An electrode design for droplet dispensing with accurate volume in electro-wetting-based microfluidics

Wei Wang, Jianfeng Chen, and Jia Zhou

View online: http://dx.doi.org/10.1063/1.4954195
View Table of Contents: http://scitation.aip.org/content/aip/journal/apl/108/24?ver=pdfcov
Published by the AIP Publishing

Articles you may be interested in

Automatic droplet transportation on a plastic microfluidic device having wettability gradient surface

Size-variable droplet actuation by interdigitated electrowetting electrode

Droplet jumping by electrowetting and its application to the three-dimensional digital microfluidics

Droplet formation in a T-shaped microfluidic junction

Electrowetting-based actuation of liquid droplets for microfluidic applications
An electrode design for droplet dispensing with accurate volume in electro-wetting-based microfluidics

Wei Wang, Jianfeng Chen, and Jia Zhou
State Key Laboratory of ASIC and Systems, Fudan University, Shanghai 200433, People’s Republic of China
(Received 5 May 2016; accepted 6 June 2016; published online 15 June 2016)

Electro-wetting-on-dielectric actuation enables droplets, the basic units of digital microfluidics, to be manipulated on a two-dimensional surface, providing a versatile platform for chemical assays or multi-step operations at micro- or nano-scale. In this paper, we carry out characterization experiments to demonstrate an electrode design to improve the reproducibility of on-chip droplet generation with no extra external apparatus. The overall reproducibility for a sequence of droplets dispensed consecutively from a non-refilling reservoir can be limited within ±0.5%. Results from the repetition of 1000 iterations offer the long-term reproducibility in the range of ±1%, indicating its validity in practical applications. Published by AIP Publishing.

Digital microfluidics (DMF), reagents-contained droplets can be dispensed and manipulated reliably on-demand with no carrier flow or micro-channel, catering to the need of reagent dosing handling in LOC applications.7–9

DMF system based on electro-wetting-on-dielectric (EWOD) is a promising technique manipulating droplets in either single- or two-plate geometries by proper control of an electrode sequence.10 While the fluidic instability is essential to induce the pinch-off process, the dispensing process is subject to many uncertainties as it is affected not only by operation parameters (e.g., actuation sequence or driving voltage) but also by many random parameters (e.g., hydrophobic coating, dielectric properties, as well as their time dependencies).10 Thus, the basic EWOD unit is not capable of precise volume handling in high-precision applications unless parameters are designed specific for every application.11,12

To improve the reproducibility of EWOD system, off-chip pressure sources are often adopted to assistant droplet dispensing13 and the system uncertainty can be further eliminated with volume metering mechanism.14 EWOD unit integrated with on-chip feedback mechanism on multilayer printed circuit board (PCB) was demonstrated by Gong et al.10 However, the extra pump or feedback set-up compromises the simplicity of EWOD systems, which is considered as one of its greatest strengths. To improve the reproducibility while maintaining the simplicity of EWOD system at the same time, an alternative method called passive dispensing was recently described by Wheeler et al.15 However, since the dispensed droplet is stuck in hydrophilic sites, difficulties can be predicted for multi-step process where several droplets react with each other. Besides, an approach was demonstrated by gradually ramping down voltage instead of abruptly switching off electrodes with extra controlling circuits.16 A summary of published studies regarding droplet dispensing is presented in Table I. It is of note that walled-in reservoirs and extra electrodes within are adopted to adjust the pressure from reservoir in time while dispensing, thus regulates the liquid volume dispensed into the droplet.11,14

While almost all operation parameters and many random parameters can affect the final volume, previous works in Table I adopted several methods to monitor and constantly adjust the dispensed volume. Our goal is to develop a EWOD device with high reproducibility while exercising its simplicity with no peripherals other than electrical connections.

Soda lime glass with indium tin oxide coating was used for a two-plate EWOD structure with a design of electrodes as shown in Fig. 1, following the fabrication procedure described elsewhere.17 About 2 μm cyanoethyl pulluan (CEP) and 80 nm Teflon-AF 1600 was spin-coated as the dielectric layer and the hydrophobic layer, respectively. Multiple studies have reported that the small gap results in a higher control sensitivity with no peripherals other than electrical connections.

To characterize the reproducibility, an image of the EWOD chip was recorded above with a CCD camera immediately after dispensing to estimate the droplet volume. The
standard deviation (SD) is divided by the mean (MN) to demonstrate the reproducibility, which is also called the coefficient of variation (CV),

\[ CV = \frac{SD}{MN} = \frac{1}{\mu} \left[ \frac{1}{N} \sum_{i=1}^{N} (x_i - \mu)^2 \right]^{1/2} \]

where \( \mu \) is the mean volume and \( x_i \) is the volume of number \( i \) droplet in total \( N \) droplets, making the reproducibility of droplets with different mean volumes comparable. Ten water droplets were dispensed from a non-replenishing reservoir each time to calculate its CV and the process was repeated 5 times for the error bar.

Herein, we propose an electrode design to stabilize the droplet shape before pinch-off occurs and the droplet dispensing process is shown in Fig. 1. Breaking electrode is designed with two outer arcs and a center dumbbell imitating the natural shape of cutting process. Typical two-step dispensing process (extrusion and breaking) is expanded to three steps (extrusion, necking, and breaking) to achieve higher controllability of the dispensing process, while necking step is added between the typical extrusion and breaking steps. The liquid is first extruded from the reservoir along the actuated sequence of electrodes in Fig. 1(a). Then, the extruded liquid on the breaking electrode is extracted from hydrophobic lateral arcs to the actuated center dumbbell to form a thin and stable neck connecting the reservoir and the creation site as in Fig. 1(b). Finally, the center dumbbell is turned to hydrophobic to pinch the liquid off at the finest location in the center dumbbell, dispensing a droplet on the creation site as Fig. 1(c). For the electrode structure in Fig. 1, \( L \) and \( W \) present the length and width of the breaking electrode, respectively, while \( a \) is the distance of two meniscus sides on the center dumbbell (denoted as neck width).

When breaking process occurs, liquid on the breaking electrode is pulled back to the reservoir and radius of its curvature \( R_b \) decreases until the two necking menisci meet and pinch off the droplet. The volume change on the creation site \( \Delta V_c \) can be expressed as a function of the flow rate from breaking electrodes to the creation site based on two-dimensional hydrodynamic equations. Consequently, the dispensed droplet volume \( V_c \) can be calculated by integrating \( \Delta V_c \) from the start until pinch-off occurs\(^{10}\)

\[ V_c = V_{c0} + \int_{t=0}^{\text{pinchoff}} f(R_b(t), V_c(t)) dt = V_{c0} + V_{cd}. \]

**TABLE I. Summary of published studies aiming at improving the reproducibility in EWOD devices.**

<table>
<thead>
<tr>
<th>Number of dispensed droplets</th>
<th>Volume variation</th>
<th>Pressure source?</th>
<th>Feedback control?</th>
<th>Walled-in reservoir with extra electrode within?</th>
<th>Medium</th>
<th>Reference</th>
</tr>
</thead>
<tbody>
<tr>
<td>6</td>
<td>0.7%–13.8%</td>
<td>No</td>
<td>No</td>
<td>No</td>
<td>Air</td>
<td>15</td>
</tr>
<tr>
<td>48</td>
<td>0.4%</td>
<td>No</td>
<td>No</td>
<td>No</td>
<td>Oil</td>
<td>11</td>
</tr>
<tr>
<td>5</td>
<td>1%</td>
<td>No</td>
<td>No</td>
<td>Yes</td>
<td>Air</td>
<td>12</td>
</tr>
<tr>
<td>12</td>
<td>1%</td>
<td>No</td>
<td>Yes</td>
<td>No</td>
<td>Oil</td>
<td>10</td>
</tr>
<tr>
<td>&gt;5</td>
<td>&lt;5%</td>
<td>Yes</td>
<td>No</td>
<td>No</td>
<td>Air</td>
<td>13</td>
</tr>
<tr>
<td>42</td>
<td>5%–6%</td>
<td>Yes</td>
<td>Yes</td>
<td>Yes</td>
<td>Oil</td>
<td>14</td>
</tr>
<tr>
<td>5</td>
<td>&lt;1%</td>
<td>No</td>
<td>No</td>
<td>No</td>
<td>Oil</td>
<td>16</td>
</tr>
</tbody>
</table>

**FIG. 1. Schematics and photos of EWOD dispensing process with our electrode design (top view).** (a) Extrusion step: The electrode sequence is actuated to extrude liquid out from the reservoir. (b) Necking step: Liquid is extracted from lateral to the media to form a thin and stable neck on the center dumbbell, connecting the reservoir and the creation site. (c) Breaking step: Center dumbbell is grounded to break the neck and pinch the droplet off from the reservoir. (d)–(f) Photos of extrusion, necking, and breaking step, respectively. \( L \) and \( W \) present the length and width of the breaking electrode, respectively, while \( a \) is the width of the finest point in the center dumbbell (denoted as neck width). The pink electrodes are applied to the driving voltage with blue ones applied to ground and the purple circular contour indicates deionized water (DI water).
As the liquid volume on actuated electrodes before necking starts is the same for a given device \((V_{c0})\), the volume variation mainly comes from the integration section \((V_{cd})\). After necking step in our design, \(R_b\) can be roughly the curvature radius of the meniscus side on the center dumbbell. The pinch-off process occurs in a flash after the center dumbbell turned to hydrophobic as little liquid needs to be pulled back to the reservoir. What’s more, the pinch-off point can be restricted within a small range due to the effect of surface tension. Thus, the final volume of the dispensed droplet can be approximated to the initial \(V_{c0}\).

Designs with different neck widths and electrode lengths are fabricated and results are illustrated in Fig. 2. The variation of droplet volume gradually decreases to \(\pm 0.5\%\) and the CV is as small as 0.38\% when the neck width reduces from 2 mm to 0.05 mm while \(L\) and \(W\) are both set as 2 mm. What’s more, CV increases when the breaking electrode elongates under 0.1 mm wide neck and 2 mm wide breaking electrode. Longer breaking electrode and wider neck mean larger \(R_b\) and more liquid on the center dumbbell. Thus, the actual pinch-off point lies in a relatively larger range and longer pinch-off time can also induce larger volume variation. As large volume fluctuation is undesirable, the center dumbbell of breaking electrode should be fabricated with a fine and short neck to gain a better reproducibility.

However, when neck width increases, uncontrollable growing fluctuation of \(R_b\) and pinch-off time change the droplet shape response, thus significantly affect the final dispensed volume with a changing drift. For ten droplets dispensed in a typical EWOD unit, the droplet volume increased monotonously and CV reached 5.42\% as the reservoir shrank to nearly empty, as shown in Fig. 3(a). This phenomenon mainly comes from the decrease of the force drawing liquid back to the reservoir. Assuming the force acting on per unit length of contact line (noted as \(F\)) remains the same around the same electrode, the overall electro-wetting force on the liquid can be calculated by integrating \(F\) as

\[
F_{\text{total}} = \int F dl = \frac{1}{2} C_d V_d^2 L_{\text{eff}},
\]

where \(C_d\) is the capacitance of dielectric layer and \(V_d\) is the voltage falling on it. The overall force induced by electro-wetting is proportional to \(L_{\text{eff}}\), the length of liquid boundary on active electrodes. When droplets are dispensed continuously without refilling the reservoir, the force pulling liquid out of reservoir is constant as the volume of each droplet changes in a relatively small range, but the draw-back force reduces rapidly as the reservoir shrinks by the volume dispensed, about 360 nl each time in this case. Consequently, less liquid is drawn back to the reservoir, resulting in the rise of individual droplet volume.

What is more, assuming the pinch-off point lies right at the middle of the neck, the liquid volume supposed to be dispensed (blue dots) and experimental results (black squares) are shown in Fig. 3(b), demonstrating an increasing gap between them. When the center dumbbell extracts to pinch off, the draw-back force would act on all liquid extruded out of the reservoir, including liquid on the creating site. Consequently, a longer pinch-off time means more liquid would be pulled back and the volume of each droplet would be smaller. The difference between the experimental results and ideal values becomes larger when neck width increases as more liquid is drawn back to reservoir in longer pinch-off time.

In experiments described above, only 10 droplets were dispensed each time, as it is enough for most portable and point-of-care usages. However, in some other applications, the reservoir is usually much larger than every single droplet, which indicates the reservoir is approximately constant in dispensing process. Experiments were carried out to test the long-term reliability and reproducibility of droplet dispensing with the electrode design. A droplet was dispensed and then transported back to the reservoir. The process was repeated until we got enough droplets. Based on our previous works\(^17,23\) a well-designed EWOD device can dispense thousands of droplets without breakup.
Results in Fig. 4 illustrate how the droplet volume varies when L, W, and a are 2 mm, 2 mm, and 0.05 mm, respectively. In Fig. 4(a), 200 droplets were dispensed and the properties of dielectric layer have no significant change. However, there is an initial period where about 20 droplets are dispensed with relatively large volume variation shown in Fig. 4(b) due to the mismatch between the reservoir and the electrode it sits on. Volume of 1000 droplets dispensed consecutively in one process was noted as one set and the overall process was repeated to get 4 sets of data. Fig. 4(c) is
presented here to demonstrate the distribution of droplet volumes. It is worth to note that more than 95% droplets fall within the range of ±1% volume variation for every single set and the variation of average volumes between every two sets is also less than ±1%. However, a slight degradation of reproducibility (from ±0.5% to about ±0.9%) was detected at times when hundreds of droplets were dispensed. After that, the variation in volume becomes larger over time until it fails to dispense a droplet. This phenomenon indicates changes occurring in the device which affects the factors in dispensing process. Since all the operational parameters remain constant throughout all steps and our previous works\(^\text{17}\) show CEP properties are stable in the long time test, it is likely that this is caused by degradation of the hydrophobic layer.

Droplet dispensing is an essential step for digital microfluidic systems and the volume of droplet is determined in the dispensing process. In this paper, uncertainties in the droplet pinch-off process have been analyzed and a design of breaking electrode is demonstrated for EWOD devices without introducing additional apparatus. The reproducibility of droplet volume falls in the range of ±0.5% for DI water when expanding the one-step breaking process into two steps: necking and final breaking. This all electrical, simple, and an efficient method brings an improved uniformity of droplet volume, demonstrating a promising prospect for practical applications.

This work was supported by the National Science Foundation of China with Grant No. 61176110, and the State Key Laboratory of ASIC and Systems (Fudan University) of China with Grant No. 2015MS001.