A Few New Findings on Phase Inversion in a Liquid/Liquid System

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Organic/aqueous systems are widely used in mixer/settler units to extract metals in solvent extraction processes. Usually a large organic to aqueous volume ratio is used to achieve a high throughput. It is a usual practice to operate the mixer/settler with the aqueous phase as the continuous phase and the organic as the dispersed phase, to achieve good separation efficiency in the downstream settlers. However, operating at a large organic to aqueous ratio can trigger a phase inversion, which reduces the separation efficiency. Thus, it is important to quantify the phase inversion boundary, and the effect of various parameters affecting the phase boundary.

Phase inversion describes the phenomenon of the spontaneous change from an organic phase dispersed in a continuous aqueous phase (denoted O/A) to an aqueous phase dispersed in a continuous organic phase (denoted A/O), or vice versa under certain physical or chemical conditions. The phase inversion is commonly characterized by plotting the critical organic phase volume fraction ϕ_0 at which phase inversion occurs against the agitation speed N (rpm) (refer to Figure 1). The organic volume fraction ϕ_0 is defined by

$$\Phi_0 = \frac{V_0}{V_T} \tag{1}$$

where V_0 is the volume of the organic phase and V_T is the total volume of the system (organic + aqueous). Every liquid/liquid system has a region in which the organic phase is continuous, a region in which the aqueous phase is continuous, and an ambivalent region where either phase can be continuous.

Although the topic of phase inversion has been researched by many authors in the past, it is still far from complete. Quinn and Sigloh (1963) studied phase inversion in a circular mixing tank using many combinations of immiscible liquids. Selker and Sleicher (1965) found that for a given liquid/liquid system there is a wide range of relative volumes in which either liquid could be in stable continuous phase. Rowden et al. (1975) noted that nonsteady mass transfer affected phase inversion in applications such as solvent extraction. They showed the presence of a large ambivalent region in a continuous liquid/liquid system as different from that in a batch mixer. Guilinger et al. (1988) studied phase inversion in a water/kerosene system. They found that in continuous operations, the residence time in the mixer and the feed fraction of the dispersed phase affect the phase inversion behavior. There have been a number of models and correlations proposed to relate phase boundaries and liquid physical properties (Quinn and Sigloh, 1963; Yeo et al., 2002), but there is little agreement between the proposed correlations.

This note reports on a few novel behaviors associated with the phase inversion process observed in an experimental study program. The study aimed at modeling an industrial plant phase inversion problem, where air bubbles and dynamical change in the agitation were known to exist.

The experiments were conducted in a circular tank of 390 mm diameter, fitted with four equally spaced baffles 32 mm wide. A six-bladed radial disc turbine of 150 mm diameter was installed 50 mm above the tank bottom. Both aqueous and organic phases streams were introduced from a feed pipe at the tank base. The impeller speed could be varied from 0 to 900 rpm using a variable-speed controller. Shell-Sol D70 solution, which is a solvent widely used in metal solvent extraction plants, was used as the organic phase and a mix of tap water and sodium chloride was used as the aqueous phase. The ionic strength obtained with the sodium chloride prevented the possible formation of emulsions. Phase inversion was monitored by a resistance probe connected to a digital multimeter.

To determine phase inversion boundaries, the volume ratio of the organic and aqueous was varied at constant agitator speeds until phase inversion was detected. A low resistance was detected in the case of an aqueous phase in a continuous

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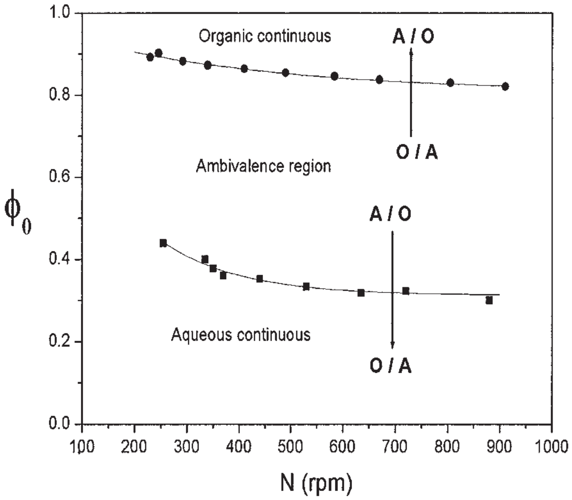

Figure 1. Phase inversion diagram of water/Shell-Sol D70 system.

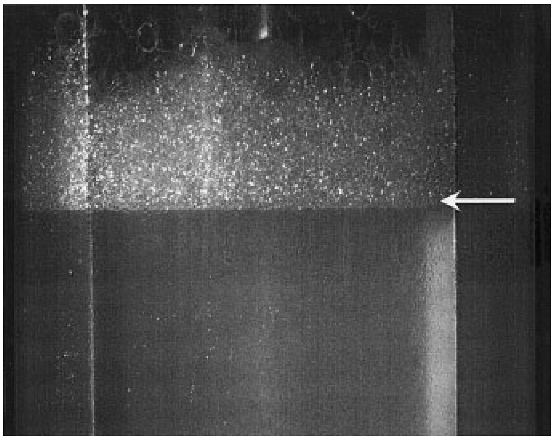

Figure 3. Clouds of droplets accumulating above the interface.

The agitator is switched off from the status of (O/A), that is, organic dispersed. and the aqueous in the continuous phase (O/A) (the interface line is indicated by the arrow).

phase (O/A), and an infinite resistance was detected when the organic phase became continuous (A/O). Although it was found that the resistance probe worked reliably, it was also possible to visually distinguish the difference between O/A and A/O shortly after the agitator was switched off. A horizontal organic/aqueous interface started to form as a result of gravitational force. Clouds of droplets could be seen accumulating below the interface in the case of an aqueous phase and the organic in the continuous phase (A/O) (Figure 2). As time progressed, droplet clouds below the interface line eventually collapsed. On the contrary, clouds of droplets were seen forming above the interface line after the agitator was switched off if the mixture was organic dispersed in an aqueous phase (O/A) (refer to Figure 3).

Figure 1 shows the phase diagram with the Shell-Sol D70/water system obtained for the standard operating conditions described by Quinn and Sigloh (1963) and Selker and Sleicher (1965). The lower curve corresponds to phase inversion from A/O to O/A. The upper curve, representing the inversion from O/A dispersion to A/O dispersion, shows that this inversion is less sensitive to impeller speed. In general, these two phase inversion curves vary with the agitator speed.

It was found that the start-up process of the agitator affected the phase dispersion status. To demonstrate this, the impeller was turned on abruptly to the test speed. The phase inversion upper curve, with the agitator started abruptly, is presented in Figure 4 (triangle symbols with dotted lines). It can be seen that the upper boundary curve shifted downward, estimated to be about 13% less than the standard condition, represented by the upper continuous curve (reproduced from Figure 1). This downward shift suggests that phase inversion became somewhat "easier" from O/A to A/O if the agitator was started abruptly. This can have implications for a full-scale industrial

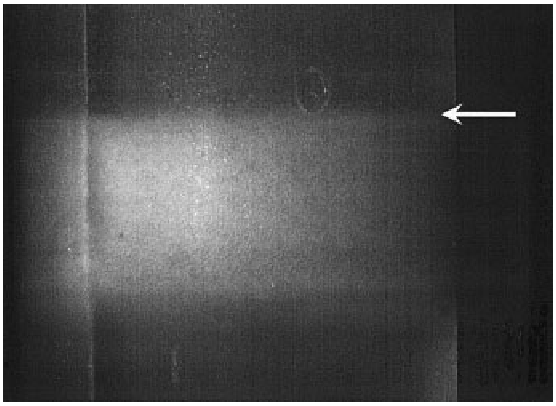

Figure 2. Clouds of droplets accumulating below the interface.

The agitator is switched off from the status of (A/O), that is, aqueous dispersed, and the organic in the continuous phase (A/O) (the interface line is indicated by the arrow).

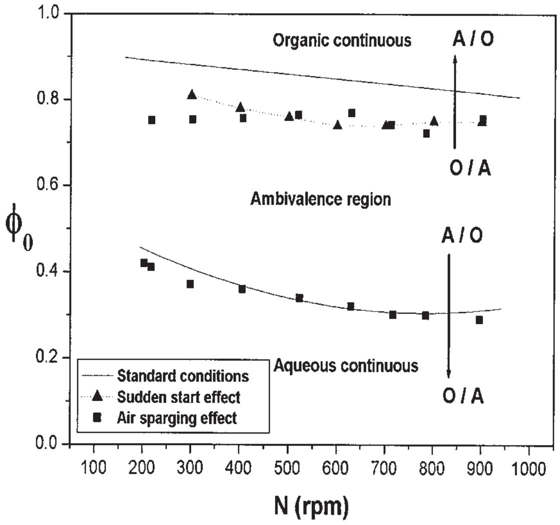

Figure 4. Effect of sudden agitator start and air bubbles on the phase boundary.

The lines correspond to the standard conditions reproduced from Figure 1; ▲ = agitator started abruptly; ■ = presence of fine air bubbles.

plant, where a sudden change in the agitation condition caused by flow instability could trigger phase inversion.

The effect of the presence of fine air bubbles was studied. Fine air bubbles were produced by injecting compressed air into a nozzle mounted in the feed pipe from the bottom of the tank. The nozzle broke the air bubbles into a fine size. One of the phase streams (O or A) with fine air bubbles injected was pumped into the tank filled with the other phase (A or O) from the tank bottom until the phase inversion was observed. The liquid was pumped at a flow rate Q_l of 34 L/min and the air sparged at a flow rate Q_g of 155 L/min. The ratio of the organic to aqueous phase at which phase inversion occurred is plotted in Figure 4 (square symbols). It can be seen that sparging air in a semibatch system did not affect the lower curve, the boundary of the A/O to O/A inversion. However, air sparging lowered the upper curve, indicating that a lower organic volume fraction was required for phase inversion from O/A to A/O when the dispersed phase was pumped. To correlate the experimental results with liquid and air flow rates for the phase inversion O/A to A/O boundary, a power law fitting of the form

$$\Phi_0 = aN^{-p(1-\varepsilon^k)} \tag{2}$$

was used for fitting the data, where ϕ_0 is the volume fraction O/A; N is the speed, ε is the gas flow rate to the liquid flow rate ratio (Q_g/Q_l); and a, p, and k are constants. The expression that best fits the results for both standard conditions case and air sparging case is

$$\Phi_0 = 1.25N^{-0.06(1-\varepsilon^{-0.11})} \tag{3}$$

It can be remarked that $\varepsilon = 0$ corresponds to the standard condition results presented by the upper curve in Figure 1 for the phase inversion from O/A to A/O.

In summary:

- A visual method to distinguish phase status is demonstrated. The phase status can be easily identified by visually inspecting the side at which the collapsing droplets are located as against the aqueous vs. organic interface line, once the agitation is turned off.
- Abrupt starting of agitation shifts the phase inversion upper curve downward, indicating that a transient flow condition affects the phase boundary.
- The presence of fine air bubbles also shifts the upper curve downward.

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