



# Luminescent and absorptive metal-coated emulsions for micro-velocimetry



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## ARTICLE INFO

### Article history:

Received 16 October 2015

Received in revised form 10 March 2016

Accepted 15 March 2016

Available online 16 March 2016

### Keywords:

Microfluidics

Velocimetry

Emulsion

Polydopamine

Core-shell

Particle

## ABSTRACT

Fluorescent latex beads have been widely used as tracers in microfluidics for the last decades. They have the advantages to be density matched with water and to be easily localizable using fluorescence microscopy. We have recently synthesized silver-coated oil droplets that are both luminescent and absorptive, by first coating the oil interface with a polydopamine layer and then depositing a silver layer by a redox process. They have a mean diameter of 6  $\mu\text{m}$  and their density has been matched to the density of water by adjusting the thickness of the metallic layer. In this work we used these particles as tracers to measure the velocity profile of an aqueous solution in a PDMS microchannel with a rectangular cross-section. This allowed us to confirm the predictions of the Stokes equation with results comparable to those of common polystyrene particles.

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## 1. Introduction

Measuring the local instantaneous velocity of a liquid at micron scale has been an important challenge these last decades [1,2] to understand e.g. the mixing properties of microfluidic devices [3] or the no-slip condition commonly assumed in hydrodynamics [4]. With the development of lab-on-a-chip applications, micro-velocimetry has been used to characterize e.g. electrokinetic flows [5], in vivo flows [6], or biomedical science [7] related flows. For a pressure driven flow in a microchannel, the mean flux can be determined by weighing the liquid going out from a device during a certain amount of time but this technique does not allow the measurement of the spatial and temporal variations of the velocity inside the channel. To address this issue, micro-particle image velocimetry ( $\mu\text{PIV}$ ) or micro-particle tracking velocimetry ( $\mu\text{PTV}$ ) have been developed [8] to measure the spatiotemporal features of complex fluid flows. They both rely on the observation of moving particles and calculation of the relative displacement of the particles during a finite amount of time. Commonly used tracers [9] in micro-velocimetry experiments are fluorescent polystyrene beads with diameters ranging from 0.2  $\mu\text{m}$  to several microns [8]. Fluorescence is used to increase the contrast between the particles and the surrounding fluid, making the tracking fast and easy.

We recently developed [10] a fabrication protocol of metal-coated particles with a liquid core that are both luminescent and

light absorptive, making them interesting candidates for multimodal observation in micro-velocimetry. The synthesis of this type of particles is easy and relies on the use of soybean oil-in-water emulsion droplets, i.e. a metastable liquid-liquid suspension. The oil droplets are first coated with a polydopamine shell [11] synthesized in-situ and then coated with a thin, nanometric, silver layer grown by electroless plating. Both diameter of the droplets and the thickness of the silver shell are adjusted to obtain particles with a density close to water, as the weights of oil and silver compensate each other.

In this work, we use both brightfield and epifluorescence microscopy to measure the velocity profile of a pressure-driven flow of an aqueous solution seeded with metal-coated droplets in a PDMS microchannel of rectangular cross section. Using these particles as  $\mu\text{PTV}$  tracers allows us to confirm the predictions of the Stokes equation in this particular geometry.

## 2. Materials and methods

### 2.1. Materials

The Poloxamer 188 block-polymeric surfactant ( $\text{HO}(\text{C}_2\text{H}_4\text{O})_{79}-(\text{C}_3\text{H}_6\text{O})_{28}-(\text{C}_2\text{H}_4\text{O})_{79}\text{H}$ ) was kindly provided by Croda France SAS. The sodium alginate was purchased from Sigma-Aldrich. Ultrapure water (Millipore,  $18.2 \text{ M}\Omega \cdot \text{cm}^{-1}$ ) was used for all experiments. All the chemicals used for the metallized emulsion preparation were purchased from Sigma-Aldrich. Fluorescent polystyrene beads (DragonGreen, diameter of 0.51  $\mu\text{m}$ ) were purchased from Polyscience.

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## 2.2. Emulsion fabrication

We first disperse by manually stirring 15 g of soybean oil in an aqueous phase containing 2.5 g of a surfactant (Poloxamer F-68, initial proportion of 30% w/w) and 2.5 g of a thickening agent (sodium alginate, initial proportion of 4% w/w). This crude, polydisperse emulsion is further sheared and rendered quasi-monodisperse in a Couette cell apparatus under a controlled shear rate ( $5000 \text{ s}^{-1}$ ), following the method developed by Mason et al. [12]. Before decantation, the emulsion is diluted to have a proportion of 1% w/w of Poloxamer F-68 and 5% w/w of oil. After several decantation and rinsing steps with a 1% w/w solution of Poloxamer F-68 to remove very small droplets, the emulsion is stored at  $12^\circ\text{C}$  in a dark Peltier-cooled cabinet.

## 2.3. Fabrication of the metallodielectric emulsions

The protocol is described on Fig. 1A and is inspired from Nocera et al. [10]. The cream of the soybean oil emulsion is rinsed twice with a Tris buffer ( $\text{pH} = 8.5$ , 20 mM) supplemented with a surfactant (Tween 20, initial proportion of 0.2% w/w in the Tris buffer). To do this rinse, we mix  $70 \mu\text{L}$  of the emulsion and  $200 \mu\text{L}$  of the Tris-Tween solution, centrifuge during 30 s at 4000 rpm and remove the aqueous phase ( $200 \mu\text{L}$ ) with a micropipet. For the polydopamine coating, we disperse  $70 \mu\text{L}$  of the rinsed emulsion in  $100 \mu\text{L}$  of a solution of dopamine (initial concentration of  $5 \text{ mg}\cdot\text{mL}^{-1}$  in the Tris-Tween solution) and we add  $85 \mu\text{L}$  of the Tris-Tween solution. Then, we add  $5 \mu\text{L}$  of potassium permanganate ( $\text{KMnO}_4$ , initial concentration of  $40 \text{ mg}\cdot\text{mL}^{-1}$  in water) in the solution to enable the transformation of dopamine in polydopamine at the surface of the droplets. The solution is stirred with a rotor (60 rpm) during 2 h at room temperature in obscurity. To remove the unreacted molecules the sample is rinsed twice, as described before, using  $180 \mu\text{L}$  of the Tris-Tween solution. Then we coat the droplets with a silver layer using an electroless plating process. To do this, we first disperse the droplets in a solution of silver nitrate ( $\text{AgNO}_3$ , initial concentration of  $18 \text{ mg}\cdot\text{mL}^{-1}$  in the Tris-Tween solution) and stir the obtained solution on a rotor (60 rpm) during 90 min at room temperature in the obscurity. In  $50 \mu\text{L}$  of the obtained dispersion, we add  $150 \mu\text{L}$  of a solution of ascorbic acid. The acid ascorbic solution has an initial concentration of  $15.5 \text{ mg}\cdot\text{mL}^{-1}$  in the Tris-Tween solution supplemented with a surfactant (polyvinylpyrrolidone, initial proportion of 0.02% w/w in the Tris-Tween solution).

## 2.4. Microfluidic device fabrication

The devices are made in PDMS (polydimethylsiloxane), using the standard soft lithography techniques [13]. In brief, we fabricate SU-8 (SU-8 3050, Microchem) masters on a silicon wafer, then proceed to PDMS molding and thermal curing at  $80^\circ\text{C}$  during 2 h (RTV 615, 1:10 ratio for the reticulating agent, RTV 615, Momentive Performance Materials). We treat the PDMS surfaces and the glass coverslip (VWR,  $50 \times 24 \text{ mm}$ ) that closes the channel with air plasma (300 mTorr, 40 s) before sealing both parts of the chip together. The microfluidic system is then physically connected with small tubing to two small glass beakers used to impose a hydrostatic pressure difference to the flow. The channel dimensions are: width ( $w$ ) =  $200 \mu\text{m}$ , height ( $h$ ) =  $65\text{--}70 \mu\text{m}$  and length ( $L$ ) = 2 cm.

## 2.5. Microscopy

Brightfield and fluorescent images particles were acquired on a Zeiss Axio Observer Z1 microscope (Oberkochen, Germany) connected to a Flash 2.8 sCMOS camera (Hamamatsu Photonics, Japan). Epi-illumination was done with a HXP 120 L metal-halide lamp and a GFP filter set (Excitation wavelength: 475 nm, Emission wavelength: 530 nm). All images were taken with a  $100\times$  oil immersion objective (NA: 1.25,  $\text{DOF} \leq 1 \mu\text{m}$ ). Exposure times were respectively set to 50 ms and 80 ms for brightfield and epifluorescence conditions.

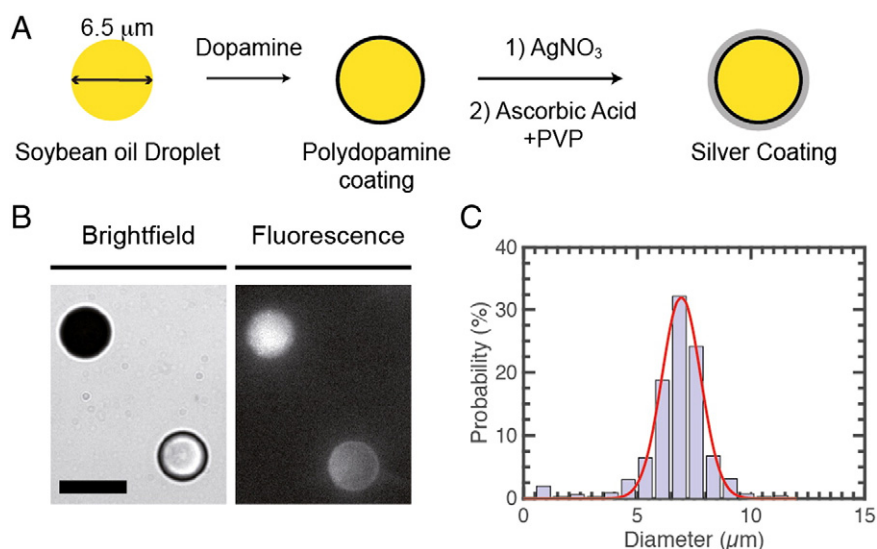
## 2.6. Numerical simulations and image analysis

All computations have been performed with Mathworks Matlab software.

## 3. Results and discussion

### 3.1. Metal-coated luminescent and absorptive oil droplets

The droplets of an oil-in-water emulsion are first coated with polydopamine and then with silver (Fig. 1A) following a fabrication protocol we developed recently [10]. The naked and monodisperse soybean oil droplets are first dispersed in an oxidative and alkaline aqueous solution of dopamine. The dopamine self-polymerizes at the surface of the droplets following a reaction scheme that involves the



**Fig. 1.** (A) Fabrication of metal-coated emulsion droplets. The naked soybean oil droplets are dispersed in an oxidative and alkaline aqueous solution of dopamine. Dopamine polymerizes at the surface of the droplets. After introducing silver nitrate, silver ions are adsorbed on the polydopamine layer. Addition of ascorbic acid, a reducing agent, and PVP, a stabilizing polymer, leads to silver-coated emulsion droplets. (B) Brightfield and fluorescence pictures of silver-coated droplets. (C) Size distribution histogram of the metal-coated emulsion. The diameter is equal to  $6.5 \pm 1.5 \mu\text{m}$ .

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