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### THE EQUILIBRIUM STATE OF COMPOUNDS OF VANADIUM, CAPABLE OF SPONTANEOUS STRUCTURE FORMATION

**Abstract.** This article presents the results of theoretical and thermodynamic analysis of equilibrium States of vanadium compounds in order to justify the possibility of xerogel synthesis in the presence of ammonia. It is shown that vanadium, which is one of the 3d-elements, has the ability to form complexes. One of the stable complexes is  $[HV_{10}O_{28}]^{-5}$ , whose properties allow to obtain nanomaterials based on vanadium.

**Key words:** complex formation, nanomaterials, template, xerogel, Sol-gel method, thermodynamic analysis, monomer forms, polymer forms, vanadium complexes.

#### Introduction

Currently, nanotechnology is attracting a lot of attention, allowing to create a number of fundamentally new production processes, materials and devices based on them. Within the framework of the known synthesis methods, nanoobjects of different morphologies can be obtained, having a variety of shapes, sizes and functional properties.

Among the known methods of obtaining nanomaterials, one can note the Sol-gel technology. Traditionally, the Sol-gel method is understood as the state of the stages, including the preparation of the precursor solution, its subsequent transfer first to Sol, and then to the gel due to the processes of hydrolysis and condensation, subsequent aging, drying and heat treatment of the product [1].

The Sol-gel method is a simplified flow chart of the synthesis. This method allows to achieve a high degree of purity of products at all stages of synthesis with a minimum of energy consumption to achieve it. It becomes possible to obtain by this method products that are characterized by a monophase crystal structure with a high degree of perfection, strictly stoichiometric composition.

The solvent removal from the gel (drying) plays an extremely important role in Sol-gel synthesis. Depending on the method of their implementation, various synthesis products (xerogels, ambigels, cryogels, aerogels) can be obtained. The General features of these products are the preservation of nanosize of structural elements at different stages of synthesis (due to changes in reaction time, temperature, concentration and chemical composition of reagents), and sufficiently high specific surface area values [2-4].

Nanomaterials synthesized on the basis of vanadium compounds are of great scientific and practical interest. Vanadium, which is a polyvalent metal, shows a high tendency to complexation. Its compounds have a wide range of properties due to spontaneous structure formation, which is the starting point for the synthesis of nanomaterials based on them in the presence of a template (the educator of complexes), which contributes to the creation of an ordered structure complex.

It should be noted that in the compounds of vanadium it is extremely difficult to distinguish a certain valence structure, and as for the formation of macroscopic gels obtained on the basis of vanadium oxide (V), the use of the term valence may generally lose its meaning.

Vanadium oxide is a unique substance forming lyotropic liquid-crystal systems. When such colloidal solution dries, xerogels with a partially ordered layered structure are formed [5].

The xerogel based on vanadium pentoxide ( $V_2O_5 \cdot nH_2O$ ) is characterized by a layered crystal structure. Using a template (i.e. an ion or a molecule embedded in a crystal structure) of different radius, it is possible to regulate the interlayer space of the structure, as a result of which the xerogel will "stretch" or "shrink" on the principle of an accordion [6]. On the other hand, due to this "flexibility" of the structure, the xerogel can intercalate (i.e. "suck") a variety of ions and molecules-guests.

The authors of [7, 8] note that in the structural spaces of the nanomaterial there are hydroxonium cations, since there is an alternative entry of the formula of xerogel  $H_xV_2O_5 \cdot nH_2O$ , which, first, shows the presence of  $V^{4+}$  and  $V^{5+}$  cations in the composition, and secondly, that protons can easily be ion-exchanged for other cations of different radius. Despite the fact that the authors used crystal  $V_2O_5$  (i.e. this is the phase in which the vanadium cations have an oxidation state of only  $5^+$ ), the appearance of the cations  $V^{4+}$  associated with the flow of the Sol-gel process in which the cations  $V^{4+}$  is an essential factor of the stage of gelation.

In addition to xerogels, the vanadium pentoxide with the participation of surface-active substances constitutes a whole range of modifications [4-15]: nanoribbon, nanotube filamentary crystals (whiskers), hybrid organic-inorganic materials. Important derivatives of vanadium (V) and (IV) oxides are nanotubes – hybrid non-organic materials containing molecular template.

These nanomaterials, due to their highly developed structure and ability to integrate into porous matrices, are of great interest from the point of synthesis of precision sorbents and catalysts, improving their special properties. Composites made by modifying porous matrices with anisotropic particles are promising in terms of their use in the separation of elements and wastewater treatment of metallurgical and other industries. In this regard, nanostructures based on 3D-element oxides, in particular vanadium, which are characterized by the ability to complex formation, are of great interest.

Despite the intensive development of nanotechnology, information on nanomaterials based on vanadium oxide is not extensive enough. Therefore, the studies conducted in this direction are timely, relevant and practically significant.

### Research methods

The literature data were studied and the thermodynamic analysis of vanadium-containing systems in the presence of ammonia and without it was carried out to assess the possibility of synthesis of xerogel of vanadium using ammonia as a template.

The thermodynamic analysis of vanadium-containing systems was carried out using the certified program of thermodynamic calculations HSC Chemistry 5.11 of Outokumpu Technology Engineering Research.

### Results and discussion

The exceptional variety of ion forms of vanadium (V) in aqueous solutions makes this area of its chemistry extremely complex. Its knowledge and correct understanding are crucial for the research on the synthesis of nanosized materials.

Vanadium belongs to the transition d-elements. The presence of an unfilled 3d-shell in vanadium atom determines the existence of its unique ability to complex formation. Complex vanadium compounds in the presence of a template under certain conditions due to various intermolecular interactions contribute to the creation of spatial highly developed layered nanoscale structures.

The electronic structure of vanadium atom is  $1s22s22p63s23p63d34s2$ . Its oxidation state in the compounds varies from -1 to +5. However, from the point of view of technological processes, the most important are the compounds of vanadium, in which it has an oxidation state of +5. Vanadium in the oxidation state + 5 has amphoteric properties and is present in aqueous solutions in both anionic and cationic forms. Depending on the pH of the medium and the total concentration of vanadium in the solution, both the diverse ionic forms of vanadium and the degree of their polymerization change [16].

In aqueous solutions in the pH range of 1-14 at a vanadium concentration of  $10^{-4}$  g-atom / dm<sup>3</sup>, there are only monomer forms of vanadium. Vanadium is present in the form of ions of orthovanadiic acid  $\text{VO}_4^{3-}$  (pH 13-14),  $\text{HVO}_4^{2-}$  (pH 8-13,5),  $\text{H}_2\text{VO}_4^-$  and  $\text{H}_3\text{VO}_4$  (pH 7-9,5) and metavanadiic acid ions  $\text{VO}_3^-$  (pH 4.5-8.5) and  $\text{HVO}_3$  (pH 3-5). With increasing acidity of the solution, a  $\text{VO}^{2+}$  cation (pH 4-1) appears, in high – acid media-a  $\text{VO}^{3+}$  cation. In the polymer form of vanadium exists in the form of dimers  $\text{V}_2\text{O}_7^{4-}$ ,  $\text{HV}_2\text{O}_7^{3-}$  (ions of pyrovanadic acid), trimer  $\text{V}_3\text{O}_9^{3-}$  (ions of metavan-diic acid). The highest degree of polymerization, equal to 10, is observed in decavanadic acid ions  $\text{V}_{10}\text{O}_{28}^{6-}$ ,  $\text{HV}_{10}\text{O}_{28}^{5-}$  and  $\text{H}_2\text{V}_{10}\text{O}_{28}^{4-}$  in the pH range 2-6 [16, 17]. The solution becomes orange color. Increasing the pH of the medium to 6 causes the formation of  $\text{VO}^{2+} \cdot \text{VO}^{3+} \cdot \text{pn}_2\text{o}$  compounds, which fall in the form of poorly filtered precipitation of polyvanadates at pH 4.5-5.0.

It should be noted that in the presence of ammonia, it is possible to change the ionic state of vanadium in an aqueous solution, which directly depends on the amount of ammonia introduced into the solution, and hence the structural characteristics of the nanomaterials obtained.

In the presence of ammonia, ions  $(\text{VO}_2)_{10}(\text{OH})_{16}^{6-}$  are not stable (the stability constant is  $\lg K = -7,5 + 0,3$ ) [17, 18, 22]. Molecules of free ammonia in the concentrated solution begin to replace OH-groups in the decavanadium ion with the formation of ammonia complexes of type  $[(\text{VO}_2)_x(\text{OH})_y \cdot n\text{NH}_3]^{y-x}$ , characterized by high solubility. Thus, vanadium complexes with a neutral ammonia molecule may appear [20].

IR-spectroscopic studies of ammonia and vanadium-containing solutions [20, 21] show that coordinated ammonia bands have four main absorption regions at 3100-3300, 1560-1650, 1150-1350 and 600-900 cm<sup>-1</sup>, characterizing valence, deformation and pendulum oscillations of atoms in the ammonia molecule.

In IR absorption spectra of vanadium-containing solutions after ammonia addition, it is possible to distinguish the bands belonging to the coordinated molecules of ammonia [16]. In the region of valent oscillations  $\nu(\text{N-H})$  there is a wide blurred band 3050-3300 cm<sup>-1</sup>, deformation oscillations – absorption bands at 1220 cm<sup>-1</sup> and 1150 cm<sup>-1</sup>. The data obtained show that at pH 4.0-6.0 in the presence of ammonia, vanadium  $[(\text{VO}_2 \cdot n\text{NH}_3)]^+$  ammonia complexes are formed. This conclusion is confirmed by the research of the authors in [19-22], which established the interaction of vanadium ions with the neutral molecule ammonia with the formation of complexes of composition  $[(\text{VO}_2 \cdot z\text{NH}_3)]^+$  where  $z = 1-4$ .

The thermodynamic analysis of V-N-H<sub>2</sub>O systems for the temperature range of 25-80 °C was carried out (figures 1, 2, table 1). Analyzing the Pourbaix diagram for vanadium-containing systems, it can be concluded that in neutral environments, the formation of various oxygen-containing compounds of trivalent and pentavalent ions.

In the system V-N-H<sub>2</sub>O (figures 1, 2) at  $E_h = (-1,15) \div (-0,75)$  b and pH 0÷4,2 vanadium is in cationic form  $\text{V}^{2+}$ . The region of existence of hydroxocomplexes of vanadium occur in the pH 1.5÷13.5 and  $E_h = (+0,3) \div (+1,45)$  V. the introduction into the system of the reducing agent resistant forms become oxides of vanadium, in the presence of oxidizing agents are stable ionic forms.

Stable form in V-N-H<sub>2</sub>O system at pH 1.8÷13.5 and potential values  $(-0,5) \div (+1,25)$  the decavanadium ion  $[\text{HV}_{10}\text{O}_{28}]^{5-}$  is b, which in the presence of ammonia, due to the electron-donor properties of the nitrogen atom, is converted into an ammonia-vanadium complex  $[(\text{VO}_2)_x(\text{OH})_y \cdot n\text{NH}_3]^{y-x}$  [14, 19]. The stability of the ion  $[\text{HV}_{10}\text{O}_{28}]^{5-}$  confirmed by the thermodynamic calculations presented in table 1.

Analysis of diagrams of the state of the V-N-H<sub>2</sub>O system at different temperatures suggests that the formation of vanadium xerogel, which is directly dependent on the presence and stability of the ion  $[\text{HV}_{10}\text{O}_{28}]^{5-}$ , is possible at room temperature.

Under certain conditions, when the dispersed phase increases, coagulation contact between the particles appears, leading to the beginning of the material structuring process. In the process of structuring gradually formed a homogeneous substance-xerogel vanadium, which is then subjected to aging and drying. At this time, the hygroscopic and part of the crystallization water is removed and the template evaporates.

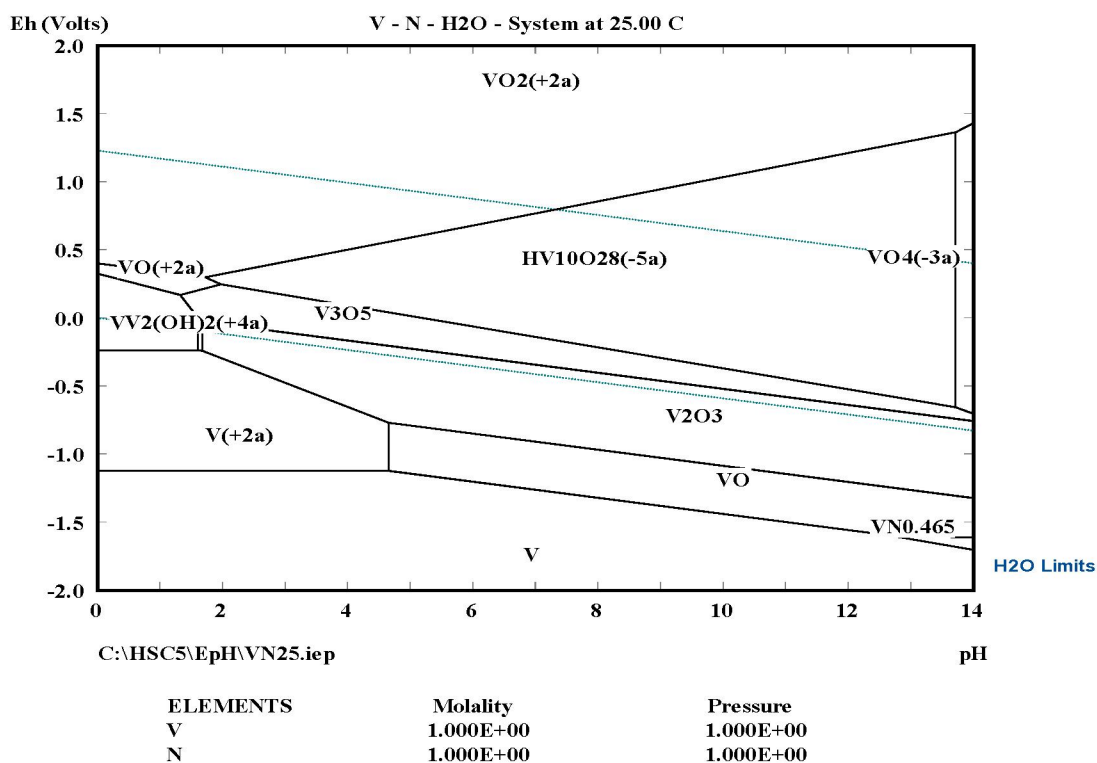
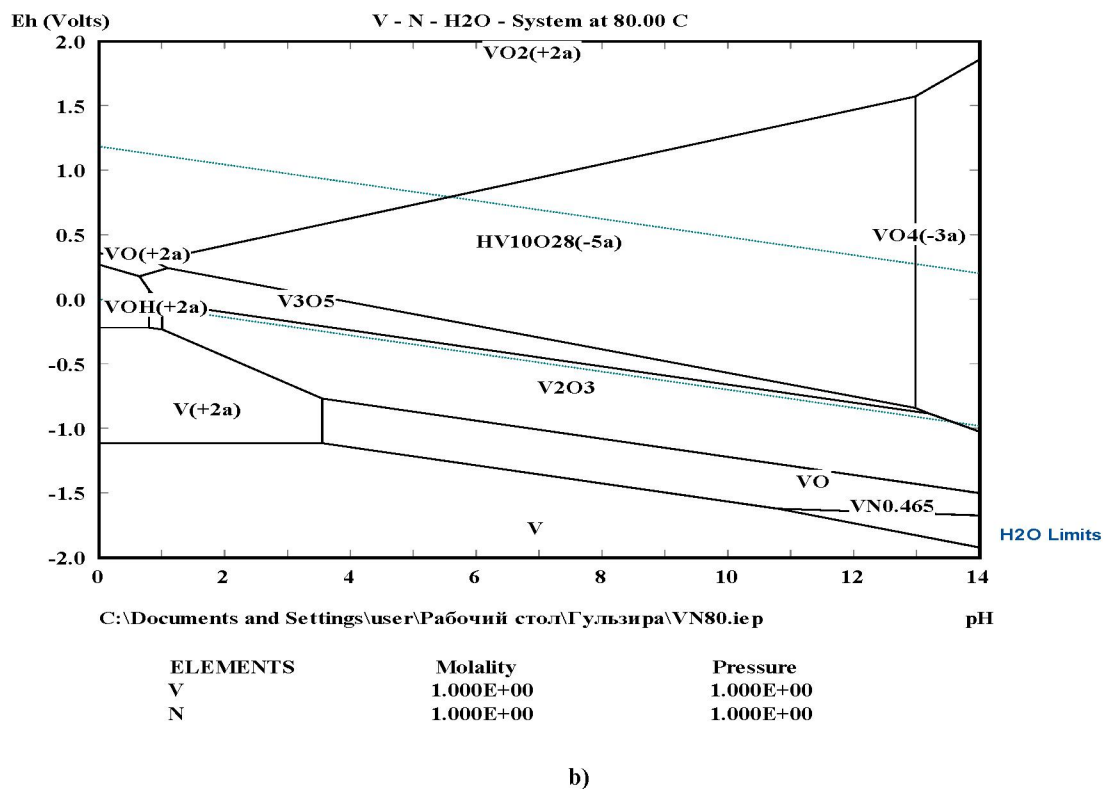
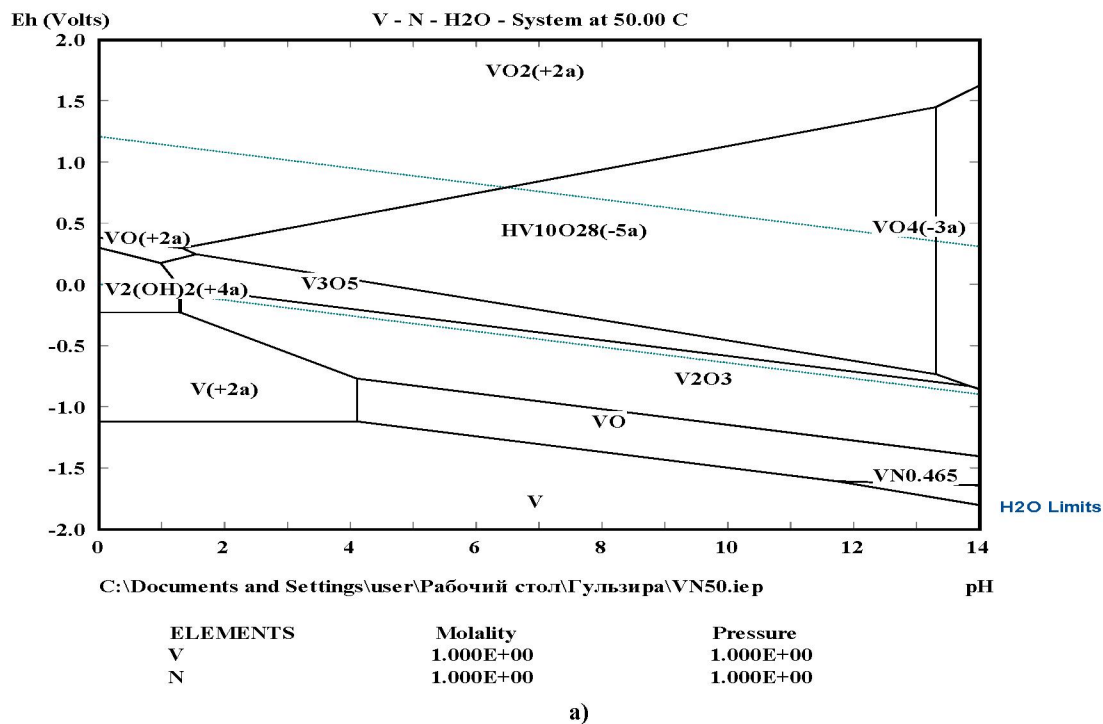


Figure 1 - diagram of Th-pH of M-T-H<sub>2</sub>O system at 25 °C

Table 1- Thermodynamic analysis of the state of equilibrium vanadium forms in the V-N-H<sub>2</sub>O system

Vanadium ions and compounds	$\Delta G^\circ$ , kJ/mol		
	25 °C	50 °C	80 °C
VN <sub>0.465</sub>	-117,772	-116,549	-115,080
VO	-401,286	-398,786	-395,789
VO <sub>2</sub> <sup>+</sup>	-587,293	-585,167	–
V <sub>2</sub> O <sub>3</sub>	-1135,619	-1135,619	-1120,529
V <sub>3</sub> O <sub>5</sub>	-1815,358	-1128,748	-1791,194
HV <sub>10</sub> O <sub>28</sub> <sup>5-</sup>	-8100,748	-8081,379	-8004,987
HVO <sub>4</sub> <sup>2-</sup>	-989,698	-983,543	–
V <sup>2+</sup>	-217,171	-216,388	-215,367
VO <sup>2+</sup>	-446,171	-442,867	-439,149
VO <sub>2</sub> <sup>2+</sup>	-606,198	-602,606	-598,515
VO <sub>4</sub> <sup>3-</sup>	-898,941	-879,333	-855,747
VOH <sup>2+</sup>	-466,959	-465,833	-459,640

The final product of this transformation is a nanomaterial with a porous, mixed-layer, twisted, spatially developed structure, which allows further influence on the properties of catalysts and sorbents.

Figure 2-diagrams of Th-pH of M-T-H<sub>2</sub>O system at 50 °C (a) and 80 °C (b)

### Summary

The results of the thermodynamic analysis of vanadium-containing systems showed that the formation of complex ions and vanadium compounds in the V-N-H<sub>2</sub>O system occurs spontaneously, since all calculated values of  $\Delta G^\circ$  in the entire studied temperature range have negative values; the formation of decavanadium complexes, in particular  $[\text{HV}_{10}\text{O}_{28}]^{5-}$  ( $\Delta G^\circ = -8004,987 \text{ kJ/mol}$ ), is preferable.

The possibility of formation of vanadium complexes with a neutral ammonia molecule is shown. In ammonia solution the molecules of free ammonia start to replace the hydroxyl ions in decavanadate June, amicitia forming complexes of the type  $[(VO_2)_x(IT^-) \cdot nNH_3]^{n-x}$ , ensure its high solubility. Choosing the conditions for the formation of vanadium-containing ammonia particles and the emergence of coagulation contact between the particles, it is possible to cause spontaneous structuring and the formation of nanomaterial in the form of xerogel vanadium.

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### РАВНОВЕСНЫЕ СОСТОЯНИЯ СОЕДИНЕНИЙ ВАНАДИЯ, СПОСОБНЫХ К САМОПРОИЗВОЛЬНОМУ СТРУКТУРООБРАЗОВАНИЮ

**Аннотация.** В данной статье изложены результаты теоретического и термодинамического анализа равновесных состояний соединений ванадия с целью обоснования возможности синтеза ксерогеля в присутствии аммиака. Показано, что ванадий, относящийся к числу 3d-элементов, имеет способность образовывать комплексы. Одним из устойчивых комплексов является  $[\text{HV}_{10}\text{O}_{28}]^{-5}$ , свойства которого позволяют получать наноматериалы на основе ванадия.

**Ключевые слова:** комплексообразование, наноматериалы, темплат, ксерогель, золь-гель метод, термодинамический анализ, мономерные формы, полимерные формы, комплексы ванадия.

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### ӨЗДІГІНЕН ҚҰРЫЛЫМДЫ ТҮЗІЛІС ЖАСАЙТЫН, ВАНАДИЙ ҚОСЫЛЫСТАРЫНАН ТЕПЕ-ТЕҢДІК КҮЙЛЕРІ

**Аннотация.** Осы мақалада тепе-тең күйдегі ванадий қосылыстарының аммиактың қатысуымен ксерогельдің синтезі болу мүмкіндіктерін негіздеу мақсатында теориялық және термодинамикалық анализ нәтижелері баяндалған. 3d-элементтер қатарына жататын ванадийдің, кешендер құруына қабілеті бар екені көрсетілген. Бірі-тұрақты  $[\text{HV}_{10}\text{O}_{28}]^{-5}$  кешені болып табылады, оның қасиеттері ванадий негізінде наноматериалдар алуға мүмкіндік береді.

**Түйін сөздер:** комплекс түзу, наноматериалдар, темплат, ксерогель, золь-гель әдісі, термодинамикалық талдау, мономерлі пішіндер, полимерлік пішіндер, ванадий кешендері.

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