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Liquid–liquid flow in an impeller–stator spinning disc reactor

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ABSTRACT

Liquid–liquid flow patterns in an impeller–stator spinning disc reactor are qualitatively described. The liquid–liquid flow is studied by contacting water and n-heptane at rotational speeds up to 900 RPM, an axial disc spacing of 1.0×10^{-3} m, and aqueous volume fractions between 0 and 1 $m^3_{AQ}\, m_L^{-3}$. The liquid–liquid flow is characterized by six different flow regimes. Each regime is characterized by its continuous liquid phase and the degree of dispersion of the dispersed liquid phase. Regime transitions depend on the formation of boundary layers on the impeller and the stator, the shear stress intensity and the aqueous volume fraction. The impeller is able to selectively pump n-heptane through the reactor, whilst it is intensively mixed with water that remains in the reactor. Combining mixing and separation in one compact reactor is an important step toward a countercurrent operated impeller-based centrifugal extractor.

1. Introduction

The multiphase flow behavior in a chemical reactor has a major influence on the selectivity and conversion of a given process. Understanding of and control over this flow behavior is therefore essential when a novel multiphase reactor is developed for liquid–liquid processes [1]. An example of such a reactor is the rotor–stator spinning disc reactor (SDR). Due to the rotation of the rotor, a velocity gradient is present between the rotor and the stator. The velocity gradient causes a shear force to act upon the liquids between the rotor and the stator. This causes an increase of the liquid–liquid interfacial area and of the turbulence intensity. Accordingly, the liquid–liquid mass transfer rate is increased [2]. The behavior of liquid–liquid and gas–liquid flow in a rotor–stator spinning disc reactor was reported previously [2–5].

This paper describes the flow behavior of water and n-heptane in an impeller–stator spinning disc reactor. In this reactor an impeller is used instead of the solid rotor in the SDR. The impeller has comparable geometry as the impeller in a centrifugal pump. In a centrifugal pump, rotation of the impeller will transfer energy from the motor to the fluid. Thereby the fluid is accelerated radially outward from the center of rotation. In the impeller–stator spinning disc reactor, the impeller has the same function. The impeller is used to transport liquids radially outwards, from the center of rotation to the rim of the impeller. A side view of the impeller–stator spinning disc reactor is given in Fig. 1. The direction of liquid flow in the impeller–stator configuration is shown in the close up of the impeller in Fig. 1.

Using an impeller instead of a solid rotor, allows for the selective through flow of the liquid with lower density while the liquid with higher density remains in the reactor [6]. This is schematically shown in Fig. 2. Accordingly, a consecutive sequence of mixing and separation is described [7]. This is an important step toward counter-current liquid–liquid extraction [8]. Information about the liquid–liquid flow behavior in the impeller–stator configuration allows for a qualitative estimation of the liquid–liquid mass transfer rate in impeller based centrifugal extractors [7]. This paper describes the liquid–liquid flow behavior in the impeller–stator spinning disc reactor by six flow regimes. These regimes are qualitatively described as a function of the rotational speed of the impeller and the aqueous volume fraction. The regime transitions are dependent on the formation of boundary layers on the impeller and the stator, the shear stress intensity and the aqueous volume fraction. Also, the requirements for selective through flow of n-heptane are discussed.

2. Experimental

2.1. Experimental set-up

The reactor consists of a cylindrical transparent polymethyl methacrylate (PMMA) housing with an inner diameter of 0.152 m and an internal height of 7.0×10^{-3} m. Both above and below the impeller the axial clearance between the impeller and the stators

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Nomenclature

Symbols

H	head (m)
N	rotational speed (RPM)
Q	volumetric flow rate ($m^3\ s^{-1}$)
r	radius (m)
Re_Ω	rotational Reynolds number ($\omega r^2 \nu^{-1}$)
SS	stainless steel
ν	kinematic viscosity ($m^2\ s^{-1}$)
V	volume (m^3)

Greek letters

γ	interfacial tension ($mN\ m^{-1}$)
ε_{AQ}	aqueous volume fraction ($m^3_{AQ}\ m_L^{-3}$)
μ	viscosity ($kg\ m^{-1}\ s^{-1}$)
ρ	density ($kg\ m^{-3}$)
σ	surface tension ($mN\ m^{-1}$)
ω	rotational disc speed ($rad\ s^{-1}$)

Sub- and superscripts

ORG	organic
AQ	aqueous
L	liquid
R	reactor
IV	inversion

equals 1.0×10^{-3} m (Fig. 1). This distance is further denoted as disc spacing. The impeller is constructed out of two concentric circular plates connected by six vanes. Two different impellers were used; their top and side views and the flow directions are schematically shown in Fig. 1. The constructional details of the impellers are given in Table 1. Impeller A is constructed out of stainless steel and has an equal radius for the top and bottom plates, both with a radius of 6.6×10^{-2} m. Impeller B is constructed out of stainless steel and has a radius of 7.3×10^{-2} m for the bottom plate, while the top plate has a radius of 6.3×10^{-2} m. The total liquid volume in the reactor, V_R, equals $6.6 \times 10^{-5}\ m^3$ for impeller A, and $6.3 \times 10^{-5}\ m^3$ for impeller B. The reactor is equipped with two inlets and one outlet. The first inlet is located in the center of the bottom stator, and is equipped with a ¼″ Swagelok® connection. The second inlet is located at 6.2×10^{-2} m from the center of the bottom stator as an orifice with a diameter of 1.4×10^{-3} m and a Swagelok® connection of ⅛″. The reactor outlet is situated in the top stator at a radius of 3.3×10^{-2} m from the center with a ¼″ Swagelok® connection. A calibrated gear pump (E-7500-09, Bronkhorst®) is used to feed the liquids to the reactor, with a maximum flow rate of $15 \times 10^{-5}\ m^3\ s^{-1}$. The impeller is propelled with a Re162 lab stirrer from IKA® laboratories. The maximum rotational speed is 1700 RPM. The rotational Reynolds number is defined as $Re_\Omega = \omega r^2 \nu^{-1}$, and thus is maximized to 7.98×10^5.

Table 1
Constructional details of the impeller configurations.

Property	Unit	A	B
Material		SS	SS
Radius top disc	m	6.6×10^{-2}	6.3×10^{-2}
Radius bottom disc	m	6.6×10^{-2}	7.3×10^{-2}
Height single disc	m	1.5×10^{-3}	1.0×10^{-3}
Height radial outflow	m	2.0×10^{-3}	2.0×10^{-3}
Empty reactor volume	m^3	6.6×10^{-5}	6.3×10^{-5}

Table 2
Density, ρ/$kg\ m^{-3}$, viscosity, μ/$kg\ m^{-1}\ s$, surface tension, σ/$mN\ m^{-1}$, and interfacial tension, $\gamma_{org\text{-}aq}$/$mN\ m^{-1}$, of water and n-heptane at 293 K. The presence of ink is not taken into account for these data [9].

Property	Water	n-Heptane
Density ($kg\ m^{-3}$)	0.99×10^3 [23]	0.68×10^3[24]
Viscosity ($kg\ m^{-1}\ s$)	1.02×10^{-3} [23]	0.39×10^{-3}[25]
Surface tension ($mN\ m^{-1}$)	72.58 [23]	20.05 [26]
Interfacial tension ($mN\ m^{-1}$)	–	51.90 [26]

2.2. Other equipment

Visual observations of the fluid flow were obtained through the bottom stator and show the space of the reactor between rotor and stator. For photographic analysis a Canon® EOS 400D camera was used in combination with a Philips® PR 9113 stroboscope. For milliseconds timescale analysis a MotionPRO® IDT Y-series high-speed camera was used with a maximal frame rate of 1800 frames per second. Light intensity was enhanced by using 4 Dedocool high-intensity light sources.

2.3. Chemicals

n-Heptane (99% from Sigma–Aldrich) was used as liquid with the lower density. PMMA is resistant to n-heptane. Water was demineralized using a Millipore Elix UV-10 and used as liquid with the higher density. For visualization purposes, $5 \times 10^{-6}\ m^3$ water soluble blue ink was added to $2.2 \times 10^{-3}\ m^3$ of water. Pelican® 4001 royal blue fountain pen ink was used. GC–MS analysis showed that the water soluble ink does not dissolve into n-heptane. All experiments were performed at ambient lab temperature, 293 ± 2 K. The density, viscosity, and surface tension of n-heptane and water at 293 K are shown in Table 2 [9].

2.4. Experimental procedure selective through flow

To study the selective through flow of n-heptane through the reactor that was explained in Fig. 2, the reactor was initially completely filled with n-heptane. After addition of a predefined water volume, the n-heptane flow is set to $12.6 \times 10^{-6}\ m^3_{ORG}\ s^{-1}$. The n-heptane flow is fed to the reactor through inlet I in the bottom stator and flows out of the reactor though the outlet in the top stator. The rotational speed was increased from 0 to 400 RPM ($Re_\Omega = 1.88 \times 10^5$) by steps of 50 RPM. Rotation of the impeller induces a flow of liquid through the impeller: both n-heptane and water will flow through the impeller.

At the impeller outlet, at the rim of the impeller, there are two possibilities which are schematically shown in Fig. 2. In Case I the density difference between water and n-heptane causes separation of the water droplets and the n-heptane. In Case II, the water droplets are entrained in the n-heptane. Thus, either selective through flow of n-heptane is accomplished (Case I, Fig. 2) or n-heptane with entrained water droplets flows out of the reactor (Case II, Fig. 2).

2.5. Experimental procedure liquid–liquid flow

To study the liquid–liquid flow between the impeller and the bottom stator, the reactor was completely filled with n-heptane in absence of rotation. The aqueous volume fraction is equal to $0\ m^3_{AQ}\ m_L^{-3}$. Then a predefined amount of water was added to the reactor through inlet 2 (Fig. 1). The aqueous volume fraction in the reactor was calculated from the reactor volume and the injected water volumes. At a given aqueous volume fraction the rotational speed was increased stepwise from 0 to 300 RPM ($Re_\Omega = 1.41 \times 10^5$), with an increase of 25 RPM per step. At each rotational speed of the