# ON THE FACTORS INFLUENCING THE HYDRODYNAMIC PERFORMANCE OF A PULSED SIEVE-PLATE EXTRACTION COLUMN: DISPERSED PHASE HOLDUP

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## Abstract

Dispersed phase holdup is studied in a low plate free area pulsed sieve-plate extraction column. The effects of pulse velocity (product of amplitude and frequency) and superficial phase velocities are studied for two liquid-liquid systems with widely different interfacial tensions. Dispersed phase holdup is found to be affected strongly with the change in pulse velocity and dispersed phase velocity. The inclusion of solute increases the dispersed phase holdup.

**Keywords:** Pulsed column, Hydrodynamics, Holdup, Pulse velocity, Propionic acid

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## 1. Introduction

The study of hydrodynamics of a liquid pulsed column is important in the design of a new liquid-liquid system with minimal of pilot scale testing [1]. The hydrodynamic behavior of the column is largely affected by the dispersed phase holdup. Dispersed phase holdup, defined as the volume fraction of the dispersed phase in dispersion, is an important property which directly affects interphase mass transfer as well as capacity of a column. It depends on a number of parameters such as superficial phase velocities, pulsation intensity, physical properties, and column geometry. Among the physical properties, one of the most important properties is the interfacial tension between the contacting phases. A number of researchers have studied the effect of operating parameters described above, however, the effect of interfacial tension on holdup is scarce. In the present study, dispersed phase holdup is studied for two binary liquid-liquid systems, kerosene-water system and *iso*-amylacetate-water system, having exceedingly different interfacial tensions. Propionic acid is used as a solute in kerosene-water system to see the effect of mass transfer on dispersed phase holdup. Most of the literature studies are carried out in columns with plate free area greater than 20%, the column studied in the present study has plate free area of only 13.5%. It is important to mention here that the study of dispersed phase holdup with water-*iso*-amylacetate and water-propanoic acid-kerosene is being reported for the first time in the literature. The effects of pulsation intensity, superficial phase velocities, interfacial tension, and addition of solute are studied.

## 2. Experimental

A photograph of the experimental system is shown in Fig. 1, while schematic diagram of the column is shown in Fig. 2. The laboratory pulsed column was 5 cm I.D. (internal diameter) glass column and had 80 number of plates. Each plate had 36 number of holes and the plate free area was 13.5%. Kerosene-water and *iso*-amylacetate-water were the two liquid-liquid systems studied for the dispersed phase holdup. Propanoic acid was used as a solute in kerosene-water system to see the effect of addition of solute on dispersed phase holdup. Kerosene and *iso*-amylacetate were of commercial grade, while propanoic acid was of analytical grade from Fischer Scientific$^{®}$ with ≥98% purity. Water available in the laboratory was employed without any purification. Physical properties of the chemicals employed are provided in Table 1.

**Table 1:** Relevant physical properties of the chemicals utilized

| Chemical | Density (kg/m$^3$) | Viscosity $\times 10^{-3}$ (Pa.s) | Interfacial tension with water (mN/m) |
|---|---|---|---|
| Kerosene | 783 | 1.67 | 44.0 |
| *Iso*-amylacetate | 865 | 0.87 | 11.5 |

In a typical procedure, initially whole of the column was filled with aqueous phase which behaved as continuous phase in each of the systems studied. Being heavier, aqueous
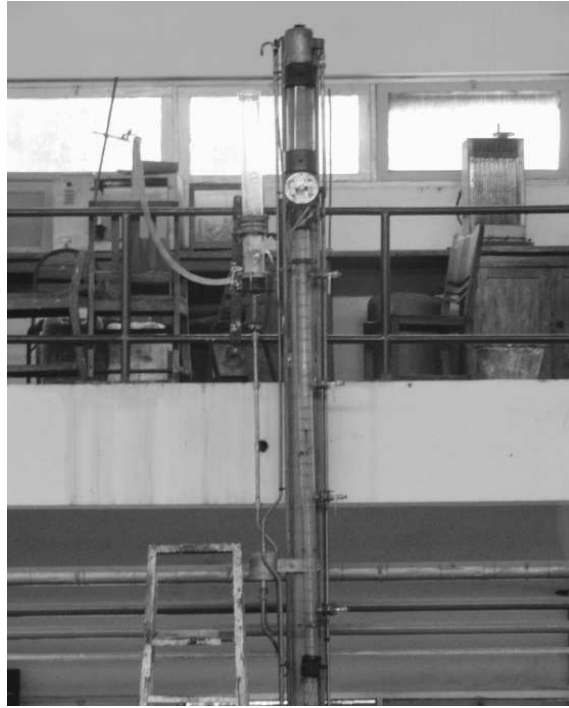
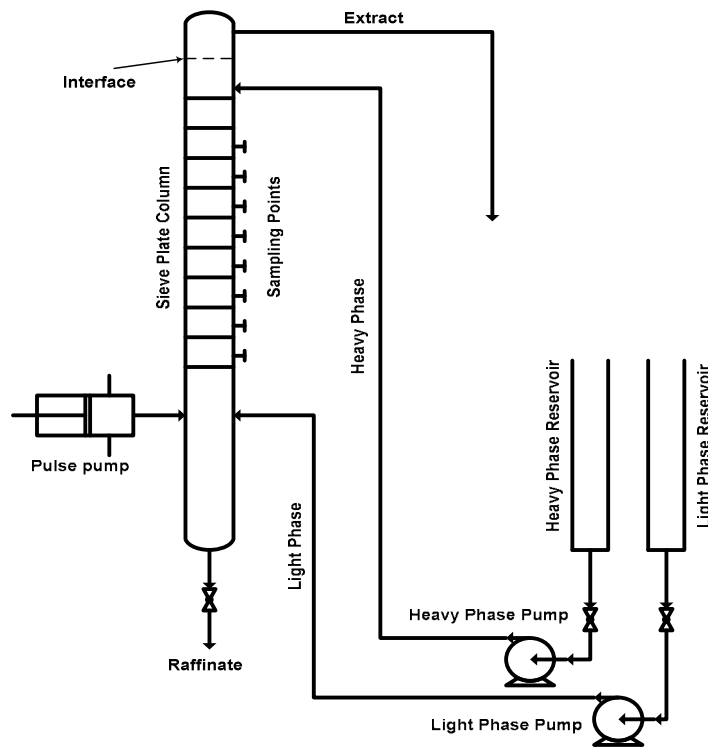**Figure 1:** Photograph of the laboratory sieve-plate extraction column



**Figure 2:** Schematic diagram for the laboratory sieve-plate extraction column

phase was allowed to flow from top to the bottom. The dispersed phase as lighter than water was then allowed to flow from the bottom to the top of the column in counter-current to the water flow. For measurement of dispersed phase holdup, sampling method [2] was adopted. There were eight sampling points in the column. 100 ml volume from each tap was taken and collected to settle for the two phases to be separated. Nearly 5 min were given between any two samplings so that each sample was taken under steady-state. In other words, a sample was taken from a tap and the column was operated for 5 more minutes and then the sample from other tap was collected. This procedure was followed for all eight samples under a constant set of pulsation intensity and phase flow rates. The volume fraction of the dispersed phase in each sample provided the value of dispersed phase holdup, here called plate dispersed phase holdup. The average of all these plate holdups was used to report the dispersed phase holdup by sampling method.

During the course of an experimental run an interface at the top was maintained as the dispersed phase was flowing from the bottom. This step was precautionary step to avoid flooding in the column. Flooding tremendously decreases efficiency of a column. Dispersed phase needs coalescence in the top reservoir before leaving the column. The interface was controlled manually by throttling the discharge valve of continuous phase flow. A wide range of experimental conditions were studied. Table 2 provides the ranges of the experimental conditions.

**Table 2:** Ranges of operating conditions for the present study

| Ranges of operating conditions | Values |
|---|---|
| $A \cdot f$ (m/s) | 3.01−32.40 |
| $u_d$ m/s) | 1.0−5.67 |
| $u_c$ (m/s) | 1.25−6.30 |
| $\gamma$ (mN/m) | 11.5−44.0 |

## 3. Results and discussion

### 3.1 Effect of pulsation intensity on the dispersed phase holdup

#### 3.1.1 No mass transfer

Fig. 3 shows the effect of pulse velocity ($A \cdot f$), defined as the product of amplitude and frequency, on the dispersed phase holdup for no mass transfer conditions. Two binary systems with different interfacial tensions are studied. In both cases, initially the holdup decreases with an increase in the pulse velocity, reaches a minimum value and then starts increasing with an increase in the pulse velocity. Before the point of minimum holdup, holdup decreases with pulse velocity while after this point holdup continues to increase till flooding occurs. The pulse velocity at the minimum holdup is characterized as

transition pulse velocity [3] and it is so called because at the point of minimum holdup, transition of mixer-settler region occurs to dispersion region. Before the transition point, mixer-settler region prevails while after the point of minimum holdup mixer-settler region is transformed into dispersion region. Sehmel and Babb [3], Bell and Babb [4], Lorenz et al. [5], and Venkatnarasaiah and Varma et al. [6] observed the same condition of minimum holdup in their study.
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It is observed that for each system with its own interfacial tension, there is a different value of transition velocity. Close observation of Fig. 3 reveals that the transition velocity is moving to the left with an increase in the interfacial tension.
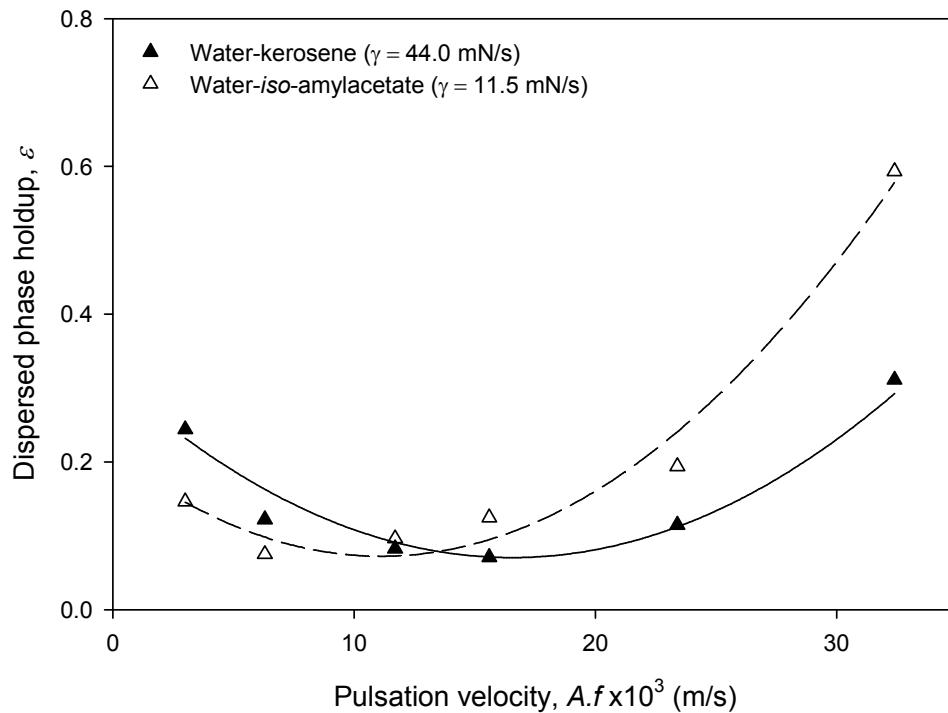


**Figure 3:** Effect of pulsation velocity on the dispersed phase holdup.
$$u_d = 2.73 \times 10^{-3}\, \text{m/s}, \ u_c = 2.86 \times 10^{-3}\, \text{m/s}.$$

The figure shows that not only the pulsation intensity but interfacial tension has a great effect on the performance of pulsed sieve-plate extraction column. It is observed that both in the dispersion and mixer-settler regions, holdup increases with a decrease in interfacial tension. However, comparing two systems of different interfacial tensions under identical conditions of operation, holdup of the system with lower interfacial tension may be less if the system with lower interfacial tension is in dispersion regime while the other is in mixer-settler regime.

The above observations may be explained in the following way. In a column while in operation, holdup of a particular system is a complex function of factors such as drop size, drop velocity, rates of drop coalescence and breakup, shear stress and drag on

drops, shape of drop, and circulatory motion of drop swarm. The above factors are written explicitly for the discussion purpose, the factors may depend on each other.

In the mixer-settler region (characterized as the formation of distinguished layer of dispersed phase beneath the sieve-plate when light phase dispersed as in the present study) for constant values of phase flows, due to low agitation, larger drop size is formed and accumulate below the sieve-plate and causes holdup to increase [7]. The phenomenon of drop accumulation was visually observed in the column. In the absence of not enough energy, the rate of drop coalescence may be greater than drop breakup and larger drops are formed which increase the holdup (in case accumulation of drops occurs). As the pulsation intensity is increased, more energy is added and drop size is reduced, and the discrete layer formed beneath the plate starts disappearing and holdup is reduced. Further increase in the pulsation intensity causes further agitation and the continuous phase may go into the dispersed phase drop body more readily increasing the density of the drop and lowering the drop velocity. A large number of drops of various diameters are formed and the column is packed with drops. The drops collide more frequently and frictional drag reduces the upward motion. Drops may start circulatory motion and accumulate in the cell. Although there is a greater chance of drop coalescence but high energy input destabilizes the drops and rate of drop breakup is also high. All these increase the dispersed phase holdup in the dispersion and emulsion regions.

The reason for moving the transition velocity to a lower value with a decrease in interfacial tension may be explained on the basis that drops are destabilized even at low energy input and mixer-settler region disappears earlier.

### 3.1.2 With mass transfer

Propionic acid also called propanoic acid in aqueous stream is used in the present study to see the effect of mass transfer on the dispersed phase holdup. Kerosene as solvent was used to see the effect of propionic acid extraction on the dispersed phase holdup. 10% by weight solution of propionic acid in water was prepared and behaved as continuous phase in the column. Dispersed phase holdup was measured mainly as a function of pulsation intensity and compared with no mass transfer condition discussed above.

Fig. 4 compares the effect of pulsation velocity on the dispersed phase holdups between mass transfer and no mass transfer conditions for water-propionic acid-kerosene system. It is observed from the figure that the dispersed phase holdup is comparatively low for the case of solute transfer than for the case of no solute transfer. The phenomenon may be explained on the basis of Maragoni effect that increases coalescence and therefore drop size is increased [6, 8]. The increase in drop size decreases the holdup for mass transfer. However, the interfacial tension of the system may also be decreased due to solute presence. This decrease in interfacial tension may decrease the drop size and reduce the dispersed phase holdup. The value of the dispersed phase holdup depends upon which phenomenon in above factors is dominant.
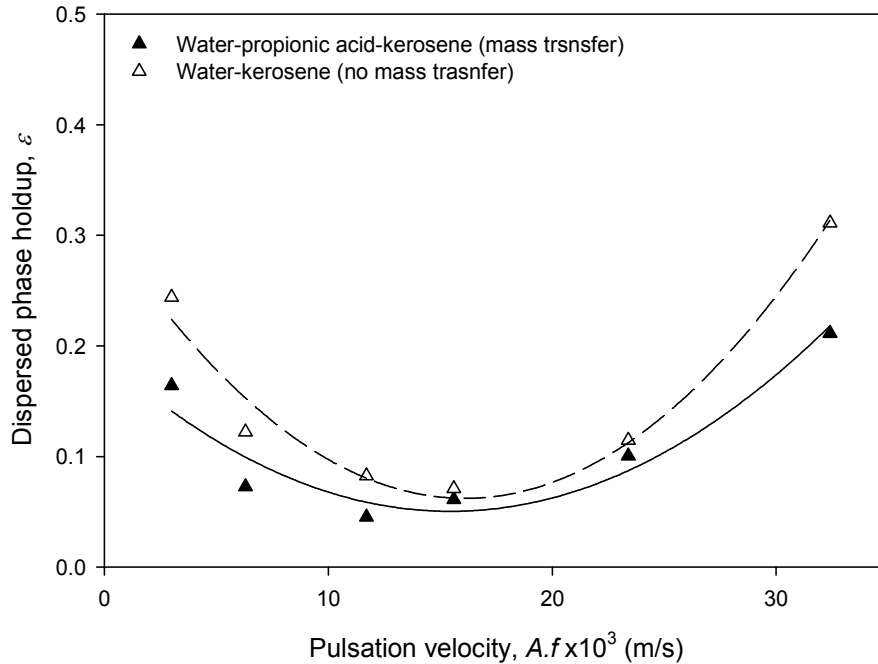
**Figure 4:** Comparison of the effect of pulsation velocity on the dispersed phase holdup with and without mass transfer for water-propionic acid-kerosene system.
$$u_d = 2.73 \times 10^{-3} \text{ and } u_c = 2.86 \times 10^{-3}.$$

## 3.2 Effect of phase velocities on the dispersed phase holdups

### 3.2.1 No mass transfer conditions

Fig 5 and Fig. 6 show the effect of phase velocities under no mass transfer conditions on the dispersed phase holdup for water-organic systems studied in the present work. It is observed that the effect of continuous phase velocity is negligible for kerosene-water system, but for the low interfacial tension system of *iso*-amylacetate-water system. For the *iso*-amylaceate-water system there is slight increase in dispersed phase holdup with continuous phase superficial velocity. In contrast to the above effects, holdup is increased with increase in the dispersed phase superficial velocity for both the systems studied. For the effect of dispersed phase velocity a number of investigators have supported the above claim. Cohen and Bayer [9], Sehmel and Babb [3], Miyauchi and Oya [10], Mishra and Dutt [11], Bell and Babb [4], Khemongkorn et al. [12], Lorenz et al. [5], and Venkatanarasaiah and Varma [6] observed substantial increase in holdup with increase in dispersed phase velocity for their conditions of experimentation.
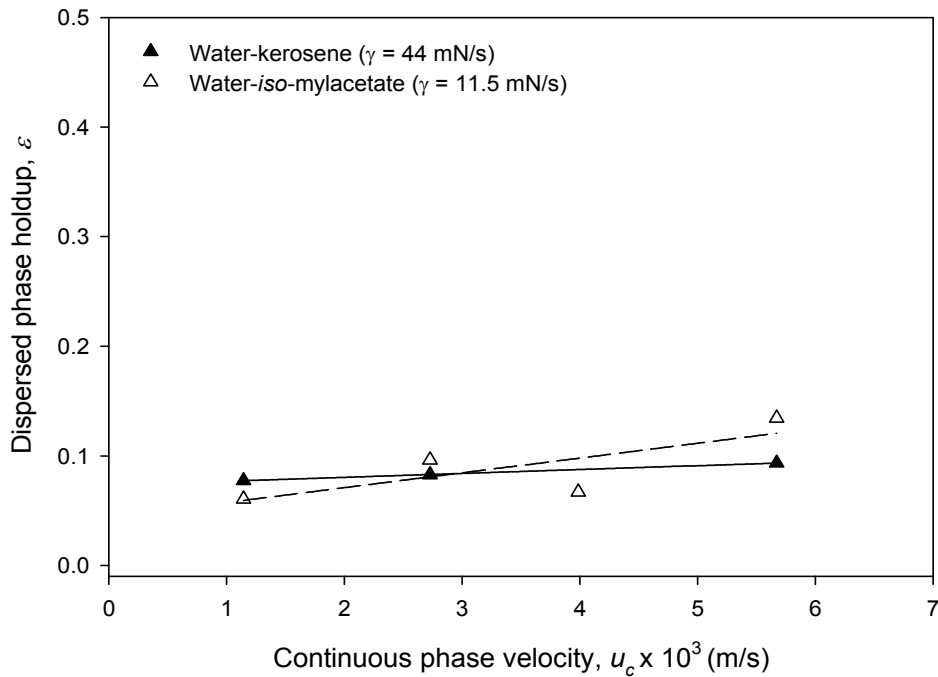
**Figure 5:** Effect of continuous phase superficial velocity on the dispersed phase holdup.
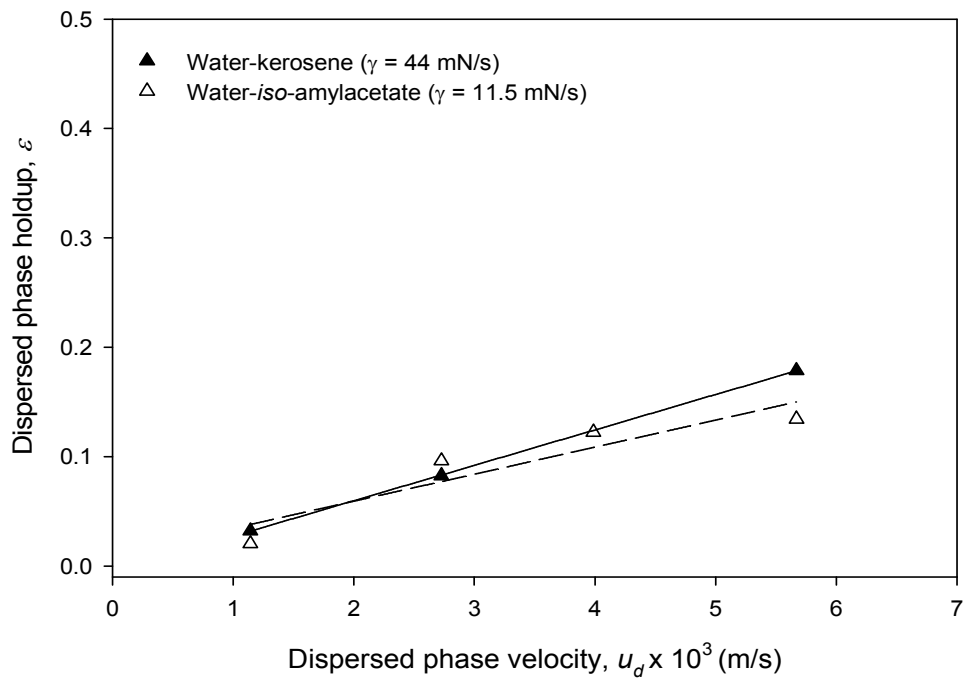$$A \cdot f = 11.7 \times 10^{-3}\,\text{m/s}, u_d = 2.73 \times 10^{-3}\,\text{m/s}.$$



**Figure 6:** Effect of dispersed phase superficial velocity on the dispersed phase holdup.
$$A \cdot f = 11.7 \times 10^{-3}\,\text{m/s}, u_c = 2.86 \times 10^{-3}\,\text{m/s}.$$

### 3.2.2 With mass transfer conditions

Fig. 7 shows a comparison for the effect of dispersed phase velocity on holdup between with mass transfer and without transfer conditions. Kerosene-water system is selected to observe the effect. Under same conditions of pulsation velocity and superficial continuous phase velocity, dispersed phase holdup is increased with an increase in dispersed phase velocity for both with and without propionic acid transfer. A higher slope of the line in the case for no mass transfer shows a greater rate of change of dispersed phase holdup with dispersed phase velocity. Similar to the effect of pulsation velocity, the dispersed phase holdup is higher with no mass transfer conditions. Same reasoning as described in Sections 3.1.1 and 3.1.2 may be described for understanding the above observation.
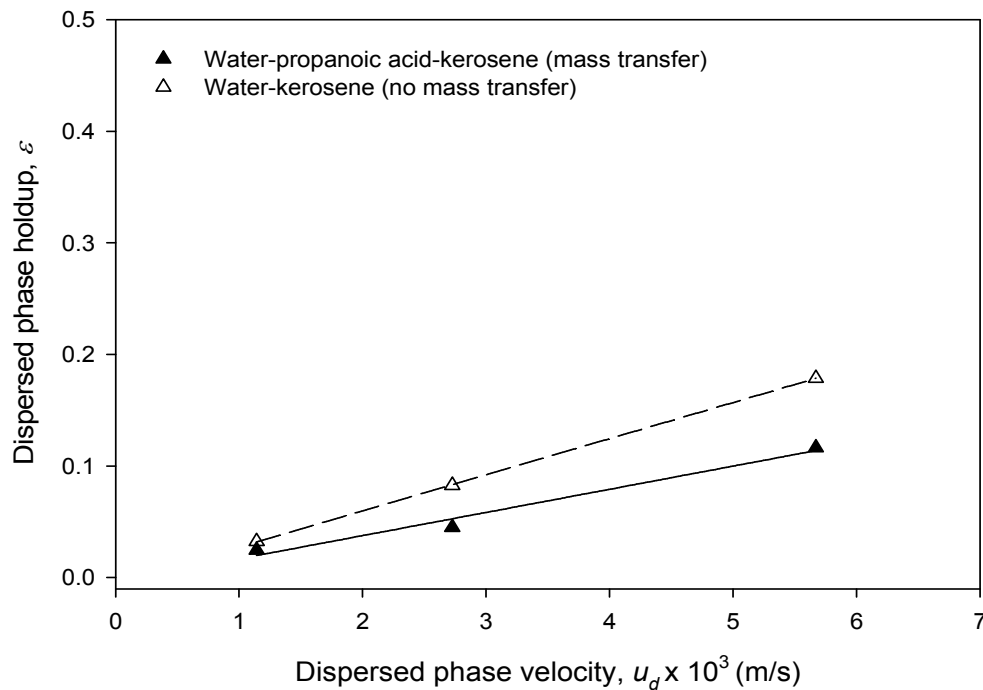


**Figure 7:** Comparison for the effect of dispersed phase superficial velocity on the dispersed phase holdup between with and without mass transfer

$$A \cdot f = 11.7 \times 10^{-3} \, \text{m/s}, \ u_c = 2.86 \times 10^{-3} \, \text{m/s}.$$

### Conclusions

- Dispersed phase holdup initially decreases with pulse velocity and after reaching a minimum holdup value (at transition velocity) it increases with an increase in pulse velocity. In other words, holdup decreases with pulse velocity in mixer-settler region while it increases in dispersion and emulsion regions of the operation of pulsed sieve-plate column. The point of minimum holdup is a function of the interfacial tension of the system. Lower the interfacial tension, lower will be the transition velocity at which minimum holdup appears.

- Dispersed phase holdup strongly increases with an increase in dispersed phase velocity, while it is virtually affected with continuous phase velocity for high interfacial tension system (kerosene-water).
- Addition of solute (propionic acid) decreases the dispersed phase holdup, however, trends of the effects of operating variables are similar to that without mass transfer.

**Nomenclature**

$A$      pulsation amplitude, m

$f$      pulsation frequency, Hz

$u_c$      superficial continuous phase velocity, m/s

$u_d$      superficial dispersed phase velocity, m/s

$\gamma$      interfacial tension, N/m

$\varepsilon$      dispersed phase holdup

**Acknowledgment**

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