

КОМПЛЕКСООБРАЗОВАНИЕ В СИСТЕМЕ ЦИНК(II)-ХРОМ(III)-НИКЕЛЬ(II)-ГЛИЦИН-ВОДА

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Актуальность исследования комплексобразования в рассматриваемой системе связана с развитием роли гетероядерных соединений в процессах электрохимического получения сплавов. Согласно известным представлениям, в гетероядерных соединениях в связывающую орбиталь больший вклад вносит более электроотрицательный атом, а в разрыхляющую – орбиталь менее электроотрицательного атома. Это способствует сближению потенциалов восстановления ионов металлов, находящихся в таком соединении и получению электрохимического сплава. Кроме того, известно, что увеличение эффективного радиуса комплексных ионов, что характерно для гетероядерных соединений, уменьшает энергию активации их электрохимического восстановления. Целью работы является получение данных по комплексобразованию в системе Zn(II)–Cr(III)–Ni(II)–глицин–вода, составу и устойчивости гетероядерных соединений. Установлению состава образующихся комплексов в исследуемой системе предшествовало получение данных по комплексобразованию каждого из указанных элементов в индивидуальных растворах. Ранее установлено, что в системе Zn(II)–Cr(III)–глицин– вода при pH 1,0 – 3,5 преобладают гетероядерные формы $[CrZn(HGly)_4Gly_4]^+$ и $[CrZn(HGly)_2Gly_6]$, при pH 2,2 – 4,0 возрастает доля накопления $[CrZnGly_8]^{3+}$. Для установления состава образующихся комплексов применяли pH– метрическое титрование и метод ядерной магнитной релаксации (ЯМР) протонов в сочетании с математической обработкой результатов (программа CPESSP). Установлено образование гетероядерных комплексов $[CrNiZn(HGly)_4Gly_4]^{3+}$, $[CrNiZn(HGly)_2Gly_6]^+$, рассчитаны их константы образования и доли накопления (HGly – глицин). Результаты работы использованы при разработке электролитов для процесса электрохимического легирования цинковых покрытий одновременно хромом и никелем.

Ключевые слова: комплексные гетероядерные соединения, цинк(II), хром(III), никель(II), глицин, pH–метрическое титрование, ядерная магнитная релаксация протонов, программа CPESSP

COMPLEXATION IN THE SYSTEM ZINC (II)-CHROME (III)-NICKEL (II)-GLYCINE-WATER

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The relevance of studying complexation in the system under consideration is associated with the development of the role of heteronuclear compounds in the processes of electrochemical production of alloys. According to the known concepts, in heteronuclear compounds, the more electronegative atom makes a greater contribution to the bonding orbital, and the orbital of the less electronegative atom, to the antibonding one. This contributes to the convergence of the reduction potentials of metal ions present in such a compound and the production of an electrochemical alloy. In addition, it is known that an increase in the effective radius of complex ions, which is characteristic of heteronuclear compounds, decreases the activation energy of their electrochemical reduction. The aim of this work is to obtain data on complexation in the system Zn (II)–Cr (III)–Ni (II)–glycine–water, composition and stability of heteronuclear compounds. The establishment of the composition of the complexes formed in the system under study was preceded by the acquisition of data on the complexation of each of the indicated elements in individual solutions. It was previously established that in the Zn(II)–Cr (III)–glycine–water system in the pH range 1.0–3.5, heteronuclear forms $[\text{CrZn}(\text{HGly})_4\text{Gly}_4]^+$ and $[\text{CrZn}(\text{HGly})_2\text{Gly}_6]^-$ prevail, at pH 2.2 - 4.0, the share of $[\text{CrZnGly}_8]^{3-}$ accumulation increases. To establish the composition of the resulting complexes, we used pH metric titration and the method of nuclear magnetic relaxation (NMR) of protons in combination with mathematical processing of the results (CPESSP program). The formation of heteronuclear complexes $[\text{CrNiZn}(\text{HGly})_4\text{Gly}_4]^{3+}$, $[\text{CrNiZn}(\text{HGly})_2\text{Gly}_6]^+$ was established, their formation constants and the fraction of accumulation (HGly - glycine) were calculated. The results of the work were used in the development of electrolytes for the process of electrochemical alloying of zinc coatings simultaneously with chromium and nickel.

Key words: complex heteronuclear compounds, zinc (II), chromium (III), nickel (II), glycine, pH-metric titration, protons nuclear magnetic relaxation, CPESSP program

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INTRODUCTION

The study of complexation processes, despite significant advances in this area, is an urgent task and the subject of modern research [1-5]. In particular, obtaining data on complexation in the volume of electrolyte and on the electrode surface is the fundamental basis for effective regulation of the processes of electro-deposition of alloys and the kinetics of reactions [6]. The choice of the object of research is associated with electrochemical problems, in particular with the development of an electrolyte and a mode of obtaining corrosion-resistant zinc coatings alloyed with various metals [7-10] based on the study of complexation processes.

It is known that complexation affects not only the stage of reagent transport to the reaction zone, but also the characteristics of the energy barrier of electron transfer. Steric effects are also important in charge transfer across the electrode-electrolyte interface. The stated considerations are confirmed by electrodeposition of zinc-chromium alloy [6].

It was previously established that in the Zn (II) – Cr (III) – glycine – water system at pH 1.0-3.5, heteronuclear forms $[\text{CrZn}(\text{HGly})_4\text{Gly}_4]^+$ and $[\text{CrZn}(\text{HGly})_2\text{Gly}_6]^-$ prevail, and at pH 2.2-4.0 – complex $[\text{CrZnGly}_8]^{3-}$ [6].

The choice of glycine as a complexing agent is due to the fact that, being a bidentate ligand, it can form heteronuclear compounds in solutions, and besides, it has surface-active and buffer properties and is effectively used in electrochemical processes [11-17].

MATERIALS AND METHODS

The experimental technique and data processing are given in [18, 19].

The study of complexation was carried out using pH-metric titration and magnetic-relaxation method using the CPESSP program [18-20].

When using pH-metric titration, the dependences of the Bjerrum function \tilde{n} on pH were subjected to mathematical processing. The function \tilde{n} can be represented as the sum of the contributions of molar properties from each component:

$$\tilde{n} = \sum \alpha_i \cdot \tilde{n}_i, \quad (1)$$

where α_i is the molar fraction of the i -th complex; \tilde{n}_i – degree of titration of the i -th complex.

The calculation of \tilde{n} was made according to the formula:

$$\tilde{n} = (10^{-\text{pH}} \cdot (V_A + V_{\text{OH}}) + C_{\text{OH}} \cdot V_{\text{OH}} - 10^{\text{pH}-14} \cdot (V_A + V_{\text{OH}})) / (C_{\text{HnL}} \cdot (V_A + V_{\text{OH}})), \quad (2)$$

where V_{OH} is the volume of added titrant alkali; V_A is the volume of the titrated aliquot.

In the method of proton magnetic relaxation, the paramagnetic complex can be characterized by the relaxation efficiency coefficient (REC) – $K_{\text{э}1}$.

$$(BT_1)^{-1} = K_{\text{э}1} \quad (3)$$

$K_{\text{э}1}$ is an experimentally determined quantity in the NM relaxation method (dimension – $\text{mol}^{-1} \cdot \text{s}^{-1} \cdot \text{l}$).

$$K_{\text{э}1} = [(BT_1)_1]^{-1} = \frac{\sum_0^m K_{\text{э}1m} B_m}{\sum_0^m B_m} \quad (4)$$

where $K_{\text{э}1m}$ is the coefficient of relaxation efficiency; B_m is the concentration of the m -th complex.

The solutions were prepared using chemically pure and analytical grade substances.

The activity of hydrogen ions was determined on a HI 2215 pH/ORP Meter pH meter. The spin-lattice relaxation time was measured on a Minispec MQ 20 pulsed NMR spectrometer with an operating frequency of 19.75 MHz. The solutions were thermostated at 25 °C.

RESULTS AND DISCUSSION

Establishment of the composition and stability of complex compounds in the system zinc(II) – chromium(III) – nickel(II) – glycine – water was preceded by obtaining data in solutions of chromium(III) – water [18], zinc(II) – glycine – water [21], chromium(III) – glycine – water [22] and zinc(II) – chromium(III) – glycine – water [23]. The study showed that heteronuclear complexes are formed in the zinc(II) – chromium(III) – glycine – water system, and at $\text{pH} > 3$, $[\text{CrZnGly}_8]^{3-}$ is the main form.

The study of the nickel(II) – glycine – water system at metal: ligand ratios of 1:1 and 1:2 confirmed the formation of complexes NiGly^+ , NiGly_2 known from the literature [24, 25]. It was also preliminary found that in the pH range 2-4 the dependences of the relaxation efficiency for the nickel(II) – glycine and nickel(II) – zinc(II) – glycine systems on pH coincide, which indicates the absence of heteronuclear glycinate complexes.

The dependences of the relaxation efficiency for the systems chromium(III) – glycine – water, chromium(III) – nickel(II) – glycine – water, chromium(III) – nickel(II) – zinc(II) – glycine – water are shown in Fig. 1-2.

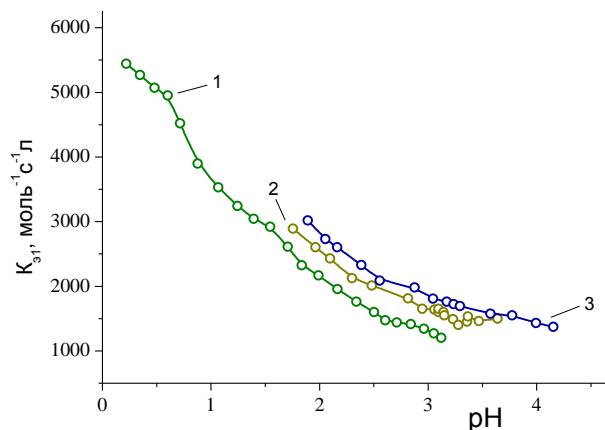


Fig. 1. Dependence of the relaxation efficiency of $(BT_1)^{-1}$ on the pH of the solution at a glycine concentration $C_{\text{HGly}} = 0.349 \text{ mol/l}$ in the following systems: 1- $C_{\text{Cr}^{3+}} = 0.045 \text{ mol/l}$; 2- $C_{\text{Cr}^{3+}} = 0.045 \text{ mol/l}$, $C_{\text{Ni}^{2+}} = 0.045 \text{ mol/l}$; 3- $C_{\text{Cr}^{3+}} = 0.045 \text{ mol/l}$, $C_{\text{Ni}^{2+}} = 0.045 \text{ mol/l}$, $C_{\text{Zn}^{2+}} = 0.048 \text{ mol/l}$

Рис. 1. Зависимость релаксационной эффективности $(BT_1)^{-1}$ от pH раствора при концентрации глицина $C_{\text{HGly}} = 0,349 \text{ моль/л}$ в следующих системах: 1- $C_{\text{Cr}^{3+}} = 0,045 \text{ моль/л}$; 2- $C_{\text{Cr}^{3+}} = 0,045 \text{ моль/л}$, $C_{\text{Ni}^{2+}} = 0,045 \text{ моль/л}$; 3- $C_{\text{Cr}^{3+}} = 0,045 \text{ моль/л}$, $C_{\text{Ni}^{2+}} = 0,045 \text{ моль/л}$, $C_{\text{Zn}^{2+}} = 0,048 \text{ моль/л}$

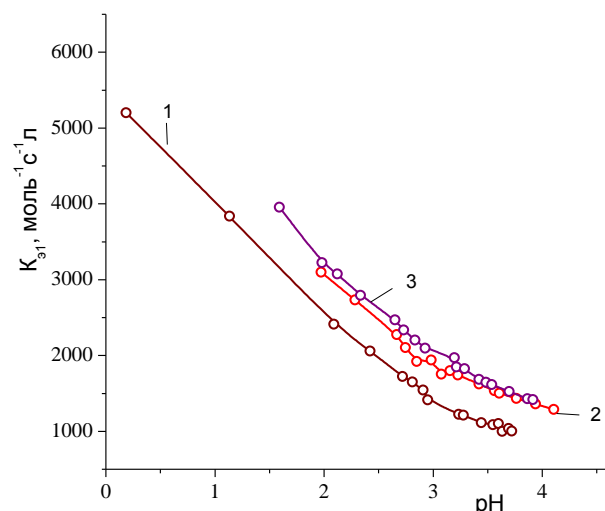


Fig. 2. Dependence of the relaxation efficiency $(BT_1)^{-1}$ on the pH of the solution at a glycine concentration $C_{\text{HGly}} = 0.901 \text{ mol/l}$ in the following systems: 1- $C_{\text{Cr}^{3+}} = 0.045 \text{ mol/l}$; 2- $C_{\text{Cr}^{3+}} = 0.045 \text{ mol/l}$, $C_{\text{Ni}^{2+}} = 0.045 \text{ mol/l}$; 3- $C_{\text{Cr}^{3+}} = 0.045 \text{ mol/l}$, $C_{\text{Ni}^{2+}} = 0.045 \text{ mol/l}$, $C_{\text{Zn}^{2+}} = 0.048 \text{ mol/l}$

Рис. 2. Зависимость релаксационной эффективности $(BT_1)^{-1}$ от pH раствора при концентрации глицина $C_{\text{HGly}} = 0,901 \text{ моль/л}$ в следующих системах: 1- $C_{\text{Cr}^{3+}} = 0,045 \text{ моль/л}$; 2- $C_{\text{Cr}^{3+}} = 0,045 \text{ моль/л}$, $C_{\text{Ni}^{2+}} = 0,045 \text{ моль/л}$; 3- $C_{\text{Cr}^{3+}} = 0,045 \text{ моль/л}$, $C_{\text{Ni}^{2+}} = 0,045 \text{ моль/л}$, $C_{\text{Zn}^{2+}} = 0,048 \text{ моль/л}$

The composition and stability constants are presented in Tables 1 and 2.

The term for the equilibrium constant is determined by the data in the "stoichiometric matrix":

$$\beta_{\text{pnmr}} = ([\text{Cr}_p \text{Ni}_n \text{H}_{2m-} \text{Gly}_m] [\text{H}]^r) / ([\text{Cr}]^p [\text{Ni}]^n [\text{H}_2\text{Gly}]^m) \quad (5)$$

(charge somitted).

Table 1

Composition and equilibrium constants of complex formation in the chromium (III) -nickel (II) -glycine-water system
Таблица 1. Состав и константы равновесий образования комплексов в системе хром(III)-никель(II)-глицин-вода

№	Стехиометрическая матрица				lgβ _{pqnmr}	Комплекс
	Cr ³⁺ (p)	Ni ²⁺ (n)	H ₂ Gly ⁺ (m)	H ⁺ (r)		
1	1	1	8	12	-1.72±0.11	[CrNi(HGly) ₄ Gly ₄] ⁺
2	1	1	8	14	-5.70±0.14	[CrNi(HGly) ₂ Gly ₆] ⁻
3	1	1	8	16	-15.14±0.22	[CrNiGly ₈] ³⁻

Table 2

Composition and equilibrium constants of complex formation in the system chromium (III)-nickel (II)-zinc (II)-glycine-water
Таблица 2. Состав и константы равновесий образования комплексов в системе хром(III)-никель(II)-цинк(II)-глицин-вода

№	Стехиометрическая матрица					lgβ _{pqnmr}	Комплекс
	Cr ³⁺ (p)	Zn ²⁺ (q)	Ni ²⁺ (n)	H ₂ Gly ⁺ (m)	H ⁺ (r)		
1	1	1	1	8	12	1.01±0.25	[CrNiZn(HGly) ₄ Gly ₄] ³⁺
2	1	1	1	8	14	-3.79±0.32	[CrNiZn(HGly) ₂ Gly ₆] ⁺

The term for the equilibrium constant is determined by the data in the "stoichiometric matrix" column:

$$\beta_{pqnmr} = ([Cr_p Zn_q Ni_n H_{2m-r} Gly_m][H]^r) / ([Cr]^p [Zn]^q [Ni]^n [H_2Gly]^m) \quad (6)$$

(charges omitted).

Calculation of the fraction of accumulation of heteronuclear complexes shows (Table 3) that they exist in the pH range 2-3.5. In this pH range, the fraction of accumulation of [CrNiZn(HGly)₄Gly₄]³⁺ varies within 0.0-0.57, the fraction of [CrNiZn(HGly)₂Gly₆]⁺ – within 0.05-0.46.

Table 3

Shares of accumulation of heteronuclear complexes depending on pH (the shares of the remaining complexes are not indicated)

Таблица 3. Доли накопления гетероядерных комплексов в зависимости от pH (Доли остальных комплексов не указаны)

pH	Доля [CrNiZn(HGly) ₄ Gly ₄] ³⁺	Доля [CrNiZn(HGly) ₂ Gly ₆] ⁺
1.891	0.57	0.05
2.050	0.54	0.11
2.385	0.39	0.3
2.556	0.20	0.4
2.876	0.05	0.46
3.045	0.02	0.41
3.236	0	0.31
3.570	0	0.13

Studies have shown that heteronuclear complexes [CrNiZn(HGly)₂Gly₆]⁺, [CrNiZn(HGly)₄Gly₄]³⁺ are formed in the system zinc(II) – chromium(III) – nickel(II) – glycine – water.

According to the well-known concepts of the theory of molecular orbitals about heteronuclear complexes [26], a more electronegative atom makes a

greater contribution to the bonding orbital, and a less negative atom to the antibonding orbital. The electrochemical activity of more electronegative metals in the case of their presence in a heteronuclear complex should be higher, i.e. they should be restored at lower overvoltages, which has a positive effect on the convergence of the reduction potentials of various metal ions during alloy formation.

In the case of electrochemical alloying of zinc coatings with chromium and nickel, the presence of a positive charge in the complex compounds [CrNiZn(HGly)₂Gly₆]⁺, [CrNiZn(HGly)₄Gly₄]³⁺ creates favorable conditions for their adsorption on the negatively charged electrode surface and charge transfer.

The data obtained can be used in the development of electrolytes for obtaining zinc coatings doped with chromium and nickel.

CONCLUSION

The data on the composition and stability of complex compounds formed in the system Zn(II) – Cr(III) – Ni(II) – glycine – water were obtained by means of pH-metry and NMR with subsequent mathematical processing of the experiment.

The heteronuclear forms of the [CrNiZn(HGly)₄Gly₄]³⁺, [CrNiZn(HGly)₂Gly₆]⁺ complexes have been determined, and their formation constants and the fraction of accumulation have been calculated.

The authors declare the absence a conflict of interest warranting disclosure in this article.

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