Electrical recognition of solvent liquids and particles in suspension with needle-plane electrodes

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Abstract. We report a measurement methodology to identify solvent liquids and the presence of suspended nanoparticles with needle-plane capacitive electrodes. Preliminary results were obtained with a low noise measurement system detecting differential electric signals of two needle-plane devices, sensing and compensating respectively. A solvent drop of 3 µl volume is placed between electrodes of the sensing device where electric field perturbations are due to the liquid evaporation process and registered as temporal electric signals. Low noise differential measurement system reduces parasitic capacitances present in the sensing device to a minimum value in order to obtain just the temporal capacitance of the liquid evaporation process, presenting a base noise around 0.15 [fF /√Hz]. The results show that different liquid samples can be recognized electrically with just 3 µl volume drop, analysing its temporal signal shape, amplitude and evaporation time.

1. Introduction

The impedance concept associated in a specific way to capacitance have had a great evolution and relevance in diverse scientific disciplines and industry as electrochemistry, biosciences, physical-chemical processes, electronics, sensors as well as applied and experimental systems and others [1,2]. Electric field measurement techniques are suitable to realize electrical characterization of diverse materials and physical-chemical processes in a non-destructive way. Electrical measurements in nanoscale materials are limited by noise, commonly impeding to obtain a measurable signal of interest. However, there are low noise measurement techniques that can be applied to increase the signal to noise ratio to a suitable level to be detected [3,4]. Also, the theoretical study and design of diverse capacitance electrode geometries is important for improving the performance of a sensor. Thus, a better quantification or measurement of a physical variable of interest can be obtained [5,6].

The proposed methodology to characterize liquids electrically is based on two needle-plane devices where a glass substrate of 150 µm thickness is over each plain electrode. A liquid sample of 3 µl is placed between the conductive needle electrode and the glass substrate of the sensing device. An AC voltage of 1 V_{RMS} with 10 KHz frequency is applied to the liquid sample. The temporal electric signal during the liquid evaporation process is monitored until it is totally evaporated. The resulting signal passes through an electronic conditioning stage and a differential electric signal goes into a Lock-in...
Amplifier (LIA) where the imaginary part of the current $\text{Im}\{I_{\text{LIA}}(t)\}$ is monitored. This temporal signal represents the electric field perturbations due to the sample evaporation process present between the needle-plane electrodes. Measurements with different liquid samples were performed in order to obtain temporal electric signals for each one. Preliminary results showed that it is possible to distinguish a particular electric response for solvent liquids with and without metallic particles.

2. Low Noise Differential Measuring System

Low Noise Differential Measuring System (LNDMS) is proposed to perform electrical characterization of liquid drop samples by means of two needle-plain devices used as compensation and sensing respectively. Both devices were supplied by a sine reference signal with frequency $f = 10$ KHz and amplitude of $1 \text{ V}_{\text{RMS}}$, both constants. Reference signal $V_{AC}(\omega)$, where $\omega = 2\pi f$, was compensated in phase and amplitude before entering to the compensation device, for the sensing device the signal enters directly to it, acting as an excitation signal. Differential signal from compensation and sensing devices was processed by the LIA in order to obtain data of the current imaginary component $\text{Im}\{I_{\text{LIA}}(t)\}$, as it is shown in the block diagram of figure 1.

The principal output configuration parameters of the LIA were set with a low pass filter wideband of $\text{WB} = 1.2$ Hz, 12 dB roll-off and time constant $t_c = 100$ ms. With these parameters the noise surrounding the reference signal frequency is reduced to a minimum value.

Needle-plain devices were placed in a parallel way, where needle electrodes are fixed in a mechanical support maintaining 15 mm horizontal separation from each other. They keep a perpendicular position to the plane electrodes which are mounted over a xyz stage in order to adjust a separation between them and the needle vertex. Needle electrodes have 5 mm height and their vertex curvature is around 100 µm. Each plain electrode is deposited over a PCB with 1 cm² surface and they are separated 5 mm horizontally from each other, as it is shown in figure 2(a).

![Figure 2. Dual Needle-Plane Electrodes. a) dimensions and b) sensing device with liquid sample.](image-url)
3. Experimental measurements

LIA’s sine reference signal $V_{AC}(\omega)$ is used as an excitation signal applied to sensing needle to plain electrodes so an electric field is generated between them. A glass substrate with 150 µm thickness is placed over each plain electrode to isolate them electrically leaving around 150 µm gap between the needle vertex and the upper glass surface. The input signal of the compensation device is equalized in phase and amplitude $G V_{RMS} \sin(\omega t \pm \phi)$, where $G$ is the gain amplification, with respect to the input signal of the sensing device, in order to get two similar signals at the differential stage input. Thus, the resultant differential signal is $\Delta V(t) = A V_{RMS} [\sin(\omega t) - G \sin(\omega t \pm \phi)]$, where $A$ is the gain of the differential stage. $\Delta V(t)$ enters to the LIA’s input where the DC imaginary component of the current $\text{Im}\{\Delta I_{LIA}(t)\}$ is obtained at its output, with the configuration parameters mentioned above. When $\text{Im}\{\Delta I_{LIA}(t)\}$ is set to a minimum value the temporal base signal has minimum offset and noise as well. In this case the signal noise was around $10\text{pA}/\sqrt{\text{Hz}}$, with no liquid sample. The base signal was set before make any measurement with a liquid sample, this way the maximum dynamic range of the LIA can be used. Experimental measurements were realized with different solvent samples. A graduated micropipette was used to place drops of around 3 µl volume for each sample. When a solvent liquid sample is placed between sensing needle electrode and glass substrate, the electric field perturbations are due to the evaporation process of the solvent and they represent temporal impedance changes. The perturbations are sensed as temporal current signals $\text{Im}\{\Delta I_{LIA}(t)\}$, lasting until the solvent sample is totally evaporated. Differential capacitance data as a function of the current data are obtained with an admittance analysis of the equivalent electric circuit of just the liquid sample. This equivalent circuit is represented as a resistance-capacitance (RC) parallel circuit from which the expression for the differential capacitance is obtained as $\Delta C(t) = \frac{\text{Im}\{\Delta I_{LIA}(t)\}}{\omega V_{RMS}}$. With this expression $\Delta C(t)$ data are calculated from those registered by the LIA as $\text{Im}\{\Delta I_{LIA}(t)\}$ of the liquid sample impedance during its evaporation process. Differential measurements allow diminish parasitic capacitances of the electrodes, conductors and connectors to a minimum value which represent a base signal offset, with no liquid sample in the sensing device. Therefore, mostly the capacitance contribution of the liquid sample can be sensed when it is placed in the sensing device as it is shown in figure 2(b).

4. Results

Measurements where performed with three different solvent liquid samples, isopropanol, ethanol, acetone and a nanoparticle suspension solvent-based. The suspension was prepared with 25 ml isopropanol volume and 52.6 mg of 500 nm copper particles, mixed in a glass container. Sample drops of 3 µl volume were used in each measurement. Graphs of $\Delta C(t)$ data were obtained for each liquid sample in a determined lapse time. Each solvent liquid presents a particular behavior of $\Delta C(t)$ due to their evaporation process, as it is shown in figure 3.
Figure 3. Graphs of $\Delta C(t)$ for Ethanol, Isopropanol and Acetone. Each one represents the evaporation process for three drops. Each graph represents the evaporation process of three drops of the same liquid solvent until each one is evaporated. It can be observed the same curve tendency, although the amplitude and the evaporation time are not the same.

Nanoparticle suspension isopropanol-based graph was compared with that of pure isopropanol of figure 3, in order to detect electrically the contribution of copper nanoparticles in the solvent evaporation process of three drops for each one, as it is shown in figure 4.

Figure 4. Graphs of $\Delta C(t)$ for Isopropanol and nanoparticle suspension solvent based.

It can be inferred that metallic nanoparticles makes the difference in their evaporation process and therefore in their curve tendency and amplitude as well. Evaporation process depends on several environmental factors as relative humidity RH, temperature T, atmospheric pressure P, flowing air, etc. Experimental measurements were performed with no controlled lab ambient conditions and liquid samples were placed manually, this could explain the differences in amplitude and evaporation time for each solvent graph. In this case the atmospheric conditions present in the measurements were with RH = 60%, P = 772 mbar and T = 23°C.
5. Conclusions

Experimental results showed that it is possible to identify solvent liquids sensing the temporal electrical signal of their evaporation process, up to 3 µl volume drops, with a dual needle-plane sensing device realizing differential measurements with a LNDMS. Evaporation process of the liquid sample, placed between a needle and an isolated plane electrodes, provokes temporal electric field perturbations that are registered as ΔC(t) data graphs. The dielectric function of the liquid solvent changes as the evaporation process takes place until all the particles are dissipated in the ambient. When the liquid is totally evaporated, the electric signal returns to its base line. Measurements presented a base noise N_{WB} around 0.15 [fF/√Hz] and a maximum signal to noise ratio S/N around 38 dB with nanoparticle suspension.

Graph analysis indicates that each liquid sample presents a particular shape of its ΔC(t) curve. This electric behavior is also presented with 500 nm particle suspension isopropanol-based, indicating that is possible to detect the presence of particles in suspension despite amplitude and evaporation time are different for each sample. Thus, the results obtained indicate that it is possible to identify electrically different liquids and whether there are metallic particles in suspension or not with just a drop of the liquid of interest with the proposed sensing device. It should be of interest to improve the sensing device in order to reduce base noise and detect fewer and smaller particles in suspension. Performing more experimental measurements with different liquid and suspension samples is needed to validate the proposed method with other types of liquids and suspensions. The measurement methodology proposed could be investigated as a possible way to identify pollutants in liquids electrically.

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