Dielectric Spectroscopy of Fluids and Polymers for Microwave Microfluidic Circuits and Antennas

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Abstract-The permittivity response from 0.5 to 40 GHz of ethyl acetate (C₄H₈O₂), ethylene glycol (CH₂OH)₂, castor oil (C57H104O9), liquid paraffin, and other fluids commonly used for microwave microfluidic antennas and circuits is reported. The response of selected polymer solids, such as polydimethylsiloxane (PDMS), acrylonitrile butadiene styrene (ABS), acrylic, nylon, and others, used in microfluidics and 3-D printing of microwave structures was also studied from 0.5 to 20 GHz. Permittivity measurements were taken with a coaxial probe. Using a nonlinear least-squares fitting routine, the Cole–Cole dielectric relaxation model parameters are found for each material under test, thereby providing an analytic expression for their permittivity response.

Index Terms—Acetone, acrylic, acrylonitrile butadiene styrene (ABS), antennas, circuits, Cole-Cole model, dielectric fluids, electronics, ethyl acetate, ethylene glycol, flexible materials, liquids, microfluidics, microwaves, nylon, permittivity, polydimethylsiloxane (PDMS), polymers, Ricinus communis, 3-D printing.

I. INTRODUCTION

IQUID dielectrics and insulating polymers are constituent elements for flexible electronics [1]-[4]. Furthermore, microfluidics integration with microelectronics has opened new areas of research and development for high-frequency electronic systems. For example, fluidic tuning of antenna output frequency, radiation patterns, and polarization have been shown in numerous works over the last decade [5]-[14]. Frequency tuning of microwave components, including filters [15], [16], phase-shifters [17], [18], power splitters, [19], [20], and oscillators [21], has also been investigated using fluids.

Notwithstanding the growing body of work of fluidic electronics noted earlier, there is a limited amount of publicly available data on the permittivity of various dielectric fluids and polymer compounds used to implement those systems. In the absence of such data, researchers often rely on permittivity data gathered at one frequency to approximate the response of their device at other frequencies. Only recently have dielectric spectroscopy studies begun to appear that report on the wideband response of dielectric fluids of interest [22]. In this work, we report on the broadband complex permittivity

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(a) (b)

Fig. 1. Coaxial probes for permittivity characterization of (a) solid samples and (b) liquid samples.

response of widely used compounds for microfluidic electronic design, including ethylene glycol and water solutions, solvents, oils, flexible substrates, and 3-D-printed plastics used in electromagnetic structures. The materials were characterized using a coaxial probe and a vector network analyzer. Analytic expressions for the permittivity response as a function of frequency for the materials under study were extracted and fit to the Cole-Cole equation using a nonlinear least-squares routine. Several of these materials were also characterized over temperature, such as the oils and the ethylene glycol (commercially known as antifreeze) and water solutions. Knowledge of the permittivity of dielectric fluids and polymers is critical for accurate modeling and full-wave electromagnetic simulation of fluidic antennas and microwave circuits prior to fabrication and measurement.

This article is organized as follows. Section II describes the permittivity characterization procedure and discusses the nonlinear least-squares method used to curve-fit the analytic Cole-Cole model to the measured data. Section III presents the measured results and tabulates the Cole-Cole model parameters for the dielectric fluids and polymers under test. Section IV concludes this work.

II. METHODS AND MODELS

Permittivity measurements of materials under test are carried out using the open-ended coaxial probe method, as shown in Fig. 1. For the characterization of solid materials, the probe

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makes contact at the surface of the object and for the characterization of liquids, and the probe is fully submerged in the substance. The amount by which the material disturbs the fringing electromagnetic fields at the open termination is measured as a change in the amplitude of the reflected signal measured by a vector network analyzer (VNA), from which the material's permittivity as a function of frequency is found. A prior calibration of the probe and VNA is required to extract the permittivity response of the material from the measured data. In this work, the coaxial probes were calibrated using air, short-termination, and distilled water measurements.

Open-ended coaxial probes from the Keysight N1501A Probe Kit are used to measure the complex permittivity of uniform sample materials over frequency at a constant temperature. The N1501A-104 probe, rated from 500 MHz to 40 GHz, is used to measure liquids, and the N1501A-104, rated from 500 MHz to 20 GHz, is used to measure solids. The probes are connected to a Keysight N5247A Vector Network Analyzer, which, prior to measurement, are calibrated based on air, short, and distilled water measurements at room temperature. An *xyz* probe connected to Fluke 80BK-A Multimeter monitors the temperature of the liquid samples so that the temperature can be recorded at the time of measurement. Liquid samples are heated to the desired temperature using a benchtop hotplate and cooled using an ice bath. Solids samples are only measured at room temperature.

Temperatures are measured to within whichever of the following is greater: ± 2.2 °C or 2% of the measured temperature. The measured ϵ' and ϵ'' have typical uncertainty within 5% of the complex permittivity magnitude [23].

For liquids, the probe requires a minimum of 5-mm probe insertion and 1 mm surrounding the probe. For solids, the probe requires the sample to have a minimum diameter of 20 mm and a thickness of $20/\sqrt{|\epsilon^*|}$ mm, where $|\epsilon^*|$ is the magnitude of the complex permittivity. Both probes used in this work require that materials have a tan $\delta > 0.05$.

Solid samples are prepared as cylindrical pucks of uniform materials that have a diameter of 25 mm and a thickness of 20 mm. Acrylic, nylon, polylactic acid (PLA), and Dremel ECO-ABS pucks are prepared using a Dremel 3D45 3-D printer. An additional acrylic puck is laser-cut from a cast sheet. A polydimethylsiloxane (PDMS) puck is prepared from a Sylgard 184 silicone elastomer kit, where the monomer and curing agent are mixed into a mold at a ratio of 10:1 and cured in the oven. Photographs of the solid material samples are shown in Fig. 2.

The Cole–Cole equation is an empirical model for the frequency response of the permittivity of liquids and polymers [24]

$$\epsilon^*(\omega) = \epsilon_{\infty} + \frac{\epsilon_s - \epsilon_{\infty}}{1 + (j\omega\tau)^{1-\alpha}} \tag{1}$$

where ϵ_s and ϵ_{∞} are the material's permittivities at zero frequency and infinite frequency, respectively, τ is the relaxation time constant, and α is a value with a range between 0 and 1. To fit the measured data to the Cole–Cole equation, a nonlinear least-squares analysis was carried out for the real and imaginary parts of the equation. The minimization



Fig. 2. Solid dielectric samples.

problem is expressed as

$$\min_{\boldsymbol{\beta}} \left(\sum_{i=1}^{n} ||\boldsymbol{r}_{i}||_{2}^{2} \right) = \min_{\boldsymbol{\beta}} \left(\sum_{i=1}^{n} ||\boldsymbol{\epsilon}_{i} - \boldsymbol{f}(\omega_{i}, \boldsymbol{\beta})||_{2}^{2} \right) \quad (2)$$

where β is the set of Cole–Cole parameters that minimize the sum, and r_i are vector residuals, defined as the difference between the measured data ϵ and the nonlinear vector model data $f(\omega_i, \beta)$. The nonlinear model vector function takes the form of the real and imaginary parts of the Cole–Cole equation, as shown in the following equation:

$$f(\omega, \boldsymbol{\beta}) = \begin{bmatrix} \operatorname{Re}\{f(\omega, \boldsymbol{\beta})\} \\ \operatorname{Im}\{f(\omega, \boldsymbol{\beta})\} \end{bmatrix}$$
$$f(\omega, \boldsymbol{\beta}) = \beta_1 + \frac{\beta_2 - \beta_1}{1 + (j\omega\beta_3)^{1 - \beta_4}}.$$
(3)

In a similar manner to the vector model function, the measured data are defined such that, for each frequency, there is a corresponding vector containing real and imaginary parts of the permittivity, as shown in the following equation:

$$(f_1, \epsilon_1), (f_2, \epsilon_2), \dots, (f_n, \epsilon_n)$$

$$\epsilon_i = \begin{bmatrix} \operatorname{Re}\{\epsilon_i^*\} \\ \operatorname{Im}\{\epsilon_i^*\} \end{bmatrix}.$$
(4)

The minimization problem is solved using a Trust Region algorithm [25], commonly used in optimization problems. The mean square error (mse) and normalized mean square error (NMSE) are computed to quantify the accuracy of each Cole–Cole model.

III. EXPERIMENTAL RESULTS AND DISCUSSION

The permittivity of the distilled H_2O was characterized to verify the measurement setup and the Cole–Cole parameter extraction methodology, as water has been extensively studied in the literature. Fig. 3 compares the measured and fit data produced here with the findings of Schwan [26], showing very good agreement.

A. Ethylene Glycol and Water Solutions

Binary ethylene glycol (1,2-ethanediol) and water solutions can be used for microfluidic electronics intended for outdoor or cold environment use. Ethylene glycol is used commonly as coolants and antifreezes. The freezing point of the solution follows an upward concave trend with solution

Work	Frequency	Temp.		Cole-Col	e Paramet	ers	Error Metrics		
	(GHz)	$(^{\circ}C)$	ϵ_{∞}	ϵ_s	au~(ps)	${lpha}$	MSE	NMSE	Max Mes. Err.
				H_2O	(100%)				
This work	0.5-40	25	5.10	79.0	8.64	0.0005	0.004	0.999	3.95
Schwan [26]	0.5 - 40	25	4.6	78.3	8.08	0.0014	-	-	-
			e	thylene g	lycol (35%))			
This Work	0.5-40	5	6.75	75.2	29.0	0.07	0.168	0.999	3.66
		25	6.33	69.6	17.3	0.081	0.437	0.998	3.40
			e	thylene g	lycol (50%))			
This Work	0.5-40	5	6.68	71.8	46.9	0.08	0.153	0.999	3.42
		25	6.90	69.5	20.7	0.08	0.345	0.999	3.38
			e	thylene g	lycol (65%))			
This Work	0.5-40	5	6.31	66.3	69.1	0.09	0.121	0.999	3.06
		25	7.04	66.0	29.4	0.09	0.278	0.999	3.18
			e	thylene g	lycol (80%))			
This Work	0.5-40	5	5.65	60.8	123	0.11	0.078	0.999	2.63
		25	7.51	56.7	69.9	0.07	0.105	0.999	2.66
			et	hylene gl	ycol (100%)			
This Work	0.5-40	5	4.86	51.6	253	0.13	0.044	0.998	1.86
		25	5.63	45.5	140	0.12	0.046	0.999	1.94
Gregory [28]	0.03-5	10	4.54	44.3	266	0.17	-	-	-
	0.03-5	25	4.70	40.75	134	0.14	-	-	-
Jordan [29]	0.1 - 70	10	4.1	45.5	-	-	-	-	-
	0.1 - 70	20	3.8	42.8	-	-	-	-	-

TABLE I $\label{eq:cole-cole} \mbox{Cole-Cole Parameters for Ethylene Glycol} \ (CH_2OH)_2 \ \mbox{Water Solutions} \ (\mbox{by \%})$



Fig. 3. Comparison of water models (T = 25 °C). Measurements and model from this work are shown as (o) and (–), respectively. Model by Schwan [26], shown as (--).

concentration [27]. The freezing points of the solutions presented here are approximately -22 °C, -38 °C, -52 °C, -43 °C, and -13 °C for volume ratio of ethylene glycol to water of 35%, 50%, 65%, 80%, and 100%, respectively. Prior studies on ethylene glycol reported the permittivity [28], [29], and the model results of those works are also presented for comparison.

The measured and modeled ϵ' , ϵ'' , and $\tan \delta$ of ethylene glycol by percent volume solutions are shown in Figs. 4–6. The extracted Cole–Cole model parameters for the solutions under test are tabulated in Table I. The Cole–Cole analytic models yield a close fit to the measured permittivity data of the solutions, evident by every each having NMSE metric near unity. The results for pure ethylene glycol are consistent with other works in the literature, and to the best of our knowledge, this is the first report of the Cole–Cole model parameters for ethylene glycol solutions of intermediate concentrations to 40 GHz.



Fig. 4. Comparison of the measured ϵ' to models of ethylene glycol and water solutions under test. Measurements are shown as (o) and (\diamond) and models as (–) and (–-) for T = 5 °C and T = 25 °C, respectively.



Fig. 5. Comparison of the measured ϵ'' to models of ethylene glycol and water solutions under test. Measurements are shown as (o) and (\diamond) and models as (–) and (–-) for T = 5 °C and T = 25 °C, respectively.

Solutions with lower ratios of ethylene glycol to water by volume produce relaxation processes that are closer to that of pure water, characteristic of their higher ϵ_s and smaller τ ,

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Work	Freq.	\mathbf{T}	Cole-Cole Parameters				Error Metrics			
	(GHz)	$(^{\circ}C)$	ϵ_{∞}	ϵ_s	au~(ps)	α	MSE	NMSE	Max. Mes. Err.	
Acetone (C ₃ H ₆ O)										
This work	0.5-40	5	3.40	21.8	3.16	0.00	0.156	0.988	1.13	
	0.5 - 40	25	1.47	21.17	2.51	0.00	0.110	0.990	1.08	
Onimisi [32]	0.1 - 10	10	8.69	22.24	92.2	0	-	-	-	
	0.1 - 10	20	4.55	21.13	40.5	0	-	-	-	
Gregory [28]	0.03 - 5	5	-	22.80	-	-	-	-	-	
	0.03 - 5	25	-	20.59	-	-	-	-	-	
			Ē	Ethyl Ace	tate (C ₄ H ₈	O ₂)				
This work	0.5-40	5	3.30	6.79	4.64	0.14	0.016	0.971	0.362	
	0.5 - 40	25	3.42	6.27	3.97	0.06	0.018	0.958	0.328	
Shrike [30]	0.01-20	15	1.98	6.27	12.56	0	-	-	-	
	0.01-20	25	1.92	6.06	11.21	0	-	-	-	
Isopropyl Alcohol (C ₃ H ₈ O)										
This work	0.5-40	5	3.06	30.51	660	0.12	0.023	0.989	0.718	
	0.5 - 40	25	3.25	21.15	273	0.10	0.016	0.995	0.786	
Sato [31]	0.1-89	25	2.48	19.34	-	-	-	-	-	
Gregory [28]	0.03-5	10	3.045	21.73	-	-	-	-	-	
	0.03-5	25	3.065	19.30	-	-	-	-	-	
Methyl Hydrate (CH ₃ OH)										
This work	0.5-40	5	6.12	36.66	70.19	0.04	0.119	0.997	0.362	
	0.5 - 40	25	6.01	34.01	52.68	0.039	0.107	0.997	0.328	
Jordan [29]	0.01-70	10	4.7	37.0	70	0.035	-	-	-	
	0.01-70	20	4.5	34.8	56	0.044	-	-	-	
Gregory [28]	0.03-5	10	5.82	35.74	70.36	0	-	-	-	
	0.03 - 5	25	5.56	32.66	50.67	0	-	-	_	

TABLE II Solvents



Fig. 6. Comparison of the measured tan δ to models of ethylene glycol and water solutions under test. Measurements are shown as (o) and (\diamond) and models as (–) and (–-) for T = 5 °C and T = 25 °C, respectively.

which is expected. Solutions with higher ratios of ethylene glycol to water by volume have a lower loss, due to lower ϵ_s and higher τ , which normally leads to a lower ϵ'' over frequency.

For all ethylene glycol solutions, ϵ_s has an inverse relationship with temperature, while the infinite frequency parameter ϵ_{∞} exhibits a weaker dependence on temperature compared with ϵ_s . The relaxation time constant τ shows a significant drop as the temperature increased for all cases. These Cole– Cole parameter temperature variations can easily be observed in Table I for all solutions. Smaller time constants broaden the dielectric relaxation process. This broadening is observed in Fig. 4, where all solutions' ϵ' have a more gradual descent with frequency for the higher temperature case. Similarly, in Fig. 5, all solutions ϵ'' peak in a broader crest for the higher temperature case.

It is notable that, at high frequencies, the ethylene glycol solutions have ideal electronic design qualities: stable ϵ' , minimal temperature variation, and minimum loss.



Fig. 7. Comparison of the measured ϵ' to models of solvents under test. Measurements are shown as (o) and (\diamond) and models as (–) and (--) for T = 5 °C and T = 25 °C, respectively.

Lower frequencies have additional design challenges, but narrow-band applications can take advantage of the high ϵ' without concern of the rapid variation in permittivity.

B. Solvents

Prior permittivity studies on solvents have resulted in a partial collection of model parameters over frequency, and some studies report on ϵ' but not ϵ'' [28]–[32]. In this work, a characterization of the permittivity of acetone (C₃H₆O), ethyl acetate (C₄H₈O₂), isopropyl alcohol (C₃H₈O), and methyl hydrate (CH₃OH) was carried out from 0.5 to 40 GHz, and a comprehensive set of the Cole–Cole parameters was found. Table II tabulates the Cole–Cole parameters from this work, and Figs. 7–9 shows the plots of ϵ' , ϵ'' , and tan δ , respectively.

Similar to the H_2O molecule, the isopropyl alcohol and methyl hydrate molecules have an orientational polarization and that impacts the relaxation process of these compounds,



Fig. 8. Comparison of the measured ϵ'' to models of solvents under test. Measurements are shown as (o) and (\diamond) and models as (–) and (--) for T = 5 °C and T = 25 °C, respectively.



Fig. 9. Comparison of the measured $\tan \delta$ to the models of solvents under test. Measurements are shown as (o) and (\diamond) and models as (–) and (--) for T = 5 °C and T = 25 °C, respectively.

which, in turn, manifests itself as a fairly pronounced variation of their permittivity over temperature and frequency [24]. Although the acetone and ethyl acetate molecules also have an orientational polarization, their dipole moment is weak, and hence, their permittivities exhibit only a weak variation with temperature and frequency.

For microfluidic electronics using dielectric fluids for tuning, the stable permittivity can result in more linear tuning around the nominal design frequency. Second, the relatively high permittivity, compared with the oils presented in Section III-C, can lead to a higher range of frequency tuning, as the effective wavelength near the dielectric fluid shortens comparatively more. The tradeoff here is the generally high loss tangent although ethyl acetate and acetone are observed to have low loss at lower frequencies.

C. Oils

Oils are of interest for microfluidic electronics because of their low loss. Castor oil ($C_{57}H_{104}O_9$) and liquid paraffin were selected for characterization from 25 °C to 50 °C, and their measured ϵ' is plotted in Figs. 10 and 11, respectively. The measured ϵ' of the oils is reported but not their ϵ'' because the loss tangent of these liquids is below the minimum rated tan δ sensitivity of the dielectric probes used in the experiments. Since generating the Cole–Cole model parameters requires accurate characterization of both ϵ' and ϵ'' , we do not report these parameters for the oils. The manufacturer's rated lower



Fig. 10. Measured ϵ' for castor oil. Maximum measurement uncertainty of ± 0.20 .



Fig. 11. Measured ϵ' for liquid paraffin. Maximum measurement uncertainty of ± 0.14 .

bound for the $\tan \delta$ sensitivity of the dielectric probes is 0.05 for solids and liquids.

Castor oil ($C_{57}H_{104}O_9$) is extracted from the castor plant (Ricinus communis) seed and is a raw material for paints, coatings, inks, lubricants, and many other products [33]. The permittivity of castor oil has been previously studied by Cataldo out to 1 GHz and Ramu out to 10 GHz [34], [35].

Liquid paraffin, commonly referred to as mineral oil, is a refined petroleum distillate with a wide range of uses, including pharmaceuticals, cosmetics, and lubricants. The permittivity of liquid paraffin has been studied at low frequencies for use as an insulator in power transformers [36], [37].

Both castor oil and liquid paraffin were observed to have some variation of ϵ' with frequency, particularly at the lower end of the band measured. Although, in general, both oils are observed to have broadband stable ϵ' , they have some sensitivity to temperature. Unlike the other dielectric fluids measured in this work, the shape of the dielectric response over frequency for oils is not altered by temperature; only an upward vertical shift is observed with increasing temperature.

D. Polymers

Polymer materials used as 3-D-printing filaments have a relatively low ϵ' and are increasingly being used to make antenna structures and antenna mounts in place of metal parts in antenna measurement systems to minimize undesired reflections. Fig. 12 shows the plots of the measured ϵ' of the materials characterized in this work: nylon, 3-D-printed

TABLE	III
PDMS	5

Work	Freq.	T	(Cole-Col	e Paramete	ers	Error Metrics			
	(GHz)	$(^{\circ}C)$	ϵ_{∞}	ϵ_s	au~(ps)	lpha	MSE	NMSE	Max. Mes. Err.	
This work	0.5-20	25	2.49	3.15	8.36	0.179	0.003	0.801	0.157	



Fig. 12. Measured ϵ' for polymers. Maximum measurement uncertainties are follows: acrylic (cast) ± 0.15 acrylic (3-D-printed) ± 0.14 ECO-ABS ± 0.12 nylon ± 0.14 PDMS ± 0.16 PLA ± 0.11 .



Fig. 13. Comparison of the measured ϵ' and ϵ'' to model response for PDMS.

acrylic, cast acrylic, ECO-ABS, PLA, and PDMS. Except for PDMS, the loss tangent of the tested polymers was below the dielectric probe's minimum tan δ sensitivity specification of 0.05. Therefore, the only material for which ϵ'' is plotted is for PDMS in Fig. 13.

The measured ϵ' of PDMS is in good agreement with previous studies [38]. Here, we report on, for the first time, the Cole–Cole parameters for PDMS to 20 GHz in Table III. Fig. 13 presents a comparison between the PDMS Cole–Cole model and its measured complex permittivity response.

IV. CONCLUSION

This work provides new permittivity characterization results and analytic model parameters for a multiplicity of fluids and solids that are of interest in the field of microfluidic electronics. Dielectric relaxation models are presented for several solvents, water solutions, oils, and plastic polymers over various temperatures. Novel models are produced for binary ethylene glycol–water solutions and PDMS. The temperature response of the permittivity for several of the compounds was also studied. All models were characterized using data from physical measurements carried out using the coaxial probe method. The analytic expressions are useful in simulation tools as they generate material properties at the desired operating points, either discrete or over a range.

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