Confocal imaging of laminar and turbulent mixing in a microscale multi-inlet vortex nanoprecipitation reactor

Yanxiang Shi  
*Iowa State University*

Rodney O. Fox  
*Iowa State University*, rofox@iastate.edu

Michael G. Olsen  
*Iowa State University*, mgolsen@iastate.edu

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Abstract
Mass production of functional nanoparticles may be realized through flash nanoprecipitation in microscale reactors such as the multi-inlet vortex reactor (MIVR). A comprehensive understanding of mixing in the MIVR is required for process control and reactor design. Mixing in the MIVR is studied using a technique coupling laser induced fluorescence with confocal laser scanning microscopy. It is shown to provide meaningful qualitative and statistical data of the scalar field for analysis and comparison with numerical simulations. Data were collected for four flow rates, showing that mixing is incomplete even at the highest flow rate.

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Confocal imaging of laminar and turbulent mixing in a microscale multi-inlet vortex nanoprecipitation reactor

Yanxiang Shi,1 Rodney O. Fox,1 and Michael G. Olsen2,a)
1Department of Chemical and Biological Engineering, Iowa State University, Ames, Iowa 50011, USA
2Department of Mechanical Engineering, Iowa State University, Ames, Iowa 50011, USA

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Mass production of functional nanoparticles may be realized through flash nanoprecipitation in microscale reactors such as the multi-inlet vortex reactor (MIVR). A comprehensive understanding of mixing in the MIVR is required for process control and reactor design. Mixing in the MIVR is studied using a technique coupling laser induced fluorescence with confocal laser scanning microscopy. It is shown to provide meaningful qualitative and statistical data of the scalar field for analysis and comparison with numerical simulations. Data were collected for four flow rates, showing that mixing is incomplete even at the highest flow rate. © 2011 American Institute of Physics. [doi:10.1063/1.3662042]

Flash nanoprecipitation (FNP)1–4 is a technique for synthesizing functional nanoparticles with narrow particle size distribution (PSD). Such nanoparticles have applications in cosmetics, pesticides, and pharmaceuticals.5–10 FNP employs supersaturation generated by mixing organic active solution with an antisolvent to gain precise control on PSD and particle morphology.11 Rapid mixing is crucial to this process to ensure a small precipitation Damköhler number.1,12

Microscale reactors such as the confined impinging-jets reactor (CIJR)12–14 and multi-inlet vortex reactor (MIVR)9,15 are used in FNP. In the CIJR, a highly turbulent region is created where the two inlet jets impinge, resulting in successful nanoparticle generation.12 The MIVR (bottom of Fig. 1) offers flexibility of operation compared to the CIJR since it does not require equal momenta from opposing streams. Previous research on the MIVR includes Liu et al.’s15 investigation into the conversion of 2,2-dimethoxypropane in a competitive reaction system, Shen et al.’s investigation of self-assembled polymeric nanoparticles, and Cheng et al.’s9 particle image velocimetry measurements and large-eddy simulations. The present work complements these studies by providing data on passive scalar mixing within the MIVR over a range of Reynolds numbers.

Planar laser-induced fluorescence (PLIF) has been a primary technique for studying turbulent mixing at the macroscale.17–19 By using a fluorescent dye in one or more of the inlet streams, mixing of a scalar in a small volume illuminated by a thin laser sheet can be measured. However, when applied to microscale reactors that are of the same dimensions as the laser sheet, PLIF fails. This difficulty can be overcome using confocal laser scanning microscopy (CLSM) where a pinhole is placed in front of the detector to reject out-of-focus light.20,21 Two rotating mirrors are used to scan the entire imaging region in a point-by-point manner using a photomultiplier tube (PMT), generating a planar image.

A sketch of the reactor and the experimental setup for confocal μ-LIF is illustrated in Fig. 1. The MIVR has a mixing chamber diameter (2R) of 6.26 mm and height (H) of 1.78 mm. Other dimensions are outlet channel diameter 2r = 1.40 mm and inlet channel width w = 1.19 mm. Four inlet channels are tangentially attached to the mixing chamber with the outlet channel protruding in the third dimension. Two syringe pumps are used to deliver the four inlet streams. Two opposing inlet streams are pure ethanol whereas the other two contain the fluorescent dye Rhodamine 6G with a concentration of 600 μg/L. Calibration showed a linear relationship between the fluorescence intensity and dye concentration up to 700 μg/L. The MIVR rests on the stage of the inverted microscope installed with a 5 × 0.15 NA objective. Excitation light is provided by a white light laser (WLL) with three laser lines (500 nm, 513 nm, and 525 nm) outputting at 100%.

Confocal μ-LIF data are collected at four inlet stream Reynolds numbers, Rej = 10, Rej = 53, Rej = 93, and Rej = 240, on two different planes, the midplane at half the chamber height and a plane within the outlet channel (see Fig. 1, where h = 0.95 mm). The CLSM scans a single measurement volume (or pixel) in 4.88 μs, thus each individual

![FIG. 1. (Color online) Schematic of experimental setup and the MIVR.](image-url)
measurement represents the temporally averaged concentration within each measurement volume over 4.88 μs. Forming a $512 \times 512$ image requires 1.28 s. To facilitate rapid collection of data for temporal and statistical analysis, the system is operated in line-scanning mode where a specified horizontal line is scanned repeatedly to form a profile. The profile presented here is along the center line of the reactor (marked by the dash-dotted line in Fig. 1). For the temporal analysis, over 10,000 line-scans were performed consecutively and 512 of those were chosen at fixed intervals to form $512 \times 512$ images providing a visualization of the temporal evolution of the flow. In these images, the vertical direction is time $t$ starting from the top.

For statistical analysis of the data, an individual measurement of any point along the profile is considered an individual realization for that point. All realizations are normalized against the pure dye signal after the background signal is subtracted. Ensemble averaged mean and root mean square (RMS) profiles of normalized dye concentration can then be calculated. Instantaneous full field images (of size $3.10 \text{mm} \times 3.10 \text{mm}$ marked by the shaded area in Fig. 1) were also collected. However, due to the finite time required to build an image, these images do not represent a true instantaneous “snapshot” of the flow and unsteadiness in the flow can distort the images. Nevertheless, they do provide a visualization of the flow at lower Reynolds numbers, but are no longer informative when the flow becomes turbulent and unsteady, as in the $Re_j = 240$ case.

Results for $Re_j = 10$ are shown in Fig. 2. At $Re_j = 10$, the flow is laminar and steady. The fluid has low tangential velocity, and the effects of viscosity are significant. Instead of spiraling towards the exit, the fluid heads nearly directly towards the exit. No large scale stirring occurs and mixing occurs only on the short interfaces between the incoming streams by

![Fig. 2. Full-field (top row) and line-scan (bottom row) results from the mid-plane (left column) and outlet (right column) at $Re_j = 10$.](image)

![Fig. 3. Results for $Re_j = 53$. Panel arrangements as in Fig. 2.](image)

![Fig. 4. Results for $Re_j = 93$. Panel arrangements as in Fig. 2.](image)

![Fig. 5. Results for $Re_j = 240$. Panel arrangements as in Fig. 2.](image)
diffusion. Fluid exits the reactor nearly as segregated as it enters. In the time-evolving profiles at the bottom of Fig. 2, the nearly straight interfaces reflect the steadiness of the flow.

At Re$_j$ = 53 the flow exhibits more intricate patterns as shown in Fig. 3, such as the inner “tai-chi” pattern and the circular streaks around it. At the midplane, the flow spirals towards the center due to the greater tangential velocity resulting in bands that grow thinner due to the fluid accelerating as it approaches the center. Also, the flow is no longer steady. As seen in the midplane time-evolving profile in Fig. 3, the interfaces move back and forth periodically, especially in the outer region. Over the 25.5 s that data were collected, approximately 5 cycles are completed. In the outlet where the fluid is more confined, the motions of the interfaces are less pronounced. The flow is still laminar, and poor overall mixing is observed.

Previous research has suggested that Re$_j$ = 93 is near the transition from laminar to turbulent flow. However, the confocal $\mu$-LIF data indicate that even at Re$_j$ = 93, the mixing is poor. The midplane of the mixing chamber is shown in the top left of Fig. 4. The outer part of the flow appears nearly at a uniform concentration, yet in the center, the fluid appears unmixed. The near uniformity in the outer region of the mixer is due to the “bands” of dyed and undyed fluid spiraling towards the center being so thin that the imaging resolution of the confocal $\mu$-LIF system is insufficient to individually resolve them. The poor mixing at Re$_j$ = 93 is evident in the results both near the center of the midplane and in the outlet. Similar behaviors are observed in the time-evolving profiles for Re$_j$ = 53 in that the wavy motion of the interfaces still exists. However, at Re$_j$ = 93 more cycles are completed within the same time period with nearly 9 cycles observed.

At Re$_j$ = 240 the flow becomes truly turbulent. As seen in Fig. 5, because of the rapidly varying flow field, CLSM fails to capture a meaningful full field image. Also, in both the full field images and the time-evolving profiles, instead of flow structures, i.e., the “bands” and the “tai-chi” pattern, that appear in previous cases, there is now everywhere a noisy blur. Meaningful results at Re$_j$ = 240 can only be obtained through statistical analysis.

The ensemble averaged mean and RMS profiles of normalized concentration are compared between Re$_j$ = 10 and Re$_j$ = 240 in Fig. 6. At Re$_j$ = 10, the concentration is nearly binary with values of either zero or one at the midplane. A similar trend is observed in the outlet. The RMS values at the interfaces between dyed and undyed fluid are due to small unsteady motions of the interfaces caused by the mechanical behavior of the syringe pumps. At Re$_j$ = 240, at the center of the midplane, a pair of peaks with mean concentrations of 0.7 are symmetric around the reactor center. Note that the entire mean concentration profile being above 0.5 is simply a consequence of the profile chosen for analysis. Different profiles would have mean concentrations below 0.5. The mean profile in the outlet suggests that while mixing is improved at Re$_j$ = 240 it is by no means complete. Complete mixing would be indicated by a mean concentration of 0.5 and an RMS concentration of zero throughout the outlet, neither of which is observed.

In summary, confocal $\mu$-LIF was applied to the MIVR to study mixing spanning the laminar through turbulent regimes. Pointwise data were ensemble-averaged to provide mixing performance data for flow rates that were too large for full-field images or time-evolving profiles to provide meaningful results. The results indicate that even at the highest flow rate investigated, the reactor does not achieve complete mixing at the outlet.

References: