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DOI:
10.1002/mop.32679

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Document Version
Publisher's PDF, also known as Version of record

Citation for published version (Harvard):
Mohammed, AM, Wang, Y & Lancaster, MJ 2020, '3D printed coaxial microwave resonator sensor for dielectric measurements of liquid', *Microwave and Optical Technology Letters*, vol. 63, no. 3, pp. 805-810. https://doi.org/10.1002/mop.32679

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Download date: 11. May. 2021

Received: 4 June 2020

DOI: 10.1002/mop.32679

WILEY

RESEARCH ARTICLE

3D printed coaxial microwave resonator sensor for dielectric measurements of liquid

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Funding information
This work was supported by Petroleum Technology Development Fund (PTDF) in Nigeria by providing the PhD scholarship to Mohammed, A. M.

Abstract

This paper presents a coaxial resonator for dielectric measurements of low and medium loss liquids at multiple resonance frequencies up to 8 GHz with the fundamental transverse electromagnetic (TEM) resonant mode at 2 GHz. The measurement is by filling the entire resonator cavity with the material under test and the permittivity of the material is readily extracted using simple equations. This technique provides an easy and accurate extraction method of dielectric properties without any analytical approximation and dedicated software algorithm as used in broadband open coaxial probes or the complex perturbation formula in resonator-based methods. This significantly reduces the risk of systematic errors from the model approximation. The measured quality factor of the resonator is 2650 to 3500 depending on the resonant mode. This allows for the measurement of samples with loss tangents up to 0.05 (Acetone taken as a reference). The device was made by 3D printing and verified by measurements of several common solvents at all four resonance frequencies. The results obtained agree well with values reported in literature. Further measurements of crude oil samples were carried out and results confirmed with values obtained using other techniques.

KEYWORDS

3D printing, coaxial resonators, liquids, permittivity, quality factor

1 | INTRODUCTION

Coaxial structures have been widely utilized in measurements of dielectric materials.[1-7] One of the most common type is the industrial dielectric probe or coaxial probe and also referred to as open-ended coaxial line[1] for liquids, solids and gel materials. Other than the conventional probe, various forms of open-end resonators have been utilized in this regard. Using the open-end coaxial cavity resonator method,[8] the sample is placed in contact with the open-end of the resonator to determine the reflection coefficient or transmission coefficient over a broadband depending on the port-type. The resonance frequency and quality factor of the resonator is modified as the electric field fringes into the material under test. These quantities are then utilized in the extraction of the dielectric properties. Based on this principle various design approaches have been reported in the literature. An open coaxial resonator with RF transceiver and microcontroller as measuring instrument was presented for moisture monitoring in paper, textile, and foil processing.[2] In order to reduce the effect of air gap associated with open-ended resonator, spring supported inner conductors were introduced.[2,3] Detailed design process of coaxial resonator has also been reported[4] and the authors later used the technique for rain sensing using a glass covered open-end.[5] A coaxial-feed open-ended cut-off circular waveguide resonator have been utilized for dielectric measurement using a reflection approach and was verified in comparison with other methods of dielectric measurements.[6] More recently, Zhu et al[7] reported a hollow coaxial cable Fabry-Perot resonator that is made for dielectric constant measurement at low-cost. The concept is to replace the insulator in the cable with air and create an opening in the hollow coaxial cable. The opening groove allows the liquid into the cavity and the reflected signal is measured accordingly.[7] Closed-end coaxial resonator has been used previously for surface resistance measurements of superconducting materials over 17 resonance frequencies up to 20 GHz.[9] The design was later modified by introducing two tuning screw to enable tuning of the different modes of the cavity. The device has been validated for microwave characterization of superconductors of larger dimensions both in linear and nonlinear regimes.[10]

In this work, a closed-end coaxial cavity resonator is proposed for the measurements of low and medium loss liquid materials for the first time to the best knowledge of the authors. The design is in such way that the entire cavity would be filled with the material under test. This makes the extraction of the permittivity very easy using a simple equation as opposed to dedicated software algorithm as in the broadband open coaxial probes or using complex cavity perturbation formula in the resonant method. These conventional approaches rely on some model approximation of the field distribution and their interaction with the material under test. This often introduces system errors. The technique demonstrated in this work does not use such approximations. As compared with the wideband coaxial probe method, the resonant method offers high sensitivity. The narrow band limitation of the resonant method is partly compensated for by using the multiple resonance techniques demonstrated in this paper.

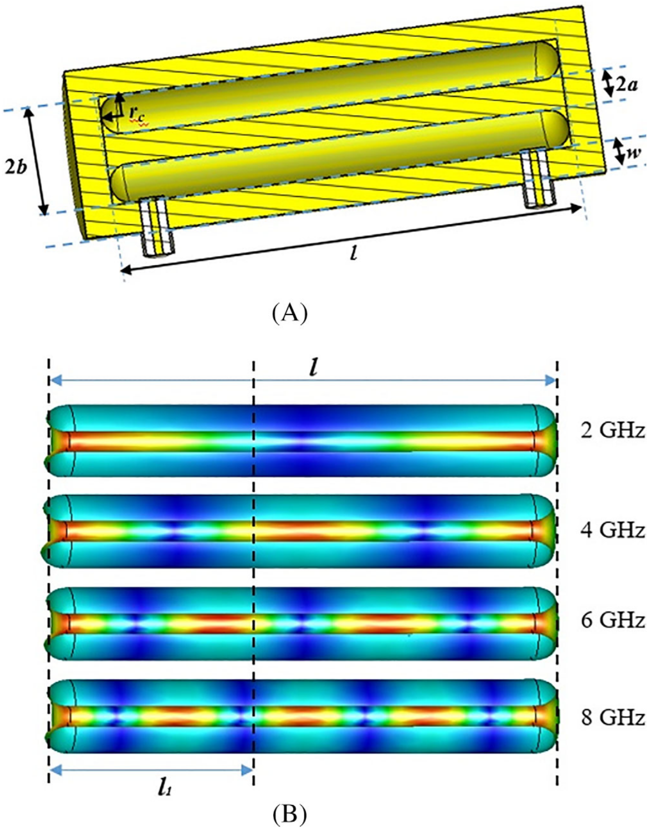


FIGURE 1 A, Illustration of the coaxial resonator internal structure with design dimensions in millimeter (mm): a = 5, b = 17.9, l = 74.59, w = 5 and r_c = 3.25; B, Simulated current distribution of the resonator at four different resonance modes [Color figure can be viewed at wileyonlinelibrary.com]

2 | DESIGN AND FABRICATION OF THE COAXIAL RESONATOR

The half-wavelength air-filled coaxial was made short-circuited at both ends to facilitate the support of the central conductor. As shown in Figure 1A, the diameter of the inner conductor of the coaxial resonator is $a = 5$ mm while the outer cavity diameter is $b = 17.95$ mm, which is approximately 3.59 times the inner radius to ensure the optimum quality factor (Q-factor).[11] The length of the coaxial resonator l is 74.59 mm, half of the wavelength at the fundamental frequency. To increase the Q-factor and mitigate effect of losses resulting from sharp corners in the cavity we have introduced round corners in the coaxial cavity resonator. Simulation shows a corner radius r_c of 3.25 mm resulted in 2.5% increase in Q-factor compared to the one without round corners. A 3 mm hole was created to enable liquid sample insertion. The coaxial resonator is coupled based on a transmission configuration and the coupling into and out of the resonator was deliberately made weak to attain insertion loss of over 10 dB in all the resonance modes. The resonator is excited with two identical 50 Ω subminiature A (SMA) connectors. The SMA open probe has a conductor diameter of 1.3 mm with 1 mm penetration length into the cavity. These design parameters provides frequency operation based only on transverse electromagnetic (TEM) modes up to 8 GHz with a fundamental transverse electromagnetic TEM-mode resonance of 2 GHz. The 8 GHz limit is determined by the onset of the first high-order nonTEM mode (TE11) and it covers four TEM-mode resonances within the frequency range. Besides the fundamental TEM modes, there are other TE and TM modes in the coaxial resonator that could be utilized for the measurement of dielectric properties of materials. However, an added complexity with the use of these higher order modes lies in the tracking of the mode after filling the liquid. This is possible but not considered in this work. In order to maintain the use of only TEM mode resonances but extend the operation frequency range, the limit of the upper frequency by TE11 mode could be pushed higher by reducing the dimension of the inner and outer conductor while maintaining the ratio $b/a = 3.59$ for the optimum Q-factor. Also, as the length of the resonator determines the frequency of the first fundamental TEM mode. Increasing the length of the resonator will be able to generate more modes (and therefore more measurement points) within the frequency range. This work only uses four TEM-mode up to 8 GHz to ease fabrication within reasonable dimensions.

The coaxial resonator is closed at both ends. To ease fabrication and assembly, the device has to be cut. However, cutting along the radial direction of the coaxial disturbs the currents and increases the losses. To minimize this effect, the cutting has to be made at a point where the current density is not at its maximum for all the concerned resonance modes. To this end, the current distributions in all four resonance modes are studied and shown in Figure 1B, based on eigen-mode simulation in CST microwave studio. Cutting the device in the middle or at the ends may significantly disturb the current of at least two modes, as it cuts through the current maxima. To avoid these maxima, the device was conveniently cut at distance $l_1 = 28.827$ mm from one end. At this cutting position, we have considered the trade-off between all four modes. This to a large extent will not have much effect on the overall performance of the device, as later confirmed by the experiment. It is noted that further trade-off may have to be made if more resonances are used either to extend the measurement frequency range or the number of data points. Having said that, as the extraction of the dielectric property relies on the differences in frequency before and after the liquid filling rather than the absolute frequency values, the impact of the cutting is not expected to be significant. The depth and round geometry of the coaxial structure presents a big challenge for CNC machining. Therefore, the device was manufactured by stereolithography (SLA) 3D printing with Accura 25 plastic materials. The device was then copper plated inside and out with a 10 μm thickness.

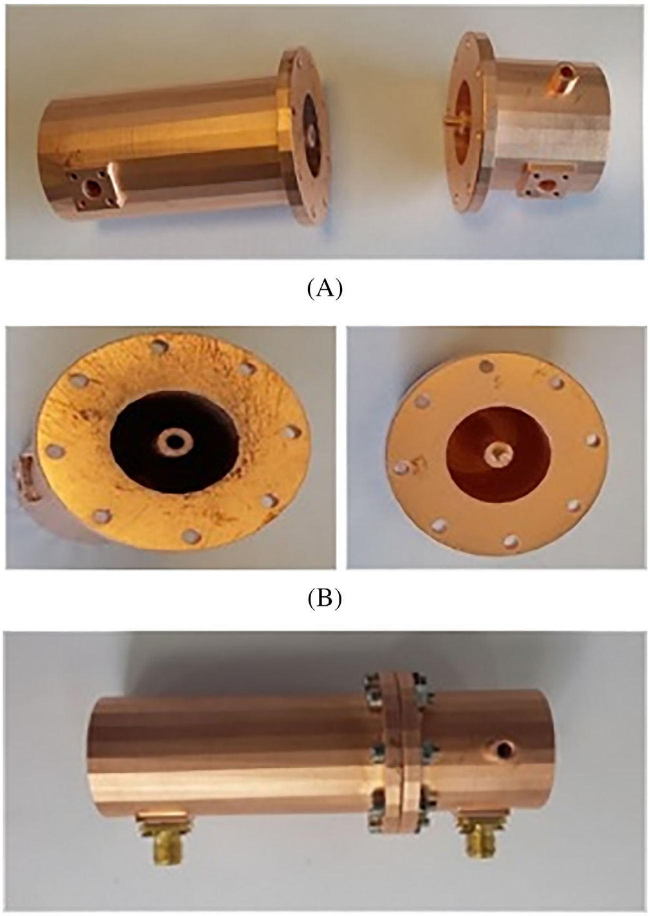
3 | MEASUREMENTS

3.1 | Coaxial resonator measurements

The coaxial resonator cavity was tested after assembling the device with eight 2.5 mm screws as shown by the photo of the prototype in Figure 2. The measurement procedure begins by calibrating the device using 85052D calibration kit over a frequency range of 250 MHz to 8.50 GHz. An IF bandwidth of 500 Hz was used with 6401 sweep point to better the accuracy of the *Q*-factor measurement using the 3 dB method. The measured and simulated broadband transmission coefficient (S_{21} response) of the empty coaxial resonator is given in Figure 3. There is a close agreement between the two results with a frequency shift of around 10 MHz (0.50%) at 2 GHz, 19 MHz (0.48%) at 4 GHz, 26 MHz (0.43%) at 6 GHz and 30 MHz (0.38%) at 8 GHz. These small shifts are a result from the fabrication tolerance of about 10 μm. The measured *Q*-factor is 2650 to 3500 for the four resonances, which is in very good agreement with the simulated *Q* of 2900 to 3850. This demonstrated good fabrication accuracy of the SLA and plating quality.

3.2 | Material measurements

For material measurements, the coaxial resonator was filled with liquid samples using a glass pipette through the 3 mm hole opening. Three common solvents of n-heptane (Analytical reagent AR grade), hexane (HPLC grade with purity of 95%) and pentane (AR grade with purity of 98%) were used as samples to verify the device. The first two samples are product of Fisher Scientific while the third is from Sigma Aldrich Ltd. The cavity is emptied after every measurement and allowed to evaporate and dry. It is further



(A)

(B)

(C)

FIGURE 2 Photo of prototyped coaxial resonator. A, Resonator in two parts; B, Inner view of the parts; C, Assembled with 2.5 mm screws and nuts [Color figure can be viewed at wileyonlinelibrary.com]

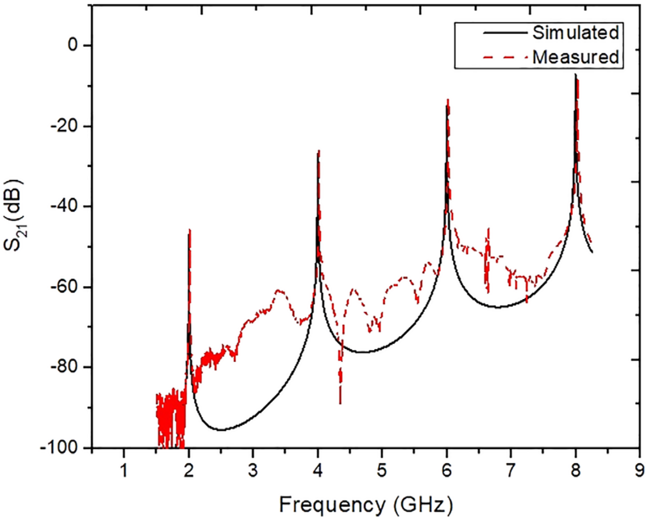


FIGURE 3 S_{21} response of measured and simulated empty coaxial resonator [Color figure can be viewed at wileyonlinelibrary.com]

heated gently with an electric dryer. The device is then cooled to the ambient temperature. The empty cavity is then measured again at all four-resonance modes before putting in a new liquid sample. The S_{21} response of the empty cavity after cleaning following the measurement of pentane and hexane is shown in Figure 4. There is very little change in the response. Further measurements of dielectric properties of light and medium crude oils as classified by American Petroleum Institute API gravity were carried out. Acetone as a medium loss sample was also measured. The S_{21} broadband response of the measured crude oils is given in Figure 5. We can clearly observe the shift of resonance peaks and the change of the magnitude of S_{21}. Following, the measurement of light crude oil, the cavity was cleaned with Isopropanol. However, this time the quality factor of the cavity drops by 7.5% due to cavity contamination. Though the resonance frequency barely changes as shown in Figure 4.

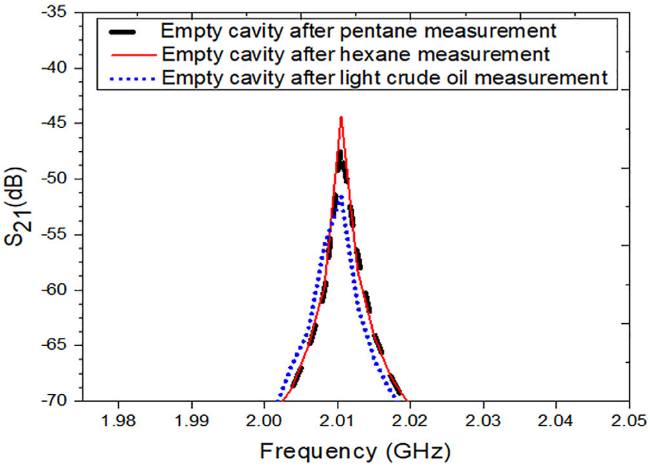


FIGURE 4 Measured S_{21} response of empty coaxial resonator after measurements with different samples and cleaning at 2 GHz [Color figure can be viewed at wileyonlinelibrary.com]

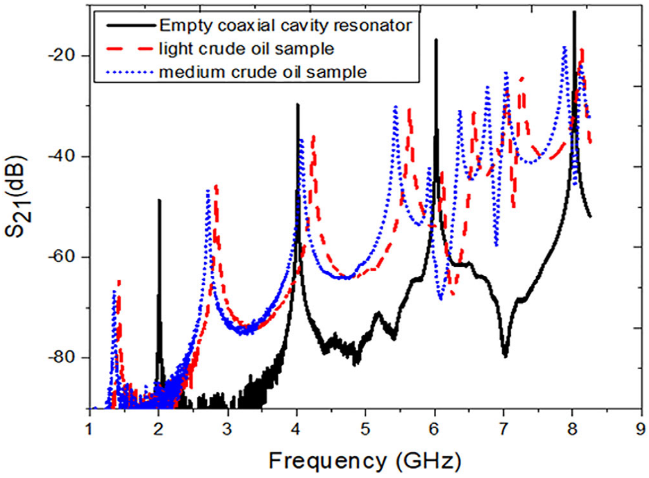


FIGURE 5 Broadband S_{21} response of the measured crude oil samples at the four resonant modes [Color figure can be viewed at wileyonlinelibrary.com]

3.3 | Extraction of dielectric properties

The resonance frequency and quality factor before and after the samples was put into the cavity were measured. The resonance frequency was used to evaluate the magnitude of the dielectric constant based on a simple and accurate relationship, without analytical approximation, given by

$$\varepsilon_r = \left(\frac{f_r}{f_s}\right)^2. \tag{1}$$

where f_r is the resonance frequency of the empty cavity and f_s is the resonance frequency with the sample. The measured Q-factor was used in extracting the dielectric loss tangent $tan\delta$ of the material.[12] Filling the cavity with dielectric medium affects the unloaded Q-factor, which is determined by dielectric losses and conductor losses and is related by

$$\frac{1}{Q_u} = \frac{1}{Q_c} + \frac{1}{Q_d} + \frac{1}{Q_r}. \tag{2}$$

$$\frac{1}{Q_l} = \frac{1}{Q_u} + \frac{1}{Q_e}. \tag{3}$$

$$Q_u = \frac{Q_l}{1 - S_{21}(f_o)}. \tag{4}$$

$$tan\delta = \frac{1}{Q_d}. \tag{5}$$

where Q_u, Q_l and Q_e are the unloaded, loaded and external quality factors respectively. The Q-factors due to conductor, radiation and dielectric of the materials are Q_c, Q_r and Q_d respectively. f_o is the resonance frequency.[12] The loaded Q-factor before and after the cavity is filled with liquid samples are measured. The unloaded Q-factors were extracted using Equation (4) in each case. The Q-factor due to radiation Q_r was estimated to be 2.4×10^6 based on simulation using (2). Neglecting Q_r, $Q_u \approx Q_c$ when the cavity in empty. The loss tangent is then evaluated using (2) and (5). The results of all the measured samples at all the resonance frequencies are given in Table 1 and is discussed in Section 4.

3.4 | Condition of accuracy

Measurements were taken within a control ambient temperature of 21 ± 1°C. It is believed the uncertainty caused by temperature variation is negligible. Potentially trapped air bubbles or gaps are a major source of error in the experiment. To minimize this, air bubbles were pushed out by tilting the device to ensure adequate removal of air. However, it is still difficult to ascertain complete air removal. The effect of air gap was analyzed using CST simulation by introducing an air gap varying from 0.5% to 7.5% of the

TABLE 1 Comparison of dielectric properties of measured samples with literature values

	2 GHz		4 GHz		6 GHz		8 GHz		Literature		
Samples	ε_r	$tan\delta$	ε_r	$tan\delta$	ε_r	$tan\delta$	ε_r	$tan\delta$	ε_r	$tan\delta$	**References**
Heptane	1.81	0.00013	1.81	0.00017	1.82	0.00024	1.83	0.00033	1.92	0.00041	at 8 GHz[13]
Pentane	1.84	0.00010	1.84	0.00013	1.83	0.00017	1.84	0.00023	1.84	0.00025	at 8 GHz[13]
Hexane	1.88	0.00011	1.88	0.00014	1.88	0.00019	1.88	0.00026	1.89	0.00031	at 8 GHz[13]
Acetone	20.98	0.016	20.81	0.031	20.97	0.043	21.02	0.055	21.40	0.047	at 2 GHz[14]
Light crude oil	2.00	0.0055	2.01	0.0055	2.02	0.0051	2.03	0.0049	2.16	0.0097	at 2 GHz[15]
Medium crude oil	2.20	0.012	2.18	0.010	2.19	0.0092	2.20	0.0085	2.31	0.0064	at 2 GHz[15]

sample volume over ε_r of 2 to 10 in step of two using the first resonant mode. Based on the method adopted to remove air gap in the cavity, the air gap (if any) is assume to be trapped at the top between the liquid and cavity wall considering its seating in Figure 2c. Based on simulations of the cavity with the air gap at this position, an air gap of 0.5% and 7.5% of the sample volume would result in $\Delta\varepsilon_r$ of 1.15% and 3.5% for $\varepsilon_r = 2$ respectively. This will go higher as the percentage of air gap and values of ε_r increases. However, this would not give a true physical representation of the air gap within cavity but an estimate of the effect of air gap in this type of measurement.

4 | RESULTS AND DISCUSSION

As given in Table 1, the measured dielectric properties of the samples under consideration are in close agreement with literature values and also in consistence to each other at all resonant frequencies. The maximum variation among the four modes is about 1.25% for all the measured samples with reference to the fundamental mode results. This has strongly validated the measurement method. However, one question lies in the re-usability of the resonator. The cavity has to be opened, cleaned, and re-assembled after every measurement. Depending on the type material measured, the cavity is subject to potential contamination or even damage. However, the fact that the dielectric constant depends on the ratio of resonance frequency before and after the sample, this may not have a significant effect on the results. This was further verified by measuring hexane sample at all the resonance modes following cavity contamination (with heavy crude oil) with measured fundamental resonance frequency of the empty cavity being 1.988 GHz and *Q*-factor of 564. Results obtained (eg, $\varepsilon_r = 1.878$ at 2 GHz) are still consistence with the earlier results given in Table 1 for the dielectric constant and in conformity with the values in literature. However, the *Q*-factor due to dielectric of the material in this case cannot be correctly evaluated. The measured *Q*-factor is far below the initial measured *Q* of 2650 and therefore, does not represent the actual ratio of energy stored and dissipated due the dielectric material. For this, the loss tangent is not extracted in this case. Therefore, from our experience and results of investigation presented here, it is believed that reopening the cavity is less of the problem. The main issue in this case is the degradation of the metal coating on the 3D printed part. This is a relatively well-known challenge for plastic 3D printing. This can be mitigated by using other fabrication method such as 3D metal printing.

5 | CONCLUSION

A coaxial half-wavelength cavity resonator was designed to undertake dielectric measurement of low-and medium loss liquid samples over a broadband up to 8 GHz. Four resonance modes are utilized. The device was fabricated by 3D printing. The change in resonance frequency and the *Q*-factor of materials under measurement were measured by filling the entire cavity. The dielectric properties were extracted using a simple and accurate formula. The results obtained have validated the capability of using the device for dielectric metrology. The re-usability of the device remains a challenge for the closed coaxial resonator especially as to the extraction of loss tangents. However, the technology provides an easy and accurate extraction method of dielectric properties without using any dedicated software algorithm as in the broadband open coaxial probes or using complex cavity perturbation formula in the resonant method. The risk of system error due to analytical approximation is therefore mitigated.

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How to cite this article: Mohammed AM, Wang Y, Lancaster MJ. 3D printed coaxial microwave resonator sensor for dielectric measurements of liquid. *Microw Opt Technol Lett*. 2020;1–6. https://doi.org/10.1002/mop.32679