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Experimental investigations of liquid–liquid disengagement in a continuous gravity settler

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ABSTRACT

Several applications in chemical and hydrometallurgical industries involve separation of dispersed liquid–liquid flows. In the present work, experimental investigations of liquid–liquid disengagement are performed in a laboratory-scale continuous gravity settler for tri-butyl phosphate (TBP)–dodecane and nitric acid solution system that is used widely in hydrometallurgical solvent extraction processes. The effects of different operating parameters (total flow rate and inlet drop size distribution) and geometrical parameters (location of baffle inlet opening position and settling area) on the phase disengagement are investigated. The dispersion-band thickness, local dispersed-phase volume fraction and the drop size distribution within the dispersion band are measured. The results are analyzed using the order of magnitude estimates of the settler residence time, convective dispersion velocities, drop-rise velocities, rates of binary and interfacial coalescence processes. Based on the measurements performed in the present work and the data reported in the literature, an empirical correlation is proposed to predict the dispersion-band thickness as a function of dispersed phase flow rate, settling area, inlet drop size distribution, ratio of density of the dispersed phase to the continuous phase, ratio of inlet baffle opening position to the end plate height and inlet dispersed phase volume fraction.

1. Introduction

Several applications involve separation of dispersed liquid–liquid flows in chemical process industry (e.g., in liquid–liquid reactions), crude oil processing (e.g., crude desalting), hydrometallurgy (e.g., solvent extraction), waste-water treatment, etc. Solvent extraction process is widely used in the hydrometallurgical applications because of its metal extraction efficiency. The equipment that are widely used for the solvent extraction are mixer-settlers, centrifugal contactors, rotating disc contactors and pulsed columns. The selection of a particular equipment is dependent on different factors like process volumes to be handled, costs (capital and maintenance), process reliability, solid tolerance capacity, etc. Centrifugal contactors are preferred when short residence time is required. However, the primary drawback of the centrifugal contactors is lower solid tolerance capacity due to the presence of the small internal channels inside the centrifuge chamber. The pulsed column is an effective contactor when intermediate residence time is required for the process. In addition, the pulsed columns consume lower floor space because the mixing and separation processes occur in the same column.

On the contrary, mixer-settlers are widely used in the processes that require longer residence time and higher throughputs. Several additional factors like lower capital investment, high process reliability, operational simplicity and low maintenance cost make the mixer-settler as a prominent candidate for the solvent extraction process. In a mixer-settler, two immiscible liquid phases enter the mixer, and the organic phase is dispersed into the aqueous phase (due to the shear generated by the impeller) in the mixing chamber (used for extraction) and then the dispersion flows into a large-sized gravity settling chamber. The dispersion undergoes binary and interfacial coalescence that occur within the dispersion band inside the continuous gravity settler and results into phase separation.

Over the years, liquid–liquid disengagement was investigated experimentally in various extraction equipment e.g., pulsed-sieve extraction column (e.g., Gourdon and Casamatta, 1991; Mirzaie et al., 2016), rotating disc contactors (RDC) (e.g., Gonçalves et al., 2016; Wang et al., 2002) and centrifugal contactors (Mincher et al., 2016; Nakahara

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Nomenclature

A_s	Settling area, m^2
A_{cs}	Settler cross-sectional area, m^2
A_d	Surface area of drop, m^2
d_{32}	Sauter mean diameter, m
d_d	Drop diameter, m
d_h	Hydraulic diameter of settler cross-section, m
Eö	Eötvös number (as defined in Table 3), –
g	Gravitational acceleration, m/s^2
H_{db}	Height of dispersion band, m
H_b	Height of dispersion inlet opening baffle, m
H_t	Height of end plate, m
h	Height of settler, m
h_o	Height of dispersion inlet baffle opening, m
l_s	Length of settler, m
N	Number of drops, –
N_D	Dispersion number (as defined in Table 3), –
Q_d	Dispersed phase flow rate, L/h (m^3/s in correlation (Eq. (11)))
Q_t	Total flow rate, L/h
Re	Reynolds number, –
r	Drop radius, m
t_{ic}	Time of interfacial coalescence, s
t_{RM}	Phase residence time in mixer, s
t_{RS}	Phase residence time in settler, s
V	Volume of settler, m^3
V_d	Volume of a droplet, m^3
u_r	Rise velocity, m/s (used in Eq. (7))
V_m	Volume of mixer, m^3
υ_d	Dispersion velocity, m/s
υ_{in}	Dispersed phase velocity at inlet, m/s
υ_r	Terminal rise velocity, m/s
w_o	Width of dispersion inlet opening, m
x^*	Dimensionless settler length (l/l_s), –
y^*	Dimensionless settler height (h/H_t), –

Greek letters

α_d	Dispersed phase volume fraction, –
α_c	Continuous phase volume fraction, –
ρ_d	Dispersed phase density, kg/m^3
ρ_c	Continuous phase density, kg/m^3
$\Delta\rho$	Density difference, kg/m^3
σ	Interfacial tension, N/m
ε_c	Dispersed phase volume fraction at coalescing interface, –
μ_c	Continuous phase viscosity (kg/ms)
Ψ	Rate of interfacial coalescence per unit area, m^3/s m^2
Γ	Rate of binary coalescence, m^3/s

Subscript/superscript

ic	Interfacial coalescence
in	Inlet
bd	Buoyancy-driven collision
sd	Shear-driven collision

Acronyms

DSD	Drop size distribution
TBP	Tri-butyl phosphate
RMSD	Root mean square deviation

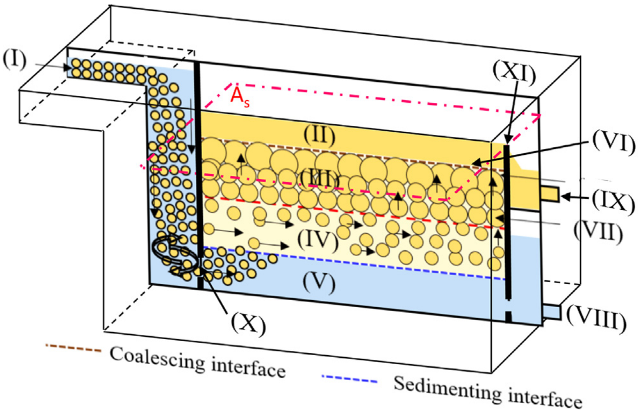

Fig. 1 – Schematic of a continuous gravity settler, (I) dispersion inlet, (II) pure disengaged organic phase, (III) dense-packed zone, (IV) sedimentation zone, (V) pure disengaged aqueous phase, (VI) interfacial coalescence front, (VII) region of binary coalescence, (VIII) aqueous phase outlet, (IX) organic phase outlet, (X) inlet baffle opening slot and (XI) end plate.

et al., 2007; Schuur et al., 2010). The important issue in the aforementioned equipment is the flooding because of the inadequate residence time provided for the phase disengagement. In the mixer-settler contactors, the formation of dispersed phase drops and mass transfer between the continuous and dispersed phase occur inside the mixer. Several researchers investigated the effects of geometrical parameters (e.g., impeller blade, impeller diameter, baffles and mixer size) (Podgórska and Bałdyga, 2001; Zhou and Kresta, 1998), operating parameters (e.g., impeller speed, system temperature and ratio of flow rates of organic to aqeous phase) (Desnoyer et al., 2003; Mlynek and Resnick, 1972) and physical properties of the phases (e.g., viscosity and interfacial tension) (Calabrese et al., 1986; Jana and Sau, 2004) on the drop size distribution (*DSD*) inside the mixer. Since the hydrodynamics of liquid–liquid flows inside the mixer is well investigated, the focus of the present work is on understanding of the flow of liquid–liquid dispersion and separation inside the continuous gravity settler. Therefore, detailed analysis of the literature on the mixer is not presented here.

Compared to the literature available on the pulsed columns, RDCs, centrifugal contactors, and mixers; there exists very less information on the liquid–liquid disengagement in continuous gravity settlers (Eckert and Gormely, 1989; Jeelani and Hartland, 1993; Padilla et al., 1996). The phase disengagement rate after extraction process is dependent on the dispersion flow rate (Q_t), *DSD* at the inlet, settling area (A_s), internals (baffles, picket fences) of the gravity settler, physical properties of the liquids, rates of binary coalescence and interfacial coalescence, etc. (Eckert and Gormely, 1989; Jeelani and Hartland, 1993; Padilla et al., 1996). The schematic of a continuous gravity settler is shown in Fig. 1. The dispersion is introduced through inlet baffle to settling region with settling area of A_s (see Fig. 1). The settler consists of three regions, the separated aqueous phase (at the bottom), organic phase (at the top) and the dispersion band (between the organic and aqueous phase). The binary (drop–drop) and interfacial (drop–interface) coalescence occur within the dispersion band and results into the phase separation. The rates of binary and interfacial coalescence are dependent on several parameters such as *DSD*, Q_t, settler residence time (t_{RS}), dispersion inlet velocity (υ_{in}), drop rise velocity (υ_r), etc. Due to the binary coalescence, the size of drops increases from the settling front (Passive Interface, *PI*) to the coalescence front (Active Interface, *AI*) along the length and height of the settler. Eventually, due to the interfacial coalescence, the drops present near the *AI* coalesce with the bulk organic phase. The pure organic phase flows over the end plate weir whereas pure aqueous phase (settled at the bottom of the settler) exits below the end plate weir. The position of the dispersion band inside the settler is maintained at a particular position by interface controller mounted on the aqueous-phase outlet. Due to the significant influence