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Application of the complex electrical module method for the determination of the relaxation parameters of dielectrics with high electrical conductivity

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Abstract. The paper submits that the method of a complex electrical module allows to investigate the characteristics of relaxation processes in dielectrics having high electrical conductivity. The method makes it possible to determine the relaxation parameters of dielectrics when relaxation peaks are absent in the frequency dependence of dielectric losses $\varepsilon''(f)$. In turn, relaxation peaks can be detected on the frequency dependences $M''(f)$. In addition, a function graph $M'(f)$ is plotted. Simultaneous approximation of the curves $M''(f)$ and $M'(f)$ in the frequency range corresponding to the maximum M'' by the Havriliak–Negami equation for the electrical module makes it possible to determine the relaxation parameters α, β, τ_0.

Keywords: electrical module, relaxation parameters, the Havriliak–Negami equation for the electrical module

Introduction

Dielectric spectroscopy, i. e., investigation of frequency dependences of dielectric permittivity and dielectric loss factor, is widely used in the physics of dielectrics. This method is used to study both synthetic (Castro et al. 2017; Kremer, Schonhals 2002; Nikonorova et al. 2016; 2019; Rychkov et al. 2005; Sazhin 1977) and biological dielectrics (Annus, Min 2021; Asami 2002; Chelidze 2002; Raicu, Feldman 2015; Romanov et al. 2008; Wolf et al. 2011). The analysis of the relaxation peaks on the frequency dependence of the dielectric loss factor makes it possible to determine the relaxation parameters of dielectrics. However, in case of high electrical conductivity, the relaxation peaks on the frequency dependence of the dielectric loss factor cannot be detected. The method of the complex electrical module, proposed in (McCrum et al. 1967), is used to detect such peaks. The purpose of the paper is to show how the complex electrical module method is applied in the analysis of dielectric spectroscopy measurements for synthetic and biological dielectrics under high electrical conductivity. The use of the method is illustrated by specific examples.

Dielectric Spectroscopy

Relaxation equations

The concept of a complex dielectric permittivity $\varepsilon^*(f)$ is used for the analysis of the relaxation properties of dielectrics. It is determined by the expression:

$$\varepsilon^*(f) = \varepsilon'(f) - i{\cdot}\varepsilon''(f),$$

where f is the frequency of the applied electric field, $\varepsilon'(f)$ is the real part of the complex dielectric constant called dielectric permittivity characterising the degree of electric field shielding, $\varepsilon''(f)$ is the imaginary part of the complex dielectric constant called dielectric loss factor characterising absorption energy transferring into the thermal form. The values $\varepsilon'(f)$ and $\varepsilon''(f)$ are determined experimentally using dielectric spectrometers. For example, the spectrometer "Novocontrol Technology Concept 81" allows to make dielectric measurements in the frequency range $f = 10^{-3} - 10^{10}$ Hz at different temperatures.

Generally, relaxation processes are described by the Havrilyak–Negami (H–N) equation (Havriliak, Negami 1966):

$$\varepsilon^*(\omega) = \varepsilon_\infty + \frac{\varepsilon_s - \varepsilon_\infty}{\left(1 + (i\omega\tau_0)^{1-\alpha}\right)^\beta} \tag{1}$$

where ε_s and ε_∞ are the static and high-frequency permittivity $\varepsilon'(f)$ respectively, i. e., ε_s corresponds $f \to 0$, ε_∞ corresponds $f \to \infty$, $\omega = 2\pi f$ is the cyclic frequency, τ_0 is the most probable relaxation time of the electrical response of molecular aggregates or sample molecules, α is the width of the relaxation time spectrum, β is the asymmetry of this spectrum. These parameters can correspond to the following values: $0 \le \alpha < 1$, $0 < \beta \le 1$. In this case, the larger the value α, the greater is the frequency dispersion of the numerical values of the relaxation times of the sample molecules τ, that is, the wider the relaxation spectrum, the smaller the value β, the greater the degree of its asymmetry. For the Debye spectrum $\alpha = 0$, $\beta = 1$.

According to Formula (1), it possible to write analytical formulas for $\varepsilon'(\omega)$ and $\varepsilon''(\omega)$ (Salnikova, Kononov 2020):

$$\varepsilon'(\omega) = \varepsilon_\infty + \frac{(\varepsilon_s - \varepsilon_\infty)(cos(\beta\varphi))}{\left[1 + 2(\omega\tau_0)^{1-\alpha} cos\frac{\pi(1-\alpha)}{2} + (\omega\tau_0)^{2(1-\alpha)}\right]^{\frac{1}{2}}}, \tag{2}$$

$$\varepsilon''(\omega) = \frac{(\varepsilon_s - \varepsilon_\infty)(sin(\beta\varphi))}{\left[1 + 2(\omega\tau_0)^{1-\alpha} cos\frac{\pi(1-\alpha)}{2} + (\omega\tau_0)^{2(1-\alpha)}\right]^{\frac{1}{2}}}, \tag{3}$$

where $\varphi = arctg\left[\dfrac{(\omega\tau_0)^{1-\alpha} sin\frac{\pi(1-\alpha)}{2}}{1 + (\omega\tau_0)^{1-\alpha} cos\frac{\pi(1-\alpha)}{2}}\right]$.

Parameters α, β, τ_0 are the fundamental relaxation parameters of the samples under study. These parameters are determined by the approximation of functions $\varepsilon'(f)$ and $\varepsilon''(f)$. For example, Figure (1a) shows the dispersion of the dielectric permittivity. In turn, Figure (1b) presents the example of function $\varepsilon''(f)$. In this image the relaxation peaks are seen very well. These four peaks are associated with different forms of internal molecular motion of macromolecules. For the analysis of the data, the experimental values $\varepsilon''(f)$ are approximated by a curve according to Formula (3). In this case, the variable parameters of the approximation are α, β, and τ_0. These parameters are selected according to the principle of the maximum coincidence of the experimental and approximating curves. This approximation is made by the software which uses the least square method.

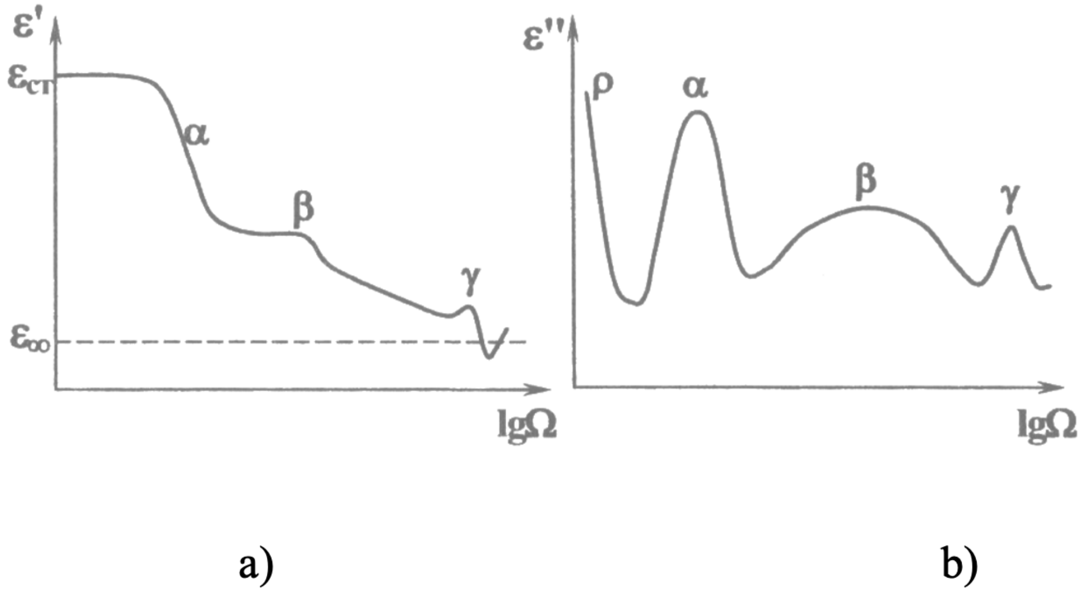


Fig. 1. Frequency *Ω* dependences for a polar polymer in a highly elastic state (Rychkov et al. 2005): a) dielectric permittivity *ε'* b) dielectric loss factor *ε'*

For dielectrics having high electrical conductivity, relaxation peaks in the *ε"(f)* plot are often not observed. To detect them, the dielectric losses associated with electrical conductivity are subtracted from the total dielectric losses (Sazhin 1977):

$$\varepsilon''(f) = \frac{1.8\times 10^{10}\sigma(f)}{f} \ ,$$

where *σ(f)* is the specific electrical conductivity of the dielectric. The remaining value is equal to the relaxation losses $\varepsilon''_{rel}(f)$:

$$\varepsilon''_{rel}(f) = \varepsilon''(f) - \frac{1.8\times 10^{10}\sigma(f)}{f} \ . \tag{4}$$

However, in case of electrical conductivity, this method does not allow to detect relaxation peaks in the $\varepsilon''_{rel}(f)$ plot. Consequently, the determination of the relaxation parameters α, β, τ_0 by this method becomes impossible.

In this case, it is advisable to use the complex electrical module method. It is known, that the application of this method allows to detect relaxation peaks. In turn, mathematical methods allow to determine the values of the relaxation parameters.

Complex electrical module method

The complex electrical module *M* (ω)* is the value of the inverse complex dielectric permittivity determined by the expression (McCrum et al. 1967):

$$M^*(\omega) = M'(\omega) + i\cdot M''(\omega).$$

The quantities *M'(ω)*, *M"(ω)* are called the real and imaginary components of the complex electrical module, respectively. They are equal to:

$$M'(\omega) = \frac{\varepsilon'(\omega)}{\varepsilon'^2(\omega)+\varepsilon''^2(\omega)} \ , \tag{5}$$

$$M''(\omega) = \frac{\varepsilon''(\omega)}{\varepsilon'^2(\omega)+\varepsilon''^2(\omega)} \ . \tag{6}$$

From Equation H-N (1) it is possible to derive formulas for $M'(\omega)$ and $M''(\omega)$ (Salnikova, Kononov 2020):

$$M'(\omega) = \frac{M_\infty M_s A^\beta (M_\infty - M_s) sin\beta\varphi}{A^{2\beta} M_s^2 + 2A^\beta (M_\infty - M_s) M_s cos\beta\varphi + (M_\infty - M_s)^2} \quad , \tag{7}$$

$$M''(\omega) = \frac{M_\infty M_s A^\beta (M_\infty - M_s) sin\beta\varphi}{A^{2\beta} M_s^2 + 2A^\beta (M_\infty - M_s) M_s cos\beta\varphi + (M_\infty - M_s)^2} \quad , \tag{8}$$

where $M_\infty = \dfrac{1}{\varepsilon_\infty}$, $M_s = \dfrac{1}{\varepsilon_s}$,

$$A = \left[1 + 2(\omega\tau_0)^{1-\alpha} sin\frac{\pi\alpha}{2} + (\omega\tau_0)^{2(1-\alpha)} \right]^{\frac{1}{2}} \quad ,$$

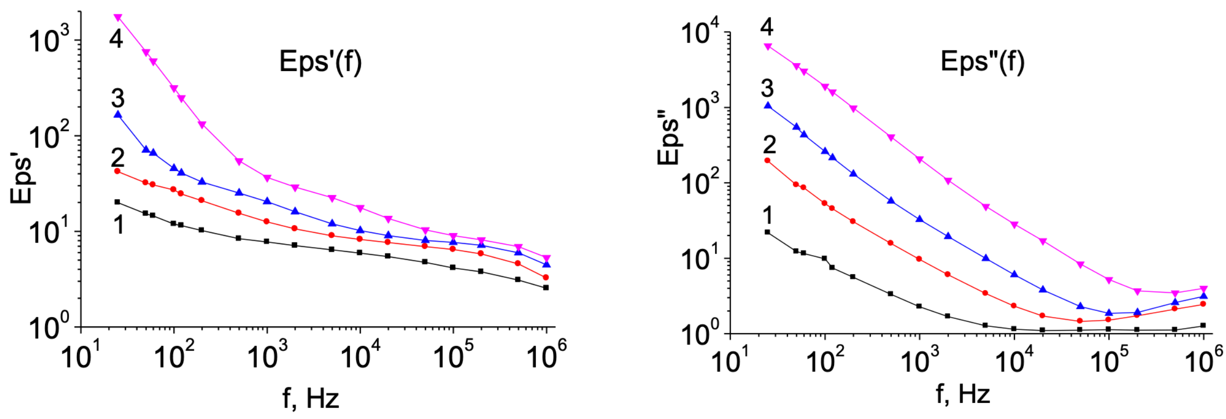
$$\phi = arctg \left[\frac{(\omega\tau_0)^{1-\alpha} cos\dfrac{\pi\alpha}{2}}{1 + (\omega\tau_0)^{1-\alpha} sin\dfrac{\pi\alpha}{2}} \right] \quad .$$

In this case, the parameters α, β, τ_0 have the same physical meaning as in Equation (1).

Experiment

Practical application of the complex electrical module method for copolyamide films SPA-3

Let us consider the practical application of the complex electrical module method to determine relaxation parameters of dielectrics with high electrical conductivity. The sample under study is aliphatic copolyamide SPA-3 films, whose dielectric spectra were derived in our previous work (Avanesyan, Salnikova 2020). If we plot the frequency dependences $\varepsilon'(f)$ and $\varepsilon''(f)$, then the diagrams shown in Fig. 2 will be obtained. It is seen that the $\varepsilon''(f)$ plot does not have relaxation peaks.



a) b)

Fig. 2. Frequency dependences for SPA-3 a) dielectric permittivity $\varepsilon'(f)$, b) dielectric loss factor $\varepsilon''(f)$ at different temperatures: 1—350 K, 2—375 K, 3—405 K, 4—440 K

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Figure (3a) shows the frequency dependence of the specific electrical conductivity σ *(f)*. After subtracting the contribution of conductivity from dielectric losses, the $\varepsilon''_{rel}(f)$ diagram changes (Fig. 3b). There are no relaxation peaks in the diagram, and negative values are explained by high electrical conductivity.

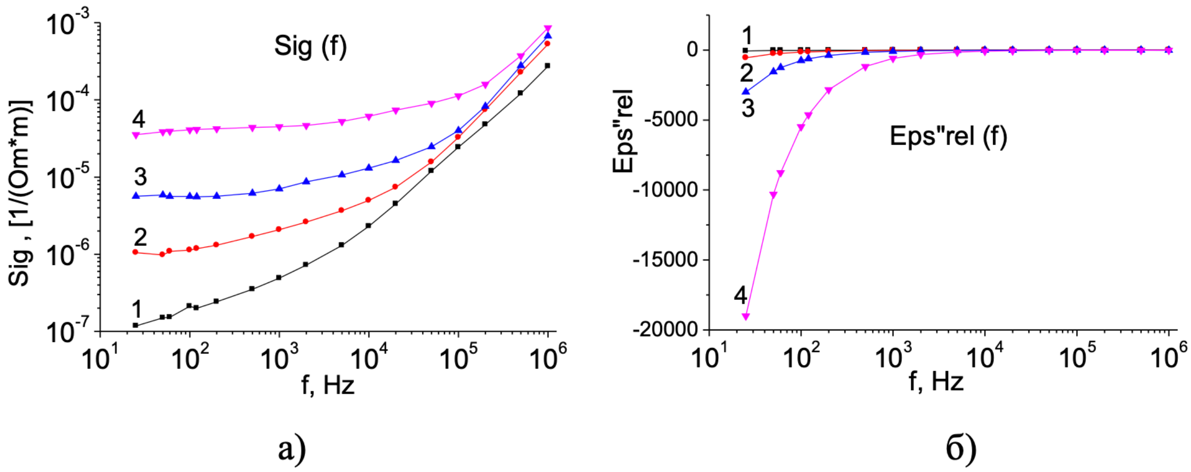

Fig. 3. Frequency dependence for SPA-3 a) specific electrical conductivity $\sigma(f)$, b) relaxation dielectric losses $\varepsilon''_{rel}(f)$ at different temperatures: 1 — 350 K, 2 — 375 K, 3 — 405 K, 4 — 440 K

Let us consider the complex electrical module for this sample. Figure 4 shows the experimental diagrams $M'(f)$ and $M''(f)$ (the values of the electrical module are calculated using Formulas (5, 6); the approximating curves are obtained using Formulas (7, 8).

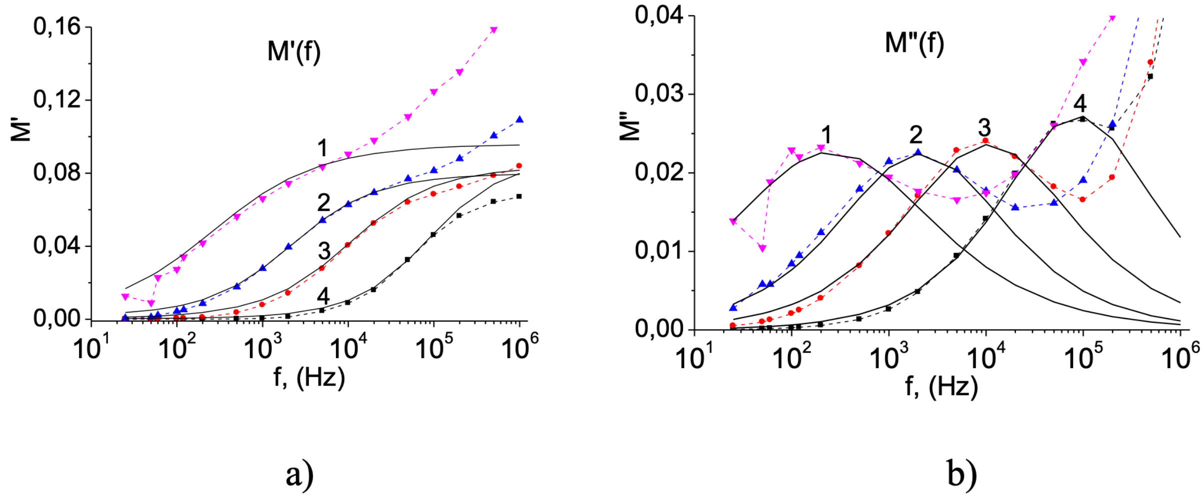

Fig. 4. Frequency dependences of the electrical module for SPA-3 a) the real component of the electrical module $M'(f)$, b) the imaginary component of the electrical module $M''(f)$ at different temperatures: 1 — 350 K, 2 — 375 K, 3 — 405 K, 4 — 440 K. Black lines mark the approximating curves obtained from H–N equation (7, 8) (Avanesyan, Salnikova 2020)

The experimental data are presented by color lines, the approximating curves are shown by black lines. Relaxation peaks are observed on the *M”(f)* dependence, and a sharp increase is observed in the *M’(f)* dependence in the frequency range corresponding to the *M”* maximum. The approximating curves are obtained using the H–N equation for the electrical module. The parameters α, β, τ_0 are selected so as to achieve the maximum coincidence of the experimental and approximating curves simultaneously for both diagrams *M’(f)* and *M”(f)*. It is seen that the simultaneous approximation of both *M’(f)* and *M”(f)* in the region of the maximum *M”* gives good results. This algorithm allows to determine the parameters α, β, τ_0. The relaxation parameters obtained by this method are presented in Table 1.

The relaxation peaks illustrated in Fig. 4b shift toward high frequencies when temperatures rise. This confirms the relaxation type of the polarisation process of the sample. With an increase in temperature, the width of the peaks decreases, but the peaks stay rather wide, their width at half maximum is two frequency decades. This means that the sample has a wide relaxation spectrum typical of polar polymers.

Table 1. Temperature dependence of the relaxation parameters α, β, τ_0 for SPA-3 (Avanesyan, Salnikova 2020)

T K	α	β	τ_0 (s)
350	0.35	0.87	1.5×10–2
375	0.29	0.93	2.5×10–3
405	0.30	0.94	2.4×10–4
440	0.27	0.97	1.0×10–5

These results show that the sample has a relatively wide ($\alpha \geq 0.27$) and almost symmetric ($\beta \geq 0.87$) relaxation spectrum typical of polar polymers.

Using the obtained values $\tau_0(T)$, it is possible to calculate the activation energy of dipole polarization.

Besides, for the complex electrical module it is possible to plot a Cole-Cole diagram, i. e., the dependence *M”(M’)*. Fig. 5 shows the diagram for SPA-3 sample.

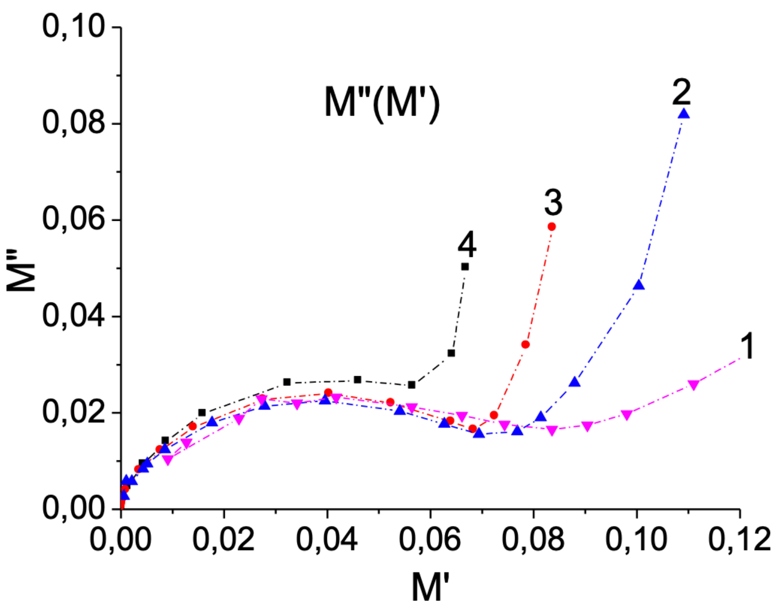

Fig. 5. Cole-Cole diagram for the complex electrical module at different temperatures for SPA-3: 1 — 350 K, 2 — 375 K, 3 — 405 K, 4 — 440 K

It is seen that the experimental points are approximated well by semicircles, the centers of which are located below the M' axis. This corresponds to polymers with a broad, symmetric relaxation spectrum, i. e., $\alpha \neq 0$, $\beta \approx 1$. With an increase in temperature, the curves approach the semicircles, which corresponds to the Debye spectrum, i. e., with an increase in T, the parameter α decreases and the parameter β increases.

Practical application of the complex electrical module method for blood serum of patients with chronic lymphocytic leukemia

Let us consider the practical application of the complex electrical method for the investigation of blood serum (BS) in patients with cancer—chronic lymphocytic leukemia (CLL) (Salnikova et al. 2020a; 2020b). BS has high electrical conductivity ($\sigma \sim 0.01$ Ohm^{-1}*m^{-1}) because it contains Na^+ and Cl^- ions. The dependences $\varepsilon'(f)$ and $\varepsilon''(f)$ constructed according to the data obtained in our work (Salnikova et al. 2020a) will result in the diagrams shown in Fig. 6. It is seen that there are no relaxation peaks in the $\varepsilon''(f)$ plot.

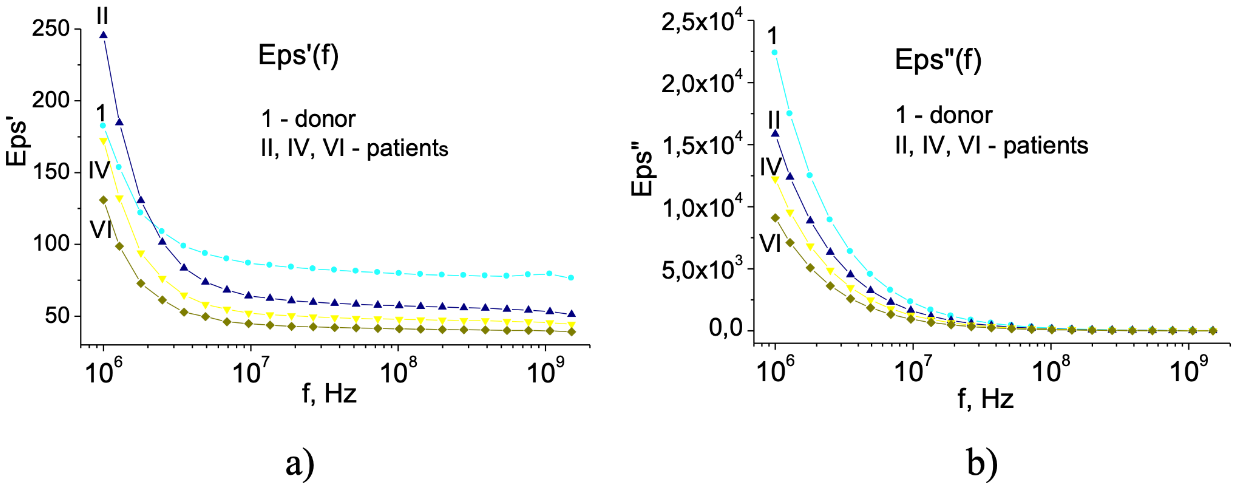

Fig. 6. Frequency dependence for BS in patients with CLL a) dielectric permittivity $\varepsilon'(f)$, b) dielectric losses $\varepsilon''(f)$

Figure (7a) shows the frequency dependence of the specific electrical conductivity $\sigma(f)$. Figure (7b) shows the frequency dependence $\varepsilon''_{rel}(f)$ obtained by subtracting the contribution of specific electrical conductivity $\sigma(f)$ from the dielectric loss factor using Formula (4). The absence of the relaxation peaks in the diagrams and negative values are explained by high electrical conductivity.

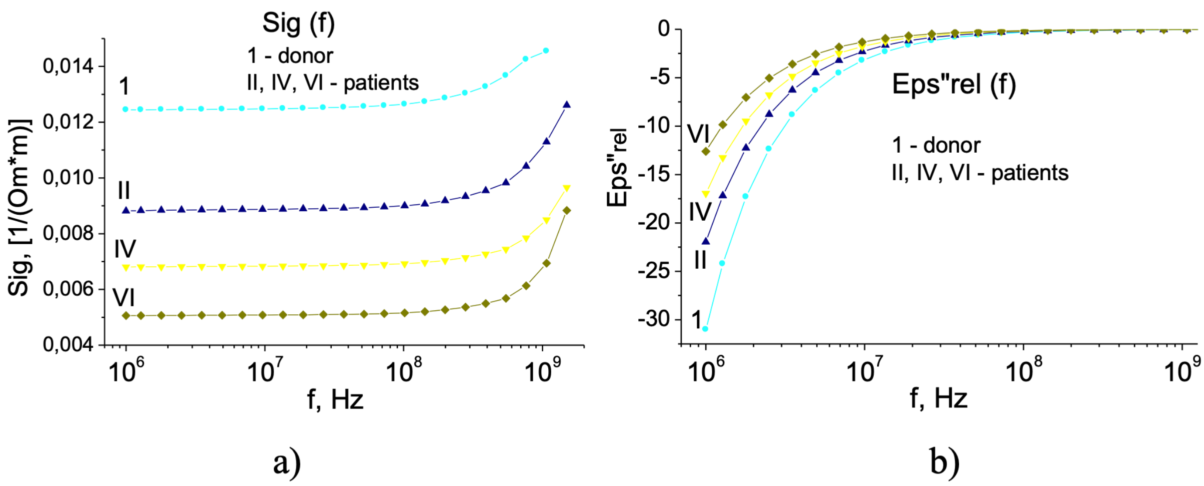

Fig. 7. Frequency dependence for BS in patients with CLL a) specific electrical conductivity $\sigma(f)$, b) dielectric losses $\varepsilon''_{rel}(f)$

Let us move on to the complex electrical module. The values of the real and imaginary components of the electrical module *M'(f)*, *M"(f)* are calculated by Formulas (5, 6) and are represented by colored lines (Fig. 8) (Salnikova et al. 2020a). Relaxation peaks are observed on the *M"(f)* dependence, and a sharp increase is observed in the *M'(f)* dependence in the frequency range corresponding to the *M"* maximum. The approximating curves obtained using the H–N equation for the electrical module Formulas (7, 8) are represented by solid black lines. It is seen that the simultaneous approximation of both *M'(f)* and *M"(f)* in the region of the maximum *M"* gives good results. This algorithm allows to determine the parameters α, β with an accuracy no worse than ± 0.01. The relaxation parameters obtained this way are presented in Table 2 (Salnikova et al. 2020a).

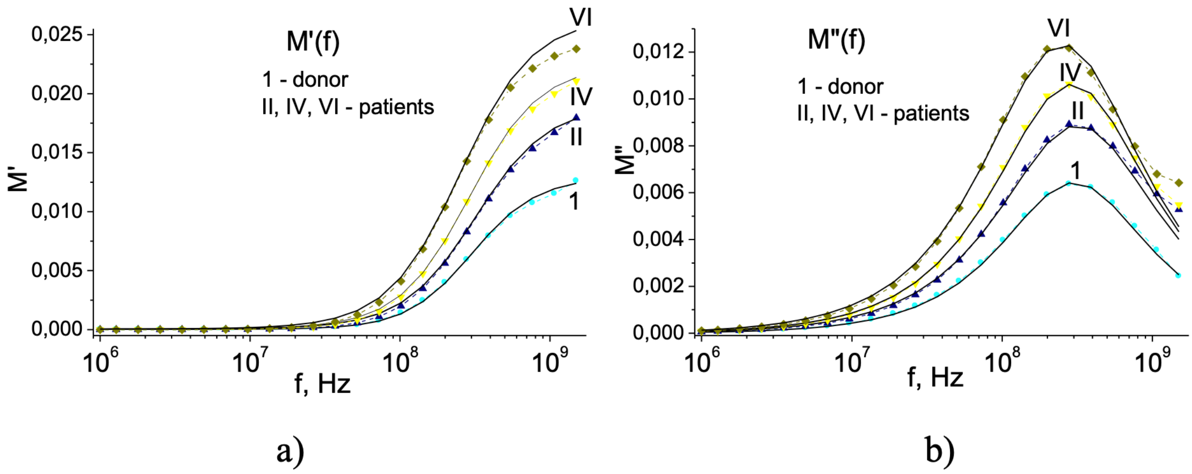

Fig. 8. Frequency dependence for BS in patients with CLL a) the real component of the electrical module *M'(f)*, b) the imaginary component of the electrical module *M"(f)*; 1 — donor, II, IV, VI — patients. Black lines indicate approximating curves obtained using the H–N equation (7, 8)

Table 2. Relaxation parameters α, β, τ_0 of BS for patients with CLL (Salnikova et al. 2020a)

Sample	α	β	τ_0 (s)
1	0	1	6.3×10–5
II	0.03	0.99	1.2×10–4
IV	0.03	0.99	1.7×10–4
VI	0.04	0.99	2.9×10–4

*1–4—donors, I–VI—patients

Figure 9 shows the Cole-Cole diagram of the electrical module *M"(M')* for BS in patients with CLL and donors (Salnikova et al. 2020b). The semicircles are observed clearly. The semicircles are remarkably different for donors and patients with CLL.

We assume that the complex electrical module method can be used for any BS samples. In our opinion, future application of this method will make it possible to diagnose and monitor various diseases by analyzing changes in the relaxation parameters α, β, τ_0 of blood serum. The parameters will not change over time for healthy individuals, while patients will see a change in the parameters. Moreover, the parameters will change more intensively if the disease gets more acute. In case of remission (weakening of the disease), these parameters will begin to return to the normal values typical for a particular individual.

Conclusion

The complex electrical module method allows to determine the relaxation parameters α, β, τ_0 of dielectrics having high electrical conductivity when there are no relaxation peaks on the frequency dependence of dielectric losses *ε"(f)*. Relaxation peaks can be found in the *M"(f)* diagram. In the *M'(f)*

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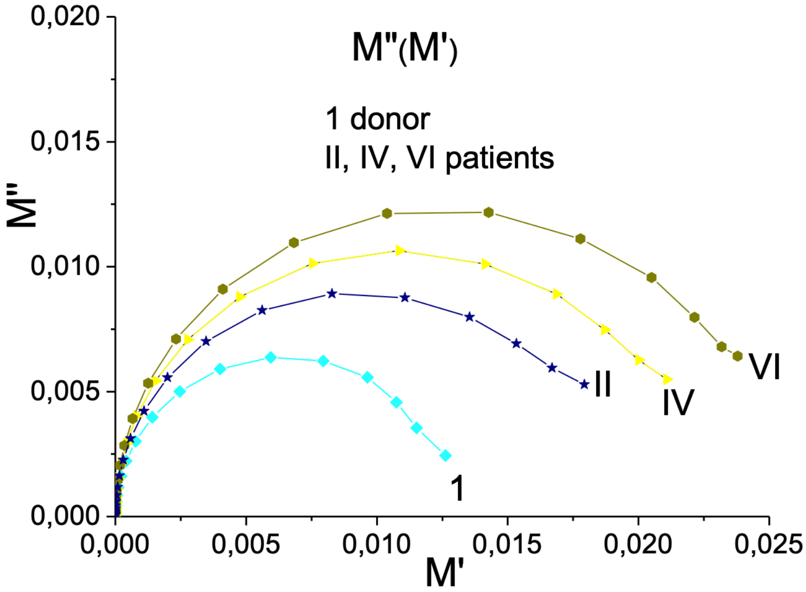

Fig. 9. Cole-Cole diagram of the electrical module for BS in patients with CLL and donors.
1–4 — donors, II, IV, VI — patients

diagram, one can find a sharp rise in the area of the maximum *M"*. Simultaneous approximation of the curves *M"(f)* and *M'(f)* in the frequency range corresponding to the maximum M" according to the Havriliak–Negami equation for the electrical module allows to determine the relaxation parameters for the sample under investigation. Thus, the method of the complex electrical module makes it possible to research the characteristics of relaxation processes hidden by the high electrical conductivity of dielectrics.

Conflict of interest

The authors declare that they have no conflicts of interest.

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