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Study of the Synthesis of Materials with Specific Properties Water and Water Systems

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Keywords: aqueous systems, crystallization, ice, melting, water.

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Abstract:

The features of water and aqueous systems used in the directed synthesis of materials with specified properties are shown. Water simultaneously plays the role of the initial medium, "building material" and "tool" in this synthesis. The conditions of directed crystallization and melting of ice are discussed as the basis for controlling the physicochemical transformations of components in aqueous systems. New methods of studying and using aqueous systems are considered - the "floating reactor" method and its based on the use of the Pettier and Seebeck effects.

Keywords: aqueous systems, crystallization, ice, melting, water.

دراسة تركيب المواد ذات الخواص المحددة للمياه والأنظمة المائية

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الخلاصة:

يستعرض هذا البحث خصائص الماء والأنظمة المائية المستخدمة في التركيب الموجه للمواد ذات الخصائص المحددة. حيث يلعب الماء دور الوسيط الأولي "مادة البناء" و"الأداة" في هذا التركيب. تتم مناقشة ظروف التبلور الموجه وذوبان

الجليد كأساس للتحكم في التحولات الفيزيائية والكيميائية للمكونات في الأنظمة المائية. يقدم البحث طرقا جديدة لدراسة الأنظمة المائية واستخدامها – طريقة «المفاعل العائم» وطريقة تعتمد على استخدام تأثيرات بيلتييه وزيبيك

الكلمات المفتاحية: الماء، الأنظمة المائية، الجليد، التبلور، الذوبان.

1. Introduction:

Water scarcity is a global problem [1, 2] and industrial water is a major contributor to water consumption [3,4]. Recycling industrial water is an important means of saving water, but the repeated use of circulating cooling water causes the scaling ions in the water to form scale on the heat exchanger and pipe walls in the form of precipitation, resulting in a decrease in the heat exchange efficiency of the equipment [5, 6].Water treatment agents are currently added to alleviate the scaling of circulating water treatment systems , as they offer certain advantages including simple operation, excellent effectiveness, and low cost [7]. The most commonly used agents include inorganic salts, organic phosphine compounds, natural macromolecules, and synthetic macromolecules. Among these scale inhibitors, inorganic salts and organic phosphine compounds have limited applications because they can easily promote the growth of bacteria and algae in the system, which can cause water eutrophication and environmental damage . Natural polymers are cheap and easy to obtain, but they have disadvantages; for example, they are needed in large amounts and are unstable at high temperatures in the process of application [8]. Thus, phosphorus-free synthetic macromolecule scale inhibitors have received extensive attention . These copolymers mostly contain carboxyl, hydroxyl, sulfonic acid, amino, and other anti-scaling functional groups, and a combination of different groups that can effectively prevent the weak hydrophilic groups from forming insoluble calcium gels. This process can also help to dissolve calcium gels, to effectively prevent scaling . IA is an unsaturated dicarboxylic acid, which contains the carbon double bond functional groups required for free-radical polymerization, as well as carboxylic acid groups on both sides of the double bond. The latter endows IA copolymers with excellent negative dispersion properties and the ability to complex with other ions [9]. Moreover, IA molecules do not contain phosphorus or nitrogen. As a result, they do not cause eutrophication of water bodies through enrichment, and are environmentally friendly. While polymers can be obtained from IA monomers through homopolymerization, and this particular polymer offers certain scale inhibition effects for calcium carbonate, its dispersion performance still needs to be improved . Sulfonic acid groups are strong acid groups with strong hydrophilicity, and they can enhance the solubility of a copolymer, effectively preventing the weak acid group from generating insoluble calcium sol gels .Electrostatic water treatment technology has additional advantageous characteristics

including low energy consumption, near-zero pollution, and high efficiency, with excellent application prospects that urgently need to be researched and developed (. In this study, SMAS, which contains sulfonic acid groups, was copolymerized with IA to obtain a scale inhibitor (IA/SMAS) with good scale inhibition and dispersion properties. The resulting product was free of phosphorus and nitrogen and environmentally friendly. The IA/SMAS polymers were characterized by various means. Afterward, the synergistic scale inhibition performance of the physical water treatment technology (electrostatic field water treatment technology) and the agent was also studied. Finally, this study also evaluated the IA/SMAS copolymer alone and its synergistic scale inhibition mechanism with electrostatic fields(10).

Aim of study

Consider new methods for studying aquatic electronics - the "floating reactor" method and a method based on the use of the Peltier and Zeppeck effects.

2. Material and methods:

An aqueous solution of the substance under study of a given volume and concentration is cooled to a temperature that guarantees the transformation of all the water in the solution into ice. The resulting ingot is then maintained at an ambient temperature of 300K and, as the melt forms, it is taken in certain portions (fractions), recording the volume of the fraction and the time of its formation. After room temperature has been established in all fractions of the melt, the concentration of the substance dissolved in water is determined in each of them. Various analytical methods are used for this - refractometry, potentiometry, and others. Based on the data obtained, graphs are plotted of the dependence of the volume of the formed melt on the time the ingot is kept at room temperature, and the concentration of the dissolved substance in the fraction on the volume of the formed melt for initial aqueous solutions of different concentrations. The nature of the curves on these graphs allows one to determine the type of phase diagram of the corresponding binary aqueous system and the conditions for separating and isolating the components from the solution. The obtained data can also be used to establish the conditions for growing crystals of these components with specified properties [11].

Moreover, the fractional melting technique can be used to grow crystals of a substance dissolved in water directly in the process of melting an ice ingot. For this, the initial solution, for example, a salt whose phase diagram of the binary system (“salt-water”) is eutectic, must

correspond to a certain composition from the hypereutectic region of this system. That is, its concentration in the initial solution must exceed the eutectic. Such a solution is cooled to a temperature below the eutectic, and then the formed ingot is subjected to fractional melting using the technique described above. In the process of melt fractions selection, the remaining unmelted part of the ingot is enriched with salt, since the melt fractions being selected at the beginning of melting have a salt concentration close to its eutectic value, i.e. lower than the initial, hypereutectic concentration. Under a certain regime of melt fractions formation and selection, salt crystals begin to grow inside the ingot that has not yet melted. In this case, the ingot itself simultaneously acts as a "container", "thermostat" and the starting material for crystal growth. At the same time, the internal structure of the ice ingot determines the conditions for the growth of salt crystals, their shape, composition and structure. Moreover, the melting process itself is inextricably linked with the crystallization process inside it. In order to consciously.

To control the above-described crystallization and melting processes and use them to synthesize materials with specified properties, it is necessary, first of all, to control and purposefully set the temperature-time parameters of these processes: the rate of cooling, heating, as well as the corresponding changes in the nature of the distribution of the concentration of components in the system. For this purpose, we have developed a set of methods based on the use of the Peltier and Seebeck effects, as well as the "floating reactor" method - a method of physicochemical transformations under hydrostatic conditions.

Traditional cooling methods are most often based on the use of freon technology. In this case, a certain volume is cooled, into which the initial solution is placed. Heat is removed from it from the surfaces that limit this solution. Ice crystallization begins from these surfaces, and the substances dissolved in water are displaced by it into the center of the solution volume. This nature of ice crystallization in the solution reduces the efficiency of separating the components and complicates the control of the processes occurring in the system. We have developed a method for controlled cooling of solutions using cooling pipes introduced into a given area of the solution volume. The heat flow through the cooling pipe is set and controlled by semiconductor thermoelectric modules operating using the Pelte and See beck effects [12]

The fundamental difference of this method is that ice crystallization in the solution begins not from its surface, but from a certain given, volume, which is determined by the shape dimensions, and material of the cold line. The intensity of crystallization and the structure of

the ice depend on the magnitude of the working current flowing through the thermoelectric modules. By changing the direction of the current, it is possible to change the direction of the heat flow between the modules and the solution, thereby combining the directed processes of crystallization and melting in a single technological cycle. Moreover, since the thermoelectric modules operate on the Peltier and Seebeck effects, the cold line (heat pipe) connecting them to the solution (reaction volume) simultaneously performs the functions of a working element transferring heat and a temperature sensor. During phase transformations that are controlled by heat flows through the cold line, for example, during crystallization.

When ice (or other components of the solution) is liquefied and melted, heat is released or absorbed, and the corresponding temperature changes, recorded by the cold pipe as a change in the heat flow through it, affect the electrical parameters of the semiconductor thermoelectric modules and their power source. In our particular device, this influence is clearly recorded by a voltage jump of the corresponding sign on the power supply unit, which maintains a specified value of the operating current through the module. Moreover, the sensitivity of the device to phase transformations is so high that it allows them to be monitored in a solution volume of less than 1 ml, for example, by applying just one drop of the solution to the surface of the cold pipe. This allows us to study phase transformations very effectively by combining precise recording of the transformation temperature with visual control of the process.

The use of thermoelectric devices (modules) for the implementation and control of various physicochemical transformations allows us to significantly increase the efficiency of the processes of separation and isolation of components in solutions, targeted synthesis of materials, for example, growing crystals with specified properties [13].

For example, we significantly modified the above-described method of fractional melting of ice. Moreover, its modification was carried out in two directions. The first direction is the development of the "floating reactor" method or the method of physicochemical transformations under hydrostatic conditions. Often, to control the processes occurring in solutions, it is necessary to operationally control the nature of the distribution of components in the volume of the system. Moreover, sometimes it is more important to control the dynamics (kinetics) of change, for example, the concentration of components in time and space, than its absolute values.

The method of such control was developed by us primarily to study the nature of the distribution of components in the process of fractional melting of ice ingots formed in aqueous solutions with different initial concentrations of components. Its essence is that the vessel with the ice ingot, after its removal from the refrigeration chamber, is lowered into another vessel with liquid, distilled water in our case, so that the vessel with ice - the "floating reactor" - floats freely in this liquid, and the design of both vessels allows, purposefully change the mass of substances in the vessels. In the case of fractional melting of ice, the sampling of each melt fraction from the inner vessel (see above) is accompanied by fixing the liquid level in the outer vessel.

The ratio of the volumes of the fractions sampled and the corresponding changes in the liquid level in the outer vessel makes it possible to determine the density of each fraction and, consequently, the concentration of the dissolved substance in it, and to plot the dependence of the concentration of components on time during the melting process. Naturally, these studies require compliance with a number of conditions. It is necessary to control the temperature, pressure, and also pre-calibrate the system using solutions with a known concentration and density.

The scope of application of this method can be significantly expanded, for example, to study processes associated with the evaporation of components, adsorption, ion exchange, etc. It can also be effectively used in combination with the previously described method of physicochemical transformations controlled by thermoelectric modules. If a coolant is introduced into the volume of the solution in a "floating reactor", it is possible to directly control the concentration of components in the phases undergoing the corresponding transformations during the crystallization or melting process, and a change in the position of the reactor relative to the coolant, determined by a change in the liquid level in the external vessel, for directional crystallization or melting of the solution components.

3. Results:

Figure 1 shows a typical picture of the distribution of the concentration of a substance dissolved in water (in this case, NaCl) by melt fractions during fractional melting of ice ingots formed from NaCl solutions with different initial concentrations

For initial compositions from the pre-eutectic region (more diluted) of the "H2O–NaCl" system, the first fractions of the melt are enriched in salt compared to the initial concentration. As the initial concentration increases (approaches the eutectic), the degree of enrichment decreases, and with an initial concentration equal to the eutectic, the distribution among the melt fractions becomes almost uniform. Exceeding the eutectic concentration in the initial solution leads to a fundamental change in the Chirac.

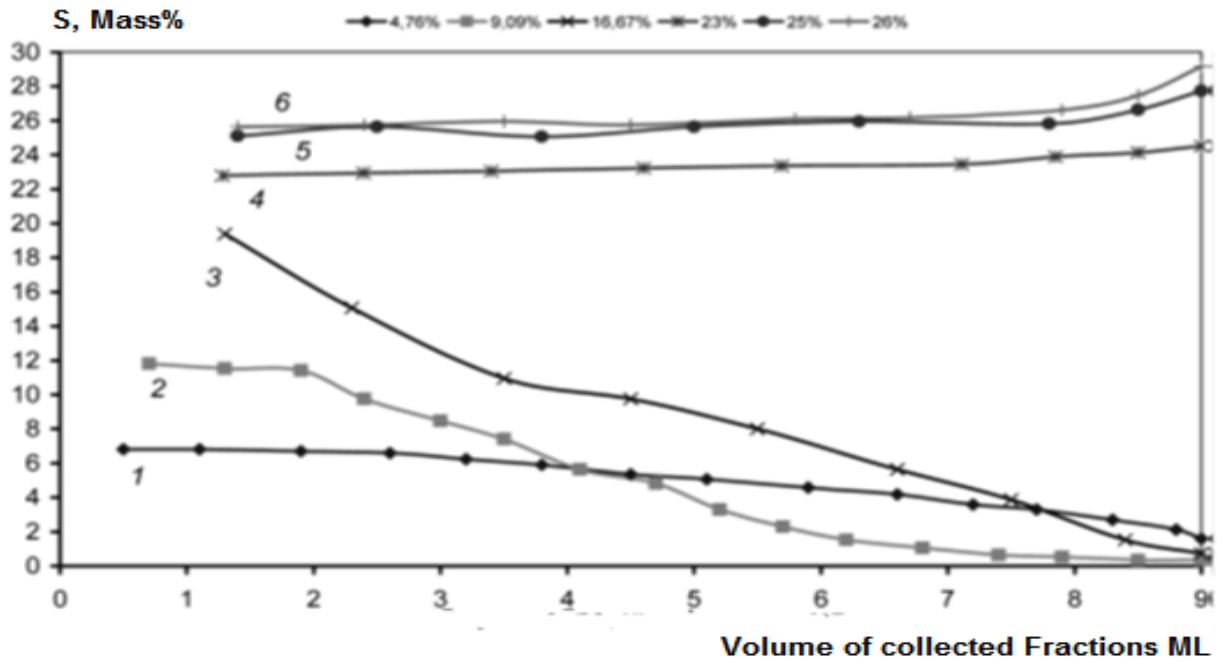

Fig. 1. Distribution of NaCl concentration by melt fractions for initial solutions of different concentrations

distribution of salt among melt fractions. The latter fractions are enriched with salt In [11, 13] it is shown that such a pattern of distribution of the concentration of a substance dissolved in water among melt fractions during fractional melting is characteristic of all substances we have studied, whose phase diagrams are eutectic. We have established that these include not only water-salt systems, but also a number of systems water–amino acid", "water–glucose", etc.

The study of aqueous solutions in the H_2O–NaCl system by the method of fractional melting in a floating reactor did not reveal any significant differences in the distribution of salt concentration among melt fractions compared to its classical version (i.e., in a stationary

vessel). At the same time, the determination of the density of melt fractions during fractional melting in a floating reactor by the method described above (see Fig. 2) showed a rather complex pattern of changes in the density of the solution in successive fractions. It is interesting that the “direct” determination of the density of fractions by weighing them on an analytical balance repeated this pattern with the only difference that the absolute values of density in this case turned out to be somewhat different from those for the floating reactor method.

Fig. 3 shows similar results for the "H_2O-fructose" system

The following can be noted regarding the complex nature of the melt density distribution by fractions (Fig. 2, 3):

1-The periodic change in density is not exclusively the result of experimental errors and inaccuracies, although they are naturally not excluded. These changes reflect, first of all, the objective picture.

.2-The objectivity of the results is confirmed by a good correlation between the dependencies obtained in two different ways - the "floating reactor" method and "direct" weighing on a scale

3-The periodicity of the change in the density of successive melt fractions is determined by te periodic pattern of distribution of components in the ice ingot formed during its crystallization.

4- The density of the melt depends on the temperature. The results obtained in the floating reactor method reflect the maximum correspondence to the melting ingot temperature. The process of weighing the selected fractions introduces inevitable changes into this correspondence. Therefore, the results, repeating qualitatively, differ somewhat in the absolute values obtained

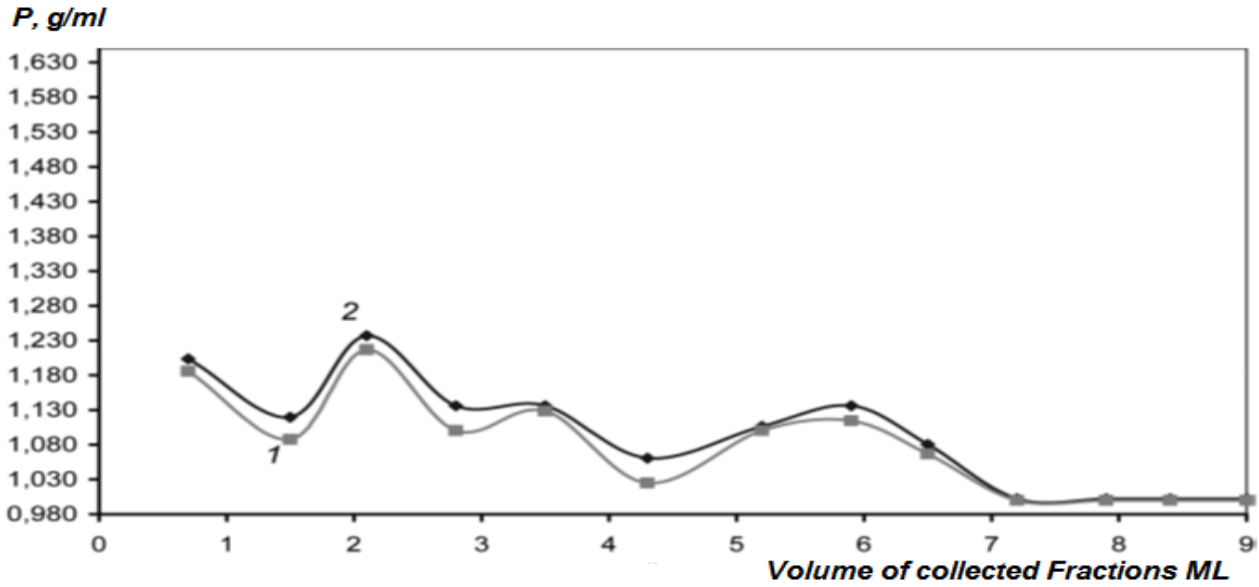

Fig. 2. Density of different melt fractions for the initial solution of 10% NaCl: 1- pycnometric determination of density; 2 - determination of density by the “floating reactor” method.

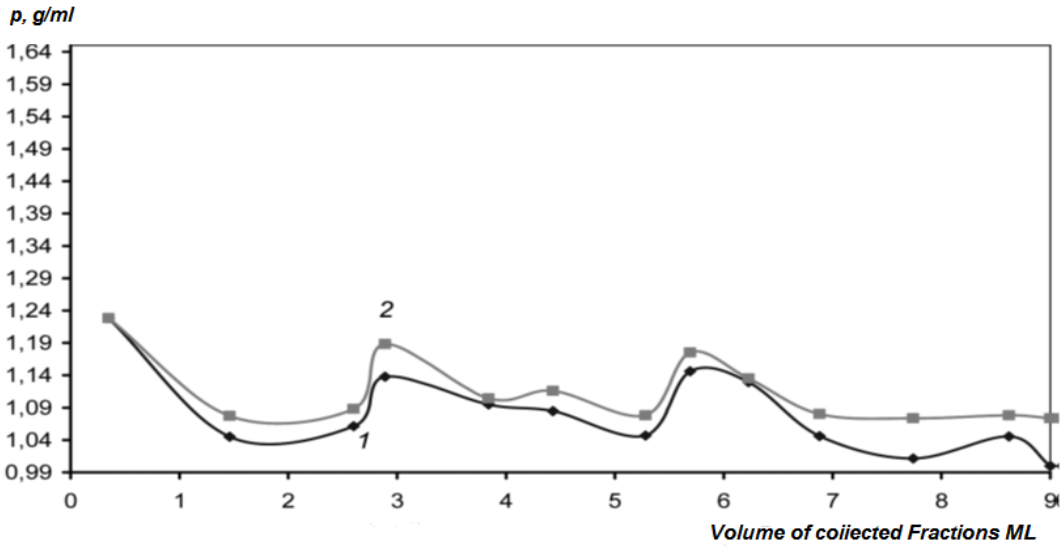

Fig. 3. Density of different melt fractions for the initial solution of 20% fructose: 1 — psychometric determination of density; 2 — determination of density by the “floating reactor” method.

quantities. It can be said that the melt, separated from the ingot, loses the “connection” with it that exists in the melting process itself.

4. Discussion

Directed synthesis, i.e. synthesis of materials with specified properties, requires control of the processes of component distribution in a given volume - distribution of their mass, density, concentration. In aqueous media, the main "instrument" of control is the state of water which is primarily determined by temperature. A change in temperature changes the structure of water, the nature of the interaction of its molecules with each other and with particles of other substances. Very indicative from this point of view are transformations in systems described by eutectic phase diagrams, for example, "salt-water". In [14], it is shown quite convincingly, on the basis of many experimental results and theoretical reasoning, that the well-known "NaCl-H_2O" solutions turn into colloidal ones when the temperature drops to 0 C° and below — to the eutectic point. We believe that this phenomenon is not unique, it characterizes many eutectic systems, which we are convinced of, for example, by the results of our own experiments on the study of the composition, structure and properties of glycine and triglycine sulfate crystals - TGS, grown from aqueous solutions at different temperatures [15].

Comparing the complex of physicochemical properties of these crystals grown in the temperature range near room temperature and near 0 °C (and below), we came to the conclusion that significant and regular changes in these properties with a decrease in growth temperature are due, first of all, to a change in the state of water in those aqueous solutions in which this growth was carried out, as well as the state of water in the crystals themselves during and after their growth. According to modern concepts, even pure water is a micro heterogeneous system built from aggregates of different composition and structure. The sizes of these aggregates fluctuate from molecular to colloidal-dispersed . As the temperature decreases, the proportion and role of colloidal-dispersed states in water increases. The same tendency is characteristic of aqueous solutions of various substances. At room and higher temperatures, their structure fully meets the conditions for the existence of true solutions, that is, solutions in which the degree of dispersion is of an ionic-molecular nature, and individual ions or molecules of dissolved substances replace individual water molecules in the three-dimensional network of its hydrogen bonds. The solution is maximally homogeneous. When the temperature decreases, the water itself is the first to heterogenize, losing its hydrophilic properties - the tendency to form a single branched network of hydrogen bonds. Its hydrophobization occurs - the tendency for self-closure of hydrogen bonds, the formation of disunited aggregates, which, even being closely arranged with each other, do not create hydrogen bonds with each other. In such a structure, the expulsion of particles of dissolved substances from the nodes of the three-dimensional network into its voids prevails, which contributes to their unification with each other into their own

aggregates. This is the mechanism of formation of colloidal-dispersed phases, isolated from each other - water and substances previously dissolved in it. Such a process develops in the temperature range from room temperature to 0 °C and below, to the eutectic point. If we compare the crystallization conditions of the water itself and the substance dissolved in it, that is, in the pre- and post-eutectic regions of the system, we can distinguish two fundamentally different mechanisms of nucleation and growth of crystals , which are determined precisely by the difference in the structure of water and aqueous solutions in these regions. In the crystallization processes of interest to us, this difference must be associated first of all with the concepts of "saturation" and "super saturation" of the solution, which determine the conditions of nucleation, growth of crystals, their composition, structure, properties.

Graphically, in the language of phase diagrams these states are associated with the location of the "liquids" lines. On the eutectic phase diagram, they connect the eutectic point with the points corresponding to the melting temperatures of the pure components that make up the system. The liquids line itself determines the state of saturation of the solution with one of the components at its corresponding concentrations and temperatures. The state of the second component in this case — namely for eutectic type systems — changes regularly along these lines, in the regions adjacent to them, from far from saturation, at higher temperatures, to saturated near the eutectic. Here, the regions of saturation and super saturation of the system for both of its components overlap. If far from the eutectic, the states of saturation and super saturation of the system for each of its components are not connected with each other and are independent of each other, the "liquidus" lines go far from each other, then in the eutectic region they come closer together, intersecting (and ending) at the eutectic point, and the saturation regions for both components overlap, superimpose on each other, "interfere". In these regions, where the solution is simultaneously saturated with both components, the conditions for the nucleation and growth of crystals, their composition, structure, and properties change sharply and fundamentally. Let us consider, first of all, the behavior of the components in limited, local regions of the solution, both in the eutectic region of temperatures and compositions, and far from it. As was indicated above, at higher temperatures (far from the eutectic) the solution is "true", i.e. it consists of individual ions and molecules, and the formation of aggregates from them is hindered by thermal motion. When the system is saturated and supersaturated with one of the components, which is necessary for the nucleation and growth of its crystals, the solution remains far from being saturated with the second component, in relation to which it is still built as "true". In this case, the most important mechanism for the construction of the crystal is the

well-known mechanism of layer-by-layer growth , in which the building material is attached to the crystal "piece by piece", mainly in the form of individual molecules or ions, the role of aggregates pre-formed in the solution is, as a rule, insignificant. As a result, conditions are created for the growth of large, perfect, flat-faced single crystals. The capture of solution components by the growing crystal is reduced to a minimum (which, by the way, corresponds to the minimum mutual solubility of components in the solid state, characteristic of such systems).

In the region of states adjacent to the eutectic, i.e. when the temperature decreases, the solution begins to become saturated with both components at once the "liquids" (lines converge). At the same time, as already indicated above, the solution begins to disintegrate (micro heterogenize) into separate micro regions, built mainly from water molecules or molecules (ions) of the dissolved with each other. Each of them is "supersaturated" with one of the components. This component, striving to form its own structure, "phase" first in the form of a prototype" of the future crystal, pushes out the molecules of the second component from this region, since in the solid state they are practically insoluble in each other. In this case, the regions adjacent to the given one are enriched and supersaturated with this very second component, pushed out of it. Such a process, under appropriate conditions, begins to develop in an avalanche-like manner, like a chain reaction, encompassing new regions of the system. This is where the mutual connection of states (regions) supersaturated with both components at once manifests itself. The decomposition of the “true”, homogeneous solution occurs, which can be called eutectic decomposition. Naturally, the conditions for the nucleation and preferential growth of crystals of one of the components in this region will be fundamentally different than at higher temperatures, discussed above. With some deviation of the solution composition from the eutectic, for example, into the hypereutectic region, it is possible to grow crystals of a substance dissolved in water, but they will not be built from individual molecules or ions, but from already formed aggregates (during eutectic decomposition of the solution) according to the so-called "block mechanism [16].

Their composition, structure and properties will differ significantly from those formed during layer-by-layer growth. As an example, we can cite the difference in the ferroelectric properties of TGS crystals grown by us purposefully according to both described mechanisms [15]. It should also be noted that the mechanism of "eutectic" decomposition of aqueous solutions is, in our opinion, essentially very close to spinodal decomposition [17], which is characteristic

mainly of solid solutions, but is also observed in liquid phases [18]. We pointed out the possibility of decomposition of eutectic solutions by the spinodal type earlier in [19]. In accordance with our ideas about the spinodal decomposition of eutectic solutions, it is possible to theoretically estimate the concentration and temperature at the eutectic point, that is, to predict it in the case where it is unknown in a given system from other sources, and, accordingly, to model in advance the conditions for crystal growth and synthesis of materials with specified properties. For such an assessment, it is necessary to compare the concentrations, densities of solutions and the densities of their individual components at different temperatures. Based on the results obtained, it is possible to estimate the volumes of the solution regions in which can simultaneously, that is, according to the described mechanism of eutectic, spinal, decomposition form supersaturated states of the system both components for The experimental basis for such assessments for us is primarily the results of directed crystallization and subsequent fractional melting of ice in the studied aqueous systems using the methods described above that we have developed.

We consider it necessary to make some comments regarding the existing ideas about the states of "saturation" and "super saturation" of the system. As a rule, they are associated with the deviation of the temperature and concentration of components in the system from certain equilibrium values that are connected to each other, for example, by the "liquids".

or "solidus" lines on the phase diagrams of these systems. Understanding these concepts as purely thermodynamic, associated with the excess of free energy of the system over its equilibrium, i.e. minimum values, usually overlook the chemical nature of these concepts, their crystal chemical aspect, interpreted in a broad sense, as an aspect of structure. The approach of the state of the system to "saturated" and "supersaturated" fundamentally changes the nature of the interaction of the particles that make it up. The most important factor in this change is the tendency of particles to "aggregate", "condense". Such a change depends both on the internal structure of the system and on the parameters of the external impact on it. In this case, the key factors in this impact are the gradients (i.e. driving forces) of those parameters that affect the system, and the nature of their distribution and change in space and time.

5. Conclusions

Two new methods for studying phase transformations in water and aqueous systems have been developed the “floating reactor” method and a method based on the use of the Peltier and Seebeck effects. They allow one to control the distribution of components during directed crystallization and melting of ice and use these processes to synthesize materials with specified properties.

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