Micro/Nano Liter Droplet Formation and Dispensing by Capacitance Metering and Electrowetting Actuation

Hong Ren, and Richard B. Fair

Department of Electrical Engineering, Duke University, Durham, NC 27708

Abstract — **In this work, a method is presented for dispensing droplets. The method exploits the built-in capacitance of an electrowetting device to meter the droplet volume and control the dispensing process dynamically. A fully automated droplet dispensing device is developed and used to load liquid samples or couple continuous flow to electro-wetting droplet handling. The reproducibility of the dispensing process is tested against dispensing parameters including dispensing volume, production rate, fluid viscosity, channel geometry and needle diameter. The relative independence of the reproducibility on those parameters indicates the wide applicability of the device in biological and chemical sample dispensing. Meanwhile, as low as 1.2% error in reproducibility is demonstrated and up to 120 droplets per minute production rate is possible. The dispensing process has been demonstrated for droplet volumes between 60nl and 1900nl or above while maintain reproducibility in an acceptable range of 10%.**

I. INTRODUCTION

The rapid development of 'lab-on-a-chip' systems and their applications in chemical and biological analysis has put new demand on the sample handling technique used. Such systems rely on the capability of metering, transportation and mixing of microscale liquid samples in microfabricated channels. Microfluidic systems based on continuous and discrete flow have been reported. Treating liquid samples as droplets has the advantages that dead volume can be decreased, and control complexities of flow rate can be eliminated.

One of the promising schemes of droplet manipulation is through electrowetting [1,2], in which a droplet is contained between two glass plates with patterned electrodes and an insulator layer over the surface. Application of voltages to the electrodes modulates the solution/insulator interfacial tension, leading to the bilateral transport of droplets in the planar microchannel.

To prevent evaporation of liquid samples, droplets should usually be formed and transported in a liquid media. In addition, variable volumes of liquid samples need to be generated reproducibly in a fast and fully automated manner. In addition, a cost effective device is required if widespread use of the technology is the goal. However, current microdispensing technologies do not combine these features sufficiently. Piezoelectric droplet generation [3] and pin-transfer [4] technology are subject to the variation of surface chemistry, and thus are limited in the flexibility and simplicity they have to adjust and regulate the dispenser output in terms of droplet size and generation rate. Syringe-solenoid based inkjet dispensing technology [5] overcomes these disadvantages, however, it suffers from high cost and is still not suitable to generate water-in-oil droplets.

We present a method and device for dispensing droplets of aqueous solutions of different viscosity in the micro/nano liter scale with high positioning accuracy and reproducibility. The device provides an excellent coupling between the outside world and an electro-wetting actuator system, while maintain its efficiency, simplicity, low cost and full automation. It includes a custom build capacitance measurement and continuous flow loading system with or without valve control. The droplet generation process is characterized in terms of reproducibility, dynamic range (dispensing volume), production rate.

II. EXPERIMENTAL

A. Principle

Schematics of dispensing system setup is illustrated in Fig.1

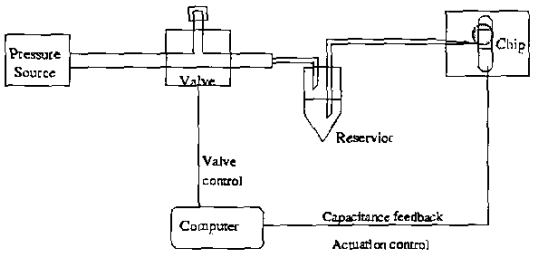

Fig. 1. Schematic of droplet dispensing system setup

With an electric motor-driven pump, liquid is deposited in planar microchannels between plates of two dimensional electrode arrays through a micro-capillary needle. The needle, with 300um outer diameter, is sandwiched between the plates and positioned at a right angle to the linear array of electrodes. The amount of liquid accumulated is metered by dynamic measurement of the capacitance between the electrolyte liquid and underlying electrode. The incremental change of the capacitance reflects the increasing foot print area of the

0-7803-7538-6/02/$17.00©2002 IEEE

deposition. Under given experimental conditions the dispensing volume is determined by knowing the variation of capacitance of the measured electrode. When the required volume is obtained, the droplet is produced by actuating the liquid and splitting it away from the inlet by electrowetting actuation [2]. Varying droplet volume is achieved by controlling the cutoff capacitance value.

B. Equipment and procedures

An electric motor driven pump provides the pressure source for loading liquid samples. A pressure regulator from Cole-Parmer is employed to adjust the pressure range from 0 to 60psig. A solenoid three-way valve controlled by a computer is used to provide continuous flow cutoff. The valve is shown in the experiment to be removable at flow rates smaller than 5ul/min. A voltage switch for droplet actuation and capacitance measurement are build out of a custom made switch board and DIO card (NI PCI 6534). Software programmed with Labview 6.0 is written to control the dispensing process.

Droplet formation was observed using a microscope and captured by a high-speed video camera with full frame size (width 720 pixels, height 380 pixels) at 30 frame/sec. Droplet size was measured by counting the pixels.

III RESULTS AND DISCUSSION

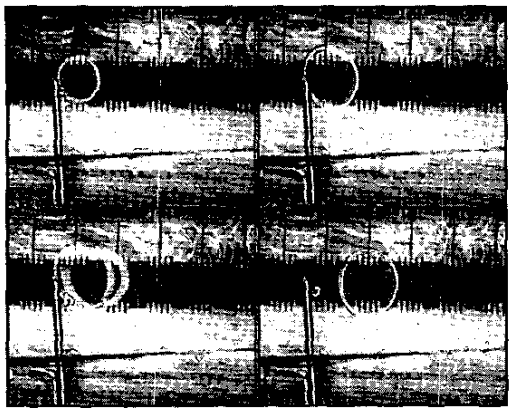

Fig.2 KCL droplet formation and generation from 300mm diameter needle on chip of 1.5mm pitch size and 600mm gap

Regular-sized droplets of KCL solution in oil were generated on 0.3mm-1.5mm pitch size electrodes with volumes varying from 60nl to 1.9ul. Automated droplet dispensing was demonstrated with time-lapse illustration showing a droplet being formed a source of KCL solution in Fig 2.

The reproducibility of droplet volumes (CV), also referred to as the precision of the dispensing process, is of vital importance to rate the quality of the entire dispensing system, the calculation of which is commonly performed by dividing standard deviation by mean of a group of measurements. The all-purpose use of the device is only possible if sufficient precision can be achieved with varying parameters. In this work, the effects of needle diameter, channel geometry (gap), dispensing volume, production rate and viscosity on reproducibility are investigated. The reproducibility is obtained by measuring the size of droplets generated sequentially in an automated manner.

A. Effect of dispensing volume on reproducibility

TABLE I
EFFECT OF DISPENSE VOLUME ON REPRODUCIBILITY

Dispense Volume (nl)	CV (%)
63	8.98
477	5.03
1900	6.13

Table 1 shows that dispensing reproducibility could be controlled within the acceptable range of 10% when the dispensing volume is within 60-1900nl. But it starts to decrease at volumes around 60nl. From experimental observation, it is believed that at smaller dispensing volumes with smaller unit pitch size, the resolution of capacitance measurement and the variation of flow rate may contribute to the volume variation.

B. Effect of production rate on reproducibility

TABLE II
EFFECT OF PRODUCTION RATE ON REPRODUCIBILITY

Production Rate (No./min)	CV (%)
8	1.2
45	5.03
60	5.14

Different production rates were realized by adjusting the flow rate with the pressure regulator. Table 2 shows that as low as 1.2% error in reproducibility can be achieved when the production rate is 8 droplets/min. This indicates that small flow rate does have a favorable effect on reproducibility control. Experimental data also demonstrate that the dispensing process is quite reliable in terms of reproducibility control when production rate is up to 60-120/min.

Figure 3 and Figure 4 show the distribution of droplet volumes at production rates of 8/min and 60/min

respectively. Consistent distributions were obtained with several groups of samples.

C. Effect of viscosity on reproducibility

TABLE III
EFFECT OF VISCOSITY ON REPRODUCIBILITY

Viscosity (Csi)	CV (%)
1	5.05
15	3.98
58	4.40

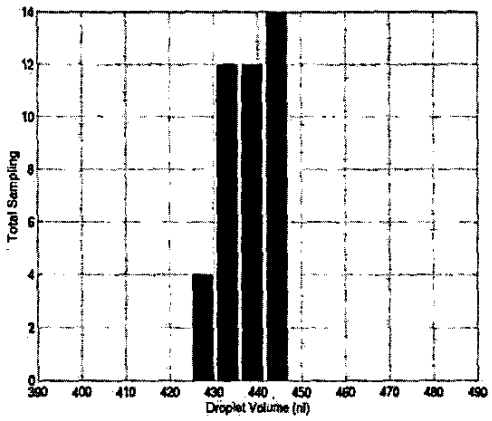

Fig.3 Distribution of droplet volume of KCL solution on chip of 1mm pitch size and 600mm gap at production rate 8/min

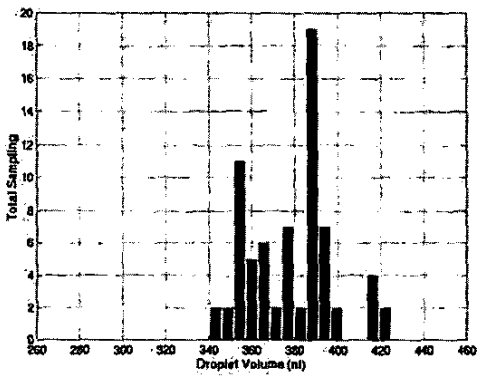

Fig.3 Distribution of droplet volume of KCL solution on chip of 1mm pitch size and 600mm gap at production rate 60/min

By dissolving sucrose concentration in KCL solution, dispensed solution with viscosity between 1 Csi and 58 Csi were made. Table 3 shows that the reproducibility of the dispensing process is almost independent from the viscosity of the dispensed solution. The viscosity range tested is believed to cover the viscosities of most chemical and biological solutions.

D. Effect of needle diameter and channel geometry on reproducibility

Two types of needles were tested, one with 300mm diameter, another with 500mm diameter. Experimental data shows that there was no significant difference in reproducibility as long as the flow rates were maintained similarly for the two types of needles. Instead, the hydrophobic property of the needle outlet as well as the position of the needle with respect to electrode pitch are relevant to the reproducibility of the dispensing process.

Channel geometry, as observed, has little influence on reproducibility. However, it is noted that when channel aspect ratio (gap/pitch size) is reduced to as small as 0.15, it becomes difficult to split droplets away from the needle outlet through electrowetting, The variation of splitting time results in large variations of droplet volume. According to experimental observation, it is favorable to keep the aspect ratio around 0.4.

IV CONCLUSIONS

A method has been presented for generating droplets in an oil-based electrowetting actuator system, thus providing liquid loading and/or continuous flow coupling to electrowetting droplet handling. The method has the advantages over conventional microdispensing techniques that allow suitable metering of variable volumes of samples while maintaining precise control of volumes electronically. The relative independence of dispensing reproducibility on microchannel structure and fluid properties (viscosity, surface tension, etc) makes it applicable to the preparation of a wide range of chemical samples. In addition, the electrically controllable capacitance metering, pump, valve as well as actuation automate the dispensing process and allow fast droplet generation. By the same merit, droplet generation-on-demand is becoming an easy task.

ACKNOWLEDGEMENT

The authors wish to acknowledge the assistance of Michael G. Pollack.

REFERENCE

[1] R. B. Fair, M.G. Pollack, R. Woo, V.K. Pamula, H. Ren, T. Zhang, and J. Venkatraman, " A microwatt metal-insulator-solution-transport (MIST) device for scalable digital bio-microfluidic systems", Tech.Digest, *IEEE Int. Electron Device Meet.*, Washington DC, USA, (Dec. 2-5, 2001).

[2] M. G. Pollack, R. B. Fair, and A. Shenderov, "Electrowetting-based actuation of liquid droplets for microfluidic applications", *Appl.Phys.Lett.* 77, (2000) p. 1725
[3] T Laurell, L Wallman and J Nilsson, "Design and development of a silicon microfabricated flow-through dispenser for on-line picolitre sample handling". *J.Micromech. Microeng.* Vol.9 (1999) 369-376
[4] Don Rose. "Microdispensing Technologies in drug discovery". *Drug Discovery Technologies.* Vol,4 No.9 Sep 1999.
[5] United States Patent: 5741554

0-7803-7538-6/02/$17.00/©2002 IEEE