

READY STEADY (BUBBLE) FLOW! PREDICTIVE CONTROL OF MIXING, MASS TRANSFER AND RESIDENCE TIMES IN SEGMENTED FLOW

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ABSTRACT

Utilizing the benefits of microfluidic segmented flow systems, e.g., enhanced mixing, mass transfer and reduced axial sample dispersion, usually requires specialist knowledge and accommodating a few experimental difficulties. The currently microfluidic segmented flow experimental setups often require more than 10 min for flow stabilization. Moreover, In order to maintain or adjust the desired residence time or mixing behaviour, repeated manual incremental adjustments of experimental conditions may be required. Here we report an image-based feedback strategy that overcomes the aforementioned challenges and allows gas-liquid and liquid-liquid segmented flows to be rapidly and precisely achieved without a need for the manual control of experimental conditions. We demonstrate how the proposed image-based cruise control method can routinely and consistently establish segmented flows. As a result, the key flow parameters such as mixing, mass transfer and residence times can be directly imposed rather than manually adjusted.

KEYWORDS

Segmented flow, cruise control, image-based feedback, mass transfer, automated microfluidic platform.

INTRODUCTION

The flow of uniformly long gas bubbles through microchannels (segmented flow) has become popular due to the shortened mixing and mass transfer times and the reduced axial dispersion. Two challenges however limit its widespread use by non-specialist users: (a) the long times required to obtain stable flow conditions and (b) the dynamic and its non-linear nature [1, 2] that makes it difficult to exert direct control over flow parameters such as the gas bubble length, L_B , liquid slug length, L_S , and gas bubble velocity, U_B (Fig. 1). The main experimental challenge is applied by the lack of directly adjusting the desired mixing and residence times in segmented flow microfluidic systems. The corresponding segmented flow parameters, that are the liquid volume and bubble velocity, dynamically adjust in response to the selected experimental conditions that are liquid flowrate, Q_L , and gas inlet pressure, P_G . Moreover, as many applications involve dynamic changes in the composition of the gas or liquid, those therefore might change the fluid viscosity and surface tension, segmented flow parameters can be expected to be affected. Different correlations that are largely limited to one experimental setup and gas-liquid pair were proposed to predict these parameters [2, 3].

Here, we demonstrate a robust strategy that (a) practically removes unwanted stabilization times and (b) provides direct control over L_B , L_S , and U_B and therefore the mixing, mass transfer and residence times for the given microchannel dimensions. Figure 1b compares the required time to establish a stable gas-liquid segmented flow for different gas delivery methods, and demonstrates our strategy reduces the experimental time 20-30 folds as compared to present manual approaches.

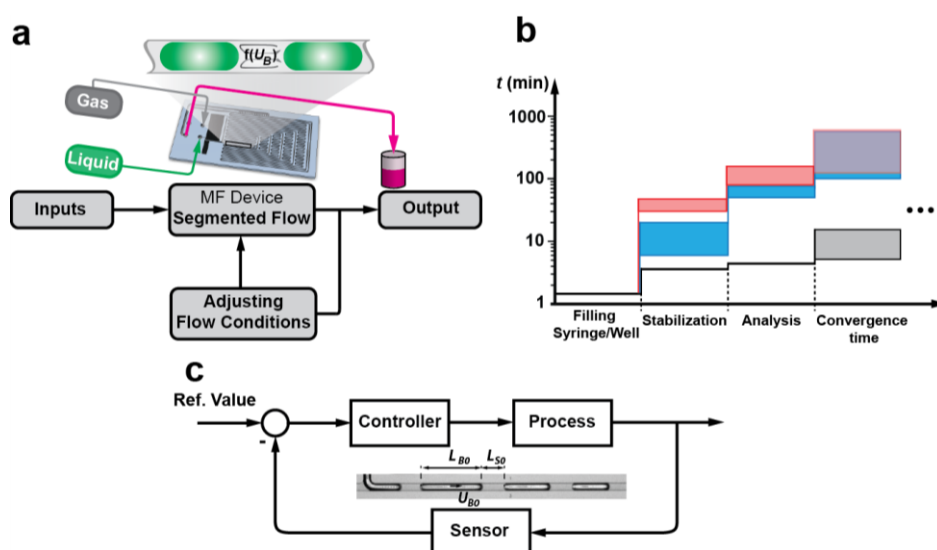


Figure 1 (a) Schematic of segmented flow systems. Mixing time inside the liquid slugs and the residence time can be adjusted by controlling U_B . (b) Comparison of the process time for different gas delivery methods in a segmented flow system. Blue: manual pressure regulator and mass flow controllers, red: syringe pump, black: this study-automated digital pressure regulator. (c) Image-based sample data feedback strategy using a modified discrete time integral controller is illustrated. The initial segmented flow parameters are highlighted within the inset micrograph.

The proposed strategy is independent of the experimental setup, microfluidic device substrate (e.g. polydimethyl–siloxane, Silicon, PMMA, glass or thermoplastics), microfluidic device design (T-junction, Y-junction or flow focusing geometries), fluid type, temperature or pressure and presence of surfactants in the liquid. The convergence time required to reach the desired segmented flow parameter to the target value defined by the user varies between 1 to 5 min.

EXPERIMENTAL

Our automated microfluidic (MF) platform employs an image-based sample data feedback strategy that consists of a modified discrete time integral controller (Fig.1c) to control L_{B0} , L_{S0} and U_{B0} (subscript “0” indicates the initial condition after T-junction) in a gas-liquid segmented flow system. Figure 2 shows the schematic of the experimental setup that includes a silicon-based microfluidic device, an automated syringe pump, an automated digital pressure regulator and a CMOS based high speed camera. The silicon-based microfluidic device (pressure drop sections: $75\mu\text{m}$ wide by $75\mu\text{m}$ deep, 60 cm long and multiphase section: $300\mu\text{m}$ wide by $300\mu\text{m}$ deep, 85 cm long) was fabricated using shadow mask lithography, front and back side DRIE and a wafer level anodic bonding to a Borofloat 33 glass wafer. A custom developed Matlab-based image processing code using Canny edge detection method was developed to analyze the acquired segmented flow images and measure the desired segmented flow parameters. The image-based feedback strategy uses a real-time image processing technique to converge the selected segmented flow parameter to the defined value, and no initial screening is needed.

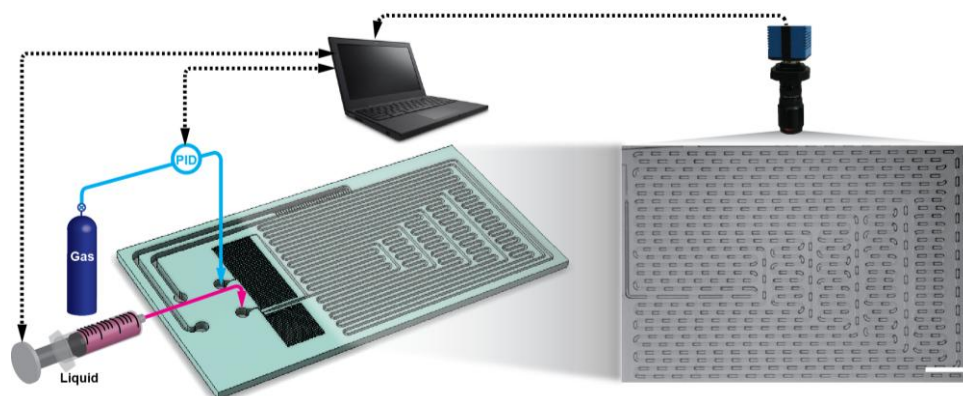


Figure 2 Schematic of the experimental setup including an automated syringe pump, a digital pressure regulator and a high speed CMOS camera. Scale bar is 5 mm.

RESULTS

The proposed cruise control strategy was utilized to converge the initial segmented flow parameters to the desired values defined by the user. Figure 3 shows the convergence plots using the image-based feedback algorithm, to maintain the initial segmented flow parameters for a syringe pump driven gas-liquid segmented flow system.

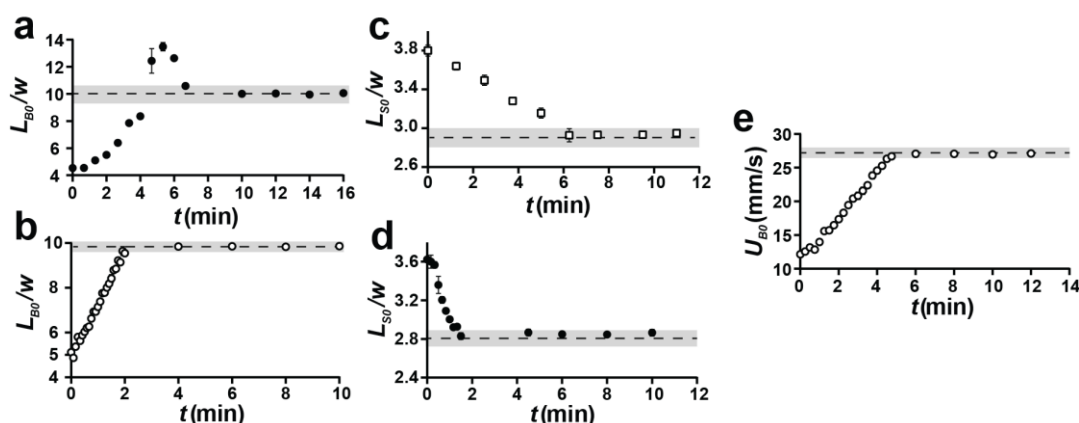


Figure 3 Convergence plots for the syringe pump driven system. (a) Initial bubble length evolution while converging to the target value $L_{B0}/w=10\pm0.5$. Initial $Q_L=25\ \mu\text{l}/\text{min}$, $P_G=1.0\ \text{psig}$, time delay=40 s, Q_L increments= $3\ \mu\text{l}$. (b) Initial bubble length evolution while converging to the target value $L_{B0}/w=9.8\pm0.2$. $Q_L=25\ \mu\text{l}/\text{min}$, Initial $P_G=1.0\ \text{psig}$, time delay=5 s, P_G increments= $0.05\ \text{psig}$. (c) Initial liquid slug length evolution while converging to the target value $L_{S0}/w=2.9\pm0.1$. Initial $Q_L=25\ \mu\text{l}/\text{min}$, $P_G=1.0\ \text{psig}$, time delay=75 s, Q_L increments= $3\ \mu\text{l}$. (d) Initial liquid slug length evolution while converging to the target value $L_{S0}/w=2.8\pm0.05$. $Q_L=25\ \mu\text{l}/\text{min}$, Initial $P_G=1.2\ \text{psig}$, time delay=10 s, P_G increments= $0.05\ \text{psig}$. (e) Initial bubble velocity evolution while converging to the target value $U_{B0}=27\pm0.5$. $Q_L=15\ \mu\text{l}/\text{min}$, Initial $P_G=1.0\ \text{psig}$, time delay=15 s, P_G increments= $0.05\ \text{psig}$. Working fluids are nitrogen and ethanol.

Figures 3a, c, and e illustrate the inlet gas pressure, P_G , constant cases and Figs. 3b and d are the liquid flowrate, Q_L , constant cases. As shown in Fig.3a, even if the measured value overshoots the target value \pm threshold value, the feedback algorithm will adjust the experimental conditions to bring the desired segmented flow parameter back to the target value. The proposed system can also be used for the case that liquid is driven by pressure head from a local reservoir close to the microfluidic device.

In a case study, the effect of U_{B0} on the mass transfer time, $(K_{La})^{-1}$, where K_{La} is the volumetric gas-liquid mass transfer coefficient, was studied for a highly soluble gas-liquid pair, CO_2 and dimethyl carbonate (DMC) (Fig.4). The image-based cruise control strategy was used to maintain L_{B0} constant while increasing U_{B0} , and thereby increasing the capillary number. Figure 4a shows carbon dioxide bubble lengths evolutions in the flow direction at a constant temperature ($T=298\text{ K}$). Between 3 to 5 min were required for the image-based feedback algorithm to converge to the defined L_{B0} . The volumetric mass transfer coefficient of CO_2 -DMC was measured as described previously [4].

Figure 4b shows the measured mass transfer times for a one order of magnitude change in U_{B0} . The decrease in the measured $(K_{La})^{-1}$ values with the increase of U_{B0} is attributed to the enhanced recirculation inside liquid slugs.

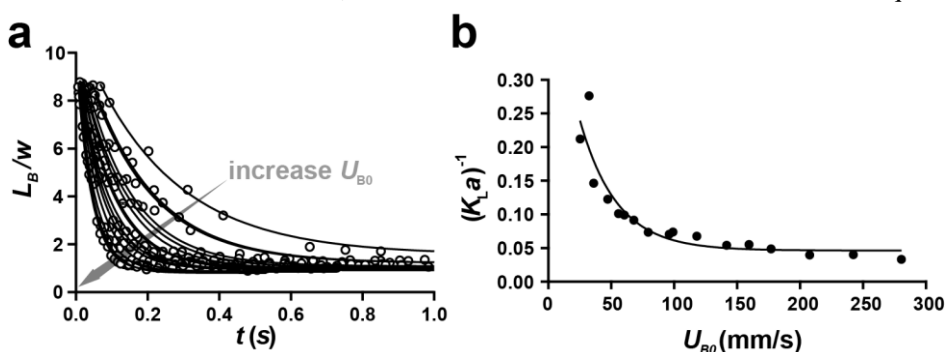


Figure 4 (a) Carbon dioxide bubble length evolution in the silicon based microfluidic device for the same L_{B0} while changing the U_{B0} . (b) Measured mass transfer times $(K_{La})^{-1}$ for CO_2 -DMC over a one order of magnitude change of U_{B0} for the same L_{B0} . The initial bubble length used for all the experiments was $L_{B0}/w=8.5\pm 0.1$.

The presented modified discrete time integral controller of the image-based feedback strategy can also be implemented into a liquid-liquid segmented flow system with different droplet breakup geometries such as T, Y and K-junctions and flow focusing. Dispersed phase plug length, continuous phase slug length and dispersed phase plug velocity can be adjusted using the automated cruise control strategy. In addition, using a single camera, multiple parallel segmented flow lines (gas-liquid or liquid-liquid) can be controlled simultaneously in a scaled-up integrated microfluidic system.

CONCLUSIONS

A cruise control strategy for gas-liquid and liquid-liquid segmented flows using a sample data image-based feedback algorithm was demonstrated. A real-time Matlab-based image-processing code in combination with an automated digital pressure regulator and an automated syringe pump were employed to control the desired segmented flow parameters in a gas impermeable (silicon) microfluidic device. The image-based feedback approach can also be extended to a head pressure-driven based flow and liquid-liquid segmented flows. As a case study the effect of initial plug velocity on the CO_2 -DMC mass transfer time was studied for a fixed initial CO_2 plug length. As it was expected, increasing the mixing strength inside the liquid slugs (i.e. increasing the stirring speed inside the liquid slugs) decreased the total mass transfer time of the CO_2 molecules from the gas bubbles into the DMC slugs. We expect the proposed cruise control strategy will enable plug'n play operation of gas-liquid and liquid-liquid segmented flow microfluidic systems in different applications that include flow chemistry, in-flow analysis and screening, and material synthesis.

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