

Measurements of electric properties using an open-ended probe blocked by a non-conducting foil



Alicja Wolny*, Kazimierz Orzechowski, Marek Rudowski

Faculty of Chemistry, University of Wrocław, Joliot-Curie 14, 50-383 Wrocław, Poland

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ABSTRACT

An open-ended coaxial probe separated from the investigated medium by a thin non-conducting layer (foil) is proposed for making dielectric permittivity measurements within the RF range. The method was originally applied by the authors for intraoperative tissue assessment, but it could also be suitable for food control (requirement of purity) or measurements in aggressive media (protection from corrosion).

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Introduction

The measurements of dielectric properties of materials play an important role in quality control, for example of petrol products [1], in nanotechnology [2], biotechnology [3,4] food control [5,6], biomedical applications for tissue characterization [7–11], and even intraoperative applications for cancer detection [12–14]. In many cases the investigated materials demonstrate a high electrical conductivity, which leads to the considerable difficulties caused by ion migration and a strong electrode polarization effect. There are many methods to subtract or diminish the electrode polarization effect: fitting of theoretically predicted equations considering a constant phase angle element in series with the sample admittance [15], modification of the electrode distance [16] or surface [17], and the use of the four-electrode method [18]. A comprehensive discussion of these questions can be found in reviews [19,20].

This paper describes a method called “the blocking of electrodes”, which consists in covering the surface of the electrodes by a layer of isolating material, separating the investigated sample and the electrodes [21–23]. Covering the electrodes has a double effect: 1/suppress the electrode polarization, 2/isolates the sensor from the investigated medium. The additional, possibly a sterile layer, could be interesting in applications, especially in food control and in medicine.

Unfortunately, the non-conducting layer covering the electrodes significantly affects the dielectric response. In this paper the electric properties of an open-ended (OE) coaxial probe and a

blocked open-ended (BEO) coaxial probe will be compared. A blocking layer is formed by polyethylene foil located between the investigated sample and the probe. The probe was tested in standard liquids of relative electric permittivity from 1.9 to 80 and in NaCl + water solution of concentration from 0.0025 mol/dm³ to 0.5 mol/dm³. The blocked open-ended probe was already used for the intraoperative detection of cancer lesions in breast-cancer surgery [13,24].

Experimental

The coaxial open-ended probe (Fig. 1) is made from gold-plated brass and Teflon placed between the inner and outer electrodes. The geometry was fitted to 50 ohm impedance calculated according to the formula [25]:

$$Z_0[\Omega] = \frac{60}{\sqrt{\epsilon_{sep}}} \ln \frac{a}{b} \quad (1)$$

where ϵ_{sep} is the permittivity of the material separating the electrodes (Teflon in our case), a, b – diameter of the outer (9.60 mm) and the inner (2.95 mm) electrode. The probe is equipped with an APC-7 connector for direct connection to the impedance analyzer. In experiments, 4191A RF Impedance Analyzer, operating in the frequency range of 1 MHz–1 GHz, was used. Before taking measurements the analyzer was calibrated according to the manufacturer's manual using the 0 Ω , 0 S and 50 Ω termination mounted on the end of the open-ended probe (OE). In the frequency range 1–5 MHz the relative error of capacity measurements is estimated as 2%, in the frequency range 5 MHz–1 GHz as 1%. During the tests the open-ended probe was in a vertical position with the

* Corresponding author.

E-mail address: alicja.wolny@chem.uni.wroc.pl (A. Wolny).

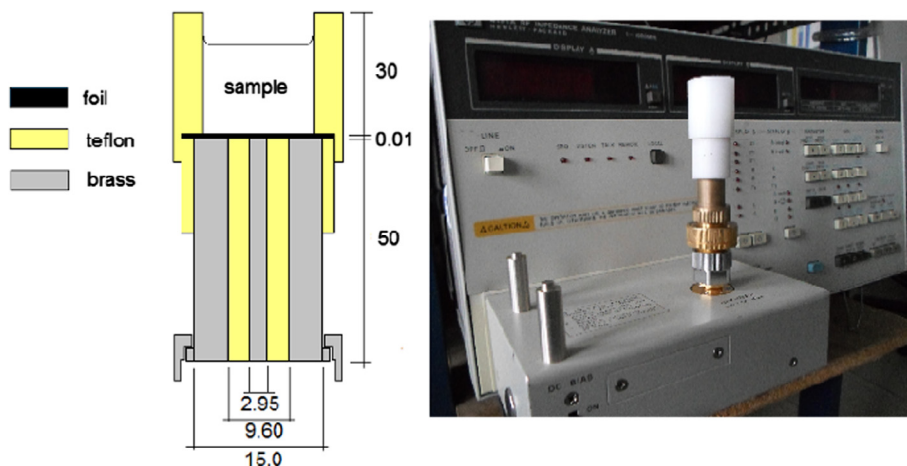


Fig. 1. The cross-section of the open-ended probe blocked with polyethylene foil (dimensions are in millimeters) and the photo of experimental set-up.

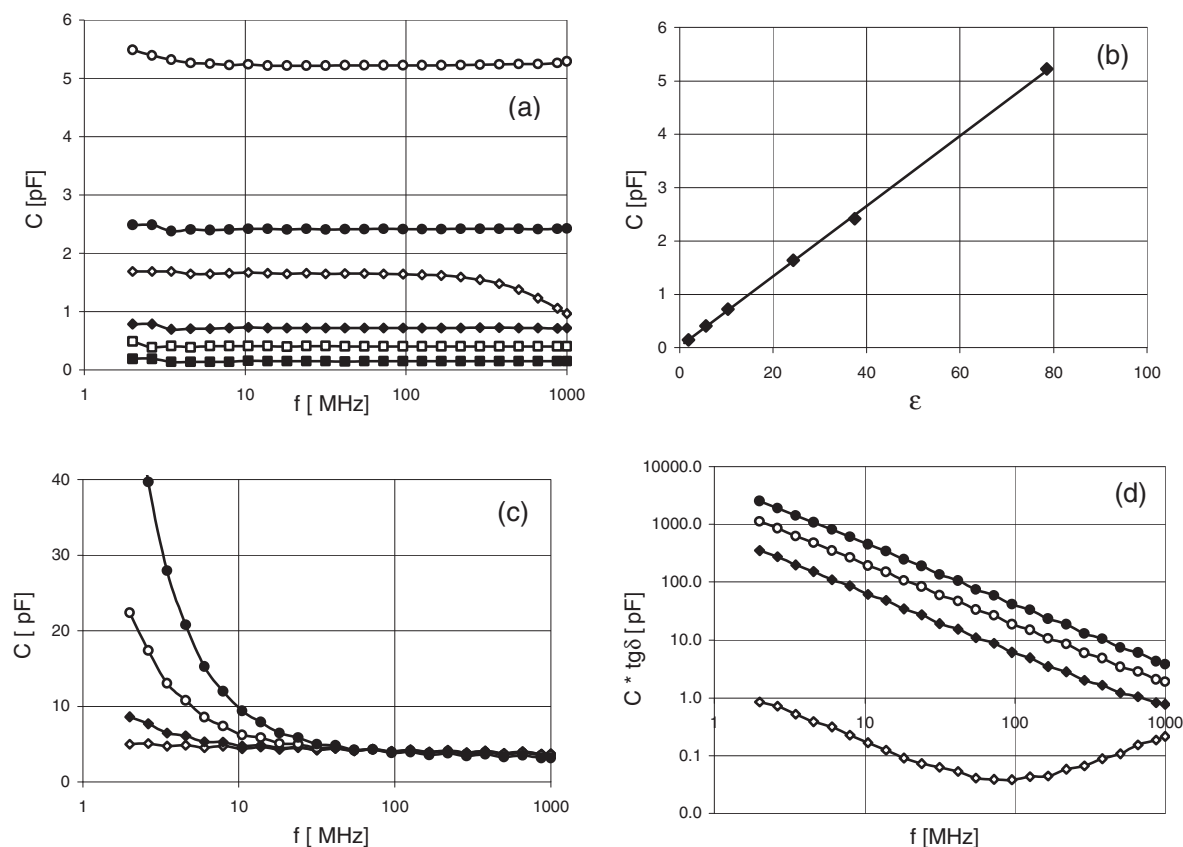


Fig. 2. Measurements performed in standard liquids by the open-ended probe. (a) capacity measured as a function of frequency in hexane (black squares), chlorobenzene (empty squares), 1,2-dichloroethane (black triangles), ethanol (empty triangles), acetonitrile (black circles), water (empty circles); (b) capacity obtained at 100 MHz as a function of permittivity; (c) and (d) capacity and the product of capacity and loss factor ($C \cdot \tan \delta$) measured as a function of frequency in NaCl + water solutions of concentrations: 0 mol/dm³ (white diamonds), 0.05 mol/dm³ (empty diamonds), 0.2 mol/dm³ (empty circles), 0.5 mol/dm³ (black circles).

sample located on the top (Fig. 1). In experiments with the blocked probe the foil was 0.01 mm thick. In experiments with the non-blocked probe, the foil was removed. Measurements were done at room temperature (25 ± 2 °C). Good quality chemicals (pure for analysis) were used as delivered.

Results and discussion

In order to determine the effect of blocking of an open-ended probe by a non-conducting layer the measurements performed

by the open-ended (OE) and blocked open-ended (BOE) probes were compared. The results are presented in Fig. 2a–d and Fig. 3a–d. In the case of the OE probe for liquids of low and moderate conductivity the measured capacity does not depend on frequency, provided that the permittivity of the tested liquid is constant in the investigated frequency range. A decrease in capacity in ethanol ($f > 200$ MHz) is related to the dipolar relaxation observed in this frequency range. Fig. 2b shows that the capacity increases linearly along with an increase in the permittivity of standard liquids, which proves that the geometrical capacity (C_0)

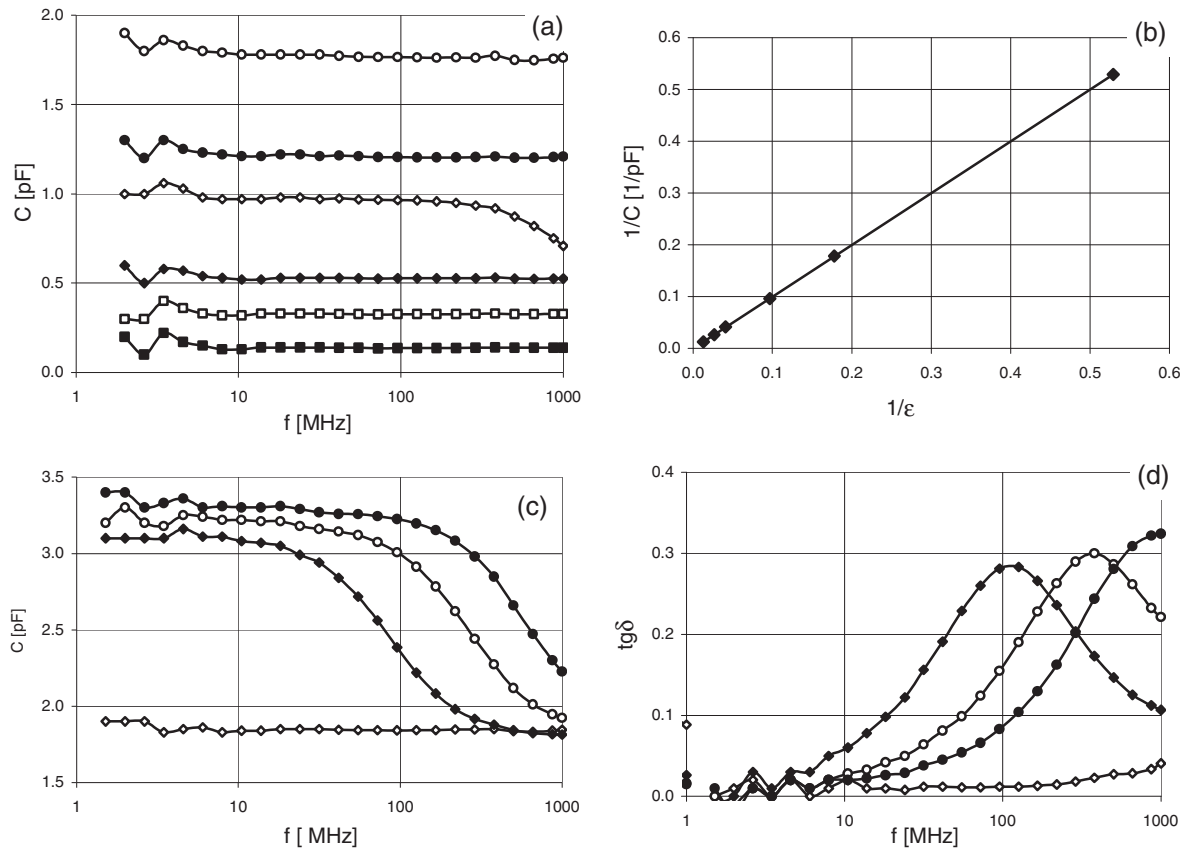


Fig. 3. Measurements performed in standard liquids by a blocked open-ended probe: (a) capacity measured as a function of frequency in hexane (black squares), chlorobenzene (empty squares), 1,2-dichloroethane (black triangles), ethanol (empty triangles), acetonitrile (black circles), water (empty circles); (b) inverse of capacity obtained at 100 MHz as a function of inverse of permittivity; (c) and (d) capacity and the loss tangent measured as a function of frequency in NaCl + water solutions of concentrations: 0 mol/dm³ (empty diamonds), 0.05 mol/dm³ (black diamonds), 0.2 mol/dm³ (empty circles), 0.5 mol/dm³ (black circles).

is constant and fringe capacities are negligible. Fig. 2c presents the capacity obtained in solutions of NaCl in water. An increase in the conductivity results in a strong increase in the measured capacity at low frequencies. This effect is related to the electrode polarization. The loss tangent in NaCl solutions investigated by the OE probe within the RF range is dominated by the DC conductivity (the product of capacity and loss tangent is linear in a double logarithmic scale, Fig. 2d).

The application of a non-conducting layer between the probe and the investigated medium (BOE probe) significantly affects the response. The results are presented in Fig. 3.

In liquids of low conductivity the capacity measured within the RF range is much lower than that in the measurements taken by means of a non-blocked probe and it does not depend on frequency (provided that the permittivity of the test liquid is constant) (Fig. 3a). Unlike the observations made for the non-blocked probe, the capacity is not proportional to the permittivity of standard liquids, but the inverse of capacity is proportional to the inverse of permittivity. In the measurements of electrolytes (water solutions of NaCl) the real and imaginary components of capacity show the dependence characteristic of a relaxation process. The relaxation frequency increases along with an increase in the conductivity of the investigated medium.

To explain the response observed in the case of the blocked probe, a simple reference circuit, presented in Fig. 4, was proposed.

The dielectric permittivity and conductivity of the investigated sample are represented by the capacity C_x (proportional to the permittivity) and the resistance R_x (proportional to the inverse of specific conductivity). In this simple model we consider that the sample possesses the DC resistivity, and we did not consider the

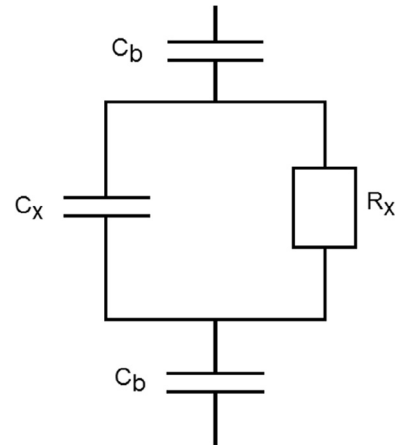


Fig. 4. The reference circuit of the blocked open-ended probe.

loses related to dipolar relaxation. The non-conducting polyethylene foil is represented by the blocking capacity C_b and negligible conductivity. The impedance of the reference circuit could be expressed as follows:

$$Z = \frac{1}{j\omega C_b} + \frac{1}{\frac{1}{R_x} + j\omega C_x} + \frac{1}{j\omega C_b} \quad (2)$$

and the admittance:

$$Y = \frac{1}{Z} = \omega C_t \text{tg}\delta + j\omega C \quad (3)$$

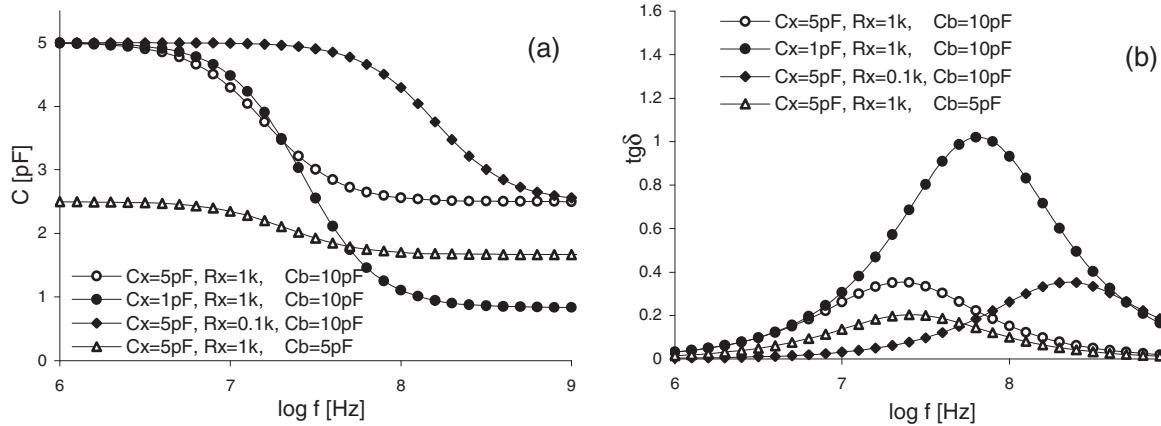


Fig. 5. Results of calculation of the capacity C and loss tangent $\text{tg}\delta$ for the selected R_x , C_x and C_b parameters, according to Eqs. (4) and (5).

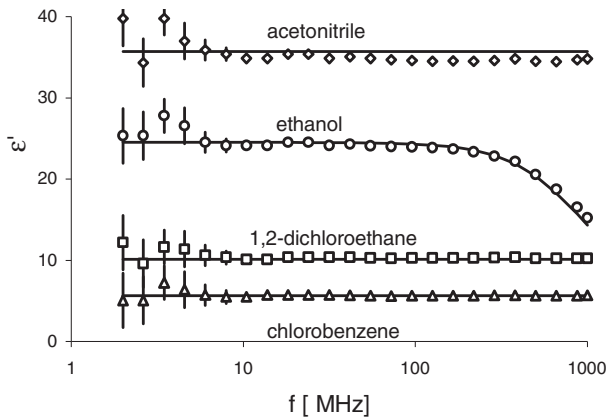


Fig. 6. The comparison of the electric permittivity measured by BOE probe and the literature data. Straight lines present literature data.

where

$$C = \frac{2C_b + \omega^2 C_x C_b R_x^2 (2C_x + C_b)}{4 + \omega^2 R_x^2 (2C_x + C_b)^2} \quad (4)$$

and

$$\text{tg}\delta = \frac{\omega C_b^2 R_x}{2C_b + \omega^2 C_x C_b R_x^2 (2C_x + C_b)} \quad (5)$$

It is convenient to analyze the properties of the circuit on the base of the calculated reference capacity $C(f)$ and loss tangent $\text{tg}\delta(f)$, by giving specific sets of values in Eqs. (4) and (5). Results are presented in Fig. 5.

An increase in C_x , reflecting an increase in the permittivity of the investigated medium (compare the curves $C_x = 1\text{ pF}$; $R_x = 1\text{ k}\Omega$; $C_b = 10\text{ pF}$ and $C_x = 5\text{ pF}$; $R_x = 1\text{ k}\Omega$; $C_b = 10\text{ pF}$), results in an increase in capacity in the high frequency region only. The absorption (presented as $\text{tg}\delta(f)$) decreases and is shifted to slightly lower frequencies. A decrease in the resistance R_x , reflecting an increase in the conductivity of the investigated medium (compare the curves $C_x = 5\text{ pF}$; $R_x = 1\text{ k}\Omega$; $C_b = 10\text{ pF}$ and $C_x = 5\text{ pF}$; $R_x = 0.1\text{ k}\Omega$; $C_b = 10\text{ pF}$), results in a shift of dispersion and absorption to higher frequencies. The low and high frequency capacity does not depend on R_x . A decrease in the blocking capacity C_b (compare the curves $C_x = 5\text{ pF}$; $R_x = 1\text{ k}\Omega$; $C_b = 10\text{ pF}$ and $C_x = 5\text{ pF}$; $R_x = 1\text{ k}\Omega$; $C_b = 5\text{ pF}$) results in a considerable decrease in capacity at low frequencies and a moderate decrease at high frequencies. The maximum of $\text{tg}\delta(f)$ is shifted to slightly higher frequencies. The shape of

experimental $C(f)$ and $\text{tg}\delta(f)$ curves, presented in Fig. 3, are similar to those obtained using the reference circuit.

By taking account of the properties of the reference circuit it is possible to explain the observed proportionality between the inverse of capacity and the inverse of permittivity of standard liquids (Fig. 3b). This is due to series connection of capacities C_b and C_x . According to Eq. (4), in the high frequency region the measured capacity C is a function of the capacity C_x (related to the permittivity of the investigated sample $C_x = C_0 \cdot \epsilon$) and the blocking capacity C_b :

$$\epsilon_x = \left(\frac{1}{C} - \frac{2}{C_b} \right)^{-1} C_0^{-1} = \left(\frac{A}{C} - B \right)^{-1}$$

where ϵ_x is the permittivity of the investigated sample, C – capacity of the BOE probe measured by the equipment, C_0 geometrical capacity of the open-ended probe, C_b – blocking capacity, A, B – constants attainable in the course of calibration. The measurements performed by us in pure test liquids (not in NaCl solutions) are located in high frequency region and could be used to test the BOE probe for permittivity estimation. Calibration constants A and B were obtained using the measured capacity of hexane and water at a single frequency of 100 MHz. These parameters were used to calculate the permittivity of chlorobenzene, 1,2-dichloroethane, ethyl alcohol and acetonitrile in the frequency range 2–1000 MHz. The results were compared with the literature data in Fig. 6.

For acetonitrile [26], 1,2-dichloroethane [27] and chlorobenzene [28] the dipolar relaxation is located at much higher frequencies and the obtained data were compared with the static dielectric constant of these liquids [29]. In the case of ethanol the $\epsilon'(f)$ data were compared with the data reported by Kaatz et al [30]. In all tested liquids the $\epsilon'(f)$ obtained by means of the blocked open-ended probe follows the literature data. Precision of the measurements is limited by the resolution of capacity measurements. The capacity of the BOE probe, used in our experiments, rarely exceeds 2 pF. An error of capacity measurements made by 4191A RF Impedance Analyzer at low frequencies could be as large as 0.1 pF that considerably affects the error of permittivity measurements (see Fig. 6).

Conclusions

It was demonstrated that the open-ended probe separated from the investigated material by a non-conducting layer, possibly formed by a sterile chemically inert foil, made it possible to investigate electrical properties of the material. It was shown that the capacity measured as a function of frequency exhibited a

pronounced dispersion and absorption caused by the layered structure of the probe. The characteristic relaxation frequency is a function of conductivity of a material. It was demonstrated that the capacity measured in the high frequency region was related to the permittivity of the sample and could be used for the determination of ϵ .

The open-ended probe blocked by a sterile polyethylene foil was already used for *in vivo* and *in vitro* examinations of cancer and normal breast tissue [13,24]. The conducted experiments show that at some particular frequency, equal to 100 MHz, it is possible to differentiate very precisely between the cancer breast tissue and the normal breast tissue (consisting mainly of fat). The experiments performed for the purposes of this paper demonstrate that the high sensitivity of the BOE probe in the breast cancer investigation results from a fortunate coincidence. The electric permittivity ϵ' of a tumor is close to 80 (at 100 MHz) and the conductivity σ approximates 1 S/m (100 MHz), whereas in the case of fat ϵ' is close to 6 and σ is about 0.03 S/m. The analysis of the results obtained in this paper shows that the electric “response” of a tumor is located in the dispersion area of the BOE probe. Both the large permittivity and the large conductivity enhance the differences between cancer and normal breast tissues, which provides the basis for the reported high sensitivity of the BOE probe with regard to cancer tissue in breast cancer investigations.

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