

# HIDRODINAMIČKE KARAKTERISTIKE EKSTRAKCIONIH KOLONA TIPA OLDŠU-REŠTON

## HYDRODYNAMIC CHARACTERISTICS OF OLDSHUE-RUSHTON EXTRACTION COLUMNS

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*U ovom radu je dat pregled eksperimentalnih istraživanja, koja su preuzeta iz literature, a odnose se na određivanje hidrodinamike u ekstrakcionim kolonama tipa Oldšu-Rešton u slučaju korišćenja različitih dvofaznih sistema tečno-tečno. Razmatran je uticaj radnih parametara kolone (broj obrtaja rotora, protoci faza i pravac prenosa mase između faza), geometrije kolone, kao i fizičkih osobina dvofaznih sistema na hidrodinamičke karakteristike ekstrakcione kolone. Od hidrodinamičkih karakteristika razmatrani su: efikasnost stupnja, srednji prečnik kapi i sadržaj dispergovane faze u radnom delu ekstrakcione kolone. Utvrđeno je da efikasnost stupnja veća kada se prenos mase ostvaruje iz dispergovane faze u kontinualnu fazu u odnosu na obrnut proces prenosa mase. Konstatovano je da je veličina kapi dispergovane faze veća kada se radi o sistemu sa većim međufaznim naponom nego kada je sistem imao umereni međufazni napon. Kada se relativna brzina faza povećavala, dolazilo je do smanjenja sadržaja dispergovane faze i efikasnosti stupnja. Broj obrtaja rotora vrlo mnogo utiče na performanse ekstrakcione kolone, dok je uticaj protoka faza zenemarljiv. Komentarisana je primena empirijskih korelacija koje su razvijene za predviđanje srednjeg prečnika kapi, raspodelu veličina prečnika kapi, kao i sadržaj dispergovane faze. Poznavanje vrednosti hidrodinamičkih parametara u Oldšu-Reštonovoj koloni je od velikog značaja u postupku projektovanja kolona pri radu sa sistemom tečno-tečno. Razmatranje eksperimentalnih rezultata, koji su preuzeti iz literature, a prikazani su u ovom radu i odnose se na hidrodinamiku kolone, može biti od značaja i praktičnog interesa pri dizajnu i projektovanju Oldšu-Reštonovih kolona.*

**Ključne reči:** srednji prečnik kapi, efikasnost stupnja, sadržaj dispergovane faze, protok faza

*This work presents an overview of research related to the determination of hydrodynamic characteristics of Oldshue-Rushton extraction columns using different two-phase liquid-liquid systems. The following hydrodynamic characteristics are examined: mean drop diameter, dispersed-phase holdup and stage efficiency. In this paper the effects of operating parameters (rotor speed, superficial velocities of the phases and directions of mass transfer between the phases) on the data of hydrodynamic characteristics, taken from the literature, are reviewed. Experimental data have shown that the stage efficiency depends very strictly on the rotor speed and the interfacial tension, but very little depends on the dispersed and continuous phase velocities. On the other hand, it was found that the stage efficiency was higher when the mass transfer of solute from the dispersed to the continuous phase was achieved compared to the mass transfer from the continuous to the dispersed phase. The drop size of the dispersed phase was larger in a system with a higher interfacial tension than in a system with a medium interfacial tension. Finally, when the relative phase velocity increases, this leads to a decrease in the dispersed-phase holdup and the stage efficiency and, as a result, the extraction column operates in a more or less stable mode. The rotor speed had the greatest effect on the performance of the extraction column, while the influence of the phase flow was negligible. Some of the empirical correlations were commented, which included the influence of physical properties of liquid systems, operational parameters and geometric parameters of the column on Sauter mean drop diameter ( $d_{32}$ ).*

**Key words:** Sauter mean drop diameter, dispersed-phase holdup, phase flows, stage efficiency

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## 1 Introduction

Liquid-liquid extraction, sometimes called solvent extraction, is a separation operation in which two immiscible phases are brought into contact, with one or more solutions transitioning from one phase to another on the basis of better solubility [1]. If the substances from the original solution are distributed differently between the two liquid phases, an appropriate degree of separation will be achieved, which is increased by multiple contact of the phases, which also happens in separation of absorption and distillation. Solvent extraction is used in the chemical and petrochemical industries, hydrometallurgy, biotechnology, nuclear technology, separations in the food industry, wastewater treatment and similar technologies [2]. Separation processes involving liquid-liquid extraction are performed in various column devices, such as: spray column, packed column, perforated-plate column, pulsed column (with packaging or with perforated plates), rotation disk contactor (RDC), Kühni column, Scheibel column, Oldshue-Rushton column and other extraction columns [3]. In our recent work, we have dealt with hydrodynamics and mass transfer in some of the above extraction columns [4-8].

The Oldshue-Rushton extraction column belongs to the group of extraction columns with mechanical agitation. This column is in the group of extraction columns with the highest efficiency that are applied in industrial practice. The Oldshue-Rushton extraction column consists of a series of sections, one section consisting of horizontal ring stators, connected by four vertical baffles, connected to the inner wall of the extraction column [9], **Figure 1**.

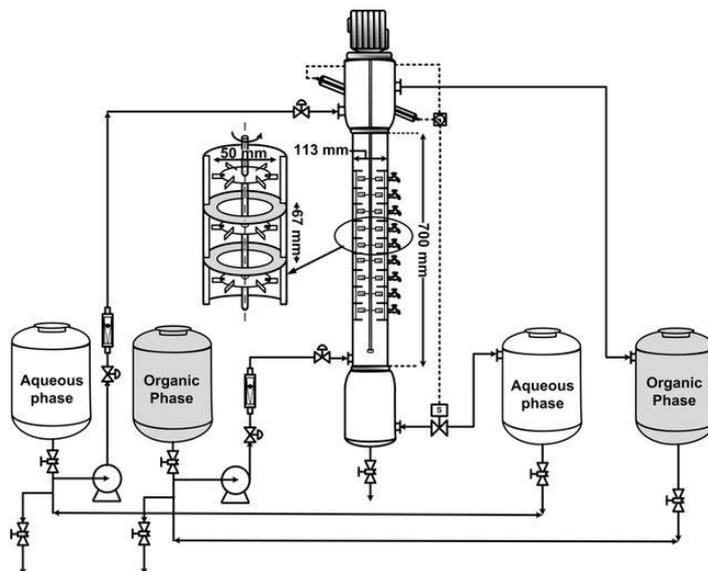


Figure 1. Schematic diagram of the Oldshue-Rushton extraction column  
(Source: et al., *Inter. J. Eng.*, 29 (2016), (8), 1047-1055 [9])

Ring stators reduce the backmixing in the column extractor. On the vertical axis, there are flat turbine mixers, which are used for dispersing and mixing liquid phases. By changing the geometry of the stator, the regime of fluid flow inside the column device is significantly affected [10]. This type of extractor can also be used to separate heterogeneous solid phase systems, as well as when a gas phase is present. In practice, the application of the Oldshue-Rushton column in uranium extraction has been written, which is one of the systems that are difficult to extract, since they have a high interfacial tension, and chemical reaction also occurs.

In liquid-liquid extraction processes, the most important hydrodynamic characteristics are: Sauter mean drop diameter, dispersed-phase holdup, flooding point and split (relative) phase velocity. These parameters depend on the geometry of the column, the superficial velocity of the continuous and dispersed phases, as well as on the physical properties of the liquid-liquid system. On the other hand, the values of hydrodynamic parameters are necessary in the calculations of the axial diffusion coefficient and the performance related to mass transfer in two-phase liquid systems.

The aim of this paper is to present a comparison of experimental results, taken from the literature, which refer to hydrodynamic characteristics in extraction columns of the Oldshue-Rushton type, and which are a function of rotor speed and liquid phase flow ratio. Different two-phase liquid-liquid systems were used to obtain these experimental data. From the experimental hydrodynamic data, the mean drop diameter, dispersion-phase holdup as a function of the operating parameters of the extraction columns were considered. Empirical correlations, developed for Sauter mean drop diameter, as well as the dispersed-phase holdup were also considered. Based on the results taken for the hydrodynamic parameters, the optimal values of the quantities that characterize the geometry of the column were determined, as well as the operational parameters that are necessary for the design and engineering of these extraction columns.

## 2 Hydrodynamics of the columns

Asadollahzadeh *et al.* [11] presented the results of investigating the influence of operational parameters such as mass transfer direction between the phases, rotor speed and dispersed and continuous phase superficial velocities on mass transfer in the Oldshue-Rushton extraction column. The number of rotor speed had the greatest effect on the performance of the extraction column, while the influence of the phase flow was negligible. An empirical correlation was developed in the literature [9] which included the influence of physical properties of liquid systems, operational parameters and geometric quantities of the column on Sauter mean drop diameter ( $d_{32}$ ). The results obtained by applying the empirical correlation were compared with the experimental results taken from the literature and taken from the present paper [9]. The correlation included experimental results for different obtained in a large number of extractors using mechanical agitation.

In research [12], the efficiency of the stage was measured in an Oldshue-Rushton column with a diameter of 11.3 cm for two two-phase systems: toluene-acetone-water (medium interfacial tension) and n-butyl acetate-acetone-water (high interfacial tension). The experiments were performed in both directions of mass transfer between phases. The influence of various parameters, such as: rotor speed, superficial velocities of the dispersed and continuous phases and the direction of mass transfer of the solution (acetone) between the phases on the stage efficiency in the extraction column was investigated. Experimental data have shown that the stage efficiency depends very strictly on the rotor speed and the interfacial phase tension, but very little depends on the dispersed and continuous phase velocities, **Figure 2a**. It was found that the stage efficiency was higher when the mass transfer of solute (acetone) from the dispersed to the continuous phase (d to c) was achieved compared to the mass transfer from the continuous to the dispersed phase (c to d). This is due to the presence of oscillations caused by the surface stress gradient, **Figure 2b**. The drop size of the dispersed phase was larger in a system with a higher interfacial tension than in a system with a medium interfacial tension, which leads to the fact that these drops pass faster through the working height of the extraction column. Finally, when the relative phase velocity increases, this leads to a decrease in the dispersed-phase holdup and the efficiency of the stage, and, as a result, the extraction column operates in a more or less stable mode [12].

In this same paper [12], an empirical correlation was developed that describes the dependence of degree efficiency on Reynolds or Froude numbers, respectively. It was found that there is a good agreement between calculated and experimental data for the stage efficiency in the extraction column. Knowledge of the behavior related to the diameter of the drops is one of the very important criteria in determining the kinetics of mass transfer when choosing extraction columns of the Oldshue-Rushton type in liquid-liquid. In the paper [10], the experimental application of multiple control of the extraction column of the Oldshue-Rushton type was presented. The influence of control of the dispersed-phase holdup during mass transfer and simultaneous control of the concentration of the solution at the outlet of d the extraction column are included in this paper. The aim of the control was set to control the outlet concentration of the solution and to maintain a high value of the dispersed-phase holdup under the safe operation of the column in terms of flooding conditions. Models of transfer functions were identified from step tests around the normal operating point. The output concen-

tration of the solution was controlled by changing the flow of the continuous phase, while the dispersed-phase holdup was controlled by changing the rotor speed. The Dahlin controller has been used in both control circuits and has been shown to work satisfactorily for both servo devices and the control controller even if the extraction process is not well represented by the transfer function. The monitoring and control process, presented in this research [10], can also be used for a new or existing extraction column device to ensure safe and optimal operation.

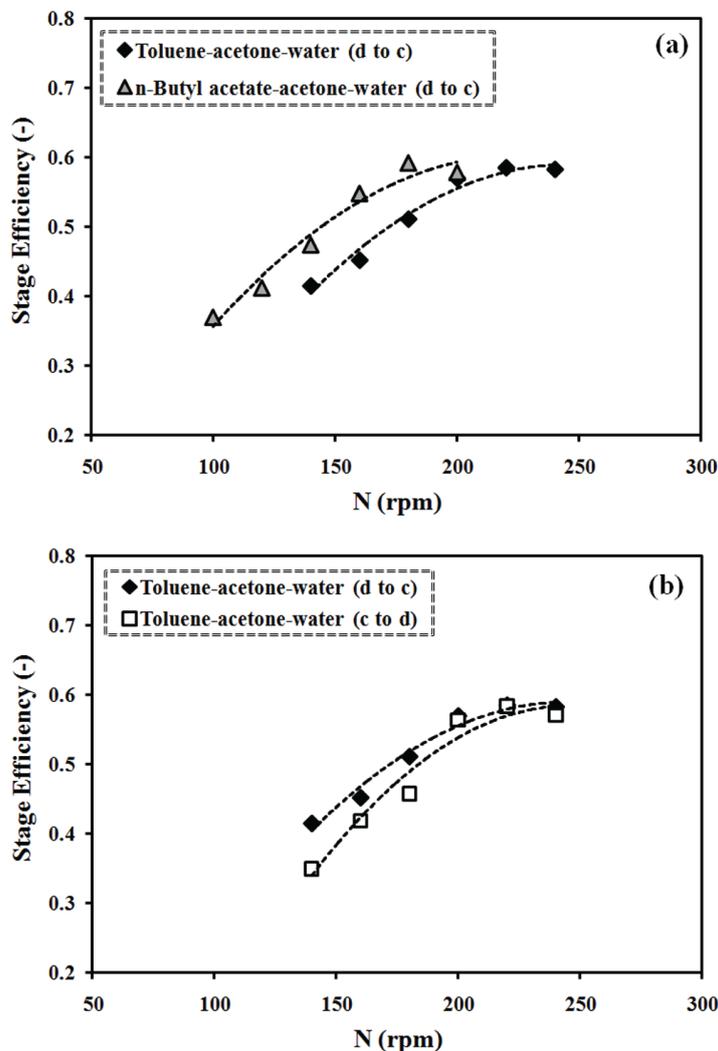


Figure 2. Effect of rotor speed on the stage efficiency with different direction of mass transfer between the phases: a) surface tension; b) direction of mass transfer ( $V_c = V_d = 0.66$  mm/s); (Source: Asadallahzadeh et al. *Chem. Ind. Chem. Eng. Q.* 2016, 22 (1), 75-83 [12]).

Sauter's mean drop diameter and drop size distribution were determined for three different liquid-liquid systems as a function of phase flow and rotor speed in a semi-industrial extraction column of Oldshue-Rushton type, [13]. It was found that the Sauter mean drop diameter and the distribution of drop diameter sizes significantly depend on the rotor speed, physical properties of liquid systems and the direction of mass transfer between phases, while only partially depend on the phase flow. The experimental data were fitted using the density probability function, i.e. the normal and log-normal functions. A mathematical approach was used to determine the parameters (constants) in these functions and thus enable the fitting of experimental data. At the same time, an empirical correlation has been proposed that gives the dependence of the Sauter mean drop diameter on the operating parameters, column geometry, and physical properties of the liquid system. Good agreement was achieved between calculated and experimental data.

Oxidative desulfurization (ODS) is an attractive complementary or alternative process for the hydrodesulfurization (HDS) process due to advantages such as mild operating conditions. In the ODS

process, the sulfur-containing compounds are oxidized first to polar sulfoxide and sulfone products and then these polar compounds are separated using solvent extraction or an adsorption process. In the paper [14], the results of sulfur extraction in a semi-industrial extraction column of Oldshue-Rushton type were presented, where the operating costs are optimized while the initial mixture (fed) at the inlet of the column was previously determined in one reactor with oxidative desulfurization. Dimetilformamid was used as a polar solvent to remove sulfur contaminated components in oxidized diesel in the extraction column. The same paper presented the influence of rotor speed (100-200 rpm) and solvent inlet flow in the column (33-165 mL/min) on the content of the dispersed phase, the degree of sulfur removal, diesel return flow, as well as solvent regeneration. The methodology of response surfaces was applied in the procedure. Operating costs during the continuous extraction process, which consist of the costs of consumed chemicals, the costs of spent energy, as well as health costs related to SO<sub>2</sub> emissions, were used as criteria for optimizing the extraction process. The best performance of the extraction process was achieved at room temperature, where the inlet flow of oxidized diesel was 99 mL/min, rotor speed 107 rpm, inlet flow of solvent was 35 mL/min. Under these conditions, the content of the dispersed phase, sulfur removal, diesel regeneration and solvent regeneration were 0.0101; 92.75; 91.80 and 96.90%, respectively. In this paper [14], a corresponding improvement in the process of sulfur removal was achieved, as well as the costs of chemical consumption

### 3 Conclusion

This study presents a comparative view of the effects of hydrodynamic characteristics of Oldshue-Rushton columns such as dispersed-phase holdup, mean drop diameter, and stage efficiency on the throughput of extraction columns. The experimental data, taken from the literature, have shown that the stage efficiency depends very much on the of rotor speed and the interfacial tension, but very little depends on the phase flows. On the other hand, it was found that the stage efficiency was higher when the mass transfer of solute from the dispersed to the continuous phase was achieved compared to the oposite transfer. The drop size of the dispersed phase was larger in a system with a higher interfacial tension than in a system with a medium interfacial tension. The experimental data presented in this work for the mean drop diameter, dispersed-phase holdup, and stage efficiency in Oldshue-Rushton columns can serve as a sound basis for the development of corresponding empirical correlations that can be used in designing these columns. The knowledge of the values of hydrodynamic characteristics in extraction column of the Oldshue-Rushton type is of great importance in the design of these columns in liquid-liquid systems. Consideration of experimental results from scientific papers taken from the literature which are presented in this paper, and relate to the determination of some hydrodynamic characteristics in the Oldshue-Rushton columns, may be of practical interest to scale-up and designing of this extractors type.

### 4 Nomenclature

- d - mean drop diameter, m
- $d_i$  - mean diameter of drops in a certain interval  $i$ , m
- $d_{32}$  - Sauter mean drop diameter,  $d_{32} = \frac{\sum_{i=1}^{NN} d_i^3}{\sum_{i=1}^{NN} d_i^2}$ , m
- N - rotor speed, rpm
- NN - total number of drops
- $V_c$  - continuous phase superficial velocity, m/s
- $V_d$  - dispersed phase superficial velocity, m/s
- $\varepsilon_d$  - dispersed-phase holdup.

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