

Temperature and Frequency Dependent Empirical Models of Dielectric Properties of Sunflower and Olive Oil

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Abstract. *In this article, a known concept and measurement probe geometry for the estimation of the dielectric properties of oils have been adapted. The new probe enables the measurement in the frequency range of 1 to 3000 MHz. Additionally, the measurement probe has been equipped with a heat exchanger, which has enabled us to measure the dielectric properties of sunflower and olive oil as well as of two commercial emulsion concentrates. Subsequently, corresponding linear empirical temperature and frequency dependent models of the dielectric properties of the above mentioned oils and concentrates have been created. The dielectric properties measured here as well as the values obtained based on the empirical models created here match the data published in professional literature very well.*

Keywords

Dielectric properties measurements, low-loss liquids, temperature and frequency dependent empirical models.

1. Introduction

Electromagnetic (EM) field has found its usage in many industrial and medical applications (e.g. [1–6]).

A new field of application of microwave (MW) power could be high-power MW-field-induced separation of Oil and Water (O-W) emulsions [7]. Oil-in-Water (O/W) emulsions, often used as cutting fluids (also called coolants or lubricants), represent an environmental risk when disposed of. Petrochemistry, automobile and metalworking industry produce large quantity of these emulsions [7]. They are hard to dispose of and the conventional separation process involves the use of environmentally harmful chemicals. Accordingly, there is a clear demand for a chemical free and environmentally friendly process.

Results of laboratory and field tests have shown that high-power MW-field-induced separation of Oil and Water (O-W) emulsions has a potential to become [8] a chemical-free alternative to the conventional separation process. The separation mechanism of this process has not been fully

understood yet and there is a need for further research. To study the separation mechanism, the following two physical phenomena have to be considered: dielectric heating and EM induction of interdroplet forces. In both phenomena, the dielectric properties of the two main emulsion components, water and oil, play an important role. While a temperature and frequency dependent model of dielectric properties of water already exists [9], a lack of professional literature dedicated to the topic of dielectric properties of oils was discovered. A brief literature survey follows.

In [10], dielectric properties of 11 commercially available cooking oils (measurements of the dielectric properties of sunflower and olive oil were not included) were presented for three frequencies and for three temperatures: 300 MHz, 1 and 3 GHz and 25, 50 and 80 °C, respectively. Dielectric properties of one of the oils were measured at 25 °C over a broad frequency range (100 Hz - 100 GHz) and the relaxation frequencies were found in the frequency range between 100 and 1000 MHz.

In [11], dielectric properties of 10 edible oils (including olive and sunflower oil) and 6 fatty acids, the main components of the edible oils, were measured over the frequency range from 100 Hz to 1 MHz. The dependency of the dielectric properties of the studied liquids at different temperatures (20 - 45 °C) and moisture content (0.02-0.31 %) was investigated as well. The dielectric constant of all studied liquids exhibits the same frequency dependency. It is constant from 100 Hz up to 500 Hz and for higher frequencies it starts to decrease. The authors correlated the dielectric constant of the oils with those of the fatty acids and with their fatty acid content. For frequencies up to 13.2 kHz, the loss factor decreases, for higher frequencies it increases. In the studied frequency range, the difference between the loss factors of the tested fluids was too small to differentiate them clearly from each other.

In [12], the collection of dielectric data for a wide range of organic solvents commonly used in microwave synthesis were presented. The approach used in [12] utilized the well-known Cole-Cole model [13] of complex permittivity ϵ^*

$$\epsilon^* = \epsilon_\infty + \frac{\epsilon_s - \epsilon_\infty}{1 + \left(i\frac{f}{f_r}\right)^{1-\beta}} + i\frac{\sigma_s}{\epsilon_0 2\pi f} \quad (1)$$

where ϵ_s is the static permittivity, ϵ_∞ is the permittivity at extremely high frequencies, f_r is the relaxation frequency, β is the dispersion parameter, ϵ_0 is the permittivity of vacuum, and σ_s is the static electrical conductivity (please note that σ_s is typically very small for oils. Dielectric losses are much higher than resistive losses in the assumed frequency range, and therefore σ_s is neglected here). According to [12], fitting of the Cole-Cole model to measured data of complex dielectric permittivity at a range of 300 kHz to 20 GHz was done with $\epsilon_\infty = 3$. This value was an average of the ϵ_∞ of the studied organic compounds and the determination of relaxation time τ and ϵ_s was not sensitive to the value of ϵ_∞ .

In [14], ten vegetable oils (included a sunflower and 5 different olive oils) were characterized with time domain reflectometry (TDR) (the probe was a section of a coaxial transmission line filled with the liquid under test and short circuited at one end) and specific data processing. The data obtained from the time domain approach was converted into frequency dependent dielectric properties from which the parameters of the Cole-Cole model were extracted for a single temperature, namely 20 °C. To further improve the accuracy of this method, the authors extracted ϵ_s first using LCR analysis (around 1 kHz) and the remaining three Cole-Cole model parameters were determined using the data (in the frequency domain from 0 to 1 GHz) from TDR. The Cole-Cole model parameter that varied the most among the tested oils was the relaxation frequency.

In this article, the dielectric properties of sunflower and olive oil as well as of two commercial emulsion concentrates were measured in the temperature and frequency range from 10 °C to 70 °C and from 1 MHz to 3 GHz, respectively. Instead of a commercial measurement system, the Agilent Dielectric Probe Kit (DPK) [16], due to reasons stated in Section 2, a measurement probe and a procedure (presented in [15] and expanded upon in more detail here) adapted from [14] were used. A description of the probe used here is presented in Subsection 2.1. Empirical models corresponding to the measured data were created and they are described in Section 3. The measured data is compared to the data published in professional literature. In this manner, this article partly alleviates the lack of published literature on temperature and frequency dependent dielectric properties of oils.

2. Measurements of the Dielectric Properties of Oils

The DPK is widely accepted for the measurements of the dielectric properties of liquids. Unfortunately, due to the probe's small outer diameter (open-ended coaxial Slim form probe 85070E-030), the DPK is not suitable for the measurements of dielectric properties at frequencies under 500 MHz [16]. The limitations of the open-ended coaxial probes at low frequencies are discussed in [17]. Furthermore, according to [16], the minimum loss tangent of

the measured liquids is $\tan\delta = 0.05$. For a dielectric material with $\epsilon'_r = 3$ (a value close to the typical value of ϵ'_r for oils), this corresponds to an imaginary part of complex relative permittivity equal to $\epsilon''_r = 0.15$. ϵ''_r of oils is often below 0.15. Therefore, the measurement of the dielectric properties of low loss liquids such as oils with the DPK at frequencies below 500 MHz would disregard two restrictions of [16] simultaneously.

2.1 Measurement Probe and Procedure

For the characterization of the dielectric properties of the emulsion concentrates, a coaxial measurement probe was built and tested. The main part of the is the coaxial transmission-line section (the length ℓ of the transmission-line section is equal to 100 mm) filled with the measured liquid and short circuited at one end. Instead of the evaluation of dielectric properties of the Liquid Under Test (LUT) for every frequency point, the general model of dielectric properties of liquids, the Cole-Cole model [18] consisting of four real parameters, was used and these parameters were extracted directly. This procedure, the probe geometry and performance evaluation were adapted with a number of modifications from Cataldo et.al. [14]. Compared with [14], the main differences in geometry, the equivalent circuit and the measurement method used in this article are:

1. The cross-section dimensions of the probe used here are given by the 7/16 coaxial standard instead of SMA. Here, the inner and the outer radii of the measurement coaxial probe are 3.5 and 8 mm, respectively. This simplifies filling and emptying of the probe with high viscosity liquids and it also makes the removal of possible air bubbles in the liquid before measurement easier.
2. Between the measurement reference plane and the probe input (the interface of the LUT region and PTFE), there are two different adapters (SMA to N, N to 7/16) and a 7/16 connector. The two adapters and the connector were measured and characterized separately using the necessary auxiliary structures. The influence of the two adapters and the connector located between the measurement/calibration reference plane and the input of the probe (the interface of the connector and the LUT) has been removed from the measurements using a de-embedding procedure [19]. The mathematical model of the measurement probe used here thus does not have to describe the effects of the adapters and consists of less parameters that need to be determined by fitting.
3. The equivalent circuit model of the probe consists of a transmission line model and an equivalent model of the short-circuit (see Fig. 2). In [14], additional circuit elements are used at the input of the probe. The reflection coefficient of the probe equivalent circuit model is denoted here as $\underline{S}_{11,\text{mod}}$.

4. The reflection coefficient was measured directly using VNA instead of TDR measurements and conversions to S-parameter using FFT. The VNA (Agilent 8720A) was calibrated using the full two-port calibration procedure and SMA female mechanical calibration standards: short, open, load and through (Agilent 85052D) at the measurement reference plane.
5. The probe presented here was equipped with a heat exchanger, which allowed the measurement of dielectric parameters at precisely set temperatures.
6. Poor contact of the inner and outer conductor, especially along the junction between the short-circuit metal plane and the outer conductor, could possibly lead to the excitation of higher order modes in the measurement probe. Special care was paid here to this problem and the design of the short-circuit geometry ensures that the above mentioned contact is good (see Fig. 1).

The measurement setup was as follows: the VNA was connected to the measurement probe with a phase-stable coaxial measurement cable. The measurement cable was mechanically fixed during and between the calibration and the measurements (in order to avoid the phase shift of the measured reflection coefficient $S_{11,meas}$ due to cable banding) the calibration and the measurements. The VNA was calibrated at the end of the measurement cable (measurement reference plane). The measurement probe was placed vertically as depicted in 1 a).

The values of the equivalent circuit elements of the non-ideal short-circuit (topology of the short circuit was adapted from [14]), R_1 , R_2 and L , were determined by the fitting of the measured reflection coefficient $S_{11,meas}$ of the probe filled with pure (99.9 %) standard liquid, namely ethyl acetate, with $S_{11,mod}$. For this purpose, the known Cole-Cole parameters of the standard liquid were inserted into the mathematical model and the only variables were R_1 , R_2 and L . Ethyl acetate was used for this purpose by virtue of its following special dielectric properties in the considered frequency range. The real part of its complex permittivity is almost constant and the imaginary part, due to the high relaxation frequency, is very low¹. Therefore, by performing the evaluation of R_1 , R_2 and L values, the main losses of the probe are due to the short circuit.

2.2 Measurements and Results

At the very beginning of the evaluation of the Cole-Cole parameters of the LUT with the new probe, control and adjustment of the LUT temperature was not used. The temperature of the measurement laboratory was considered to be exactly 20 °C. A noticeable mismatch was shown between the computed and the measured reflection coefficient and this mismatch could not be compensated by the modi-

¹compared with the other two available pure liquids, ethanol and 2-propanol

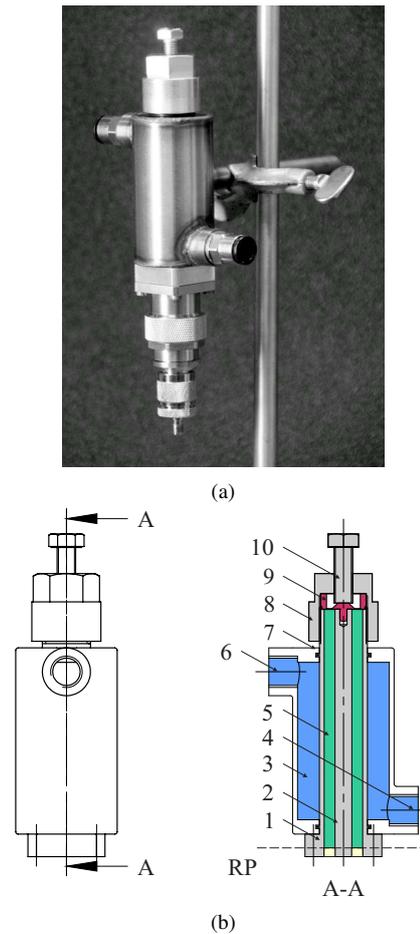


Fig. 1. Picture of the probe with the heat exchanger (a). During the measurement, the probe was equipped with a thermal insulation, which is omitted here in order to show the probe's geometry. Simplified technical drawing and section of the measurement probe with the heat exchanger (b). (1) and (2) are the outer and the inner conductor of the coaxial probe, respectively, (3) water, (4) water inlet, (5) LUT, (6) water outlet, (7) heat exchanger, (8) nut, (9) short-circuit, (10) bolt.

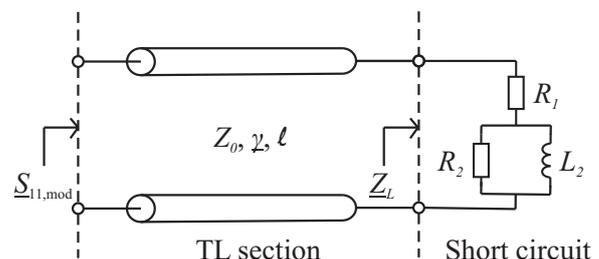


Fig. 2. The equivalent-circuit model of the measurement probe, where l, Z_0, γ is the physical length, characteristic impedance and the complex propagation constant of the coaxil transmission line [22], respectively.

fication of the values of R_1 , R_2 and L alone. The mismatch disappeared with the addition of a fourth fitting parameter T (temperature). It turned out that the value of T , obtained by fitting, was slightly higher, by about 0.6 °C, than the considered laboratory temperature. After ensuring that the tem-

perature of the LUT was constant, using the heat exchanger, the evaluated values of Cole-Cole parameters of the standard liquids agreed well with the values listed in [20, 21], see Tab. 1.

The LUT temperature control proved to be very important. For the measurement of dielectric properties of the LUT at a certain temperature, the following approach was used:

1. The probe was filled with ethyl acetate and was heated/cooled to the desired temperature.
2. The reflection coefficient was measured.
3. The measurements of the reflection coefficient of the probe filled with ethyl acetate were used for the evaluation of R_1 , R_2 , L and the actual temperature T of the LUT.
4. The steps 1 to 2 were repeated for 2-propanol and for liquids of unknown dielectric properties.
5. The evaluated Cole-Cole parameters (using the values of R_1 , R_2 , L determined in the 3rd step) of 2-propanol were compared with the values from [20,21] and if the values matched, the Cole-Cole parameters of the liquids of unknown dielectric properties were evaluated as well.

The evaluation of the Cole-Cole parameters of LUT is analogical to the evaluation of the values of R_1 , R_2 and L . Again the measured reflection coefficient $\underline{S}_{11,meas}$ of the probe filled with LUT was fitted with $\underline{S}_{11,mod}$ [14]. For this purpose, the known values of R_1 , R_2 and L were inserted into the mathematical model and the only remaining variables were the Cole-Cole parameters [14]. The MATLAB[®] curve fitting procedure "lsqcurvefit" (based on the trust-region-reflective algorithm) has been used.

In order to verify the presented measurement approach, the Cole-Cole parameters of two pure (99.9 %) liquids with well known dielectric properties, namely ethanol and 2-propanol, were measured. The Cole-Cole parameters evaluated here using the measurement probe and procedure, described in Section 2.1, are compared to the values published in literature [20,21] in Tab. 1. The compared data match very well, which confirms that the measurement probe works and the procedure is valid.

Please note that the thermal expansion of the measured liquids as well as of the probe parts has been considered and estimated. The thermal expansion has been neglected here, due to negligible values of the estimated decreased/increased volumes/lengths. Furthermore, the probe and the LUT had been partly precooled/preheated before the upper cap of the probe has been closed and the measurement performed. The measurement probe is not pressure tight and so it can be assumed that a potential expanded LUT volume would probably rise up above the upper cap. But no such effect has been observed during the measurements.

Ethanol			
Parameter	Reference values		Values measured here
	[20]	[21]	
ϵ_s [-]	25.16 ± 0.04	25.07	25.103 ± 0.014
ϵ_∞ [-]	4.486 ± 0.025	4.2	3.709 ± 0.089
f_r [MHz]	834 ± 4	1110	873.0 ± 10.0
β [-]	not available	0	0.0240 ± 0.0045
2-propanol			
Parameter	Reference values		Values measured here
	[20]	[21]	
ϵ_s [-]	20.26 ± 0.06	19.0	20.487 ± 0.089
ϵ_∞ [-]	3.447 ± 0.016	3.2	3.321 ± 0.030
f_r [MHz]	358 ± 2	545	354.9 ± 5.2
β [-]	not available	0	0.0385 ± 0.0025

Tab. 1. Comparison between the Cole-Cole parameters of ethanol and 2-propanol at 20 °C evaluated here and published in [20, 21]. The values of the short-circuit equivalent-circuit elements are $R_1 = 1.17 \cdot 10^{-5} \Omega$, $R_2 = 4.84 \Omega$ and $L = 0.084 \text{ nH}$.

Parameter	Sunflower oil		Olive oil	
	a	b	a	b
ϵ_s [-]	$-5.073 \cdot 10^{-3}$	3.334	$-4.706 \cdot 10^{-3}$	3.232
ϵ_∞ [-]	$4.559 \cdot 10^{-4}$	2.266	$2.690 \cdot 10^{-4}$	2.269
f_r [GHz]	$1.755 \cdot 10^7$	$7.332 \cdot 10^6$	$1.627 \cdot 10^7$	$1.254 \cdot 10^6$
β [-]	$-1.294 \cdot 10^{-3}$	$5.405 \cdot 10^{-1}$	$-1.177 \cdot 10^{-3}$	$5.295 \cdot 10^{-1}$
Parameter	Emulsion A concentrate		Emulsion G concentrate	
	a	b	a	b
ϵ_s [-]	$-3.765 \cdot 10^{-3}$	3.252	$-3.809 \cdot 10^{-4}$	3.482
ϵ_∞ [-]	$-3.996 \cdot 10^{-4}$	2.262	$9.891 \cdot 10^{-5}$	2.336
f_r [GHz]	$1.243 \cdot 10^7$	$-3.059 \cdot 10^7$	$1.160 \cdot 10^7$	$7.824 \cdot 10^7$
β [-]	$-4.385 \cdot 10^{-4}$	$5.589 \cdot 10^{-1}$	$9.214 \cdot 10^{-6}$	$4.318 \cdot 10^{-1}$

Tab. 2. Linear function coefficients of temperature dependent Cole-Cole parameters $f(T) = aT + b$ of the measured edible oils and emulsion concentrates. Temperature T is given in [°C].

3. Empirical Models

The Cole-Cole model is a frequency dependent model of the dielectric properties of liquids at a certain temperature. Cole-Cole parameters of four different oils were evaluated based on the measurements at five different temperatures: 10, 20, 40, 60, 70 °C. For every liquid and every temperature, three independent measurements were performed. Since the evaluated Cole-Cole parameters showed an almost linear dependency on temperature, their temperature dependencies were approximated with linear functions [23, 24]. The coefficients of these linear functions are listed in Tab. 2. In Figs. 3 and 4, the Cole-Cole parameters evaluated here are plotted with the corresponding error bars and linear models. Error bars represent 3σ , where σ is the standard deviation computed from the data obtained by three independent measurements. As in [12], from all four Cole-Cole parame-

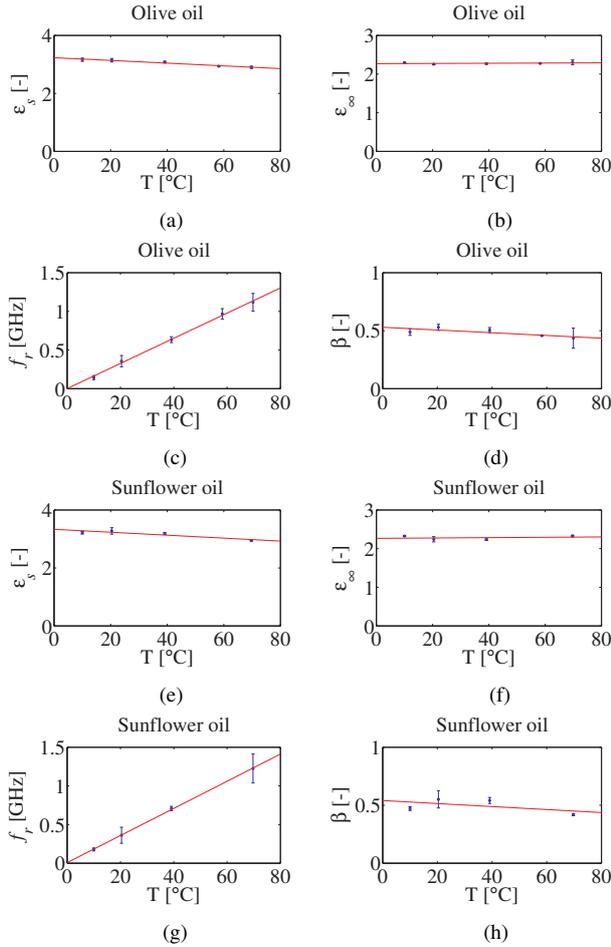


Fig. 3. Temperature dependent Cole-Cole parameters (a) ϵ_s , (b) ϵ_∞ , (c) f_r and (d) β of olive oil and (e) ϵ_s , (f) ϵ_∞ , (g) f_r and (h) β of sunflower oil. Dots, error bars and solid lines represent measured values, 3σ and linear models, respectively.

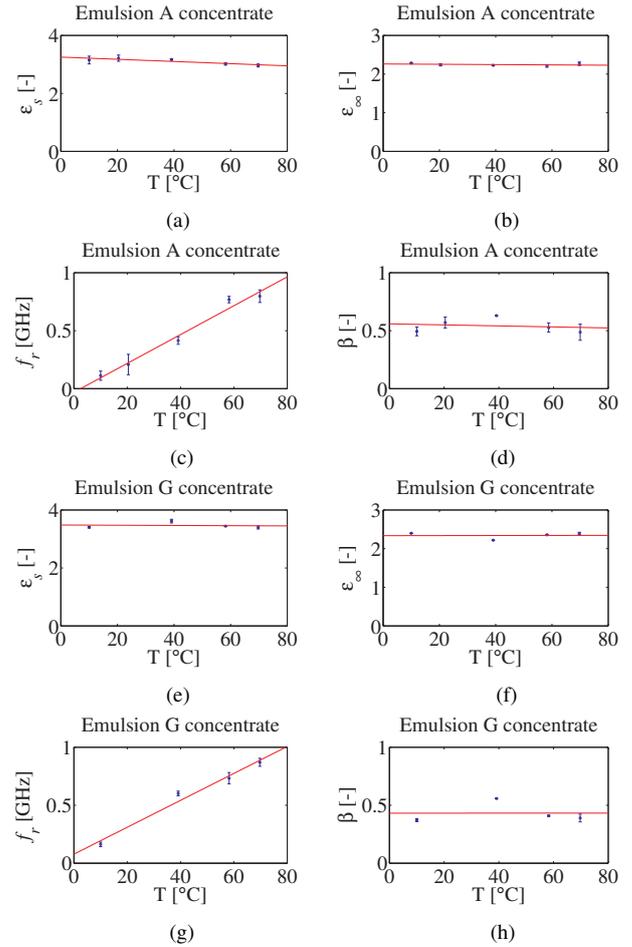


Fig. 4. Temperature dependent Cole-Cole parameters (a) ϵ_s , (b) ϵ_∞ , (c) f_r and (d) β of Emulsion A concentrate (e) ϵ_s , (f) ϵ_∞ , (g) f_r and (h) β of Emulsion G concentrate. Dots, error bars and solid lines represent measured values, 3σ and linear models, respectively.

ters, the most temperature dependent one is f_r and as noticed in [12], this parameter increases linearly with temperature. The decrease of ϵ_s with increasing temperature is similar to that of water. The Cole-Cole parameter ϵ_∞ is independent of temperature as noticed in [12] and has almost the same value for both measured oils and emulsion concentrates.

Temperature and frequency dependent dielectric properties of the two edible oils and of the two emulsion concentrates evaluated from the empirical models created here are plotted in Fig. 6.

4. Comparison to Published Data

The values of static permittivity ϵ_s evaluated here match well the values published in [11]. The value of ϵ_s of olive and sunflower oil published in [11] is 3.07 and 3.065, respectively. Here, ϵ_s for both oils at the temperature $T = 25^\circ\text{C}$ is equal to 3.114 and the relative difference between the values published in [11] and here is approximately 1.5 %.

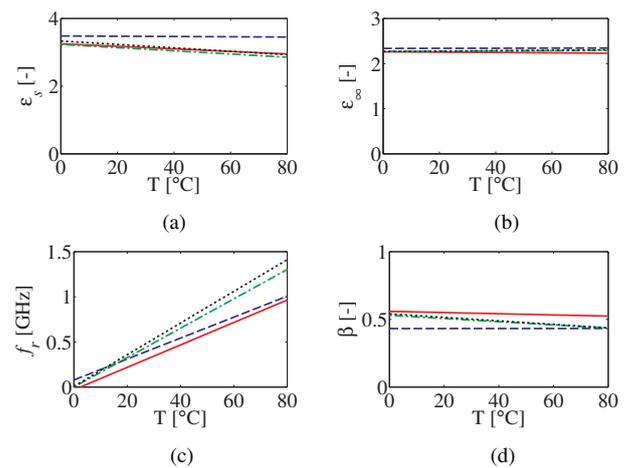


Fig. 5. Comparison of temperature dependent Cole-Cole parameters (a) ϵ_s , (b) ϵ_∞ , (c) f_r and (d) β of emulsion concentrates and edible oils. Solid, dashed, dotted and dash-dot lines correspond to emulsion concentrate A, emulsion concentrate B, sunflower oil and olive oil, respectively.

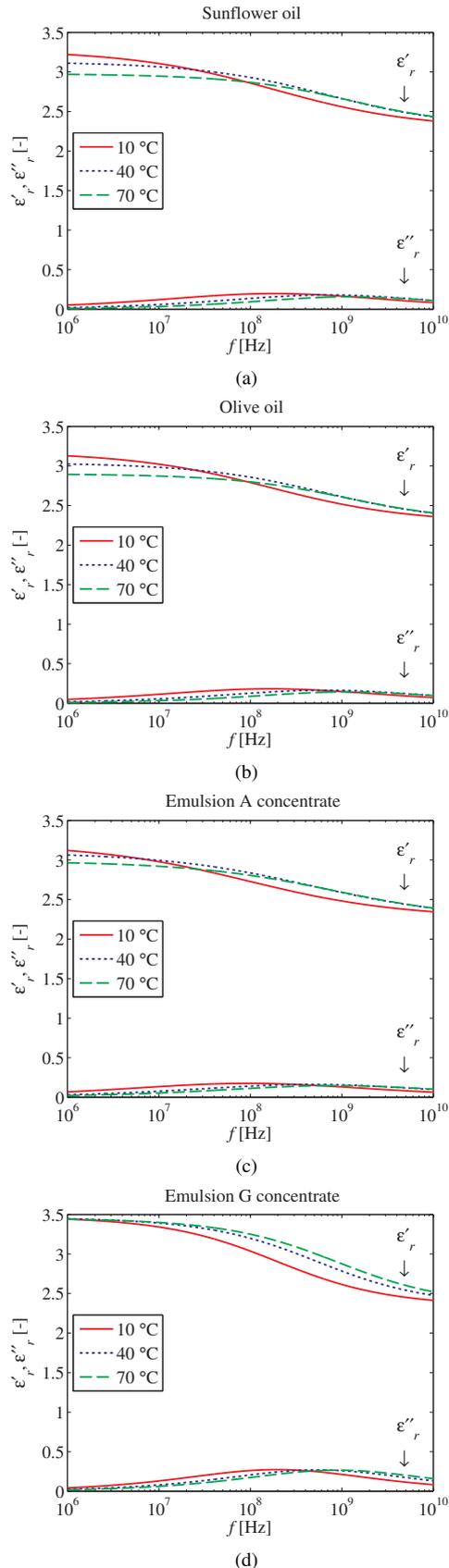


Fig. 6. Measured frequency and temperature dependent dielectric properties of edible oils: (a) sunflower oil, (b) olive oil and of emulsion concentrates: (c) Emulsion A concentrate, (d) Emulsion G concentrate.

In [11], the measured data of ϵ_s of soybean oil for 25 and 75 °C is available and it is 3.115 and 2.91, respectively. These values correspond well to the values of ϵ_s of oils measured here at these two temperatures, namely 3.114 and 2.88 (for both oils the same values of ϵ_s were measured). The relative difference between the values for $T = 75$ °C published here and in [11] is approximately 1 %.

At the temperature $T = 25$ °C, for all liquids measured here the relaxation frequency f_r lies in a frequency range of 250 to 500 MHz. This is consistent with the observation made in [10]. In [14] as well as here, the Cole-Cole model parameter that varied the most among the tested oils was the relaxation frequency f_r . The relaxation frequency shows a linear temperature dependence, which is consistent with an explanation presented in [12]. The Cole-Cole parameter ϵ_∞ evaluated here was almost the same for all four measured liquids and it has shown almost no dependence on temperature. This behavior was qualitatively explained in [12] as well.

Sunflower oil				
Cole-Cole model parameters				
Comment	ϵ_s [-]	ϵ_∞ [-]	f_r [MHz]	β [-]
evaluated in [14]	3.12	2.40	292	0.31
evaluated here	3.23	2.27	358	0.51
Olive oil				
Cole-Cole model parameters				
Comment	ϵ_s [-]	ϵ_∞ [-]	f_r [MHz]	β [-]
evaluated in [14]	3.08	2.39	315	0.33
(ac. = 0.3 %)				
evaluated in [14]	3.14	2.38	288	0.36
(ac. = 1.2 %)				
evaluated in [14]	3.19	2.36	259	0.44
(ac. = 1.6 %)				
evaluated in [14]	3.19	2.34	249	0.42
(ac. = 4.0 %)				
evaluated here	3.14	2.27	327	0.51

Tab. 3. Comparison between the values of Cole-Cole parameters of olive and sunflower oil evaluated here and in [14].

5. Summary

In this article, dielectric properties of sunflower and olive oil as well as of two commercial emulsion concentrates were measured in the temperature and frequency range from 10 to 70 °C and 1 to 3000 MHz, respectively, and corresponding empirical temperature and frequency models have been created. These models together with the existing frequency and temperature dependent model of the dielectric properties of water represent the necessary background for further analysis of the studied dielectric heating, emulsion separation, as well as for the study of effective dielectric properties of W-O emulsions. Furthermore, there is a continuously increasing number of users of multiphysical simulations and for some of them the empirical models presented here could be of great significance.

Acknowledgements

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