Micro-scale Laser-induced Fluorescence Thermometry for Multiphase Flow of Immiscible Liquids

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ABSTRACT

In this work an implementation of laser induced fluorescence thermometry technique at the micro-scale for application in multiphase flow of two immiscible liquid is presented. The selected fluid phases are water and n-decane. One key limiting factor for dye selection is that each dye must be soluble in only one of the liquid phases. The selected fluorescent dyes are sulforhodamine 101 (SR) and eosin Y (EY) in water, and pyrromethene 597-8C9 in n-decane. The technique uses ratiometric two-color two-dye approach for water and two-color one-dye approach for n-decane. The two imaging wavelength bands are 540–572 nm and 610–640 nm. The technique yields a measurement sensitivity of 1.7% K⁻¹ in water and ~0.1% K⁻¹ in n-decane in the temperature range 20–60 °C with a spatial and temporal resolution of $5.2 \,\mu$ m and 1 s, respectively. The sensitivity and uncertainty level achieved herein are satisfactory and near expected values for the aqueous phase. For the non-aqueous phase, modifications in the optical setup and/or choice of fluorescent dye(s) are needed to improve the sensitivity of the measurement technique.

1 Introduction

Microfluidic devices are used in miniaturized systems for analysis and synthesis of chemicals, and preparation and testing of biological samples. In these devices, mass and momentum transport and chemical reactions occur in micro-scale geometries under controlled conditions. One of the important variables to be controlled is temperature and its spatiotemporal variations, since temperature and its gradients experienced by the fluids play a key role in transport processes and chemical reactions (Stone & Kim, 2001). Microfluidic devices are also used for flow in porous media research, where optically transparent artificial microfabricated porous media, termed *micromodels*, are fabricated to mimic real porous media (Gerami *et al.*, 2019; Kazemifar *et al.*, 2015, 2016; Li *et al.*, 2017). Heat transfer in porous media plays a key role in many environmental and energy-related problems. Applications include heat transfer in geothermal energy systems (Dong et al., 2015), thermal recovery processes in hydrocarbon reservoirs (Kovscek, 2012), geological CO₂

sequestration (Pruess, 2008), thermal remediation of contaminated soil (Vidonish et al., 2016), fuel cells (Andersson et al., 2016), and biological tissues (Nakayama & Kuwahara, 2008).

Studying heat transfer processes in microfluidic devices and in porous media requires advanced measurement techniques for precise measurement of temperature with microscale resolution. While common macro-scale temperature probes such as thermocouples (TC) (Zhang et al., 2006) and resistance temperature detectors (RTD) (Chung & Kim, 2008) can be miniaturized using microfabrication techniques, they are not ideally suited for microscale applications due to several limitations. Specifically, they can only provide discrete point measurements. In addition, they do not directly measure the fluid temperature, instead they provide only the surface temperature measurements.

Optical temperature measurement techniques are alternatives that can potentially provide highresolution fluid temperature measurements without the aforementioned limitations (Wang et al., 2013). One of such techniques is laser induced fluorescence (LIF) that can be used to measure variables such as concentration, pH and temperature in fluids (Crimaldi, 2008). LIF thermometry is based upon the temperature sensitivity of the absorption and emission characteristics of fluorescent dyes and has been implemented in both macro- (Sakakibara & Adrian, 1999) and micro-scale configurations (Natrajan & Christensen, 2009). The technique has also been applied in aqueous (Koegl *et al.*, 2020; Natrajan & Christensen, 2009; Sakakibara & Adrian, 1999; Shafii *et al.*, 2010) and non-aqueous (oils and alkanes) solutions (Deprédurand *et al.*, 2008; Perrin *et al.*, 2015). The range of applications include investigation of temperature fields during solidification of an aqueous ammonium chloride solution (Shafii *et al.*, 2010), droplet evaporation (Chaze *et al.*, 2017; Koegl *et al.*, 2020), and droplets impinging on heated surfaces (Castanet *et al.*, 2020; Dunand *et al.*, 2012).

To the best of our knowledge, there are no instances of application of LIF thermometry to *simultaneously* measure the temperature of two immiscible liquids: an aqueous and a non-aqueous solution. Specifically, the implementation of this technique is intended for use in multiphase flow in porous media. The methodology proposed herein would enable investigation of processes applicable to geothermal energy systems and enhanced oil recovery, among others.

2 Methodology

2.1 Principles of LIF Thermometry

For a dye of concentration *C* illuminated with an incident light flux of intensity I_0 , the fluorescence power emitted per unit volume *I*, is

$$I = I_0 C \phi \varepsilon \tag{1}$$

where ε is the absorption coefficient of the dye and ϕ is its quantum efficiency (QE). The variation in *I* with temperature is attributable to temperature dependence of quantum efficiency and absorption coefficient, *i.e.*, $\phi \varepsilon = f(T)$. However, spatial and/or temporal variations in I_0 and *C* can be misinterpreted as changes in temperature. One way for mitigating this effect is using the ratiometric or two-color technique, which can be implemented with one dye (two-color one-dye, 2c-1d) or two dyes (two-color two-dye, 2c-2d) (Chaze *et al.*, 2016). In the ratiometric approach, the fluorescence signal intensities at two wavelength bands are recorded, whereby taking the ratio of the two signals, the dependence on illumination intensity can be eliminated. In the 2c-1d approach, two distinct emission bands of the *same* dye are selected for imaging wherein the emission intensity for one of the wavelength bands should be nearly insensitive to changes in temperature. In equation 2, V_1 and V_2 are the image intensity in camera 1 and 2, and subscripts *A* and *B* refer to two distinct wavelength bands.

$$F_{2c-1d} = \frac{V_1}{V_2} = \frac{I_A}{I_B} = \frac{I_0 C \phi_A \varepsilon_A}{I_0 C \phi_B \varepsilon_B} = \frac{(\phi \varepsilon)_A}{(\phi \varepsilon)_B}$$
(2)

In this example, the emission intensity of wavelength band *A* is assumed to be sensitive to temperature $(\phi \varepsilon)_A = f(T)$, while wavelength band *B* is insensitive or weakly sensitive to temperature, $(\phi \varepsilon)_B \neq f(T)$.

In the 2c-2d approach, each of the two imaging wavelength bands should coincide with an emission band of one of the dyes. Ideally, camera 1 should pick the emission only from dye *A*, and camera 2 should pick the emission only from dye *B*, without any crosstalk. However, in many cases complete spectral separation of the emission from the two dyes may not be possible. As a result, camera 1 will pick emissions from dye *B*, and camera 2 will pick emissions from dye *A*. This effect can be accounted for by measuring functions Π_A and Π_B , where Π_A represents the fraction of emission from dye *A* recorded by camera 2, while Π_B represents the fraction of emission from dye *B* by camera 1 (Eq. 3).

$$F_{2c-2d} = \frac{V_1}{V_2} = \frac{I_A + \Pi_B I_B}{I_B + \Pi_A I_A} \neq f(I_0)$$
(3)

The ideal dye candidates for this approach can be excited by the same laser wavelength while their emissions spectra have no overlap.

2.2 Experimental Apparatus

An Olympus IX-73 inverted microscope coupled to a Karin TwinCam image splitter and two Andor Zyla 5.5-megapixel sCMOS cameras (2560×2160 pixels, 6.5μ m pixel size) are used for imaging. The image splitter allows for simultaneous imaging of the field of view at two wavelength bands. The recording spectral band for camera 1 and camera 2 are 540–572 nm and 610–640 nm, respectively (Fig. 1). Using a 10× 0.25-numerical aperture (NA) objective, imaging is carried out at a magnification of 0.65 μ m/pixel, with a field of view of ~2 mm in diameter. A Quantel Evergreen dual-head pulsed Nd:YAG laser (200 mJ/pulse maximum energy) at 532 nm coupled to the microscope is used as the excitation source for the fluorescent dyes.



Fig. 1 Optical setup for two-color fluorescence thermometry.

Data acquisition is performed using a NI-9212 module attached to a NI-cDAQ 9171 compact chassis for temperature measurements. A BNC 575 timing unit is used to synchronize the cameras and the laser, and control the lamp and Q-switch timing for the Nd:YAG laser. Images are recorded using Andor SOLIS software, while data acquisition is carried out in MATLAB. All image and data post-processing is carried out in MATLAB.

A calibration module, consisting of an aluminum block with internal drilled channels is used, to record the fluorescence signal from the fluorescent dyes at known temperatures. Water from a

temperature-controlled bath is circulated in the channels using an external pump (**Fig. 2**). The dye flows through a channel attached to the heat sink and is sealed using an O-ring and a 1-mm-thick microscope glass slide clamped to the aluminum heat sink. Two T-type thermocouples are inserted into the channel to record the dye temperature during image acquisition. Using this setup, the temperature sensitivity of aqueous and non-aqueous dye solutions are quantified.



Fig. 2 Experimental setup for temperature calibration of fluorescence signal for different dye solutions.

Registration of the images of the two cameras is performed using a microscope scale as a target and using the imregister function in MATLAB, as shown in Fig. 3.



Fig. 3 Image registration process showing the captured image from camera 1, camera 2, and the superimposed image from the two cameras.

2.3 Fluorescent Dyes

The dyes are selected based on the following primary criteria:

- 1. Absorption band in 532 nm so that all dyes can be excited with the same Nd:YAG laser
- 2. Soluble only in either the aqueous or the non-aqueous phases.

In addition, for each fluid phase, at least one dye should have temperature-dependent fluorescence spectra. It is notable that some of the most commonly used dyes for LIF thermometry such as Rh B and Rh 6G (Crimaldi, 2008; Sutton et al., 2008) are soluble in both water and oil/alkanes and hence are not suitable for this study.

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Solution of Sulforhodamine 101 (SR101) and (Eosin Y) EY is used as the aqueous phase. The absorption and emission spectra of SRh 101 and EY are presented in Fig. 4, where each spectrum is normalized with respect to its maximum value. EY has peak absorption and emission at 525 nm and 546 nm, respectively. SR 101 has peak absorption and emission at 578 nm and 593 nm, respectively. In the current optical setup (shown in Fig. 1) camera 1 records signal primarily from EY (dye A), while camera 2 records emitted light from both SR (dye B) and EY. Thus, Π_A correction function is used to account for the emissions from dye A (EY) picked up by camera 2.



Fig. 4 Absorption and emission spectra of Sulforhodamine 101 (SRh) in water and Eosin Y (EY) in water. The grey shaded areas show the imaging wavelength for camera 1 (540–572 nm) and camera 2 (610–640 nm). Data from: Chroma Spectra Viewer.

Solution of pyrromethene 597-8C9 in n-decane is used as the non-aqueous phase. The absorption and emission spectra of PM 597-8C9 in decane is presented in Fig. 5, where each spectrum is normalized with respect to its maximum value. The peak absorption and emission for PM 597 occur at ~525 nm and ~600 nm, respectively. The maximum emission consists of a relatively wide band near 575–600 nm.



Fig. 5 Absorption and emission spectra of Pyrromethene 597-8C9 (PM 597-8C9) in n-decane. The grey shaded areas show the imaging wavelength for camera 1 (540–572 nm) and camera 2 (610–640 nm). Data from: Labergue *et al.*, (2010)

3 Results and Discussion

The images recorded by the two cameras at each temperature are transformed based on the image registration process described in §2.2. The average intensity of an 8×8-pixel window in the center of the image is used for calibration. This yields an effective spatial resolution of ~5.2 μ m. At each temperature 10 images are acquired at an imaging frequency of 10 Hz, yielding a temporal resolution of 1 s.

Figure 6 shows the effect of consecutive excitations on the fluorescence signal. The intensities from individual cameras and their ratio are normalized to facilitate comparisons. For these measurements, the dye solution was excited continuously with 500 laser pulses at room temperature. In both fluids, the ratiometric approach seems to work well in minimizing the impact of pulse-to-pulse laser energy variations. For the aqueous solution (Fig. 6a), the standard deviation of signal for camera 1 and camera 2 is 3.7% and 3.1%, respectively, while the ratio has a standard deviation of 1.9%. In the non-aqueous solution (Fig. 6b), the standard deviation of signal for camera 2 is 3.3% and 3.7%, respectively, while the ratio has a standard deviation of 0.7%. In the aqueous solution (Fig. 6a) there is a slight downward trend in the ratio signal with increasing laser pulses. This could be due to change in temperature during data acquisition or photobleaching in one or both dyes which require further investigation. In the non-aqueous

solution (Fig. 6b), there are some instances where the signal from individual cameras is $\sim 15\%$ lower than average. This is most likely due to abrupt changes in laser pulse energy as the ratio signal remains within $\sim 1\%$ of the average.



Fig. 6 Effect of consecutive excitation on recorded fluorescence signal for each camera and their ratios. **(a)** 1.0 mg/L Sulforhodamine 101 and 1.6 mg/L Eosin Y in water **(b)** 5 mg/L Pyrromethene 597-8C9 in n-decane. The intensities from individual cameras and their ratio are normalized with their average values to facilitate comparisons

Error! Reference source not found. shows the calibration curve for the aqueous solution consisting of 1.0 mg/L SR and 1.6 mg/L EY, the 5.0 mg/L solution of PM597 in n-decane. In this implementation of the 2-color 2-dye approach the measurement sensitivity for the aqueous solution is ~ $1.7 \text{ }\%\text{K}^{-1}$. Also, the measurement sensitivity for the non-aqueous solution in this 2-color 1-dye approach is ~ $0.1 \%\text{K}^{-1}$. The measurement uncertainty for both cases is ~1% based on one standard deviation from the 10 images used at each temperature.

The previous studies using LIF thermometry in aqueous solutions report a sensitivity of ~2-4% K⁻¹ in 2c-2d configuration and ~1% K⁻¹ in 2c-1d configuration. Sakakibara & Adrian, (1999) used solution of Rhodamine B and Rhodamine 110 in water, excited with a 488-nm Argon ion laser in a 2c-2d macroscopic planar configuration and obtained a sensitivity of ~1.7% K⁻¹. Natrajan & Christensen, (2009) used Rhodamine B and Sulforhodamine 101 in water excited with a pulsed 532-nm Nd:YAG laser in a 2c-2d microscopic configuration and obtained a sensitivity of 2.7 % K⁻¹. Shafii *et al.*, (2010) used fluorescein and Kiton Red dyes in water excited with a 514.5-nm Argonion laser in aqueous ammonium chloride solution in macroscopic planar configuration obtaining



Fig. 7 Temperature dependence of the normalized fluorescence intensity of 1.0 mg/L SR and 1.6 mg/L EY in water, and 5.0 mg/L of PM597-8C9 in n-decane. The markers represent one standard deviation in normalized intensity.

a sensitivity of ~4% K⁻¹. Collignon *et al.*, (2022) used a solution of Rhodamine 560 and Kiton Red dye in water with a laser light excitation at 532 nm, achieving a temperature sensitivity of ~3% K⁻¹. Castanet *et al.*, (2020) and Chaze *et al.*, (2017) used sulforhodamine 640 and disodium fluorescein 27 in a 2c-2d configuration with 532 nm pulsed laser reporting a sensitivity of ~3% K⁻¹. Dunand et al., (2012) used 2c-1d LIF thermometry with fluorescein 27 dye and 532 nm laser reporting a sensitivity of ~0.8% K⁻¹.

Compared to aqueous solutions, there are relatively fewer studies using LIF thermometry in liquids immiscible with water such as oils and alkanes. Deprédurand et al., (2008); Labergue et al., 2010; Perrin et al., (2015) used 2c-1d LIF thermometry with pyrromethene 597-8C9 in alkanes yielding a sensitivity of 0.8–1 % K⁻¹ for studying heat transfer and evaporation of fuel droplets. Prenting et al., (2021) quantified sensitivity of a several fluorescent dyes in organic solvents. They reported a sensitivity of ~0.6% K⁻¹ in 2c-1d configuration for PM597 in o-xylene excited at 532 nm. The reported sensitivity is quite lower than reported values in the literature. This indicates that the variation of fluorescence intensity within both wavelength bands is similar.

4 Summary and Future Work

The results presented herein demonstrate the principles and the feasibility of LIF thermometry to obtain spatially and temporally resolved temperature fields for multiphase flow two immiscible

liquids. In this implementation, 2-color 2-dye approach with a solution of Eosin Y and Sulforhodamine 101 in water is used in for aqueous phase, and 2-color 1-dye approach with a solution of Pyrromethene 597-8C9 in n-decane is used for the non-aqueous phase. The achieved spatial and temporal resolution of the measurements are $5.2 \,\mu$ m and 1 s, respectively. The measurement sensitivity for the aqueous and non-aqueous phase are $1.7 \,\% \text{K}^{-1}$ and $0.1 \,\% \text{K}^{-1}$, respectively, with an uncertainty of <1% in both cases. The sensitivity and uncertainty level achieved herein are satisfactory for the aqueous phase. However, for the non-aqueous phase, modifications in the optical setup and/or choice of fluorescent dye(s) are needed to improve the sensitivity of the measurement technique.

The future planned work is focused on fine tuning experimental parameters to improve the sensitivity and spatiotemporal resolution of the measurements. Based on the frequency of the pulse laser, the temporal resolution can theoretically be improved to 1/30 s enabling investigation of transient processes. The intended application for this technique is to investigate heat transfer in multiphase flow in porous media.

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