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Comparison of normal phase operation and phase reversal studies in a pulsed sieve plate extraction column

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ABSTRACT

The hydrodynamic characteristics of a pulsed sieve plate extraction column (PSPEC) was studied experimentally using two different liquid phase systems, namely water/kerosene and 30%TBP (tributyl phosphate) in NPH (normal paraffin hydrocarbon)/0.3 M HNO_3. The aqueous phase as the dispersed phase and the organic phase as the continuous phase (phase reversal) and vice versa (normal phase operation) studies in a pulsed sieve plate extraction column 0.076 m in diameter and 1 m height are presented in this paper. The hydrodynamic properties like drop size and holdup are characterized as a function of various operating parameters namely pulse velocity, dispersed phase and continuous phase velocity and duty cycle of pulsing. Flooding in the column was also investigated for the changes involving flow ratio of continuous phase to that of the dispersed phase for both insufficient and excessive pulsing. It has been observed that phase reversal mode of operation is not efficient as compared to normal phase operation for the PSPEC.

Keywords: Pulsed sieve plate extraction column (PSPEC); Phase reversal; Holdup; Drop size; Flooding; Duty cycle

1. Introduction

Liquid–liquid extraction usage in industry for separation processes has been hampered by the poor extraction efficiencies associated with gravity-operated towers. The relatively small driving force for phase dispersion in this column is because of the small density difference between the two liquids. To aid phase dispersion an external means of agitation were provided, which evolved new class of liquid–liquid extractors i.e. pulse column introduced by Van Dijck in 1935. Various methods have been developed for mechanically agitating extractor column for increasing degree of turbulence which in turn increases rate of extraction (Goldberger and Benenati, 1959). The pulsed sieve plate extraction columns are especially attractive for fuel reprocessing operations as it can be operated remotely. Moreover, they have fewer moving parts and short residence times (Van Dijck, 1935; Jaradat et al., 2011). Its mechanical simplicity contributes to easier maintenance and their short residence time minimizes the solvent degradation due to intense radioactivity of the irradiated fuel.

The study of hydrodynamic characteristic plays an important role in the design of PSPEC. The literature on the PSPEC has been primarily concerned with the evaluation of the column operating variables (flow rates, nature of continuous phase, pulse amplitude, frequency); geometrical parameters (the number, spacing, and material of construction of the perforated plates; plate perforation diameter and per cent free area; column height and diameter); and fluid properties (viscosity, density, interfacial tension) for determination of the hydrodynamic characteristics like holdup, drop size and flooding.

The two phase flow is found in a wide range of industrial applications in column contactors. In a system of two immiscible liquids, usually an aqueous and an organic liquid, there are two general types of dispersions which can occur in the system. Water-in-oil (w/o) dispersion is a dispersion formed when the aqueous phase is dispersed in the organic phase and oil-in-water (o/w) dispersion is a dispersion formed when the organic phase is dispersed in the aqueous phase (Torab Mostaedi et al., 2008).

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Nomenclature

A	amplitude of pulsation (m)
D	column diameter (m)
f	frequency of pulsation (Hz)
Af	pulse velocity (m/s)
d	perforation diameter (m)
h	plate spacing (m)
T	period of the function
V_c	superficial velocity of continuous phase (m/s)
V_d	superficial velocity of dispersed phase (m/s)
V_c+V_d	throughput of the column (m/s)
DC	duty cycle (%)
d_{32}	Sauter mean diameter (m)
v_c	volume of the continuous phase (m^3)
v_d	volume of the dispersed phase (m^3)

Subscript

c	continuous phase
d	dispersed phase

Greek letters

α	fractional free area
τ	time duration that the function is active
φ	holdup (%)

However, there is often an ambiguity in the usage of the terms; phase reversal and phase inversion associated with two phase flow. The phase reversal refers to a phenomenon where, the dispersed phase reversed to become the continuous phase and vice versa under conditions determined by system properties, phase ratio and energy input. A few authors [Li and Newton (1957), Sobotik and Himmelblau (1960) and Ikeda and Suzuki (1995)] have studied the phase reversal phenomenon for evaluating the effect of plate wetting characteristics. A controlled phase reversal is a desirable and essential step in certain industrial processes like extraction and stripping. It is intentionally created in the column by simply interchanging the continuous and dispersed phases to determine its effects.

Phase inversion is the phenomenon wherein, under particular system conditions, the dispersed phase coalesces to form the continuous phase and simultaneously the continuous phase breaks into droplets to form dispersed flow (Torab Mostaedi et al., 2008; Tidhar et al., 1986; Ioannou et al., 2005). There is a significant change in pressure drop and viscosity of the two-phase mixture in the ambivalent zone marking the transition between the two dispersions (De et al., 2010; Yeo et al., 2002). An uncontrolled phase inversion has to be prevented in all processes. The main difference between phase reversal and phase inversion is that the former is intentionally created whereas the latter is accidental phenomenon.

The objective of this research is to contribute more data of hydrodynamic parameters needed for developing and/or improving design strategies and scale up procedures of pulsed columns. In present study emphasis is given on the finding experimentally hydrodynamic parameters using an original system. Geier (1954) studied the general aspects of pulsed column used for the reprocessing of nuclear spent fuel to recover uranium and plutonium in the Purex process. In the Purex process, the aqueous phase is the dispersed phase to the extraction column; the organic phase becomes the dispersed phase to the stripping column. This paper aims towards understanding basic behaviour of phase reversal operation in a pulsed sieve plate extraction column (PSPEC) so as to study possible applicability in the Purex process.

2. Materials and methods

2.1. Materials

Depending on the operational conditions, either of the two fluids involved can form the dispersed phase.

System-1: The continuous heavy phase is 0.3 M nitric acid (aqueous phase), and the dispersed phase is 30% tributyl phosphate (TBP) in NPH (organic phase) for normal phase operation and vice versa for phase reversal studies.

System-2: Normal phase operation uses kerosene (organic) as dispersed and water as continuous phase conversely phase reversal studies were carried out using water (aqueous phase) as the dispersed phase and kerosene (organic phase) as the continuous phase.

Table 1 gives the properties of experimental test systems for which the hydrodynamic studies have been carried out. It is well known fact that the operation of the column depends on the phase properties like, density difference and interfacial tension (Montazer-Rahmati et al., 2006). The systems used in this work are kerosene/water (with a low interfacial tension and low density difference) which can be considered as the dummy system in comparison with the original system 30% TBP in NPH/0.3 M HNO_3 (with a slightly higher interfacial tension and higher density difference).

2.2. Methods

2.2.1. Measurement of drop size

The videographic technique was used to measure drop size in the active length of the column. The videos of column were taken from two different rectangular sections at bottom (at height of 7.5 cm) and top (at height of 90 cm). Initially these two sections were filled with the continuous phase to nullify the diffraction effect. Camera used in the measurement of drop size was Canon SX20 IS. The most of the videos were taken on AV (aperture value) programme with SM (supermicro) mode. The shutter speed was adjusted at 2500–3000 and frames at *f*/7.5 to *f*/8. The videos were converted into approximately 100 images using KM player and finally converted into pdf for drop size measurement.

2.2.2. Holdup measurement

Holdup is the total volume of dispersed phase that contacted with the continuous phase in the active length of the column and it is directly related to the drop size and free surface area. Holdup was measured by the displacement method. For normal phase operation, holdup is measured at the top disengaging section where dispersed phase is organic phase whereas in phase reversal, holdup is measured at the bottom disengaging section. All parameters are maintained constant till the interface between the two phases attains steady state. Once steady state is reached all inlet and outlet valves are shut off and the drops of dispersed phase are allowed to settle at the top and bottom. The level difference was measured at the top and bottom of the column for normal phase operation and phase reversal respectively and by using cross-sectional