

Determination of slip and characteristic velocities in reactive extraction with experiments in the Oldshue-Rushton column and presence of samarium and gadolinium metals



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ABSTRACT

The slip and characteristic velocities of the reactive system (samarium, gadolinium and D2EHPA) were investigated in the pilot plant Oldshue-Rushton extraction column. The experiments were carried out at the extraction and stripping stage. The influences of the agitation speed, dispersed as well as continuous phase velocity were studied. An increase in dispersed phase velocity led to the increase of the slip velocity while it decreased with agitation speed and continuous phase velocity. More buoyancy and faster upward movement of larger drops accelerate the slip velocity in the stripping condition as compared with the case of extraction condition. The experimental data were compared with the proposed correlations by Asadollahzadeh et al., and the related deviation was within 26% from these equations. Therefore, the modified correlations for prediction of slip and characteristic velocities were proposed with the experimental data and the data from the physical systems in the literature.

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1. Introduction

Rare earth elements play a significant role in the manufacture of catalysts, fluorescent lamps, permanent magnets, super alloys, medicines and several other appliances [1–3]. Samarium is one of the rare earth elements that is applied for the manufacture of Sm-Co permanent magnets [2]. It is also used as a samarium iodide catalyst for speeding up decomposition of plastics, dechlorination of PCBs and ethanol dehydration [4]. The other rare earth element is gadolinium whose organic compounds are used for magnetic resonance imaging. It can reduce relaxation times and thereby enhance signal intensity with its high magnetic moment [5]. It is also added to optical glasses meant for use in the optical fibers. It has a potential application as the functional material in magnetic refrigeration [6]. In addition, samarium, gadolinium, and dysprosium oxides are used in the nuclear industry in the radiation shielding [7]. The large primary and secondary sources of rare earth elements in the world have led to considerable research into suitable extraction and separation processes including ion exchange [8], solvent extraction [9,10] and chemical precipitation [11] to produce individual rare earth elements.

Among these technologies, solvent extraction is the most widely used technique in which commercially available extractant, such as D2EHPA [12,13]; HEH/EHP [14]; Cyanex 272 [15]; Cyanex 301 [16]; Cyanex 302 [17]; Cyanex 923 [18]; Aliquat 336 [19] and sec-octyl phenoxy acetic acid [20] for extraction and separation of samarium from gadolinium, are utilized.

Mixer-settler contactors are used as the standard extraction equipment in the rare earths processing plant. The actual equipment arrangement depends on the nature of the feed and product requirement [9,21]. However, large space requirement, large solvent inventory, long residence time, and poor sealing of the system can be listed as the disadvantages of mixer-settlers. To achieve better flexibility and extraction efficiency, agitated extraction columns such as rotating disc column (RDC) [22,23], asymmetric rotating disc column (ARDC) [24], Kühni column [25], Scheibel column [26] and Oldshue-Rushton column [27,28] were scrutinized in the light of the solvent extraction processes. Nonetheless, the applications of the Oldshue-Rushton extractor were not observed in the literature for rare earth separation and purification from the primary and secondary resources.

Information on hydrodynamic parameters in the Oldshue-Rushton column such as droplet size, holdup, slip and characteristic velocities is important for the scale up and the design of this extraction column. The slip velocity controls the mass transfer coefficient when a solute is transferred between the two phases,

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Nomenclature

| | |
|----------|---|
| d_{32} | Sauter mean diameter (m) |
| d_i | Drop diameter (m) |
| d_r | Rotor diameter (m) |
| g | Acceleration due to gravity (m s^{-2}) |
| N | Rotor speed (s^{-1}) |
| R | Reaction parameter (–) in Eq. (7) |
| V_c | Continuous phase velocity (m s^{-1}) |
| V_d | Dispersed phase velocity (m s^{-1}) |
| V_k | Characteristic velocity (m s^{-1}) |
| V_s | Slip velocity (m s^{-1}) |

Greek Letters

| | |
|--------------|--|
| $\Delta\rho$ | Density difference between phases (kg m^{-3}) |
| μ | Viscosity (Pa s) |
| ρ | Density (kg m^{-3}) |
| σ | Interfacial tension (N m^{-1}) |
| φ | Dispersed phase holdup (–) |

Subscripts

| | |
|---|------------------|
| c | Continuous phase |
| d | Dispersed phase |

while the holdup together with the drop size determines the interfacial area for mass transfer conditions [29,30]. Another important hydrodynamic parameter is the characteristic velocity requiring a good knowledge so as to predict the flooding time and maximum volumetric capacity of an extraction column [31].

No information for the design and scale up of Oldshue–Rushton columns in reactive extraction conditions is available in the literature. In this research work, the effects of operating parameters such as rotor speed, dispersed and continuous superficial velocities on the slip velocity and the characteristic velocity in reactive extraction conditions with the presence of samarium, gadolinium and D2EHPA extractant were investigated.

The previous correlations in the literature for the physical systems without reaction condition were studied and the comparison between them and the experimental results was evaluated in this column. In addition, the modified correlations for prediction of slip and characteristic velocities were proposed in this research work.

2. Experimental

2.1. Materials

Commercial grade of kerosene was used as a diluent in the extraction process without further purification and the organic

solvent (di-(2-ethylhexyl) phosphoric acid (D2EHPA)) from Aldrich, was employed as an extractant. Commercial salts ($\text{Sm}(\text{NO}_3)_3 \cdot 6\text{H}_2\text{O}$, $\text{Gd}(\text{NO}_3)_3 \cdot 6\text{H}_2\text{O}$, Middle East Ferro Alloy Company, 99.9% purity) were used as rare earth metals in the experiments. The aqueous phase solutions were prepared by dissolving these salts in the deionized water.

2.2. Apparatus

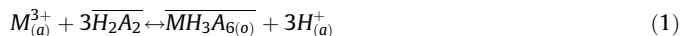
The samarium and gadolinium content in the aqueous stream was measured using ICP-AES spectroscopy and a Perkin-Elmer model 5500 inductively coupled plasma atomic emission spectroscopy. The density measurements were performed using the pycnometer method. The viscosity data were obtained by DVI-Prime viscometer and the interfacial tension data were measured by a Krüss tensiometer. Table 1 shows the physical properties of the two phases.

2.3. Pilot plant Oldshue-Rushton column

The schematic diagram of the pilot plant Oldshue-Rushton column is shown in Fig. 1 and the general characteristics of the column are also listed in Table 2.

The extraction column consisted of a cylindrical shell and a baffled stator. Moreover, it was equipped with 6-blade impellers with accurate speed control. The main section was made of Pyrex glass, 113 mm I.D, and the height of the column was 700 mm. The value of the free area was 25% at the nine stages as compared with the column cross-sectional area. The stirrer was located in the center of each compartment with 6-blade impellers 50 mm in diameter and these impellers were driven by an electric motor via a variable gearbox. The two phases were introduced counter-currently in the column through the circular perforated distributors with the light phase entering from the bottom, and the heavy phase from the top of the column. The interface was maintained at the required level by means of an optical sensor as previously described [32].

The aqueous phase of the reactive test system consists of water, samarium nitrate, gadolinium nitrate and nitric acid, whereas the solvent kerosene and the cation exchanger D2EHPA form the organic phase. D2EHPA is a surface-active component and extracts samarium and gadolinium via an interfacial reaction as follows:



where H_2A_2 and M^{3+} represent the D2EHPA and samarium or gadolinium, respectively. The bars in Eq. (1) indicate the components in the organic phase.

In extraction runs, the nitrate solution of samarium and gadolinium with 500 ppm concentration, adjusted at 1.5 pH in water and 0.12 M D2EHPA diluted with kerosene were used as the continuous phase and dispersed phase, respectively.

In stripping runs, nitrate solution with 0.1 M concentration and the loaded organic phase of samarium and gadolinium with

Table 1
Physical properties of the phases used in the system, at 25 °C.

| Properties | | $\rho(\text{kg m}^{-3})$ | $\mu(\times 10^3 \text{ kg m}^{-1} \text{ s}^{-1})$ | $\sigma(\times 10^3 \text{ N/m})$ |
|------------------|------------------|--------------------------|---|-----------------------------------|
| Extraction Stage | Continuous phase | 1005 | 0.9216 | 22.1 |
| | Dispersed phase | 789 | 1.2587 | |
| Stripping Stage | Continuous phase | 995 | 0.9204 | 23.5 |
| | Dispersed phase | 795 | 1.2674 | |

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