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Auto-Tuning and Self-Calibration of High-Sensitivity Radio Frequency Interferometers

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Abstract

We demonstrate a technique that automatically tunes the sensitivity of a radio-frequency (RF) interferometer with a tunable liquid attenuator by accurately changing its liquid volume. The obtained effective quality factor (Q_{EFF}) of the interferometer is up to 1×10^8 at ~ 5 GHz. The Q_{EFF} is ~ 100 times higher than previously reported results. When material-under-test (MUT), i.e., methanol-water solution in this work, is used for the tuning, a self-calibration and measurement process is demonstrated from 2 GHz to 7.5 GHz at a methanol concentration level down to 5×10^{-5} mole fraction, which is 100 times lower than previously reported results. Further investigations are needed to achieve better system stability and higher sensitivity.

Index Terms

Dielectric measurement; microwave sensors; interferometer

I. INTRODUCTION

Accurate and rapid measurement of the dielectric properties of aqueous solutions is essential for studying the hydration structures and dynamics of macromolecules [1] and ions [2]. Such measurements are also promising for many biomedical applications [3]. As a result, various measurement techniques have been investigated, including the use of high quality factor (Q) resonators [4] and broadband transmission line structures [5]. Among these approaches, tunable radio-frequency (RF) interferometers, similar to the setup in Fig. 1(a), have demonstrated high sensitivity operations with an effective $Q_{eff} = f_0 / f_{3dB}$ up to 3.0×10^6 and a frequency coverage up to ~ 3 decades [6].

For a matched design, the measured transmission coefficient S_{21} of the interferometer in Fig. 1(a) is

$$S_{21} = K_1 \exp(-\gamma_{MUT} l_1) + K_2 \exp(-\gamma_{REF} l_1) \quad (1)$$

where K_1 and K_2 include the effects of all the components in material-under-test (MUT) and reference (REF) paths except MUT and REF liquid sections; γ_{MUT} and γ_{REF} are the

propagation constants of the sections, and their length is l_I (Fig. 1(b)). For high sensitivity MUT measurement at frequency f_0 , it is necessary to fine tune the interference process and to obtain very low $|S_{2I}/\min$ values (Fig. 1(a)). The tuning is achieved with high resolution tunable attenuators. Currently, significant experience is needed with available commercial devices. It is often challenging to keep the interferometer stable over a long period of time. Thus, rapid measurements are important. Furthermore, calibration liquids are needed to remove the unknown coefficients, i.e. K_I and γ_{REF} in (1), in order to quantify MUT permittivity. The accuracy of the calibration liquid properties and the measurement repeatability are significant concerns, especially when the analyte concentration level is very low. De-embedding approaches could avoid the use of calibration liquids by measuring different calibration structures [5]. But measurement repeatability of different structures is a challenge. In this work, we demonstrate that liquid-based attenuators can be exploited to automatically tune RF interferometer sensitivity at high resolutions. Also, MUT solutions can be used to calibrate the measurement system, and enable rapid and quantitative measurements of MUT dielectric properties. No additional calibration standards or de-embedding structures are needed. Thus, the above mentioned problems are addressed.

II. RF Interferometer with Liquid Attenuators

Figures 1(b) and (c) show the layout of a well-based tunable liquid attenuator built on a coplanar waveguide (CPW), which is fabricated with Duroid 5870 laminate. When MUT liquid, i.e., methanol-water solution, is used for tuning, the well is simultaneously a sample holder. A syringe pump shown in Fig. 1(a) is used to infuse or withdraw MUT liquid to or from the well with a pre-determined volume V , which effectively tunes the attenuation value. The attenuation resolution, which depends on initial h_{liq} (Fig. 1(c)), V , and MUT properties, can be very high since V can be very small, as indicated in Fig. 2. The curves are calculated with eqs. (2) and (3) in [6] at 3 GHz. Compared with digitally tunable attenuators, Fig. 2 indicates that the liquid attenuator has better resolution. It also has larger dynamic range as well as potentially smaller insertion loss and lower cost.

To evaluate the performance of the attenuator and the interferometer in Fig. 1, the well is initially filled with $\sim 600 \mu\text{L}$ deionized (DI) water, which corresponds to an h_{liq} of $\sim 4 \text{ mm}$. The $|S_{2I}/\min$ in Fig. 1(a) is manually tuned to $\sim -70 \text{ dB}$, the corresponding f_0 and $|S_{2I}/\min$ are the “starting point” in Fig. 3. Then the attenuator in Fig. 1(c) takes over the job for tuning $|S_{2I}/\min$. DI water is withdrawn from the well at $\sim 54 \text{ nL}$ per step with ~ 1 second time interval between two steps. As a result, both $|S_{2I}/\min$ and f_0 will change and reach a new $(f_0, |S_{2I}/\min)$ point in Fig. 3 until $(f_{0,\min}, |S_{2I}/\min, \min)$ is obtained. Further water withdrawal increase $|S_{2I}/\min$ until it goes back to $\sim -70 \text{ dB}$. The pump is then switched to infuse water to the well. Thus, curve 2 in Fig. 3 is obtained. The procedure is repeated and stopped at the “ending point”. The whole process took about 33 minutes.

Figure 3 shows that the water attenuator can automatically tune $|S_{2I}/\min$ of the interferometer in Fig. 1 to a very low level. Thus, very high frequency resolution and high measurement accuracy can be achieved. Nevertheless, significant $f_{0,\min}$ drift occurs during repeated infusion/withdrawal operations. In ~ 33 minutes, the drift is $\sim 40 \text{ kHz}$ at a rate of $\sim 1.2 \text{ kHz/minute}$. It seems that the drift is mainly induced by microwave absorption of the CPW line

and DI water in Fig. 1(c). Additionally, $|S_{21}|_{\min,\min}$ fluctuates up to ~ 4.87 dB, which is likely due to VNA thermal noise [3].

In Fig. 3, the interferometer has very high frequency resolution, consequently high sensitivity, at $(f_{0,\min}, |S_{21}|_{\min,\min})$. Figure 4 shows three curves measured on the VNA in Fig. 1(a) around 5 GHz. To make the figure clearer, only the $|S_{21}|$ curve with $(f_{0,\min}, |S_{21}|_{\min,\min})$, i.e., curve 1, and two adjacent curves, i.e., 2 and 3, are included. At point (5.0695322 GHz, ~ -138.88 dB), the measured Q_{eff} is more than 1×10^8 , which is exceptionally high and indicates outstanding frequency resolution. The Q_{eff} is comparable to or higher than those reported for optical resonators [8]. It is worth pointing out that lossy water, which significantly deteriorates the Q of resonators, is used in the measurement.

Unfortunately, it is difficult to keep the interferometer with such a high Q_{eff} stable for a long time. Thus, calibrating the system with standard liquids or de-embedding structures to quantify MUT permittivity is challenging. However, the interferometer is fairly stable over a short period of time. For instance, it takes about 3 seconds to obtain the 3 curves in Fig. 4. Assuming the same f_0 drift rate as in Fig. 3, a total frequency drift would be ~ 60 Hz. Thus, making use of successive measurements with the same MUT liquid but of different volumes is promising to provide a rapid and self-calibrating operation.

To obtain quantitative $\epsilon = \epsilon' - j\epsilon''$ of MUT, the coefficients $K_{1,2}$ and propagation constants $\gamma_{MUT,REF}$ in (1) as well as h_{liq} for $|S_{21}|_{\min,\min}$ in Fig. 4 need to be obtained. As a result, 4 different measurements need to be used for data extraction. High sensitivity/accuracy consideration suggests using the measurements close to the one with $|S_{21}|_{\min,\min}$, i.e., curve 1 in Fig. 4, where MUT $V_{\min}$ is also uncertain. Using (1), we have

$$S_{21,\min,\min} = K_1 \exp(-\gamma_{V_{\min}} l_1) + K_2 \exp(-\gamma_{REF} l_1) \quad (2)$$

Similarly, for $|S_{21}|$ at the same f_0 but measured immediately after drawing V (curve 2 in Fig. 4) or before drawing V (curve 3), the S -parameters are expressed as

$$S_{21,next} = K_1 \exp(-\gamma_{V_{\min} - \Delta V} l_1) + K_2 \exp(-\gamma_{REF} l_1) \quad (3)$$

$$S_{21,last} = K_1 \exp(-\gamma_{V_{\min} + \Delta V} l_1) + K_2 \exp(-\gamma_{REF} l_1) \quad (4)$$

Thus, we have

$$\frac{S_{21,last} - S_{21,\min,\min}}{S_{21,next} - S_{21,\min,\min}} = \frac{\exp(-\gamma_{V_{\min} + \Delta V} l_1) - \exp(-\gamma_{V_{\min}} l_1)}{\exp(-\gamma_{V_{\min} - \Delta V} l_1) - \exp(-\gamma_{V_{\min}} l_1)} \quad (5)$$

Additionally, another S_{21} curve next to curves 2 or 3 is needed. The formula for calculating propagation constant $\gamma = \alpha + j\beta$ can be found in [6]. It can be proven that $V_{\min}$, ϵ'_{liq} and ϵ''_{liq} can be solved using (2)–(5) when mismatches in Fig. 1 are ignored [9].

III. Measurement Results

We first validate the self-calibration method by measuring 0.005 mole fraction methanol-in-DI water (MUT) and comparing with the results reported in [10]. The liquid attenuator in Fig. 1(c) is also used as a sample holder. Approximately 600 μL MUT is infused to establish an initial h_{liq} in Fig. 1(c). Then the manual phase shifters and attenuators are used to tune $|S_{21}|_{\min}$ to ~ -80 dB at the desired frequency point. After that, the computer controlled syringe pump automatically infuses or withdraws MUT solutions to or from the well, ~ 54 nL per step. The measured scattering parameters are recorded until $|S_{21}|_{\min, \min}$ is identified. Repeat the process 4 times for the same frequency point before move to the next frequency point.

Using (2)–(5), the extracted complex permittivity at different frequencies of the methanol-water solution is shown in Table I. They agree with the results in [10] reasonably well. Thus, our auto-tuning and self-calibration measurement procedures are validated.

Then, methanol-water solutions at 5×10^{-4} and 5×10^{-5} mole fractions are measured similarly at 3 GHz to further demonstrate the high sensitivity capabilities. The methanol concentrations are 10 and 100 times lower than that in [10], respectively. The obtained permittivity data, averaged from five independent measurements, are shown in Table II. As expected, lower methanol concentration MUT has ϵ closer to that of water. The values are reasonable even though there are no published data for comparison. Thus, accurate permittivity values or small permittivity changes can be automatically measured without the need for additional calibration liquids or de-embedding procedures. Table III shows that our main results compare favorably with recently published dielectric spectroscopy results.

The high resolution liquid attenuator is also useful for investigating the group delay (GD) and phase properties of the interferometer. Both depend on the balance of the two signal transmission paths [14] in Fig. 1(a). Figure 5 shows typical measurement results. The positive group delay increases continuously from several nano-seconds to ~ 0.526 ms around $|S_{21}|_{\min}$. After further withdrawal of MUT, the group delay is tuned to negative values.

IV. Discussions and Conclusions

The demonstrated capabilities to automatically tune sensitivity and self-calibrate system operation make the interferometer in Fig. 1 unique and promising for various applications, including sensing molecules and ions at low-concentration levels in liquids. However, further work is needed to improve the stability of the liquid attenuator and interferometer so that less $f_{Q, \min}$ drift in Fig. 3 and smoother curves in Figs. 3 and 4 are achieved. The main factors that need to be addressed include mechanical vibrations (e.g., disturbance due to syringe pump operation) and environment variations. The performance of the VNA, such as its frequency resolution and dynamic range, also need to be evaluated in the context of interferometer operations.

In summary, we demonstrated that infusing or withdrawal liquid to or from a well can achieve high resolution attenuation tuning. The obtained effective quality factors of the interferometer are up to 1×10^8 . The group delay of the interferometer can be tuned from nanosecond level to millisecond level, at positive or negative polarities. When MUT sample is used, the automatic tuning process also self-calibrates the measurement system and yields MUT permittivity values. Methanol-water solution is measured down to 5×10^{-5} mole fraction concentration level.

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Fig. 1.

(a) A schematic of the proposed RF interferometer with liquid attenuators. Another attenuator in the REF path can be replaced by a cable. The additional manually-tunable attenuators (R) and phase shifters (ϕ) are introduced for additional tuning flexibility. (b) Top view and (c) cross section of the liquid attenuator in (a), where $w=0.13\text{mm}$, $g=1.935\text{mm}$, $t=17\mu\text{m}$, $h_{well}=20\text{mm}$, h_{liq} : tunable, $h_{sub}=0.787\text{mm}$, $w'=2.2\text{mm}$, $g'=0.9\text{mm}$, $l_1=10\text{mm}$, $l_2=15\text{mm}$, $l_3=32\text{mm}$, $l_4=40\text{mm}$, $\epsilon_{sub}=2.33$, ϵ_{liq} : to be measured.

Fig. 2.

Calculated $|S_{21}|$ of the attenuator in Fig. 1(b) when infuse (positive change) or withdraw (negative change) lossy liquids. h_{liq} is referenced to the initial h_{liq} at which the characteristic impedance values are 48.677 Ω , 44.033 Ω , 43.717 Ω and 54.116 Ω . A change of 54 nL liquid volume induces 360 nm h_{liq} . When h_{liq} is tuned from -1000 nm to $+1000$ nm, the impedance value changes are below 0.017 Ω . Thus, the attenuator is reasonably matched.

Fig. 3. Measured trajectory of $(f_0, |S_{21}|_{\min})$ in Fig. 1(a) for repeated water infusion and withdrawal operations.

Fig. 4. Measured interferometer outputs when MUT is removed from the well at 54 nL/step. Curve 2 is one step after curve 1, which is one step after 3.

Fig. 5. Typical positive and negative group delays (τ_g) and corresponding phases (ϕ) (blue square for positive and red circle for negative) tuned with the attenuator.

TABLE I

Extracted complex permittivity of methanol-water solution at 0.005 mole fraction and comparison with the data in [10].

Freq. (GHz)	This work		[10]	
	Real	Imag	Real	Imag
2	77.1327±0.0063	7.6241±0.0034	77.1304	7.6212
3	76.1358±0.0005	11.2783±0.0004	76.1417	11.2692
4	74.8036±0.0056	14.7401±0.00005	74.8056	14.7372
6	71.2439±0.0021	20.9998±0.0097	71.2586	20.9673
7.5	68.0076±0.0016	24.9204±0.0004	68.0224	24.9164

TABLE II

Measured complex permittivity of methanol-water solutions at 0.005 (#0), 0.0005 (#1), 0.00005 (#2) mole fractions and pure DI water (#3).

#	Real	Imag
0	76.1358±0.0005	11.2783±0.0004
1	76.5192±0.0017	11.1125±0.0023
2	76.5479±0.0023	11.1048±0.0047
3	76.5462±0.0004	11.1097±0.00006

TABLE III

A comparison with some recent dielectric spectroscopy work.

Reference	Q_{EFF}	Min. Concentration	Max. Error
[11]	15–75	pure solutions	<1%
[12]	7–11	nitrocellulose	NA
[13]	NA	1.25 vol%	0.0066(ϵ') @ $4 < \epsilon' < 5$
This work	$\sim 1 \times 10^8$	5×10^{-3} mol%	0.0097(ϵ'') @ $\epsilon'' = 20.99$