Split Ring Resonator-Based Microwave Fluidic Sensors for Electrolyte Concentration Measurements

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Split Ring Resonator (SRR) based Microwave Fluidic Sensors for Electrolyte Concentration Measurements

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Abstract— **A differential microwave sensor, based on a pair of uncoupled microstrip lines each one loaded with a split ring resonator (SRR), is applied to the measurement of electrolyte concentration in deionized (DI) water. For that purpose, fluidic channels are added on top of the SRR gaps, the most sensitive parts of the structure. The operating principle is based on the measurement of the cross-mode insertion loss, highly sensitive to small asymmetries caused by differences between the reference liquid and the liquid under test (LUT). In this work, the reference liquid is pure DI water (the solvent), whereas the solution, DI water with electrolyte content, is injected to the LUT channel. The proposed sensor is able to detect electrolyte concentrations as small as 0.25 g/L, with maximum sensitivity of 0.033 (g/L)⁻¹. The sensor is validated by measuring the concentration of three types of electrolytes, i.e., NaCl, KCl and CaCl₂. Finally, the sensor is applied to monitor variations of total electrolyte concentration in urine samples.**

Index Terms— **Microwave sensors, differential sensors, microfluidics, permittivity measurements, microstrip technology, split ring resonator (SRR), electrolyte concentration.**

I. INTRODUCTION

Electrolytes are chemical substances that dissociate into cations and anions when dissolved in polar solvents, such as water, thereby modifying the electric and electromagnetic properties of the solvent. Particularly, changes in the conductivity and loss factor (or loss tangent) of the solvent are expected by the presence of electrolytes in it. Therefore, electrolyte concentration can be measured by means of methods or approaches sensitive to the conductivity or loss factor of the solution, and particularly through microwave techniques.

Electrolytes are physiologically important since they play key roles in various vital functions, such as blood pH and pressure control, body hydration, nerve and muscle functions, etc. [1]. The most important primary ions of electrolytes in blood and urine are sodium (Na⁺), potassium (K⁺), calcium (Ca²⁺), chloride (Cl⁻) and bicarbonate (HCO₃⁻). Imbalances in the concentration of certain ions in blood or urine (the so-called anion gap [2]) are indicative of certain pathologies. For instance, in urine the anion gap is defined as [Na⁺] + [K⁺] − [Cl⁻], where [*] denotes concentration, and typical values in urine samples not subjected to physiological pathologies should be comprised between 0 and −33 mEq/L. Ion concentration in blood and urine are typically measured by means of electrochemical systems, such as ion selective electrodes (ISE) [3], able to provide the individual concentration of specific ions, and consequently the anion gap. Measuring the total concentration of ions does not determine the anion gap, but may be indicative of an overall excess or defect of electrolytes, as compared to the typical levels found in "healthy" samples. Therefore determining the total electrolyte concentration in serum, plasma or urine through real-time low-cost systems (as compared to chemical systems) may be useful as a complementary diagnosis method.

Within this context, this paper is focused on the measurement of electrolyte concentration in DI water using microwaves. The sensor is based on the variations of the complex dielectric constant (mostly related to variations in the conductivity or loss tangent) of DI water experienced by the presence of electrolytes dissolved in it. Such variations can be sensitively detected by means of a differential technique based on the measurement of the cross-mode insertion loss in a pair of microstrip lines loaded with split ring resonators (SRRs), and equipped with fluidic channels for liquid injection. The main advantage of this resonator-based approach is the high sensitivity and capability to detect small concentrations of electrolytes in water.

Many sensing structures based on resonator-loaded lines have been proposed, and they can be categorized in three main groups: (i) frequency variation sensors [4]-[13], (ii) coupling modulation sensors [14]-[22] and (iii) frequency-splitting sensors [23]-[30]. In frequency variation sensors, the output variable is the frequency position of either a notch or a peak in the frequency response of a transmission line loaded with a resonant element (sometimes, the notch depth, or the peak magnitude, is also used as an additional output variable). Sensors based on this approach have been applied to different types of measurements, but, particularly, they have been used for dielectric characterization of solid [8]-[10] and liquid [6],[12],[13] samples. Symmetry disruption is the physical

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principle in coupling modulation sensors [31],[32]. In such sensors, a transmission line is symmetrically loaded with a symmetric resonator uncoupled to it. Such uncoupling is achieved by using resonant elements exhibiting a symmetry plane of different electromagnetic sort than the symmetry plane of the line [31], [32]. However, by truncating symmetry (e.g., by means of a relative linear or angular displacement between the resonator and the line, or by means of an asymmetric dielectric loading), line-to-resonator coupling is activated, thereby generating a notch in the frequency response (with depth intimately related to the level of asymmetry). In such coupling modulation sensors the output variable is thus the notch depth, and they have been mostly applied to the measurement of spatial variables [14]-[22],[33]. Finally, in frequency splitting sensors, a transmission line is symmetrically loaded with a pair of (not necessarily symmetric) resonators coupled to it. Under perfect symmetry, a single notch at the fundamental resonance frequency of the resonant elements appears. However, if the resonators are asymmetrically unloaded, frequency splitting arises. In these sensors, similar to differential sensors, the output variables are the differential frequency and the differential depth of the generated notches, and these frequency-splitting sensors have been mainly applied to differential permittivity measurements [29],[30].

The previous sensors exhibit, in general, acceptable sensitivities and dynamic ranges, but they exhibit limited resolution, not being able to detect small variations of the variable of interest. This aspect is critical for the measurement of solute concentration in highly diluted solutions. Therefore, for the accurate measurement of electrolyte content in DI water solutions exhibiting concentrations in the range 1-10 g/L or even below, as pursued in this paper, novel strategies, highly sensitive to small concentrations, are needed. The above-mentioned sensing approach, based on the measurement of the cross-mode insertion loss in a pair of lines, each one loaded with a resonant element, provides the necessary sensitivity and resolution for the intended application. This approach was first demonstrated in [34],[35] by considering open complementary split ring resonators (OCSRRs) [36] as loading resonators (NaCl was the considered electrolyte in [35]).

In this paper, it is demonstrated that sensitivity can be improved by replacing OCSRRs with SRRs. Moreover, sensor validation is carried out by considering three electrolytes, i.e., NaCl, KCl and $CaCl_2$. An advantage of using SRRs instead of OCSRRs as sensing element is the fact that SRRs do not require vias, hence easing fabrication and contributing to enhance resolution, intimately related to the "level" of symmetry achieved when the differential structure is loaded with identical loads in both resonators (approaching to "perfect symmetry" is more feasible if vias are not present). In the last part of the paper, it is demonstrated that the proposed sensor may be useful to detect variations of total ion concentration in biological samples, particularly urine. This may be of interest for real-time monitoring of potential dysfunctions or anomalies, e.g., during disease treatment in hospital environments, related to extreme ion concentrations in such samples.

II. STRUCTURE OF THE PROPOSED SENSOR, SENSING PRINCIPLE AND FABRICATION

The proposed differential sensor consists of two parts: (i) the microwave structure, a *FR4* substrate with a pair of uncoupled 50 Ω lines each one loaded with a SRR, and (ii) the fluidic channels plus the mechanical accessories for liquid injection to the channels and for channel attachment to the substrate. The topology of the microwave structure is depicted in Fig. 1, where relevant dimensions are indicated. The dielectric constant and thickness of the considered substrate are $\varepsilon_r = 4.4$ and $h = 1.6$ mm, respectively. The fluidic channels are made of polydimethylsiloxane (PDMS), and are identical to those used in [30]. Channel dimensions are designated by h_{ch} (height), w_{ch} (width) and l_{ch} (length). The mechanical parts consist of a polyether ether ketone (PEEK) structure, that accommodates the fluidic connectors (necessary for liquid injection to the channels), and screws, used to fix the PEEK-PDMS assembly on the substrate. In order to avoid substrate absorption, a dry film of clear polyester, with an estimated thickness of 50 μm and dielectric constant of 3.5, has been deposited on top of the SRRs. The top and lateral views of the channel plus mechanical parts are depicted in Fig. 2. The photograph of the whole sensing structure is shown in Fig. 3.
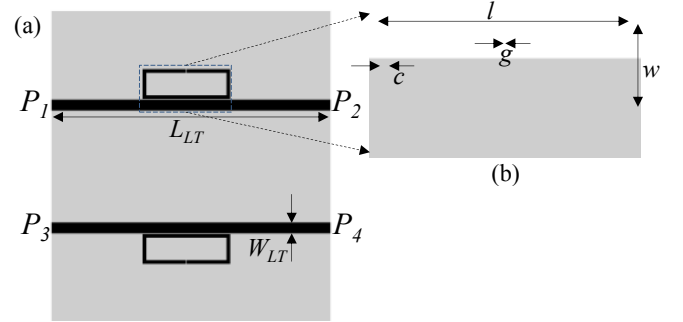

Fig. 1. Topology of the microwave structure of the proposed sensor (a) and zoom of resonator (b). Dimensions are (in mm): $L_{LT} = 76.5$, $W_{LT} = 2.79$, $l = 24$, $c = 1$, $w = 8.17$, $g = 0.2$. The ground plane is depicted in grey.
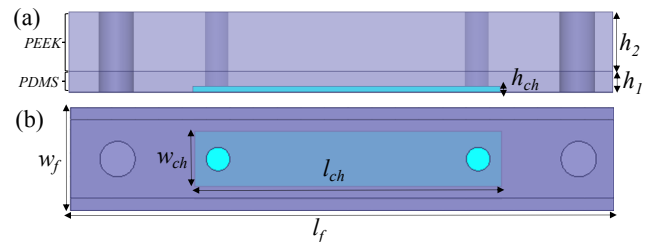

Fig. 2. Lateral (a) and top (b) views of mechanical and fluidic parts of the microwave sensor and relevant dimensions. Channel dimensions are: $h_{ch} = 1.5$ mm, $l_{ch} = 26$ mm, $w_{ch} = 4.6$ mm; other relevant dimensions are: $l_f = 46$ mm, $w_f = 12.6$ mm, $h_1 = 3$ mm, and $h_2 = 9$ mm.
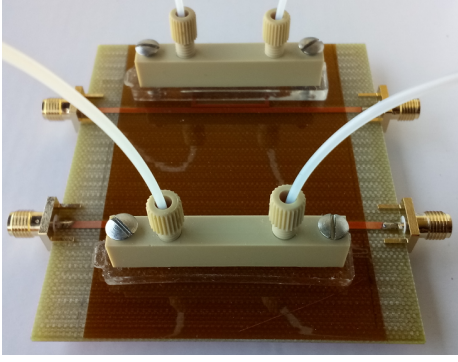
Fig. 3. Photograph of the proposed sensor including the microwave structure, the fluidic channels (plus mechanical accessories) and connectors.

The working principle of the proposed differential sensor is symmetry disruption, caused by an asymmetric dielectric load in the SRRs. Thus, in a sensing operation, the reference liquid is injected in one of the channels (designated as REF channel), whereas the liquid under test (LUT) is injected in the other channel (LUT channel). The output variable in the proposed sensor is the cross-mode insertion loss (or cross-mode transmission coefficient), highly sensitive to small asymmetries and, hence, very convenient to detect small differences between the REF and the LUT liquids. This is necessary in order to measure small concentrations of electrolytes in DI water (the purpose of this work). Thus, the considered REF liquid will be pure DI water (the solvent), whereas the LUT is the DI water solution with electrolyte content.

According to the port definition of Fig. 1, the cross-mode transmission coefficient is given by [37]

$$S_{21}^{DC} = \frac{1}{2}(S_{21} - S_{43}) \qquad (1)$$

This result is valid as long as the SRR-loaded lines are uncoupled. If the structure exhibits perfect symmetry, it follows that $S_{21}^{DC} = 0$ (note that the cross-mode insertion loss, i.e., the cross-mode transmission coefficient expressed in dB, should be ideally $-\infty$ under perfect symmetry). However, if symmetry is disrupted, as occurs by injecting pure DI water in the REF channel and the electrolyte solution in the LUT channel, the cross-mode insertion loss should take finite values, and the maximum value should be indicative of the level of asymmetry. Therefore, the maximum level of the cross-mode insertion loss is considered the output variable in the proposed sensing structure.

In practice, perfect symmetry is not achievable, as long as sensor fabrication involves several ("imperfect") processes, including substrate etching, dry film deposition, and mechanical fabrication of the PDMS-PEEK assembly. Thus, certain level of cross-mode insertion loss is expected even by loading the REF and LUT channels with identical liquids. Let us designate the maximum of the modulus of the cross-mode transmission coefficient experimentally found with balanced loads as $|S_{21}^{DC}{}_{BAL}|$. It is important to minimize $|S_{21}^{DC}{}_{BAL}|$ as much as possible, since it has direct impact on sensor resolution. Note that tiny differences between the REF liquid

and the LUT liquid, which should provide a very small value of $|S_{21}^{DC}|$, may be obscured by $|S_{21}^{DC}{}_{BAL}|$. Therefore, an accurate and rigorous sensor fabrication process, providing small $|S_{21}^{DC}{}_{BAL}|$, is demanded for resolution optimization.

III. EXPERIMENTAL VALIDATION

The fabricated sensor has been validated by considering DI water solutions with different types of electrolytes, particularly, NaCl, KCl and $CaCl_2$. These electrolytes have been considered since they are present in blood and urine and they play key roles in several vital functions.

The first set of experiments has been done by injecting DI water solutions with different concentrations of NaCl in the LUT channel. In all the cases, pure DI water has been injected in the REF channel. The measured cross-mode insertion loss for the different NaCl concentrations is depicted in Fig. 4. Note that the cross-mode insertion loss resulting when the LUT channel is filled with pure DI water (balanced loading) is also included. $S_{21}^{DC}{}_{BAL}$, expressed in dB, is found to be -38.95 dB, which is a very reasonable value. NaCl concentrations as small as 0.25 g/L can be perfectly resolved by virtue of the small value of $S_{21}^{DC}{}_{BAL}$. By increasing the electrolyte concentration, the cross-mode transmission coefficient (or insertion loss) also increases, as expected. In Fig. 5, we have depicted the dependence of the maximum value of the cross-mode transmission coefficient, $|S_{21}^{DC}|_{max}$, with the concentration of NaCl, i.e., [NaCl]. For small concentrations, the sensitivity, i.e., the variation of $|S_{21}^{DC}|_{max}$ with [NaCl], is very high, and it progressively decreases as [NaCl] increases. The maximum value of the sensitivity is found to be 0.033 $(g/L)^{-1}$, i.e., higher than the maximum sensitivity of the sensors proposed in [35], based on a similar approach, but using OCSRRs as sensing elements (in [35] the sensors were applied to the measurement of NaCl concentration, as well). From the results of Fig. 5, we have obtained the calibration curve, from which the concentration of NaCl can be inferred by measuring $|S_{21}^{DC}|_{max}$. Such curve, with a correlation coefficient of $R^2 = 0.99966$, is

$$[NaCl]g/L = -0.43 + 0.45e^{\left(\frac{S_{21}^{DC}}{0.03649}\right)} + 2 \cdot 10^{-5}e^{\left(\frac{S_{21}^{DC}}{0.0091}\right)} \quad (2)$$
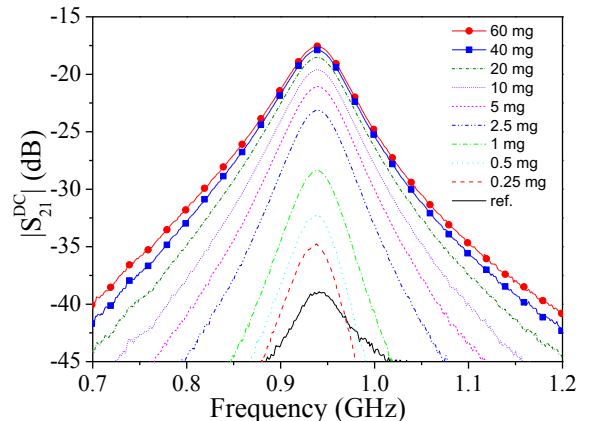

Fig. 4. Cross-mode insertion loss for different values of NaCl concentration.
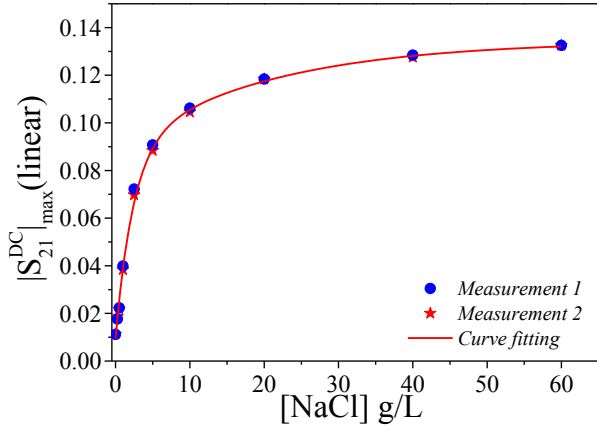
Fig. 5. Variation of $|S_{21}^{DC}|_{max}$ with [NaCl].

Repetitiveness of the results is important for the practical use of the proposed sensors. Therefore, we have repeated the previous measurements by subsequently injecting in the LUT channel the DI water solutions with the NaCl concentrations indicated in Fig. 5. The results, also included in Fig. 5 and identified as *Measurement 2*, reveal that measurements are repetitive by virtue of the small differences with the first experimental campaign (*Measurement 1*).

In the second set of experiments, KCl has been considered as solute. The variation of the cross-mode insertion loss and $|S_{21}^{DC}|_{max}$ with KCl concentration are depicted in Figs. 6 and 7, respectively. Repetitiveness of the results is also demonstrated (see Fig. 7). In this case, the calibration curve, with $R^2 = 0.99931$, has been found to be

$$[KCl]g/L = -0.11 + 0.14e^{\left(\frac{s_{21}^{DC}}{0.02347}\right)} \quad (3)$$
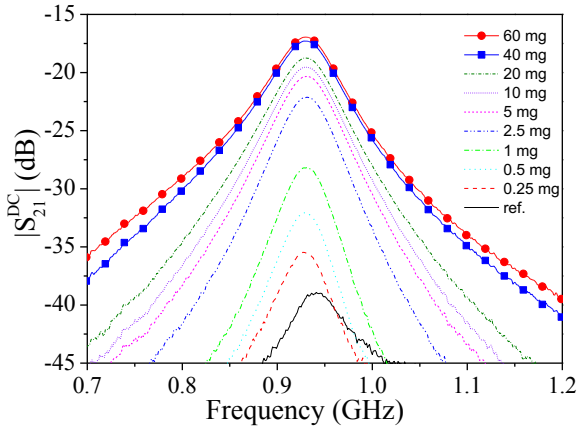
and the maximum sensitivity is 0.032 (g/L)$^{-1}$

Fig. 6. Cross-mode insertion loss for different values of KCl concentration.

Fig. 7. Variation of $|S_{21}^{DC}|_{max}$ with [KCl].

Finally, we have prepared several solutions of CaCl$_2$ in DI water, and we have also measured the cross-mode insertion loss, as well as $|S_{21}^{DC}|_{max}$, for the different solute concentrations. The results are depicted in Figs. 8 and 9. The calibration curve, with $R^2 = 0.99905$, in this case is

$$[CaCl]g/L = -1.76 + 1.29e^{\left(\frac{s_{21}^{DC}}{0.04221}\right)} + 3 \cdot 10^{-6}e^{\left(\frac{s_{21}^{DC}}{0.00848}\right)} \quad (4)$$

and the maximum sensitivity is 0.021 (g/L)$^{-1}$

Fig. 8. Cross-mode insertion loss for different values of CaCl$_2$ concentration.
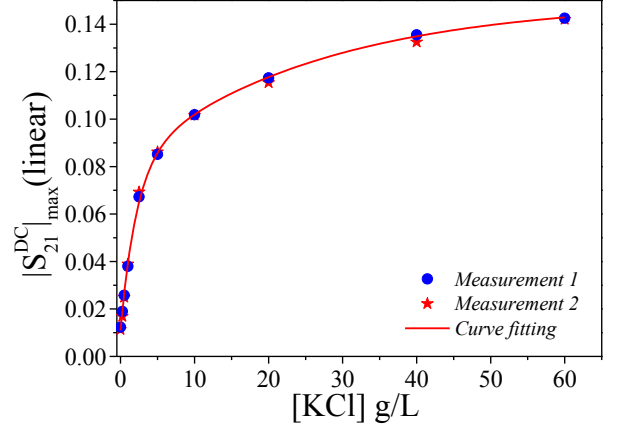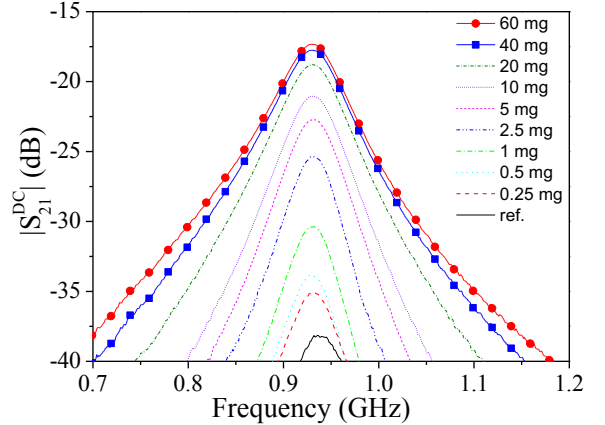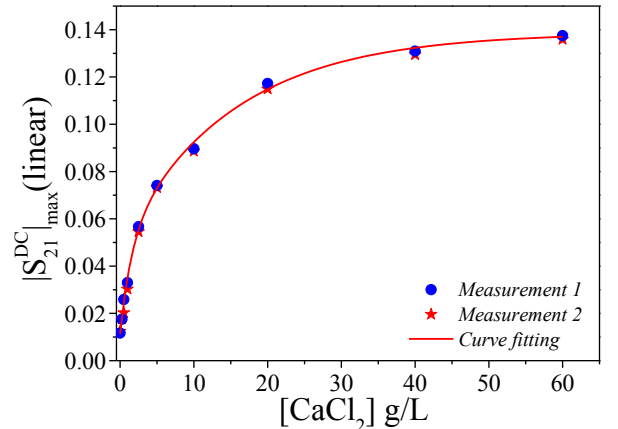

Fig. 9. Variation of $|S_{21}^{DC}|_{max}$ with [CaCl$_2$].

IV. Discussion and Comparison to Other Fluidic Sensors for solute concentration measurements

Note that the dependence of $|S_{21}^{DC}|_{max}$ with the electrolyte concentration is not identical for KCl, NaCl and CaCl$_2$, but similar. The relatively high sensitivity of the reported sensors is in part because the presence of electrolytes in DI water modifies substantially its conductivity (or loss tangent), provided the electrolyte molecules dissociate into anions and cations when dissolved in water. For a certain electrolyte concentration, ion concentration is determined by the molecular mass of the electrolyte. Therefore, since the conductivity depends on ion concentration, we cannot expect identical dependences of the cross-mode insertion loss with the electrolyte concentration for the different electrolytes considered. Moreover, conductivity depends also on ion mobility, also related to the atomic mass. Indeed, at intermediate concentrations (between 2.5 g/L and 20 g/L), the electrolyte that produces higher values of the cross-mode insertion loss is NaCl, which is coherent with the low value of the atomic mass of Na, as compared to K and Ca. However, the differences are not significant. On the other hand, for very small values of the electrolyte concentration (< 2.5 g/L), the accuracy is expected to be limited, and for large values (> 20 g/L), $|S_{21}^{DC}|_{max}$ exhibits a quasi-saturation effect, and the dependence of the conductivity on ion concentration and mobility may be more complex. These facts may explain that at low and high electrolyte concentrations there is not a clear dependence of the cross-mode insertion loss with the ion concentration and atomic mass. Nevertheless, for a given electrolyte, the very good correlation coefficient that results by curve fitting, plus the repetitiveness of the results, reveals that the corresponding calibration curve is useful to determine its concentration.

Besides the work [35], focused on the measurement of NaCl concentration in DI water by using a pair of OCSRR-loaded lines, other papers report solute concentration measurements in liquid solutions. In [38], the considered solute is also NaCl, whereas in [39]-[42], the proposed sensors are used for the measurement of glucose concentration in liquid solutions. Finally, in [43], both NaCl and glucose concentration measurements in DI water are reported. Table I compares these sensors in terms of maximum sensitivity, resolution and dynamic range. It is remarkable the sensor reported in [40], with a very good value of the sensitivity, taking into account that the considered solute is glucose, a substance that does not modify the water conductivity (or loss factor) as electrolytes do. If we focus on the sensors devoted to NaCl concentration measurements, we conclude that the sensor proposed in this paper provides the best combination of sensitivity and resolution. Moreover, sensor fabrication is simple (only a single metal layer is etched) and vias are not required. Therefore, the reported differential sensing structure, based on SRRs is very promising for the measurement of electrolyte content in very diluted liquid solutions.

TABLE I
COMPARISON OF VARIOUS MICROWAVE FLUIDIC SENSORS FOR SOLUTE CONCENTRATION MEASUREMENT IN LIQUID SOLUTIONS

Ref.	Max. sensitivity (dB/g/L)	Resolution (g/L)	Dynamic range (g/L)
[35]	4.3	0.25	80
[38]	0.005	2	10
[39]	0.003	1	300
[40]	1.75	1.5	5.5
[41]	0.017	10	150
[42]	0.003	5	300
[43], Glucose	0.055	1	100
[43], NaCl	1.609	0.5	100
This work, NaCl	**12.27**	**0.25**	**60**

V. Characterization of urine samples

To further validate the proposed sensor, we have characterized several horse urine samples, with different levels of electrolyte concentrations (Na$^+$, K$^+$ and Cl$^-$), which have been externally measured by means of an ISE electrochemical system (the electrolyte concentration measurements and the urine samples have been provided to us by the Biochemistry and Molecular Biology Department of Universitat Autònoma de Barcelona). From the measured values of the individual electrolyte concentrations (i.e., Na$^+$, K$^+$ and Cl$^-$) for each sample, expressed in mEq/L, we have obtained the total concentrations by a simple addition. Note that the total concentration in mEq/L is proportional to the total ion concentration, a quantity of interest as it may be indicative of certain pathologies if certain limits are surpassed. The real-time monitoring of possible variations in the total ion concentration in urine (or even in blood) may be also of interest to detect possible dysfunctions or anomalies, e.g., during disease treatment in hospital environments. Note that with the proposed sensor system, ion concentration variations as compared to a reference sample can be easily measured.

The urine samples provided to us exhibit the total ion concentrations indicated in the right hand side column of Fig. 10. By considering as reference liquid the urine sample with the highest level of ion concentration (615.8 mEq/L), the cross-mode insertion loss that results by injecting the different samples to the LUT channel are those depicted in Fig. 10. It should be mentioned that for monitoring changes in total ion concentration with time in a real scenario, the reference sample should be, obviously, the urine at the beginning of the test campaign. In our case, we have (arbitrarily) considered that the reference sample is the one with 615.8 mEq/L ion concentration, as mentioned above.

The variation of $|S_{21}^{DC}|_{max}$ with the total ion concentration is depicted in Fig. 11. The calibration curve, with $R^2 = 0.99422$, is (the total ion concentration has been designated as [*])

$$[*]mEq/L = -46.62 + 517.26e^{\left(\frac{-S_{21}^{DC}}{0.037}\right)} + 428.68e^{\left(\frac{-S_{21}^{DC}}{0.0036}\right)} \quad (5)$$

and the maximum sensitivity has been found to be -0.00058 (mEq/L)$^{-1}$. From these results, we can envisage the application of this sensing principle to monitor electrolyte concentration variations in biological samples (e.g., urine and blood) in a real scenario.
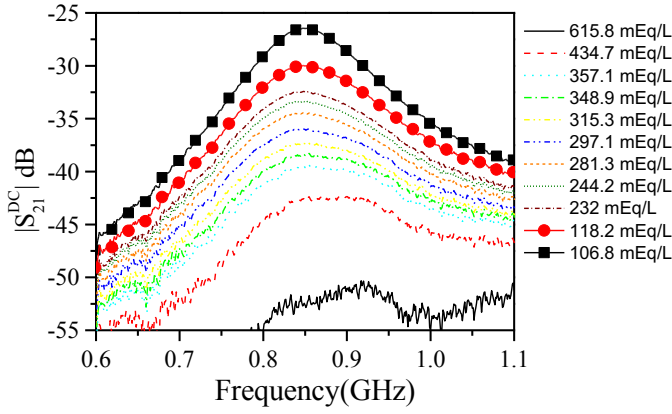
Fig. 10. Cross-mode insertion loss for different values of total ion concentration in urine samples.
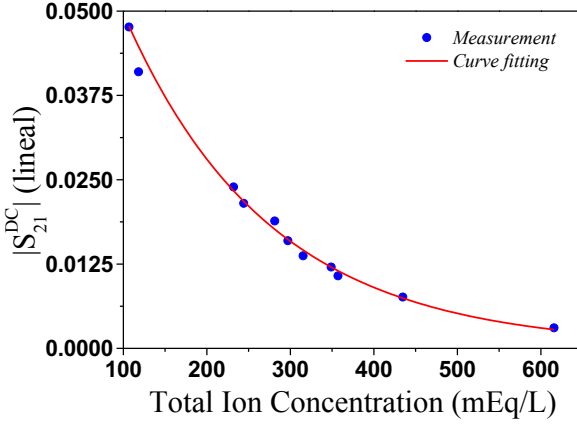

Fig. 11. Variation of $|S_{21}^{DC}|_{max}$ with total ion concentration in urine.

VI. CONCLUSIONS

In summary, novel differential microwave fluidic sensors for electrolyte concentration measurements in diluted aqueous solutions, based on pairs of uncoupled lines loaded with split ring resonators (SRRs), have been reported. The working principle of the sensor is the variation of the cross-mode insertion loss generated by an asymmetric dielectric load of the SRRs. Thus, by loading the fluidic channel of one of the SRRs with the reference liquid (the solvent), and the other fluidic channel with the liquid under test (the solution), it follows that solute concentration determines the cross-mode insertion loss level, which is used as output variable. Sensor validation has been carried out by considering three types of electrolytes: NaCl, KCl and CaCl$_2$. Repetitiveness of the results has been demonstrated, and it has been found that the dependence of the cross-mode insertion loss with electrolyte content does not experience significant variations when the different electrolytes are considered. The combination of sensitivity and resolution of the reported sensor for NaCl concentration measurements in DI water, as compared to those of other sensors (also focused on NaCl measurements), has been found to be very competitive. With these high sensitivities and small resolutions, these differential sensors are of general interest for measuring small solute concentrations in liquids, as well as to detect changes in the properties of liquids (related to variations in the complex

dielectric constant), as compared to a reference. In the last part of the paper, the sensor has been applied to the characterization of urine samples with different levels of ion concentrations, and the potential of the approach for real-time monitoring of total ion concentration in biological samples in a real scenario, has been pointed out.

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