^{-1}$
 x = average value

Greek letters

ϵ = bed porosity
 ϵ_L = total liquid holdup
 $\epsilon_{L,r}$ = residual liquid holdup
 λ = subscript denoting the phase, either G for gas or L for liquid
 μ = viscosity, $\text{Pa} \cdot \text{s}$
 ρ_G = density of the gas phase, $\text{kg} \cdot \text{m}^{-3}$
 ρ_L = density of the liquid phase, $\text{kg} \cdot \text{m}^{-3}$

Literature Cited

- Al-Dahhan, M. H., and M. P. Dudukovic, "Pressure Drop and Liquid Holdup in High Pressure Trickle-Bed Reactors," *Chem. Eng. Sci.*, **49**, 5681 (1994).
- Al-Dahhan, M. H., M. H. Larachi, M. P. Dudukovic, and M. P. Laurent, "High-Pressure Trickle-Bed Reactors: A Review," *Ind. Eng. Chem. Res.*, **36**(8), 3292 (1997).
- Attou, A., C. Boyer, and G. Ferschneider, "Modelling of the Hydrodynamics of the Cocurrent Gas-Liquid Trickle Flow Through a Trickle-Bed Reactor," *Chem. Eng. Sci.*, **54**, 785 (1999).
- Charpentier, J. C., and M. Favier, "Some Liquid Holdup Experimental Data in Trickle-Bed Reactors for Foaming and Nonfoaming Hydrocarbons," *AIChE J.*, **21**, 1213 (1975).
- Christensen, G., S. J. McGovern, and S. Sundaresan, "Studies on Trickle-Bed Hydrodynamics: Multiple Hydrodynamic States in the Trickle Regime," *AIChE J.*, **32**, 1677 (1986).
- Dombrowski, H. S., and L. E. Brownell, "Residual Equilibrium Saturation of Porous Media," *Ind. Eng. Chem.*, **46**(6), 1207 (1954).
- Ellman, M. J., N. Midoux, G. Wild, A. Laurent, and J. C. Charpentier, "A New, Improved Liquid Hold-Up Correlation for Trickle-Bed Reactors," *Chem. Eng. Sci.*, **45**, 1677 (1990).
- Ergun, S., "Fluid Flow Through Packed Columns," *Chem. Eng. Prog.*, **48**, 89 (1952).
- Gianetto, A., G. Baldi, V. Specchia, and S. Sicardi, "Hydrodynamics and Solid-Liquid Contacting Effectiveness in Trickle-Bed Reactors," *AIChE J.*, **24**(6), 1087 (1978).
- Holub, R. A., M. P. Dudukovic, and P. A. Ramachandran, "A Phenomenological Model for Pressure Drop, Liquid Holdup, and Flow Regime Transition in Gas-Liquid Trickle Flow," *Chem. Eng. Sci.*, **47**, 2343 (1992).
- Holub, R. A., M. P. Dudukovic, and P. A. Ramachandran, "Pressure Drop, Liquid Holdup, and Flow Regime Transition in Trickle Flow," *AIChE J.*, **39**(2), 302 (1993).
- Iliuta, I., F. Larachi, and B. P. A. Grandjean, "Pressure Drop and Liquid Holdup in Trickle Flow Reactors: Improved Ergun Constants and Slip Correlations for the Slit Model," *Ind. Eng. Chem. Res.*, **37**, 4542 (1998).
- Kan, K. M., and P. F. Greenfield, "Multiple Hydrodynamic States in Cocurrent Two-Phase Downflow Through Packed Beds," *Ind. Eng. Chem. Proc. Des. Dev.*, **17**, 482 (1978).
- Kan, K. M., and P. F. Greenfield, "Pressure Drop and Holdup in Two-Phase Cocurrent Trickle Flows Through Beds of Small Packings," *Ind. Eng. Chem. Proc. Des. Dev.*, **18**, 740 (1979).
- Larachi, F., A. Laurent, N. Midoux, and G. Wild, "Liquid Saturation Data in Trickle Beds Operating Under Elevated Pressure," *AIChE J.*, **37**(7), 1109 (1991).
- Larachi, F., I. Iliuta, M. H. Al-Dahhan, and M. P. Dudukovic, "Discriminating Trickle-Flow Hydrodynamic Models: Some Recommendations," *Ind. Eng. Chem. Res.*, **39**, 554 (2000).
- Larkins, R. P., R. R. White, and D. W. Jeffrey, "Two-Phase Concurrent Flow in Packed Beds," *AIChE J.*, **7**, 231 (1961).
- Levec, J., A. E. Sáez, and R. G. Carbonell, "The Hydrodynamics of Trickle Flow in Packed Beds. Part II: Experimental Observations," *AIChE J.*, **32**, 369 (1986).
- Levec, J., K. Grosser, and R. G. Carbonell, "The Hysteretic Behaviour of Pressure Drop and Liquid Hold-Up in Trickle-Beds," *AIChE J.*, **34**, 1027 (1988).
- Midoux, N., M. Favier, and J. C. Charpentier, "Flow Pattern, Pressure Loss and Liquid Holdup Data in Gas-Liquid Downflow Packed Beds with Foaming and Nonfoaming Hydrocarbons," *J. Chem. Eng. Jpn.*, **9**, 350 (1976).
- Sáez, A. E., and R. G. Carbonell, "Hydrodynamic Parameters for Gas-Liquid Cocurrent Flow in Packed Beds," *AIChE J.*, **31**(1), 52 (1985).
- Sie, S. T., "Scale Effects in Laboratory and Pilot-Plant Reactors for Trickle-Flow Processes," *Rev. Inst. Fr. Pet.*, **46**(4), 501 (1991).
- Specchia, V., and G. Baldi, "Pressure Drop and Liquid Holdup for Two Phase Concurrent Flow in Packed Beds," *Chem. Eng. Sci.*, **32**, 515 (1977).
- Turpin, J. L., and R. L. Huntington, "Prediction of Pressure Drop for Two-Phase, Two-Component Concurrent Flow in Packed Beds," *AIChE J.*, **13**, 1196 (1967).
- Wammes, W. J. A., S. J. Mechelsen, and K. R. Westerterp, "The Influence of Pressure on the Liquid Hold-Up in a Cocurrent Gas-Liquid Trickle-Bed Reactor Operating at Low Gas Velocities," *Chem. Eng. Sci.*, **46**(2), 409 (1991a).
- Wammes, W. J. A., J. Middelkamp, W. J. Huisman, C. M. deBaas, and K. R. Westerterp, "Hydrodynamics in a Cocurrent Gas-Liquid Trickle Bed at Elevated Pressures," *AIChE J.*, **37**, 1849 (1991b).

Manuscript received May 22, 2002, and revision received Nov. 13, 2002.