

# SINGLE POINT DETECTION METHOD FOR SURFACE CHARACTERIZATION OF CHANNELS IN MICROFLUIDIC CHIPS

Eric R. Castro<sup>1</sup>, Mark D. Tarn<sup>1,3</sup>, Pavlina Ginterová<sup>1,2</sup>, Pavel Neužil<sup>1,2\*</sup> and Andreas Manz<sup>1</sup>

<sup>1</sup>KIST Europe GmbH, GERMANY

<sup>2</sup>Palacký University, CZECH REPUBLIC

<sup>3</sup>University of Hull, UNITED KINGDOM

## ABSTRACT

Here we report a single point detection method for the characterization of surface properties in microfluidic channels. Our method couples segmented flow (SF) with laser induced fluorescence (LIF) to assess the hydrophobicity of microfluidic channels by analyzing the shape of a fluorescent plug as it moves past a detector. Differences in plug shape are shown to correspond to differences in surface conditions and allow us to distinguish between hydrophobic, partially hydrophobic and hydrophilic surfaces.

**KEYWORDS:** Segmented flow, LIF, surface properties, contact angle

## INTRODUCTION

The surface modification of microfluidic channels is often an important part of chip fabrication. Researchers routinely modify channel surfaces to be either more hydrophobic or hydrophilic depending on the application. Assessment of the surface modification is conventionally done by goniometry (i.e. contact angle measurement). Such measurements are, however, impossible to perform directly inside a closed microfluidic chip.

In SF or two-phase flow, arrays of aqueous droplets separated by plugs of an immiscible liquid phase, usually an oil, are generated inside a microfluidic channel, typically by means of a T-junction [1, 2]. Aqueous droplet arrays of this kind have been used in many applications, such as reaction vessels [3], to segregate cells [4] or to contain separated analytes to prevent diffusional broadening [5]. Optical detection methods such as LIF have been used previously to measure droplet fluorescence, size and reproducibility [4, 5]. Our present work focuses on using the shape of moving droplets to characterize the dynamic surface properties of channels.

## THEORY

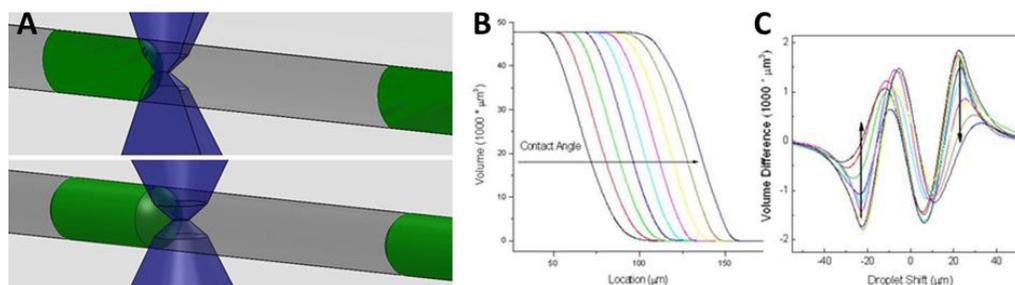


Figure 1: A) SolidWorks model of droplet interaction with a laser. B) Interaction volume vs. location of droplet plot for droplets with different contact angles. C) Plots of the residuals of the model and fit to the Boltzmann equation.

The interaction of the laser beam with a droplet inside a channel was modeled using SolidWorks software. To simplify the model, we have assumed a channel with a circular cross-section. A droplet was generated inside the channel and moved stepwise past a conical region representing the laser light (Figure 1a). The interaction volume between the droplet and laser was calculated by SolidWorks. The hydrophobicity of the channel surface was altered by changing the contact angle of the droplet. The model was used to generate plots of the interaction volume vs droplet position for a range of contact angles between  $40^\circ$  and  $120^\circ$  (Figure 1b). The droplet profiles so obtained were fit to the Boltzmann function (1).

$$f(x) = \frac{A_1 - A_2}{1 + e^{\frac{x-x_0}{dx}}} + A_2 \quad (1)$$

Residuals values between fit and model were plotted (*Figure 1c*). The residual plots show more clearly than the droplet profiles the effect of the contact angle on the droplet shape. We concluded from these results that channels with different hydrophobicities would result in measurable changes in droplet shape.

## EXPERIMENTAL

The first set of experiments to confirm the results of the model were performed using a 40 cm piece of fused silica capillary. The hydrophobicity of the capillary surface was modified by chemical vapor deposition (CVD) using the silanizing reagent FAS-17 (Sigma, USA). A detection window was made 10 cm from one end of the capillary by burning the polyimide coating and placed into a home-built LIF detection system (*Figure 2a*). A second window, 10 cm from the other end of the capillary, was placed in the viewing field of a digital microscope with a CMOS sensor (Aigo, China) for simultaneous droplet imaging. An LED with a nominal wavelength of 470 nm (Thorlabs, USA) was used as the source for fluorescence excitation. The sampling rate that could be achieved using the microscope software was too low for continuous imaging of the high velocity droplets produced for SF. For this reason, a stroboscopic imaging technique was used, in which the LED was operated in “burst” mode synchronized to the sensor frame rate. The frequency of the bursts was set so that there was only a short pulse of light during each capture, essentially freezing the motion of the droplet in the channel. SF was generated inside the capillary using an acrylic T-junction to mix hexadecane and an aqueous fluorescein solution. The fluorescence signal was monitored with a photo multiplier tube (PMT) and an oscilloscope. In a second set of experiments, segmented flow was generated inside a glass microfluidic droplet generator (Micronit, Netherlands). The chip was placed in the viewing field of an inverted microscope with a 50× objective (*Figure 2b*). Droplet fluorescence was monitored first using a CCD camera (Jenoptik, Germany) and the aforementioned flashing method. The camera was then replaced with a PMT to obtain traces like in the capillary experiments.

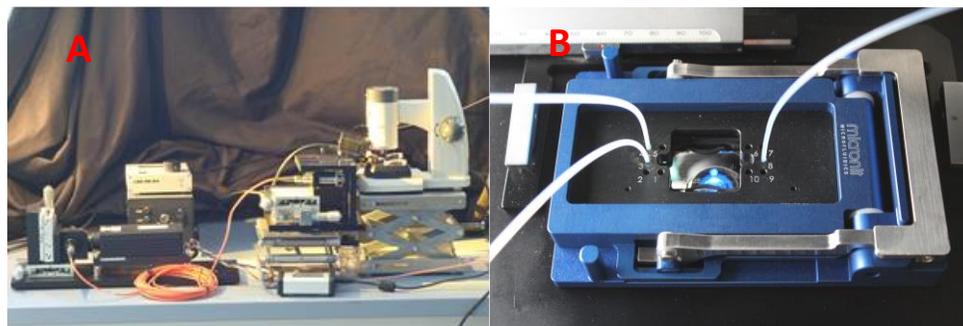


Figure 2: A) Instrument setup for capillary experiments. B) Micronit droplet generator chip and holder.

## RESULTS AND DISCUSSION

In the capillary, the hydrophobicity of the FAS-17 modified (hydrophobic) capillary was confirmed by the droplet shape in the images (*Figure 3a*). The droplet front and back profiles obtained from the oscilloscope traces for both silanized and untreated (hydrophilic) capillaries were fit to the Boltzmann function (*Figure 3b*). The residual plots for the modified and unmodified capillaries also exhibited measurable differences as predicted by the model (*Figure 3c,d*). This can be seen most clearly at the beginning of the interaction. For example, in the hydrophilic capillary the rise of profile from the experimental data is slower than that of the Boltzmann function, leading to a greater deviation from the model than in the hydrophobic capillary. The comparison of both residual plots (*Figure 3c*) shows a larger “peak” for the hydrophilic capillary in this region.

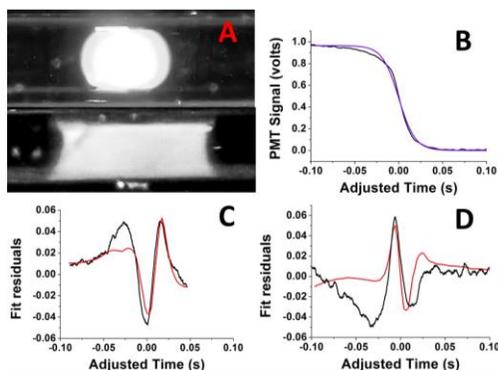


Figure 3: A) Fluorescence images of droplets in a hydrophobic (top) and hydrophilic (bottom) capillary. B) Profile of droplet back in a hydrophobic capillary. C) Residual fits for droplet profile front in a hydrophobic (red) and hydrophilic (black) capillary. D) Residual plots for droplet back in a hydrophobic (red) and hydrophilic (black) capillary.

Based on these results, we have also extended the method to microfluidic channels with non-circular cross-sections. A commercial glass chip with a T-junction was used to generate SF in a channel with a cross section shown in Figure 4a. Fluorescence traces from these experiments were analyzed as described for the capillary experiments. Surface information was also obtained from the fluorescence signal measured on-chip. Droplet asymmetry from partial wetting, for example, was observed in the PMT trace.

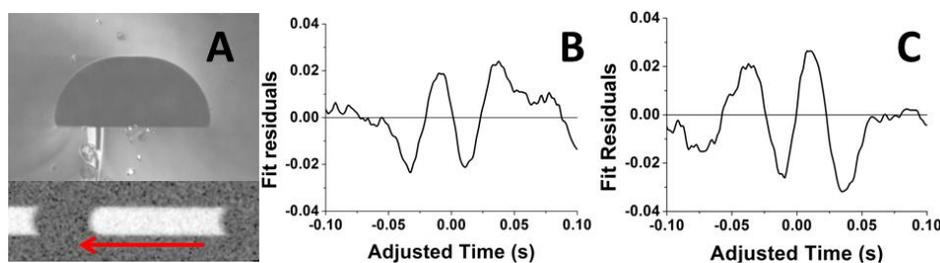


Figure 4: A) Images of cross-section of Micronit microchip channel (top) and fluorescent droplet in the channel (bottom, arrow indicates direction of flow). B) Residual plot for droplet front. C) Residual plot for droplet back.

## CONCLUSION

We conclude that SF coupled to LIF is a powerful method to extract information about the *dynamic* surface conditions of microfluidic channels. Such an approach could be a valuable tool for assessing coating or surface modification efficiency in microfluidic devices.

## REFERENCES

- [1] P. Garstecki, M.J. Fuerstman, H.A. Stone, G.M. Whitesides, "Formation of droplets and bubbles in a microfluidic T-junction - scaling and mechanism of break-up," *Lab Chip*, 6, 437-446, 2006.
- [2] H. Song, J.D. Tice, R.F. Ismagilov, "A microfluidic system for controlling reaction networks in time," *Angew. Chem. Int. Ed.*, 42, 768-772, 2003.
- [3] I. Shestopalov, J.D. Tice, R.F. Ismagilov, "Multi-step synthesis of nanoparticles performed on millisecond time scale in a microfluidic droplet-based system," *Lab Chip*, 4, 316-321, 2004.
- [4] A. Huebner, L.F. Olguin, D. Bratton, G. Whyte, W.T. Huck, A.J. de Mello, J.B. Edel, C. Abell, F. Hollfelder, "Development of quantitative cell-based enzyme assays in microdroplets," *Anal. Chem.*, 80, 3890-3896, 2008.
- [5] M. Wang, G.T. Roman, M.L. Perry, R.T. Kennedy, "Microfluidic Chip for High Efficiency Electrophoretic Analysis of Segmented Flow from a Microdialysis Probe and in Vivo Chemical Monitoring," *Anal. Chem.*, 81, 9072-9078, 2009.

## CONTACT

\* P. Neuzil; phone: +49-681 9382 228; pavel@kist-europe.de