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Smart Sensing Applications of Radio Frequency Resonators based on Complex Permittivity

복소 유전율에 기반한 공주파 공진기의 스마트 감지 응용

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Graduate School of
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이 논문을 공학박사 학위논문으로 제출함

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기계항공공학부

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Abstract

Complex permittivity is an intrinsic property of any dielectric material representing its ability to store electrical charge when electric field is applied. They are unique to the material and have been used in a wide range of applications to characterize a wide variety of materials from building material to biological tissues. In this work, these dielectric properties are utilized to perform sensing at radio frequency towards smart sensing applications namely, soil moisture sensing in plant factories and on-chip Polymerase Chain Reaction (PCR) for point-of care devices in the bio-medical field. For sensing, radio frequency resonant circuit of inductor - capacitor pair is used. The interdigital capacitor structure responds to dielectric property changes, and this change when coupled with the spiral inductor structure, results in resonant frequency change of the circuit. This resonant frequency shift is a function of the

dielectric environment hence leads to sensing of any changes. In case of moisture sensing application, the sensor is developed to be embedded in the plant growth substrate, in the root zone area. It works in inductive coupling with the resonator sensor, located outside of the substrate. The changes in the gravimetric moisture contents at the root zone leads to change in the capacitance of the embedded resonator and hence its resonant frequency. This information is transferred to the outside interrogating resonator and hence moisture contents can be monitored remotely. The second application towards on-chip PCR, requires the sensor to process small volumes of test samples. For this a miniature resonant circuit with well structure over the capacitor region, and with a protective coating over the circuit tracks, is developed. Various concentrations of different sized PCR products are tested, and the detection range is determined. The detection principle being the same as in the previous case. For both cases, a

theoretical analysis is performed to support the measurement data. The sensors are fabricated with inexpensive and simple fabrication techniques and material, hence easily mass producible. Sensing at radio frequency brings in the possibility of integrating with wireless sensor network in Internet-of-things which is becoming a common industry norm these days.

Keyword : radio frequency, complex permittivity, inductive coupling, gravimetric water content, polymerase chain reaction.

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Chapter 1
Introduction

1.1. Study Background

Today the demand for miniature devices, passive or battery less and wireless sensors which can be easily integrated in telemetric systems for continuous monitoring and recording of physiological data or in other words, smart sensors, is ever increasing. RF resonant circuits are very versatile; they are passive circuits, require no power supply, simple and compact structures and can be fabricated with simple and inexpensive techniques. Hence, they are very good candidates for sensing in a variety of applications. Passive LC resonant circuits are especially advantageous; as they have long service life, no wired connections are required, can be used in any kind of harsh environments. Additionally, LC sensors can achieve high energy efficient transmission due to lower operating frequency and near- field coupling**.** Previous works have demonstrated the varied applications of LC sensors in so many engineering and technology fields. Biomedical field have utilized the LC resonant sensors as implantable devices for in vivo monitoring of various physiological conditions pertaining to disease state, such as monitoring intraocular or gastrointestinal pressure [1, 2, 3, 4] or permanent or bio-degradable sensors for monitoring heart condition of chronic or acute disease [5]. Cancerous tumor detection is another medical field which have been explored, a study of tissue characterization and differentiation based on the contrast in the electrical properties between tissue in healthy and diseased state was presented [6]. LC sensors have also been applied as environmental monitoring sensors as demonstrated by [7]. Measuring pressure at high temperatures, this application has been developed by a number of studies, one such example is [8] where wireless passive LC pressure sensor based on low-temperature co-fired ceramic (LTCC) technology with high sensitivity was demonstrated. Another example of application of LC sensors for temperature measurement in harsh environments was demonstrated [9], where the sensor was realized using LTCC technology and ferroelectric ceramic was used as the dielectric which has temperature sensitive permittivity. An interesting application where LC sensors are applied is humidity monitoring for wide range of fields from food packaging to building construction materials [10, 11]. Here, a humidity sensitive material, such as polyimide is used a dielectric layer for the capacitor. Another interesting thing to note here is that the dielectric constant of water is much higher than the dielectric constant of the test materials, this contrast leads to high sensitivity of measurement.

1.2. Purpose of Research

Radio Frequency (RF) technologies based sensing is finding ever increasing applications in various fields. One major reason behind this is that RF or microwaves can reveal unique properties of materials. These are reflected by measurement of complex permittivity or dielectric constant which gives an insight of the composition or concentration of the constituents of the material under test. Dielectric spectroscopy (DS) is the branch of RF engineering which deals with material characterization, or in other words, studying the molecular arrangement and physical structure of the test material and how these molecules behave in this arrangement. DS is a well-established technique and has been applied to so many fields like polymers, nanoparticles, colloidal sciences, bio-medical and pharmaceuticals, to name a few. DS measures the dielectric properties of the medium as a function of frequency and is based on the interaction between the electric charge entities in the medium and the externally applied electric field.

It is important to look at the varied applications of DS to see the importance of this technique and fully understand its features and extend the scope to newer applications. It is interesting to see that how DS has proved useful for so many different applications. The agricultural applications of DS have been reviewed by Nelson [12] which include work about the dielectric property measurement of specific rice and wheat crops for evaluating selective dielectric heating of insects. Dielectric measurements on fresh fruits and vegetables for the purpose of determining their maturity index was also demonstrated. Not only DS can be used in detecting biomolecules, like Lee et al demonstrated label-free detection of stress markers [13], but DS also help in understanding molecular dynamics of these bio-molecules [14].

In this work the feasibility of application of LC resonant circuits as sensor to various engineering fields requiring smart sensors are explored. Although two very different fields of application are explored, the underlying detection mechanism remain the same; electrical properties monitored by the RF sensor. The LC sensor is used to detect the dielectric property change in the test environment or the test sample as a function of its resonant frequency. The changes in dielectric constant are directly related to the sensing quantity being monitored. The capacitor measures the dielectric property change. The inductor windings serve two purposes, to form a resonant circuit with the capacitor and to magnetically couple with an external loop. Via mutual inductive coupling, wireless measurement can be performed. The applications include soil moisture sensing in smart farms or plant factories and on-chip Polymerase Chain Reaction (PCR) for point-of care devices in the bio-medical field.

In case of moisture sensing application, the scope lies in the fact that the field of soil moistures sensing is dominated by invasive probe type wired sensors requiring direct power supply. Although these work on the principle of low-frequency dielectric spectroscopy, but certainly the above limitations lead to scope of improvement. The presented sensor is miniature and developed to be embedded in the plant growth substrate in the root zone area at the beginning of crop cycle, thus eliminating the need for regular invasive measurements. It works in inductive coupling with the resonator sensor, located outside of the substrate. The changes in the moisture contents at the root zone leads to change in the capacitance of the embedded resonator and hence its resonant frequency. This information is transferred to the outside interrogating resonator and hence moisture contents can be monitored remotely. The details of experiment and analysis are presented in following chapter.

The second application is towards on-chip PCR; the field is well worked on in terms of achieving all PCR process steps on miniature chip. Many researchers have demonstrated the same, however the detection process is still largely optical and not label-free in many cases. This issue is being addressed in this work, to present a simple and label-free resonant sensor. Also, this application requires the sensor to process small volumes of test samples. For this a miniature resonant circuit with well structure over the capacitor region, and with a protective coating over the circuit tracks, is developed. Various concentrations of different sized PCR products are tested, and the detection range is determined. The detection principle being the same as in the previous case. The details of experiment and analysis are presented in following chapter.

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Chapter 2

Complex Permittivity: Study and Measurement

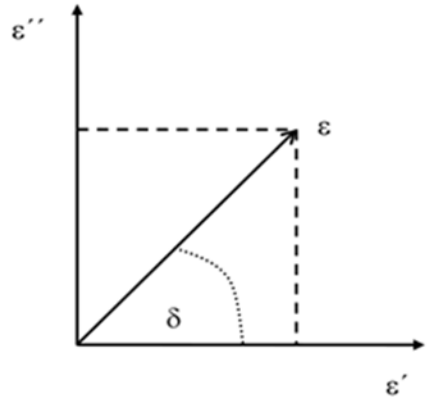
2.1. Complex Permittivity

The term permittivity in electromagnetism is associated with dielectrics, a material which can be polarized by external applied electric field. The charges are displaced slightly according to the direction of the applied field. Permittivity represents the ability of any dielectric to store electric in terms of polarization of the medium. Lower permittivity means a lower ability to store charge and a higher ability to store charge for higher permittivity. The permittivity of vacuum or free space is the lowest possible and is a universal constant of value 8.85 x 10^{-12} F/m represented as ε_0. The permittivity of all other dielectric materials are represented relative to that of the vacuum or free space, ε_r called relative permittivity also referred to as dielectric constant [1]. The permittivity or dielectric constant is a complex quantity represented below and is dependent on the angular frequency, ω.

$$\varepsilon(\omega) = \varepsilon'(\omega) + \varepsilon''(\omega)$$

Where, ε' is the real part of permittivity and represents the stored charge in the medium with applied field and the imaginary part

ε'' is the dielectric loss and represents the energy dissipated in the polarization of the medium. Following is the complex plane representation of the permittivity. Alternatively, the dielectric loss can also be represented by the loss tangent tan as shown below.



$$\tan\delta = \frac{\varepsilon''}{\varepsilon'}$$

Figure 2.1 Complex dielectric permittivity

As mentioned above the response of dielectrics to external field is frequency dependent and it takes finite time for the medium to respond to the applied, represented by relaxation time. Thus, depending on the frequency range different polarization mechanism in the medium comes to play, as shown in figure below [2].

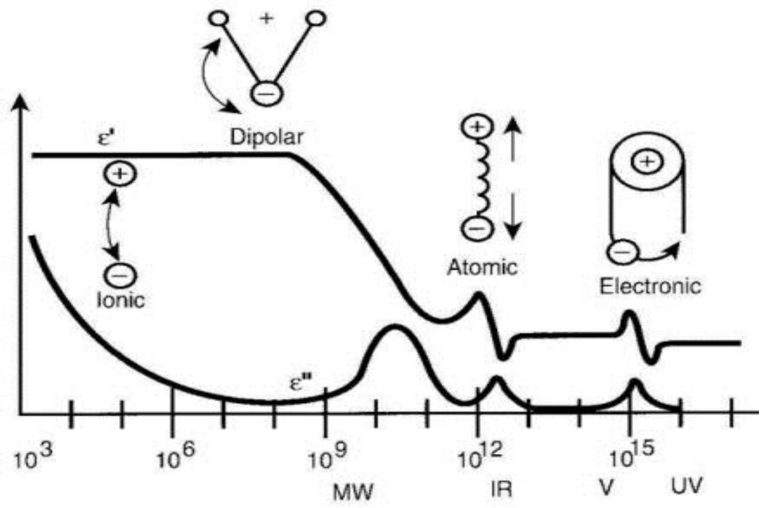


Figure 2.2 Frequency response of dielectric mechanisms

Ionic polarization occurs at lower microwave frequencies and is due to ionic conduction and space charge polarization. Dipolar polarization is due to the rotation of internal dipoles, which are randomly oriented and have no net polarization, rotate to align with external field. As the frequency of applied field is increased, the above dielectric responses are too slow to follow the changing field. Atomic polarization is due to stretching of the bond between neighboring positive and negative atoms in the field direction. Electronic polarization occurs within an atom when the electric field displaces the electron cloud surrounding the nucleus.

Dielectric spectroscopy (DS) is the branch of RF engineering which deals with material characterization, or in other words, studying the molecular arrangement and physical structure of the test material and how these molecules behave in this arrangement. DS is a well-established technique and has been applied to so many fields like polymers, nanoparticles, colloidal sciences, bio-medical and pharmaceuticals, to name a few. DS measures the dielectric properties of the medium as a function of frequency and is based on the interaction between the electric charge entities in the medium and the externally applied electric field.

For developing a permittivity based sensor it is important to have knowledge of the dielectric properties of the target material. For this purpose, dielectric spectroscopy measurements were performed for the case of both applications under study. Material preparation, measurement technique and DS data are discussed in following sections.

2.2 Dielectric properties of heterogeneous medium

The concepts of permittivity seem fairly easy to apply when homogeneous medium are the material under test. However, when considering heterogeneous systems, the dynamics of the medium changes and the contribution to dielectric response due individual constituents and due to the dielectric boundary layer at the inter phase of these constituents need to be considered. This type of systems is represented by Maxwell- Wagner effect and a number of studies have been performed to characterize these. One such model was developed by Koji Asami [3], where the effective dielectric constant of a two-phase system is related to the dielectric constant of its constituents as below. Where, Ɛ* is the complex effective dielectric constant of medium, Ɛa* is the dielectric constant of the solvent and Ɛp* is the dielectric constant of the solute. The materials under investigation in the application this work can be represented by this model and theoretical analysis of this model give good deal of information of what can be predicted of the experimental results or to verify them, as seen in the following chapters

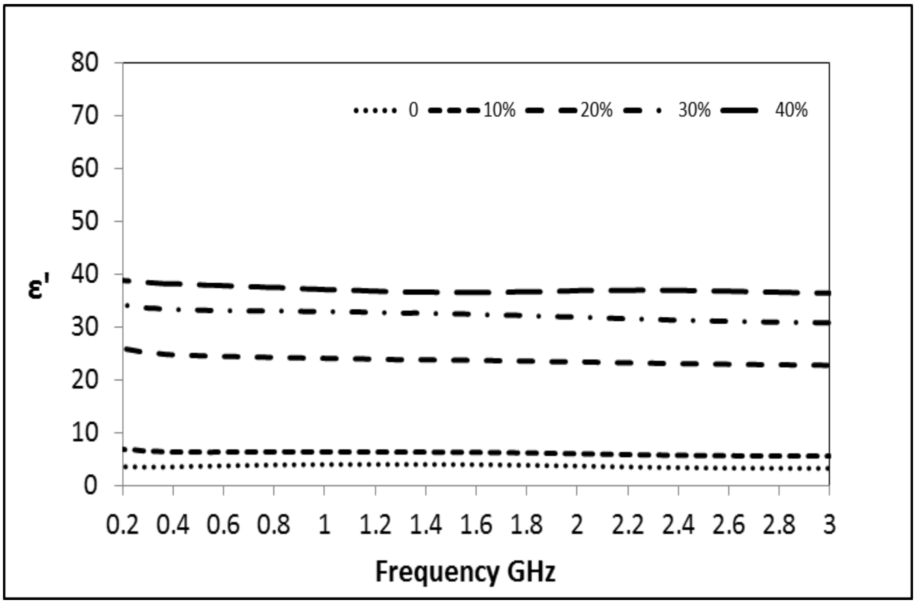
$$\frac{\varepsilon^* - \varepsilon_a{}^*}{\varepsilon^* + 2\varepsilon_a{}^*} = \Phi \frac{\varepsilon^* - \varepsilon_p{}^*}{\varepsilon^* + 2\varepsilon_p{}^*}$$

2.3. Dielectric properties of plant growth substrate

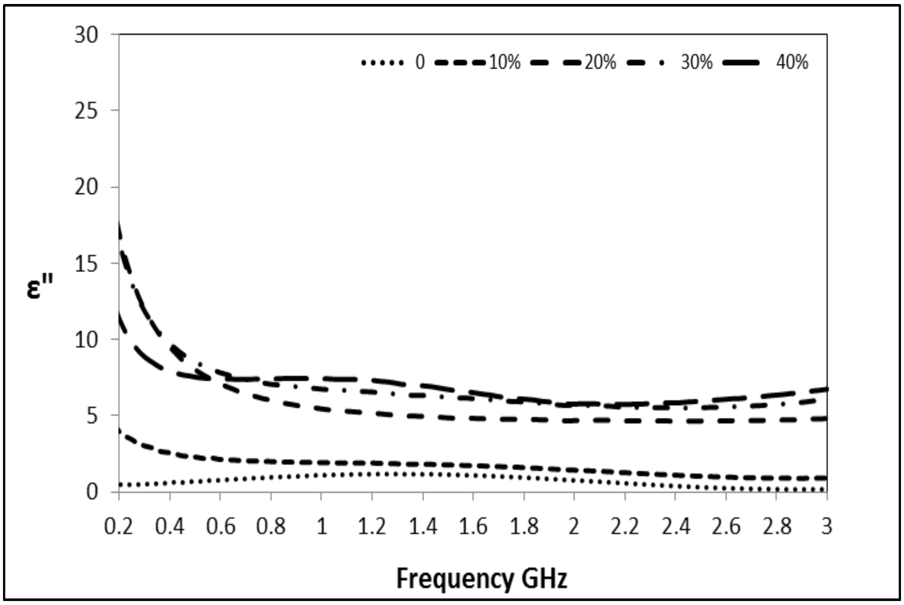
The background for the dielectric measurement lies in the study and measurement of soil moisture which has been performed by a number of researches. The principal work has been carried out by Topp et al [4], where a detailed analysis of DS for a number different kind of soils have been performed. The widely recognized conclusions from this work include, the equivalent dielectric constant of wet soils strongly dependent on its water content, the dielectric constant is almost independent on the soil density, texture and salt content and also temperature. An empirical relation between dielectric constant and volumetric water contents was developed and verified with experimental data. In the present work, instead of soil commercially available plant growth substrate was used due its wide usage in greenhouses as thy many advantages. Coconut peat based commercially available horticulture soil (zeolite 5%, pearlite 8%, vermiculite 12%, coco peat 66.6%, fertilizer 0.4%) has been used as the test medium, henceforth referred to as substrate. Vast studies have been carried out to investigate coco peat based substrates on the growth of a number different vegetables, fruits and flowering plants. These substrates not only have suitable, physical, chemical and biological properties, they were also found to have higher productivity for smaller irrigation amounts, suppressing plant disease, besides being environment friendly and even helping environmental curation by removal of anthropogenic chemical pollutants [5, 6, 7, 8, 9]. However, it suffers from the limitation of low aeration, which affects oxygen diffusion in the medium. Studies show that adding other medium like perlite, zeolite not only significantly improved the chemical physical properties of the growing medium like air-filled porosity, electrical conductivity and ph, but also improved nutrients uptake in plant and reducing environment pollution and can also be recycled for many crop seasons, thus reducing production cost [10, 11].

Dielectric properties of this substrate were measured for different water content, to understand and estimate the response to radio signal prorogation through it. The measurement setup consisted of Keysight dielectric probe connected to Keysight impedance analyzer [12] at the EMTI lab, Korea Radio Promotion Association, Seoul [13]. Test sample were prepared for different gravimetric water contents, mass of water per mass of dry soil (W/W). Substrate moisture content was varied from dry state (0% W/W) to fully saturated state, which in this case is 50% W/W, in steps of 10% W/W.

Figure 2.3 Measurement setup for complex dielectric properties measurement of the coco peat agriculture substrate.



(a)



(b)

Figure 2.4 Measurement of dielectric properties of the coco peat based agriculture substrate. (a) real part ε′ and (b) imaginary part ε″ of the complex dielectric constant for different water contents in the substrate

The complex dielectric properties were measured over a range of 100MHz to 3GHz, as shown in figure 2.4. Both the real and imaginary components of dielectric constant do not change much from 500 MHz onwards. Also, there is significant change in dielectric constant value form dry state of substrate, 0% w/w when permittivity is around 2 to fully saturated state, 50% w/w, when it is almost 58. However, the permittivity does increase linearly with linear increase in substrate water content. This measurement data will be used in later chapter to design and apply LC sensor to moisture sensing application.

2.4 Dielectric properties of Deoxyribonucleic acid molecule in aqueous solution

On-chip polymerase chain reaction (PCR) detection or in other words, Deoxyribonucleic acid (DNA) detection is another application that explored in this work, as DNA is the product of PCR. DNA is an important molecule as it is the blueprint of all life on earth. Every aspect and characteristic of everything living is determined by the arrangement of DNA molecules in the cells. PCR is an amplification technique for cloning specific parts of DNA sequence of interest, thus it holds special interest in study and detection of DNA. Another reason is that usually in almost all applications of DNA, the target DNA available is in very minute quantities, so it is important to amplify the amount of target

DNA present. DNA molecules have interesting structure and dielectric properties which have been studied by so many researches and different phenomenon has been observed in these DS studies based on the range of frequency or length of DNA chains under test. Besides, the sample preparation and the process of PCR is in itself a technique which requires detailed preparation and experimentation. Thus dielectric property measurement for DNA will be discussed in following chapter along with PCR sample preparation and experimentation protocol and results.

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Chapter 3

LC Resonators

3.1. The LC Resonators

The LC resonant circuit is a very well-known resonant circuit comprising of an inductor and a capacitor. In a series arrangement, the resonance is achieved at the particular frequency where the inductive and capacitive reactance are equal to each other and this resonant frequency f_0 is represented as

$$f_0 = \frac{\omega_0}{2\pi} = \frac{1}{2\pi\sqrt{LC}}$$

Where, L and C are the inductance and capacitance respectively. At $f_{0,}$ the current is maximum and the circuit impedance is minimum. For frequencies below $f_{0,}$ the circuit is capacitive and frequencies above f_0, the circuit is inductive.

The LC resonator has some very interesting characteristics which makes it very attractive to use present smart sensing applications. Consider the capacitor part, if all other elements in the circuit remain same, the resonant frequency reflects the change in capacitance alone, which in turn reflects the changes in the dielectric environment surrounding the capacitor, as shown in figure below. Thus, the capacitor measures the dielectric property change. The inductor windings serve two purposes, to form a resonant circuit with the capacitor and to magnetically couple with an external loop.

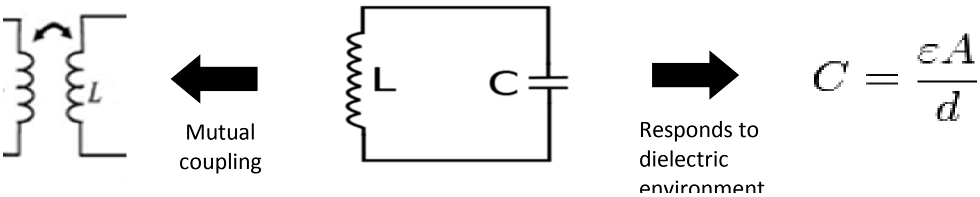

Figure 3.1 Functions of the elements of LC resonator in smart sensing

3.2 Circuit element design

The LC resonator at radio frequency for present sensing application is realized with planar spiral inductor and planar interdigitated capacitor. The design crieria are well establised by previous works. Using the design equation presented by S. Mohan et al [1], the spiral can be designed to desired operating frequency and size requirements. This design formula is based on a modified Wheeler formula [2].

$$L_{mw} = K_1 \mu_0 \frac{n^2 d_{\text{avg}}}{1 + K_2 \rho}$$

Where, ρ is the fill ratio defined as $(d_{out} - d_{in})/(d_{out} + d_{in})$ and $d_{avg} = 0.5*(d_{out} + d_{in})$, d_{out} and d_{in} being the outer and inner diameter of the spiral. K_1 and K_2 are layout dependent coefficient and were determined as 2,34 and 2.75, respectively for square spiral geometry.

For the design of interdigitated capacitor, the expression used is shown below [3], here A is the square area and d is the separation between any two fingers, the nits of measurement being microns. N is the number of fingers and ε_r is the relative dielectric constant of the substrate used for fabrication of the circuit elements.

$$C = 0.2249 \frac{\epsilon_r A (n-1)}{d} \qquad \text{(pF)}$$

The design and development of the LC resonator is performed with the help of simulation tool high frequency structure simulator HFSS [4]. With the design frequency of around 900 MHz, the circuit is finally optimized to the following dimensions. The reason for choice of such an operating frequency is based on the regulations by the International Telecommunication Union which regulates the use of frequency bands in radio spectrum. Where all other bands have specific and licensed applications, the industrial, scientific and medical (ISM) band is allowed to be used for unlicensed operation and depending on the country, it is allocated around the frequency of 1 GHz [5].

The final circuit elements are realized a follows. The spiral inductor has 1.5 turns, maximum outer dimension is 4200 microns and the width and the spacing between conductor tracks is 100 microns. The effective inductance is 23.4 nH. By HFSS simulation it is also verified that the designed inductor has stable inductive reactance around the design frequency of 1 GHz, as is evident from the plot of complex admittance as function of frequency, as shown in figure below.

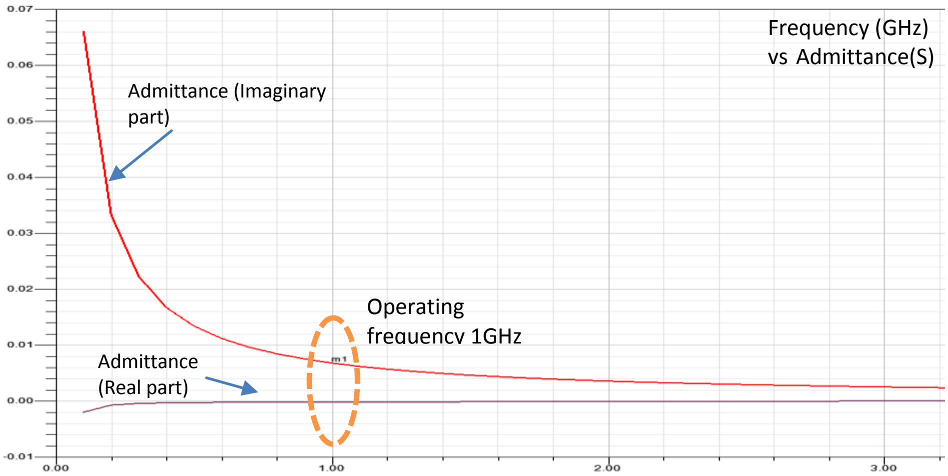

Figure 3.2 Simulated performance of spiral inductor

In the same fashion the interdigitated capacitor is designed to with 16 number of fingers, width and spacing between conductor track is 100 microns and the effective capacitance is 1 pF at design frequency. Again, by HFSS simulation it is also verified that the designed capacitor has stable inductive reactance around the design frequency of 1 GHz, as is evident from the plot of complex admittance as function of frequency, as shown in figure below

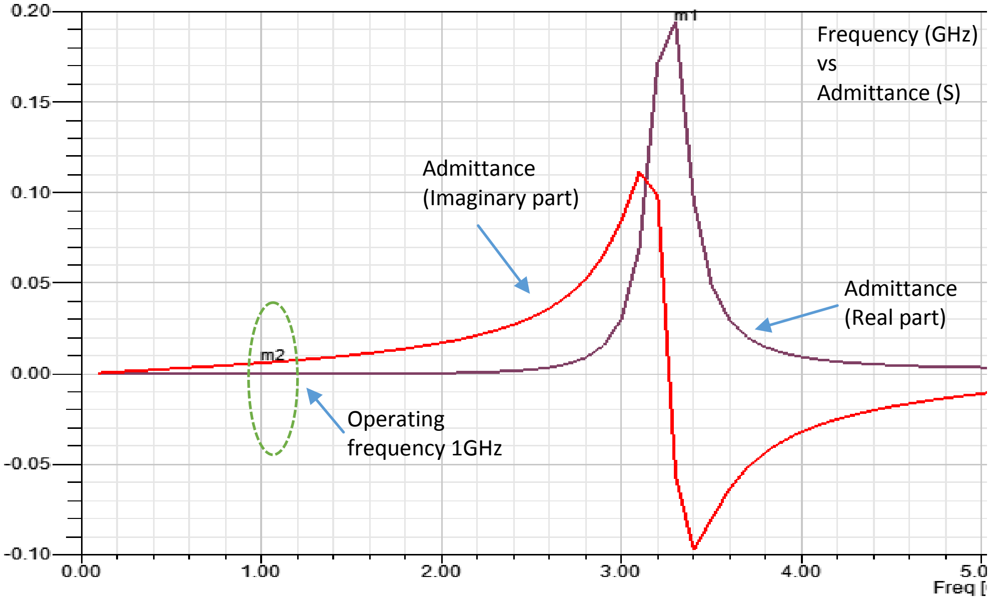

Figure 3.3 Simulated performance of interdigitated capacitor

The substrate used for the design of the LC resonator is Rogers RT/duroid 5880 [6] with specifications as follows, relative dielectric constant of substrate 2.2, thickness of substrate 30 mils. Copper sheet thickness on both sides is 17 microns

3.3 Equivalent circuit of LC

In this section the equivalent circuit is developed and circuit parameters are obtained. The purpose is to verify the response of the designed circuit and also to evaluate the LC resonator in the test environment when developing it as a sensor for different sensing applications. Because any distributed element like the spiral inductor or the interdigitated capacitor are not purely inductive or capacitive, it is important to check its equivalent circuit performance as well. For example, spiral inductor is dominantly inductive in the range of operating frequency but it also has resistive and capacitive parasitic element [3] as shown below. Here, L is equivalent inductance of the spiral circuit and the rest of the elements are parasitic. This equivalent circuit should be modified as per the spiral inductor in the specific application, for example, in present work the resonator does not have any ground plane, thus the parasitic elements C_1 and C_2 can be ignored.

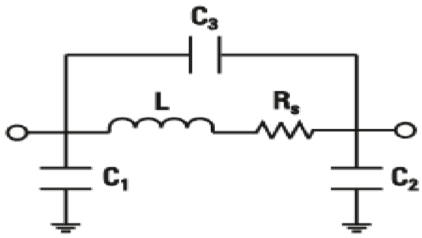

Figure 3.4 Circuit element equivalent of spiral inductor

The equivalent circuit of the 1 GHz LC sensor is developed as below. The software tool to design this circuit is Advanced Design system (ADS) [7]. The performance of this circuit is simulated and compared with circuit designed previously using HFSS

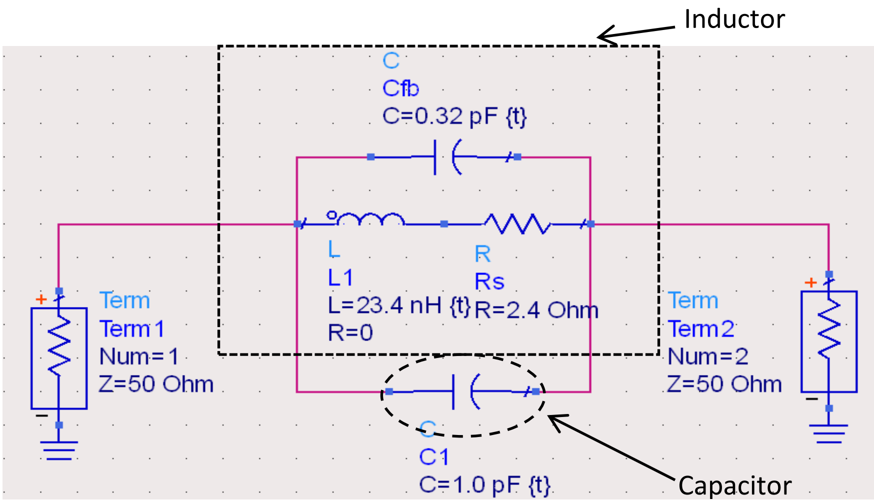

Figure 3.5 Circuit element equivalent of LC resonator

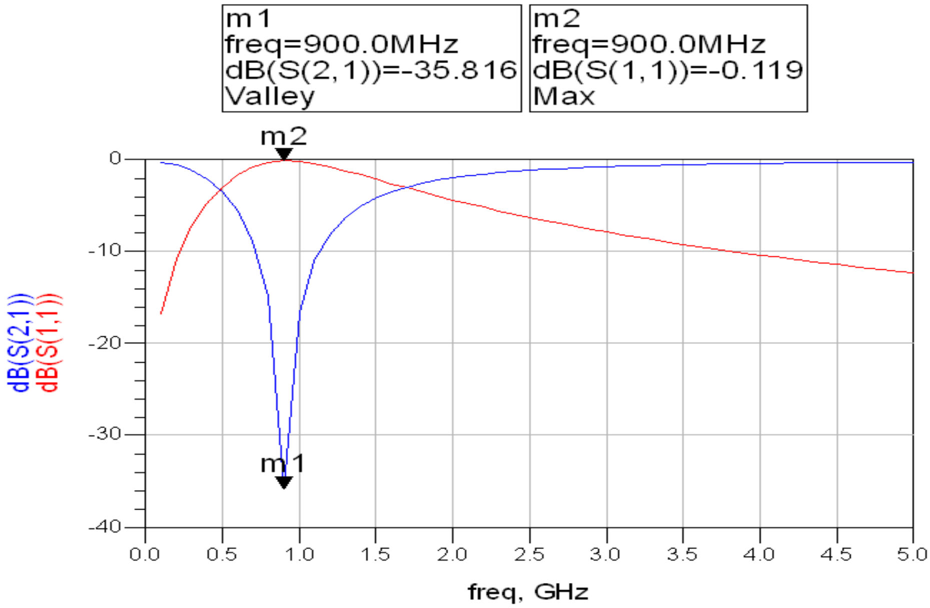

Figure 3.6 Resonant Circuit of LC resonator from equivalent circuit

The resonant behavior is shown by the plot of two port Scattering parameters and the resonant frequency from equivalent circuit analysis is close to the resonant frequency from structure analysis. Thus this kind of equivalent circuit can be used to analyze the physical sensor for any specific test condition.

Reference

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[2] H. A. Wheeler, “Simple inductance formulas for radio coils”, Proceedings of the Institute of Radio Engineers, Oct. 1928, Vol. 16, No. 10, pp. 1398–1400

[3] I. Bhal, “Lumped elements for RF and microwave circuits”, ARTECH HOUSE

[4]https://www.ansys.com (courtesy department of Electrical Engineering, Seoul National University)

[5]http://life.itu.int/radioclub/rr/art1.pdf

[6]https://www.rogerscorp.com/acs/products/32/RT-duroid-5880-Laminates.aspx

[7]https://www.keysight.com (courtesy Applied Electromagnetic Laboratory, Seoul National University)

Chapter 4.

Embedded LC Sensor for moisture detection

4.1. Introduction

Soil moisture content is a critical parameter in many applications of soil mass including agronomy. Needless to mention, a key factor in the life of plants, water is a large part of plant tissue; it is required for photosynthesis and nutrient transport. Right moisture conditions help in better development of plant root system, but excessive moisture could lead to growth of harmful pathogens.

A wide variety of techniques has been applied towards soil moisture measurement from ground penetrating radar, neutron scattering, gamma rays, soil resistivity sensing, optical techniques, heat flux sensors and dielectric property of soil based techniques [1, 2]. In spite of this huge range of technologies available soil moisture sensing has been largely developed as a proximal technology, revolving around dielectric techniques, namely time domain reflectometry sensors (TDR) and frequency domain reflectometry (FDR).

TDR use precise electrical pulses in the frequency range of radio spectrum which travels along transmission lines and waveguides. These are inserted inside the soil and the impedance along these is a function of the bulk dielectric constant of the soil. The dielectric constant is measured by the time it takes for the pulse to travel along the waveguide path. The dielectric constant is in-turn a function of the water content in soil. Topp et al. [3] proposed an empirical relationship between dielectric constant and volumetric water content of soils with several textures. In case of FDR sensing, the sensor consists of two or more capacitors, usually rod shaped, which are inserted in the soil. These capacitors use soil as dielectric and thus the response is reflective of soil moisture content. TDR based sensing suffers from disadvantage of bulky equipment and high initial cost of setup [4]. FDR sensors are invasive, where a few centimeters long and thick needles are inserted into the soil bed [5] and local soil moisture can be determined. But this invasiveness can hamper the accuracy of measuring moisture content in the actual root zone area of the soil. Besides some plants, for example ornamental crops, require very specific conditions to thrive, any smallest changes in these might disturb their growth. Finally, FDR techniques are also not economical for multiple site measurement [6].

The thing to be noted, sensing based on the bulk dielectric properties of soil in the radio frequency spectrum is very advantageous as the relative dielectric constant of water is 80 and that of any general soil sample is in the range of 2-4 [7]. Another advantage of working in the radio frequency spectrum, the response is mainly dependent on the moistures content and not so much on other soil-specific properties like salinity, texture, temperature etc.

4.2 LC sensing approach

In this work a new approach towards soil moisture sensing is proposed for accurately monitoring the water contents in the root zone area of plants. The embedded resonator sensor whose resonant frequency shifts based on the surrounding soil moisture contents, thus it can monitor plant root zone water. Via magnetic coupling, the sensor transmits this information wirelessly to an outside coupling resonator. This detection system is pictorially depicted below.

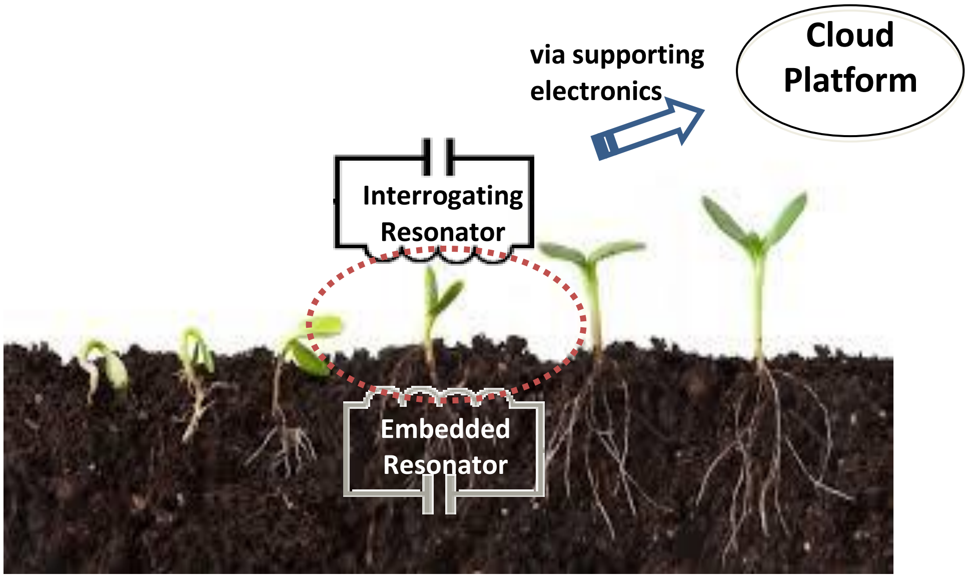


Figure 4.1 Proposed sensing approach using coupled resonators

The dielectric properties of the plant growth substrate were measured using dielectric spectroscopy in previous chapter, they are shown below. It was observed that dielectric constant is independent of frequency in the range of frequencies measured. Thus the choice of operating frequency for the LC resonator is very flexible. Keeping design parameters in mind and opting for a reasonable size for practicality and ease of fabrication, The LC resonator is designed to resonate at 500 MHz.

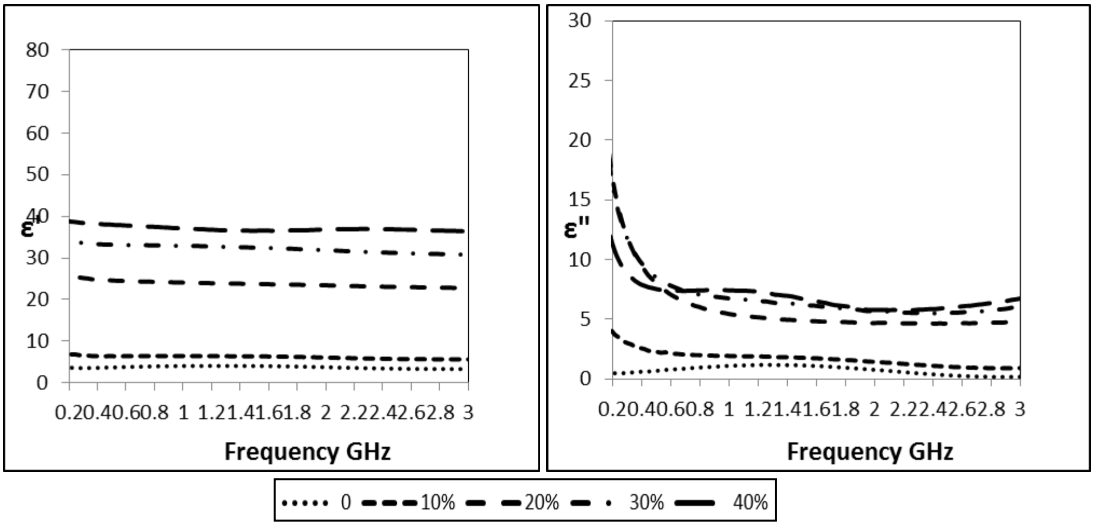


Figure 4.2 Dielectric spectroscopy of plant substrate for different moisture contents

The design process has been discussed in chapter 3 and for this application the design frequency is selected as 500 MHz. The LC resonator is designed with spiral inductor 1.5 turns, maximum outer diameter of 1300 millimeters (mm), the interdigitated capacitor has 16 fingers of length 9mm and for both of these the width of copper track and spacing between them is 300 microns. There is no ground plane and the substrate is same, Rogers RT/duroid 5880, which has glass reinforced

Polytetrafluoroethylene substrate sandwiched between two copper sheets of thickness 17 microns.

4.3 Modelling embedded sensor performance

For modelling the performance of the embedded sensor in real test conditions, the LC is simulated in the dielectric environment of substrate with different gravimetric moisture content, from dry state to fully saturated one. This varying dielectric environment is provided with dielectric information of these from the measurement data to HFSS [8]. LC is embedded in the middle of 10 cubic centimeter plant substrate. Following is the result of simulation analysis plotted as resonant frequency response of LC sensor to different moisture content of substrate.

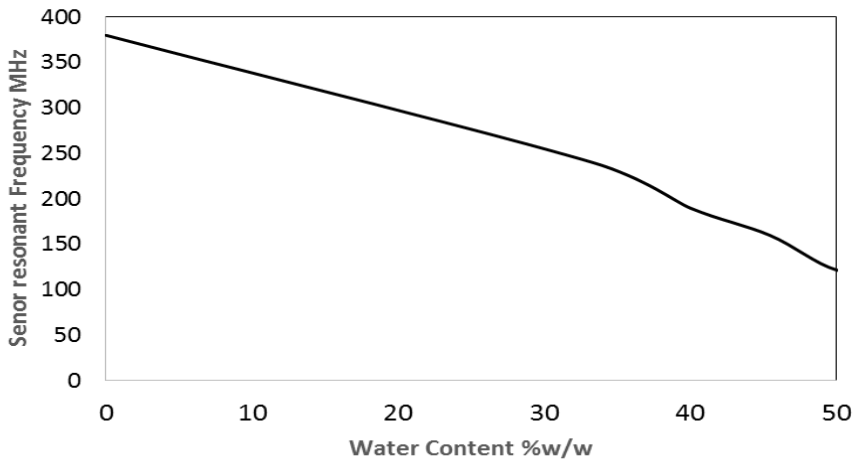


Figure 4.3 Embedded LC sensor resonant frequency in response to substrate moisture

It can be seen that resonant frequency decreases linearly in response to linear increase in gravimetric moisture content. Based on this response, it is clear that LC sensor is responding to changes in substrate moisture content only. However, there could be losses induced due water molecules to electromagnetic wave when mutual coupling link is used to couple embedded LC to outside interrogator LC. Based on this assumption the following equivalent circuit is proposed for the embedded LC, where capacitive and resistive elements represent the varying moisture dielectric environment.

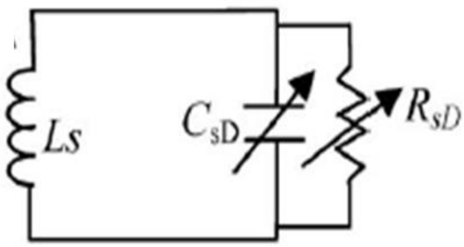


Figure 4.4 Proposed equivalent circuit of embedded LC

To verify this assumption for equivalent circuit is equivalent circuit parameters are extracted and analyzed with the help of ADS tool. The equivalent circuit and corresponding extracted parameters are shown below.

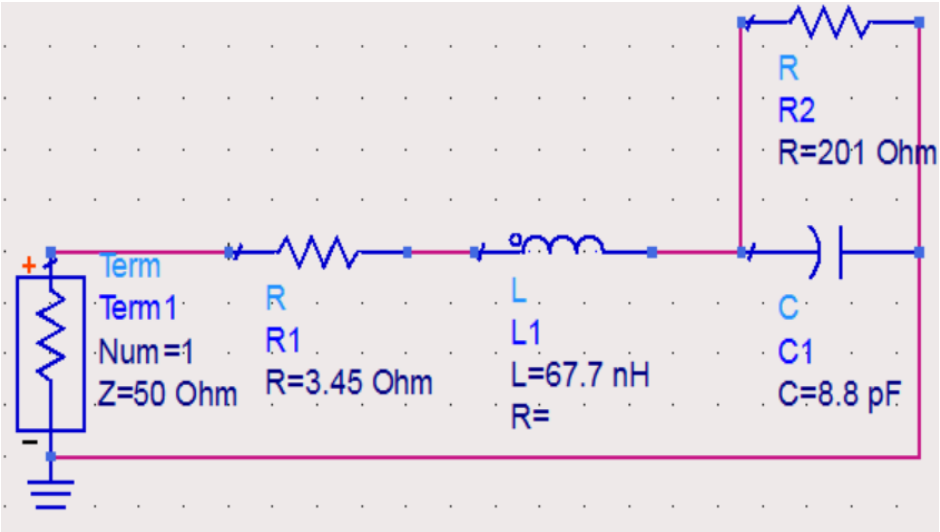
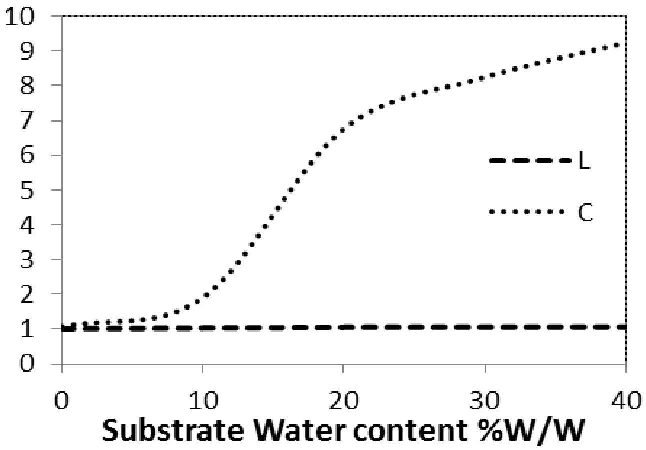


Figure 4.5 Verified equivalent circuit of embedded LC

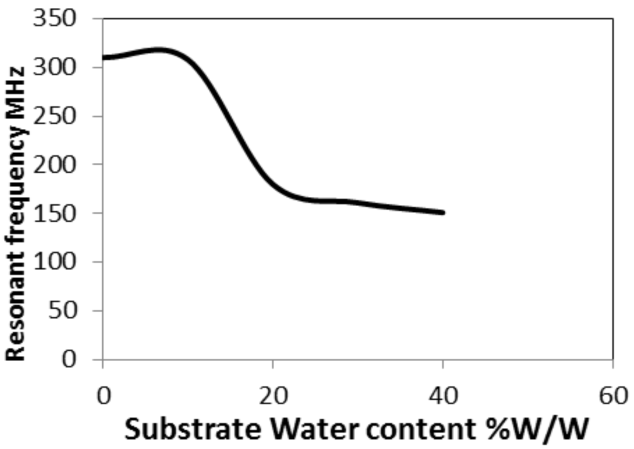
Substrate water contents% w/w	L nH	C pF	R Ohm	Resonant Frequency MHz
0	65	1.4	191.2	310
10	66.5	2.5	197	308
20	67.7	8.86	201	180
30	68.1	10.8	205.6	161
40	68.4	12.1	206.3	151

Table 4.1 Extracted equivalent circuit elements of embedded LC

From the extracted values of equivalent circuit, it is clear that the change in the resonant frequency of LC is chiefly due to the capacitor, which in turn is changing due to the change in dielectric constant of substrate with changing moisture content. As the moisture content is increasing, increase in % W/W, the capacitor value increases, thus decrease in resonant frequency. The resistive loss is present, as proposed earlier, however it does not change significantly for different water content. To summarize, the embedded LC sensor responds well to changing substrate water content and the circuit component responsible for this is response is capacitor.



(a)



(b)

Figure 4.6 (a) Normalized circuit elements L and C, (b) LC response to changing water content

To summarize, figure above shows yet again that inductor does change significantly with changing substrate moisture, only capacitor does and it reflects exactly inversely the resonant frequency curve.

4.4 Modelling interrogating LC performance

For modelling the performance of the interrogating LC sensor in real test conditions, the LC is simulated in the dielectric environment of substrate with different gravimetric moisture content, from dry state to fully saturated one. This varying dielectric environment is provided with dielectric information of these from the measurement data to HFSS. LC is embedded on top of a 10 cubic centimeter plant substrate. In this case, the lossy shunt resistance element is ignored as interrogating LC is in free space, on top of the substrate and not embedded. The equivalent circuit for this case is shown below.

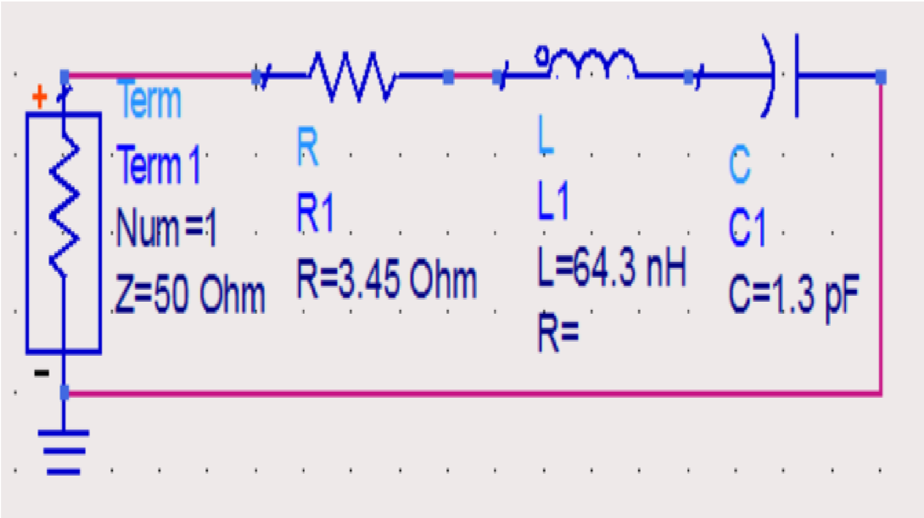


Figure 4.7 Verified equivalent circuit of interrogating LC

Substrate water contents% w/w	L nH	C pF	R Ohm	Resonant Frequency MHz
0	64.9	1.31	3.45	548
10	64.3	1.3	3.45	550
20	64.7	1.31	3.45	547
30	64.7	1.32	3.45	545
40	64.3	1.31	3.45	546

Table 4.2 Extracted equivalent circuit elements of interrogating LC

When the interrogating LC is present alone, there is no mutual coupling element thus, the plant substrate acts only as a substrate to this circuit and the local changes in this substrate near to the circuit are not significant to cause any significant change its resonant frequency, concluding this LC does not perform any sensing application by itself.

4.5 LC sensor fabrication

The sensor fabrication is simple standard micro electro-mechanical system (MEMS) fabrication, it requires single – mask photolithography and wet etching processes. The process flow is show below.

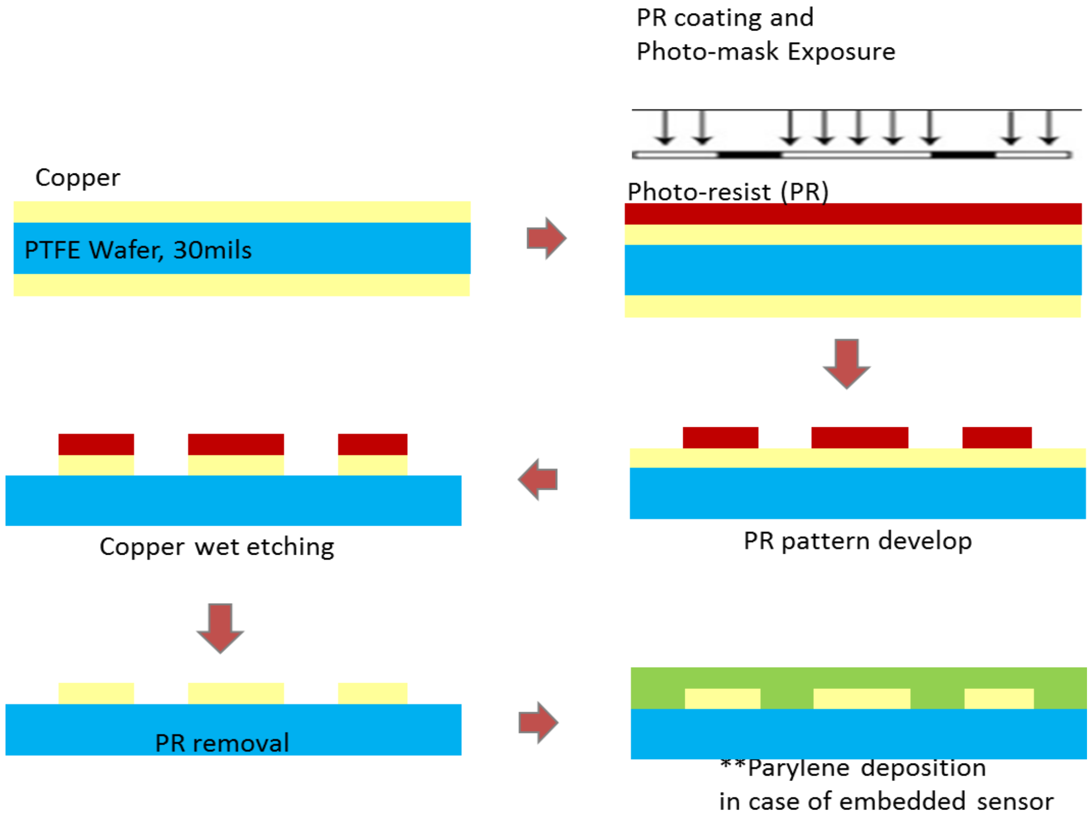


Figure 4.8 process flow for LC fabrication

The fabricated sensors are shown below. The embedded LC is a closed loop series circuit, as it is intended to stay in the plant substrate for entire crop cycle, which could be a couple of months. For this reason, it is coated with a layer of Parylene [9], a durable material which gives good conformal coatings and has low dielectric constant almost same as the circuit substrate. The interrogator LC is one port series circuit as it will be connected to outside measurement equipment to convey the embedded LC sensor's real-time measurement data.

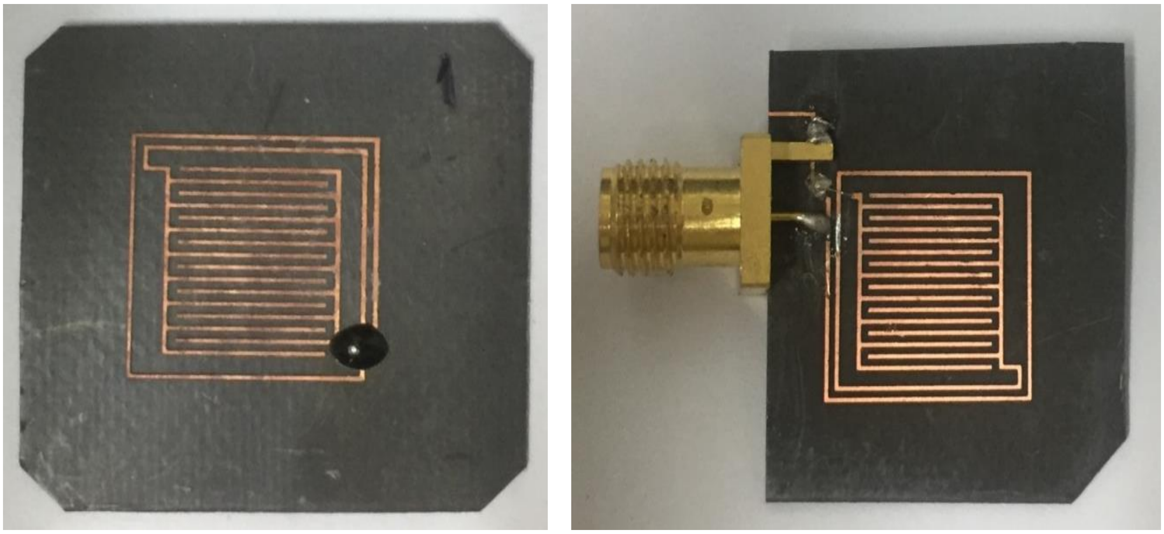


Figure 4.9 Fabricated sensor, left is embedded LC and right is interrogating LC

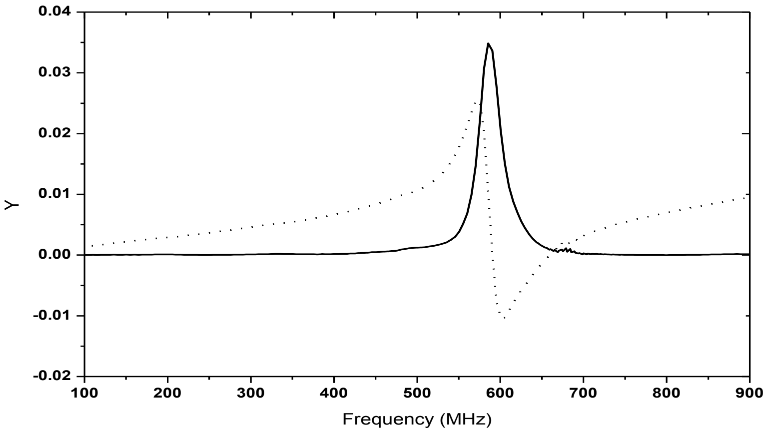
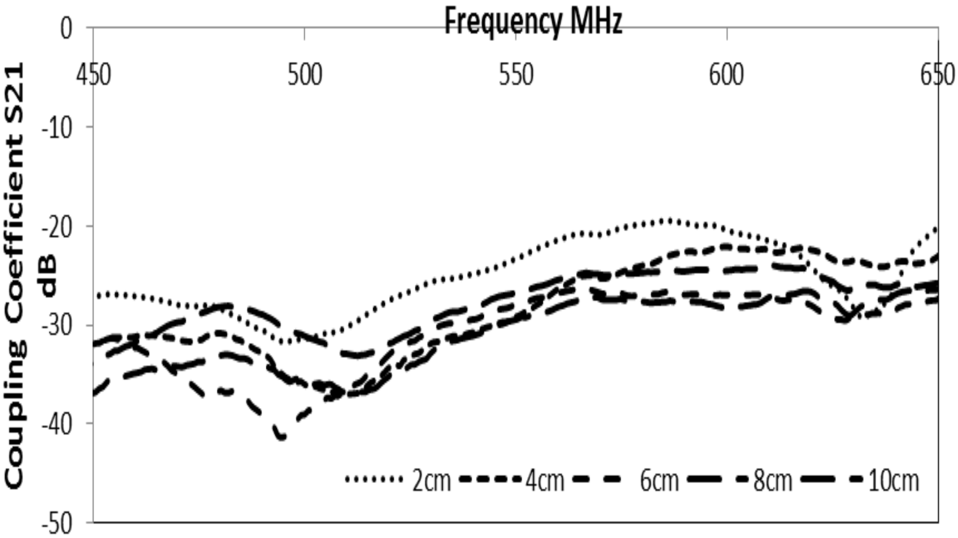


Figure 4.10 Measured [Y] parameter of the fabricated sensor, the peak value represents resonant frequency

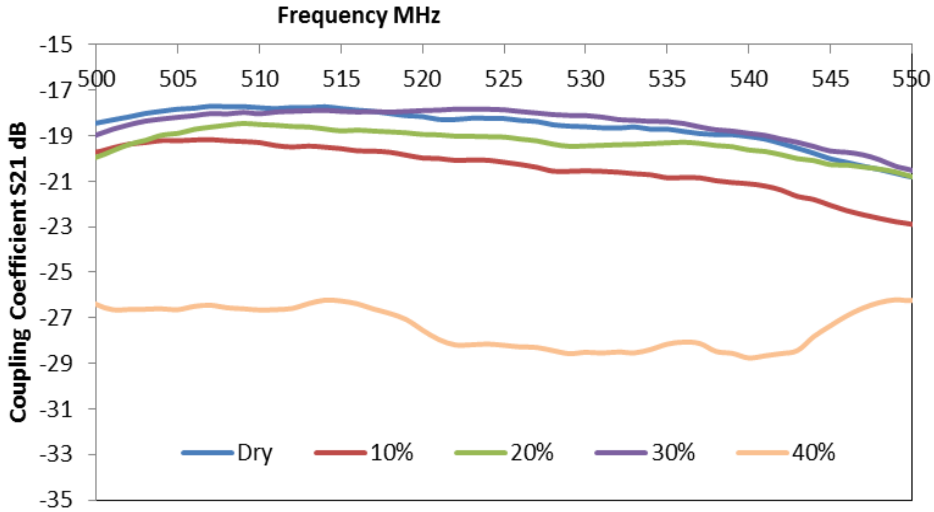
4.6 Mutual coupling

The mutual coupling between the embedded and interrogating LC is verified by measurement in free space as well in the wet plant substrate environment. For maximum coupling, both the LC are oriented exactly same and vertical separation between them is increased in increments. The mutual coupling is represented by the two port scattering or S parameter measurement. All measurements show sufficient amount of coupling, even in the case of wet substrate as the dielectric medium between the two.



(a)

(b) In free space



b) In wet substrate dielectric environment

Figure 4.11 Mutual Coupling between embedded and interrogating LC with varying vertical separation and substrate moisture contents

4.7 LC sensor testing and result

For testing the embedded LC sensing, the LC sensor was coated with thin layer of parylene to protect the conducting tracks from the wet sensing environment, and the wire- bonding forming the closed LC circuit covered with epoxy. This was then embedded in the soil substrate at a depth of 4 cm. The water contents of substrate were varied from 0% w/w to 50 % w/w and the corresponding coupled signal measured with the outside LC connected to network analyzer (NA). As a control case, outside LC resonance was measured when there was no embedded LC present, for different water content.

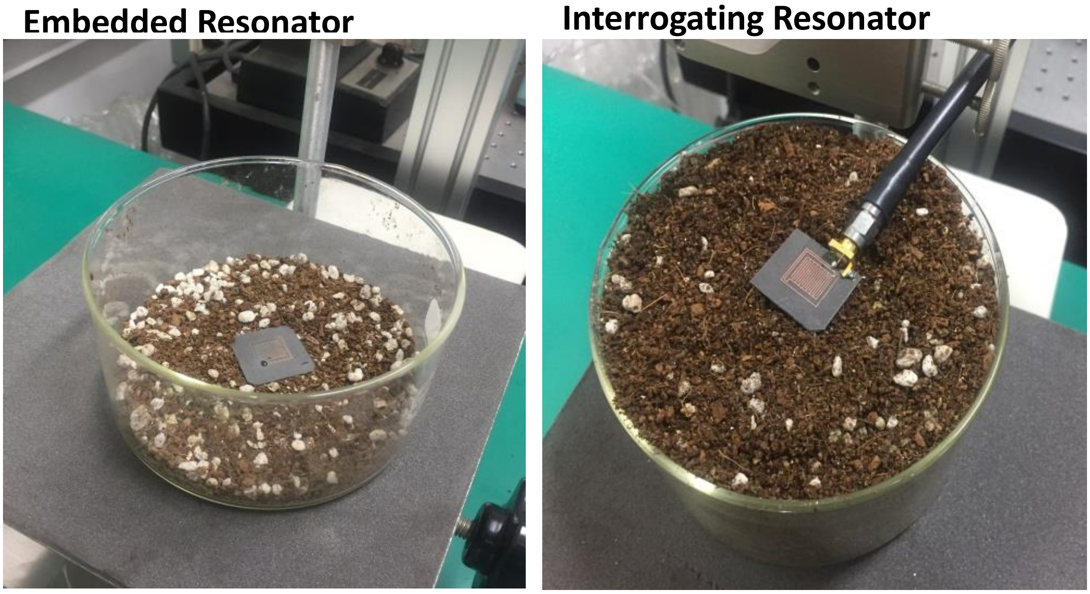
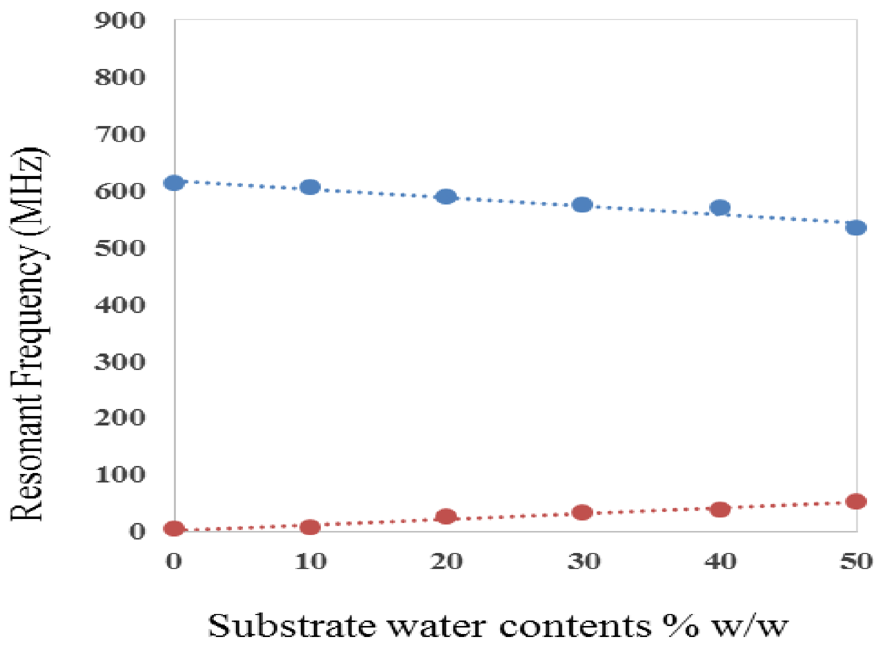
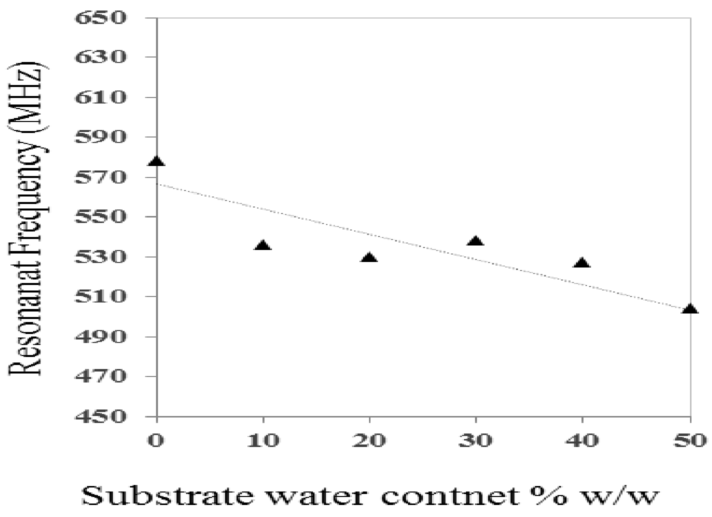


Figure 4.12. Experimental setup for measurement of substrate water contents using embedded LC sensor. Left, embedded LC sensor and right, outside coupling LC resonator connected to NA

The resonant frequency of the outside interrogating LC sensor is acquired by measuring the complex admittance parameters at the input port of the interrogating LC, using single port NA measurement. The figure below shows the measurement data for real test scenario when sensing LC is present and control test case when sensing LC is not present in the substrate. Clearly, as show in fig. 4.13a, a linear increase in the substrate water content, decreases the resonant frequency of the interrogating LC linearly and this is co-related. This shift in resonant frequency is in accordance with the equivalent circuit discussed earlier where the embedded LC sensor and the moist substrate with certain water contents, represents a specific load to the interrogating LC. Hence the water contents in the substrate and measured resonant frequency are correlated. However, for the case of no sensing LC present, the load seen by the interrogating LC is random irrespective of the specific substrate water contents, as seen in fig. 4.13b. This would be due the local water diffusion in the substrate near the LC surface being non-uniform which in turn is because the inhomogeneous nature of this substrate. Hence there is no specific or co-related trend in the measured resonant frequency with increasing water contents of the substrate.



(a)



(b)

Figure 4.13. Measurement Data (a) resonant frequency of outside LC due to shift in embedded LC frequency with changes in moisture content (blue line) and dielectric constant for corresponding case of water content at 600 MHz (b) outside LC resonant frequency when no embedded LC is present

References

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[8] https://www.ansys.com (courtesy department of Electrical Engineering, Seoul National University)

[9] https://www.parylene.com/

Chapter 5

LC sensor for on-chip PCR detection

5.1 Point of Care diagnostics

Point of care or PoC refers to medical diagnostic testing outside clinical laboratories. This facilitates rapid detection of analytes near patient thus leading to better disease diagnosis, monitoring, and management. PoC devices give quick feedback on medical tests. In today's ever-shrinking world, it is very important to rapidly detect and prevent pathogenic infections. Thus PoC is getting lot of attention with many kinds of research going on to develop this field. For this field to achieve its full potential, it's very crucial to be able to detect deoxyribonucleic acid (DNA) molecules because DNA is vital for all living beings. It is important for inheritance, coding for proteins and the genetic instruction guide for life processes. The roadmap for the development and reproduction and death for any organism or cell is with DNA. Identification of the genes that trigger major diseases or the creation and manufacture of drugs to treat these devastating diseases.

5.2 Polymerase Chain Reaction (PCR)

PCR in simple words means copying specific DNA sequences.

The three basic steps in the process are splitting a DNA template into its two single strands (denaturation); adding short segments of complementary DNA called primers to initiate replication of a chosen DNA sequence (annealing); and adding DNA polymerase to synthesize the complementary strand (extension). These steps are repeated serval times to amplify the sequence. The reason PCR is important for DNA detection is that real test samples for PoC devices have trace amounts of DNA which is not enough for any reliable output. PCR process means multiple laboratory functions on a single chip. For PoC applications it means to be able integrate and automate all steps necessary for molecular analysis. To a major extend all this has already been achieved [1]. Another interesting work integrated digital microfluidics with PCR Lab on chip system with picoliter droplets which form the unit for PCR with each droplet holding upto 1 copy of DNA [2] was demonstrated. However, the method used for DNA detection after PCR is fluorescence detection. Another work integrated on-chip PC with capillary electrophoresis [3] but PCR product detection was performed with optical detection system. The scope lies in the detection in real time, label-free using non-optical techniques target DNA after PCR amplification. In this work the focus is on the detection of PCR product and on-chip PCR process have not been attempted.

5.3 PCR Materials and Method

The target DNA sequence (PCR product) is random sequence of lengths 900 base pairs (bp) and 3000bp. Calf Thymus DNA [4] is used as the template. The primers, Forward and Reverse case, for both 900bp and 3000bp designed and supplied by Bioneer Korea. PCR Buffer and Premix (DNA polymerase + nucleotides) also from Bioneer, Korea. PCR Equipment used TaKaRa Gradient Thermal Cycler TP600 [5]. The PCR recipes for both 900 and 3000 bp cases are optimized as below.

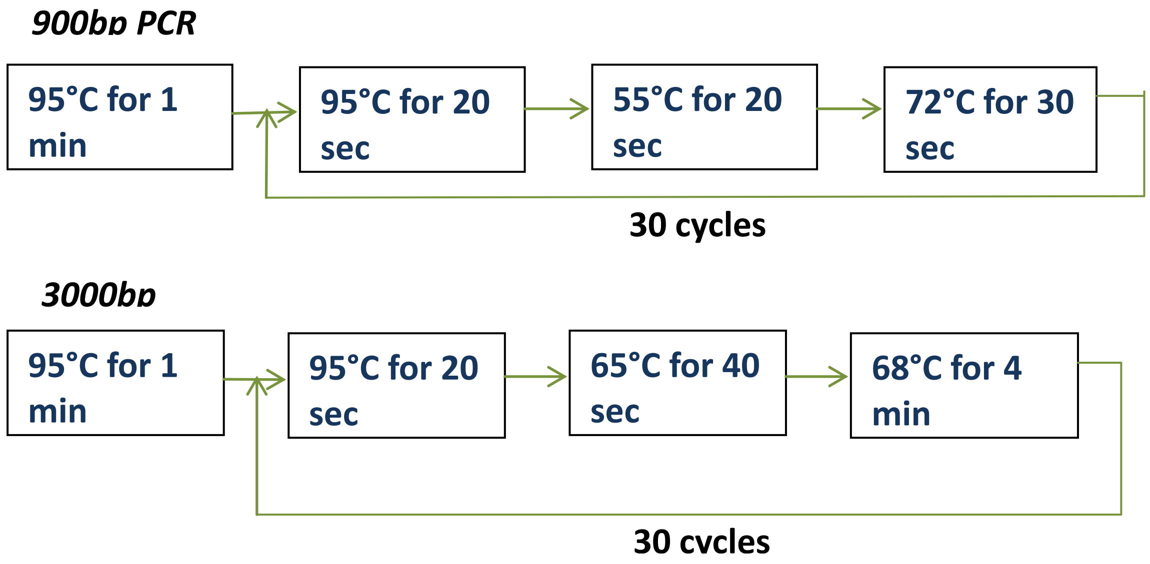
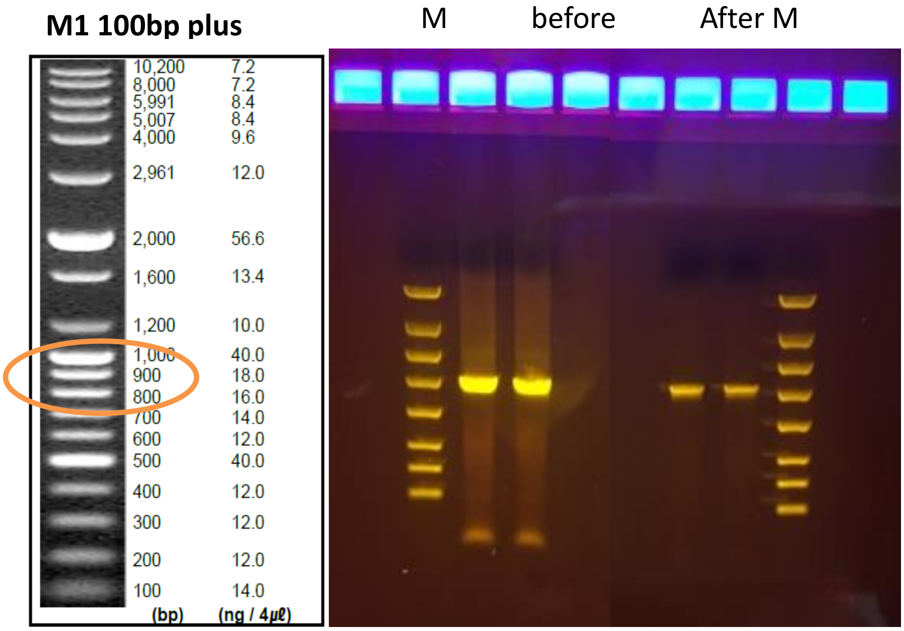


Figure 5.1 PCR recipes for DNA

After PCR is finished the PCR product, the amplified DNA is purified and quantified using gel electrophoresis FlashGel System [6], electrophoresis dock and power supply, gel cassettes, loading dye, marker ladder M1 and M2 (100 – 4k bp). Electrophoresis is performed for 7 minutes at 134 volts. PCR product is also quantified using Nano drop [7] and result shown in table below. These show good result of PCR.



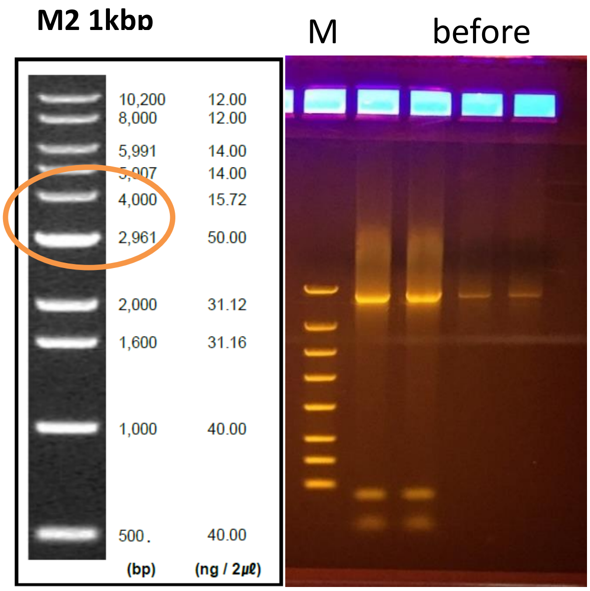


Figure 5.2 Gel electrophoresis for PCR product verification, top for 900 and bottom for 3000 bp case

Case	DNA concentration	Unit
900bp	11	ng/μl
3000bp	12.4	ng/μl

Table 5.1 PCR Product Purification: Nano Drop Result

5.4 LC Sensor for PCR detection

The sensor designed in chapter 3 is suitable for this application as its compact in size. Only thing needs to be taken care is the sample size should be always same on the capacitor part to have reliable measurement data. For this purpose, an Su8 wall is fabricated around the capacitor part of the LC. This bring additional fabrication steps. Firstly, wet etching for low aspect ratio structures which is 17:100 in this case, is difficult. Besides the nature of the substrate requires stress compensation, because the substrate is made by bonding of the substrate with copper at elevated temperature as they have different thermal coefficients of expansion. After etching this leads to a shrinkage of substrate, which means a miss-alignment for the final mask process of Su8 wall fabrication. To compensate this, a two step mask process is used. The second mask process is same as discussed in chapter 3, however the first mask process is used for after-etch stress compensation, where most of copper is etched and the substrate is annealed in nitrogen environment. This not relieves after-etch stress but also helps in achieving good fabrication accuracy. After this Su8 wall structure is fabricated and verified using surface profiler. Another aspect that needs to be considered is that buffer solution for PCR have many ions, these could degrade the copper track of the LC sensor, thus a parylene coating is used again, as in the previous application of plant soil moisture sensing. Parylene is suitable for this application as well because it does not cause any surface adhesion of many different biological molecules or particles [8].

Before fabrication, the LC sensor performance is verified using simulation analysis. For this, LC is simulated with water on top of the capacitor part, inside the su8 wall which is completely filled. The thickness of this wall is varied. From the simulation analysis result shown below, minimum sample size for capacitor saturation is 4 microliters, corresponding to a height of 42 microns for (case 1) Su8 wall, sample height above this does not affected resonant frequency. For all other cases, the resonant frequency remains same. However, if this sample size is not maintained, the same LC will resonate at different frequency for different sample size of the same material.

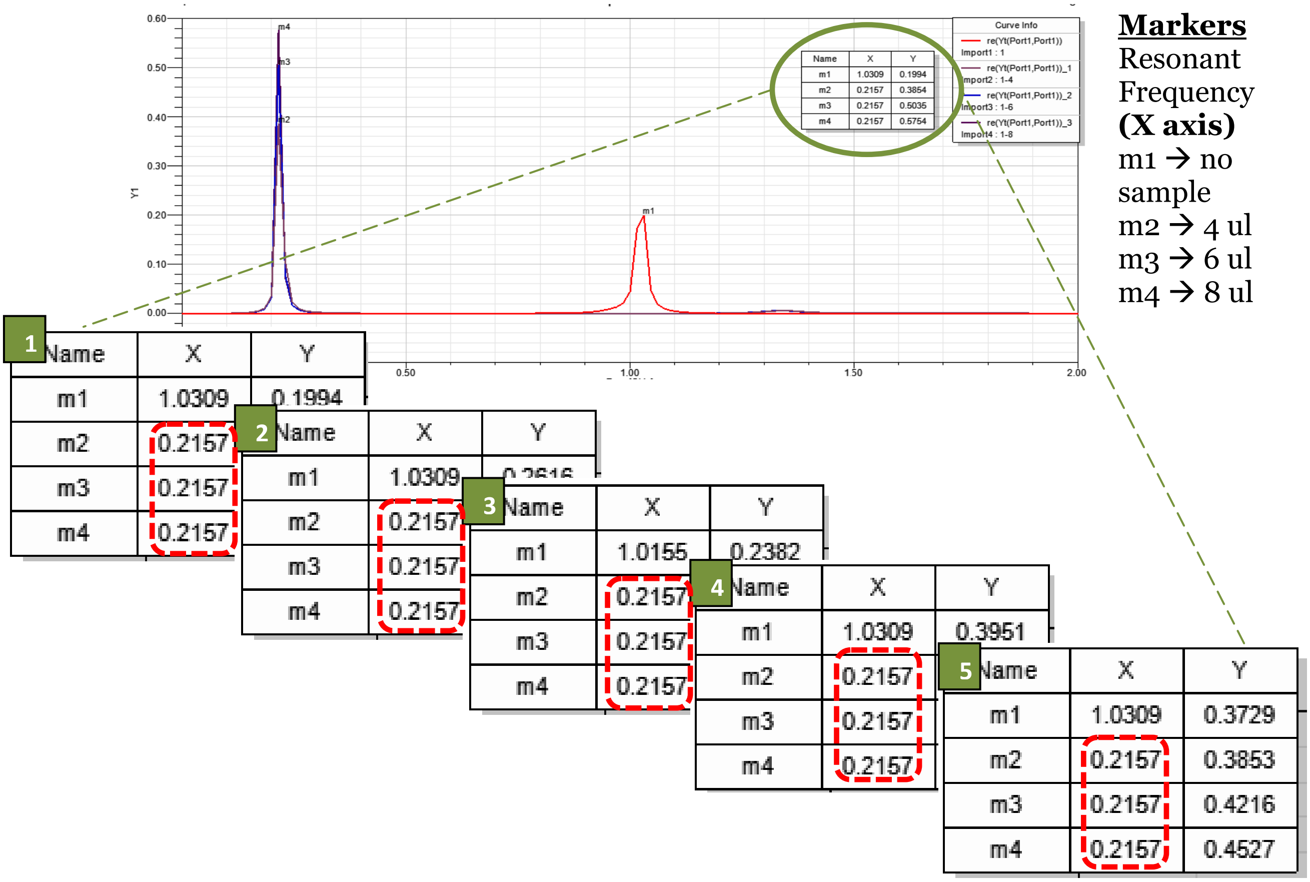


Figure 5.3 Sample size optimization for thickness of sample solution

The fabricated sensor is shown below. It is compact, approximately 1 by 1 centimeters in size. Due the Su8 wall, the test sample size is always constant over the LC resonator, as can be seen in figures below.

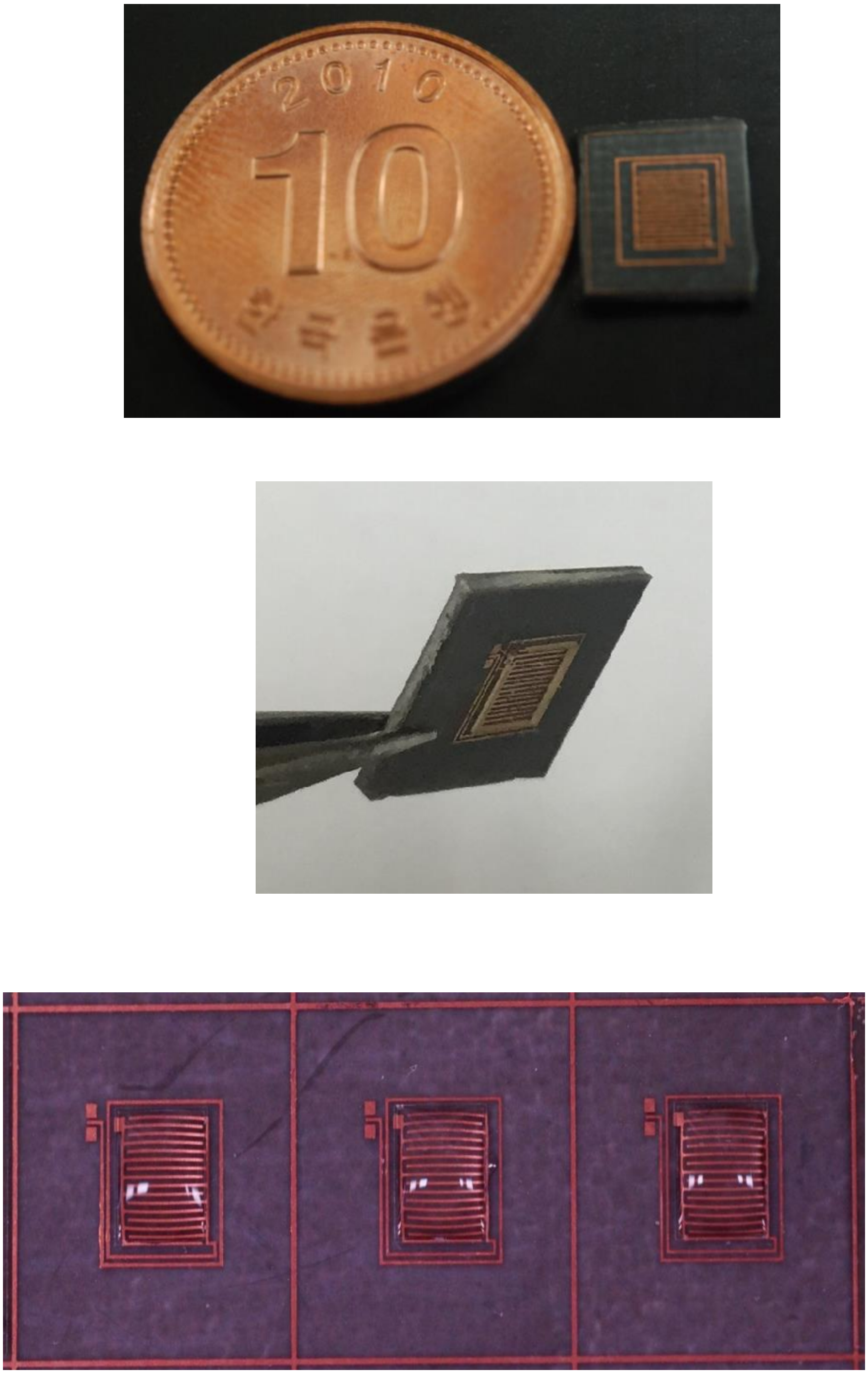


Figure 5.4 Fabricated LC Sensor with Su8 wall

5.5 Sensor Sensitivity

To estimate the sensitivity of the LC sensor, a control experiment is setup where the resonant frequency of the sensor is measured with different test solutions with known dielectric constants. Sensitivity extracted by measuring each of the standard solution, including ethanol, isopropyl alcohol, phosphate buffer and sodium hydroxide solutions with the sensor and plotting permittivity value at respective resonant frequency. The sensitivity plot is as below and the slope is -2.8, showing that the for an increase in the dielectric constant of the test environment by one unit, the resonant frequency of the LC will shift down by 2.8 MHz.

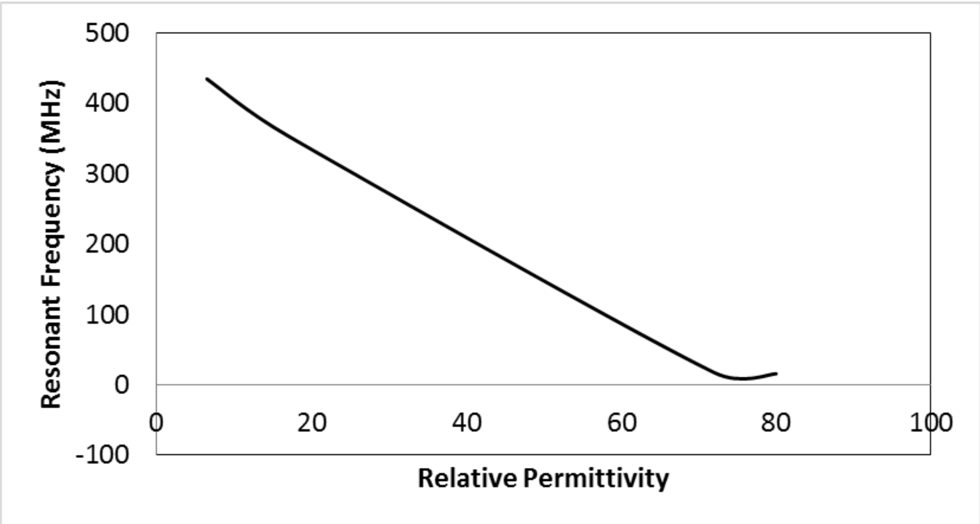


Figure 5.5 Sensitivity plot of the LC sensor

5.6 Testing DNA solutions

Network analyzer with probe station is used to measure the miniature LC sensor. The parameter under test is shift in resonant frequency Δf (difference in sensor resonant frequency with and without PCR sample) Multiple sets of readings for different PCR cycle sample are obtained. Example of one such data set for each case is shown below

For 900 bp

PCR cycles	Initial Resonant Frequency (MHz)	Resonant Frequency with Sample (MHz)	Shift in Resonant Frequency Δf (MHz)
10	1040.2	198.8	841.4
20	1022.4	178.2	844.2
30	1041.8	186	855.8

For 3000 bp

PCR cycles	Initial Resonant Frequency (MHz)	Resonant Frequency with Sample (MHz)	Shift in Resonant Frequency Δf (MHz)
10	1025.7	173.8	851.9
20	1034.6	181.6	852.4
30	1029.6	172.8	856.8

The overall result is summarized as below. It can be seen that the divergence of the sets is very high for the sample of low number of PCR cycles.

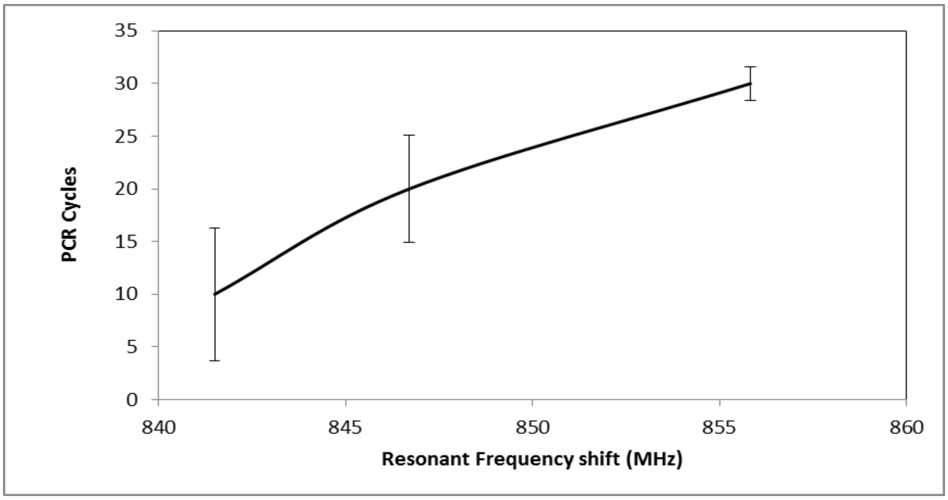


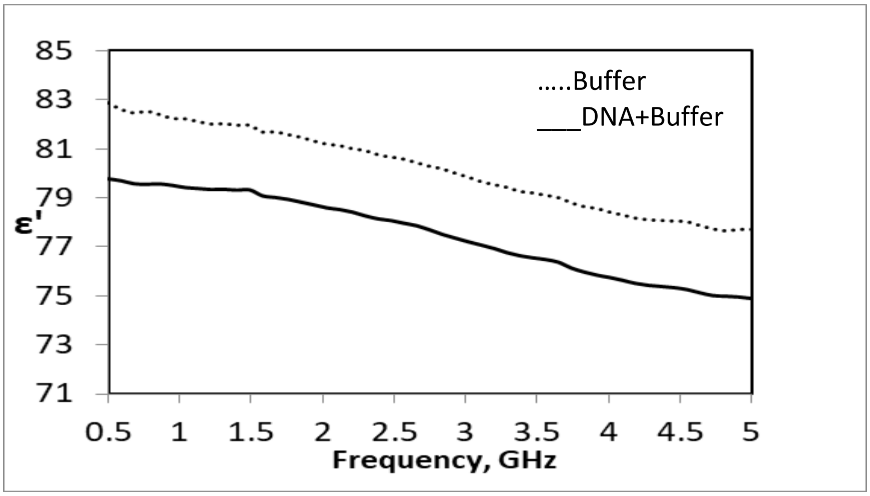
Figure 5.6 LC resonator response to different PCR concentration

Average tendency of data sets and standard deviation were analyzed. As the number DNA molecules increase, with more pcr cycles, deviation in data sets narrows. To understand in depth this trend of measurement data, dielectric properties of DNA are revisited and theoretical modelling approach is used.

5.6 Discussion

The DNA molecule has very interesting structure and thus dielectric properties. DNA is formed by the chains of nucleotides which are formed of sugar, base and phosphate group in helical conformation [9]. The two strands of the DNA helix are such oriented that their dipole moment cancel out. But when it is dissolved in solution an induced dipole moment is formed due to re-organization of charges [10].

Many researches have been published [11, 12, 13] which studies the dielectric behavior of DNA in solution, however it is difficult to summarize these and a make a single conclusion pertaining to this work. This is so due to the broad distribution of DNA chain lengths tested, also the DNA concentration varied greatly too from 0.1 to 7 milligrams per milliliters. Thus dielectric property measurement experiment is prepared to measure the dielectric properties of DNA specifically for the case of present experiment setup. The measurement technique and method is same as that performed in previous chapter 2. A high concentration PCR product DNA solution, obtained after 30 cycles for 900 bp, is used for testing. Also, the buffer, used for PCR, alone is tested.



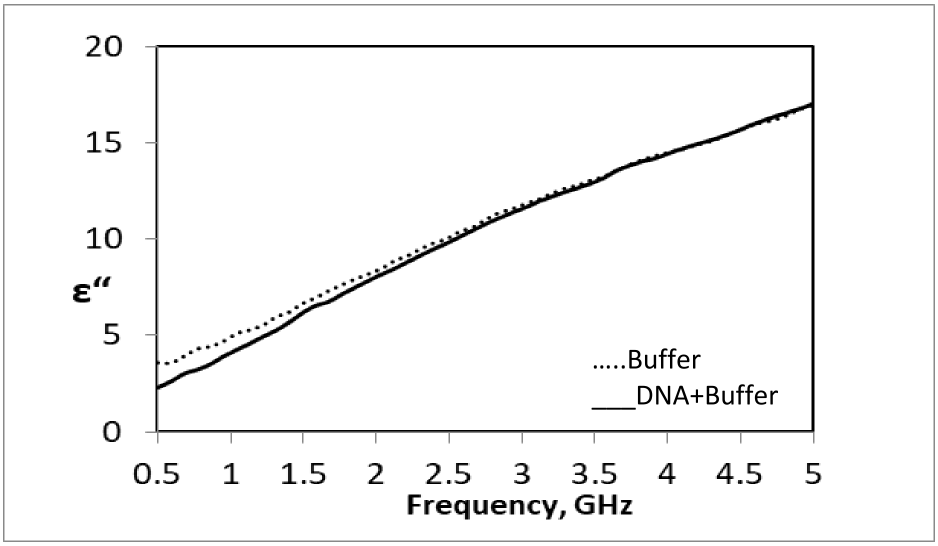


Figure 5.7 Measured complex dielectric constant of 900 bp DNA solution. Top is real part and Bottom is imaginary part

The Maxwell-Wagner model, discussed in chapter 2 is then used to estimate the effective dielectric constant of different concentrations of DNA solution, corresponding to different PCR cycles. A MATLAB program is used for this calculation [14]. The difference in dielectric constant, $\Delta\varepsilon r$ for each case is calculated, as shown below.

PCR cycle	%w/v DNA	ϕ	ε	$\Delta\varepsilon r$
10	3.98E-12	2.34E-12	82.8729 - 3.5923i	0
20	4.07E-09	2.39E-09	82.8729-3.5923i	0
30	4.17E-06	2.45E-06	79.7965-2.294i	3.076

The model estimates that in case of DNA solutions, obtained after 10 and 20 cycles of PCR, the change is dielectric constant from that of buffer solution alone, is negligible. However, for the case of 30 cycle PCR product, there is an appreciable change in dielectric constant. This explains the measurement data corresponding to 20 and 30 cycle PCR solutions with LC, showing large dispersion in data sets. This can be explained alternatively by the following curve.

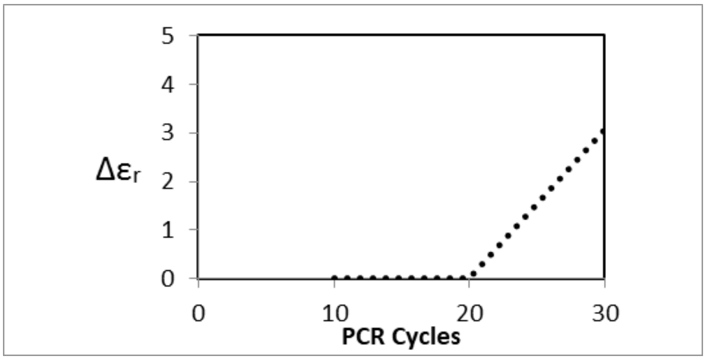


Figure 5.8 Change in dielectric constant of DNA solution for different PCR cycles

All the above experiments and studies conclude that dielectric sensing could be useful for on-chip PCR sensing application, however a high concentration of DNA is required or in other words, higher number of DNA cycles should be performed.

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Chapter 6

Conclusion

In this work newer applications of LC resonator are explored. Both the field of moisture sensing in greenhouse and on-chip PCR detection for PoC devices are fields requiring smart sensing capabilities. LC resonators prove suitable to be applied to these applications, however integrating the basic LC into such systems or applications is a future scope for this works.

Application-specific study, measurement and analysis of complex permittivity was undertaken in this work which opened newer avenues to explore while designing the sensor and highlighted the importance of application specific dielectric spectroscopy.

For the case of application in water content sensing, good laboratory performance of the sensor was achieved. Future work aims at on site testing. Also aims at developing a wireless read-out circuit. This would lead to a very simple and compact measurement system, operable in any kind of field location with swift sensing capability.

Proof of concept experiments and analysis for DNA detection for PoC application in real time have been performed. However, detailed experiments for higher DNA concentration are also required. Bio-recognition elements like antibodies and aptamer enhance the specificity of electrochemical sensors. Finally, a simple 'smart card' like measurement system by coupling the resonant sensor to read-out circuit antenna, is it possible to achieve a very simple measurement system, which is quick and easy to use anywhere can be incorporated with near-field wireless coupling is a promising future work for DNA sensor

Passive LC resonant sensors do not require wired connections or external power supplies, and thus, they can readily be applied to complex measurements in harsh environments. Furthermore, these sensors can realize high-speed data collection and perform highly efficient energy transfer within smaller frequency ranges and working distances. Thus, research into passive LC sensors is advantageous for measuring various physical, chemical, and biological parameters. With expanding needs for effective sensors and the development of micro/nano processing technology, it will be useful to apply this simple sensor to newer applications.

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Abstract

복소 유전율은 전기장이 인가된 때 전하를 저장하는 능력을 나타내는 유전체(절연체) 고유의(본래의) 특성이다. 이것은 재료마다 독특하며, 빌딩건축자재에서 생체조직에 이르기까지 광범위한 응용분야에서 특성을 나타내며 사용되어져 왔다. 본 고(연구)에서는 이러한 유전(절연) 특성들이 스마트 센싱 응용분야들, 즉 식물원(농원)에서 토양 수분 감지나 생의학 분야에서 표적치료(Point-of Care)장치를 위한 중합효소 연쇄반응(PCR) 온칩에서 무선 주파수의 감지를 수행하는데 적용되어졌다. 감지방식에 대하여 인덕터-커패시터 쌍으로 구성된 무선 주파수 공진 회로가 사용되었다. 인터디지털 커패시터 구조는 유전 특성의 변화에 반응하고 이 변화는 나선형 인덕터 구조와 결합되어질때 회로의 공진주파수 변화를 유도한다. 이 공진 주파수 이동은 유전(절연) 환경의 기능이므로 어떠한 변화에 대한 감지로 이어진다.(감지할 수 있다.) 수분감지 응용분야의 경우에 센서는 뿌리에 있는 식물 성장 기질에 내장되도록 개발되어, 기질의 외부에 있는 공진기 센서와 유도결합하여 동작한다. 뿌리영역에서의 수분의 중량 분석에 대한 변화는 내장된 공진기의 정전용량의 변화로 이어지며 공진 주파수의 변화를 만들어 낸다. 이 정보는 외부에 위치한 측정공진기로 전송되어 수분향량이 원격으로 모니터링 되어질 수 있다. 두번째 응용인 온칩 PCR은 센서가 테스트 시료들의 소량을 철리하도록 요구한다. 커패시터 영역에 있어 잘 구조화되고 회로 경로(판)에 대한 보호코팅을 가진 이 소형화 회로가 개발되었다. 다양한 직접물들을 가진 다양한 크기의 PCR 제품들이 테스트되었고 감지 범위가 결정된다. 감지원리는 이전 응용의 경우와 동일하며, 두가지 응용 실험 모두에서 측정 데이터를 확인하기 위하여

이론적 분석이 수행된다. 센서는 저렴하고 간단한 제조기술과 재료로 제조되고 있기 때문에 대량 생산이 용이하다. 무선 주파수에서의 감지(센싱)는 사물인터넷(IoT)에서 무선 센서 네트워과 통합 가능성을 제공하면서 최근 일반 산업 표준이 되고 있다.