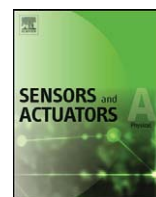




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A capacitive sensor for non-contact nanoliter droplet detection

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ABSTRACT

This paper reports on a sensor for the detection of microdroplets in flight. The presented sensor is based on a capacitive principle, which allows for non-contact monitoring of a complete droplet dispensing process. In the presented experiments the change in capacity caused by liquid droplets in the range of a few nanoliters passing through the electric field of the sensor is studied. From the capacitive change the droplet presence can be deduced with a reliability of 100%, which means that every single droplet dispensed within the experiments caused a significant signal change. In addition, the sensor signal is sensitive to the droplet's volume V , dielectric constant ϵ_r (epsilon) and velocity v . It turns out that every specific droplet exhibits a characteristic "fingerprint" signal depending on these parameters. Especially the droplet volume correlates very well with the peak value of the extracted signal. Therefore, the calibrated sensor is able to determine the volume of dispensed droplets in the range from 20 to 65 nl with a resolution of less than 2 nl. Furthermore, the printed circuit board (PCB) technology applied for fabrication of the sensor enables a very cost efficient and flexible realisation of the whole sensor unit. The non-contact capacitive principle prevents contamination and loss of media. Therefore, the proposed approach is well suited for high precision droplet presence detection and low cost online monitoring of liquid volumes in microdispensing processes for various applications.

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1. Introduction

Selective application of very small amounts of liquid has gained more and more attention over the past years. In the field of biomedicine, pharmacy and medical research for example, the use of chemical reagents portioned to several nanoliters to perform thousands of bio-chemical experiments daily (e.g. for combinatory chemistry or pharma research) has become a routine application [1]. But the demand for controlled small droplet application has also significantly increased in the field of conventional mechanical engineering and industrial production. Highly accurate proportioned quantities of adhesives, lubricants, cooling agent, etc. are required in certain fully automated industrial processes. For this purpose mostly contactless dispensing systems are used, which deliver single droplets with volumes in the range from pico- to nano-liters at high spatial resolution. Contactless dispensing systems deliver the dispensed liquid as free flying single droplets or jets. To enable an online quality control of production processes involving contactless dispensing systems it is desirable to monitor each individual droplet on its way to the target.

According to the small volumes of liquid and high evaporation rates, the quality control for these processes implicates certain

problems, thus high costs. Gravimetric and optical techniques routinely applied for droplet detection and measurement [2–4] are often unsuitable for compact integration and parallelization on liquid handling robots and for a direct quality control in fully automated processes.

The sensor presented in this paper is different from earlier work studying interaction of a droplet in contact to an electrode [5] or droplet counting [6]. In this paper the sensor principle is contactless and the droplets are much smaller (several hundred microns). Thus, the droplets can be used for any purpose after the sensing event. Furthermore, the use of standard printed circuit board technology like described in the following offers a cost efficient way for the fabrication of the whole sensor unit including the sensor capacitor and the signal conditioning circuit.

2. Working principle

The proposed droplet detection method is based on a media sensitive and volume dependent capacitive sensing principle as sketched in Fig. 1. A free flying droplet is introduced between the two plates of an open plate capacitor driven by an AC voltage. Plate capacitors are sensitive to changes in the geometrical arrangement of the plate electrodes or to alteration of dielectric characteristics [7].

A dispensed droplet, which passes through an open plate capacitor leads to a change of capacity due to its certain dielectric constant

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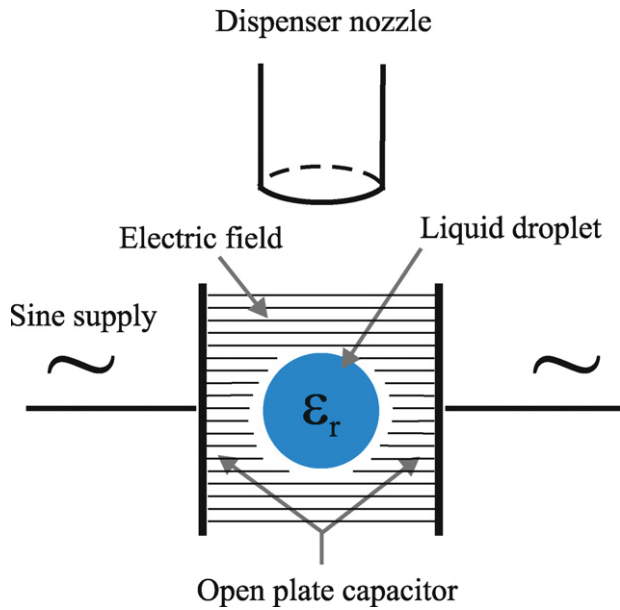


Fig. 1. Principle sketch of the capacitive measurement concept. A droplet passes through the electric field of an open plate capacitor.

[5,8]. This can be seen from the basic equation describing the capacitance of a plane parallel plate capacitor:

$$C = \epsilon_0 \epsilon_r \frac{A}{s} \quad (1)$$

where C is the capacitance of the capacitor, ϵ_0 is the electric permittivity of the vacuum, ϵ_r is the dielectric constant of the dielectric, A is the area of the plate electrodes and s is the distance of the plates. In case of this basic equation the volume of the dielectric has the same value as the space between the two capacitor plates. It is obvious that the capacitance depends on a geometric factor of the dielectric and its dielectric constant.

A droplet, however, which does not touch either of the capacitors electrodes, has to be considered as a dielectric which does only partly fill the dielectric space. Therefore, Eq. (1) has to be adapted to this geometrical condition. Fig. 2 shows three examples of capacitor arrangements and the corresponding equations to determine their capacitance.

An equation to calculate the change in capacity ΔC caused by a spherical shaped droplet as shown in Fig. 2c is derived from [5]

$$\Delta C = 4\pi\epsilon_0 \frac{r^3 \epsilon_r - 1}{s^2 \epsilon_r + 2} \quad (2)$$

Here r is the radius of the droplet, s is the distance of the capacitor plates and ϵ_r is the dielectric constant of the medium and ϵ_0 is the permittivity of the vacuum [9]. In case of pure water the value of the dielectric constant amounts to $\epsilon_r \sim 81$ ($f < 1$ GHz). Thus,

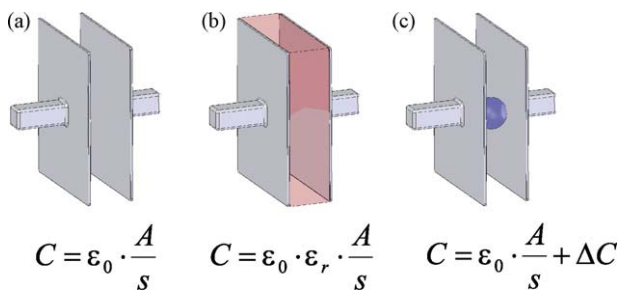


Fig. 2. Different capacitor arrangements with the corresponding equations to calculate their capacity.

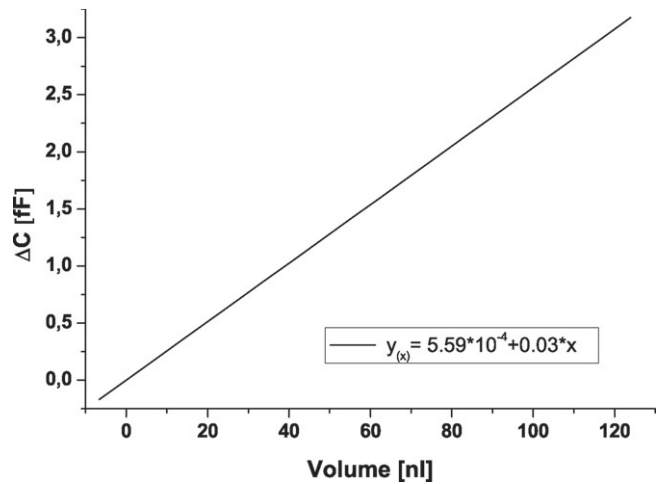


Fig. 3. Change in capacity ΔC versus the causative volume of pure water droplets according to Eq. (2).

the capacity change caused by a water droplet can be calculated straightforward using Eq. (2). For an open plate capacitor with a plate distance $s = 1$ mm this yields a droplet size dependent capacity change as displayed in Fig. 3.

It is shown that the expected changes in capacity caused by droplets in the range from 5 to 120 nl, which corresponds to droplet radii from 106 to 306 μm , are quite small and scale down to $\Delta C = 0.1$ fF.

Strictly speaking the shown results can be applied to exactly spherically shaped droplets only. However, since dispensed droplets often appear as spherical shaped bodies, the results from Eq. (2) provide a reasonable first approximation.

3. Fabrication

The capacitive detection of free flying droplets in flight requires a measurement capacitor, which enables a droplet to pass through its electric field.

An open plate capacitor has to be created, which conforms to the required geometrical conditions. To cope with the given requirements in a cost effective way, a certain feature of standard PCB technology is consulted. The implementation of the sensor capacitor is realised by the use of a standard PCB through connection (via), which can easily be set on a PCB at any position, in any size and without additional cost. This via is sliced by mechanical milling to create two opposite, cylindrical half shell electrodes, which are connected to the electric circuit of the sensor by standard strip lines. An advantage of cylindrical half shell electrodes is the increase of the capacitive interaction with spherical shaped bodies compared to plane parallel electrode capacitors [10].

The sensor circuit, which is completely arranged on the sensor PCB, and the open capacitor are fabricated in only one fabrication procedure, which establishes a very cost effective basis for a further completion of the sensor. Furthermore, the mentioned fabrication technology enables an easy alteration of the sensor size and shape to keep the sensor flexible for an adaption to any dispenser design and type. Fig. 4 presents the sensor PCB prototype adapted to a PipeJet™ dispenser [11], which was used for the whole work throughout this paper solely. The enlarged aspect visualises the mentioned sensor capacitor, fabricated by a standard through connection. The integration on the dispenser is realised by a special designed mechanical fixture, which clamps the sensor PCB underneath the dispenser unit, see Fig. 5.

Since the accuracy of capacitive measurement improves with the homogeneity of the electrical measurement field, the droplets

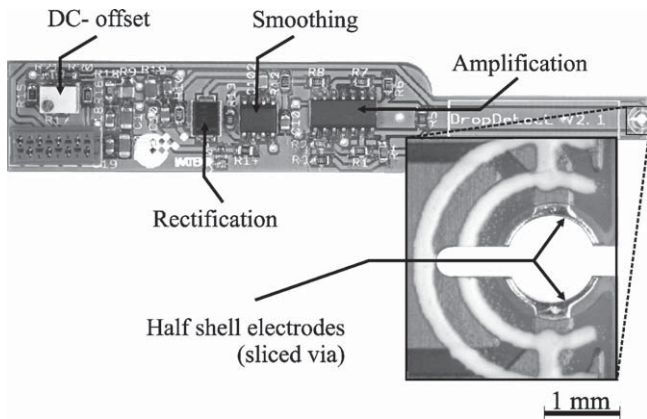


Fig. 4. Sensor PCB prototype adapted to the PipeJet™ dispenser; enlarged aspect—the sensor capacitor fabricated by a sliced standard through connection.

should pass centrally through the capacitor [5]. CFD (computational fluid dynamics) simulations and experiments to visualise electrical fields using dielectrophoresis to align yeast cells along the field lines [12] have shown that the central electric field of the cylindrical half shell electrode arrangement is nearly homogeneous, see Fig. 6. Thus, a major requirement to the sensor fixture is to adjust the dispenser nozzle concentrically to the capacitor's half shell electrodes to direct the flight path of the dispensed droplets to the centre of the capacitor.

A very important task, which has to be considered in designing the sensor capacitor is contamination or wetting of the electrode plates due to malfunctions in the dispensing process (satellites, deviation from the expected flight path, etc.).

The capacitor size, especially the distance of the capacitor plates (here the diameter of the used via) has to be well adapted in respect to the expected droplet volume and the accuracy of the dispensing system. Wetting of the capacitor plates would influence the measurement strongly and lead to unusable and inaccurate results.

The used PipeJet™ dispenser enables to vary the volumes of single dispensed droplets in the range from 10 to 100 nl by simple variation of its driving parameters (for more information see [13]). These volumes correlate with droplet radii from $r_{\min} = 133 \mu\text{m}$ to $r_{\max} = 288 \mu\text{m}$.

A distance of the capacitor plates (here the inner diameter of the sliced via) of $d_{\min} = 1.2 \text{ mm}$ ($r = 0.6 \text{ mm}$) facilitates a contamination free process for droplet sizes in the expected range. Further coat-

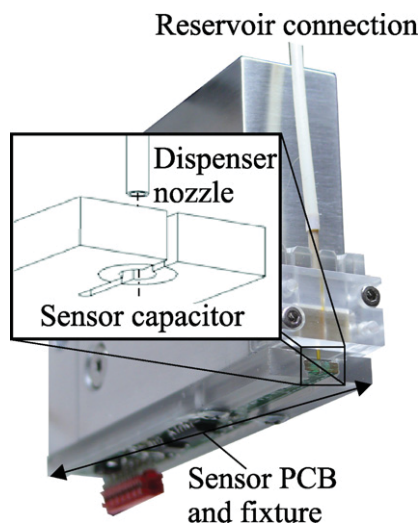


Fig. 5. PipeJet™ dispenser with implemented capacitive sensor.

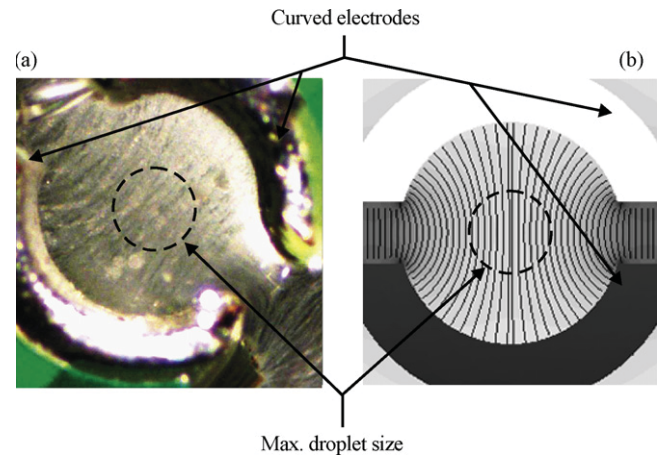


Fig. 6. Visualisation of the electric field of the cylindrical half shell electrode capacitor versus the maximal possible droplet size. (a) Yeast cells aligned at the electric field by dielectrophoresis; (b) computational simulation of the field lines (CFD).

ings, encapsulations or protections of the sensor capacitor are not processed yet.

4. Electrical conditions

The detection of capacity changes can be realised by a variety of methods [14]. In case of the presented sensor the detection principle is based on detuning of an active high pass filter. Fig. 7 shows a principle sketch of the basic transducing concept.

Since the frequency response of an electrical filter depends on the magnitude of the certain filter components, especially the filter's capacitor, an alteration of the capacitance leads to a shift in the frequency characteristic of the unit. While a droplet passes through the electrical field of the capacitor, its value changes for the time of flight of the droplet through the electric field. Therefore, the frequency characteristic of the filter shifts depending on the geometry and the material specific dielectric constant of the droplet.

Due to the constant sine wave supply frequency the amplitude of the passing signal changes and lead to the output signal. This measurement method enables to attain material and volume dependent signals contactless from dispensed, free flying microdroplets in flight.

To extract significant signals the sensor capacitor is supplied by an alternating voltage of $V_{pp} = 20 \text{ V}$ at a constant sine frequency of $f = 156 \text{ kHz}$.

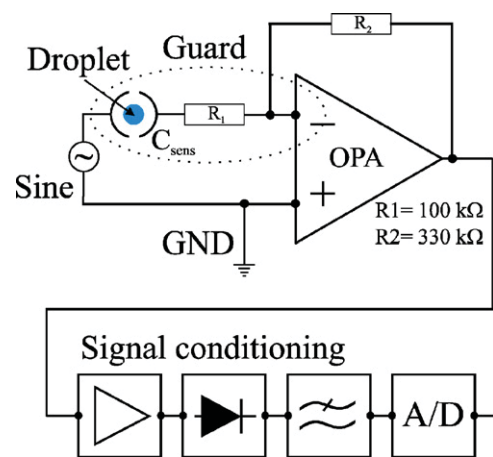


Fig. 7. Sketch of the signal generating unit, an inverting high pass filter.

Due to the occurring, very small changes in capacitance caused by single droplets, see Fig. 3, the generated signal has to be conditioned by an adapted analogue circuit. The depicted transducing unit is followed by further amplification steps, a diode rectification and an active smoothing of second order according to Butterworth.

The signal is amplified by three successive amplification steps. Due to the very small capacity of the measurement capacitor ($C_{\text{Sens}} \approx 50 \text{ fF}$) the signal has first to be amplified to an utilizable level by the first operational amplifier (OPA). The second OPA includes the addition of a DC offset voltage, which shifts the negative half waves of the signal out of the operation range of the used OPA. This is necessary to increase the sensitivity of the last amplification step. Therefore, the third step is able to amplify the tiny changes on top of a relatively large signal.

This signal conditioning is not very commonly used and one might rather consider a differentiator circuit or a lock-in principle for this purpose. However, the presented approach provides the benefits of easy guarding and avoids parasitic capacitances, which can be in the order of magnitude of the sensor capacitor [15,16]. This circuit works with a minimum amount of electronic components, which implicates very low space consumption and high cost efficiency.

Critical aspects, which have to be considered for capacitive measurement, are the effects of leakage current to the capacitive transducer and the influence of parasitic capacitance. To interrupt leakage surface- or bulk-currents, so-called guard rings are necessary, which act as third electrode. A guard ring is a conductive ring or area surrounding the transducing unit, which has to be connected to a low impedance point that is on the same potential as the input [17]. Due to the used inverting amplifying transducer unit, the input is virtually connected to the circuit's ground potential, thus the guard can be connected simply to ground as mentioned above [15]. The guard ring in case of the presented sensor is realised as an extensive ground area which surrounds the transducing unit completely, as well as the strip lines and the input pins of the transducers OPA with a distance of $300 \mu\text{m}$ [18].

The complete electric conditioning circuit is placed on the sensor PCB, shown in Fig. 4 and results in an output connector where the analogue signals can be sampled by a computer. (The sample delay for all experiments presented in this paper was $\Delta t = 8 \mu\text{s}$.)

5. Measurement setup

The characterisation measurement setup to evaluate the presented sensor consisted of the PipeJet™ dispenser (BioFluidix, Type R2b+, $500 \mu\text{m}$ dispensing tubes [13]), combined with the adapted sensor holder and the sensor PCB prototype shown in Fig. 4. The PipeJet™ dispenser is a piezo-electric driven dispenser, which enables the generation of single droplets in the nanoliter range on demand.

The volume and velocity of the droplets can be varied easily by adjusting the extension length and extension velocity of the piezo-electric actuator [11].

The correlation of the sensor performance to the actually dispensed single droplet volume requires a highly precise volume determination. Therefore, a highly precise gravimetric balance is used to measure the droplet volumes passing through the sensor independently. An additional optical observation of the dispensing processes was realised by a stroboscopic camera [19]. Fig. 8 shows a block diagram of the whole measurement setup.

The functionality of the sensor is based on a constantly alternating sine voltage supply of the sensor capacitor. This supply voltage is generated on a special designed supply board. The main part of the board is the XR2206 IC component from EXAR, an integrated circuit element, which generates adjustable oscillating waves. The amplitude of the generated sine signal is amplified by a simple one

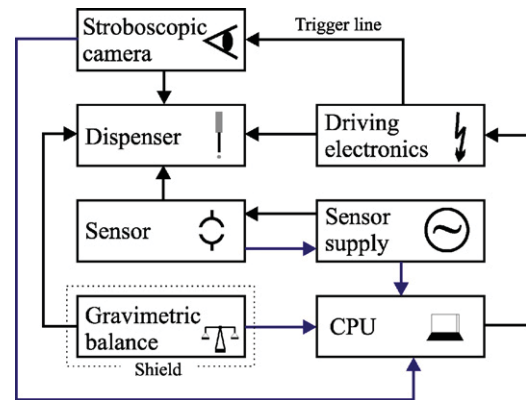


Fig. 8. Block diagram of the measurement setup.

step post-amplifying circuit. The whole supply board is packed in an external case, where the sensor can be connected via an 8 pole micromatch connector. The analogue signals are logged through the supply PCB, where they can easily be sampled by a guarded coaxial cable, connected to the BNC connector at the supply case.

Further tasks of the board are to provide the positive and negative supply voltage for the sensors active components (OPA), to provide the voltage for the DC offset and to enable the addition of an offset voltage to the sensor output signal.

In order to reduce high evaporation rates and environmental influences like air movement, etc. the flight path of dispensed microdroplets should be kept as small as possible. Therefore, the droplets have to be detected as close as possible to the dispenser's nozzle cut-off, which is realised by the adapted sensor fixture, see Fig. 5.

Fig. 9 shows a stroboscopic image of the front view of the sensor at the flight of a dispensed droplet through the sensor capacitor. After passing through the sensor capacitor the droplets impinge on the weighing table of the gravimetric balance. This measurement setup allows for the correlation of each single dispensed droplet to its actually dispensed mass.

The expected droplet volumes of a few nanoliters correspond in case of pure water to masses in the range of micrograms. The precise volume determination of such small quantities is realised by the SC2 high definition balance from Sartorius, which features a resolution of $\Delta m = 100 \text{ ng}$ and satisfies the challenge of the given requirement [20].

To attain precise and reliable results, the balance has to be set on a vibration damped table and has to be covered by a wind shield case to damp environmental influences on the measurement. High humid conditions inside this case inhibit the influence and falsification of the weighted mass due to evaporation. But the environmental influence has to be considered anyway. Special software algorithms evaluate the weighted mass of the dispensed

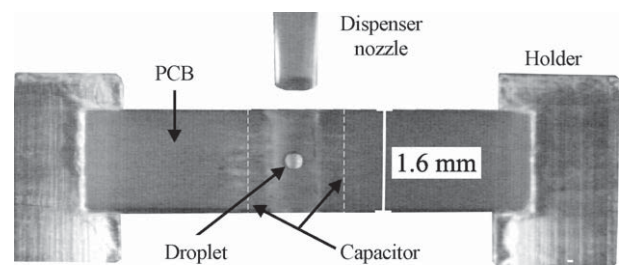


Fig. 9. Stroboscopic image of the sensor's front view at a dispensing cycle; a dispensed droplet is passing through the sensor capacitor.

single droplets in respect to the actual evaporation rates and vibrations.

6. Experimental results

The requirements for droplet detection can be very diverse. The easiest purpose is to determine the presence or absence of a droplet. In the best case, a so-called droplet counter delivers information about the number of dispensed droplets and identifies absent droplets. The performance of the presented sensor is far beyond this level. Significantly more information can be extracted from the analogue sensor signal like presented in the following.

To characterise the capacitive sensor nanoliter droplets of pure water were generated with the PipeJet™ and dispensed through the measurement capacitor's electrical field. A typical time dependent signal, generated by a droplet of pure water is shown in Fig. 10. The signal is correlated to pictures of the droplet's flight through

Sensor frontview

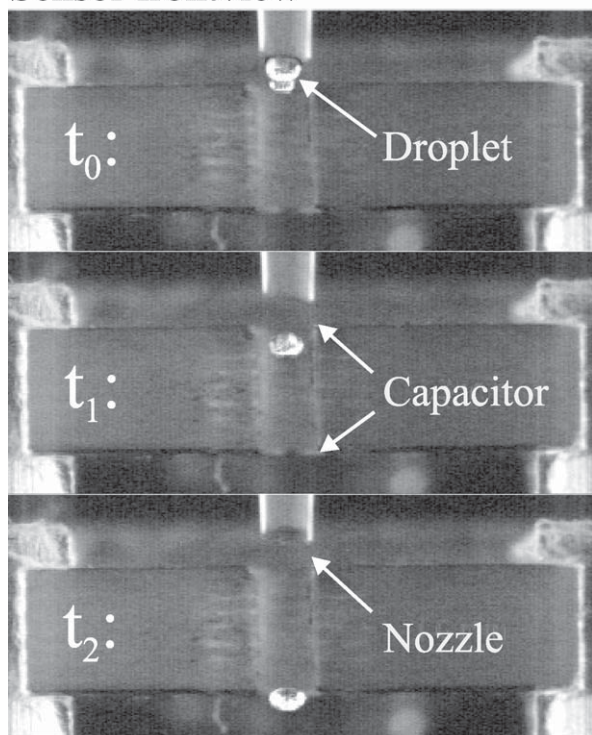


Fig. 10. Typical signal characteristic, generated by a droplet of pure water correlated to three significant positions of a droplet during the flight through the sensor capacitor.

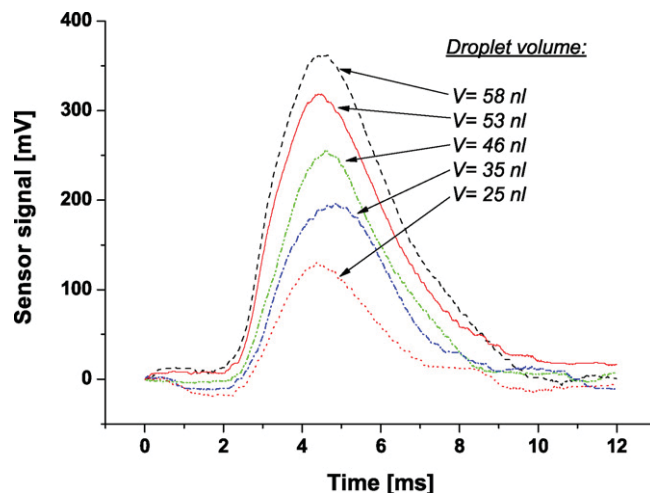


Fig. 11. Time dependent signals generated by droplets of pure H₂O of different size. Droplet volumes are determined by gravimetrical measurement.

the sensor capacitor at three different positions. The entrance behaviour of the droplet is characterised by a small falling edge from time t_0 to t_1 (cf. Fig. 10). The droplet's flight through the capacitor from time t_1 to t_2 results in a steep signal increase. The falling edge of the signal after t_2 results from the voltage decay in the electronic circuit to its initial value, after the droplet has passed.

In further experiments the sensitivity of the signal with respect to droplet size changes was investigated.

Therefore, the driving parameters of the dispensing process were kept constant and only the piezo-extension length of the actuator was varied to generate droplets of different size at constant velocity [11].

Fig. 11 displays the results from this experiment. Obviously, there is a significant correlation between the peak value of the signal and the droplet volume in the range from 25 to 57 nl. To determine the average correlation of the signal's peak value and the droplet volume a series of 100 single droplets was dispensed for volumes of 25, 35, 45, and 55 nl.

The peak value of each recorded signal was determined and is displayed as function of droplet volume in Fig. 12. Based on this data a linear fit was calculated, which can be considered as volume calibration for the sensor. To determine the predictive value of the fit, an error assessment was performed by statistical interval estimation

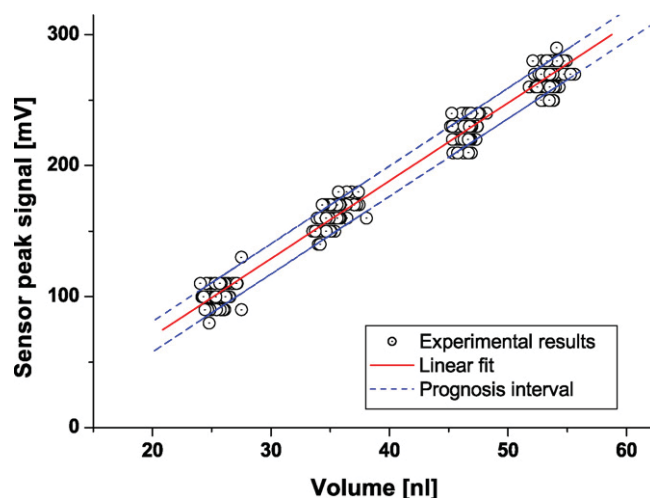


Fig. 12. Peak value of the sensor signals versus gravimetrically measured droplet volume including error assessment boundaries. Correlation coefficient $r_{x,y} = 0.992$.

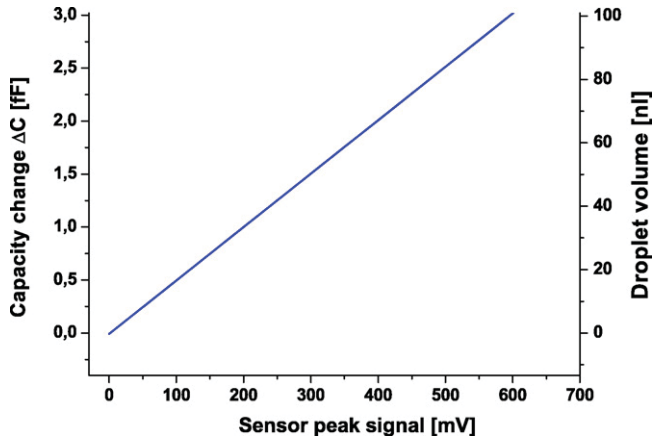


Fig. 13. Relation from theory (Eq. (2)) to the experimental results represented by the linear fit given by Eq. (3).

[21]. The calculated prognosis interval, with an error probability of 5%, is depicted in Fig. 12 as dashed lines.

This interval signifies that 95% of all droplets of a given size will cause peak signal values within these limits.

A relation connecting theory to the experimental result is shown in Fig. 13. It is derived by setting Eq. (2) equal to the experimentally gained linear fit given in Eq. (4). Thus, the graph relates the average sensor peak value to the change in capacity as given by Eq. (2).

Further investigations were focussed on the influence of the hitherto constant parameters, which are the droplet velocity and the dielectric constant of the liquid. For a first test, droplets of equal volume but different velocity and dielectric constant were measured with the sensor. Fig. 14 compares three sensor signals generated by the first experiments, where droplets of one volume but different media, thus dielectric constant, and different velocity were dispensed through the sensor capacitor. The generated signals show different peak values and shifts in the time dependent signal characteristics. The difference is obvious for liquids of different dielectric constants (oil and water in Fig. 14). Furthermore, also a change in velocity influences the sensor signal significantly, which is proven by the different signals produced by water droplets of nearly identical volume but different velocity. This indicates that the sensor is not only sensitive to volume, but also to other droplet parameters.

The depicted average exit velocity of the droplets can be estimated from the graph in Fig. 10, taking the spatial dimensions of the sensor into account

$$V_{av} \approx \frac{t_2 - t_1}{s_{sensor}} \text{ [m/s]} \quad (3)$$

where $(t_2 - t_1)$ is the time the droplets need to pass through the sensor and s_{sensor} is the sensors thickness (1.6 mm). Thus, the sensor might also be applied to provide estimates of the droplet velocity.

Finally, to determine the reproducibility of the sensor signal at constant droplet parameters, another series of 100 water droplets was dispensed at a constant velocity and target volume (the CV of the droplet volumes was determined independently by gravimetric measurement to be about 2%). Fig. 15 shows the overlay of 100 single signals, which visualises the high reproducibility of the sensor performance. The standard deviation of the signal peak values is $CV = 3.2\%$ (Fig. 15).

This also includes the fluctuation of the sensor initial output voltage and random variations of the droplet size. Thus, the reproducibility of the sensor signal itself (i.e. given a stabilized voltage and ideally equal size droplets) might be significantly better than 3.2%.

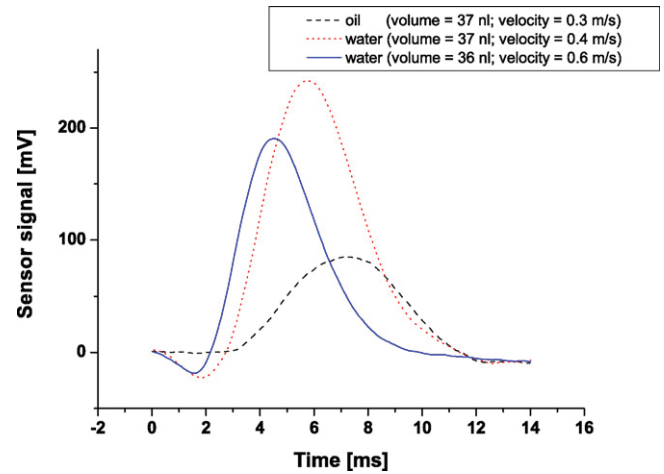


Fig. 14. Influence of the material specific dielectric constant and the droplet velocity to the sensor signal. Three sensor signals, generated by droplets of identical volume but different dielectric constant (water $\epsilon_r \sim 81$; oil $\epsilon_r \sim 2.6$) and velocity.

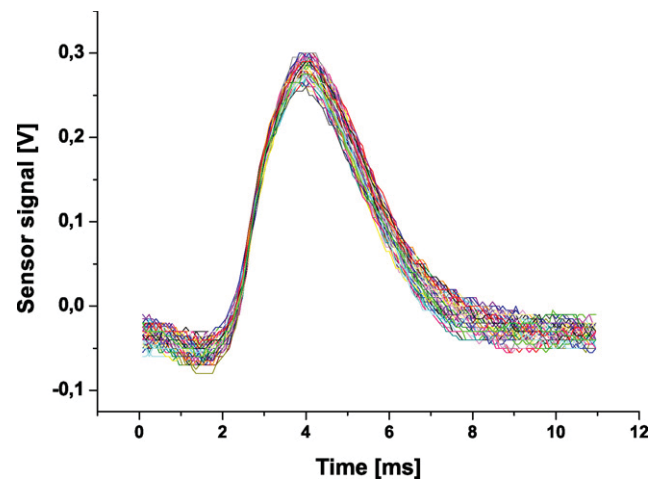


Fig. 15. Correlation of 100 single signals, generated by droplets of pure H₂O at the same dispensing parameters and conditions (correlation coefficient $r_{\text{signal characteristic}} = 0.990$; $CV_{\text{max peak value}} = 3.2\%$).

7. Discussion

It is evident from the presented experiments, that the capacitive droplet sensor delivers an analogue signal which is highly sensitive to the droplet volume and the liquid type (especially the dielectric constant has a strong influence on the signal, see Eq. (2)). In the experiments in particular the dependence of the peak signal value on the droplet volume has been studied, because the signal shape looks very similar for droplet of different size. Taking the peak signal value as quantitative measure, it turns out that there is a very good linear correlation of the peak voltage with the droplet volume with a correlation coefficient of $r_{x,y} = 0.992$ (cf. Fig. 12). The linear regression derived from this experiment is given by the following equation:

$$U_{\text{peak}} = 49.38 \text{ mV} + 5.95(\text{mV/nl})V_{\text{droplet}} \quad (4)$$

Eq. (4) gives the volume calibration of the sensor, if the peak voltage is considered only. Obviously the sensor has a sensitivity of approximately 6 mV/nl. Thus, in principle a resolution of 1 nl should be achievable. However, the average standard deviation of $\Delta U = 7.68 \text{ mV}$ calculated for the water droplets of different size, shows that the reproducibility of the signal (or the experimental conditions) does not allow for resolving the volume with 1 nl

precision. Nevertheless, the applied statistical assessment of the data, shown in Fig. 12, describes that the sensor enables to determine the volume of 95% of all detected droplets with an accuracy of $\Delta V \pm 1.9$ nl. The confidence limits of the calculated prognosis interval are given by Eqs. (5) and (6) in [mV] [19]

$$U_{\max} = -37.04 \text{ mV} + 5.89(\text{mV/nl})V_{\text{droplet}} + 6.82 \times 10^{-4}(\text{mV}^2/\text{nl}^2)V_{\text{droplet}}^2 \quad (5)$$

$$U_{\min} = -61.72 \text{ mV} + 5.99(\text{mV/nl})V_{\text{droplet}} - 6.82 \times 10^{-4}(\text{mV}^2/\text{nl}^2)V_{\text{droplet}}^2 \quad (6)$$

Therefore, the calibrated sensor can be used without further modification to quantitatively measure water droplet volumes in the range from 20 to 60 nl with a resolution of about 2 nl within a confidence interval of 95%.

Regarding the sensitivity of the sensor with respect to other parameters like for example droplet velocity, liquid type, etc. only a brief test has been performed within this experimental study. The results for droplets of identical volume, shown in Fig. 13, confirm that the sensor is also sensitive to other parameters. Even the qualitative signal shape can change with liquid properties. Most likely the dielectric constant (water ($\epsilon_r \sim 81$); oil ($\epsilon_r \sim 2.6$)) is the most important parameter to be considered in this context (see also Eq. (2)). The extent which other parameters do influence the signal remains to be investigated in further studies.

8. Conclusion

The concept of capacitive non-contact droplet detection by an open plate capacitor was successfully demonstrated. It could be shown that the presence or absence of a droplet in a dispensing process can easily be detected. The high reproducibility of the sensor signals can be used to enable a direct control of the stability and reproducibility of droplet volumes in a non-contact dispensing process. Furthermore, quantitative information can be extracted from the sensor signals. A high correlation of the signal peak value to the droplet volume was demonstrated. A calibrated sensor can be used to determine droplet volumes in the range from 20 to 60 nl with a resolution of about 2 nl.

The presented approach to fabricate the sensor capacitor integrated with the readout electronics in standard PCB technology enables its cost efficient realisation. Therefore, this sensor concept can be considered to be well suited for routine and low cost monitoring of non-contact dispensing processes in automated liquid handling systems.

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Biographies

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Peter Koltay studied physics at the Universities of Freiburg (Germany) and Budapest (Hungary) and obtained his PhD from the University of Freiburg in 1999. Afterwards he joined the laboratory of Prof. Zengerle at the Institute for Microsystem Technology (IMTEK) of the University of Freiburg as a faculty member. In 2005 he founded the company BioFluidix GmbH to commercialize non-contact dispensing technologies developed at IMTEK. His research interests are especially related to the development of non-contact liquid-handling technologies, sensors for droplet detection and quality control, modeling of droplet and bubble dynamics, design and fabrication of passive direct methanol fuel cells and simulation of microfluidic devices by numerical methods.