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БІОМЕДИЧНА ІНЖЕНЕРІЯ

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EFFECT OF pH ON THE STABILITY OF COORDINATION COMPOUNDS OF CO(III) WITH DIAMINOETHANOL LIGANDS IN NON-AQUEOUS SOLUTIONS

I.S. Кузеванова, С.В. Повальчук, А.О. Зулфігаров, О.О. Андрійко, В.А. Потаскалов, Н.С. Власенко. Вплив зміни рН середовища на стійкість координаційних сполук Co(III) з діетаноламіном в неводних розчинниках. Виконано синтез внутрішньокмплесної (ВКС) сполуки кобальту(III) з діетаноламіном [Co(DetmHdetm)] та гетероядерної кмплесної сполуки (ГМК) 2Co-Ni з діетаноламіном [Ni(CoDetmHdetm)₂](NO₃)₂. Стійкість отриманих сполук досліджувалась в неводному розчиннику (диметилформаміді) за різних значень рН (від кислого до лужного середовищ). За допомогою методів потенціометричного титрування та електронних спектрів поглинання були визначені межі значень рН середовища, де кмплесні сполуки не піддавались деструкції. За результатами проведених досліджень було визначено, що значенні рН вище 7 змін координаційного оточення металів (Co(III), Ni(II)) не відбувається. Коли рН зменшється нижче 7 (при додаванні хлоридної кислоти), внутрішню кмплесна сполука Co(III) зазнає руйнування. Ліганд H₂detm змінюється на Cl⁻ та частково протонізується залишок діетаноламіну. Крім того, відбувається перетворення цис-N,N ізомеру внутрішню кмплесної сполуки в транс-N,N ізомер і як наслідок змінюється симетрія координаційної сполуки. У випадку гетерометалічного кмплесу 2Co-Ni, внаслідок зниження рН нижче 7 молекули розчинника починають реагувати з продуктами часткової деструкції кмплесної сполуки. В результаті відбувається утворення нового кмплесу, в якому координаційне число нікелю(II) зростає з 4 до 6. Водночас, при підвищенні рН більше 10 гетерометалічний кмплес залишається стабільним. Має місце лише частковий процес гідролізу розчинника. Наведені схеми перетворень [Co(DetmHdetm)] та [Ni(CoDetmHdetm)₂](NO₃)₂, що описують поведінку кмплесних сполук в неводному розчиннику (диметилформаміді) при зміні рН від кислого до лужного середовищ. За результатами виконаних досліджень, отриманий матеріал можна використати при розробці новітньої технології створення каталітичних матеріалів на основі продуктів термічної деструкції кмплесних сполук кобальту(III)-нікелю(II). Очевидно, що повинен підтримуватися діапазон рН від 7 до 10 при синтезі компонентів каталітичних матеріалів, щоб уникнути забруднення кінцевих продуктів залишками деструкції кмплесних сполук та розчинника.

Ключові слова: діетаноламін, внутрішньокмплесна сполука кобальту(III), гетерометалічний кмплес кобальту(III)-нікелю(II), диметилформамід, вплив рН на стійкість

I. Kuzevanova, S. Povalchyk, A. Zulfigarov, A. Andriiko, V. Potaskalov, N. Vlasenko. Effect of pH on the stability of coordination compounds of Co(III) with diaminoethanol ligands in non-aqueous solutions. Inner complex compound of cobalt(III) with diaminoethanol, [Co(DetmHdetm)], and polynuclear complex compound 2Co-Ni with diaminoethanol, [Ni(CoDetmHdetm)₂](NO₃)₂, were synthesized. Stabilities of the obtained compounds were investigated in non-aqueous (dimethylformamide) solutions at different values of pH (from acid to alkaline). Methods of potentiometric titration and electron absorption spectra were used to determine the range of pH values where the compounds do not decompose. As a result of these studies, we found that at the upper value of pH=7, the coordination surrounding of the metals (Co(III), Ni(II)) does not change. When the pH value becomes lower than 7 (with adding HCl acid), the inner complex of Co(III) begins to decompose. The ligand H₂detm is replaced by Cl⁻ and partial protonization of diethanolamine occurs. Also, transformation of N,N-cis isomer of inner complex compound of cobalt(III) to N,N-trans isomer takes place, thus reducing the symmetry of the complex. As for the

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polynuclear complex compound 2Co-Ni, when pH value becomes lower than 7, molecules of solvent begin to react with the products of partially destroyed complex. As a result, new complex compound is formed with the increase of coordination number of nickel(II) from 4 to 6. However, in alkaline solutions where pH values are up to 10, the heterometal complex compound of 2Co-Ni remains stable. Only partial hydrolysis of the solvent occurs in these conditions. The schemes of transformations of $[\text{Co}(\text{DetmHdetm})]$ and $[\text{Ni}(\text{CoDetmHdetm})_2](\text{NO}_3)_2$ occurring in non-aqueous (dimethylformamide) solutions at different values of pH (from acid to alkaline) are presented in the paper. The results of this study can be used for the preparation of precursor solution for the technology of catalytic materials production. Evidently, the range of pH from 7 to 10 must be maintained in order to avoid the contamination of the final products by the decomposed wastes of complexes and solvent.

Keywords: diaminoethanol, Cobalt(III) inner complexes, Cobalt(III)-Nickel(II) heterometal complexes, dimethylformamide solutions, pH range of stability

Introduction

The article is devoted to preparation of polynuclear complex compounds of cobalt (III) with aminoalcohol ligands and further studies of their structure as well as electrocatalytic properties in the reactions of intercalation /deintercalation of lithium in carbon materials and reactions of discharge-ionization of hydrogen on carbon nanotubes. Heterometals complex cobalt(III)-nickel(II) with monoethanolamine deposited on the surface of carbon nanotubes exhibits high catalytic properties in the reaction of discharge-ionization of hydrogen, comparable in efficiency to platinum-based catalysts used in modern systems for electrochemical storage of hydrogen. So, the creation of manufactured technology of these catalytic materials are very important nowadays. But the stability of coordination compounds of Co(III) with aminoethanol ligands wasn't investigated for depending of the range of pH environment. This parameter is very important, because it straightly influence on the structure of catalytic materials.

Analysis of recent research and publications

The properties of 3d metal compounds attract considerable attention mainly due to their catalytic activity in various reactions [1 – 5], which is sometimes comparable to the activity of traditional catalysts based on iron family metals [6, 7, 8]. The 3d metal compounds which to allow catalyzing main part of chemical technology processes continue investigating to develop more effective composition and maximum universal methods of causing [9, 10, 11]. In particular, it was shown [12] that the products of pyrolytic destruction of polynuclear complex compounds of cobalt(III)-nickel(II) with aminoalcohol ligands grafted onto the surface of carbon materials are catalytically active relatively to some electrolytic reactions such as reduction of oxygen, intercalation of lithium into graphite and discharge-ionization of hydrogen. Synthesis of heterometal complex compounds 2Co-Ni with diaminoethanol was elaborated for the preparation of catalytically active centers in [13]. The conditions of temperature treatment were determined. The structure of coordination compound 2Co-Ni was investigated which allowed to predict the composition of active catalytic centers on the surface after the pyrolytic decomposition [14]. The non-aqueous solutions of the polynuclear complexes are used as precursors for grafting the complexes on the surface of catalytic materials before the thermal treatment. The question of stability of the complexes in such solution and its dependence on the acid-base properties of the solvent is not sufficiently studied yet.

The aim and objectives of the study

Thus, the aim of the present work was to establish the range of acidity (pH) where the solutions are stable and investigate the mechanisms of decomposition of heterometal complex compounds 2Co-Ni with diaminoethanol beyond the range of stability.

Experimental Methodology

Cobalt(II) nitrate and Nickel(II) nitrate, Alkali hydroxide, Diaminoethanol (DEA) and Dimethylformamide (DMFA) were used for preparation of the complex compounds. Synthesis of polynuclear complex compound 2Co-Ni with diaminoethanol $[\text{Ni}(\text{CoDetmHdetm})_2](\text{NO}_3)_2$ were performed in two steps.

The first, the inner complex compound (ICC) of cobalt(III) with diaminoethanol $[\text{Co}(\text{DetmHdetm})]$ (where $\text{H}_2\text{detm} - \text{HN}(\text{CH}_2\text{CH}_2\text{OH})_2$; H_2detm^- and Detm^{2-} – deprotonated aminoalcohol molecules) were obtained by oxydation of Co^{2+} to Co^{3+} with ambient oxygen in alkaline media according to the reaction:



The second, the polynuclear complex compound 2Co-Ni with diaminoethanol were synthesized as follows. Nickel(II) nitrate was dissolved in moderately hot DMFA. After cooling solution to room temperature, it was mixed with the before prepared solution of inner complex compound of cobalt(III).

The resulting mixture remained aged for three days. Reaction (2) describes the process of formation of the heterometal polynuclear complex compound:



Elemental chemical analysis was performed by gas chromatographic method on Carlo Erba Elemental Analyzer 1106 (for C, N, H) and by induction-couple atomic emission spectroscopy method on Jobin Yvon-Horiba 180 Ultrace (for Co, Ni).

Investigation of the stability of $[\text{Co}(\text{DetmHdetm})]$ and $[\text{Ni}(\text{CoDetmHdetm})_2](\text{NO}_3)_2$ in the solutions with various pH was made with pH-meter Akvilon pH410. Variation of the pH was achieved by potentiometric titration with aqueous solutions of HCl and NaOH.

The presence of Co^{2+} ions due to the possible incomplete oxidation (reaction (1)) was checked by the method of Ox-RedOx titration with potassium hexacyanoferrate (III) in ammonia buffer.

The structure transformations in the solutions were studied by electron absorption spectra method (EAS) using VIS spectrometer UNICO2100.

Results and discussion

The results of elemental chemical analysis of the obtained complexes are given in Table. One can see that the experimentally obtained values are in good agreement with the calculated values for the composition of complex compounds of cobalt(III) and nickel(II) according to the reactions (1) and (2). So, it confirms the accepted formulas of both complex compounds.

Results of elemental chemical analysis coordination compounds Co(III) and Ni(II) with diaminoethanol

Compound	C, %		N, %		H, %		Co, %		Ni, %	
	Calc.	Exper.	Calc.	Exper.	Calc.	Exper.	Calc.	Exper.	Calc.	Exper.
$[\text{Co}(\text{DetmHdetm})]$	36.09	36.15	10.53	10.45	7.14	7.23	22.18	22.25	–	–
$\{\text{Ni}[\text{Co}(\text{DetmHdetm})_2]\}(\text{NO}_3)_2$	26.74	26.69	11.7	11.8	5.29	5.33	16.44	16.57	8.22	8.31

After the synthesis, the solution of ICC in DMFA had the value of pH=10.96. The potentiometric titration curve of this solution by hydrochloric acid is shown in Fig. 1. The inflection point at pH 5.6 on the titration curve is observed followed by the descending part.

The RedOx titration with potassium hexacyanoferrate (III) in ammonia buffer [15] did not detect the ions of Co^{2+} in the solution.

The method of electron adsorption spectra was used to study the transformations when pH decreases in course of adding the hydrochloric acid to the non-aqueous solution of inner complex compound of cobalt(III) with diaminoethanol. The electron spectra are shown in Fig. 2.

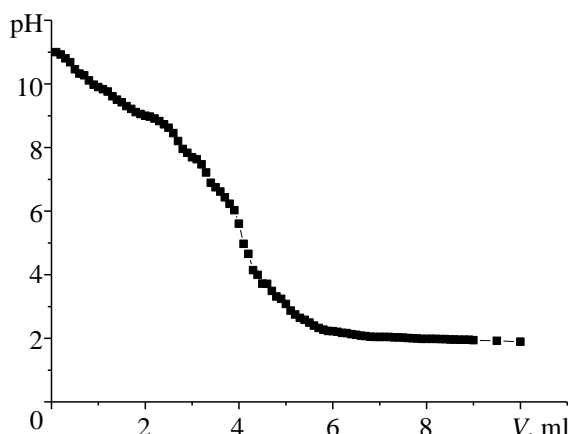


Fig. 1. Curve of potentiometric titration ($C(\text{HCl}) = 0.1 \text{ mol/l}$) of $\text{Co}(\text{DetmHdetm})$ dimethylformamide solution

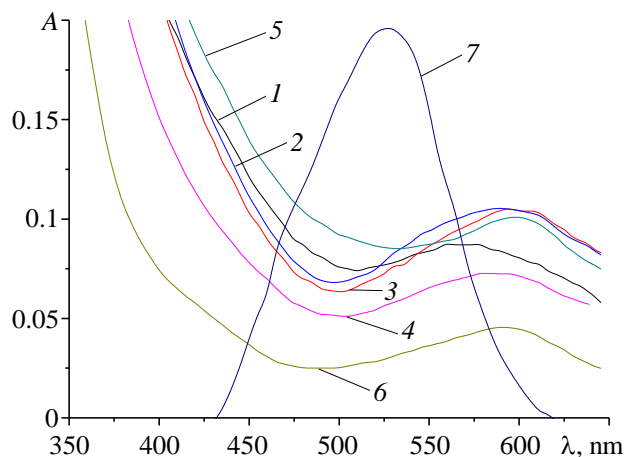
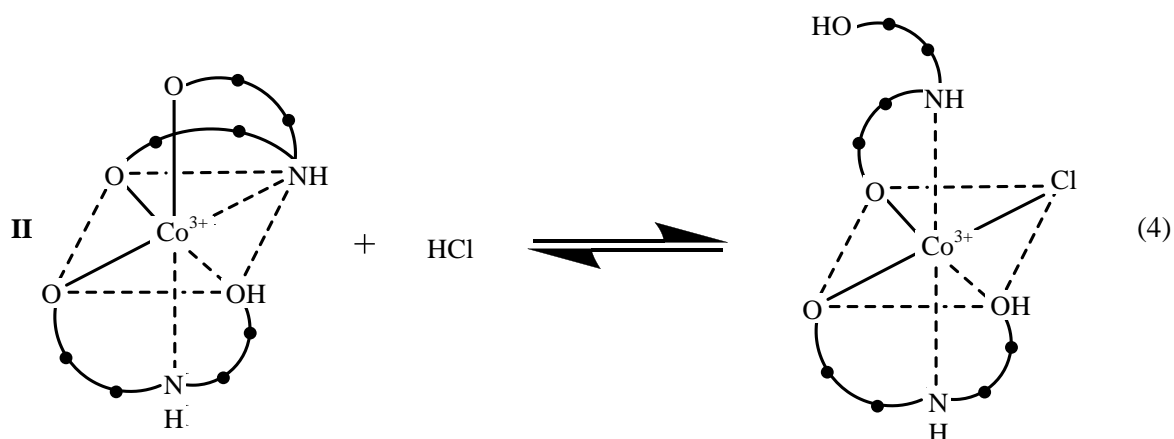
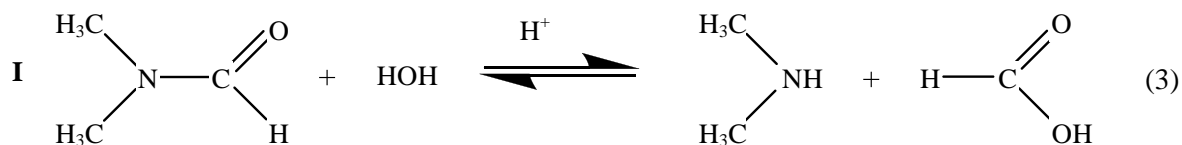


Fig. 2. The electron spectra in acid media of ICC cobalt(III) with diaminoethanol and reactant (1 – pH 10.96; 2 – pH 6.03; 3 – pH 4.01; 4 – pH 3.17; 5 – pH 2.08; 6 – pH 1.31; 7 – DMFA solution of Co^{2+})

EAS of the samples corresponding to different points of potentiometric curve (pH 10.96; 6.03; 4.01; 3.17; 2.08; 1.31) detect the bathochrome shift of absorption maximum Co^{3+} related to d-d transition ${}^1A_{1g} \rightarrow {}^1T_{1g}$ of cobalt (III) from 575nm (for initial ICC Co^{3+} spectra) to 600 nm starting at pH 6.03.

Further on, the intensity of spectra curve decreases with the addition of acid but absorption maximum does not shift. These results permit to describe the probable scheme of the destruction of ICC Co^{3+} with diaminoethanol in acid media that by two step reaction:



The first step (reaction (3)) is the partial hydrolysis of solvent. Further addition of the acid results in the destruction of aminoalcohol complex compound when the ligand H_2detm is replaced by Cl^- and partial protonization of diaminoethanol takes place. Also N, N-cis isomer of the inner complex of cobalt(III) transforms into the N,N-trans isomer with symmetry reduction of the complex [16] (reaction (4)).

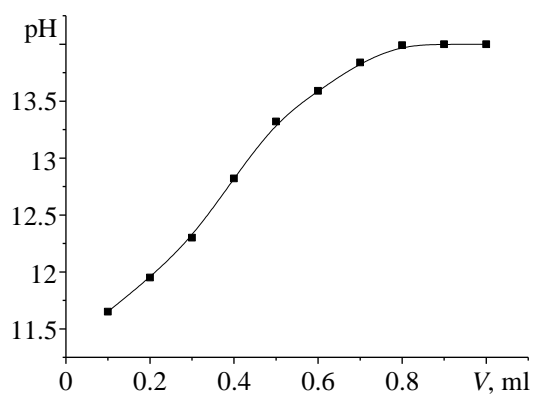


Fig. 3. Curve of potentiometric titration ($C(\text{NaOH}) = 0.1 \text{ mol/l}$) of $\text{Co}(\text{DetmHdetm})$ dimethylformamide solution

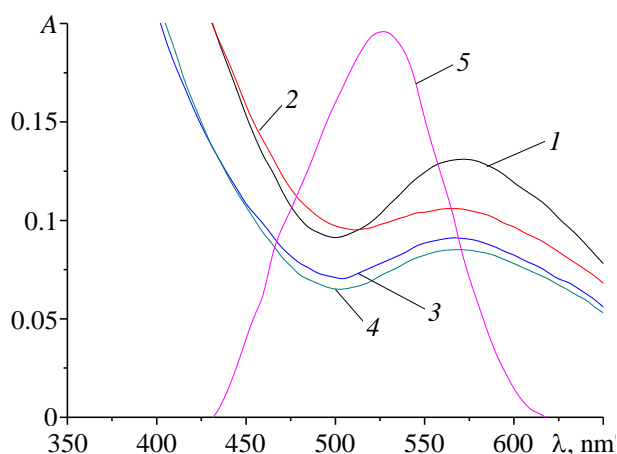
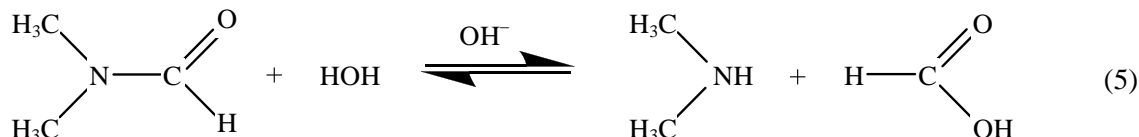


Fig. 4. The electron spectra in alkaline media of ICC cobalt(III) with diaminoethanol and the precursor of its synthesis (1 – pH 10.96; 2 – pH 12.5; 3 – pH 13.5; 4 – pH 14; 5 – DMFA solution of Co^{2+})

One inflection point at pH=12.7 is observed on the titration curve of the solution of inner complex of cobalt (III) with diaminoethanol by sodium hydroxide (Fig. 3). Again, no traces of Co^{2+} in the solution was detected by Ox-RedOx titration with potassium hexacyanoferrate (III) in ammonia buffer.

However, no shift of the absorption maximum of Co^{3+} at 575nm is observed in electron absorption spectra (Fig. 4) when pH increases to 14.

The presence of only one equivalent point on the potentiometric curve and absence of the peak's shift in alkali solution evidences that the complex is stable in these conditions. The partial hydrolysis of DMFA (reaction (5)) may occur:



Further, we have studied the influence of pH on the stability and transformations of heterometal complex (HMC) compound cobalt(III)-nickel(II) with diaminoethanol in non-aqueous solutions.

The pH value of the solution of HMC in DMFA after the synthesis was equal to 9.34.

Fig. 5 represents the potentiometric titration curve of the solution of $[\text{Ni}(\text{CoDetmHdetm})_2](\text{NO}_3)_2$ by hydrochloric acid. One inflection point at pH 5.7 can be detected, with inclined descend before this point. Again, no traces of Co^{2+} was found by the method of Ox-RedOx titration with potassium hexacyanoferrate (III) in ammonia buffer.

Analyzing the EA spectra of the solutions corresponding to the different points of the titration curve (Fig. 5), one can observe the bathochrome shift of the absorption maximum of Co^{3+} in HMC from 570 nm to 605 nm, which corresponds to d-d transition ${}^3\text{A}_{2g} \rightarrow {}^3\text{T}_{2g}(\text{F})$ of nickel(II). The spectra became significantly changed at the pH less than 6.05.

Most probably, it indicates that adding 2 molecules of dimethylamine (it appears because of hydrolysis DMFA) to the squared heterometal complex of cobalt(III)-nickel(II) with diaminoethanol results in the formation of new octahedral complex compound. Further adding of acid does not shift the absorption maximum even when pH 1.5 reaches (Fig. 6).

The obtained results make possible to present most probable scheme of destruction of HMC 2Co-Ni with diaminoethanol in

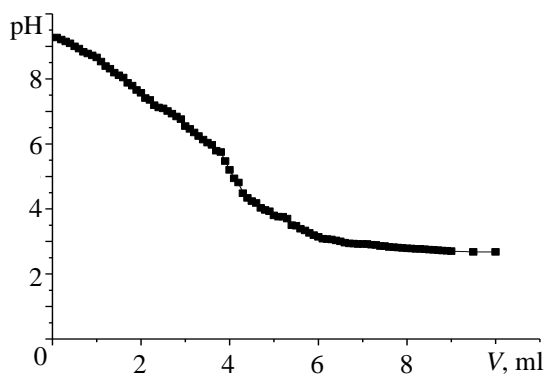


Fig. 5. Potentiometric titration curve ($C(\text{HCl}) = 0.1 \text{ mol/l}$) of $[\text{Ni}(\text{CoDetmHdetm})_2](\text{NO}_3)_2$ in dimethylformamide solution

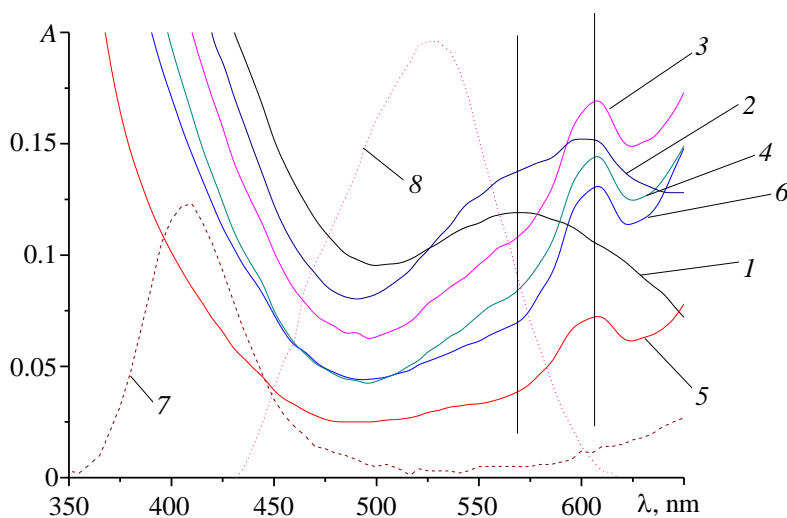


Fig. 6. Electron spectra of HMC cobalt(III)-nickel(II) with diaminoethanol in acid media (1 – pH 9.34; 2 – pH 6.05; 3 – pH 3.98; 4 – pH 2.9; 5 – pH 2.68; 6 – pH 1.5; 7 – DMFA solution of Ni^{2+} ; 8 – DMFA solution of Co^{2+})

acid media. It seems to proceed in two steps. The first is partial hydrolysis of solvent (like before, reaction (3)) and the second is the formation of new complex compound (reaction (6)):

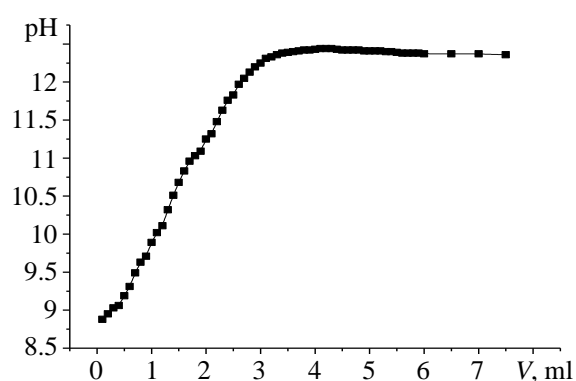
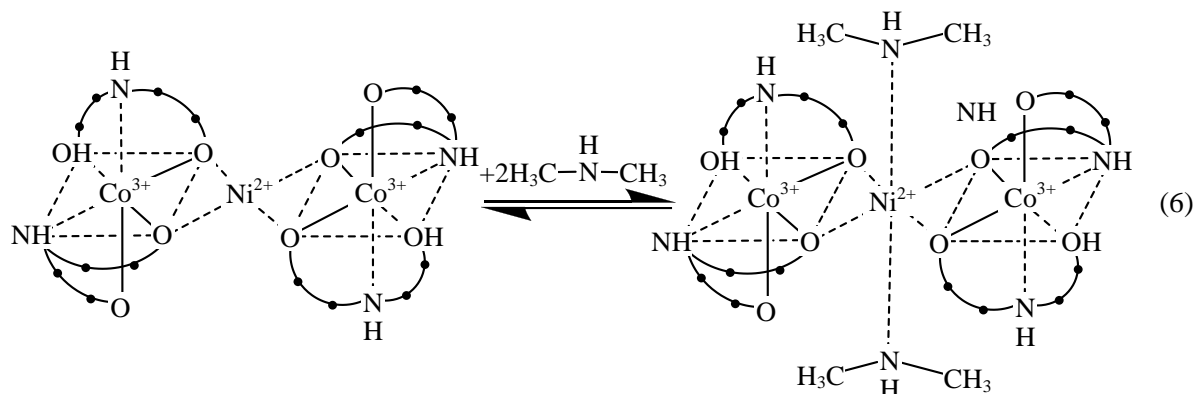


Fig. 7. Potentiometric titration curve of $[Ni(CoDetmHdetm)_2](NO_3)_2$ in dimethylformamide solution with alkali solution ($C(NaOH) = 0.1 \text{ mol/l}$)

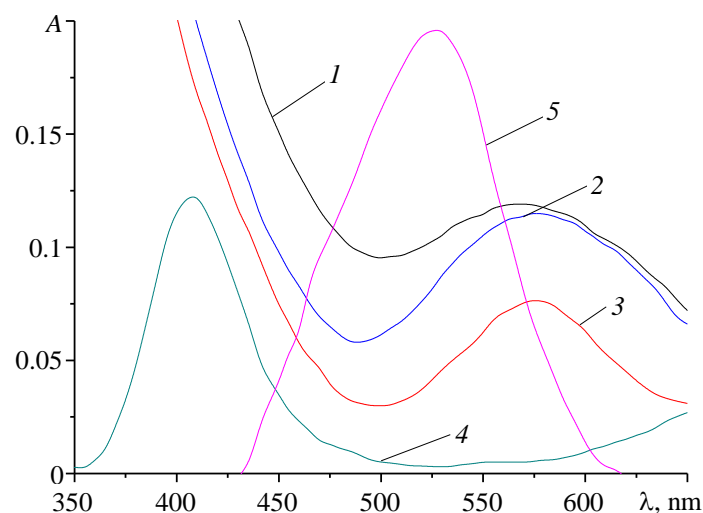


Fig. 8. The electron spectra of HMC cobalt(III) with diethanolamine in alkali media (1 – pH 9.34; 2 – pH 10.5; 3 – pH 12.4; 4 – DMFA solution of Ni^{2+} ; 5 – DMFA solution of Co^{2+})

Fig. 7 represents the titration curve of the solution of heterometal complex of cobalt(III)-nickel(II) by sodium hydroxide. One inflection point is observed in this curve at pH 11. Again, no traces of Co^{2+} ions were found.

The displacement of the absorption maximum of Co^{3+} at 575nm (corresponding to d-d transition ${}^1A_{1g} \rightarrow {}^1T_{2g}$) is not observed in the spectra of Fig. 8 when value of pH increases to 12.4. The ions Co^{2+} were not detected after the end of titration.

The presence of only one equivalent point on the titration curve and no displacement of absorption maximum of HMC cobalt(III)-nickel(II) proves that the complex compounds in alkali environment are stable and indicates only partial hydrolysis of DMFA (reaction (5)).

Comparing the obtained results with the data of [15], we must admit that the effect of the solvent is not crucial. The complex remains stable in a neutral and weak base environment and decomposes in the acid media, both in alcohol and in DMFA solutions. However, in case of DMFA, hydrolysis of the solvent may result in the change of the composition of the precursor solution, which should be taken into account.

Conclusions

The results of the studies permit to define the range of acidity (pH) where the inner complex compound of cobalt(III) with diaminoethanol and heterometal complex $2Co-Ni$ with diaminoethanol in non-aqueous solutions are stable.

As follows from the electron absorption spectra and the results of Ox-Redox titration, the coordination environment of metals (Co(III), Ni(II)) does not change at pH moderately higher than 7. Even when the value of pH increases over 10, the compounds in DMFA solutions remained stable. However, at these conditions, the process of solvent hydrolysis begins to develop. The schemes of corresponding transformations occurring with Co(DetmHdetm) and $[\text{Ni}(\text{CoDetmHdetm})_2](\text{NO}_3)_2$ which are affected by acid or alkali, are presented.

The obtained results indicate the necessity of pH control of the reaction media when the solutions of heterometal complex compounds are used as precursors for preparation of the catalytic materials. A range of pH from 7 to 10 is recommended in order to avoid the contamination of the catalysts with the products of solvent and complex destruction reactions.

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