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## Liquid-liquid mixing using micro-fluidised beds

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#### ABSTRACT

This study experimentally investigates the application of a solid–liquid micro-fluidised bed as a micro-mixing device. The experiments were performed in a borosilicate capillary tube with an internal diameter of 1.2 mm (i.e. near the upper-limit dimension of a micro-fluidic system) using borosilicate particles with a mean diameter of 98 µm. Refractive index matching technique using sodium iodide solution was employed to achieve a transparent fluidised bed. Mixing performance of the micro-fluidised bed in terms of mixing time was investigated using a dye dilution technique. Experiments were carried out in the creeping flow regime at Reynolds numbers ranging between 0.27 and 0.72. It was demonstrated that the micro-fluidised bed mixing time sharply decreases as the Reynolds number increases. That is because at relatively high Reynolds numbers, the particle oscillation is stronger creating larger disturbances in the flow. The energy dissipation rate in micro fluidised bed was estimated to be four orders of magnitude less than other passive micro mixers which operate in the turbulent regime. It was also demonstrated that the ratio of mixing time and the energy dissipation rate for fluidised bed micro-mixer was found to operate at much lower Reynolds numbers compared to other passive mixers, with a mixing time of the order of few seconds. © 2013 The Institution of Chemical Engineers. Published by Elsevier B.V. All rights reserved.

*Keywords*: Micro-fluidised bed; Micro-mixer; Liquid–liquid mixing; Mixing time; Refractive index matching technique; Dye dilution technique, Energy dissipation

#### 1. Introduction

Application of micro-fluidic devices for processing of multiphase flows in areas such as medical diagnostics, chemical analysis, power generation and fuel processing, invariably relies upon the physical or chemical interaction between at least two fluid phases. Such interaction is achieved through effective mixing. However, at length-scales associated with micro-fluidic devices where systems operate in a laminar regime with Reynolds numbers typically less than 1, the mixing process is rather poor as it is principally governed by molecular diffusion.

Generally, active or passive techniques are employed to achieve effective mixing at micro-scales (Hessel et al., 2005; Nguyen, 2012). In active mixing external forces including ultrasound, acoustic, electrokinetic, and magneto-hydrodynamic forces are used to induce mixing. In these mixers the transversal disturbances generated by external fields lead to instabilities at the interface between the two mixed phases. Passive mixing on the other hand is accomplished by maximising the mixing contact area and/or reducing the mixing path through "Streaming Techniques" where the flow path is restructured using geometrically-based methods.

There are two main mechanisms namely the diffusive transport and chaotic advection which drive mixing in passive micro-mixers. Diffusive mixing is often improved by decreasing the striation thickness through optimisation of the geometrical designs to create parallel or sequential laminations, sequential segmentation and focusing of fluid flow. Similarly, chaotic advection is generated by modifying the channel shape for stretching, folding and breaking of the laminar flow. The aim is to promote the streamlines to cross each other periodically (i.e. a spatially periodic flow). For example, the use of obstacles in passive mixers is demonstrated to be effective in generating such advections (Nguyen, 2012; Wang et al., 2002). At Reynolds numbers greater than 100, vortices are generated as the liquid is passing by an obstacle. These vortices disrupt the laminar flow pattern and induce transversal

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Nomenclature	
C <sub>1</sub>	proportionality constant in Eq. $(3)$ (m <sup>-1</sup> )
Cy	fitting parameter in Eq. (6)
d	capillary internal diameter (m)
D	molecular diffusivity (m/s²)
Ι	grey scale intensity of individual pixel in
	shadow image
$I_{x,\infty}$	intensity for perfect mixing
I <sup>*</sup>	normalised intensity of individual pixel in
	shadow image
L	length of capillary (m)
L <sub>x</sub>	chord length (m)
М	percentage mixing index
Ре	Peclet number
Re	Reynolds number
$Q_d$	dye stream flow rate (μL/min)
QT	total flow rate (μL/min)
Q <sub>c</sub>	clear stream flow rate (μL/min)
tm	mixing time (s)
t <sub>diff</sub>	diffusion time, $d^2/D$ (s)
х	distance from the centre of capillary in horizon-
	tal direction (m)
у	distance from the inlet of capillary in vertical
	direction (m)
Greek symbols	
γeffective	effective shear rate (1/s)
$\gamma_{ ext{total}}$	total shear rate (1/s)
$\delta_0$	striation thickness (m)
$\Delta P$	pressure drop (kPa)
$\sigma_{\rm I,0}$	deviation from concentration profile for perfect
	mixing at inlet (y = 0)
$\sigma_{\mathrm{I},\mathrm{y}}$	deviation from concentration profile for perfect
	mixing at y
$\sigma_{\mathrm{I},\infty}$	deviation from concentration profile for perfect
	mixing at infinity
ε	energy dissipation rate (w/kg)
η	mixing efficiency
11	kinematic viscosity $(m/s^2)$

kinematic viscosity (m/s<sup>2</sup>)

transport, in turn, improving the mixing. At Reynolds numbers less than 100, the lateral mass transport induced by the obstacles leads to mixing. Besides the fabrication complexity, the main drawback associated with the presence of obstacles, however is the rise in pressure drop across the bed.

Clearly, the existing passive and active techniques suffer from fabrication complexity which limits their mass productions, high fabrication and maintenance costs, and high energy dissipation rates (Hessel et al., 2005; Nguyen, 2012). To overcome the shortcomings of the above techniques, a fluidising-based micro-mixer system is considered here.

It is well-established that fluidisation provides efficient mixing and intensification of mass and heat transfer. In recent years, a number of studies focusing on miniaturised fluidised beds have emerged (Doroodchi et al., 2012; Potic et al., 2005). The major focus of these studies was to establish the fluidisation hydrodynamic characteristics of the fluidised beds both experimentally and theoretically. The focus of this work however is to experimentally investigate the mixing performance of two miscible fluids in a miniaturised solid–liquid fluidised bed in terms of mixing time and mixing efficiency. The relative performance of the fluidising-based micro-mixer compared with typical existing passive micro-mixers is also established.

It should be highlighted that apart from non-reacting flows the abovementioned miniaturised fluidised-beds can be also used as effective platforms in reacting flow systems, for example micro-reactors, where the mixing agents undergo a series of chemical reactions.

#### 2. Experimental

A schematic diagram of the experimental setup is presented in Fig. 1. The setup consisted of (i) a 30 cm glass capillary tube with an internal diameter of 1.2 mm (i.e. a dimension near the upper-limit dimension of 1 mm for micro-fluidic systems), (ii) a fluid reservoir, (iii) two syringe pumps, (iv) a LED light source and focusing lens, (v) a CCD camera, and (vi) a data acquisition system. The bed material was clear borosilicate glass sphere from Cospheric LLC with a size range of 90–106  $\mu$ m and mean diameter of 98  $\mu$ m, particle density of 2230 kg/m<sup>3</sup>, and refractive index of 1.47–1.48. A 52  $\mu$ m wire mesh was used as a distributor.

For optical diagnostics, the refractive index of the fluidising medium (i.e. sodium iodide solution) and bed material were matched (forming a transparent fluidised bed) minimising the noise generated by the light reflections from the surface of particles. Dye dilution technique (Nguyen, 2012) was used to determine the mixing performance at various operating conditions given in Table 1. In this work, the tracer dye stream (i.e. dye + sodium iodide solution) was fed in the centre from the bottom of the capillary tube through a 30G<sup>1</sup>/<sub>2</sub> needle whilst the sodium iodide solution (56 wt%) was fed annularly passing through the fluid reservoir. The two streams have similar density (1800 kg/m<sup>3</sup>) and viscosity (0.0018 Pa.s). A LED lamp combined with a biconvex focusing lens was used to illuminate the region of interest using a back lighting technique. A cuvette filled with the sodium iodide solution was placed around the capillary tube to reduce the light refraction at the curved surface of the tube. An IDT XS3 high speed camera was then used to capture the dye dilution profile across the channel cross section at a rate of 50 Hz. 2000-3000 images were captured using IDT Motion Studio software whilst MAT-LAB software was employed for image analysis. Background images were also obtained at dye flow rates of zero at the start of each experiment. For benchmark purposes, the same experiments were carried out in the empty capillary tube (i.e. fluidised bed with no particles).

The variation of the dye concentration profile along the length of capillary tube has been calculated by processing the images recorded using the procedure described above. The criterion used to evaluate the performance of the liquid-liquid mixing within micro-mixers was the uniformity of the dye intensity. The concentration of the dye was estimated from the shadow image generated by the diffusing jet of opaque dye stream using image processing in MATLAB software. The background images obtained at the start of each experiment was averaged and subtracted from individual mixing images. In a shadow image, the pixel intensity is inversely proportional to the concentration of dye at that point. Therefore a negative of each image was taken after the background subtraction. The background intensity was non-uniform. This non-uniformity implies that direct calculation of local concentration using extinction of the backlight is erroneous. To compensate for the gradient in backlight, normalisation of image intensity based

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