Optical Probe Technique for Two-Phase Flow Measurements in an Extraction Column

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Introduction

Accurately predicting the dispersed phase droplet behavior in liquid-liquid two-phase flow continues to complicate the design and scaling-up of extraction columns. Although many methods have been developed to measure the drop velocity, drop size distribution and holdup of the dispersed phase, the design and scaling-up of extraction columns continues to rely heavily on pilot testing rather than model prediction. Among these measurement techniques, the laser scattering¹ and photographing² methods can only be applied to nonlight systems. Fluorescence³ and sampling⁴ methods can destroy the multiphase flow field, ultrasonic technology⁵ cannot measure the drop velocity and size distribution, and resistance probe⁶ can only be applied to systems with a conductive dispersed phase and insulated continuous phase. A new type of inline measurement technology, the optical probe, has been used to measure local dispersed phase holdup, bubble/drop size distribution and bubble/drop velocity of many multiphase systems including gas-liquid,^{7,8} gas-liquid-liquid,⁹ and gas-liquid-solid¹⁰ systems. The differences in physical properties of liquid-liquid phase systems, however, are too small to detect by conventional measurement technologies and few results using the optical probe technique10-12 have been published. Furthermore, for simplification many extraction column experiments assume drop velocity is equal to drop terminal velocity, neglecting the influence of pulse energy input (pulse amplitude and frequency) and flow rates on drop velocity. In this experiment, the optical probe technique was used in a pulsed-sieve-plate extraction column (PSEC) to calculate the drop velocity and size distribution of a liquid-liquid phase system. The influences of energy input and flow rates of the two-phases on drop size and distribution were taken into account and a correlation to calculate the Sauter mean diameter of droplets was developed based on the results.

Experimental Setup

The PSEC body was a glass cylinder 2 m in effective height and 150 mm in diameter with 25 mm plate spacing. Teflon plates were inserted into the column every fourth plate to enhance the mass transfer performance by the interface renewal effect. 13 The experimental system was 30% TBP (in kerosene)nitric acid with water as the continuous phase. The holdup of the dispersed phase ϕ , was measured using the volumetric replacement method. The refractive indices of the continuous and dispersed phases at 25°C are 1.34 and 1.43, respectively. The pulse energy input to the PSEC varied with pulse amplitude A ranging from 0.5-2.5 cm, and a pulse frequency f ranging from $1.0-2.0 \text{ s}^{-1}$. The real drop velocity and drop size distribution of the dispersed phase drops were measured using a dual optical probe, which contained two separate optical fibers with conical tips. The difference in refractive indices of the two liquids allowed the probe tips to differentiate between the values of light intensity reflected from the two passing phases. The drop velocity and size distribution were obtained by comparing the optical signals reflected from the two fibers. The experimental setup is shown in Figure 1. The optical probe was located between the first and second stainless steel plates (counting from the lower Teflon plate). In a previous experiment, 14 we confirmed that d_{32} in this region can be used to represent d_{32} along the entire column. The diameter of the optical fiber was 62.5 µm. Two infrared laser beams were passed through the two separate optical fibers, reflected at the conical tips, and collected to form electronic signals.

Measurement Principle of Dual Opticoptical Probe

Figure 2 is a typical voltage output signal curve of the dual optical probe when the same drop passes across the two fibers separately.

The top and bottom curves are the signals from fibers I and II, respectively. The two optical fibers, when in contact with the continuous phase, have high voltage signal values until a dispersed phase drop passes across the conical tip leading to a

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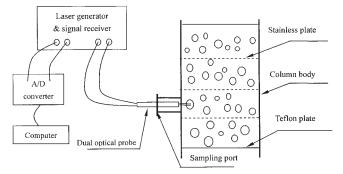


Figure 1. Experimental setup of infrared laser.

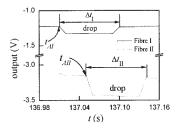


Figure 2. Signals of dual optical probe (A = 1.0 cm, $f = 1.0 \text{ s}^{-1}$, $u_x = 0.00133 \text{ m/s}$, $u_y = 0.00133 \text{ m/s}$).

decrease in the voltage output level as seen in Figure 2. When the fiber tip is again immerged in the aqueous phase, the voltage output signal recovers to the initial high level. Figure 3 shows a drop crossing the tip of fiber I at time t_{AI} and fiber II at time t_{AII} , creating a similar voltage output curve for both signals with delay of arrival time $(t_{AII}-t_{AI})$. The drop velocity is obtained using the distance between the two fibers, H, and the delay of arrival time $(t_{AII}-t_{AI})_d$, as shown in Eq. 1 and the cut chord of

$$u_d = \frac{H}{(t_{AII} - t_A)_d} \tag{1}$$

$$d_c = u_d \Delta t_I \tag{2}$$

the drop d_c , is calculated using the obtained velocity and Eq. 2.

Results and Discussion

Energy input and flow rate influence on droplet velocities in a PSEC

Figure 3 shows the influence of flow rates on drop velocity under constant 1.0 cm pulse amplitude and $1.0~\rm s^{-1}$ pulse frequency.

The signal curves of fibers I and II are very similar except for the time delay. The time delay values, $(t_{AII} - t_{AI})_d$, of droplets with different sizes were similar in duration at constant pulse energy input, consistent with the conclusions made by Kentish et al.¹⁵ Figure 4 shows the influence of the energy input on drop velocity under the same flow rates.

The values of $(t_{AII} - t_{AI})_d$ of droplets with different sizes at different energy input conditions were not the same, and droplet velocities were more sensitive to changes in pulse energy input than flow rates. From the experimental results, the rela-

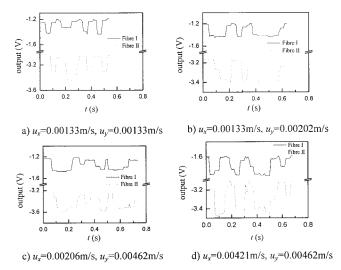


Figure 3. Influence of flow rates on signal curves (A = 1.0 cm, $f = 1.0 \text{ s}^{-1}$).

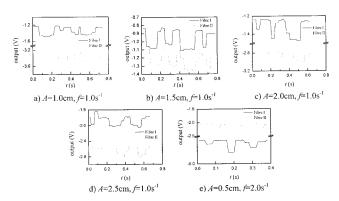


Figure 4. Influence of varying pulse amplitude and frequency on signal output curves ($u_x = 0.00206$ m/s, $u_y = 0.00462$ m/s).

tionship between energy input and time delay, was expressed as

and the drop velocities calculated using Eqs. 1 and 3 were expressed as

$$u_d = \begin{cases} 0.050 \text{ m/s} & Af < 2.0 \text{ cm/s} \\ 0.025 \text{ m/s} & Af \ge 2.0 \text{ cm/s} \end{cases}$$
 (4)

Energy input influence on drop size distribution and mean diameter

The cut chord d_c of a droplet crossing the dual optical probe was calculated using Eqs. 1–4. The calculation of drop diameter d, was simplified by assuming d_c is equal to d. Figure 5 shows the influence of energy input on drop size distribution. The drop size distribution became narrower and drop size decreased with the increase of pulse energy input. Drop size decreased more significantly with decreased pulse frequency

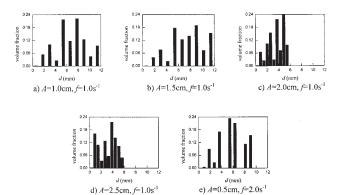


Figure 5. Influence of energy input on drop size distribution ($u_x = 0.00133 \text{ m/s}, u_y = 0.00462 \text{ m/s}$).

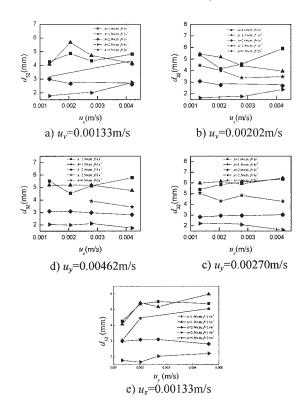


Figure 6. Influence of phase flow rates on Sauter mean diameter, d_{32} .

than pulse amplitude. The influence of phase flow rates on Sauter mean diameter d_{32} is shown in Figure 6.

Similar to drop size distribution, the Sauter mean diameter d32 decreased with the increase of pulse energy input, but again more significantly under higher pulse frequencies than pulse amplitudes. d32 slowly increased with the increase of superficial velocity of the dispersed phase, u_y at constant pulse energy and continuous phase superficial velocity, u_x . Varying the dispersed phase superficial velocity, u_x had a much smaller effect on d_{32} at constant pulse energy and dispersed phase superficial velocity. Taking into account both pulse input energy and phase flow rates Eq. 5 was derived from experimental results to calculate d_{32} , and shows a Sauter mean diameter dependence on Af^2 similar to Kumar and Hartland's 16 results.

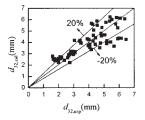


Figure 7. Comparison of $d_{\rm 32}$ between experimental data and calculated results.

In Figure 7, a comparison of the experimental results and predicted results using Eq. 5 for d_{32} showed an average deviation of $\pm 20\%$.

$$d_{32} = 6.5 \cdot \left(\frac{u_x}{1 - \phi} + \frac{u_y}{\phi}\right)^{0.72} / \sqrt{Af^2}$$
 (5)

Notation

A =pulse amplitude, cm

 d_{32} = Sauter mean diameter, mm

 $d_c = \text{cut chord, mm}$

 $f = \text{pulse frequency, s}^{-1}$

H = distance between two probes, m

 t_A = arrival time, s

 $u = \text{superficial flow rate, m} \cdot \text{s}^{-1}$

 $u_d = \text{drop velocity, m/s}$

 Δt = residence time of a drop passing through a single probe, s

 ϕ = holdup of the dispersed phase

Subscripts

cal = calculated data

d = drop

I = probe I

II = probe II

x = continuous phase

y =dispersed phase

exp = experimental data

Literature Cited

- Triballier K, Dumouchel C, Cousin J. A technical study on the Spraytec performances: influence of multiple light scattering and multimodal drop-size distribution measurements, *Experiments in Fluids*. 2003, 35:347–356.
- Bardin-Monnier N, Guiraud P, Gourdon C. Residence time distribution of droplets within discs and doughnuts pulsed extraction columns via Lagrangian experiments and simulations. *Chem. Eng. J.* 2003, 94:241–254.
- Weiss C, Kohler V, Marr R. Fluorescence capillary photometry for characterization of liquid-liquid dispersions. AIChE J. 1997, 43:1153– 1162.
- Steiner L, Balmelli M, Hartland S. Simulation of hydrodynamic performance of stirred extraction column. AIChE J. 1999, 45:257–267.
- Bonnet JC, Sorrentino JA. Holdup measurements in a Scheibel column by means of an ultrasonic technique. J. of Chem. Technol. and Biotechnol. 1994, 61:201–207.
- Bao X-J, Fan Z, Chen J-Y. The impedence method for dispersed phase holdup in solvent extractors. *Petroleum Processing and Petrochem. Eng.* 1994, 25:54–57 (in Chinese).
- Chabot J, Lee S-L-P, Soria A, Lasa HI. Interaction between bubbles and fibre optic probes in a bubble column. C. J. of Chem. Eng. 1992, 70:61–68
- Fordham E-J, Simonian S, Ramos R-T, Holmes A, Huang S-M, Lenn C-P. Multi-phase-fluid discrimination with local fibre-optical probes:

- II. Gas/liquid flows. *Measurements Sci. and Technol.* 1999, **10**:1338–1346.
- Fordham E-J, Simonian S, Ramos R-T, Holmes A, Huang S-M, Lenn C-P. Multi-phase discrimination with local fibre-optic probes: II. Three phase flows. *Measurements Sci. and Technol.* 1999b, 10:1347–1352.
- Wang T-F, Wang J-F, Yang W-G, Jin Y. Experimental study on bubble size distribution in there-phase circulating fluidized beds. J. of Chem. Industry and Eng. (China). 2001, 52:197–203.
- Fordhan E-J, Simonian S, Ramos R-T, Holmes A, Huang S-M, Lenn C-P. Multi-phase discrimination with local fibre-optic probes: I. Liquid/liquid flows. Measurement Sci. and Technol. 1999, 10:1329–1337.
- Hamad F-A, Pierscionek B-K, Bruun H-H. A dual optical probe for volume fraction, drop velocity and drop size measurements in liquidliquid two-phase flow. *Measurement Sci. and Technol.* 2000, 11:1307– 1318.
- Li H-B, Luo G-S, Wang J-D. Mass transfer performance in a coalescence-dispersion pulsed sieve plate extraction column. *Chem. Eng. J.* 2000, 78:225–229.
- Tang X-J. Mass transfer enhancement and modeling of coalescencedispersion pulsed-sieve-plate extraction column. PhD. Thesis, Tsinghua University, Beijing, 2004.
- Kentish S-E, Stevens G-W, Pratt HRC. Measurement of drop rise velocities within a Kuhni extraction column, *Industrial Eng. Chemistry Res.* 1997, 36:4928–4933.
- Kumar A, Hartland S. Prediction of drop size in pulsed perforatedplate extraction columns, *Chem. Eng. Communications*. 1986, 44:162– 182.

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