**Characterization of gas-liquid flow photoreactors at the micro- and milli-scale**

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**Highlights**

* Photon flux per liquid volume increases with gas transport fraction.
* Optical pathlength is smaller in gas-liquid phase in comparison to single phase.
* Actinometry has potential for quantification of scattering and liquid residence time.

**1. Introduction**

Flow photo microreactors are a promising tool for gas-liquid photoreactions such as photooxidations and fluorinations due to their small penetration depth and the promotion of segmented flow which leads to good mass transfer of the gas in the liquid [1,2]. However, reaction condition optimization in microreactors and scale-up to milli-reactors requires an extensive parameter study [4,5]. Nevertheless, the optimal light source intensity, reagent concentration and total flow rate are determined by photon transport and hydrodynamics in the employed gas-liquid photoreactors. When these phenomena are understood at a fundamental level, the optimization and the scale-up procedure become faster and cost efficient. Chemical actinometry is an important tool to characterize photoreactors and was previously used to determine the optical pathlength and photon flux in single liquid phase in a microstructured reactor [6]. However, the observations found in single-phase may not apply to two-phase flow due to the light scattering at the gas-liquid interface and hydrodynamics determined by the presence of the gas bubbles. The aim of this study is to experimentally determine the optical pathlength and photon flux in gas-liquid micro- and milli-scale photoreactors and compare it with the case of single liquid phase.

**2. Methods**

The microreactor used in this work consists of a glass plate with a serpentine channel characterized by a volume of 0.6 mL and a diameter of 1 mm. The milli-scale reactor is a Corning Advanced-Flow G1 Photo Reactor which is composed of heart-shaped elements and is characterized by a volume of 8 mL and a channel height of 1.1 mm. Both reactors are irradiated by green Light-Emitting Diodes (LEDs). The photon flux and the optical pathlength are experimentally determined by visible-light actinometry following a methodology previously reported [6]. The actinometric measurements are performed in single liquid phase and in gas-liquid two-phase flows. The total flow rate is 1.3 mLmin‑1 in the microreactor and between 35 and 42 mLmin-1 in the milli-scale reactor. Nitrogen is used as inert gas with volumetric gas transport fractions (*β*G) comprised between 0.2 and 0.9 in the microreactor and between 0.2 and 0.5 in the milli-scale reactor.

**3. Results and discussion**

To ease the comparison, the values of the photon flux per liquid volume obtained in two-phase flow are normalized by the values found in single phase. As shown in Figure 1, the photon flux in two-phase flow experiences an exponential increase with the gas transport fraction reaching 240% at *β*G=0.9 in the microreactor and 54% at *β*G=0.5 in the milli-scale reactor. This increase can be caused by light scattering. Another possibility is that the liquid residence time is higher than considered in calculations due to the slip and occurrence of local back flow through the liquid films [7]. Currently, a residence time distribution investigation is carried out in order to experimentally determine the mean liquid residence time. Moreover, it was found that the optical pathlength in two-phase flow is 31% smaller at *β*G=0.9 compared to the single phase microreactor and 17% smaller in the milli-scale reactor irrespective of the gas fraction.

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**Figure 1.** The variation of normalized photon flux per liquid volume with the gas transport fraction (*β*G) in the microreactor and the milli-scale reactor. The insets illustrate the flow pattern at *β*G = 0.88 in the microreactor and at *β*G = 0.52 in the milli-scale reactor.

**4. Conclusions**

Our study shows that the presence of gas bubbles has a significant impact on the amount of photons received by the liquid phase and on the distance travelled by photons within the reactor at both micro- and milli-scale. The correlation of the actinometric measurements with the photon transport and hydrodynamics could extend the application of actinometry to the quantification of light scattering and liquid residence time in two-phase photoreactors.

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