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Capacitive Solvent Sensing with Microfluidics Chip

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Capacitive sensor,
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Coplanar electrode

Abstract: Solvents are widely used in daily life as well as in almost all laboratories. Identification and detection of these solvents used in many areas have great importance in terms of health, toxicology and environment. For this reason, the main purpose of our study is to be able to identify the solvents in practical ways. In this study, gold microelectrodes were used to define the capacitance detection of various solvents in the frequency range of 20 kHz - 2 MHz. Moreover, the electrical noise and signal output of the solvents at different frequencies were investigated. Measurements were made by micro-channels integrated on microelectrodes for reducing noises and detection with high sensibility.

Mikroakışkan Çip ile Kapasitif Çözücü Tespiti

Anahtar Kelimeler
Kapasitif sensör,
Mikro kanal,
Eş düzlemsel elektrot

Özet: Çözücüler, hem günlük hayatta hem de hemen hemen tüm laboratuvarlarda yaygın olarak kullanılmaktadır. Birçok alanda kullanılan bu çözücülerin tespiti ve tanımlanması, sağlık, toksikoloji ve çevre açısından büyük önem taşımaktadır. Bu nedenle, çalışmamızın temel amacı, çözücülerin pratik yollarla tespit edilebilmesidir. Bu çalışmada, altın mikro elektrotlar kullanılarak 20 kHz – 2 MHz frekans aralığındaki çeşitli çözeltilerin kapasitans ölçümü ile tanımlamaları yapılmıştır. Ayrıca, farklı frekanslardaki çözücülerin elektriksel gürültü ve sinyal çıktıları incelenmiştir. Ölçümler, gürültüyü azaltmak ve yüksek hassasiyette algılama yapabilmek için mikro elektrotlar üzerine entegre edilen mikro kanallarla yapılmıştır.

1. Introduction

All materials have different dielectric properties depending on their atomic and molecular structures. These properties show changes with frequency [1]. This enables the characterization of materials depending their electrical properties. Dielectric spectroscopy is rising as a new branch of science exploiting the dielectric properties of objects to detect them.

Solvents are everywhere; we drink them, burn them, use them for cleaning or putting them in our pills etc. Therefore, they are very important in our life and it is very important to detect and identify solvents sensitively. There are a lot of capacitive sensing devices [2-4] some of which have also been commercially successful. In this study, we tried to detect solvents exploiting different dielectric behaviors under applied AC electric signal. We scanned a wide frequency range investigating the noise stability of different frequencies. Moreover, we investigated different methods of sending the solvent on the sensing area. One of the them is to pour a droplet on to the sensor with a micro pipette; the other one is dipping the sensor into 50 ml of solvent in a petri dish; and the last one is sending the solvent to the sensor via a microchannel.

The best performance was achieved with microchannels. Thanks to developing rapid prototyping methods, microchannel fabrication was easier than before which make applications of microfluidics more common. Combination of microfluidics with several environmental elements enables high sensitive sensor applications. Integration of fiber optics with micro channels enabled the world's easiest optical flow cytometry when used with viscoelastic fluids [5]. Making different compartments for mixing and sensing is also possible in microfluidics when micro valves and pumps are integrated to the chip. In one of the recent research studies, detection of TNT from water was

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done utilizing the UV fluorescent silica nano particles in a micro total analyzing system [6].

2. Material and Method

2.1. Electrode fabrication

There are lots of electrode types used in dielectric spectroscopy that is also called as electrochemical impedance spectroscopy (EIS). From carbon rod electrodes to liquid mercury electrodes, there are a lot of alternatives [7]. Micro electrode is one of them with rising popularity in micro electrodes.

Recently, micro electrodes have been widely used with increasing number of available micro fabrication facilities. There are some advantages of micro electrodes in comparison to the other electrodes [8]. One of the advantages is their small size decreasing the contact area so the parasitic effects causing noise. Hence, the effect of electric double layer decrease which has low value in micro electrodes because of decreasing electrode surface area. Electric double layer cut the electric field lines disabling signal to get through the sample. The other advantage is the low sample requirement due to small size. In this study, we used 2 finger coplanar electrodes [9]. However, there is also a multiple finger device design which is called interdigitated micro electrodes having higher capacitance because of bigger area of electrodes. These devices also have lots of applications in combination with other techniques [10].

All the fabrication processes were realized in class 100 and 1000 clean room facilities at National Nanotechnology Research Center (UNAM). We used the lift off method for the patterning of electrodes on the glass slide. A schematic presentation of fabrication process was introduced in Figure 1. Firstly, conventional cleaning protocol was used for removing organic contaminants from the glass surface. The glass slide surface was cleaned with acetone and IPA in a sonicator bath for 5 minutes. Between sonication steps, the substrate was washed with DI water and dried under nitrogen flow. After wet cleaning, it was put on 110 °C hot plate for 10 minutes for the evaporation of any wet remnant on the surface. After cleaning, photo pattering process was started. First, HMDS was spin coated on the surface at 4000 rpm for 50 s for the better adhesion of photo resist on to the surface. Then, photo resist was spin coated at 5000 rpm for 40 s. Finally, photo resist coated glass slide was baked at 110 °C hot plate for 50 s.

We designed our photo mask in CAD software and printed out the mask at 3600 dpi printer which corresponds one dot per 7.5 µm that is good enough resolution for our 50 µm electrodes. The printed mask was cut out and taped on a soda lime glass surface where printed area of the mask film would be in soft contact with substrate. We used EVG620 mask aligner for UV exposure, so the soda lime glass that the mask was taped on is the blank 5 inches mask glass that is used with EVG 620 mask aligner. The photo resist coated glass surface was exposed with 40 mJ UV in soft contact mode and developed using AZ400K developer for 40 s. After patterning of photo resist on the surface, we passed to the metallization process. VAKSIS thermal evaporator system was used for the deposition of the Chromium and Gold. Gold was used as the electrode material because of its inertness as usual. In this study, we used the Chromium as an adhesion layer for gold since; the gold layer is peeled off easily without chromium. So, at 6 x 10^{-6} torr vacuum 20 nm of Chromium and 50 nm of the Gold layer were deposited on the glass surface. Then, the substrate was dipped into acetone for 3 h and sonicated for 1 min for totally lifting off the remaining photo resist. Therefore, we fabricated 40 µm width and gap gold micro electrodes. Representation of electrode fabrication process is shown in Figure 1.

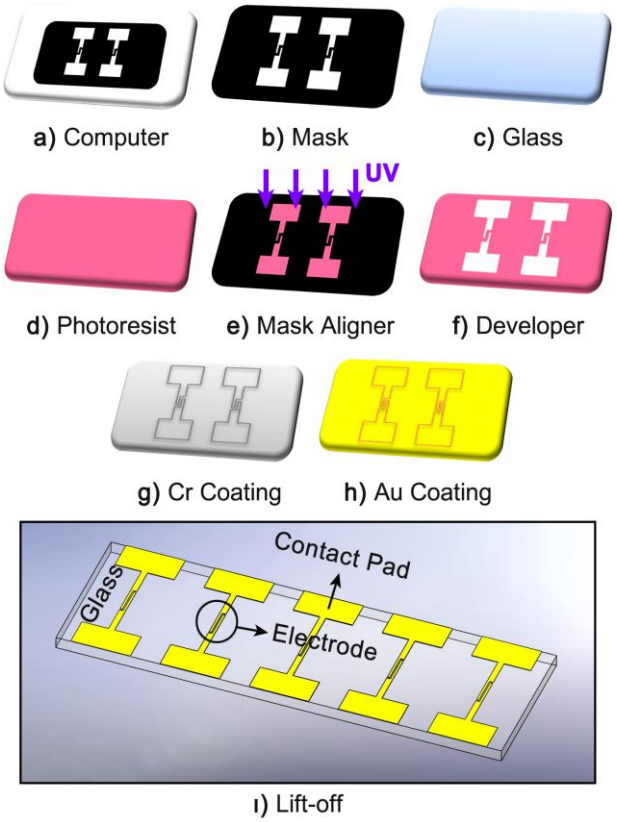

Figure 1. From designing of mask to patterning of photo resist on glass slide, a complete fabrication process

Figure 1 from designing of mask to final patterned metalized of glass slide, a complete fabrication process a. The mask is designed in computer b. The mask is printed out with high resolution printer c. cleaned plain glass slide d. spin coated photo resist on glass slide e. UV light exposed on PR thorough the mask with mask aligner f. UV exposed area on PR surface is peeled off in developer solution g. whole slide is coated with chromium in thermal evaporator h. gold is coated on chromium in thermal evaporator i. slide is dipped into acetone which etches PR and

everything coated above the PR peel off leaving the metalized area only on plain glass surface where PR doesn't remain.

2.2. Micro channel fabrication

Micro channels were fabricated from PDMS (Sylgard 184 Dow Corning). We fabricated the microchannel using published technique of the researchers of this study [11]. First, an acetate sheath attached on a glass slide with double sided tape and PDMS was spin coated on the acetate sheet at 1000 rpm. PDMS was mixed at 10/1 ratio with curing agent and degassed to remove bubbles. After spin coating the acetate sheet with PDMS, it was cured at 100 °C for 1 hour on a hot plate. Then acetate sheet was peeled from the glass slide and put on another glass slide reversely. Hence, spin coated PDMS layer sandwiched between acetate and glass slide. We designed channel geometry in CAD 2007 software and cut the PDMS layer using Epilog Zing laser engraver. Glass-PDMS-acetate block was put inside the laser cutter and laser beam ablate the acetate and PDMS at desired geometry as drawn in CAD. Then Laser cut PDMS layer was transferred to a PDMS block finishing the channel fabrication process. Here, the second PDMS block was prepared with the same method as mentioned above.

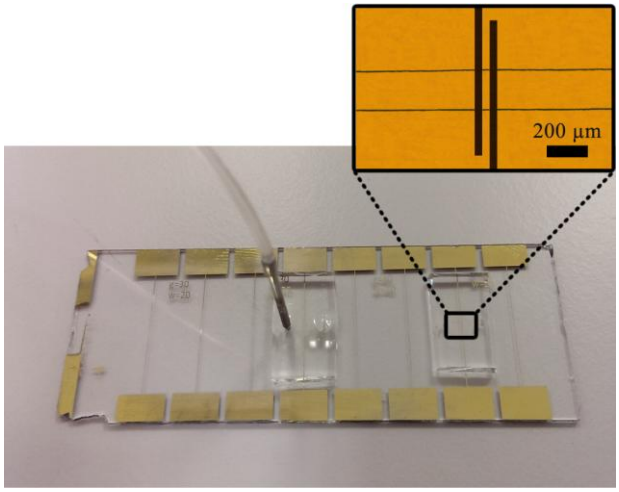

Figure 2. Micro channels bound to micro electrodes on glass slide

First PDMS was prepared mixing with its reagent at 10/1 ratio and stirred. After degassing process in a desiccator, it was poured on a glass petri dish and left for hardening at 100 °C for 3 h. Hardened PDMS was pulled up and cut with a razor blade and laser cut PDMS layer was plasma bonded on the PDMS block. Because chemical bond between plasma bonded PDMS layer and PDMS block is stronger than physical bond between PDMS layer and acetate sheet, the acetate sheet is easily peeled off leaving the PDMS layer bond to PDMS block forming the micro channel structure. Finally, slide carrying micro electrodes and micro channel are bonded using plasma activation again and microfluidics chip fabrication ended. Whole photo of the chip with micro structure is shown in Figure 2.

2.3. Measurement and setup

After fabrication of the device, measurement was done with a technique that was previously used in a published article by the current researchers [12]. The electrical measurement was done with Agilent e4980a LCR meter. We wrote a LabVIEW code for controlling the instrument and data acquisition by connecting the LCR meter to the computer via USB 2.0 port. Electrical connection between LCR meter and electrodes was done with RG 79 coaxial cable. There is a representation of the experimental setup in Figure 3.

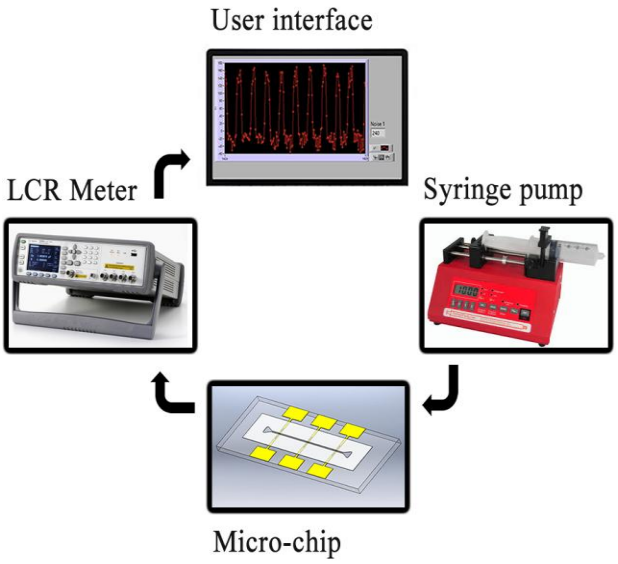

Figure 3. Experimental setup for solvent sensing using a microfluidic device

Three different techniques were tried for the electrical measurement. First, we put a droplet on top of the electrodes and found that capacitance value was increasing continuously. Hence we decided not to use this technique as it was not stable. In the second method, we dipped the microfluidics device into the 50 ml of solvent poured in a glass petri dish and realized that the results were better than those with the first method. The capacitance value was not increasing or decreasing continuously, but fluctuating in a reasonable band. In the last method, we sent the solvent through the micro channel and we had the best results in this way.

Here, we used the parallel capacitance and resistance as the equivalent circuit model because there is a small electrical double layer on the electrodes and solutions are highly insulative [13]. Electrical double layer formation and conductivity are induced by the ion in the solution. However, in this study, we used very pure solvents which means there are extremely low contaminants and so the ions.

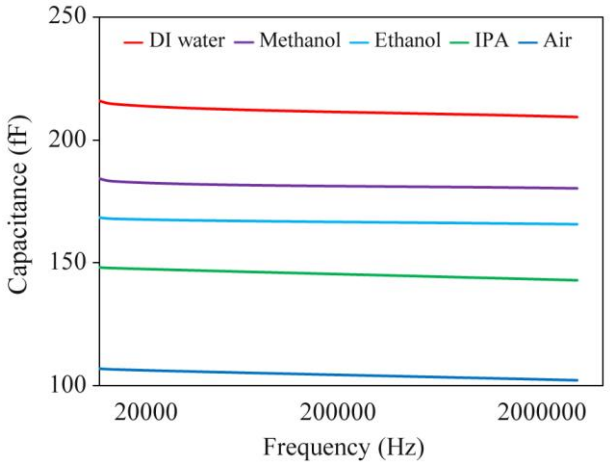

Figure 4. Wide frequency characteristics of different solvents

The solvents were sent through the channel with a syringe pump at constant flow rate of 1 µl/min. LCR meter was run with LabVIEW code which makes frequency sweep at a wide range. Capacitance results were recorded to an excel file for later evaluation. Figure 4 presents are the frequency sweep results of parallel capacitance (Cp) values of dry, DI water, IPA, methanol and ethanol.

According to known dielectric constants of the solvents given in the Table 1, Cp results are at the right where they were supposed to be. As a well-known fact from Fundamental Physics, there are two parameters affecting the total capacitance namely geometry of the device and dielectric constant of the material between electrodes. Here only dielectric material changes because there is no change at the system like extending or shrinking. Hence, variance at the sensor reading comes only from the material.

Table 1. Relative dielectric constants [14]

DI Water	80.1
IPA	17.9
Methanol	32.7
Ethanol	24.5

At low frequency, physical background of the experiment is more complex than high frequencies. Because there are different effects like electrical double layer and non-faradaic currents which are very dominant at low frequencies. Capacitance change from the dry value of the sensor with different solvents is shown in Figure 5. It is seen that our sensor is able to distinguish even IPA and Ethanol which have relatively close dielectric constants.

3. Conclusion

We were able to fabricate solvent sensor using micro fabrication techniques. Sensors with low frequency performance were not reliable due to complexities in low frequencies. There may also occur some faradaic currents that we don't want in such a capacitive detection situation. Moreover, our parallel capacitor and resistor model can't be enough in such a complex situation so there has to be addition of some other elements in series and parallel to meet complex physical requirements. But high frequency response of the system is promising. It is very suitable to use such a system at solvent sensing application because it gives clear results after 10 kHz. Here we used a bench top heavy LCR meter and the measurement instrument; if it can be reduced in weight and volume, it would have possible commercial application because of its mobility. Today, fake drink production is especially a big problem causing several deaths. Here, this method may be a good candidate for portable and easy detection of those drinks. Because, the system made a clear distinguishing between different types of alcohols like ethanol and methanol.

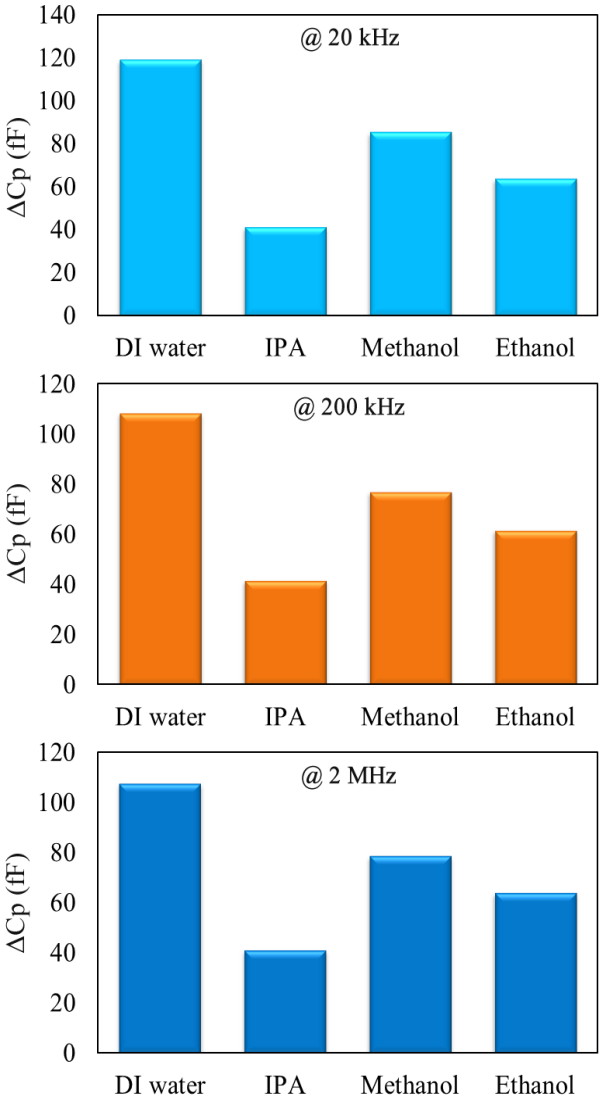

Figure 5. Capacitance change of different solvents

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