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Comparison of the effect of impurities with different structural organization on the electrical properties of a frozen moisture-containing dispersed system

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Abstract. This article presents the results of experimental studies into the frequency dependences relating to the specific electrical conductivity of frozen water-containing dispersed systems. The focus is on real Arctic soil models in which ice with dissociating impurities of acids and non-dissociating impurities of saccharides acts as a dispersed phase. The studies were conducted at temperatures from 130 to 270 K and the frequency of the external electric field from 25 Hz to 1 MHz. A frozen dispersed system with impurities of orthophosphoric, sulfuric, nitric acids, fructose, and sucrose is examined for static and high-frequency conductivity, relaxation time, and orientation and ionic defect conductivity based on the Debye equation, the Jaccard model, and the theory of ionic thermal polarization. Comparison of how effectively impurities affect the specific electrical conductivity of a frozen dispersed system is given. A model of frozen water-containing systems is proposed to explain the electrical properties of pure systems and systems with impurities.

Keywords: water-containing dispersed system, specific electrical conductivity, ice, dissociating impurities, activation energy, relaxation time

Introduction

Frozen water-containing dispersed systems (WDS), in which ice with dissociating and non-dissociating impurities acts as a dispersed phase, are widespread in nature, in particular in the circumpolar Arctic region. When studying the electrophysical properties, such soils are modeled with fine-grained quartz powder with a low specific electrical conductivity of 10^{-15} S/m (Koposov 2004; Petrenko, Whitworth 2006), while the specific electrical conductivity of ice and distilled water is 1000 times greater (Zatsepina 1974).

According to the WDS model (Volkov, Koposov 2021), charges in an electric field can move through a film of bound water or through a layer of ice. In this case, the charge of the bound water film is positive. It appears at the boundary with the granules of the dispersed medium, as well as at the boundary of bound water and ice. The atoms of the dispersed medium particles will move towards the ice (Koposov 2004; Koposov, Tyagunin 2013; Tonkonogov 1998; Volkov, Koposov 2021), and an excess of negative charges will appear on the intergranular ice layer due to orientation defects and ions. This causes an increased concentration of negative carriers in the pure ice.

When dissociating impurities are added to water, the impurity ions will be embedded in the crystal structure of the ice during freezing. H^+ ions increase the concentration of either D^+ defects or H_3O^+ ions (Koposov 2004; Tonkonogov 1998; Zatsepina 1974). The negative ions of the acid residue surround the positive D^+ or bound H_3O^+ ions.

The conductivity in this kind of WDS is due to the presence of defect carriers, orientation and ionic defects in the ice structure with inclusions of impurity molecules, ionic defects of acid residues, and static defects of large molecule inclusions. Carrier generation depends on the humidity and temperature of the sample, as well as the type and number of defects through which conduction is carried out.

The first phenomenological theory of the electrical properties of ice was proposed by Jaccard (1959). The main provisions of this theory are described in a review (Tonkonogov 1998) and a monograph (Petrenko, Whitworth 2006).

Methods

Fine-grained quartz powder was used as a soil model, acting as a dispersed medium in a frozen moisture-containing dispersed system. The dispersed phase was ice with dissociating impurities of monobasic HNO_3, dibasic H_2SO_4, and tribasic H_3PO_4 acids, and non-dissociating impurities of saccharides — monosaccharide $C_6H_{12}O_6$ and disaccharide $C_{12}H_{22}O_{11}$. The concentration of impurities ranged from 10^{-6} to 10^{-2} M, with the WDS humidity of 12%.

The acid residue of the dissociating impurity is NO^{3-}, SO_4^{2-}, and $PO_4^{3-.}$ In the ice structure, it can replace a crystal lattice node or be embedded in it (Koposov 2004; Volkov, Koposov 2021). Saccharide molecules are much larger than the structural units of the crystal lattice of ice, and the molecules exist as inclusions in the structure of the dispersed phase.

The specific electrical conductivity was determined using a measuring stand based on an E7-20 immittance meter (Belarus). The studied frequency range of the immittance meter ranges from 25 Hz to 1 MHz with an adjustable frequency step. A WDS sample with an admixture of various concentrations was placed in a measuring cell-a flat capacitor connected to the terminals of an immittance meter. Cryostatization was carried out using liquid nitrogen in a thermostat by selecting the heater power of the chamber in which the measuring cell was placed. Thus, the measurements were carried out in the temperature range from 130 to 270 K.

Taking into account the measurement of the conductivity G of a flat capacitor, the specific electrical conductivity of a substance was calculated using the formula

$$\sigma = \frac{(G - G_0)d}{S}, \tag{1}$$

where G_0 and G are the conductivities of an empty and filled cell, respectively, S is the area of the electrodes, and d is the distance between the cell plates.

The relative error in determining the values of specific electrical conductivity at a frequency of 1 kHz did not exceed 3%.

Results and Discussion

Based on the calculated values of σ according to equation (1), frequency dependences σ = f(ω) were obtained at a given temperature (Fig. 1). An example of frequency dependences of specific electrical conductivity for a WDS sample with a humidity of 12% is given a) with an admixture of orthophosphoric acid with a concentration of 10^{-4} M at different temperatures and b) with an admixture of fructose of various concentrations at temperatures of 250, 210, and 170 K.

Characteristics of electrical properties included the following parameters: low-frequency (static) σ_S and high-frequency σ_∞ specific conductivities and temporary relaxation of specific electrical conductivity τ. The characteristic parameters of the dependencies σ_S and σ_∞ were determined through a multinomial representation of the dependencies σ(ω) or σ(1/ω) by the free term for ω→0 and 1/ ω→0 (Volkov, Koposov 2018; Volkov et al. 2016).

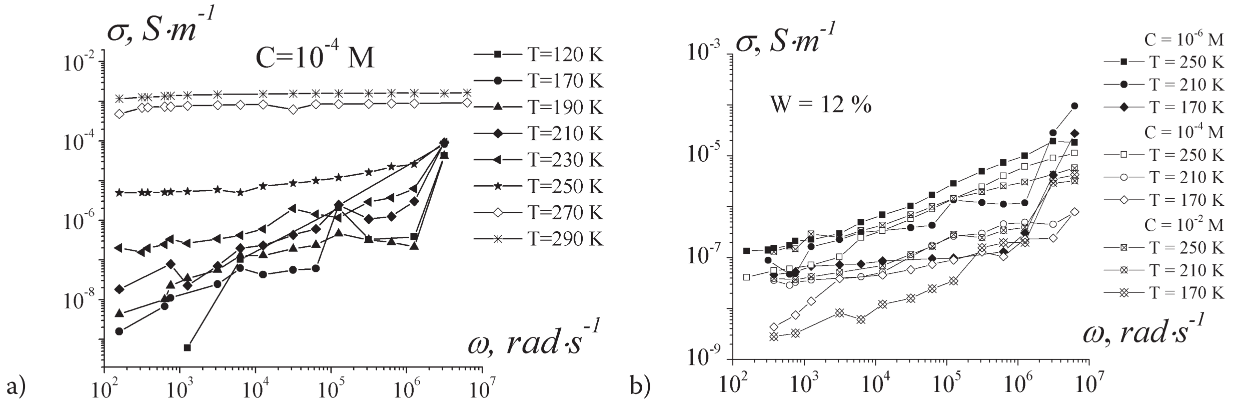

Fig. 1. Frequency dependence of the specific electrical conductivity of WDS with humidity W = 12%
(a) with an admixture of H_3PO_4 at a concentration of 10^{-4} M at different temperatures
and (b) with an admixture of fructose of various concentrations at different temperatures

It should be noted that for some moisture-containing dispersed systems, the dependence σ(ω) in the first approximation (Koposov 2004; Volkov, Koposov 2018; 2021; Volkov et al. 2017) is satisfactorily described by the Debye frequency dispersion

$$\sigma(\omega) = \sigma_s + \frac{\sigma_\infty - \sigma_S}{1+\omega^2\tau^2}. \tag{2}$$

In this case, the selection of the parameters σ_S and σ_∞ and the relaxation time τ was carried out using software (Volkov 2020), which allows approximating the experimental curves with a given function. Using software, the experimental curves (Fig. 1) were approximated by a function of the form (2) to determine static and high-frequency electrical conductivity, as well as relaxation time.

Fig. 2 shows the temperature and concentration dependences of these characteristics for all the impurities studied at a sample humidity of 12%. : For saccharide impurities, a higher temperature is observed than for acid impurities at the beginning of carrier generation, determined by the intensity of the increase in σ_S values and, as a result, a higher activation energy. The concentration dependence for non-dissociating impurities is weak, and for acid impurities, the values of both static and high-frequency conductivity increase as impurity concentration rises. The differences between the temperature dependences of the high-frequency components of the specific electrical conductivity of various types of impurities are insignificant; for saccharide impurities up to 200 K, there is a range of constant values associated with insignificant defect generation. The relaxation time values for saccharide impurities are lower in comparison with dissociating impurities, and the concentration dependence is weak.

The behavior of the electrical properties of WDS samples, primarily with dissociating impurities, is undoubtedly related to the activity of the electrolyte solution. According to (Popova 2007; Vasilev 2002), the activity a of an aqueous electrolyte solution can be calculated using the formula

$$a = f \cdot C, \tag{3}$$

where f is the average activity coefficient, and C is the molar concentration of the impurity in the solution. The average activity coefficient for the A_mB_n electrolyte is related to the activity coefficients of individual ions f_1 and f_2:

$$f = \sqrt[m+n]{(f_1)^m \cdot (f_2)^n}\,. \tag{4}$$

The values of the activity coefficients of individual ions were calculated using the Debye and Hückel formulas (Popova 2007):

$$\begin{cases} \lg(f) = -A \cdot Z_i^2 \cdot \sqrt{\mu}, \text{ with } \mu < 0.01 \\ \lg(f) = -\dfrac{A \cdot Z_i^2 \cdot \sqrt{\mu}}{1 + Bb \cdot \sqrt{\mu}}, \text{ with } 0.01 < \mu < 0.1 \end{cases}, \tag{5}$$

where A and B are constants depending on the temperature and dielectric constant of the solvent (for water, A = 0.5, B = 0.33), Z is the ion charge, and b is the empirical constant, v ≈ 3 Å. For more accurate

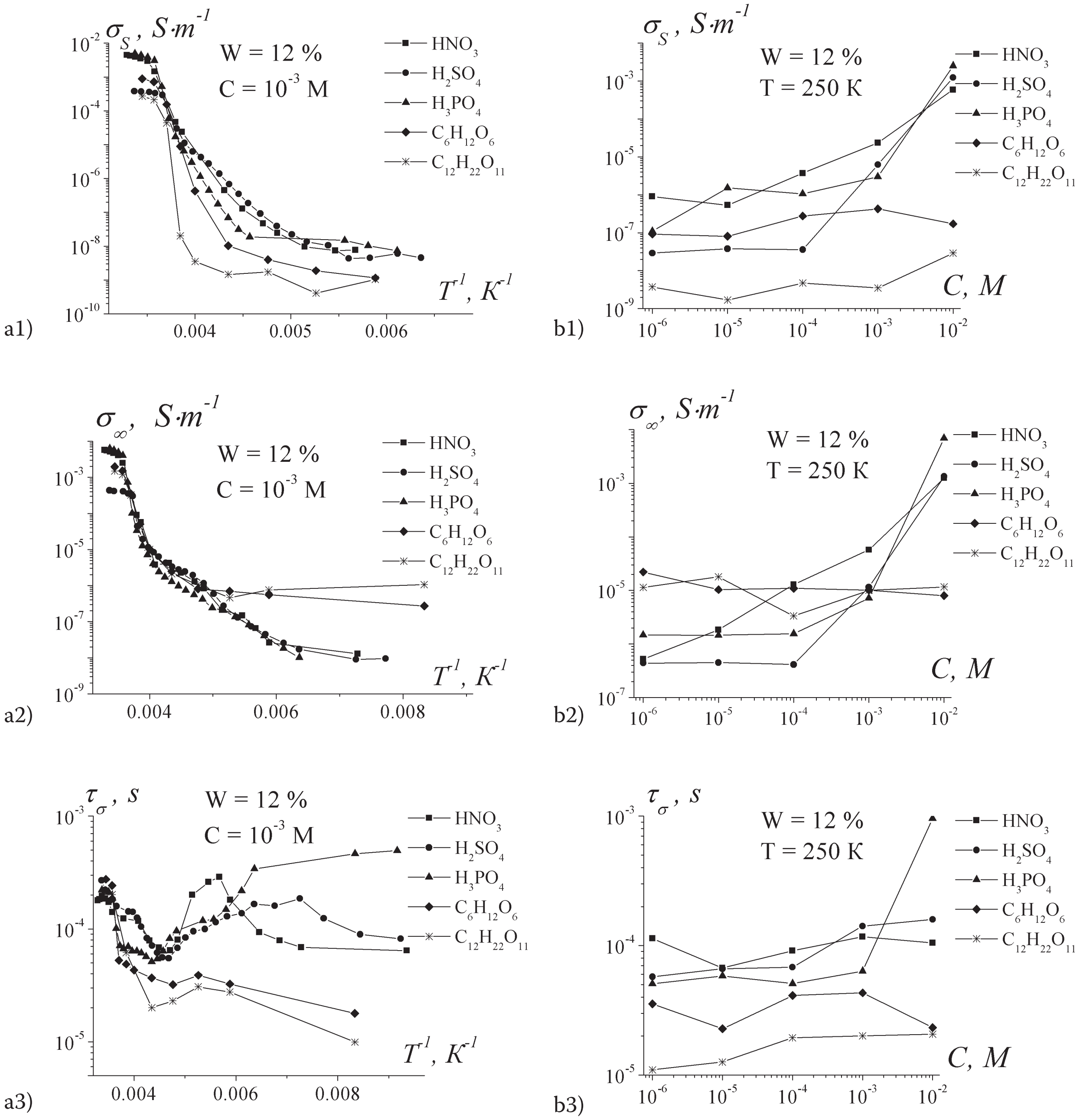

Fig. 2. Dependences of static σ_S, high-frequency σ_∞ specific electrical conductivity, and relaxation time τ of WDS with various impurities on a) temperature at C = 10^{-3} M and b) impurity concentration at T = 250 K

calculations, tabular data were used (Lure 1989; Ravdel, Ponomareva 2002) at the studied impurity concentrations, 0 °C temperature, and normal pressure. In equation (5), m is the ionic strength of the solution, which determines the magnitude of the electrostatic interaction between ions, calculated using the expression

$$\mu = 0.5 \cdot \sum C_i \cdot Z_i^2 \, . \tag{6}$$

The values of the calculated activity of the solutions and the ionic strength of the solution with acid residues are shown in Table 1 (for t = 0 °C).

For the concentration dependences of the characteristic parameters with dissociating impurities, the intensive growth of the parameters begins with concentrations of 10^{-5} M for the HNO_3 impurity, 10^{-4} M for H_2SO_4, and 10^{-3} M for H_3PO_4. This is due to the degree of dissociation of the corresponding

Table 1. Values of the activity of solutions of dissociating impurities and the ionic strength of the solution

	a, m/l			K_d		
C, M	NO_3^-	SO_4^{2-}	PO_4^{3-}	HNO_3	H_2SO_4	H_3PO_4
10^{-6}	9.99×10^{-7}	9.94×10^{-7}	9.79×10^{-7}	43.6	1×10^{3} 1.2×10^{-2}	7.52×10^{-3} 6.31×10^{-8} 1.26×10^{-12}
10^{-5}	9.97×10^{-6}	9.80×10^{-6}	9.35×10^{-6}			
10^{-4}	9.92×10^{-5}	9.39×10^{-5}	8.14×10^{-5}			
10^{-3}	9.76×10^{-4}	9.28×10^{-4}	5.56×10^{-4}			
10^{-2}	9.29×10^{-3}	5.93×10^{-3}	2.60×10^{-3}			

acids. Despite the fact that the activity of sulfuric acid is greater than that of nitric acid, the latter is more likely to dissociate into a proton and an acidic residue, while sulfuric acid dissociates into a proton, HSO_4, then H^+, and SO_4^2. The amount of sulfuric acid residue ions is determined by the impurity concentration. Orthophosphoric acid of medium activity dissociates into H^+, H_2PO_4, HPO_4^2, and PO_4^3 in solutions, and the existence of the H_2PO_4 ion is highly probable.

According to Jaccard's theory (Jaccard 1959), the following expressions are valid for electrical conductivity:

$$\frac{e^2}{\sigma_{\delta r}} = \frac{e_{or}^2}{\sigma_{ion}} + \frac{e_{ion}^2}{\sigma} \tag{7}$$

and

$$\sigma_\infty = \sigma_{or} + \sigma_{ion}\,, \tag{8}$$

where σ_{or} and σ_{ion} are the contributions of orientation (L and D) and ionic (H_3O^+ and OH^-) defects to electrical conductivity, respectively, $e_{or}/e = 0.38$ and $e_{ion}/e = 0.62$. Then

$$\frac{\sigma_\infty}{\sigma_S} = (\sigma_{or} + \sigma_{ion})\left(\frac{0.62^2}{\sigma_{ion}} + \frac{0.38^2}{\sigma_{or}}\right) \text{ and } \frac{\sigma_\infty}{\sigma_{\delta r}} = 0.62^2 + 0.38^2 + 0.62^2\frac{\sigma_{or}}{\sigma_{ion}} + 0.38^2\frac{\sigma_{ion}}{\sigma}.$$

By entering the notation $X = \frac{\sigma_{or}}{\sigma_{ion}}$, we obtain a quadratic equation for finding X:

$$0.62^2X^2 + \left(0.62^2 + 0.38^2 - \frac{\sigma_\infty}{\sigma_S}\right)X + 0.38^2 = 0 \tag{9}$$

Let us analyze the dependences of the ratio of orientational and ion defect conductivities on the temperature and impurity concentration shown in Fig. 3.

The predominance of orientation defects is typical for all types of impurities in the region of negative temperatures. Depending on this ${}^{\sigma_{or}}\!/_{\sigma_{ion}} = f\left({}^{1}\!/_{T}\right)$, an extremum is observed near T = 190 K for impurities of dissociating acids and T = 250 K for saccharide impurities. This inflection corresponds to the beginning of ionic defect generation; for saccharide impurities, it is primarily associated with the appearance of a liquid phase. From the concentration dependence ${}^{\sigma_{or}}\!/_{\sigma_{ion}} = f(C)$ at T = 250 K, it follows that for acid impurities, the generation of ionic defects begins at a concentration of C < 10^{-4} M, for saccharides at C < 10^{-5} M, and for sucrose impurities, the dependence is weaker than for fructose impurities. The concentration dependence is primarily related to a decrease in the melting point and pre-melting of the dispersed phase with an increase in the impurity concentration.

The effect of impurities of various concentrations on the values of orientational and ionic defect conductivities, depending on the temperature and concentration of the impurity, is shown in Fig. 3. An admixture of acids makes a positive contribution to the conductivity of orientation and ionic defects in the region of negative temperatures, in contrast to impurities of saccharides. Moreover, the maximum

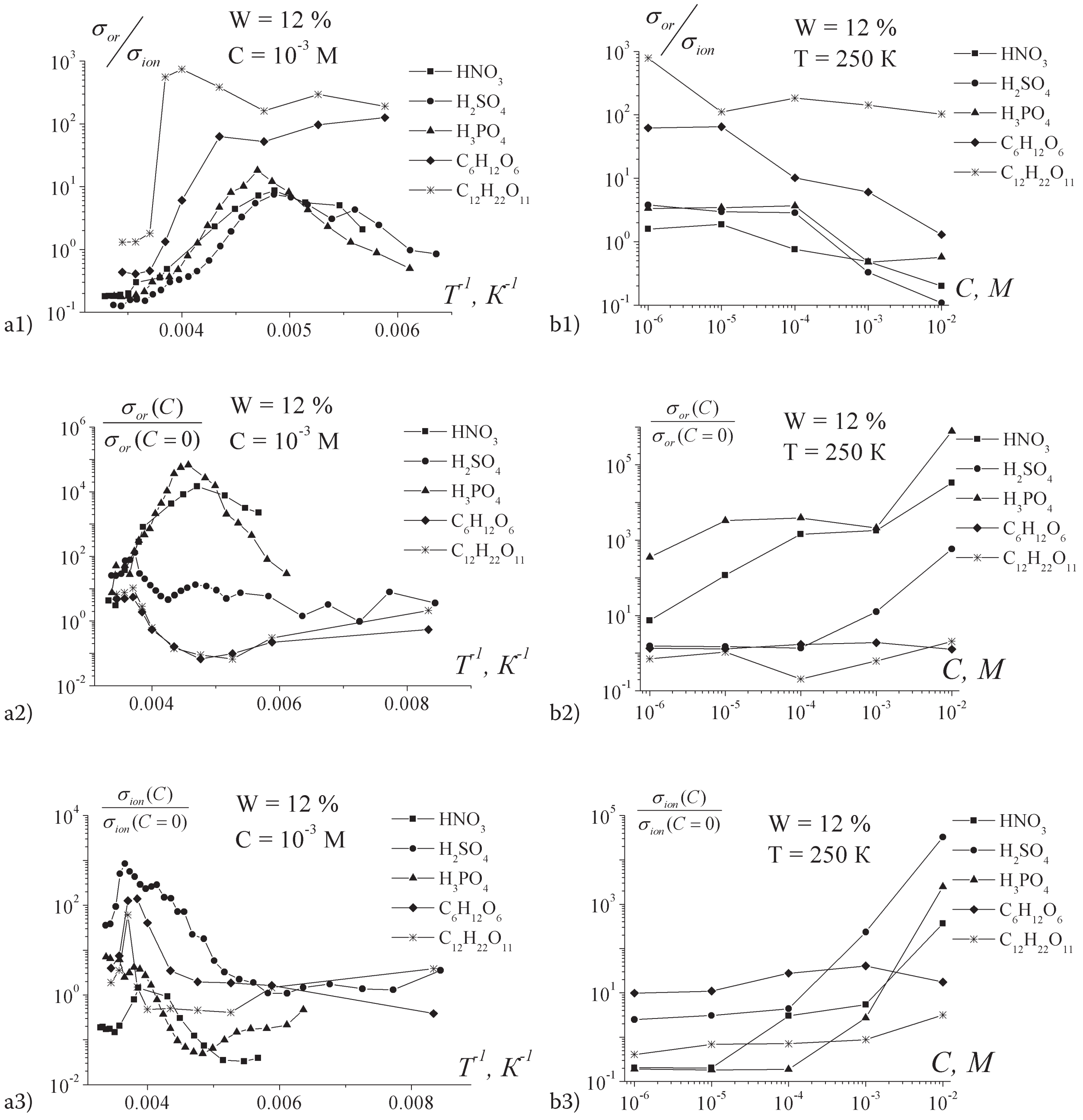

Fig. 3. Dependence σ_{or}/σ_{ion} of the effectiveness of the impurity effect on the conductivity values for orientation and ion defects of WDS with various impurities on a) temperature at C = 10^{-3} M and b) impurity concentration at T = 250 K

effect is on σ_{or} for impurities of nitric and orthophosphoric acids, and on σ_{ion} for sulfuric acid. For dissociating impurities, the contribution to σ_{or} and σ_{ion} depends on the impurity concentration, while for non-dissociating impurities, the concentration dependence is weak.

The graphs obtained confirm the effect of the impurity on the specific electrical conductivity depending on the activity of the ions of the acid residue.

In accordance with the theory of ion relaxation polarization (Koposov 2004; Koposov, Tyagunin 2013), electrical conductivity

$$(\sigma_{\infty} - \sigma_s) = \frac{q^2 \delta^2 n}{12 \tau k T}, \tag{10}$$

where q is the ion charge, n is the ion concentration, δ is the ion jump length, and τ is the relaxation time. Then

$$(\sigma_\infty - \sigma_s)\cdot T\cdot \tau = \frac{q^2\delta^2 n}{12k}\ . \tag{11}$$

The dependence $(\sigma_\infty - \sigma_s)\cdot T = f(T)$ (Fig. 4) then contains information about the temperature dependence of the ion concentration n(T). Therefore, $(\Delta\sigma_\infty^{imp} - \Delta\sigma_s^{imp})\cdot T\cdot \tau$, provides information about the dependency $\Delta n^{imp}(T)$.

In the region of negative temperatures near 0 °C, a sample with an admixture of nitric acid is a conductor for a given concentration, which is atypical of other dissociating impurities. The concentration dependence (Fig. 4b) suggests that the carrier concentration rises with increasing impurity concentration from $C=10^{-4}$ M. For WDS samples with non-dissociating impurities, the temperature dependence is identical, and the concentration dependence does not appear.

Equations (7)–(11) make it possible to determine the concentration of orientation defects using the formula

$$n_{or} = \frac{\sigma_{or} m}{0.38^2 e^2 \tau}\ . \tag{12}$$

The values of the relaxation time of the specific electrical conductivity t were in accordance with Fig. 2. The obtained dependences of the concentration of orientation defects are very informative and shown in Fig. 4. An increase in the sample temperature naturally leads to a rising concentration of orientation defects. For impurities of sulfuric and orthophosphoric acids, an extremum is observed near T = 250 K, associated with the pre-melting of the sample. For impurities of non-dissociating acids up to T = 230 K, there is a slight increase in the number of orientation defects, which is related to the structure of the impurity molecules and the location in the crystal lattice of the ice.

The activation energies obtained from Fig. 4 coincide with the activation energies of orientation defects from Fig. 3, which allows us to confirm the satisfiability of formula (12).

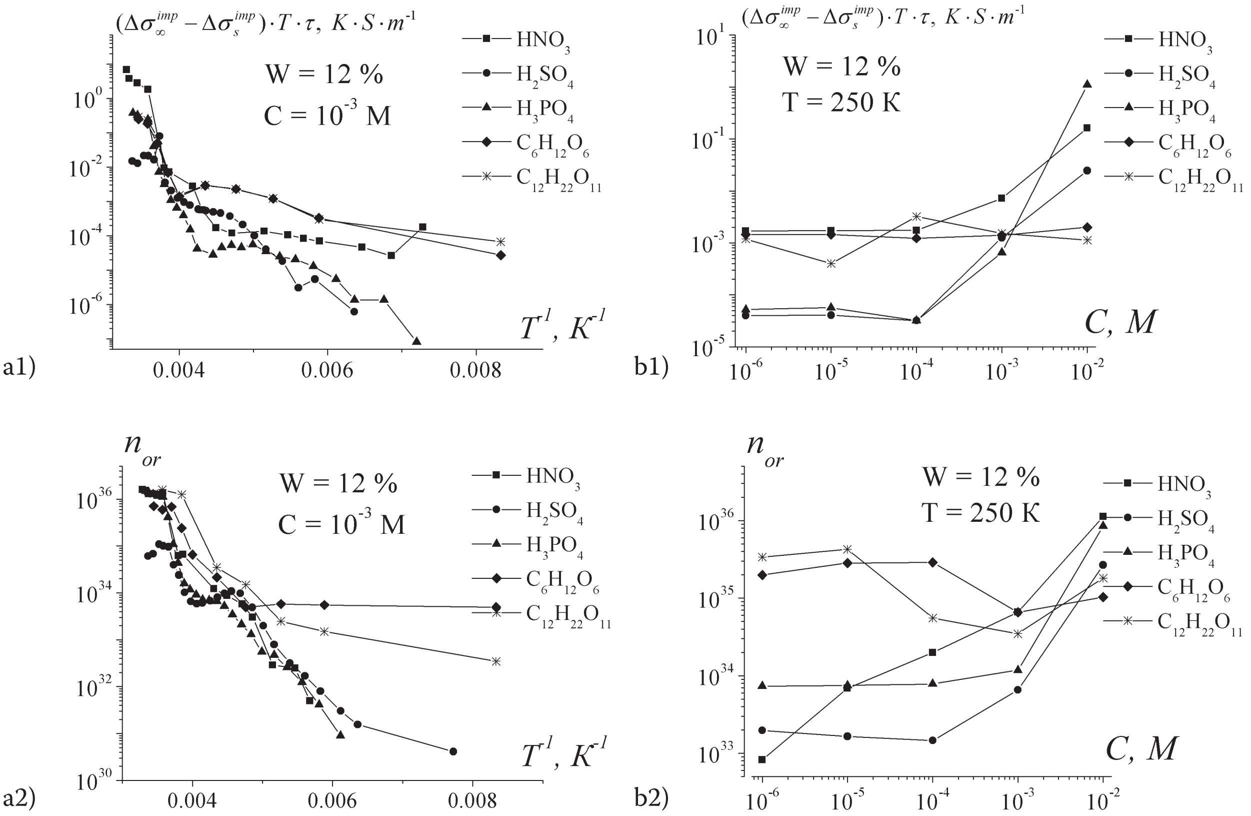

Fig. 4. Dependence of impurity carrier additives and the number of orientation defects of the studied samples on a) sample temperature and b) impurity concentration

Depending on the type of impurity, the values of activation energies vary from 0.4 eV for non-dissociating impurities to 0.6 eV for acid impurities. For acid impurities, an increase in the impurity concentration leads to more orientation defects, and for sulfuric and orthophosphoric acid impurities at T = 250 K in the range from 10^{-6} to 10^{-4} M, the concentration dependence does not appear. For saccharide impurities, a rise in the impurity concentration leads to a decrease in the number of orientation defects. Such a difference in the temperature and concentration dependences of impurity additives and orientation defects for different types of impurities is due to the different position of impurity molecules in the ice structure, and for dissociating acids, it is also due to the different activity of electrolyte solutions. Similar dependences will be observed for the concentration of ionic defects.

In addition to the effect of impurities on the values of σ_S, σ_∞, σ_{or}, and σ_{ion} shown in Figs. 2 and 3, comparisons of the reduced values of the specific electrical conductivity to bulk ice in relation to the reduced specific electrical conductivity for bulk ice are presented, which is of undoubted interest. The values can be calculated using the formula

$$\sigma^{imp}_{s/\infty} = \sigma_{s/\infty} \frac{\rho_{ice} V_{cell} (1+W)}{m_{WDS} W} \quad , \tag{12}$$

where ρ_{ice} is the bulk ice density, V_{cell} is the cell volume, and m_{WDS} is the mass of the dispersed system 'fine-grained quartz — an aqueous solution with an impurity'.

The temperature and concentration dependences of the values $\frac{\sigma_S^{imp}}{\sigma_S^{imp}(C=0)}$ and $\frac{\sigma_\infty^{imp}}{\sigma_\infty^{imp}(C=0)}$ are shown in Fig. 5, a) and b) respectively.

The graphs confirm what has been said about the effectiveness of the impurity effect on static and high-frequency specific electrical conductivity, as well as ionic and orientation defect conductivity.

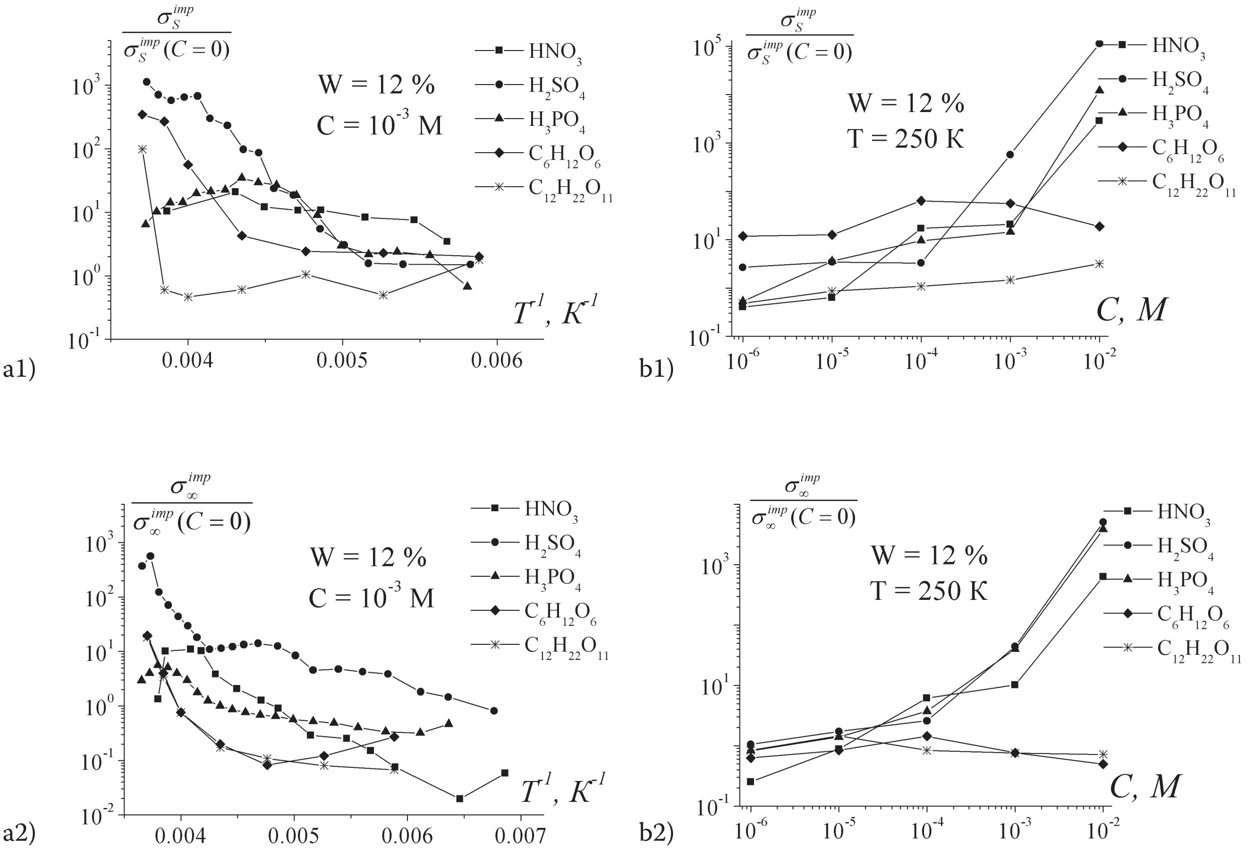

Fig. 5. Dependence $\frac{\sigma_S^{imp}}{\sigma_S^{imp}(C=0)}$ and $\frac{\sigma_\infty^{imp}}{\sigma_\infty^{imp}(C=0)}$ on a) sample temperature and b) impurity concentration

Conclusion

A comparison of the electrical characteristics of frozen WDS with a matrix of fine-grained quartz powder with various types of impurities suggests a number of conclusions. The specific electrical conductivity is affected by the ability of an impurity to dissociate. Dissociating impurities have a greater effect on the magnitude of the static and high-frequency components of electrical conductivity. They are characterized by an increase in parameters with increasing concentration, while the concentration dependences of the parameters of non-dissociating impurities are weak. Orientational and ionic defect conductivities are most pronounced for dissociating impurities in the temperature range of 200–260 K. As the number of impurity molecules rises, the concentration of orientational and ionic defects increases. The effect of an impurity on the values of static and high-frequency electrical conductivity reduced to bulk ice indicates a different effect of different types of impurity.

This is due to several factors. Firstly, as mentioned above, impurity molecules (and their acid residues) occupy different positions in the structure of the crystal lattice of ice. Ions of the acid residue of nitric acid replace the ice lattice site; ions of sulfuric and orthophosphoric acid residues are embedded in the lattice, and molecules exist as inclusions. Secondly, the dissociation of different acids releases a different number of H+ protons, which primarily affects the conductivity of orientation defects. Thirdly, the studied impurities have different activities, with the lowest observed in non-dissociating impurities.

The electric current in moisture-containing dispersed systems has two paths: through a film of bound water and through an ice layer (Fig. 6).

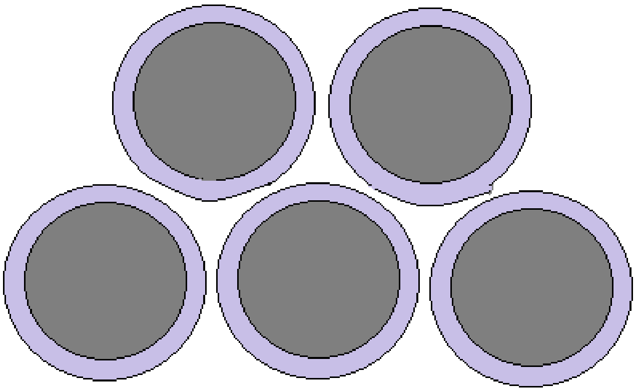

Fig. 6. Model of water-containing dispersed systems

The peculiarity of the bound water film is not so much in its small geometric dimensions, but in the fact that it has a positive charge that appears both at the boundary with granules and at the boundary with ice. Another feature is the displacement of foreign atoms towards the ice. Thus, an excess of negative charges is located on the intergranular ice layer. These negative charges are associated with orientation defects and OH-ions. From what has been said it should be assumed that there is an increased concentration of negative carriers in pure ice.

Conflict of Interest

The authors declare that there is no conflict of interest, either existing or potential.

Author Contributions

All the authors contributed equally to this work.

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