Real-time Microwave Sensor for KCl, MnCl₂ and CuCl Solutions Concentration with Ag Patterns Printed on Flexible Substrates

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Abstract. A novel microwave sensor for real-time chloride concentration analysis is reported. The sensor response to deionised water, KCl, MnCl₂ and CuCl solutions at 1M and 0.1M concentration was tested. The sensing element, in the form of a silver pattern, was printed on polyimide flexible laminate substrate to suit a broad range of applications. The developed system confirmed the viability of using microwaves for real-time chloride solutions monitoring as the reflected signals represented by S_{11} parameters were unique with clearly observed shifts in the resonant frequencies and amplitude changes when placed in direct contact with 20 µl of each solution.

1. Introduction

The composition of chloride solutions must be determined with high accuracy for many applications, such as food and pharmaceutical industries, chemical processing and environmental monitoring, where the content of impurities in water is an important factor. Salinity is a very important parameter for oceanics, marine environment monitoring, seasonal climate prediction, mariculture, and solar engineering [1]. Notably, the detection of Cu^{2+} may serve as an early warning indicator of the onset of corrosion [2].

Traditional methods of aqueous solutions composition monitoring are lab-based and include standard UV-Vis measurements, flame photometry [3], mass spectrometry, ion-sensitive electrodes and amperometric sensors [4, 5]. On the other hand, a range of modern real-time monitoring approaches exist; for example, fibre-optic sensors, MEMs, lab-on-chip and biosensors. Interestingly, salinity measurement in water environments with a long period grating based interferometer was recently reported [6].

Unfortunately, these techniques are inadequate for real-time continuous monitoring of solution concentrations and a novel approach is required. This paper presents novel methods of water salinity determination based on microwave sensing.

2. Microwave sensing

Microwave sensing is a novel but rapidly developing technology which has been successfully used as a sensing method for various industrial applications including fluid level measurements [7], material

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moisture content [8, 9], for continuous process monitoring of biogas plants [10], in the food industry for the verification of the vegetable oil types [11] and in the healthcare industry, for example for real-time monitoring of glucose in diabetic patients [12, 13] and for non-invasive monitoring of bodily fluids [14, 15].

Microwave planar printed patterns for various sensing applications are increasingly used due to their versatility, flat profile and low weight. Their design can be tailored to suit particular applications, coupled with reliability and cost-efficiency. They are easily manufactured using common methods for printed circuit board production, and their impedance can be matched to the input line by altering the micro-strip line feed configuration.

To clarify the principle behind electromagnetic wave sensing suggested in this work for water quality control, it is worth mentioning that microwave sensors in the form of planar printed patterns operate based upon the fact that an object under test, e.g. a water sample, when placed into the vicinity or in direct contact with a microwave sensor, interacts with the electromagnetic waves in a unique manner, which can be specifically correlated with the properties of this material.

By considering how reflected (S_{11}) microwave signals vary at discrete frequency intervals, the change in the signal can be linked to the composition of the object under test.

3. Experimental procedure

3.1. KCl, MnCl$_2$ and CuCl solutions preparation
Potassium chloride KCl (Sigma 60134), manganese chloride $MnCl_2$ (Sigma 2212789) and cuprous chloride CuCl (Sigma C-3294) were dissolved in deionised water at 1M and 0.1M concentrations. All chemical compounds produced clear solutions except CuCl, which is not soluble in water and produced a pale green cloudy suspension.

3.2. Microwave sensor structure
The layout and dimensions of the sensor pattern is shown in Figure 1. Silver was used as the conductive metal material for both the bottom layer, which acted as a ground plane, and the top pattern to maintain chemical neutrality when the device is placed in contact with analyte solution. The thickness of the Ag layers was 35 μm. The microwave sensor was designed on DuPont™ Pyralux® AP Polyimide Flexible Laminate substrate of 2 mil thickness.

A distinct feature of IDE type sensors is their superior sensitivity to change close to the sensor surface, with this sensitivity decaying rapidly with distance away from the surface, as shown in Figure 2. This is advantageous as it reduces significantly the chance of undesirable factors influencing sensor response.

3.3. Measurement setup
The microwave sensor was attached to a Rohde and Schwarz ZVA24 vector network analyser (VNA) via a coaxial cable, as shown in Figure 3. An SMA type Molex connector specified for 50Ω impedance was used. The data (60,000 points for each measurement) was captured in the 0-15 GHz frequency range for the reflected (S_{11}) signals at a constant temperature of 18 °C. The results are repeatable with less than 5% deviation. Notably, average sensor responses are depicted in the graphs shown in following section.
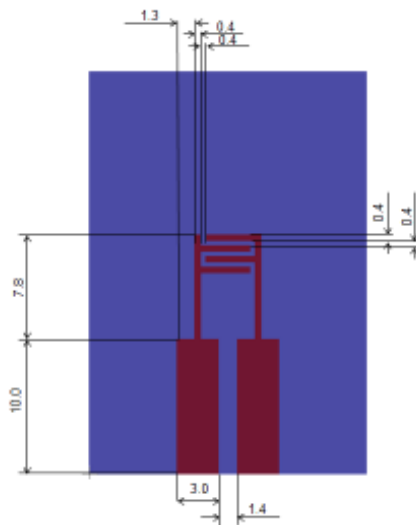
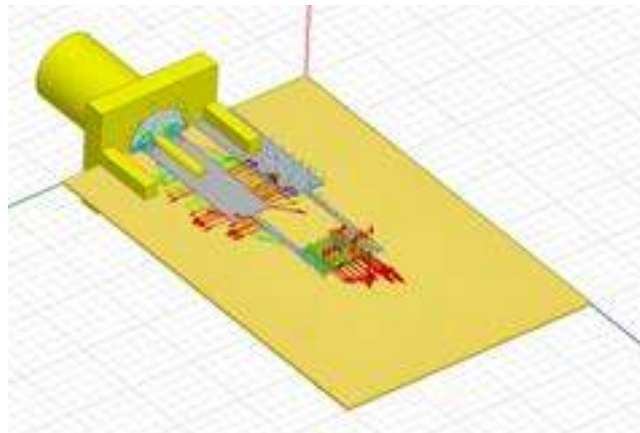**Figure 1**. Ag printed pattern layout. All dimensions are in mm.

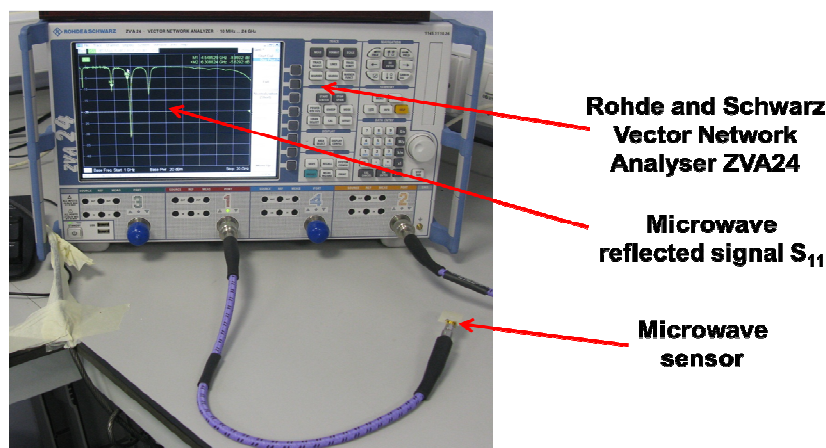**Figure 2**. HFSS model of microwave sensor field distribution.

Figure 3. Measurement setup showing VNA and a microwave sensor connected via coaxial cable.

4. Results and discussion

The spectra for the microwave sensor were recorded in the 0-15 GHz frequency range when in contact with 20 μl of deionised water and KCl, $MnCl_2$ and CuCl solutions of 1M and 0.1M concentrations. The ability of this sensor to distinguish the presence and concentration of various chloride solutions was only evident in the change in the amplitude and resonance frequency of the signal occurring at frequencies below 4 GHz.

Figure 4 illustrates the S_{11} signal response of the microwave sensor in the 0-2.5 GHz range when in contact with deionised water and CuCl solutions in 1M and 0.1M concentrations. One may clearly see that the microwave spectrum is different for each solution with both resonant peaks at around 1 and 2 GHz experiencing a frequency shift as well as the change in the signal amplitude.

Similarly, Figures 5 and 6 illustrate the S_{11} signal response of the microwave sensor when in contact with deionised water and KCl and MnCl solutions in 1M and 0.1M concentrations respectively. These graphs also show that the microwave spectra are different for each solution with both resonant peaks experiencing a frequency shift as well as the change in the signal amplitude. Thus, the developed sensor demonstrated a distinct ability not only to detect the presence of various chloride solutions in deionised water (as evident from Figure 7), but to measure their concentrations as well.
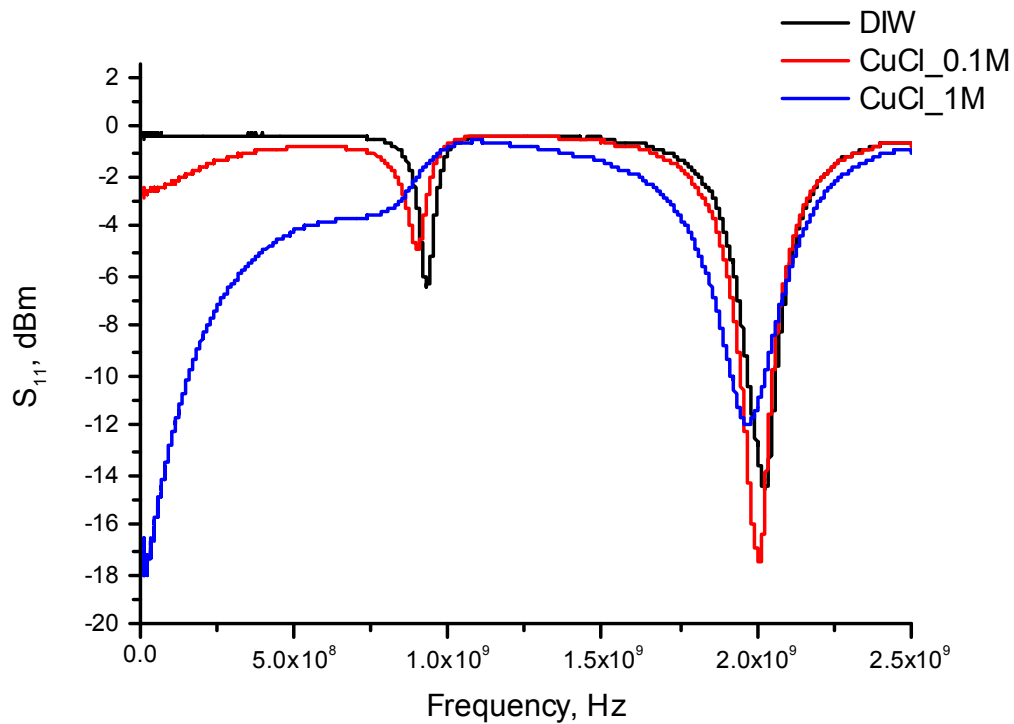
Figure 4. S_{11} signal response of the microwave sensor in the 0-2.5 GHz range when in contact with deionised water and CuCl solutions in 1M and 0.1M concentrations.
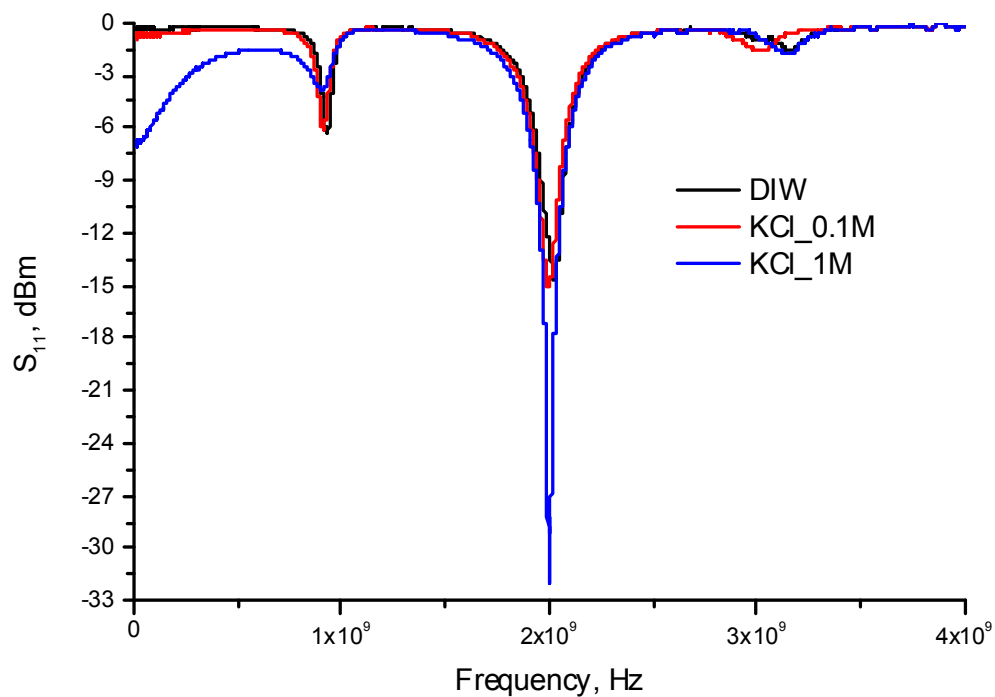

Figure 5. S_{11} signal response of the microwave sensor in the 0-4 GHz range when in contact with deionised water and KCl solutions in 1M and 0.1M concentrations.
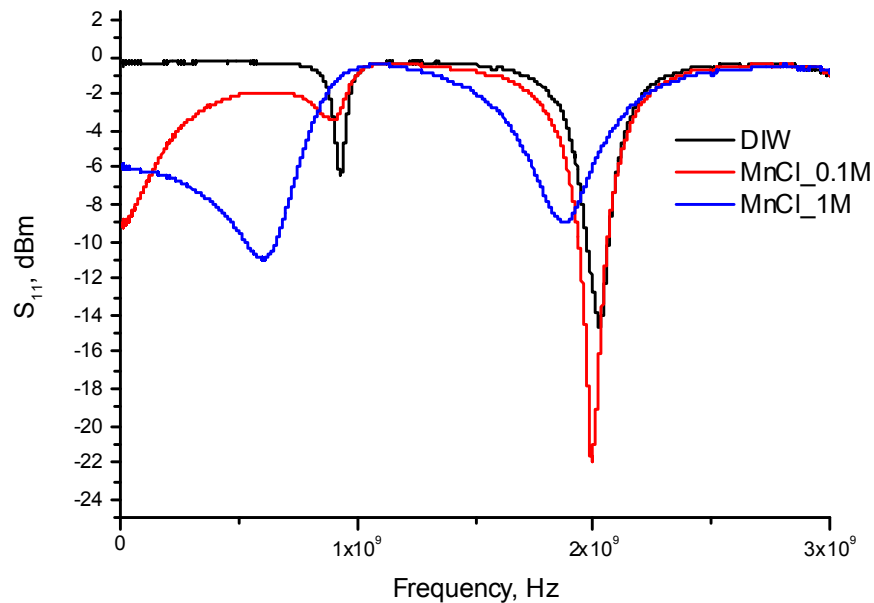
Figure 6. S_{11} signal response of the microwave sensor in the 0-3 GHz range when in contact with deionised water and MnCl solutions in 1M and 0.1M concentrations.
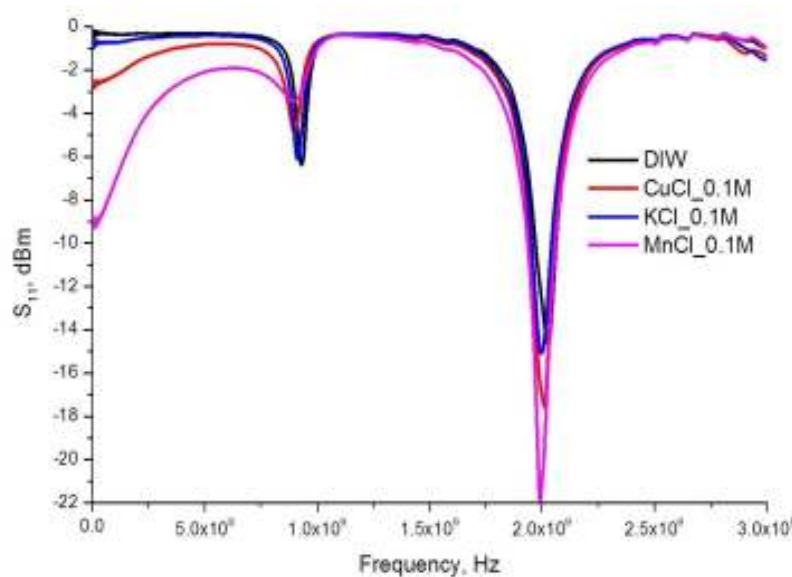

Figure 7. Comparing the microwave sensor responses to different solutions with the same concentration, confirming its ability to distinguish between different types of the solutions as well as their concentrations.

Conclusion

A novel sensor with a silver planar pattern was developed to verify the potential applicability of the microwave sensing approach for real-time monitoring of various chloride solutions presence and concentrations. The sensor responses tested in microwave frequency range revealed that the resonant

peaks amplitude and frequency change once the deionised water and KCl, $MnCl_2$ and CuCl solutions of 1M and 0.1M concentrations in 20 μl volumes were placed in contact with the sensor pattern. Each solution produced a characteristic signature spectrum, which would be used for subsequent testing of unknown chloride solutions and concentrations. A distinct advantage of this novel sensor is that minute solution volume is required for the test – a feature not available with counterpart modern methods.

Acknowledgments

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