ELECTRORHEOLOGY OF SUSPENSIONS OF VARIOUSLY PROTONATED POLYANILINE PARTICLES UNDER STEADY AND OSCILLATORY SHEAR

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ABSTRACT:
Electrorheological (ER) and dielectric properties of silicone-oil suspensions of polyaniline (PANI) particles protonated with phosphoric and tetrafluoroboric acids to various doping level have been investigated. The particle conductivity was thus varied between the order of 10^{-9} S/cm and 10^{-4} S/cm. The dynamic yield stresses obtained at controlled shear rate mode viscometry, the storage moduli from the oscillatory shear experiments and the dielectric relaxation times from frequency dependences of dielectric constant and loss factor were used as criteria of rigidity or elasticity of ER structures and particle mobility in the electric field. The conductivity of suspension particles plays a decisive role in their ER behaviour. The ER efficiency increased as conductivity of dispersed particles raised, irrespective of the type of employed acid used for the protonation of PANI.

ZUSAMMENFASSUNG:
Elektrorheologische (ER) und dielektrische Eigenschaften von Suspensionen aus Silikonöl und Poly(anilin)-Partikeln, die mit Phosphor-und Tetrafluorborsäure in unterschiedlicher Stärke protoniert wurden, wurden untersucht. Die Leitfähigkeit der Partikel wurde in der Größenordnung von 10^{-9} S/cm und 10^{-4} S/cm variiert. Die dynamische Fließpannung, die durch Messungen in dem Modus der kontrollierten Scherrate erhalten wurde, der Speichermodul aus oszillatorischen Scherversuchen und die dielektrischen Relaxationszeiten aus der Frequenzabhängigkeit der dielektrischen Konstanten und des Verlustfaktors wurden als Kriterium der Starrheit oder der Elastizität der ER-Strukturen und der Partikelbeweglichkeit in dem elektrischen Feld verwendet. Die Leitfähigkeit der Suspensionspartikel spielt eine entscheidende Rolle in ihrem ER-Verhalten. Die ER-Wirksamkeit nahm mit der Leitfähigkeit der dispergierten Partikel zu, unabhängig von der zur Protonierung von PANI verwendeten Säure.

RÉSUMÉ:
Nous avons étudié les propriétés electrorhéologiques (ER) et diélectriques de suspensions de particules de polyaniline (PANI) protonées avec des acides phosphoriques et tétrafluoroboriques à divers degrés de dopage. La conductivité de la particule a donc ainsi été variée entre 10^{-9} S/cm et 10^{-4} S/cm. Les contraintes dynamiques de seuil obtenues en mode de vitesse de cisaillement contrôlée, les modules élastiques obtenus à partir des expériences de cisaillement oscillatoire et les temps de relaxation diélectrique obtenus à partir de la dépendance fréquentielle de la constante diélectrique et du facteur de perte, ont été utilisés comme critères de rigidité ou d'élasticité des structures ER et de mobilité de la particule dans le champ électrique. La conductivité des particules en suspension joue un rôle décisif dans leur comportement ER. L'efficacité ER augmente avec la conductivité accrue des particules dispersées, sans relation avec le type d'acide utilisé pour la protonation des particules PANI.

KEY WORDS: polyaniline, electrorheology, steady shear, oscillatory mode, protonation

1 INTRODUCTION

Electrorheological (ER) effect [1], known as an outstanding change in rheological behaviour of ER suspension after the application of external electric field has been attracting strong attention since its discovery 60 years ago. The particles randomly dispersed in the non-conducting carrier medium are polarized in the presence of the electric field, resulting in particle fibrillation with string-like or columnar structures oriented along the field direction. In the electric field of intensity of several kilovolts per millimetre, this abrupt change sets in order of milliseconds. Thus, a dramatic increase in viscosity of several orders of

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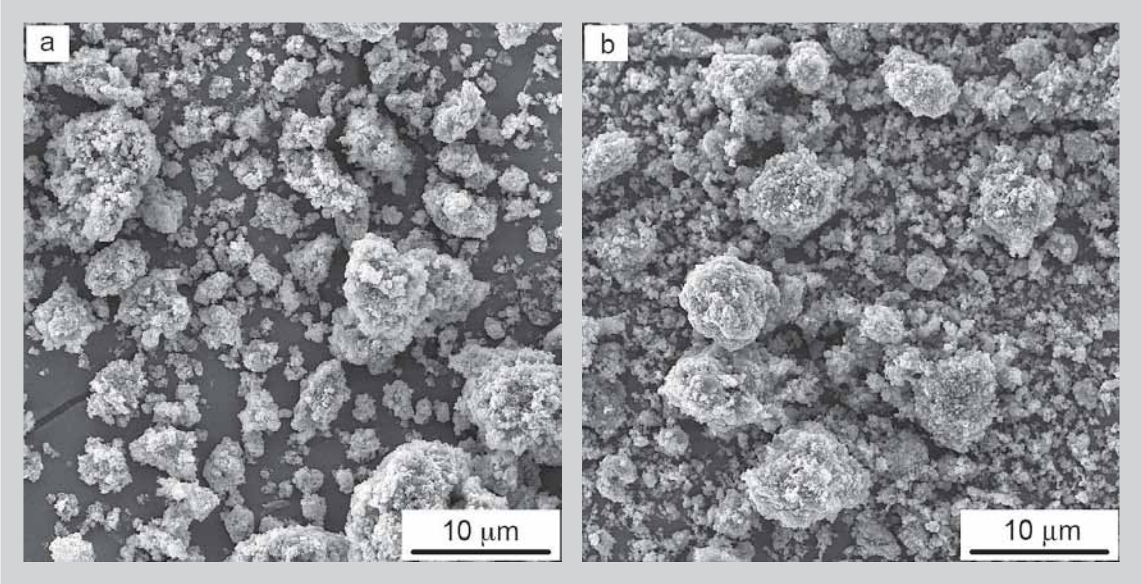

Figure 1: SEM micrographs of PANI powders protonated by different types of acids – PA5 (a) and TA5 (b).

magnitude occurs. Usually a yield stress also appears. As a result, these ER structures are also resistant to various types of forces. Nevertheless, as the shearing increases, the structures are gradually destroyed and viscosity decreases again. When the electric field is switched-off, the polarization of particles and their organization disappear and rheological properties of these smart fluids return to the original field-off state.

This phenomenon offers a manifold use in various hydraulic engineering applications and robotic control systems, such as clutches, gearboxes, valves or dumpers. Therefore, it has stimulated serious attention in the research of ER mechanism in both academic and industrial areas in the last three decades. The main ideas were reviewed in several papers [2–11]. Over time, originally water-based ER fluids [12, 13] were mostly replaced by anhydrous systems, which have higher temperature stability and do not evoke corrosion. Among them, a group of conducting polymers [14, 15] including e.g. polyaniline (PANI) or polypyrrole [16] seems to be promising.

In this paper, the ER behaviour of the suspensions made of PANI powders has been investigated. This polymer used in many studies [17–20] excels among other semiconducting ER materials due to low density, good thermal stability and controllable electric and dielectric properties by protonation [21–23]. From ER point of view, especially the electric behaviour of PANI makes the polymer so attractive. Its chemical structure given by alternating of σ- and π-bonds and the presence of charge carriers allows controlling conductivity of PANI by the protonation with various organic and inorganic acids to various extents [24–26]. As known from the literature, the conductivity of PANI can be also influenced efficiently by the reaction conditions during the polymerization of aniline [27]. In addition to the conductivity, the protonation usually increases the polymer density, changes the contact angles of PANI particles [19], and may influence the crystallinity [28]. The morphology of produced PANI, including nanofibers, nanotubes, granules, or microspheres, is also significantly affected by the way of polymer preparation [22, 29].

In our previous paper, the attention was focused on the investigation of the influence of protonation degree of PANI particles on the time dependences of the formation of ER structures [30]. The present study elucidates the factors controlling the rheological characteristics, dynamic yield stress and elastic modulus of the ER structures in silicone-oil suspensions of PANI particles protonated by two acids, phosphoric (PA) and tetrafluoroboric (TA) acids, providing a gradual increase in the conductivity as the acid concentration used for the protonation of PANI increases [23].

2 EXPERIMENTAL

2.1 CONTROLLED PROTONATION OF PANI WITH PA AND TA

PANI powder was prepared by the oxidation of 0.2 M aniline hydrochloride with 0.25 M ammonium peroxydisulfate in water at 20°C [31]. The obtained PANI salt (conductivity 4.4 S/cm) was transformed into the PANI base (conductivity $3.6 \cdot 10^{-9}$ S/cm) by two-day immersion in a fivefold molar excess of 1 M ammonium hydroxide (50 mL per 1 g of PANI salt), filtering the precipitate and drying in air. The portions of 0.5 g of PANI base were suspended in 100 mL of aqueous solutions of either PA or TA of various molar concentrations C_A. After 24 h the particles were separated, rinsed with acetone, and dried as above. The density of the powder was determined by weighing compressed pellets on Santorius R160P balance in air and immersed in decane.

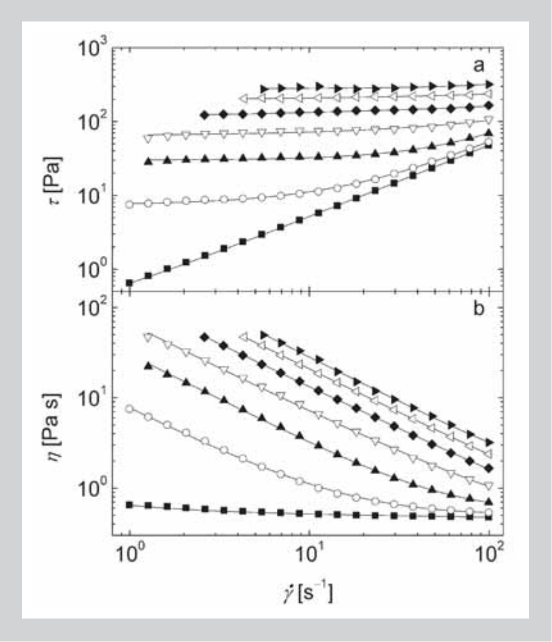

Figure 2 (left): Double-logarithmic plot of the shear stress, τ (a) and shear viscosity, η (b) versus shear rate, γ for the suspension of the sample PA5 at various electric field strengths, E (kV/mm^{-1}): (■) 0, (○) 0.5, (▲) 1.0, (▽) 1.5, (◆) 2.0, (◁) 2.5, (▸) 3.0. Lines are fits of Herschel-Bulkley Equation.

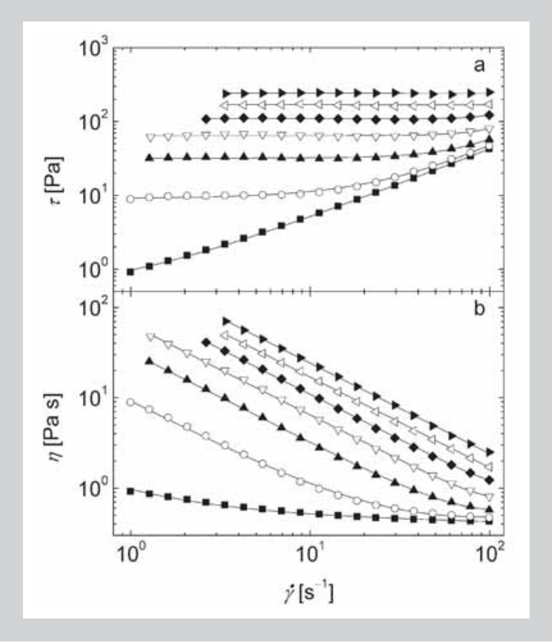

Figure 3: Double-logarithmic plot of the shear stress, τ (a) and shear viscosity, η (b) versus shear rate, γ for the suspension of the sample TA5 at various electric field strengths, E (kV/mm). Points are denoted in Figure 2.

2.2 PARTICLE MORPHOLOGY

The shape and size of the particles of protonated PANI powders were characterized by scanning electron microscopy (SEM, VEGA II LMU, Tescan, Czech Republic). The shape of particles is more or less spherical and the size is in order of several micrometers (Figure 1). Both types of doped particles resemble each other and the effect of different dopant seems to be negligible from morphological point of view.

2.3 CONDUCTIVITY MEASUREMENT

The DC conductivity, σ, of protonated PANI powders pressed at 700 MPa into pellets 13 mm in diameter and 1 mm thick, was determined either by a two-point method at $\sigma < 10^{-5}$ S/cm (Keithley 6517 electrometer) or by a four-point method at higher conductivities (a current source SMU Keithley 237 and a Multimeter Keithley 2010 voltmeter with a 2000 SCAN 10-channel scanner card) (Tab. 1).

2.4 SUSPENSION PREPARATION

Suspensions (10 wt%) were prepared by mixing PANI powders with corresponding amount of silicone oil (Lukosiol M200, Chemical Works Kolín, Czech Republic; η_c = 200 mPa·s, ρ_c = 0.965 $\text{g/cm}^{3,}$ $\sigma_c \approx 10^{-11}$ S/cm, ϵ'_c = 2.6). At first, the samples were stirred mechanically and then treated in an ultrasonic bath for 30 s before each measurement.

Table 1: The density, ρ and conductivity, σ of PANI base reprotonated in aqueous solutions of PA or TA of various molar concentrations C_A.

Sample	**CA [mol/l]**	ρ∀ ∀ [g/cm³]	σ∀ [S/cm]
B	0	1.132	3.6×10^{-9}
PA1 / TA1	0.0001	1.144/1.149	$4.7\times10^{-9}/4.9\times10^{-9}$
PA2 / TA2	0.0005	1.114/1.154	$6.8\times10^{-9}/4.9\times10^{-9}$
PA3 / TA3	0.001	1.112/1.142	$2.7\times10^{-8}/9.0\times10^{-9}$
PA4 / TA4	0.005	1.145/1.163	$5.8\times10^{-7}/1.6\times10^{-8}$
PA5 / TA5	0.01	1.264/1.157	$4.7\times10^{-5}/1.2\times10^{-5}$
PA6 / TA6	0.05	1.333/1.168	$3.1\times10^{-3}/2.5\times10^{-4}$

2.5 RHEOLOGICAL MEASUREMENT

Both steady shear rate viscometry (in the range of shear rates from 1 to 100 1/s) and dynamic oscillatory measurements in linear viscoelasticity region (angular frequency range from 0.5 to 50 rad/s) using a rotational rheometer (Bohlin Gemini, Malvern Instruments, UK) with parallel plates 40 mm in diameter and a gap of 0.5 mm were carried out at the temperature of 25°C. The chosen geometry was connected to a DC high-voltage source TREK (TREK 668B, USA) providing the electric field strength E = 0.5–3.0 kV/mm. Before each measurement, the electric field was applied for 60 s to obtain equilibrium ER structures.

2.6 DIELECTRIC MEASUREMENT

The angular frequency dependences of dielectric properties (ϵ', ϵ'') of 10 wt% suspensions were measured with a Hioki 3522 RCL HiTester in the range 62.8–6.28 10^5 rad/s. The dielectric data were used for the calculation of the relaxation time of PANI suspensions.

3 RESULTS AND DISCUSSION

To characterize the rigidity and elastic properties of the reinforced chain-like structure of polarized PANI particles in the electric field, the dynamic yield stress and the storage modulus evaluated from viscometry and dynamic oscillatory experiments have been used.

3.1 CONTROL SHEAR RATE VISCOMETRY

In the absence of electric field, where hydrodynamic properties of the suspensions depend on the size and shape of suspended particles and their aggregates and their interactions with suspension medium, the shear stress depended practically linearly on the shear rate and the

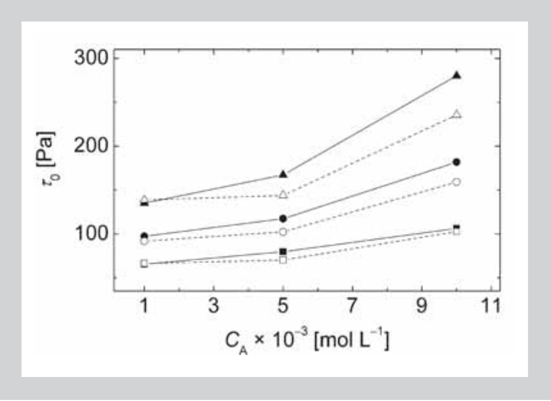

Figure 4 (left): Dependence of the dynamic yield stress, τ_o on the molar concentration of acid used for protonation of PANI particles at various electric field strengths, E (kV/mm): (■□) 2.0, (●○) 2.5, (▲△) 3.0. PA (solid) and TA (open symbols).

shear viscosity was almost independent of the shear rate. When the electric field was applied, the low-shear apparent viscosity significantly rose by several orders of magnitude and the flow curves became highly pseudoplastic.

The characteristic flow curves of the suspensions of the samples PA5 and TA5 at various electric field strengths are shown in Figures 2 and 3. A plateau region of the shear stresses at low shear rates is due to the balance between electric and shear forces. At higher shear rates, when the hydrodynamic forces begin to dominate over the electrostatic ones, these organized particle structures are gradually destroyed and the suspension tends to return to a field-off nearly Newtonian behaviour. The dynamic yield stresses, τ_o were estimated by extrapolation of the shear stresses to the zero shear-rate using the Herschel-Bulkley Equation [32]

$$\tau = \tau_o + \eta_{pl}\gamma^n \tag{1}$$

Here η_{pl} is a plastic viscosity and the exponent n characterizes the pseudoplastic decrease of the viscosity of the system with the rate of shear. It is clear that this function approximates the course of experimental points very well (Figures 2 and 3). At low acid concentrations ($C_A < 0.001$) the yield stresses for PA and TA samples did virtually not differ and had practically similar values. Only samples with three higher protonation degrees at 0.001, 0.005 and 0.01 mol/L (PA3 / TA3, PA4 / TA4 and PA5 / TA5) proved to be interesting for a detailed study. In the doped samples PA6 / TA6 the current passing through the suspensions was too high; short-circuit between electrodes set in and the measurement failed.

The plots of the yield stress on the molar acid concentration reveals that, at the electric field strengths 2.5 and 3.0 kV/mm and at the same molar acid concentration used for PANI protonation, the ER intensity of the samples PA4 and PA5 is higher than that of TA4 and TA5 (Figure 4). At lower electric field strengths the yield stresses at all three acid concentrations merge. For that reason the dynamic oscillatory experiments and final comparison of the effect of acid protonation were performed only at E = 2.0, 2.5 and 3.0 kV/mm.

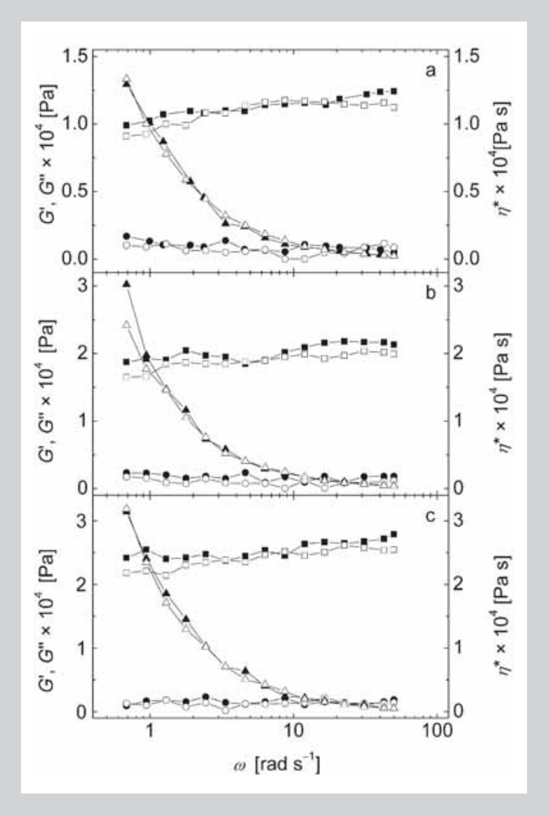

Figure 5 (right above): The angular frequency, ω dependence of the storage modulus, G' (■□), loss modulus, G'' (●○) and complex viscosity, η^* (▲△) for suspensions PA3 and TA3 at various electric field strengths, E (kV/mm): (a) 2.0, (b) 2.5, (c) 3.0. PA (solid) and TA (open symbols).

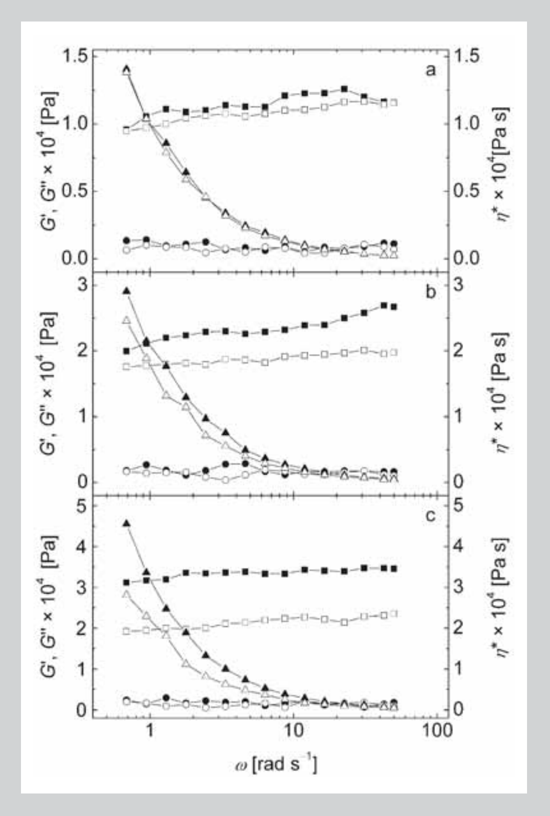

Figure 6 (right below): The angular frequency, ω dependence of the storage modulus, G' (■□), loss modulus, G'' (●○) and complex viscosity, η^* (▲△) for suspensions PA4 and TA4 at various electric field strengths, E (kV/mm). Points are denoted in Figure 5.

3.2 DYNAMIC OSCILLATORY EXPERIMENTS

At the zero electric field strength, the loss modulus is higher than the storage one and the suspension behaved rather like a fluid. On the other

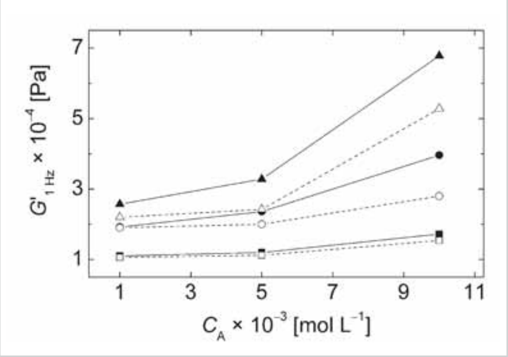

Figure 7 (left): The dependence of the storage modulus, G' (in frequency f = 1 Hz) on the conductivity of the samples. Points are denoted in Figure 4.

hand, when electric field was applied, the storage modulus dramatically increased and dominated over the loss one in the whole frequency range because of high elasticity of the system. A steep decrease of complex viscosity with frequency means a strong pseudoplasticity of polarized particle structure. At all three applied electric field strengths, the courses of the angular frequency dependences of the storage modulus, *G'*, the loss modulus, *G''* and the complex viscosity, η^* of the PA3 and TA3 samples are alike and the different effect of protonation is not apparent (Figure 5). In case of the samples PA4 and TA4 at the electric field strengths 2.5 and 3.0 kV/mm especially the storage modulus for suspension of particles doped with the PA predominates (Figure 6). At the highest used molar acid concentration the differences between samples PA5 and TA5 became more pronounced.

From the above discussion it is apparent that also the elasticity of the ER structure of the particles protonated with the solutions of the same concentration of PA and TA significantly differs. The storage modulus at the frequency 1 Hz (G'_{1Hz}) chosen as a criterion of this characteristics displays similar behaviour as the yield stresses of the suspensions (Figure 7).

3.3 THE ROLE OF PARTICLE CONDUCTIVITY

The results suggest that the doping effect of various acids on rigidity or elasticity of the ER structure of polarized particles depends not only on the protonation degree of PANI particles. According to the polarization theory [33], in the suspensions of low conducting particles permittivity plays the main role in their polarizability. For more conducting materials, however, the effect of particle conductivity prevails and becomes a crucial factor for intensity of polarization forces [34, 35]. In the case of the PA and TA samples under investigation, where particle conductivity was rather high, we found that unlike the molar acid concentration, the particle conductivity, σ is a main factor controlling mechanical properties of the chain structure of the polarized particles in the electric field. As a result, the correlations of τ_o or G'_{1Hz} with σ provided common dependences for both used acids (Figure 8).

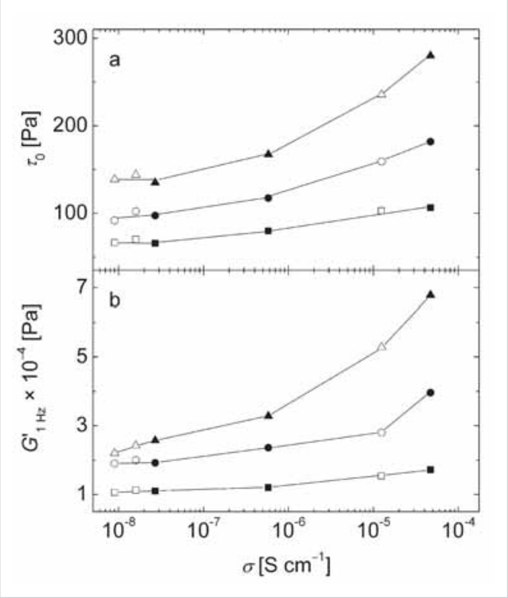

Figure 8 (right above): The dependence of the dynamic yield stress, τ_o (a) and storage modulus, G' (in frequency f = 1 Hz) (b) on the conductivity of the samples. Points are denoted in Figure 4.

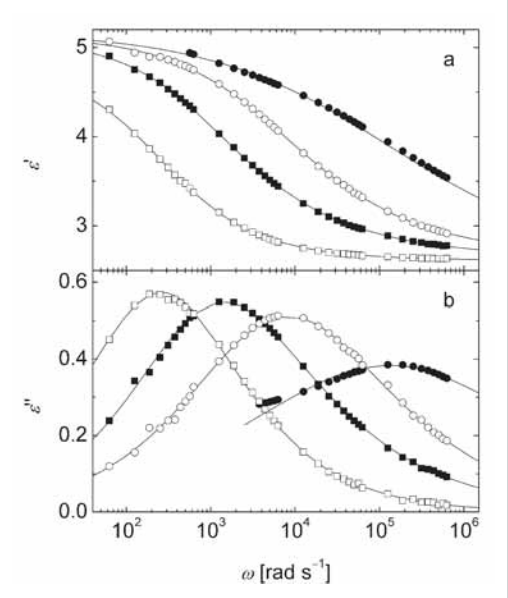

Figure 9 (right below): Frequency spectra of relative permittivity, ϵ' (a) and dielectric loss factor ϵ'' (b) for PANI suspensions protonated either PA (solid) or TA (open symbols). Molar concentration of acid C_A (mol/L) used for reprotonation of PANI base: (■□) 0.005, (●○) 0.01. Lines are fits of Havriliak-Negami Equation.

3.4 PARTICLE MOBILITY

The rate of particle polarization is related to a dielectric relaxation time, t_{rel}, inversely proportional to dielectric relaxation frequency, ω_{rel}, at which the inflection on the permittivity decrease sets in and the dielectric loss factor, ϵ'', attains a local maximum. The angular frequency spectra of relative permittivity, ϵ', and dielectric loss factor, ϵ'', of the suspensions of PA and TA samples show a distinct shift of the relaxation frequency with protonation degree to higher values (Figure 9). It appeared that the corresponding relaxation times decrease with particle conductivity regardless of the acid used for protonation (Figure 10). Thus, it may be assumed that, also in case

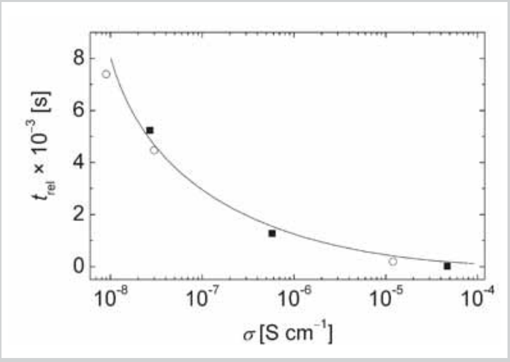

Figure 10: The dependence of relaxation time, t_{rel} on the conductivity, σ of the PANI particles. PA (solid) and TA (open symbols).

of the higher conducting particles, the conductivity is an important factor controlling their mobility in the electric field. Measured dielectric values were approached by virtue of Havriliak-Negami empirical Equation [36].

$$\epsilon^* = \epsilon'_\infty + \frac{\left(\epsilon'_o - \epsilon'\right)}{\left(1+\left(i\omega t_{rel}\right)^a\right)^b} \tag{2}$$

Here, ϵ'_o and ϵ'_∞ are the limit values of relative permittivity at the frequencies below and above the relaxation frequencies, t_{rel} is a relaxation time, a is the scattering degree of t_{rel} and b is related to the asymmetry of the t_{rel} spectrum. Large values of a mean a great scattering of t_{rel}. When a differs much from zero and b significantly from unity, the t_{rel} spectrum becomes more asymmetrical. In our case, however, the spectra are practically symmetric and the parameter b tends to one. Thus the Havriliak-Negami Equation changes to the Cole-Cole Equation

$$\epsilon^* = \epsilon'_\infty + \frac{\left(\epsilon'_o - \epsilon'_\infty\right)}{1+\left(i\omega t_{rel}\right)^a} \tag{3}$$

often successfully used for evaluation of dielectric properties of ER suspensions [37, 38].

4 CONCLUSIONS

Our findings revealed, that protonation of PANI particles with various acids at a same relatively high protonation degree may provide material with different ER property. On the other hand, conductivity of particles proved to be a universal factor controlling both viscoelastic properties of the suspension material as well as particle mobility in the electric field.

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