# **DEVELOPMENT OF NONINTRUSIVE MEASUREMENT TECHNIQUE OF FLOW RATE AND PRESSURE DROP FOR EXTENDED NANOSPACE CHANNEL FLOWS**

**S. Kubori\* 1 , Y. Kazoe<sup>2</sup> , K. Mawatari<sup>2</sup> , Y. Sugii<sup>2</sup> and T. Kitamori<sup>12</sup>**

*<sup>1</sup>Department of Bioengineering, School of Engineering, The University of Tokyo, Japan and <sup>2</sup>Department of Applied Chemistry, School of Engineering, The University of Tokyo, Japan*

# **ABSTRACT**

We developed a method to measure  $\mu$ g/s order flow rate driven by MPa order pressure in extended nanospace ( $10^1$ - $10^3$ m). Highly pressurized mass flow rate was measured by an electric balance of 1 µg resolution. The obtained flow rate in 500 nm extended nanochannel showed lower value than predicted, which suggests specific fluid dynamics affected by structured water near channel surface and electric double layer in extended nanospace. This study is important to reveal basic science in the space and establish fundamental technology of extended nanofluidic systems for chemical analysis.

**KEYWORDS** : Nanofluidics, Extended nanospace, Pressure-driven flow, Flow rate

## **INTRODUCTION**

Recently, integrated micro chemical systems have been further downscaled to extended nanospace. The extended nanospace is the surface-dominant space due to its small size and is the transitional region between single molecules and continuum phase. Many studies report unique properties of water in this space such as higher viscosity and lower permittivity of water compared to bulk space [1]. Our recent NMR studies observed higher proton mobility in extended nanospace than that in bulk space and proposed a model where water molecules form a network near the surface (proton transfer phase)[2]. Therefore the effects of structured water and electric double layer (EDL), which is the layers of counter ions attracted to the charged surface, on the liquid flow will be dominant and fluid dynamics will be different from that of bulk phase. Thus, in order to realize novel devices using these properties, understanding fluid dynamics governed by EDL and proton transfer phase is strongly required. However, conventional flow measurement techniques using tracer particles are subject to measurement errors by Brownian motion and electrokinetic potential along channel. A method using streaming current was proposed for the flow rate measurement [3]. However, it is difficult to obtain the absolute flow rate since this method detects electric signals.

Mass flow rate measurement is required as an absolute measurement method which monitors the mass of liquid flown through the channel. Previous studies for the flow rate measurement are 1 mg/s order resolution for 1000 microchannels [4]. Flow rate in extended nanochannel is ng/s order which can be increased to  $\mu$ g/s order by fabricating 10<sup>3</sup> order channels. However,  $\mu$ g/s is 3 order lower than the mg/s flow rate of conventional technique. In this study, we develop a measurement system of the  $\mu$ g/s order flow rate and the pressure drop in extended nanochannel by monitoring the mass of liquid flown through the channel.

#### **EXPERIMENTAL**

A measurement system was developed to obtain the  $\mu$ g/s order flow rate in extended nanospace. Figure 1(a) shows a schematic of measurement system. A  $10^5 - 10^6$  Pa order pressure was applied to the extended nanochannel using a high pressure controller. The mass flow rate through nanochannels was measured by an electric balance of high resolution 1 µg. The whole system is on a vibration isolator. The error in applied pressure was measured by connecting a manometer directly to high pressure controller and is estimated to be  $+/- 5$  kPa, which is negligible compared to applied pressure. The extended nanochannels were fabricated on a fused-silica substrate by electron beam lithography and plasma etching. 1  $\mu$ g/s order flow rate was obtained by fabricating 350 nanochannels (shown in figure 1(b)). Applied pressure must be nearly equal to the pressure drop only in extended nanochannels. So the number of nanochannels and channel size were optimized and 98 % of applied pressure is consumed in extended nanochannels.

In order to measure  $1 \mu g/s$  order flow rate, error factors around interface between the microchip and the electric balance. The error factors, which must be suppressed, were the fluctuation of measured mass owing to vibration of an injection tube, the buoyancy of injection tube and the evaporation of water, which are order of  $\mu$ g-mg similar to or much more than the  $\mu$ g/s flow rate. The fluctuation error of the tube was 3000 µg evaluated by putting plastic tube into collected water, which was much larger than the balance resolution (1 µg). This is caused by the tube of low rigidity, and hence a steel tube, glass cup to fix tube and rubber were employed to suppress the fluctuation to less than  $1 \mu g/s$ . In addition, steel tube dipped into water is given buoyance and the counteracting force affects the measured mass. Therefore the buoyance effect was theoretically calculated from the density of water and the diameters of water cup and steel tube. The measure flow rate is expected to be 1.7 % higher than the true value and the effect was corrected from the measured value. Finally, water evaporation  $(1.42 \mu g/s)$ 

from the water cup is in the same order as the measured flow rate  $(1 \mu g/s)$ . Therefore silicon oil was employed to cover liquid surface which suppressed evaporation effect to  $0.0275 \mu g/s$ . After suppressing the  $\mu$ g-mg error factors around the interface, the total measurement error in the system was calculated to be 2.80 % for 1  $\mu$ g /s and 10 minutes measurement. Measurement accuracy of  $1 \mu g/s$ , which is  $1/1000$  of conventional measurement, was achieved for the first time. This system is identically applicable to mass flow rate measurement in extended nanospace.



*Figure 1: (a) Schematic illustration of the measurement system of flow rate and pressure drop in extended nanospace. (b) A schematic illustration of nanochannels and microchannels. D, W and L indicate depth, width and length respectively.*

# **RESULTS AND DISCUSSION**

The developed system was verified using microchannel without specific effects by wall surfaces. The depth, width and length of the microchannel are 47.1  $\mu$ m, 102  $\mu$ m and 60 mm respectively. 1 kPa order pressure must be applied to obtain  $\mu$ g/s flow rate in microchannel. The high pressure controller, however, has  $+/-$  5 kPa error in applied pressure as described above and is not capable for the experiment. Thus a low pressure controller  $(\sim 500 \text{ kPa})$  was employed for precise pressure control of an accuracy of  $+/-$  0.1 kPa. The sample liquid is DI water.

Figure 2(a) shows the relationship between the time elapsed after applying pressure and the fluid mass collected in water cup on the balance. Applied pressures are 0.5, 1, 2, 5, 10 kPa. Linear relationship was observed for each pressure which indicates applied pressure is applied to the channel without any leakage. Figure 2(b) shows the relationship between the applied pressure and the mass flow rate, obtained by linear fitting to the results shown in Figure 2(a). The quantitative agreement was observed between measured values and theoretical ones based on Hagen-Poiseuille equation using bulk viscosity at room temperature. The flow rate as low as  $3.75 \mu g/s$  was measured for 0.5 kPa which was within 8 % of theoretical value. Therefore, the measurement system of  $\mu$ g/s order mass flow rate in extended nanospace was established for the first time.



*Figure 2: (a) The relationship between time elapsed after applying pressure and collected mass in microchannel. Applied pressures are 0.5, 1, 2, 5, 10 kPa. Linear relationship was observed for each applied pressure. (b) Measured mass flow rate as function of applied pressure in microchannel. The quantitative agreement between experimental and theoretical values was observed.*

Mass flow rate measurement in extended nanochannel was performed. The depth, width, length and number of the extended nanochannel are 507 nm, 520 nm, 400  $\mu$ m and 350 respectively. The sample liquid is DI water. Figure 3(a) shows the relationship between the time elapsed after applying pressure and the fluid mass collected in water cup on the balance. Applied pressures are 0.5, 1 and 1.5 MPa. Linear relationship was observed for each pressure as well as microchannel. Figure 3(b) shows the relationship between the applied pressure and mass flow rate. The experimental flow rate was much lower than theoretical one based on Hagen-Poiseuille equation using bulk viscosity at room temperature. A previous study reports that water viscosity in extended nanochannel increases by 4-5 times [1]. Our results suggest that water viscosity increased by 5 times on an assumption that the flow rate decrease is attributed to viscosity increase, which matches with the previous study.

The origin of this unique flow is considered to be from structured water and electric double layer. As described in previous section, structured water model as long as 50 nm from surface is proposed. Flow rate is expected to be lower due to this structure in extended nanospace. Traditional EDL model also has significant effect in the space. Reduced flow rate of pressure-driven flow is reported in small sized channels where specific interfacial area is large and EDL effect is dominant. This phenomenon is called electro-viscous effect whose impact is expected to be much larger for extended nanochannel. Structured water is dominant where channel size is less than 800 nm. The size effect of electric double layer can be controlled by changing the layer thickness which is subject to ion concentration of sample liquid. In future work, we will investigate the relationship between structured water and electric double layer, and those effects on flow rate by changing electric double layer thickness and channel size.



*Figure 3: (a) The relationship between time elapsed after applying pressure and collected mass in extended nanochannel. Applied pressures are 0.5, 1 and 1.5 MPa. Linear relationship was observed for each applied pressure. (b) Measured mass flow rate as function of applied pressure in microchannel. The experimental flow rate was much lower than prediction.*

### **CONCLUSION**

The mass flow rate measurement system using high pressure controller and electric balance of 1 µg resolution was developed to measure  $\mu$ g/s order mass flow rate in extended nanospace. The error factors of  $\mu$ g-mg around interface of the system were suppressed and the total error of the system was 2.80 % of measured flow rate theoretically. The system for measuring ug/s order mass flow rate was verified using microchannel. Reduced flow rate compared to theoretical value in extended nanospace was observed which suggests the specific effect in the space. We will investigate the relationship between structured water and electric double layer and those effects on flow rate to reveal basic science for developing novel devices using the space and establish fundamental technology of extended nanofluidic systems for chemical analysis.

# **ACKNOWLEDGEMENT**

This work was supported by Core-to-core program and Grant-in-Aid for Specially Promoted Research of Japan and Society for the Promotion of Science (JSPS)

### **REFERENCES**

- [1] A. Hibara *et al.,* "Nanochannels on a Fused-Silica Microchip and Liquid Properties Investigation by Time-Resolved Fluorescence Measurements," *Anal. Chem.*, vol. 74, pp. 6170-6176, Dec. 2002.
- [2] T. Tsukahara *et al.,* "NMR Studies of Structure and Dynamics of Liquid Molecules Confined in Extended Nanospaces," *J. Phys. Chem.*, vol. 113, pp. 10808–10816, Jul. 2009.
- [3] K. Morikawa *et al.,* "Streaming potential/current measurement system for investigation of liquids confined in extendednanospace," *Lab Chip*, vol. 10, pp. 871–875, Jan. 2010.
- [4] P. Nath *et al.,* "A System for Micro/Nano Fluidic Flow Diagnostics," *Biomed. Microdevices*, vol. 7, pp. 169–177, Sep. 2005.