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Article

Design and implementation of a non-invasive real time microwave sensor for assessing water-hardness in heat exchangers

Kah Hou Teng[1, 2,*], Andy Shaw[1, *], Muhammad Ateeq[1], Ahmed Al-Shamma'a[1], Stephen Wylie[1], Salim Newaz Kazi[2], Bee Teng Chew[2], Patryk Kot [1]

[1] Department of Built Environment, Liverpool John Moores University, Byron Street, Liverpool, L3 3AF, United Kingdom

[2] Department of Mechanical Engineering, University of Malaya, 50603 Kuala Lumpur, Malaysia

[*] Ph.D. researcher and corresponding author, Department of Built Environment, Liverpool John Moores University, Byron Street, Liverpool, L3 3AF, United Kingdom.

Phone: 0151 231 2244

Email: K.H.Teng@2016.ljmu.ac.uk/alex_teng1989@hotmail.com

[*] Professor and corresponding author, Department of Built Environment, Liverpool John Moores University, Byron Street, Liverpool, L3 3AF, United Kingdom.

Phone: 0151 231 2420

Email: A.Shaw@ljmu.ac.uk

Abstract

A non-invasive-monitoring of concentration and dielectric properties of calcium hardness in heat exchanger cooling water was conducted with a 2.5 GHz microwave cavity resonator designed and fabricated locally for the experiment. The principle of electric dipole moment theories were used to analyse the sample solution that occurs as a function of calcium ion content. Artificial different of water hardness were prepared by mixing $CaCl_2$ in deionized water. The sample was centrally positioned in the electric field of the TM_{010} mode of a resonant cylindrical cavity. COMSOL simulation package was used to compare and validate the experimental cavity resonator frequency. Transmission signal (S_{21}) measurements via Vector Network Analyser (VNA) at different concentrations were observed a linear relationship in amplitude with different frequency changes. In addition, calcium absorption provides a first order change in material polarisation (i.e. real permittivity), and second-order transitions associated dielectric losses (i.e. imaginary permittivity). These research findings introduce a novel technique of real time monitoring of water hardness concentration by using non-invasive microwave sensor.

Keywords: Non-invasive, microwave cavity resonator, real-time monitoring, water-hardness, heat exchanger

Nomenclature

VNA	Vector network analyser
EM	Electromagnetic
S_{11}	Reflection coefficient
S_{21}	Transmission coefficient
Ɛ’	Dielectric constant
Ɛ’’	Dielectric loss
f_{nml}	Resonant frequency a TM_{nm0}
c	Velocity of light
μ_r	Relative permeability of the material
ε_r	Relative permittivity of the material
D	Depth of the cavity
p_{mn}	mth root of the Besel function of the nth order
b	Radius of the cavity.

V_c	Volume of cavity resonator
V_s	Volume of under test dielectric sample
f_s	Shifted frequency
Q_{LS}	Q-value for the sample inside the cavity resonator
Q_{L0}	Q-value for the empty cavity resonator loaded with coupling loop
f_0	Resonant frequency
Δf	Bandwidth of the reflection curve

1. Introduction

Accumulation of insulated layer of deposit such as calcium carbonate, calcium sulphate and calcium phosphorus on piping surfaces in process plants and domestic equipment leads to detrimental effect on plant operation and equipment performance [1, 2]. As a consequence, effective monitoring method is crucial to ensure that water used in cooling equipment, boiler, or other industrial equipment is satisfactorily controlled so that the desired results, quality and efficiency are achieved to maintain plant operation performance [3]. It is important to establish a monitoring system for water quality in industrial plant. This is to ensure that the plant productivity is maintained, energy savings are achieved and the operation of the plant is compliant with the environmental regulations. The continuous monitoring also means a reduced risks of sudden break down of boiler, heat exchangers, cooling tower, etc. during an industrial operation [2, 4]. Cotemporary practice is to use the traditional manual methods in industrial water cooling systems. A conventional monitoring typically comprises of plant operators or technicians conducting schedule chemical tests and comparing the results to specified chemical control limits [5]. The schedule tests can detect the properties such as pH, conductivity, suspended solids, alkalinity, hardness and others [6]. From the evaluation of the test results, the plant operator can manually regulate a chemical feed pump or broken down valve by estimating the necessary degree of changes to ensure the smooth production. From a sustainability point of view, a non-invasive on-line monitoring system can provide an automatic and continuous monitoring that is real time, faster, efficient and reliable. Nowadays, the industrial is very much interested in finding an automated monitoring and quality control of cooling water system than the existing manual operation system. This will significantly contribute to the process and production of the industry [7]. Hence, an automated continuous on-line monitoring method is much desired by the industry [8].

Over the last decade, rapid technological developments have been noticed in electronics and microprocessor technology. There are wide ranges of instrumentations available to monitor the quality of cooling water particularly calcium contains which is the main fouling ingredient. However, the existing technologies require highly skilled and trained workers and is costly to manufacture and maintain [9]. Examples include ion chromatography, ion selective electrode and Flow Injection Analysis (FIA). At present, there is no system which can completely fulfill needs of the industry, specifically, their requirement to carry out real time monitoring of the cooling water condition in an efficient and cost-effective way [10]. Therefore, effective and reliable real-time monitoring system is essential.

Microwave sensing technique has a potential to cope with the present industrial real-time monitoring demand [11]. It is an innovative technology and has been expeditiously developed over the last few decades to be used as a sensing and monitoring technique for various industrial applications. These include but not limited to the detection of material moisture content and material characterization in construction industry. Examples include moisture detection in concrete blocks, bitumen characterization [12, 13], monitoring of fluid level in process industries [14], for continuous process monitoring of plants [15], and in the healthcare industries, for example a real-time monitoring of glucose in diabetic patients [16] and for non-invasive monitoring of body fluids [17].

Various research studies have been carried out on using microwave sensor to detect the constituents concentrations in water solutions [18]. Principally it is done by considering the variation in transmitted (S_{21}) microwave signals at discrete frequency intervals and linking it with the change in the composition of the solution under investigation. This change is realized through the change in the signal and the unique spectrum. The change in the hardness of water will affect its dielectric properties. It can be recognized by comparing the complex permittivity at several frequencies of the pure water and the one with higher water hardness concentration [19]. The EM wave technique could suit this application, coupled with reliability and cost-efficiency.

2. Experimental Design and Methodology

2.1 Microwave theory and application

Materials can be studied from the data of their interactions with microwaves. This interaction can be realized in the form of a unique signal spectrum known as reflection coefficient (S_{11}) and transmission coefficient (S_{21}). Generally, these measurement quantities vary with the change in the parameter such as permittivity and conductivity of the materials [20]. Permittivity is basically a measurement of the response of a dielectric medium to the applied microwaves that can be detected through its changing electric field. It depends on the material's ability to polarise in response to the applied field [21]. The two primary parameters that define permittivity are known as dielectric constant and dielectric loss of material [22].

i.) Dielectric constant (Ɛ'): The phenomena of energy storage and reduction in the wave velocity when EM wave passes through the material which is always used to distinguish dielectric constant values of different materials. Different dielectric constant values are observed, because of the changes in polarisation inside the material.

ii.) Dielectric loss (Ɛ''): It is the loss of electromagnetic energy propagating inside a dielectric material. The reduction of the wave magnitude is due to the rotation and oscillation of the molecules in response to the applied electric field and loss of energy owning to the friction between the molecules.

Changes in the materials concentration, type, percentage etc. will be followed by the alteration of its permittivity yielding a unique signal spectrum when it comes in contact with microwave. In this way, the material is characterised over the range of discrete frequencies.

2.2 Design of cylindrical microwave cavity resonator

The EMW cavity resonates at particular frequencies depending on the dimensions of the cavity. In addition, permittivity of the material (lossy / low loss materials) has an impact on the resonant peak generated and its quality inside the cavity. Resonance occurs when the magnetic and electric fields form a perpendicular/standing wave inside the cavity. This combination of an electric and a magnetic field inside the cavity is known as a mode. The term Transverse Electric (TE) and Tranverse Magnetic (TM) are used to name each of the modes depending on the direction of its electric and magnetic components. Each mode has its own resonant peak/frequencies. Also, the cavity can have more than one modes depending on its design and dimensions. The cylindrical microwave cavity resonator designed in this work along with the experimental setup is shown in Figure 1. It was fabricated from aluminium and has 4 parts (the top, body and two ports to launch microwave inside the cavity). Sample solution was prepared in low-loss quartz tube (outer diameter of 15mm) with thin wall and inserted in the middle of cavity as in Figure 1. The cavity was designed such that its fundamental mode of operation (i.e. TM_{010}) occurs at close to 2.5 GHz. Equation (1) can be used to calculate the resonant frequency for a particular mode in a cylindrical cavity [23].

$$f_{nml} = \frac{c}{2\pi\sqrt{\mu_r \varepsilon_r}}\sqrt{\left[\left(\frac{p_{mn}}{b}\right)^2 + \left(\frac{l\pi}{d}\right)^2\right]} \quad (1)$$

Where, c is the velocity of light, ε_r is the relative permittivity of the material, μ_r is the relative permeability of the material, p_{mn} is the mth root of the Besel function of the nth order, b is the radius of the cavity and d is the depth of the cavity.

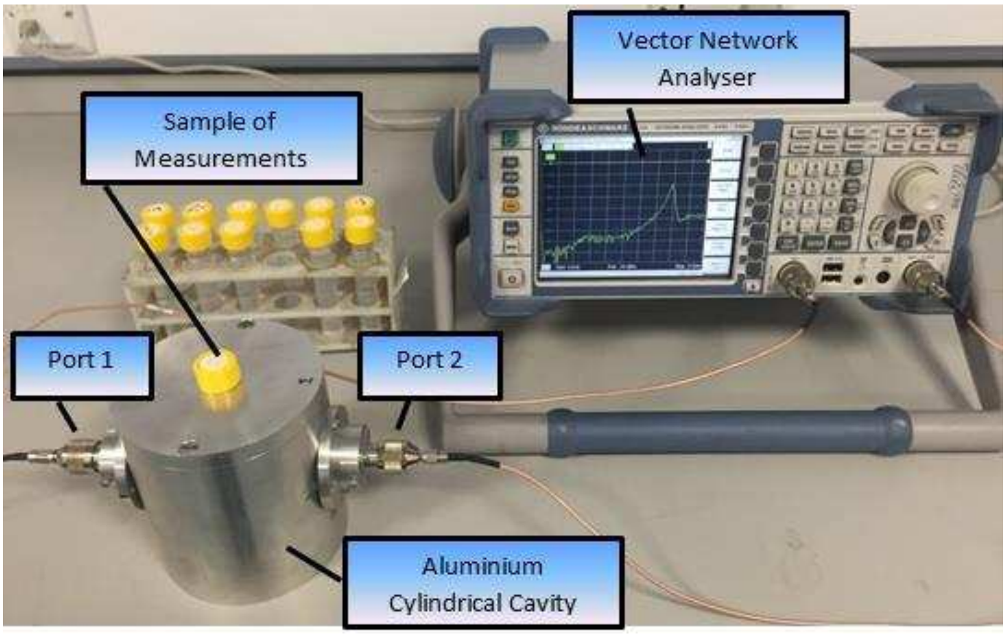

Figure 1 Microwave cavity resonator measurement system used in experiment

The fundamental mode enables high measurement sensitivity due to the strong fields inside the cavity. Each mode generates a resonant peak with a quality factor Q. A high Q indicates a sharp resonant peak that readily analyse the sample and hence improves the accuracy of the sensor. Furthermore, TM_{010} chosen in the current study for dielectric property measurement of the samples yields a high intensity, uniform electric field near the cavity's central axis. The electric filed in this mode is normal to the direction of propagation of wave and parallel to the wall of the sample tube, resulting in only minor modification of the local electric field when the quartz tube is introduced into the cavity [24]. The distribution of electric field magnitude for the TM_{010} mode is shown in Figure 2. For maximum sensitivity of the dielectric property measurement, the sample needs to be placed in the region of maximum electric field (i.e. on axis) [24]. COMSOL simulation package was used to validate the cavity resonant mode and the frequency.

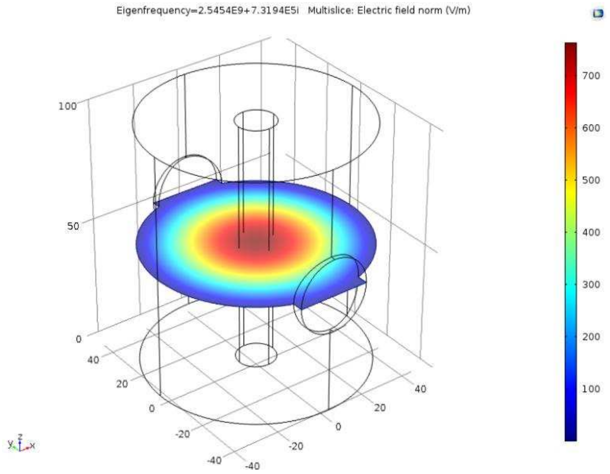

Figure 2 COMSOL model of the aluminium cavity resonator showing the distribution of the electric field magnitude in TM_{010}.

2.3 Sample preparation

Experiments were conducted to detect varying concentration of calcium hardness. Synthetic hard water was prepared from a combination of de-ionized water and calcium chloride for various concentrations. In addition, different chemicals were introduced to understand the effects of anion and cation on the microwave measurements. Temperature measurements of samples were taken at near room temperature (25 ± 0.5 ^{0}C), measured by using K-type thermocouple with Picolog recorder. Three sets of microwave measurements were taken and are listed below the samples as presented in Table1, 2 and 3:

i.) Measurement of different concentrations of calcium hardness in $CaCl_2$

ii.) Measurement of varying cation effects on chloride contents.

iii.) Measurement of varying anion effects on calcium hardness.

Table 1: List of samples for monitoring and analysis of water hardness

Sample type	Concentration, Molar	Temperature, °C
Calcium Chloride ($CaCl_2$)	0	25 ± 0.5
	0.001	25± 0.5
	0.005	25± 0.5
	0.01	25± 0.5
	0.05	25± 0.5
	0.1	25± 0.5

Table 2: List of samples for monitoring and analysis of anion effects in water hardness

Sample type	Concentration, Molar	Temperature, °C
Calcium Chloride ($CaCl_2$)	0.1	25± 0.5
Calcium Bromide ($CaBr_2$)	0.1	25± 0.5
Calcium Nitrate ($CaNO_3$)	0.1	25± 0.5

Table 3: List of samples for monitoring and analysis of cation effects in water hardness

Sample type	Concentration, Molar	Temperature, °C
Calcium Chloride ($CaCl_2$)	0.1	25± 0.5
Sodium Chloride (NaCl)	0.1	25± 0.5
Potassium Chloride (KCl)	0.1	25± 0.5

2.4 Data Acquisition and Analysis

Experiments were conducted to measure samples with different concentrations and compositions of chemicals. All the microwave measurements were carried out as the transmission signal, S_{21}. R&S®ZVL Vector Network Analyser, 9 kHz – 6 GHz (Rohde & Schwarz) was used to transmit the microwave signal and capture the microwave response. The measurements were automated and were recorded by LabVIEW software (National Instrument). The R&S®ZVL is a network analyser for use in research studies due to its powerful measurement capabilities and tremendous increase of the analysis efficiency. However, for the purpose of feeding the microwave energy to a commercial microwave sensor, a more portable and cost-effective microwave source can be developed which may deliver the necessary VNA functionalities alongside the range of the required measurement frequency. A microwave input power of 1mW (0 dBm) was launched into the cavity. Full two-port calibration was carried out to ensure the accuracy of the measurement.

To analyse the acquired samples data, first order cavity perturbation theory was used. It had to calculate the dielectric constant and dielectric loss of the materials under test (MUT). Complex dielectric property (complex permittivity) of materials can be defined by the Equation 2 [25].

$$\varepsilon = \varepsilon' - j\varepsilon''$$
(2)

Where, ε' is the dielectric constant of the material in an applied electric field and ε'' is the dielectric loss property of the material. The real and imaginary parts of the permittivity of MUT were determined by the frequency shift and change in Q-value presented in Equations 3, 4 and 5 [26].

$$\varepsilon' = 1 + 0.539\left(\frac{V_c(f_o - f_s)}{V_s f_o}\right)$$
(3)

Where, ε' is the dielectric constant, V_c is the volume of the cavity, V_s is the volume of the sample under test dielectric sample, f_o is the resonant frequency of the empty cavity and f_s is the resonant of the loaded cavity.

$$\varepsilon'' = 0.269 * \frac{V_c}{V_s} * \left(\frac{1}{Q_{LS}} - \frac{1}{Q_{L0}}\right)$$
(4)

$$Q = \frac{f_0}{\Delta f}$$
(5)

Where, ε'' is the dielectric loss, Q_{LS} is the Q-value for the sample inside the cavity resonator, Q_{L0} is the Q-value for the empty cavity resonator loaded with coupling loop, f_0 is the resonant frequency and Δf is the bandwidth or shift in the frequency. The bandwidth in the vicinity of the resonance can be measured for the Q factor [27].

2.5 Numerical Simulation

Numerical simulations were conducted by using COMSOL simulation package. Numerical study was conducted to simulate the resonant frequency for the empty and sample filled cylindrical cavity. The simulation frequency range was set at 2.2–2.9 GHz at the interval of 0.001 GHz steps to acquire a better resolution and accuracy of the simulated results comparable to the experimental results.

3. Results and Discussion

3.1 Effect of concentration

Transmission coefficient (S_{21}) measurements were obtained directly from the network analyser. The resonance frequency occurs when there is a sharp 'valley' in S_{21} as energy from port 1 of VNA will be dissipated in the chamber. Results of varying concentration of $CaCl_2$ were obtained and shown in Figure 3. A close analysis of Figure 3 shows there is a change in the magnitude and frequency of the spectrum at around 2.5 GHz when different concentrations of samples were tested. The results also demonstrate the sensitivity of the designed sensor. The analysis of Figure 3 shows that the spectra of the samples with the molar concentration of 0 and 0.001 overlap each other making them indistinguishable. However, the difference of molar concentration of $CaCl_2$ greater than 0.005 can be easily identified. Hence, an improvement is required in terms of detecting a minute change in the concentration equal or less than 0.001 molar.

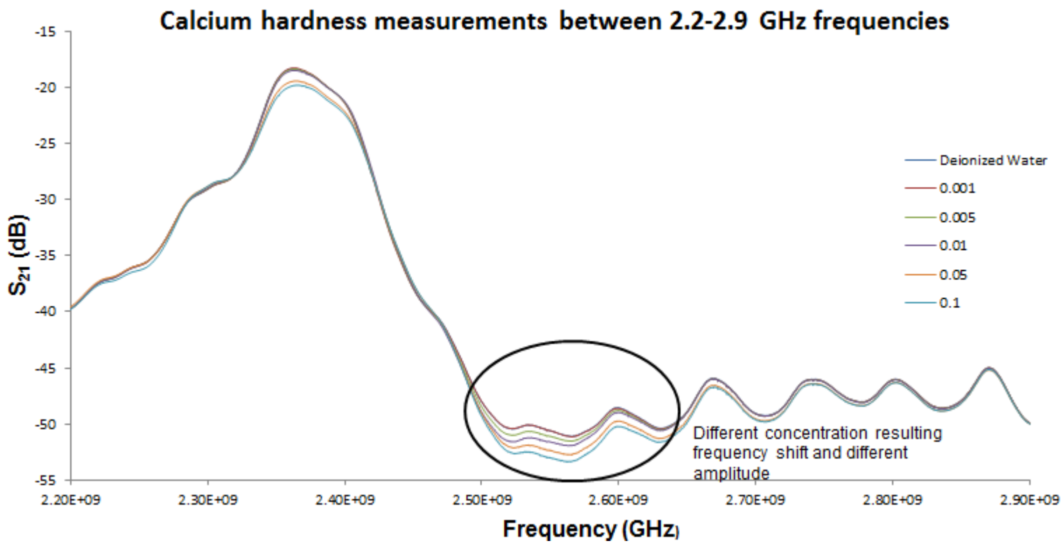

Figure 3 Microwave measurements under different $CaCl_2$ concentration

Varying step change in the concentration of $CaCl_2$ was used to show the sensitivity of the measurement sensor ranging from a small to a large change. To elaborate the frequency shift and the amplitude change, the varying molar concentration was plotted against them. The results are shown in Figure 4 and Figure 5 respectively.

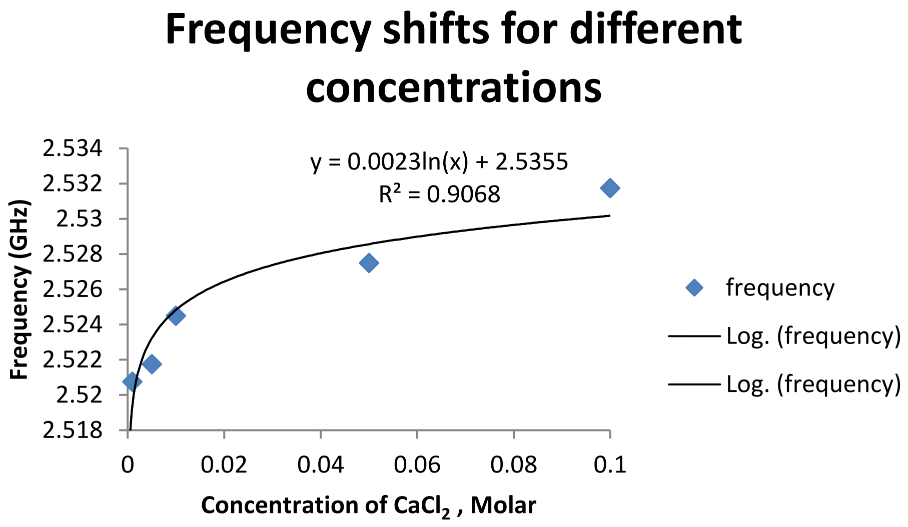

Figure 4 Frequency shifts at varying $CaCl_2$ concentrations

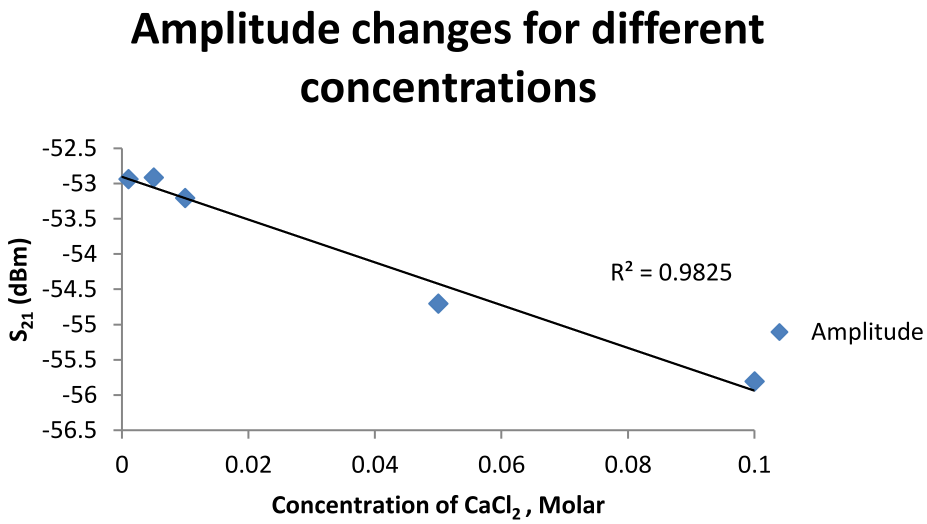

Figure 5 Amplitude changes for varying $CaCl_2$ concentration

The results in Figures 4 & 5 show an acceptable change in both the frequencies and amplitudes for different samples. The R-square values demonstrate this with respect to the change in the concentrations. Amplitude changes for varying $CaCl_2$ concentration show a good linear regression for the measurements of change in concentration, hence showing the effectiveness of the microwave sensing technique. On the other hands, frequency shifts at varying $CaCl_2$ concentration shows acceptable logarithm regression.

In addition, permittivity measurements were used to strengthen the findings of the present investigation. Equations 3 & 4 were used to calculate both the dielectric constants and loss values of the various samples. Figure 6 shows the results of change in the dielectric constant of a solution. It illustrates a decrease in dielectric constant of a solution with the increase in its hardness.

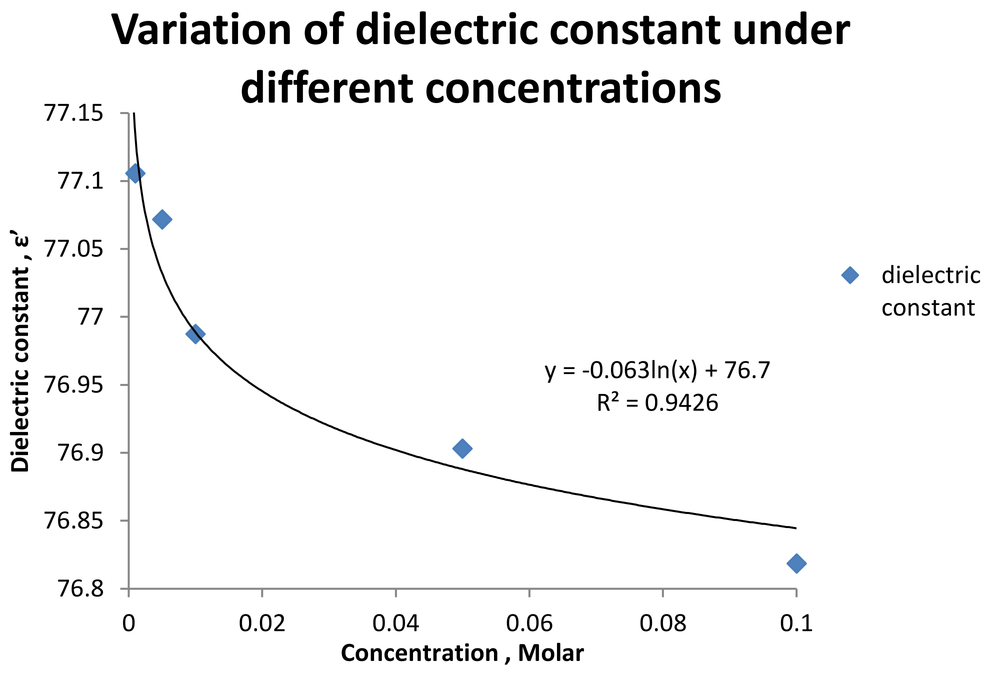

Figure 6 Variation of dielectric constants under different concentrations

On the other hand, Figure 7 shows an increase in dielectric loss of solution with the increasing of the hardness of the solution, i.e. increase in the molar concentration of $CaCl_2$. The change of a dielectric constant value with the concentration could be explained and it could be due to the electric field between calcium and chloride ions which dissociate in the solution [28]. The electric field orients the polar water molecules resulting oxygen face towards the calcium ion and hydrogen face towards the chloride ion. The orientation of these polar water molecules make its own electric field which cancels out most of the electric field and that would exist and effectively lowers the applied external field. This is the possible reason of lowering the dielectric constant as the concentration increases [29].

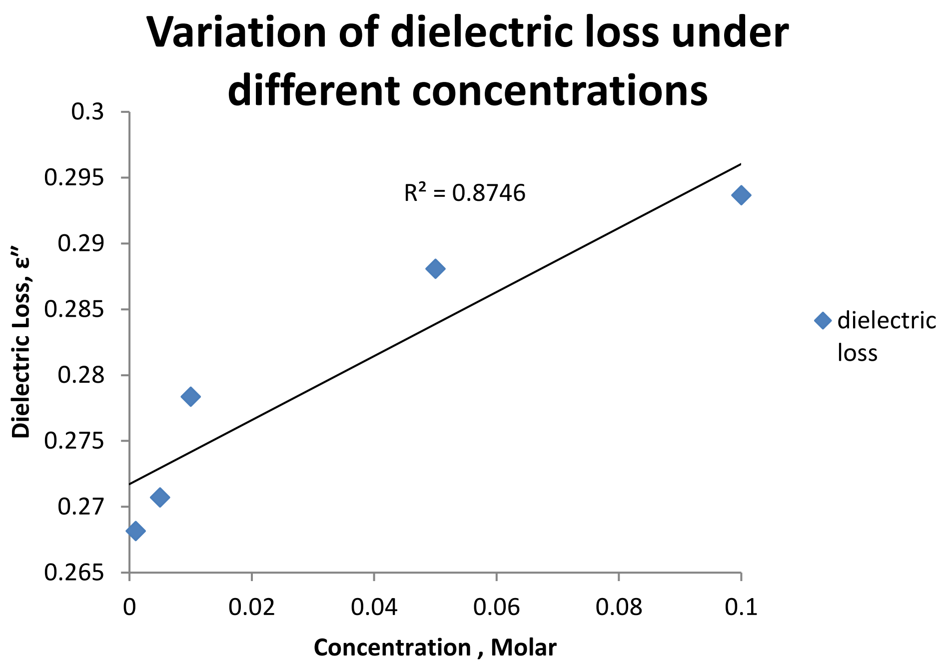

Figure 7 Variation of dielectric loss under different concentrations of $CaCl_2$

With regards to the variation in the dielectric loss with concentration, it is believed that the motion of ions in electrolyte solution is the key factor [30]. The dielectric loss increases with the increasing of the concentration. This is supported by the fact that higher numbers of ions in the electrolyte solution, burdens the dipole movement, prevents the water molecules to oscillate freely at higher frequencies. In other words, it increases the drag to the rotation of the water molecules. This causes higher friction and results in the increase of a dielectric loss value [31].

3.2 Effect of different cations and anions in water hardness

To enable people to draw a concrete conclusion on the potential of microwave technique to detect various hardness concentrations, an experimental work to analyse the effect of cation and anion on EM wave spectrum, these investigations are performed. Figure 8 illustrates the microwave signal spectrum under the effect of different cation with chloride based in the same molar concentration. Three chemicals namely, calcium chloride, sodium chloride and potassium chloride were used in this investigation. The area of interest is highlighted in Figure 8. It can be seen clearly that at the region around 2.5 GHz, amplitude and frequency changed when different cations were employed. However, no obvious change was observed at around 2.4 GHz frequency (highlighted in Figure 8).

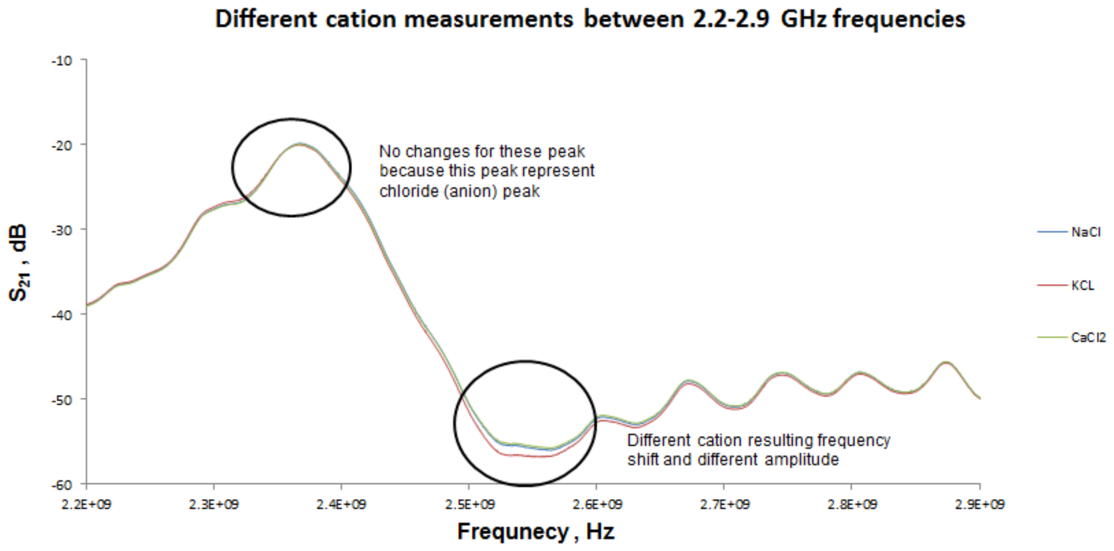

Figure 8: Frequency spectrum measurements for different chloride based cations

Figure 9 demonstrates the microwave frequency spectra under the effect of different calcium based anions. The measurements were carried out using 3 different anions i.e. chloride, bromide and nitrate based anions. It is indicated at the highlighted region around 2.5 GHz that there are no changes in term of amplitude and frequency for different calcium based anions. This emphasize that the 2.5 GHz frequency region shows some promising results in terms of using the microwave sensing technique to monitor the hardness of the solution.

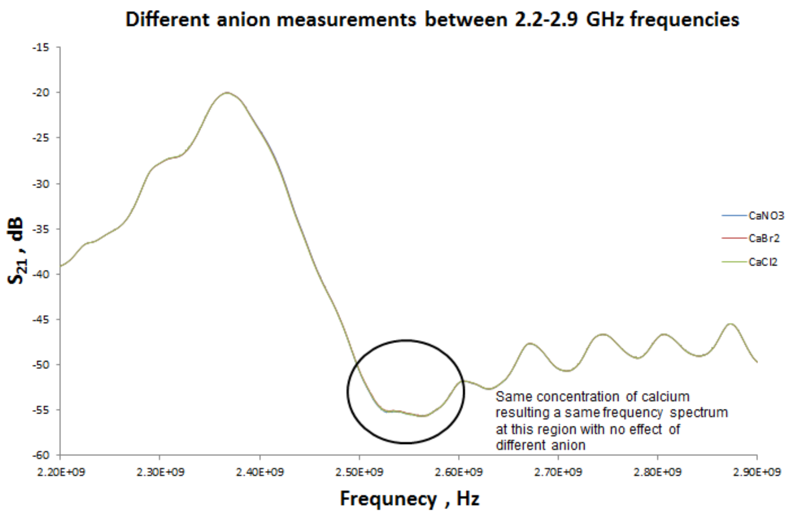

Figure 9: Frequency spectrum measurements for different calcium based anion.

3.3 Repeatability of the technique and validation of the experiments

To validate the accuracy of the sensor the simulated and experimental results were compared. COMSOL simulations were carried out to compare the simulated and experimental results of an empty and water-filled cavity. As seen in Figure 10, transmitted signals (S21) were reported relatively comparable to the practical measurement. However, it is observed that the results could not exactly match because of the ideal environment in simulations. On the other hands, practically the measurement instrumentations might experience energy loss while the signal is transported from port 1 to the cavity through a co-axial cable and received at port 2.

In addition the accuracy, reliability and consistency are important factors in any scientific measurement. For this purpose, repetition test was performed by measuring the water sample 10 times over few day periods. Root mean square (RMS) error was calculated and was found to be >99% for the repetition. Figure 11 shows the repetitive measurements for 10 de-ionized water measurements.

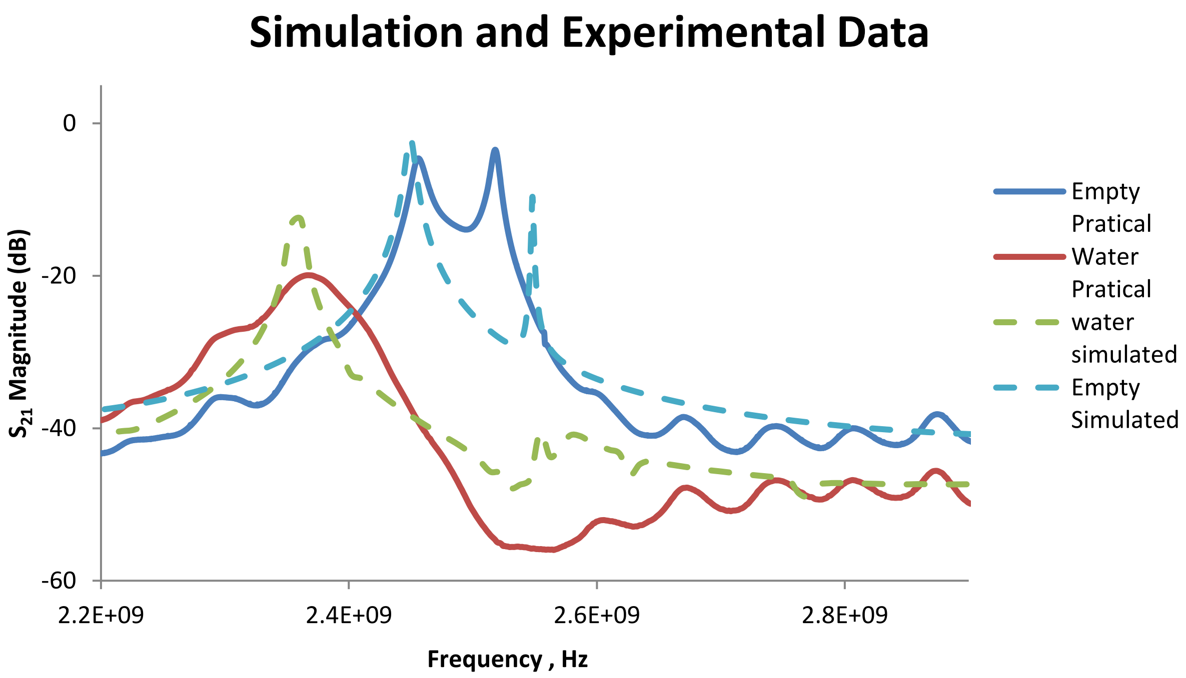

Figure 10 Comparison of simulated and experimental results

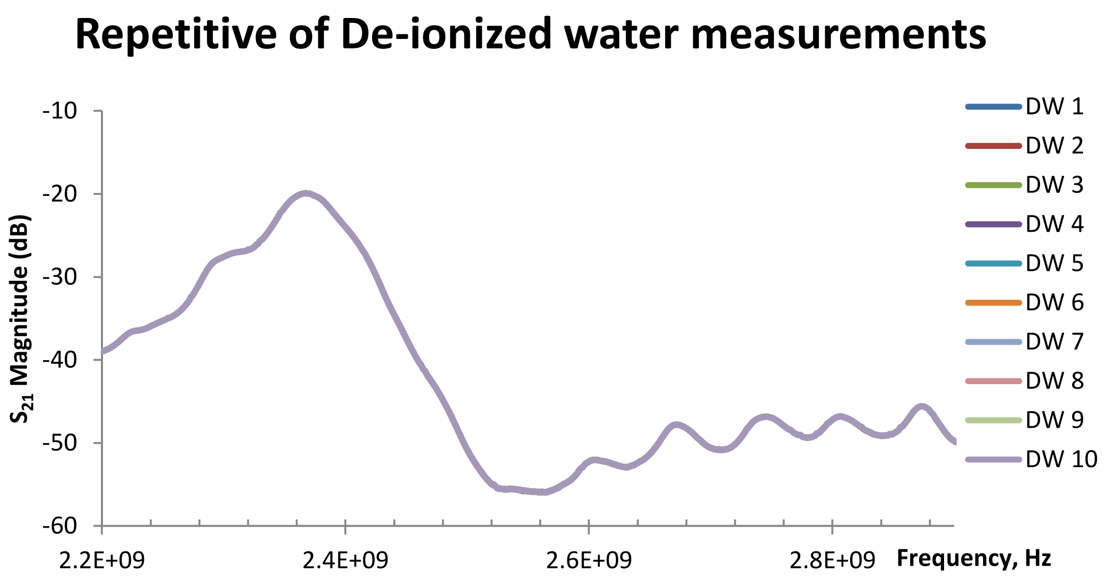

Figure 11 Repetitive of frequency measurements for de-ionized water

Conclusion

In these experiments, a microwave cavity resonator has been successfully designed and simulations show that it's theoretical and real practical data are well validated. On the top of it, a novel non-invasive real time electromagnetic wave sensor for assessing water-hardness was taken under consideration. In summary, it is observed that electromagnetic wave sensor acts successfully for water-hardness monitoring in industrial applications. In addition, this electromagnetic wave sensor technique could be potentially the finger prints for the chemical elements which could be addressed in the future research. These incredible findings could be a good practice for real-time monitoring systems for cooling-water assessments in industrial heat exchangers.

Conflict of interests

The authors declare that there is no conflict of interest regarding the publication of this paper.

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