

**THE EXAMINATION OF IRON AND CALCIUM CHLORIDE SOLUTIONS BY PHOTON
CORRELATION SPECTROSCOPY**

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**ИЗУЧЕНИЕ РАСТВОРОВ ХЛОРИДОВ ЖЕЛЕЗА И КАЛЬЦИЯ МЕТОДОМ ФОТОННОЙ
КОРРЕЛЯЦИОННОЙ СПЕКТРОСКОПИИ**

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***Аннотация.** В работе представлены результаты изучения растворимости солей хлоридов железа и кальция в растворителях различной природы (вода, этиловый спирт, бензол) при комнатной температуре и нагревании. Для исследования размеров образующихся частиц был использован метод фотонной корреляционной спектроскопии. Выявлено различное поведение солей хлоридов железа и кальция в трех растворителях. Отличия полученных зависимостей объясняются различной природой образования исследуемых солей, их структурой, а, следовательно, и свойствами.*

***Abstract.** Being aware of substances solubility is significant for solving numerous practical issues. Solubility depends on the solute and solvent nature, temperature, and other factors. The study on substance solubility within various solvents makes it possible to choose a state in which the substance under analysis or the final synthesized product will be predominantly accumulated in the solid phase; this gives way to the process of its separation from the reaction mixture by means of simple decantation. The data collection provides the proper choice of solvent recrystallization. In addition to the preliminary study on an analyte's solubility, it is necessary to perform the quantitative evaluation of dissolution rate, which represents the kinetic parameters of the chemical reaction [1]. The kinetic data give us a chance to clarify the reaction mechanism, to identify the correlative functions between the molecules reactivity and their structure; besides, they provide the future prospect in the realm of chemical synthesis reactors planning, as well as modernizing old manufacturing processes and developing the new ones [2].*

Unfortunately, nowadays there exist no theoretical data on the solubility concerning the enormous amount of different nature salts in various solvents – including such metal chlorides as iron and calcium, which are widely spread in diverse spheres of human activity, e.g., in food industry and metallurgy, medicine, construction, and other areas. Calcium chloride is used for regulating water hardness and calcium transport deficiencies within the body. This salt also appears to be applicable as an efficient hydrophobic substance. Iron chloride can be used as a good catalyst for organic or inorganic substances production, and what is more, it is commonly used as a well-precipitated component [3].

In relation to this problem, the following aim was set: to consider the theoretical grounds for the process of iron and calcium chloride salts dissolution within the solvents of different nature and to carry out experimental analysis of the mentioned processes at room and heated temperature via photon correlation spectroscopy.

The research method. The employed methods include the method of photon correlation spectroscopy (FCS), which displays a number of advantages: it proves to be informative for the study of sedimentation processes dynamics occurring in aqueous systems upon the application of heat. High accuracy and rapidity contribute to the strong points of the method, which is also significant for not perturbing in the object under analysis [4].

The research materials. Based on the initial samples, the analyzed solutions were prepared; we used iron and calcium chlorides plus solvents, such as water, ethanol, and benzol. The particle size of the dispersion phase was obtained for the prepared solutions allowing us to construct the functional connection between solubility and temperature. For this purpose, the analyzed sample was put in a 4 ml circular quartz cell. The experiment was conducted on UNICOR-SP photon correlation spectrometer: monochromatic radiation He-Ne laser (output power ~ 50 MW). Passing through the cell with the analyzed sample, the dispersed phase particles were dissipated and fixed by a photodetector; the signal was fed to the multichannel digital correlator PhotoCorSp embedded into one of the motherboard connectors in a computer. The angle between the laser beam passing through the cell and the photo-integrated block makes 90°. By means of «PhotoCor-Sp» software, it became possible to monitor the process of measurement, as well as to calculate the results. The observation target was to accumulate the correlation function. It has an exponential form, which is being used as the scattered light spectrum.

Fig.1 shows the particle size alteration in iron and calcium chloride aqueous solutions of different concentrations upon heating. The functional correlations describe the solubility enhancement coordinated with increasing temperature. As for 47% and 2% solutions, they were originally characterized by larger particles formation followed by size reduction upon the solution heating. This means that the salt solubility is being increased.

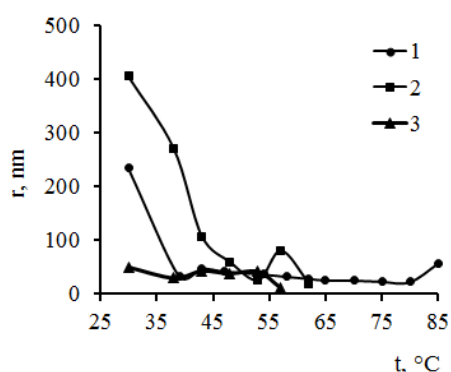


Fig. 1. The particle size alteration for iron and calcium aqueous solutions upon heating: 1–2 % (mas.) $FeCl_3$; 2–47 % (mas.) $FeCl_3$; 3–2 % (mas.) $CaCl_2$

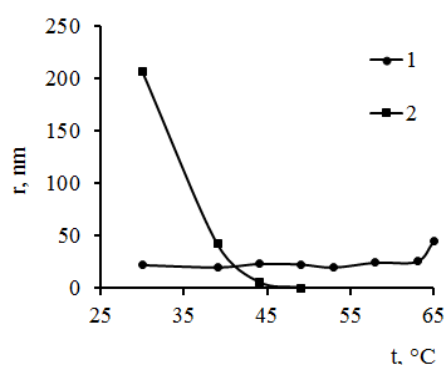


Fig. 2. The particle size alteration for 2,5 % (mas.) iron and calcium alcohol solutions upon heating

Having compared the particle size alterations for 2% iron and calcium chlorides aqueous solutions upon heating, we noted solubility increase in both of the cases. Besides, we observed the formation of calcium chloride molecular solution at the temperature of 50°C.

Fig. 2 shows the particle size alteration for iron and calcium chlorides alcohol solutions upon heating. In case of calcium chloride, the particle size is being sharply decreased upon the application of heat. Likewise, there has been observed the tendency towards the formation of the molecular solution at the same temperature of 50°C. In providing alcohol iron chloride, there appears to be practically no impact on the particle size alteration upon heating within a range from 30° to 63 °C; also, there is a slight increase in particle size at the temperature of 65°C .

Concerning benzol-based dissolution (Fig. 3), there has been 'expressive' dependence in the case of 0.5% iron chloride solution: it manifests the dynamic nature of both increase and decrease solubility. The largest particles are formed at 55°C. As for 0,5% and the 2,2% iron chloride solutions, a gradual decrease in solubility takes place. However, both solutions are characterized by the peak confirming the largest particles formation at 55 °C. Analyzing the behavior of two benzol-based solutions of iron and calcium chlorides, the opposite tendencies have been observed: the solubility of calcium chloride is being increased, and the solubility of iron chloride is being reduced upon heating.

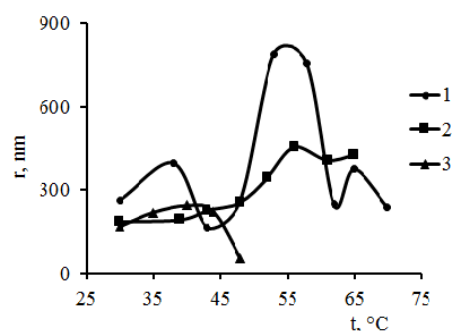


Fig. 3. The particle size alteration for benzol-based iron and calcium solutions upon heating:
1–0,5 % (mas.) $FeCl_3$; 2–2,2 % (mas.) $FeCl_3$; 3–2,2 % (mas.) $CaCl_2$

Conclusion. The behavior of iron and calcium chlorides is different for all the three solvents (water, alcohol, benzol). The solubility of calcium chloride is increased in all three solvents upon heating. In the case of iron chloride, there is an increase of solubility for water and a decrease for alcohol- and benzol-based solutions. The differences between the obtained correlations dependencies are explained by the diverse nature of formation, the structure, and the properties, respectively. Calcium chloride is a strong hygroscopic sorbent providing homolytic and heterolytic properties. Iron chloride is also hygroscopic characterized by paramagnetic properties.

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