A new online monitoring method for water-in-oil droplet based microfluidic devices

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Abstract—The online water-in-oil (w/o) droplet monitoring is essential for the uniformity of the droplet based microfluidic devices. In this work, a new contactless impedance detection (CID) method for online w/o droplet monitoring in microfluidic devices is proposed. In the proposed method, a new CID sensor, which can work at low working frequency and overcome the unfavorable influence of coupling capacitance, is developed to obtain the impedance information of the w/o droplet. Based on the obtained impedance information, measurement models are established to realize online measurement of drop length and dispersed phase holdup. Then the coefficients of variation (CVs) of the two measured parameters are calculated to evaluate the uniformity of the droplets for online monitoring. Online monitoring experiments of w/o droplet were carried out in a microfluidic device with a 500 μm × 500 μm microchannel. The experimental results verify the effectiveness of the proposed droplet monitoring method. The new developed CID sensor can realize impedance measurement at a working frequency lower than 50kHz. The drop length and dispersed phase holdup are useful parameters for online monitoring of droplets. With the established measurement models, the maximum relative error of droplet length measurement is less than 10% in the droplet length range of 1.5mm-3.5mm. And the maximum error of dispersed phase holdup measurement is less than 5% in the dispersed phase holdup range of 0.20-0.67.

Index Terms—microfluidic, droplet monitoring, contactless impedance detection, droplet length, dispersed phase holdup

I. INTRODUCTION

In the past two decades, droplet based microfluidics has gradually become a significant category in the research field of microfluidics. It can be used to fabricate intricate droplet based particles and generate droplets as microscale flow reactor [1-7]. Therefore, droplet based microfluidic devices which can generate water-in-oil (w/o) droplets have received great attention for applications in diagnostics, cellomics, proteomics, drug discovery and synthetic biology [3-6]. For these devices and applications, the parameter monitoring of droplets is important for the uniformity of the generation of droplets [5-8]. However, it is difficult and complicated to predict the parameters of droplets theoretically [1-9]. So, to better control the parameters of droplets, the online w/o droplet monitoring is essential for droplet based microfluidic devices [10-15].

Currently, optical methods such as laser-induced fluorescence, UV-vis absorption, infrared and Raman spectroscopies are commonly used methods for droplet monitoring [10-15]. These methods are non-invasive and have accurate measurement performance. But because they require off-chip instrumentation and are high-cost, they still cannot meet the growing requirements of online droplet monitoring in microfluidic devices [11-15]. In addition to optical methods, conductance detection methods are also used in microfluidic devices and they have good real-time performance [11-16]. However, conventional conductance detection methods are usually contact methods, which means the sensors are directly in contact with the fluid. That brings the drawbacks of polarization effect and electrochemical erosion [17-19]. Meanwhile, these contact sensors may disturb the generation and flow of the w/o droplets [10]. Besides, most of the contact conductance sensors are made of hydrophilic metal, which may cause unfavorable influence on the stability of the w/o droplets (the microchannel to generate and keep w/o droplets is supposed to be hydrophobic) [1-7]. So, to better meet the requirements of online w/o droplet monitoring in microfluidic devices, new monitoring methods, which can implement contactless measurement and has the advantages of good real-time performance, should be developed.

The emergence of contactless conductivity detection (CCD, also known as capacitively coupled contactless conductivity detection, C2D) technique provides a new approach for online w/o droplet monitoring in microfluidic devices [17-20]. Because the electrodes of the CCD sensor is not directly in contact with the fluid, the electrode polarization and electrochemical erosion effect can be avoided.
Up to now, the conventional CCD technique is mainly applied in the field of electrophoresis for ion concentration detection in capillaries [17-19]. The research works concerning the application of CCD technique in online w/o droplet monitoring – is limited [14-15, 20]. Cahill and co-workers extended the CCD technique to the contactless impedance detection (CID) technique and realized the online conductivity measurement of droplets in segment flow [20]. But in their work, the parameters of the droplet were not measured. Duarte and co-workers used an embedded CID sensor to measure the length of droplet [14]. But to reduce the capacitive reactance of the coupling capacitance, the working frequency of their sensor is relatively high, which will increased the unfavorable influence of the stray capacitance on measurement results. Therefore, although the above pioneering research has achieved significant progress, the related research is not sufficient. To better apply CCD (CID) technique to online w/o droplet monitoring, more research work should be undertaken.

This work aims to propose a CID method for online w/o droplet monitoring in microfluidic devices. By introducing an LC circuit, a new CID sensor is developed to obtain the impedance information of the w/o droplet. It can work at low working frequency and overcome the unfavorable influence of the coupling capacitance. With the obtained impedance information, droplet length measurement model and dispersed phase holdup measurement model will be developed for online measurement of droplet length and dispersed phase holdup of w/o droplets. Then, the coefficients of variation (CVs) of measured droplet length and dispersed phase holdup are calculated to evaluate the uniformity of the droplet and hence implement droplet monitoring. Online monitoring experiments of w/o droplets will be carried out to verify the effectiveness of the new proposed CID based monitoring method.

II. THE LOW-WORKING FREQUENCY CID SENSOR IN MICROFLUIDIC DEVICES

To show the universality of the proposed method, the microfluidic device used in this work was fabricated in a common way. A T-junction metal mold with a thickness of 500 µm was fabricated to facilitate the stripping of the polydimethylsiloxane from the mold. A portion (20 g) of PDMS prepolymer A and B with a ratio of 10:1 was poured on the mold. After baking at 85°C for 90 minutes, a PDMS block with a T-junction channel was formed. All channels were with the width of 500 µm. After baking, the PDMS block with the channel was carefully peeled off from the mold and was punched three 1.5 mm holes, two as the inlets (one for continuous phase and the other for dispersed phase) and one as the outlet of the microfluidic chip. Then, the channel side of the PDMS block was bonded to a pre-prepared PDMS film with a thickness of 20 µm via oxygen plasma treatment to form the microfluidic chip shown in Fig.1a. Finally, the microfluidic chip and a pre-prepared PCB with two electrodes of the CID sensor (the widths of the two electrodes were 1 mm and the gap between the two electrodes was 0.5 mm) were both plasma-activated and bonded together to form the microfluidic device shown in Fig.1b. Between the channel and the electrodes, there is a PDMS film with a thickness of 20 µm.

![Fig.1. The microfluidic device with the CID electrodes. (a) The microfluidic chip: (b) The schematic diagram of integrated CID based microfluidic devices. (c) The equivalent circuit of the device.](image)
requirements is developed. Fig. 2 shows the construction of the new developed CID sensor.

![Equivalent Circuit](image1)

At this working frequency, the overall impedance of the detection path is \((1 - 4\pi^2 f^2 L C_3) Z_x\) and \(I_{out}\) is:

\[
I_{out} = \frac{U_{in}}{(1 - 4\pi^2 f^2 L C_3) Z_x} \tag{4}
\]

Thus, according to Eq. (3) the amplitude of \(I_{out}\) is:

\[
|I_{out}| = \frac{|U_{in}|}{(1 - 4\pi^2 f^2 L C_3) |Z_x|} \tag{5}
\]

Because \(L, C_1, C_2\) are constants \((f)\) is pre-determined by Eq. (3), and \(|U_{in}|\) is also a constant. So, it can be known from Eq. (4) that the amplitude of \(I_{out}\) is inversely proportional to that of \(Z_x\). With the introduction of the LC circuit, the background impedance of coupling capacitance is eliminated. Meanwhile, according to Eq. (3), by adjusting the value of \(L\) and \(C_3\), the CID sensor can work at low working frequency to limit the influence of stray capacitance to a negligible level.

### III. THE RESPONSE OF THE NEW CID SENSOR TO DROPLETS

To verify the effectiveness of the new CID sensor, the response of the CID sensor to droplets is investigated. In this work, the value of \(L\) is set as 700.0mH, and the value of \(C_3\) is set as 12.0 pF. The values of \(C_1\) and \(C_2\) are approximately 0.8 pF. According to Eq. (3), the working frequency is 48.65 kHz.

Fig. 3 shows the experimental setup. Mineral oil with 3% EM90 was used as the continuous phase and aqueous solution with red food dye was used as the dispersed phase to generate the w/o droplets. Two syringe pumps (Pump 11 Elite, Harvard Apparatus) were used to pump the continuous phase and dispersed phase into the inlets of the microfluidic devices. After pumping these two fluids into the inlets of the microfluidic devices, the w/o droplets were generated in the T-junction geometry, then flew downstream and passed through the electrodes of the CID sensor. A digital camera with microscope was used to capture the photos of the droplets and an excitation/sampling unit (Zurich Instruments) was connected with the CID sensor to provide excitation signal, record the impedance information, and send the data to the computer.
The impedance information obtained by the developed CID sensor at the continuous phase flow rate of 500 µL/h and three different dispersed phase flow rates: (a) Dispersed phase flow rate of 300 µL/h. (b) Dispersed phase flow rate of 500 µL/h. (c) Dispersed phase flow rate of 700 µL/h.

Fig. 5 shows the impedance information obtained by the developed CID sensor at the continuous phase flow rate of 500 µL/h and three different dispersed phase flow rates (corresponding to the conditions in Fig. 4). It can be known that the impedance information obtained by the CID sensor is also periodic and show good accordance with the observations from Fig. 4. Meanwhile, as shown in Fig. 5, a w/o droplet is detected as a positive peak in the output current signal obtained by the CID sensor. This is because the conductivity and permittivity of a droplet are higher than those of the mineral oil, which means the amplitude of the impedance of a droplet is smaller than that of the oil phase. So, the output current increases when a droplet passes through the CID electrodes.

Besides, when the continuous flow rate is fixed at 500 µL/h, with the increase of the dispersed flow rate, the period of impedance information decreases. The height of each peak in the signal of the impedance information also increases (this result has also been obtained by Duarte and co-workers [14]).

The above analysis shows that the development of the CID sensor is successful. The sensor can effectively realize the impedance measurement of w/o droplets in microfluidic device, at a low working frequency of 48.65 kHz. The impedance information measured by the CID sensor can reflect the parameters of the droplets, which indicates the potential of the sensor in online monitoring of droplets.

IV. NEW ONLINE MONITORING METHOD FOR WATER-IN-OIL DROPLET BASED MICROFLUIDIC DEVICES

The control of droplet length and monitoring of droplet uniformity are the key points of droplet-based microfluidic applications. To better control droplet length and monitor its uniformity, online monitoring is carried out with the developed CID sensor. Because droplet length itself is the aim of control, and the droplet length and dispersed phase holdup can reflect the uniformity of droplets, the online monitoring of w/o droplets here is on the basis of the online measurement of these two parameters.
The new CID sensor
Output current
Total flow velocity
Feature extraction
Droplet length measurement model
Droplet length
Dispersed phase holdup measurement model
Online monitoring results of w/o droplets

Fig. 6. The scheme of the proposed online monitoring method.

Fig. 6 shows the scheme of the proposed online monitoring method. With the impedance information (amplitude of the output current) obtained by the new CID sensor, features which can reflect the interested parameters are extracted. With the features and the additional introduced total flow velocity, measurement model of droplet length is developed for online measurement of droplet length. Meanwhile, with the obtained features, measurement model of dispersed phase holdup is also developed for online measurement of dispersed phase holdup. With the online measurement results of droplet length and dispersed phase holdup, the uniformity of droplets is evaluated to implement the online monitoring.

A. The measurement of droplet length

To realize the measurement of droplet length with the output current of the CID sensor, the first step is to extract suitable features. Because the peaks of the output signal are resulted from the droplets, the parameters of peaks contain useful information of the droplet length and can be extracted as features. Among the characteristics of the peaks, peak width is a commonly used characteristic to reflect the volume of droplet [11, 14-15]. However, due to the complexity of contactless detection, the current output signal may slightly increase before the arrival of the droplet (at the position of the electrodes of the CID sensor). This brings difficulty for finding the beginning and the end of a peak, and hence makes the peak width hard to determine. Therefore, instead of peak width, the full width at half maximum $W$ is used as the feature to build the link between the output current signal and the droplet length. The full width at half maxima $W$ is calculated as the time difference between the two points in one peak with the amplitude values of $(I_{\text{max}} + I_{\text{min}})/2$, as shown in Fig. 7.

Fig. 7. The features of the output current of the CID sensor.

As the full width at half maximum $W$ reflects the time that the droplet staying at the sensing position of the CID sensor, the product of full width at half maximum and the total flow velocity $u_t$, i.e., $\varphi = W u_t$, can be used to establish the measurement model of drop length. To study the relationship between $L_d$ and $\varphi$, preliminary experiments were carried out. The droplet length was changed by changing the flow rates of the continuous phase and the dispersed phase. The reference value of the droplet length is obtained from the photos of droplets captured by the digital camera. The total flow rate is obtained from the micropump. Fig. 8 shows the relationship between $L_d$ and $\varphi$.

From Fig. 8, two observations can be obtained: (1) The $\varphi$ is relevant to the droplet length $L_d$. With the increase of $L_d$, $\varphi$ increases. (2) In the droplet length range of 1.2-3.3 mm, the relationship between $\varphi$ and $L_d$ shows good linearity. These indicate that the extracted feature is effective, and it is reliable to develop the droplet length measurement model with $\varphi$.

The pre-developed measurement model of droplet length is given in Fig. 8. Fig. 9 shows the flowchart to measure the droplet length in practical online monitoring. First, the full width at half maximum $W$ is extracted from the output current of the CID sensor. With the extracted $W$ and the introduction of the total flow velocity $u_t$, $\varphi$ is obtained. Finally, with the obtained $\varphi$ and the droplet length measurement model, the droplet length can be obtained.
The new CID sensor
Output current
Full width at half maximum W
Total flow velocity $u_t$
Droplet length measurement model

Fig. 9. The flowchart to measure droplet length.

**B. The measurement of dispersed phase holdup**

The dispersed phase holdup $\alpha$ is the ratio of the dispersed phase volume to the total volume of the fluid. Because the oil and water are all incompressible fluids, this ratio is proportional to the ratio of dispersed phase flow rate to the total flow rate, i.e.

$$\alpha = \frac{Q_d}{Q_t}$$  \hspace{1cm} (6)

Where $Q_d$ is the flow rate of the dispersed phase. $Q_t$ is the total flow rate calculated by:

$$Q_t = Q_d + Q_c$$  \hspace{1cm} (7)

Where $Q_c$ is the flow rate of the continuous phase.

Meanwhile, the generation of the droplets is generally periodic, and the oil and water are incompressible fluids, indicating that the dispersed phase holdup $\alpha$ is approximately equal to the ratio of the droplet volume $V_d$ to the total volume of the fluid $V_t$ in one period, i.e.

$$\alpha = \frac{V_d}{V_t} = \frac{V_d}{V_d + V_c}$$  \hspace{1cm} (8)

Where $V_c$ is the volume of the continuous phase in one period.

As the cross-sectional area of the channel is fixed, the volume of the droplet $V_d$ in one period is approximately proportional to the length of droplet $L_d$ in one period and the total volume in one period $V_t$ is proportional to the total length of the fluid $L_t$ in one period (the sum of the length of droplet and the length of the continuous phase between droplets), i.e.

$$V_d \approx S L_d$$

$$V_t \approx S L_t$$  \hspace{1cm} (9, 10)

Where $S$ is the cross-sectional area of the channel.

By substituting Eq. (9) and Eq.(10) to Eq.(8), the dispersed phase holdup $\alpha$ can be rewritten as:

$$\alpha \approx \frac{S L_d}{S L_t} = \frac{L_d}{L_t}$$  \hspace{1cm} (11)

As the droplet length can be calculated from $\varphi$, it is necessary to extract another feature that can reflect the total length $L_t$. Among the features of the output current signal, the time period $T$ is one of the basic features. It reflects the frequency of droplet generation. So, the period $T$ is extracted as another feature. $L_t$ is approximately the product of the period $T$ and the total velocity $u_t$:

$$L_t = T u_t$$  \hspace{1cm} (12)

Here, $T$ is calculated by the time difference of the two nearest peak points with the value $I_{max}$ in the amplitude of the output current, as shown in Fig. 7.

Meanwhile, because $L_t$ is equal to $T u_t$ in one period and the droplet length $L_d$ is the linear function of $W u_t$, the dispersed phase holdup can be calculated just from $\beta$, which is defined as the ratio of $W$ to $T$:

$$\beta = \frac{W u_t}{T u_t} = \frac{W}{T}$$  \hspace{1cm} (13)

That means the measurement of dispersed phase holdup can be realized without the introduction of the total flow velocity $u_t$.

**Fig. 10. The relationship between $\beta$ and $\alpha$**

To study the relationship between $\beta$ and $\alpha$, preliminary experiments were carried out. Similarly, dispersed phase holdup is also changed by changing the flow rates of the continuous phase and the dispersed phase. The reference value of dispersed phase holdup is calculated as the ratio of the dispersed phase flow rate to the total flow rate (both obtained from the micropump). Fig. 10 shows the relationship between $\beta$ and $\alpha$, with linear regression adopted. From Fig. 10, again two observations can be obtained: (1) The ratio of the two extracted features $\beta$ is relevant to the dispersed phase holdup $\alpha$. With the increase of $\alpha$, $\beta$ increases. (2) In the dispersed phase holdup range of 0.17-0.67, the relationship between $\beta$ and $\alpha$ shows good linearity. These indicate the effectiveness of the two extracted features and show that it is reliable to develop the model with $\beta$ to realize dispersed phase holdup measurement.

The developed measurement model of the dispersed phase holdup is given in Fig. 10. Fig. 11 shows the flowchart to measure the dispersed phase holdup. With the impedance information of w/o droplet in the microchannel obtained by CID sensor, the time period $T$ and full width at half maximum $W$ are extracted and $\beta$ is calculated. Then, with the dispersed phase holdup measurement model, the dispersed phase holdup can be obtained from $\beta$. 
uniformity of droplets (the actual CV of droplets) should be presented by experiment.

V. EXPERIMENTAL RESULTS

A. Online measurement results of droplet length and dispersed phase holdup

To verify the effectiveness of the proposed method, practical online monitoring experiments were carried out. The range of dispersed phase flow rates was 200 to 700 µL/h and the range of the continuous phase flow rate was 333 to 800 µL/h. Correspondingly, the investigated droplet length was from 1.2 to 3.3 mm and the dispersed phase holdup was from 0.2 to 0.67. Fig. 12 shows the measurement results of droplet length. Fig. 13 shows the measurement results of dispersed phase holdup.

With the two CVs, the droplets can be monitored. For most applications of microfluidic devices, droplets with CV less than 3% can have good performance [7,15]. But in this work, the CVs calculated with the measured droplet length and dispersed phase holdup may not be equal to the actual CV of droplets. So, in practical monitoring, the quantitative standard of the two CVs to ensure a good uniformity may be different. To verify the effectiveness of the proposed method and implement online monitoring of droplets, the correlation between the CVs of the two measured parameters and the

C. The online monitoring of droplets

As aforementioned, the drop length and dispersed phase holdup can reflect the uniformity of droplets. Here, the online monitoring is implemented with the two parameters. The coefficients of variation (CVs) of the two parameters are used to evaluate the uniformity of the droplets. The definitions are:

\[
CV_L = \frac{\sigma_L}{\mu_L} \quad (14)
\]

\[
CV_\alpha = \frac{\sigma_\alpha}{\mu_\alpha} \quad (15)
\]

Where \(CV_L\) is the CV of the measured drop length and \(CV_\alpha\) is the CV of the measured dispersed phase holdup. \(\mu_L\) and \(\mu_\alpha\) are the mean values of the measured droplet length and the dispersed phase holdup. \(\sigma_L\) and \(\sigma_\alpha\) are the standard deviations of the measured droplet length and dispersed phase holdup, respectively. The definitions of \(\sigma_L\) and \(\sigma_\alpha\) are:

\[
\sigma_L = \sqrt{\frac{1}{M-1} \sum_{m=1}^{M} (L_{dm} - \mu_L)^2} \quad (16)
\]

\[
\sigma_\alpha = \sqrt{\frac{1}{M-1} \sum_{m=1}^{M} (\alpha_m - \mu_\alpha)^2} \quad (17)
\]

Where \(M\) is the number of measured droplets. \(L_{dm}\) and \(\alpha_m\) are the measured droplet length and dispersed phase holdup of the \(m\)th droplets, respectively. \(m = 1, 2, \ldots, M\).

Fig. 11. The flowchart to measure dispersed phase holdup.

Fig. 12. The online measurement results of droplet length.

Fig. 13. The online measurement results of dispersed phase holdup.
B. Online monitoring results of the w/o droplets

As mentioned above, the uniformity of the droplets is also the aim of monitoring and it is evaluated by the CVs of drop length and dispersed phase holdup. In the online monitoring experiment, the two CVs of the two measured parameters are calculated according to Eq. (14)-Eq. (17). Fig. 14 and Fig. 15 shows two examples of the generated w/o droplets in the experiment. The dispersed phase flow rate and continuous flow rate of Fig.14 are all 500 µL/h. The dispersed phase flow rate and continuous flow rate of Fig.15 are 500 µL/h and 700 µL/h, respectively.

For the case in Fig.14, the CVs of the measured droplet length and the dispersed phase holdup are 0.54% and 0.61%, respectively. It can be seen from Fig.14 that the uniformity is good (the actual CV of droplet length calculated from the photos of droplets is less than 3.0 %). Notation: 3.0 % is a widely used standard/threshold of the actual CV to evaluate the uniformity of the droplets [1-2, 7, 15, 32-33]. If the actual CV is less than 3.0 %, the droplets are regarded as uniform.). For the case in Fig.15, the CVs of the measured droplet length and the dispersed phase holdup are respectively 0.88% and 1.78%, which are significantly higher than those of the case in Fig. 14. It can be seen from Fig.15 that the uniformity of this case is not as good as the case in Fig. 4 (the actual CV of droplet length calculated from the photo of droplets is greater than 3.0 %). So, for both the two cases, the value of the calculated CVs can reflect the actual uniformity of the droplets, which is in accordance with the actual CVs calculated from the photos. The CVs of the droplet length and dispersed phase holdup measured by the proposed method can reflect the actual uniformity of droplets, which means that the proposed method is effective.

Experimental results show that there exists approximate linear relationship between the calculated CV and the actual CV of the droplet length. Generally, for cases where the calculate CV is small, the actual CV is small. And, for cases where the calculate CV is large, the actual CV is large. However, the fluctuation and the discreteness of the experimental data are relatively large. At the current stage, it is difficult to develop the relationship rigorously. According to the current experimental conditions and the obtained experimental results, we have found that the value of 0.8% (of the calculated CV) could be regarded as a threshold/boundary value to ensure the uniformity of w/o droplets. If the value of the calculated CV is less than 0.8%, the corresponding actual CV is less than 3%, which ensures good uniformity of the droplets. It is indicated that the proposed method can qualitatively evaluate the uniformity of the droplets and implement online droplet monitoring.

VI. CONCLUSION

This work proposes a new CID based method for contactless online w/o droplet monitoring in microfluidic devices. In the new-proposed method, a new CID sensor is developed to obtain the impedance information of the w/o droplet in microchannel. By introducing an LC circuit, the new CID sensor can overcome the unfavorable influence of coupling capacitance at low working frequency. With the obtained impedance information, two features (the full width at half maxima and the time period) are extracted to develop the measurement models of drop length and dispersed phase holdup. Online monitoring of w/o droplets is implemented by evaluating the variation of the measured drop length and dispersed phase holdup.

Online w/o droplet monitoring experiments were carried out in a T junction microfluidic device (500µm height and 500µm width). Experimental results verify the effectiveness of the proposed method. The CID sensor can obtain the impedance information of w/o droplets at low working frequency (48.65 kHz). The extracted features can effectively reflect the characteristics of the impedance information and the developed measurement models have good measurement performance. The maximum relative error for droplet length measurement is less than 10% in the drop length range from 1.2mm to 3.3mm. The maximum measurement error for dispersed phase holdup is less than 5% in the dispersed phase holdup range from 0.20 to 0.67. Results also indicate that the drop length and dispersed phase holdup are useful parameters for online monitoring of w/o droplets.

This work not only presents an online monitoring method for w/o droplet in microfluidic devices, but also provides an effective approach for dispersed phase holdup measurement in microfluidic devices. However, the research is not sufficient and more research should be undertaken in the future. To investigate the relationship between the calculated CVs and the actual CV, and to study the influence of experimental setup/conditions (such as parameters of the microchannel, geometrical parameters of the CID sensor, etc.) on this relationship will be our further research work.

REFERENCES


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