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Electric polarization of polyelectrolyte and colloid media: dielectric versus electro-optic approach

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Abstract

The theories of dielectric dispersion and of electric birefringence as a representative of electro-optic methods are considered and it is shown that they both depend in a similar way simply on the real part of the complex electric polarizability of the macromolecules or the particles. The latter also contains the particle permanent dipole moment. Experimental data on dielectric dispersion, electric birefringence and electric light scattering of strongly elongated, rod-like poly(tetrafluoroethylene) particles are compared and an attempt is made to extend the dielectric dispersion curve to lower frequencies using electric birefringence and electric light scattering data. Further, the experimental data on dielectric dispersion, electric light scattering, electro-orientation and dipolophoresis for the more complicated *Escherichia coli* particles are compared. Again, the possibility to extend the 10 kHz–100 MHz dielectric dispersion curve down below 1 Hz by using electric light scattering data is examined. The good matching of the dielectric dispersion and electric light scattering frequency curves found in the overlapping frequency range (10 kHz–5 MHz) essentially enhances the chance that dielectric dispersion below 1 MHz is related to α dispersion and not to electrode polarization. Thus it is not only possible to obtain additional information on the mechanism of polarization at lower-frequency dielectric dispersion, but also to extend our knowledge about the effective dielectric properties of biological complex fluids to frequencies essentially below 1 MHz. This could be important for the understanding of the effect of low-frequency electromagnetic fields on living matter.

Keywords: Electric polarizability; Dielectric dispersion; Birefringence, electric; Light scattering, electric; Polyelectrolytes; Colloids; Escherichia coli; Poly(tetrafluoroethylene)

1. Introduction

Electric polarization of polyelectrolyte and colloid media is attracting increasing attention of many researchers in the fields of polymer and colloid sciences, biophysics, material sciences, etc. One of the main problems here is the elucidation of the mechanisms of polarization, which in a considerable frequency range are strongly related to the particle charge and the ionic environment. So not only the particle electric properties could be determined, but it is also possible to obtain more information on the reaction of different types of complex fluids to electric fields in a broad range of electric field strengths, frequencies, particle and electrolyte concentrations, etc. This might be of considerable practical interest for the biotechnology, the understanding of the effect of electromagnetic fields on biological systems, in-

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cluding the human body, electrophysiology, etc. There have been successful, interesting attempts to measure one and the same sample under the same experimental conditions and to compare the frequency dependencies obtained by different methods for studying electric parameters. A good example with increasing mismatch in frequency dependence is provided by measurements of latex particles by dielectric dispersion and cell rotation techniques [1]. This is a very interesting comparison between a macroscopic and a microscopic method of studying the electric properties of particles. Other examples are linked with the combination of electric light scattering, electric birefringence and electric dichroism of purple membranes [2], electric light scattering (electro-orientation) with dipolophoresis of biological cells [3], electric birefringence, electric light scattering and electric conductivity of microemulsions [4], etc. The last two examples are interesting since the measurements of all the experimentally studied quantities are carried out simultaneously.

Independently of the considerable progress made in the last years in the experimental methods for the electrical characterization of particles there remains some pronounced retardation in the comparative investigations of the advantages and disadvantages of the different methods. This concerns especially dielectric dispersion and electro-optic methods, which are most widely used. This holds also for dipolophoretic (dielectrophoretic) cell translation, cell rotation and other electric methods [5]. One of the rare comparative discussions on dielectric and electro-optic theories was carried out by Ookubo et al. [6]. The only dielectric and electro-optic measurements known to us of one and the same batch of PTFE particles, unfortunately at very different concentrations, were carried out by Osborn et al. [7] and Foster et al. [8].

The aim of this paper is to examine the existing dielectric and electro-optic theories, especially the way they can be best compared. This may be a basis for combining dielectric and electro-optic experimental data. This possibility is further illustrated by a simple model particle (PTFE, strongly elongated, rod-like) and by a complex biological cell (*Escherichia coli*), using published experimental data of other authors and unpublished data from our laboratory, which is currently in press or in preparation. This is only the first step in extending the dielectric dispersion curves to lower frequencies by using electro-optic dispersion data.

The advancement in this field is additionally retarded by the considerable divergence in the way in which the various authors describe and name the different quantities. A good example of this is the electric polarizability and its various components, especially in cases of complex heterogeneous particles, like biological cells.

2. Theory

For the simplest model of strongly elongated, rod-like particles one can write [6]:

$$\Delta\varepsilon^* = c\alpha_3^*/(3\varepsilon_\sigma) \tag{1}$$

where $\Delta\varepsilon^*$ is the permittivity of the disperse system in excess of the disperse medium, c is the number of particles (polymer molecules) per unit volume, and α_3^* is the complex polarizability of the particle (macromolecule) along the third (long) axis, which is given by the expression [9]:

$$\alpha_3^* = \frac{\mu^2/(kT)}{1+j\omega\tau_r} + \frac{\alpha}{1+j\omega\tau} + \alpha_3(\infty) \tag{2}$$

where μ, α and $\alpha(\infty)$ are the permanent dipole moment, the electrical polarizability increment and the high-frequency electrical polarizability of the particles respectively, and kT is the thermal energy. The first term in Eq. 2 corresponds to the orientational polarization with rotational relaxation time τ_r related to the rotational diffusion coefficient D_r by the expression:

$$\tau_r = 1/(2D_r) \tag{3}$$

The second term corresponds to the induced polarization, where τ is a harmonic mean, given by

$$(1/\tau) = (1/\tau_i) + (1/\tau_r) \tag{4}$$

The relaxation time τ_i is the intrinsic relaxation time of the induced polarization taking into account the modification of the translational motion of the counterions along the rod by the rotational motion of the particle.

For particles with a more complicated structure, expressions for the effective permittivity ε_e and effective conductivity σ_e have been suggested [3]:

$$\varepsilon_e^{\parallel(\perp)} = \varepsilon_h + \frac{\varepsilon_s^{\parallel(\perp)} - \varepsilon_m^{\parallel(\perp)}}{1 + \left[\nu/\nu_\alpha^{\parallel(\perp)}\right]^2} + \frac{\varepsilon_m^{\parallel(\perp)} - \varepsilon_h}{1 + \left[\nu/\nu_\beta^{\parallel(\perp)}\right]^2} \tag{5}$$

$$\sigma_e^{\parallel(\perp)} = \sigma_h + \frac{\sigma_s^{\parallel(\perp)} - \sigma_m^{\parallel(\perp)}}{1 + \left[\nu/\nu_\alpha^{\parallel(\perp)}\right]^2} + \frac{\sigma_m^{\parallel(\perp)} - \sigma_h^{\parallel(\perp)}}{1 + \left[\nu/\nu_\beta^{\parallel(\perp)}\right]^2}$$

where ε_s, α_s, ε_m, α_m, ε_h and α_h are the low (static), middle and high frequency values, respectively, of the effective ε and α, the index $\parallel(\perp)$ stands for parallel and perpendicular with respect to the rod axis, ν_α and ν_β are the characteristic frequencies of the α and β dispersions, respectively, corresponding to the relaxations of the polarization of the diffuse double layer and of the Maxwell–Wagner polarization.

As an example of an electro-optic effect one can take the electric birefringence Δn. If one wants to define the complex electric birefringence Δn^* analogously to the complex permittivity, it is possible to write [10]:

$$\Delta n^* = \Delta n - j\Delta K \tag{6}$$

where ΔK is the "biabsorption" or, as it is currently known, electric dichroism. Since the expressions derived are valid for both electro-optic effects and electric light scattering, when degrees of orientation are low [5], we shall further only concentrate on the theory of electric birefringence.

Best comparable to dielectric spectroscopy is the frequency-domain electric birefringence. For this one can write [6]:

$$\Delta n = \Delta n_{dc} + \Delta n_{2\omega} \tag{7}$$

where Δn_{dc} is the dc component and $\Delta n_{2\omega}$ the Δn component, alternating at 2ω and

$$\Delta n_{2\omega} = \mathrm{Re}\left(\Delta n_{2\omega}^* e^{2i\omega t}\right) \tag{8}$$

The following expressions are also useful [6]:

$$\Delta n_{dc}/\Delta n_{st} = \mathrm{Re}(\alpha_3^*)/\alpha_3(0) \tag{9}$$

where Re designates the real part of α^*, $\alpha_3(0)$ is represented as:

$$\alpha_3(0) = \mu^2/(kT) + \alpha + \alpha_3(\infty)$$

and Δn_{st} is represented as:

$$\Delta n_{st} = cE^2\Delta\alpha^{op}\alpha_3(0)/(30n\varepsilon_\sigma kT) \tag{10}$$

where $\Delta\alpha^{op}$ is the optical anisotropy factor of the particle, n is the refractive index of the disperse system and E is the electric field strength.

A useful analysis for understanding the orientational behaviour, which is closely related to the electro-optic behaviour of particles and biological cells, is in particular made by Miroshnikov et al. [3], who give an expression for the moment of orienting forces M, which depends in a similar way on α^* as the above expression for $\Delta n_{dc}/\Delta n_{st}$ (9):

$$M = \mathrm{Re}(\Delta\alpha^*)E^2\sin 2\theta \tag{11}$$

where $\Delta\alpha^* = \alpha_\parallel^* - \alpha_\perp^*$ and θ is the angle between the electric field and the long particle axis, i.e. the axis $\parallel$. For a particle with a rotational ellipsoid form and of volume V, longest axis a and shortest axis b:

$$V = \frac{4}{3}\pi ab^2$$

the components of the electric polarizability of the particle are given by:

$$\alpha_\parallel^* = \frac{1}{4}V\frac{\tilde{\varepsilon}_1^*(\varepsilon_2^* - \varepsilon_1^*)}{\varepsilon_1^* + A_2(\varepsilon_2^* - \varepsilon_1^*)}$$

and

$$\alpha_\perp^* = \frac{1}{4}V\frac{\tilde{\varepsilon}_1^*(\varepsilon_2^* - \varepsilon_1^*)}{\varepsilon_1^* + A_1(\varepsilon_2^* - \varepsilon_1^*)} \tag{12}$$

where ε_2^* and ε_1^* are the complex permittivities of the particle and the medium, $\tilde{\varepsilon}_3^*$ indicates the complex conjugated value and A_2 and A_1 are the depolarization factors of the ellipsoid:

$$A_2 = \frac{ab^2}{2(a^2 - b^2)^{3/2}} \times \left(\ln\frac{a + \sqrt{a^2 - b^2}}{a - \sqrt{a^2 - b^2}} - 2\frac{\sqrt{a^2 - b^2}}{a}\right)$$

$$A_1 = \frac{1 - A_2}{2}$$

It may be worthwhile, when discussing the theory of dielectric and electro-optic response of particles to an electric field, to have also in mind the translational motion of the particles, when placed in a non-homogeneous electric field: grad E. This phenomenon was introduced originally by Pohl [11] as *dielectrophoresis* (translational motion of non-charged particles in a non-homogeneous electric field). Later this name was used (in our opinion) incorrectly for any type of charged particle movement in a non-homogeneous electric field. So in this case some effective dielectric properties of the particles have to be understood which are complicated to define and understand (easy to misunderstand). Therefore it seems better to go back to the more general term [5,12] *dipolophoresis*, which includes, as a particular case, dielectrophoresis for induced dipoles in non-charged dielectric particles and also all cases of induced and permanent dipole charged particles as studied, for example, by electro-optic methods. Using the notations used above one can write for the dipolophoretic force [3]:

$$\vec{F}_{\mathrm{dp}} = \alpha^{*} \operatorname{grad} E^{2} \tag{13}$$

where $\alpha^{*} = \alpha_{\parallel}^{*}$, when the rod-like particle moves with its longer axis oriented parallel to the applied electric field and $\alpha^{*} = \alpha_{\perp}^{*}$, when the particle moves with its shorter axis oriented parallel to the electric field.

It can be expected that for strongly elongated rod-like particles without a permanent dipole moment the expressions for the different effects are mathematically comparable (at least qualitatively). So if the frequency ranges from dielectric and electro-optic dispersion dependences are sufficiently overlapping, a permittivity dispersion curve extended to lower frequencies may be constructed. In doing so it should be taken into account that unlike the electro-optic frequency dependence, the corresponding permittivity dependence is always increasing when going to lower frequencies.

3. Experimental

Since the aim of this study is to compare the results obtained by dielectric and electro-optic measurements, the presentation of the methods will be limited only to frequency (dispersion) dependence measurements.

Dielectric measurements in the last 10 years have been mainly performed using computer controlled impedance analysis. An example of a good quality impedance analyser is the Hewlett-Packard Model 4192A. The main problem for these measurements is the electrode polarization which considerably perturbs measurements at lower frequencies. One way of overcoming this problem is the utilization of dielectric cells with variable interelectrode distance (Osborn et al. [7]). Another approach is to measure the time response of the system when subjected to an electric pulse well defined in time followed by Fourier transformation to obtain the frequency dependence. An example of this approach is the pseudo-random noise method (Minakata et al. [13]), which is based on the multiple application to the sample of a pseudo-random noise signal containing all frequencies of the investigated frequency range.

The electro-optic measurements are in most cases made with home-made instruments which do not differ essentially from that described by Fredericq and Houssier [14], except in the computer treatment of the electro-optic signal and in some cases in the computer guidance of the experiment (Mayer et al. [15]).

The dipolophoretic (dielectrophoretic) measurements are made in two main modes: microscopic and macroscopic. In the microscopic mode (microdipolophoresis) [12], the dipolophoretic mobility of the single particle is measured. In the macroscopic mode, the yield of the particles normally on the electrode of the highest field strength is measured and from there the dipolophoretic mobility may be deduced [11].

There are some successful attempts to perform measurements simultaneously of two or more quantities, like electric light scattering (electro-orientation) and dipolophoretic displacement [3], electric birefringence, electric light scattering and conductivity [4], etc. Such measurements are of great interest for the understanding of the interrelation (and its utilization) of different methods for studying electric parameters of colloid particles and macromolecules.

4. Materials

Here attention will be paid mainly to two quite different objects. The first are the aqueous dispersions of PTFE whiskers: very strongly elongated, rod-like particles as an example of a simple model system. The second being *E. coli* suspensions as an example of particles of very complicated structure.

The poly(tetrafluoroethylene) (PTFE) fibres (whiskers) are prepared by emulsion polymerization. The samples studied in the laboratories of Prof. Hoffman (Bayreuth) and Prof. Stoylov (Sofia) were prepared in Bayreuth by S. Angel [16] and the samples studied in the laboratory of Dr. Foster (Philadelphia) were prepared by du Pont (E. I. du Pont de Nemours). The weight average length of the "Bayreuth" preparation (0.54 μm) is similar to the volume average length of the "du Pont" sample, 0.5 μm. The polydispersity of the Bayreuth sample is greater than that of the du Pont sample and it contains a greater number of short particles and small spherical particles (diameter around 100 nm), which leads to its much lower number average length of 0.21 μm, as compared to the du Pont sample with a number average length of 0.4 μm. The diameter of the fibres in both cases are around 20–30 nm. Perfluoro-surfactants were used in the polymerization in order to obtain strongly anisodiametric latex particles and stable suspensions.

The *E. coli* strain used in the electric light scattering was a hybrid between *E. coli* K-12 and *E. coli* B. It was cultivated in LB medium (yeast extract "Difco" 0.5%, tripton Difco 1%, NaCl 1%) till a concentration of 4×10^8 cells/ml. The cells were washed once in sterile water and once with 270 mM sucrose. After the last washing the cells were resuspended in 2 ml 270 mM sucrose (10^{10} cells/ml).

The *E. coli* for the dielectric measurements was a strain K-38, grown in shaken cultures at 38°C (Asami et al. [17]). The cells were washed once with the suspending medium, containing 10 mM NaCl and 1 mM $MgCl_2$. The osmolarity of the suspending medium was adjusted to ca. 0.3 M by addition of sorbitol.

5. Results and discussion

The dielectric measurements on PTFE by Osborn et al. [7] show, as is the case with most linear polyelectrolytes, two dispersions: one at lower frequencies (kHz range) and one at higher frequencies (MHz range). In Fig. 1 their data are represented as $\log(\varepsilon - \varepsilon_\infty)$ versus $\log \nu$ by filled circles. It should be specially noted that these are the results for a PTFE particle concentration of 3 wt.% and a conductivity of 2.7×10^{-4} S/cm. The electric birefringence study carried out by Foster et al. [8] for four conductivities of PTFE systems in the range 7.3×10^{-5}–7.2×10^{-4} S/cm does not show the low frequency dispersion. It should be well noted that in this case the concentration is much lower, i.e. 0.2 wt.%. Independent measurements of the alternating component have shown that there is a slight dispersion about 200 Hz. It is difficult to reconciliate all these data while bearing in mind the similarity of the frequency dependence of permittivity and electro-optic effects, some aspects of which are given in the previous part on Theory and as an appendix in the paper of Foster et al. [8]. In Fig. 2 are shown electro-optic (electric light scattering) data on the frequency dependence of PTFE for different PTFE concentrations and different ionic strengths [18]. The curves strongly suggest that differences between Fig. 1 and electric birefringence results of Foster et al. [8] may be mainly due to the different PTFE concentra-

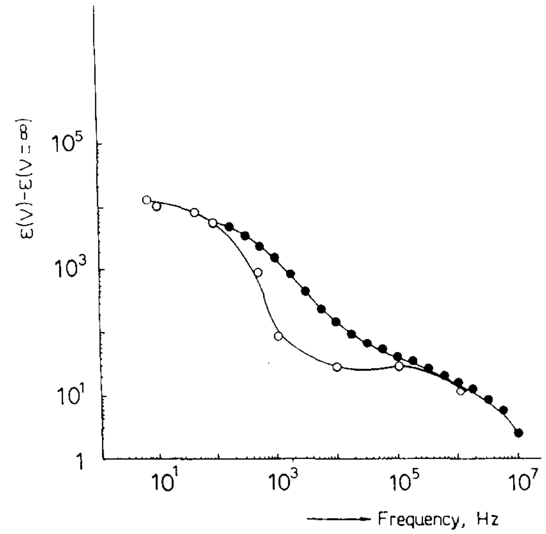

Fig. 1. Permittivity $\varepsilon(\nu) - \varepsilon(\nu = \infty)$ vs. frequency ν (at logarithmic scale) for a suspension of PTFE fibres: (●) dielectric dispersion data [7] and (○) electric light scattering data [18] recalculated for permittivity. Recalculation is made by coinciding electro-optic and dielectric data for frequencies 10^2 and 7×10^5 Hz.

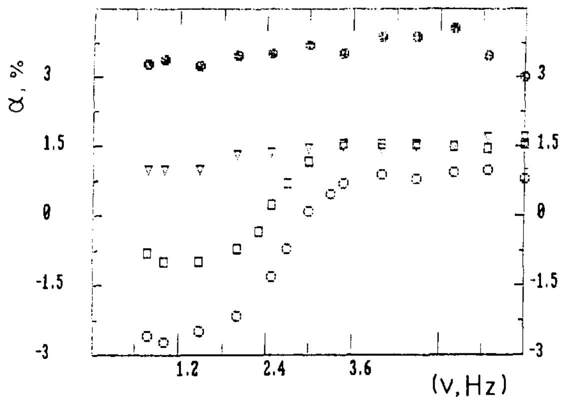

Fig. 2. Electric light scattering effect α vs. frequency ν for a suspension of PTFE fibres (electric field strength 77 V/cm) for different PTFE concentrations: (■) 0.05; (▽) 0.1; (□) 1 and (○) 4 wt.%. Suspensions conductivities, respectively: 0.6, 1.2, 4.5 and 13×10^{-5} S/cm [18].

tions. So the lower frequency dispersion at higher PTFE concentrations should be mainly related to the so called electro-optic "anomaly" (Hoffmann et al. [19]), the amplitude and the frequency of which get lower with decreasing particle concentration (Baumann et al. [20]). The higher frequency dispersion, its amplitude and critical frequency dependences on particle and electrolyte concentrations are mainly related to the relaxation properties of the diffuse double layers of the particles. It should be possible to present this through the surface conductivity of the particle like suggested by Foster et al. [8]. For a better understanding of this dispersion, an additional, more detailed study in broader ranges of particle and electrolyte concentration could be very useful as well as further refinements of surface conductance models taking into account other polarization mechanisms.

It is important to note that the results shown in Fig. 2 are fully confirmed by the extensive electric birefringence measurements on one and the same sample in Bayreuth (Baumann et al. [10]; S. Angel [16], etc.) and in Strasbourg (Stoylov et al. [21]).

In Fig. 1 an attempt is made to combine the data for permittivity (filled circles) with the data from Fig. 2 for a PTFE concentration of 4% (the lowest curve) obtained for similar samples and under not essentially different experimental conditions. The curves might be regarded as approximately corresponding to the frequency dependence of the electric polarizability presented in arbitrary units. They are obtained by fitting the electro-optic curve to the permittivity curve. Full coincidence is postulated at frequencies of 10^2 and 7×10^5 Hz in the overlapping interval. The curves are traced as permittivity versus frequency dependence for a broader frequency range. The recalculation of electro-optic data into permittivity data are shown with open circles in Fig. 1. The recalculation is made assuming that both permittivity and absolute value of electric light scattering are proportional to the real part of the complex particle polarizability and that the coefficients of proportionality in the two cases do not depend on the frequency. So the recalculated permittivity values are equal to the product of the absolute value of the electro-optic effect and the coefficient of proportionality of the electro-permittivity versus real part of the complex electric polarizability divided by the coefficient of proportionality electro-optic effect versus real part of complex electrical polarizability.

The case with bigger, slightly elongated particles of heterogeneous structure is much more complicated. A good, and important for the microbiology and biotechnology, example is *E. coli*. In Fig. 3 the frequency dependence of the electro-orientational effect (electro-turbidity) is shown [3]. The dipolophoretic yield changes are shown for the same frequency range [3] in Fig. 4. It is seen that up to four dispersions (Fig. 3) are seen in the frequency dependence of the electro-orientational effect. In general, the dipolophoresis frequency dependence

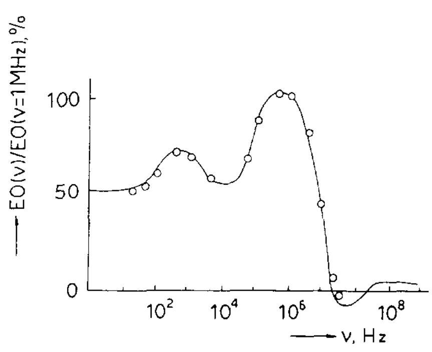

Fig. 3. Dependence of relative (to value at 10^6 Hz) electro-orientational effect EO(ν)/EO (ν = 1 MHz) on frequency for *E. coli* [3].

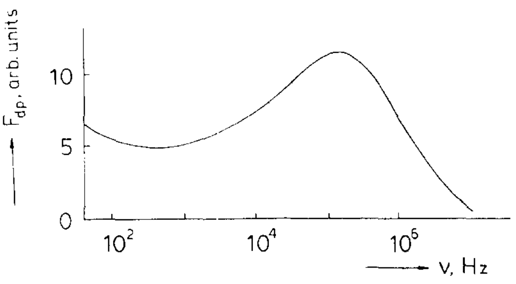

Fig. 4. Experimental dependence of the dipolophoretic force in arbitrary units on frequency [3].

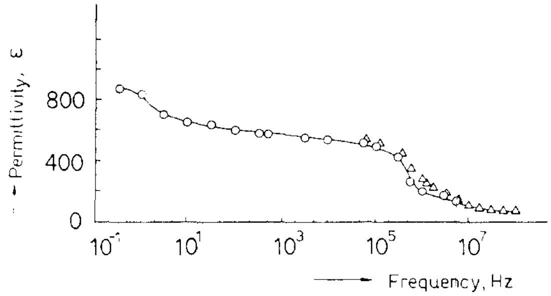

Fig. 6. Permittivity ε vs. frequency ν for *E. coli*: (△) dielectric measurements [17] and (○) electric light scattering data [22] (see Fig. 5) recalculated for permittivity. Frequencies at which dielectric and permittivity experimental data coincide are 5×10^4 and 5×10^6 Hz.

repeats the electro-orientation frequency dependence. In the latter case, the lowest part of the frequency dependence of the electro-orientation effect seems to have been lost.

In Fig. 5 electric light scattering frequency dependences (Peikov et al. [22]) for two different ionic strengths are shown. It is seen that in the frequency ranges of Figs. 3 and 4 there is a correspondence in the form of the curves. In the frequency range below 200 Hz, a new (5th) dispersion is seen in Fig. 5, which surprisingly changes its sign with the ionic strength. Following the critical frequency of this dispersion (about 2 Hz), it can be suggested that it is related to a permanent dipole-moment-like orientation. The strong ionic strength dependence could be taken as evidence for its interfacial origin. For example, this "permanent" dipole moment might be related to the heterogeneity of the electro-kinetic potential of the *E. coli* cell on the scale of the cell dimensions [23]. It is of great interest to compare this data with the dielectric spectroscopy study of Asami et al. [17] on a different strain of *E. coli*, under similar experimental conditions. Both the comparison of the electro-optic and of the dielectric frequency dependences in the range 10 kHz–5 MHz show a good matching. So an extension of the dependence of the complex electric polarizability (see e.g. Eq. 7), which is equivalent to the extension of the permittivity frequency dependence, down to below 1 Hz might be much more instructive than the similar procedure for the PTFE suspension considered in the beginning of this part of the paper. In Fig. 6 the dielectric spectroscopy and electric light scattering data are combined as in the case of Fig. 1 to produce a dielectric dispersion curve essentially extended to low frequencies (below 1 Hz). Although the strains and experimental conditions are not very comparable, there is are enough evidence (Miroshnikov et al. [3]) that this comparison has considerable reliability, although some effect of electrode polarization at the lowest frequencies cannot be excluded. The comparison of Figs. 5 and 6 illustrates quite convincingly that dispersions are much clearer displayed by electro-optic effects. The main cause for this is the possibility of dispersions of different

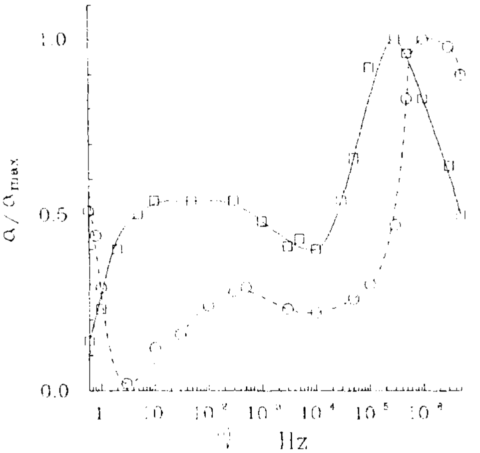

Fig. 5. Relative electric light scattering $\alpha(\nu)/\alpha_{max}$ vs. frequency ν for *E. coli* for two different ionic strengths (given as conductivities): (□) $K=3\times10^{-5}$ S/cm; (○) $K=8\times10^{-4}$ S/cm. Particle concentration $c=1.9\times10^7$/ml; $E=25$ V/cm.

signs (rising and falling) only in the case of the electro-optic frequency dependences.

6. Conclusions

This paper is only the first step in the joint approach to the investigation of the frequency dependence of the electric polarization of complex fluids of general and biological interest through the methods of dielectric spectroscopy and electro-optics. More theoretical efforts are needed for a more adequate and clearer presentation of dielectric and electro-optic, basic experimentally measurable parameters through the complex polarizability of the particles of the fluid.

Of essential importance is the right choice of both model simple particles on the one side and more complicated particles on the other and their subsequent detailed experimental investigation under the same experimental conditions by different dielectric and electro-optic methods. Here utilization of the time domain variants of both types of methods should also not be underestimated (Minakata et al. [13]; Walther et al. [24]). Thus it should be possible not only to advance in the understanding of the different mechanisms of the electric polarizability in aqueous complex fluids, but also to obtain the frequency dependence of the electric properties of various materials in much broader ranges than it is accessible only for one type of methods. In case of biological samples, this could also be helpful for a further understanding of the mechanism of the effect of electromagnetic fields on living matter.

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