Wireless Microfluidic Sensor for Metal Ion Detection in Water
Yu Liang, Mingsheng Ma,* Faqiang Zhang, Feng Liu, Tan Lu, Zhifu Liu,* and Yongxiang Li

ABSTRACT: In the present work, a wireless microfluidic sensor based on low-temperature cofired ceramic (LTCC) technology for real-time detection of metal ions in water is proposed. The wireless sensor is composed of a planar spiral inductor and parallel plate capacitor (LC) resonant antenna, which integrates with the microchannel in the LTCC substrate between the capacitor plates. Aqueous solutions of Pb(NO₃)₂, Cd(NO₃)₂, Mg(NO₃)₂, Ca(NO₃)₂, NaNO₃, and KNO₃ with concentrations of 0–100 mM were tested with the sensors. The metal ion and its concentration in water can be tested by the amplitude of the reflection coefficient (S₁₁) and the resonance frequency (f₀) of the wireless microfluidic sensor. The metal ion species can be distinguished from the wireless response behavior of the sensor. The detection limit of the sensor for the selected metal ionic solutions could reach as low as 5 μM. The normalized sensitivity of the sensor is 0.47%, which is higher than that of the reported liquid microfluidic sensors based on microwave resonators. The wireless microfluidic sensor of this study is promising for rapid and convenient detection of heavy metal ion pollutants in the industrial wastewater.

1. INTRODUCTION

Water is one of the most important natural resources on the earth.¹ There are alkali metal ions such as Na⁺ and K⁺ and alkaline earth metal ions such as Mg²⁺ and Ca²⁺ in the water, which are essential nutrient elements for the living organism. However, some heavy metal ions such as Pb²⁺, Hg²⁺, and Cd²⁺ are considered as the typical water pollutants because they are nonbiodegradable and can accumulate in organisms through the food chain.³ They can also lead to various diseases and damage human organs such as the kidney, liver, and lung.⁴ As human activities resulted in great pollution to the global water resource environment in recent years, various metal ions have been found in drinking water besides Na⁺, K⁺, Mg²⁺, and Ca²⁺.⁵ Therefore, the development of an accurate, portable, and fast detection method for different kinds of metal ions in water is of vital importance, especially for monitoring water pollution from the factory wastewater.

The conventional methods for analyzing metal ions include atomic absorption spectroscopy,⁶ X-ray fluorescence spectroscopy,⁷ inductively coupled plasma mass spectroscopy (ICP–MS).⁷ Although these techniques have high sensitivity, good selectivity, and low detection limits, the complex equipment and time-consuming process severely restricted their outdoor applications.⁸ In recent years, the microfluidic technology has been demonstrated to detect metal ions in water by high efficiency, low cost, and portable ways.⁹ The reported microfluidic sensors for detecting metal ions are mainly based on the electrochemical or optical mechanism, which depended on the chemical reactions or fluorescent labeling.¹²−¹⁵ With rapid development of wireless communication technology, the wireless sensors based on the capacitive–inductive (LC) resonant circuit have shown some advantages in analyzing the liquid. Reuel et al. presented a wireless passive microfluidic sensor based on LC resonant circuits to simultaneously measure sweat loss volume and conductivity.¹⁶ Dong et al. developed an LC wireless sensor, which can detect the size variation of microtissues during long-term culturing and drug exposure assays.¹⁷ Ebrahimi et al. designed a microfluidic biosensor based on a complementary electric-LC resonator to analyze organic samples and quantify the glucose concentration.¹⁸,¹⁹ It has been considered that the technological convergence with wireless communication is the focusing area of microfluidic technology innovation. On the other hand, the reported microfluidic sensors were usually fabricated with polydimethylsiloxane (PDMS),¹⁰ silicon, and glass.¹¹ However, it is difficult for these materials to integrate with the wireless communication module in one chip. Low-temperature cofired ceramic (LTCC) technology has become a promising alternative to silicon, glass, and PDMS for fabricating microfluidic chips because it possesses many excellent properties such as high temperature stability.
chemical resistance, biocompatibility, and high mechanical strength. In addition, the LTCC process can construct complex three-dimensional structures, which enable the integration of microchannels, microreactors, and microheaters, with passive and active electronic components including interconnects, RLC elements, sensors, actuators, and microprocessors in a single module. The integration of LC wireless sensing structures with the microfluidic channel by LTCC technology had been achieved in our previous work, and it showed good performance for sensing the organic solvent and glucose solution.

In this work, the LC wireless microfluidic sensor based on LTCC technology has been further optimized for sensing the metal ions in the water, and the wireless sensing performance to the metal ion solutions with different kinds and concentrations is presented. The response mechanism of the metal ions in the proposed wireless sensors is discussed.

2. RESULTS AND DISCUSSION

To demonstrate the wireless response of the sensor for the different metal ion solutions, the selected metal ion solutions including heavy metal ions (Pb^{2+} and Cd^{2+}), alkaline earth metal ions (Mg^{2+} and Ca^{2+}), and alkali metal ions (Na^{+} and K^{+}) with the concentrations from 0 to 100 mM were tested by the typical sensor, respectively. Figure 1 shows the amplitude of S_{11} versus resonance frequency of the typical sensor for the tested metal ion solutions. For all the metal ion solutions, the resonance frequencies of the sensor were almost the same with the increase in concentration of the metal ion solutions when the concentrations were within 5 mM. When the concentration of the metal ion in water solution increased to above 5 mM, the resonance frequencies of the sensor decreased with the increase in concentration of metal ions. The amplitude of S_{11} gradually decreased when the concentrations of the solutions increased. However, the variation in the amplitude of S_{11} for different kinds of metal ion solutions is obviously different, which can be used to distinguish the solutions of different metal ions.

Figure 2 shows the variation of the amplitude of S_{11} (ΔS_{11}) versus the concentrations of metal ions. ΔS_{11} is the difference of the amplitude of S_{11} between the tested solutions and the deionized water. As the concentrations of the metal ions increased, the values of ΔS_{11} increased significantly within 5 mM and remained constant after 50 mM. It suggests that the sensor is good for detecting the metal ions with low concentration. The inset in Figure 2 shows that there was a linear relationship between the concentrations of metal ions and the ΔS_{11} for Pb(NO_{3})_{2}, Ca(NO_{3})_{2}, and KNO_{3} solutions when the concentration range was 0–1 mM. The different slopes of the fitted curves can be used to distinguish the three types of the tested metal ions.

The three types of metal ion solutions can also be distinguished by the variation of the resonance frequency (Δf) when the concentration was above 5 mM, as shown in Figure 3. Δf is the difference of the resonance frequency between the tested solutions and the deionized water. It can be seen that the values of Δf increased with the increase in the concentrations of the metal ions. At the same concentration, the sensor presents the biggest Δf for Ca(NO_{3})_{2} solution, and the smallest Δf was found in KNO_{3} solutions. Compared with the trend in ΔS_{11}, there is no saturation in Δf at high concentrations, and thus, the Δf can be used to detect a wider range of concentrations.

For the fair comparison with the reported studies, the sensitivity of the sensor based on the variation of the resonance
The dielectric constant ($\varepsilon$) and conductivity ($\sigma$) of Pb(NO$_3$)$_2$, Ca(NO$_3$)$_2$, and KNO$_3$ solutions with different concentrations were measured, as shown in Table 2. For all the three types of solutions, the dielectric constant decreased slowly in the solution, leading to the conductivity lower than that of Pb(NO$_3$)$_2$, Ca(NO$_3$)$_2$, and KNO$_3$ solutions. The main factor that affects the conductivity in the tested solution may contribute to the decrease in the resonance frequency. In addition, the increase in conductivity in the tested solution can weaken the coupling of electromagnetic energy between the sensor and the reader antenna. As a result, the amplitude of $S_{11}$ decreased with the increase in the concentrations. The main factor that affects the conductivity of the Pb(NO$_3$)$_2$, Ca(NO$_3$)$_2$, and KNO$_3$ solutions is the valence and the migration rate of the ion in the solution. K$^+$ is a monovalent ion, while Ca$^{2+}$ and Pb$^{2+}$ are two divalent ions. The radius of K$^+$ is 1.33 Å, which is bigger than that of Ca$^{2+}$ (0.99 Å) and Pb$^{2+}$ (1.32 Å). This leads to K$^+$ moving slowly in the solution, leading to the conductivity lower than Pb$^{2+}$ and Ca$^{2+}$, as shown in Table 2.

In order to show a comprehensive image on the selectivity of the sensor, a method of data dimensionality reduction called principal component analysis (PCA) was used. It has been reported that the PCA method is a commonly used dimensionality reduction method, which enables a reduction in data and description of a given multidimensional system by means of a small number of new variables. Harnsoongnoen et al. proposed a planar microwave sensor for the detection of salts, sugars, and organic acids in aqueous solution, and they used the PCA method to distinguish organic and inorganic salts, sugars, and organic acids in aqueous solution, and they used the PCA method to distinguish organic and inorganic items.

Table 1. Comparison of the Proposed Wireless Microfluidic Sensor with Other Reported Microwave Microfluidic Sensors

| types of resonator | container materials | fair (GHz) | normalized sensitivity (%) | ref's |
|--------------------|---------------------|------------|---------------------------|-------|
| split-ring resonator (SRR) | PET | 2.0 | 0.091 | 32 |
| split-ring resonator (SRR) | Teflon | 2.559 | 0.11 | 33 |
| complementary split-ring resonator (CSRR) | PDMS | 2.0 | 0.31 | 34 |
| substrate-integrated waveguide (SIW) | glass | 2.31 | 0.062 | 35 |
| circular substrate-integrated waveguide (CSIW) | glass | 4.4035 | 0.044 | 36 |
| quarter-mode substrate-integrated waveguide (QMSIW) | PDMS | 5.791 | 0.11 | 37 |
| LC | LTCC | 0.250 | 0.47 | this work |

The variations of the resonance frequency and the amplitude of $S_{11}$ of the sensor can be attributed to the different dielectric constants and conductivities of the tested metal ion solutions. The dielectric constant ($\varepsilon$) and conductivity ($\sigma$) of Pb(NO$_3$)$_2$, Ca(NO$_3$)$_2$, and KNO$_3$ solutions with different concentrations were measured, as shown in Table 2. For all the three types of solutions, the dielectric constant decreased slowly while the conductivity gradually increased with the increase in the concentrations. In addition, the dielectric constant of these three solutions is basically the same, but the conductivity is obviously different. The resonance frequency of the sensor was related to the dielectric constant of the solution. However, for each solution samples, the resonance frequency of the sensor significantly decreased when the concentration was above 5 mM, as shown in Figure 1. It is indicated that the increase in the conductivity in the tested solution may contribute to the decrease in the resonance frequency. In addition, the increase in conductivity in the tested solution can weaken the coupling of electromagnetic energy between the sensor and the reader antenna. As a result, the amplitude of $S_{11}$ decreased with the increase in the concentrations. The main factor that affects the conductivity of the Pb(NO$_3$)$_2$, Ca(NO$_3$)$_2$, and KNO$_3$ solutions is the valence and the migration rate of the ion in the solution. K$^+$ is a monovalent ion, while Ca$^{2+}$ and Pb$^{2+}$ are two divalent ions. The radius of K$^+$ is 1.33 Å, which is bigger than that of Pb$^{2+}$ (1.32 Å) and Ca$^{2+}$ (0.99 Å). This leads to K$^+$ moving slowly in the solution, leading to the conductivity lower than Pb$^{2+}$ and Ca$^{2+}$, as shown in Table 2.

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In this work, the variables for the PCA method included metal ion species, solution concentration, dielectric constant, conductivity, resonance frequency, and the amplitude of $S_{11}$. The tested data for these variables were reduced in dimensionality and determined by two principal components, which were named components 1 and 2, as shown in Figure 4. For all the solutions with different concentrations, they can be divided into three groups, as marked in the blue (I), red (II), and yellow (III) areas. In part I, component 1 was characterized by the metal ionic solutions with low concentration (0.005–0.2 mM). In part III, the metal ionic solutions with high concentration (5–100 mM) made contribution to component 2. It indicated that it is difficult to distinguish the metal ions in parts I and III according to the contribution of individual component 1 or 2. This can be attributed to the dielectric constant and the conductivity of the different metal ion solutions. It can be seen that the dielectric constant and the conductivity of the different metal ion solutions were very close to each other when they were at the same concentration, especially for the samples with low concentration, as shown in Table 2. In part II, the concentrations of the samples were 0.5 and 1 mM, and the different metal ion solutions with the same concentration made different contributions to component 1 and component 2, so
that the different metal ions could be distinguished according to the combination of components 1 and 2.

The sensing mechanism of the wireless microfluidic sensor can be concluded in Figure 5. Pb(NO₃)₂ solution is taken as an example, and the equivalent circuit and model diagram when the Pb(NO₃)₂ solution flows through the microchannel in the sensor are presented. Cₓ and Rₓ are the capacitance and resistance between the two plates of the capacitor in the sensor, respectively. Cₛ is the capacitance of the LTCC substrate (P1 and P3 parts). According to the liquid-filled circuit model of the plate capacitor, the actual capacitance of the sensor (Cₛ) can be expressed as follows

\[
Cₛ = \frac{C₁²}{C₁ + f_1(εᵢ)} \left[ \frac{1 - \exp\left(\frac{-r}{f_1(σ)(C₁ + f₁(εᵢ))}\right)}{1 + \exp\left(\frac{-r}{f_1(σ)(C₁ + f₁(εᵢ))}\right)} \right]
\]

It can be seen that the Cₛ value is related to the dielectric constant (εᵢ) and conductivity (σ) of the liquid in the microchannel. When the conductivity of the tested liquid is very low, the Cₛ value is mainly determined by the dielectric constant of the liquid. When the conductivity is higher than a certain value, the Cₛ value would increase with the increase in the conductivity. The valence and the migration rate of the ion in the liquid can affect the conductivity; therefore, for the electrolytic liquid sample, the Cₛ value was dependent on the ion valence and the migration rate of the ion. The resonance frequency of the proposed sensor was almost the same when the concentration of the tested solutions decreased in part I, as shown in Figure 4, because the dielectric constant of the tested solutions hardly varied. When the concentrations of the metal ions are above 5 mM (part III), the increased conductivity of the tested solutions can result in the decrease in the resonance frequency. Part II can be recognized as a transition zone, where the dielectric constant and conductivity have different effects on different metal ions, and thus, the three types of metal ion solutions can be distinguished as shown in Figure 4.

3. CONCLUSIONS

In this paper, a wireless microfluidic sensor based on LTCC was introduced. Pb(NO₃)₂, Cd(NO₃)₂, Mg(NO₃)₂, Ca(NO₃)₂, NaNO₃, and KNO₃ solutions with concentrations from 0 to 100 mM were measured by the typical sensor. The detection of metal ion solutions in a low concentration range of 0–5 mM can be achieved through the change in the amplitude of S₁₁, while the variation of resonance frequency can be used for the high concentration measurement. The normalized sensitivity of the sensor based on the variation of resonance frequency presented higher performance than that of the reported microwave microfluidic sensor. The PCA method was used to analyze the metal ion solutions with the different concentrations. The proposed wireless microfluidic sensor had advantages of real-time monitoring, low cost, and high temperature resistance, which makes it a promising sensor for monitoring metal ions in industrial wastewater.

4. EXPERIMENTAL SECTION

4.1. Sensor Design. The schematic diagrams of the designed wireless microfluidic sensor are shown in Figure 6a. The LC circuit consists of a planar rectangular spiral coil (L₁) and a plate capacitor (C₁) series connection. The response behavior of the sensor is totally dependent on the interaction of the tested liquid and the electric field distribution of the capacitor. Since the sensitivity of the sensor is determined by the extent of electric field penetration inside the tested liquid, the microchannel should be located where the maximum electric field occurs. The shape of the capacitive plate in the proposed sensor was designed according to the microchannel morphology to make the electric field largely concentrating in the microchannel. Thus, the optimized sensor would present much better sensitivity than our previous work. The resonance frequency (fᵢ) of the LC circuit can be calculated using eq 3, in which L₁ and C₁ can be calculated using eqs 4 and 5, respectively.

\[
fᵢ = \frac{1}{2π\sqrt{L₁C₁}}
\]

\[
L₁ = 1.39 \times 10⁻⁶(d_{out} + d_{in})N^{5/3} \log \left(\frac{d_{out} + d_{in}}{d_{out} - d_{in}}\right)
\]

\[
C₁ = \frac{ε_{eff}S}{D}
\]

where d_{out} and d_{in} are the external and internal diameter of the planar spiral inductor, respectively. N is the number of turns. ε₀ is vacuum dielectric constant (8.85 × 10⁻¹² F/m). ε_{eff} is the effective dielectric constant between the two plates of the plate capacitor. S is the area of the capacitor plate. D is the distance between the two capacitor plates. The geometry parameters of the designed sensor are shown in Table 3, where t₁, t₂, t₃, and t₄ are the thickness of P1, P2, P3, and P4, respectively.

Since different kinds and concentrations of metal ions in water solutions possess different dielectric constants and conductivities, the capacitance and resistance between the two capacitor plates can be variable when different liquid samples flow through the microchannel in the sensor. It can result in the change in the wireless response signal of the sensor. The equivalent circuit diagram of the mutual inductive coupling between the sensor and the external reader antenna.
coupling between the LC wireless microfluidic sensor and the external reader antenna is shown in Figure 6b. The equivalent input impedance \( Z_{in} \) of the reader antenna can be expressed as follows:  

\[
Z_{in} = R_0 + j2\pi L_0 \left[ 1 + \frac{k^2 f^2}{1 + \left( \frac{f}{f_r} \right)^2} \right]
\]

where \( R_0 \) is the parasitic resistance of the readout coil, \( L_0 \) is the inductance of the readout coil. \( Q \) is the quality factor of the LC circuit. \( k \) is the coupling coefficient between the sensor and the external readout coil. \( f_r \) is the resonance frequency of the LC circuit. The input impedance is related to the reflection coefficient \( S_{11} \) when it is measured using a vector network analyzer as follows:  

\[
S_{11} = \left| \frac{Z_{in} - Z_0}{Z_{in} + Z_0} \right|_{Z_{in}=50\Omega}
\]

where \( Z_0 \) is the intrinsic impedance of the vector network analyzer. When \( f = f_r \), the reader antenna and LC sensor can form a strong inductive coupling, and the amplitude of input impedance reaches the maximum which corresponds to a local minimum of \( S_{11} \). Therefore, the resonance frequency of the wireless sensor can be experimentally determined from \( S_{11} \).

### 4.2. Sensor Fabrication.

The designed wireless microfluidic sensor was fabricated by the LTCC process. The LTCC materials with a dielectric constant of \( \varepsilon_r = 6.2 \) and dielectric loss tangent of \( \tan \delta = 0.001 \) (at 10 GHz) were produced in our laboratory. The thickness of the LTCC green tapes was about 50 \( \mu m \). Silver paste (Dupont, LL612, Wilmington, DE, USA) was used to print the LC circuit on the LTCC green tapes using the screen-printing machine (KEKO, P-200A, Slovenia). The details about the LTCC material and process can be found in our previous work. It is worth to be mentioned that the construction of a uniform microchannel is very important for the LTCC microfluidic devices. Most of the reported studies used a sacrificial material to achieve a flat microchannel structure during the LTCC process. A special lamination process with no sacrificial material was used in this work. First, the LTCC green tapes were stacked to form the layer part 1 (P1) at a laminating pressure of 5 MPa, and the laminating temperature was 58 °C with a holding time of 60 s. Second, the LTCC green tapes with the microchannel (P2) were stacked together with P1 under the laminating condition with 5 MPa, 55 °C, and 40 s. Finally, the layer part 3 (P3) and bottom layer part 4 (P4) were stacked together with the top two parts (P1 and P2) under the same laminating condition used in step 2. The layer parts named P1, P2, P3, and P4 are shown in Figure 6a. The laminated green samples were heated at 450 °C for 120 min to remove the binders and then sintered at 900 °C for 30 min in air. Figure 7 shows the photograph of the typical sensor sample, and the internal microchannel was observed by X-ray nondestructive inspection (YXON, Y. CT Solution, Germany). It can be seen that there is no obvious crack and deformation in the microchannel. The final size of the typical sensor after sintering is about 41 mm × 28 mm × 1.1 mm.

### 4.3. Sensor Test.

The wireless measurement platform used in this work is shown in Figure 8. The external reader antenna was connected to the vector network analyzer (Agilent, E5061B, USA) for obtaining the \( S \)-parameters, and the laptop was used for real-time recording of the data from the vector network analyzer. The tested liquids were pumped into the microchannel of the sensor evenly using a peristaltic pump (Shenchen, Lab 2015, China). Each liquid sample pumped into the sensor was held steadily for 1 min before collecting data. Since the alcohol can be volatized rapidly and is completely miscible with deionized water, the residual metal ion solution in the microchannel can be taken away and make the microchannel clean quickly. Therefore, after each measurement, the microchannel of the sensor was flushed with alcohol.
and put in an oven at 65 °C drying for 30 min to ensure that the sensor recovered to its original state.

Analytical grade reagents, KNO₃, Mg(NO₃)₂·2H₂O, Ca(NO₃)₂·4H₂O (Shanghai Titan Scientific Co., Ltd.), Pb(NO₃)₂, NaNO₃ (Sinopharm Chemical Reagent Co.), and Cd(NO₃)₂·4H₂O (Aladdin Industrial Corporation) were used to prepare the tested water solutions with different concentrations. A multiparameter analyzer (Rex, DZS-708L, China) was used to measure the conductivity of the liquid samples. The dielectric properties of the liquid samples were measured using a coaxial probe measurement kit (Agilent, China) was used to measure the conductivity of the liquid samples. The dielectric properties of the liquid samples were measured using a coaxial probe measurement kit (Agilent, N1500A, USA). Since the coaxial probe method could exhibit obvious uncertainty in the dielectric property measurement when the tested frequency is below 500 MHz,⁴⁶ the tested frequency for the dielectric constant in this work is at 500 MHz.

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The manuscript was written through contributions of all authors.

## Notes

The authors declare no competing financial interest.

# Acknowledgments

The authors would like to acknowledge the supports from the National Key Research and Development Program of China (2018YFB0202503) and the National Natural Science Foundation of China (61971407). M.M. acknowledges the Youth Innovation Promotion Association of CAS and Shanghai Rising-Star Program (20QA1410200).

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