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Short Communication

Visualization and characterization of gas–liquid mass transfer around a Taylor bubble right after the formation stage in microreactors

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HIGHLIGHTS

- Visualization of mass transfer around Taylor bubbles in microreactor.
- Contributions of the bubble formation stage to the overall gas–liquid mass transfer.
- The enhancement by the recirculation in the gas bubble at the formation stage was studied.
- New insight into bubble mass transfer right after the formation stage.
- Basis for elaborating a complete model, accounting for the bubble formation stage.

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ABSTRACT

The gas–liquid mass transfer occurring in Taylor flows right after the bubble formation stage were investigated in a flow-focusing microreactor. The colorimetric technique proposed by Dietrich et al. (2013) was used for locally visualizing and characterizing the gas–liquid mass transfer. Thanks to this method, the liquid-side mass transfer coefficients k_L were measured at the moment right after the bubble is detaching from the gas film near the cross-junction of the microreactor. Experiments were carried out for several flow conditions ($95.7 < Re < 226.1$, $0.0043 < Ca < 0.010$, $0.4 < We < 2.3$, $Bo = 0.044$) and bubble size ($2.34 < L_b/l < 5.59$). The results have demonstrated that the contribution of mass transfer right after the bubble formation stage is reasonably larger to those obtained at the bubble flowing-stage.

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1. Introduction

When compared with conventional reactors, microreactors present several advantages, such as high surface-to-volume ratio, excellent mass and heat transfer performances and narrow residence time distribution. Hence, they become an efficient technology for organic synthesis (Ehrfeld et al., 2000; Dietrich, 2011; Köhler and Cahill, 2014; Darvas et al., 2014). Gas–liquid reactions occupy a key place in scientific research and industrial application fields dealing with flow

chemistry. In such systems, it is essential to perfectly characterize and control the mass transfer between both phases insofar as, depending on chemical kinetics, it can become the limiting step and thus induce a decrease of the chemical conversion or selectivity.

In recent years, gas–liquid flows in micro- and milli-channels have been the subject of an extensive literature (Garstecki et al., 2006; Dietrich et al., 2008; Pohorecki et al., 2008; Sobieszuk and Pohorecki, 2010; Zhao et al., 2010; Kashid et al., 2011; Roudet et al., 2011; Wang et al., 2013; Pan et al., 2014; Zhu et al., 2014; Kastens et al., 2015). Most of these works are devoted to the understanding and modeling of the hydrodynamics associated with the Taylor flows, as this kind of flow enables to achieve the best gas–liquid mass transfer rates required for implementing

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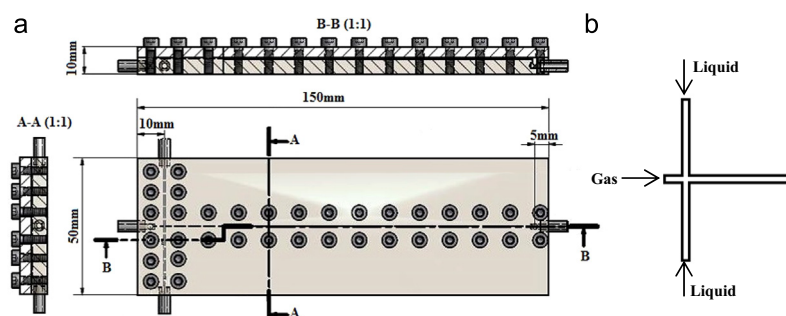


Fig. 1. (a) Geometry of the microchannel (Cross section: $A=l^2=500 \times 500 \mu\text{m}^2$) and (b) schematic representation of the cross-junction.

gas–liquid reactions. When investigated, the gas–liquid mass transfer characteristics are globally measured, namely by analyzing the solute concentration of samples collected at the outlet of microreactors. Such a method might lead to an inaccurate characterization, the sample collection and phase separation times being not usually taken into account. Few studies have also studied the separate contribution to mass transfer of the three characteristic stages, which are the bubble-forming, the bubble-flowing and the phase separation ones. Herein, it is necessary to propose online direct and more local approach to overcome these limitations and to isolate the contribution of the different stages. In this perspective, Tan et al. (2012) and Yang et al. (2014) have recently developed in situ methods, based on the time-dependent variations of the bubble sizes, to investigate the mass transfer of bubbly flows in T-junction and/or co-flowing microchannels. These authors have demonstrated that the mass transfer during the formation stage contributes significantly to the overall transferred solute, in particular with respect to the associated short mass transfer times. At present, there exists no report dealing with the direct visualization of mass transfer at microscale and just after the bubble pinch-off. Dietrich et al. (2013) have proposed a colorimetric technique (based on an oxygen-sensitive dye and without any laser excitation) to study the gas–liquid mass transfer of bubbles flowing in a millimetric square channel. In keeping with this work, the present study aims at applying this method to visualize and quantify the gas–liquid mass transfer occurring right after the bubble formation stage in a flow-focusing microchannel.

2. Materials and methods

2.1. Principles of the colorimetric technique

The method used was the colorimetric technique proposed by Dietrich et al. (2013). Resazurin (CAS 62758-13-8, molecular mass: $229.19 \text{ g mol}^{-1}$) was chosen as the dye which reacts with oxygen in the presence of potassium hydroxide (KOH) and glucose solution. In the reduced form, named dihydroresorufin, the solution is colorless, while in presence of oxygen, the oxidized form, named resorufin, is characterized by an intense pink color. To make possible the visualization of the colored field, the kinetics for the oxidation reaction should be quasi-instantaneous and the kinetics of the back reaction slow (few minutes). For that, an optimal composition of the catalyzer was determined, taking into account the balance between the oxidation and reduction kinetics, and also the requirements in terms of adequate color intensity levels. This led to potassium hydroxide and glucose solutions both diluted at 30 g L^{-1} in deionized water (the conductivity of the deionized water is $51.2 \mu\text{S m}^{-1}$), and to a concentration of resazurin fixed at 1 g L^{-1} ($\sigma_L=55.6 \text{ mN m}^{-1}$, $\mu_L=1.12 \text{ mPa s}$, $\rho_L=1004.5 \text{ kg m}^{-3}$). The latter concentration was 10

times larger than the one used by Dietrich et al. (2013) in order to conserve the same quality of images.

2.2. Description of the experimental set-up

The experimental set-up consisted of a straight horizontal channel of square cross section $A=l^2$ where $l=500 \mu\text{m}$ (4 times smaller than Dietrich et al. (2013)). The channel was curved in a polymethyl methacrylate (PMMA) plate (5 mm thick) and roofed over in a watertight manner by another plate (5 mm thick). The geometry of the channel is presented in Fig. 1.

Gas (air) and liquid phases (solution with dye, glucose and potassium hydroxide, flushed by nitrogen before the inlet of the channel) were both delivered from a 60 mL syringe in a syringe pump (Harvard Apparatus, PHD 22/2000, USA). Gas flow rates ranged from 0.6 to 1.2 mL min^{-1} and liquid flow rates from 2.4 to 4.0 mL min^{-1} in order to obtain a Taylor flow regime. The associated superficial gas j_G and liquid velocities j_L are: $0.040 \text{ m s}^{-1} \leq j_G = Q_G/l^2 \leq 0.080 \text{ m s}^{-1}$ and $0.160 \text{ m s}^{-1} \leq j_L = Q_L/l^2 \leq 0.267 \text{ m s}^{-1}$. All of the experiments were conducted at a room temperature of 293 K and atmospheric pressure.

2.3. Characterization of gas–liquid flow hydrodynamics and mass transfer

The shadowgraph method was applied to investigate the gas–liquid hydrodynamics and mass transfer inside the microchannel. The channel was lighted by a LitePad HO LED backlight (Rosco[®]) and the images were recorded by a monochromatic high-speed camera (Photron SA3) at 4000 frames per second. Image resolution was $10 \mu\text{m}$ per pixel and the region of interest $10 \times 1.5 \text{ mm}^2$. The software ImageJ (version 1.38e, National Institute of Health, USA) was used to analyze the recorded images. The following hydrodynamic characteristic parameters were measured: bubble length L_B , bubble velocity U , and the length of the unit cell L_{UC} .

The transfer of oxygen from air bubbles into resazurin solution was investigated over the range of gas and liquid flow rates defined in Section 2.2, which covered only the Taylor flow regime ($95.7 < Re < 226.1$, $0.0043 < Ca < 0.010$, $0.4 < We < 2.3$, $Bo = 0.044$). As the camera is monochromatic, the different levels of pink coloration taken by the dye solution, which depended only on the quantity of oxygen transferred, were represented by 255 gray levels on the acquired images.

The gas–liquid mass transfer characteristics were determined by an image post treatment algorithm implemented on the software Matlab (R2011b), as the one used by Dietrich et al. (2013). It consisted of two steps: (i) the determination of the calibration curve, and (ii) the image processing which enabled to transform the acquired gray-level images into the equivalent oxygen concentration fields. Note that the term “equivalent” is used as in reality the oxygen concentration is null as consumed by the instantaneous oxidation reaction. For the calibration process,

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