ISSN 2518-1491 (Online),
ISSN 2224-5286 (Print)

«ҚАЗАҚСТАН РЕСПУБЛИКАСЫ
ҰЛТТЫҚ ҒЫЛЫМ
АКАДЕМИЯСЫ» РҚБ

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ХАБАРЛАРЫ

ИЗВЕСТИЯ

РОО «НАЦИОНАЛЬНОЙ
АКАДЕМИИ НАУК РЕСПУБЛИКИ
КАЗАХСТАН»

NEWS

OF THE ACADEMY OF SCIENCES
OF THE REPUBLIC OF
KAZAKHSTAN

SERIES
CHEMISTRY AND TECHNOLOGY

1 (462)

JANUARY – MARCH 2025

PUBLISHED SINCE JANUARY 1947

PUBLISHED 4 TIMES A YEAR

ALMATY, NAS RK

«ҚР ҰҒА Хабарлары. Химия және технология сериясы»
ISSN 2518-1491 (Online),
ISSN 2224-5286 (Print)
Меншіктенуші: «Қазақстан Республикасының Ұлттық ғылым академиясы» РҚБ (Алматы қ.).
Ақпарат агенттігінің мерзімді баспасөз басылымын, ақпарат агенттігін және желілік басылымды қайта есепке қою туралы ҚР Мәдениет және Ақпарат министрлігі «Ақпарат комитеті» Республикалық мемлекеттік мекемесі **28.02.2025 ж.** берген **№KZ63VPY00113743** Куәлік.
Тақырыптық бағыты: *химия және химиялық технология*
Мерзімділігі: жылына 4 рет.
http://chemistry-technology.kz/index.php/en/arhiv

«Известия НАН РК. Серия химии и технологии».
ISSN 2518-1491 (Online),
ISSN 2224-5286 (Print)

Собственник: Республиканское общественное объединение «Национальная академия наук Республики Казахстан» (г. Алматы).

Свидетельство **№KZ63VPY00113743** о повторной регистрации периодического печатного издания информационного агентства, информационного агентства и сетевого издания, выданное Республиканским государственным учреждением «Комитет информации» Министерства культуры и информации Республики Казахстан **28.02.2025 г**.

Тематическая направленность: *химия и химические технологии*

Периодичность: 4 раза в год.

http://chemistry-technology.kz/index.php/en/arhiv

News of the National Academy of Sciences of the Republic of Kazakhstan. Series of chemistry and technology.
ISSN 2518-1491 (Online),
ISSN 2224-5286 (Print)

Owner: RPA «National Academy of Sciences of the Republic of Kazakhstan» (Almaty).

The certificate of registration of a periodical printed publication in the Committee of Information of the Ministry of Information and Social Development of the Republic of Kazakhstan **No. KZ66VPY00025419,** issued **29.07.2020.**

Thematic scope: *organic chemistry, inorganic chemistry, catalysis, electrochemistry and corrosion, pharmaceutical chemistry and technology.*

Periodicity: 4 times a year.

Editorial address: 28, Shevchenko str., of. 219, Almaty, 050010, tel. 272-13-19

http://chemistry-technology.kz/index.php/en/arhiv

NEWS
OF THE NATIONAL ACADEMY OF SCIENCES OF THE REPUBLIC OF KAZAKHSTAN
SERIES CHEMISTRY AND TECHNOLOGY
ISSN 2224–5286
Volume 1. Number 462 (2025), 144–156

https://doi.org/10.32014/2025.2518-1491.273

МРНТИ 31.15.37

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DETERMINATION OF COAGULATION THRESHOLDS OF MOLYBDENUM-VANADIUM BLUE SOLS

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Abstract. Molybdenum-vanadium blue sols are colloidal dispersions known for their unique optical and chemical properties, making them important in catalysis, sensors, and materials science. The article discusses the coagulation threshold of the sol and the effect of electrolytes such as potassium nitrate, potassium chloride, and calcium nitrate on sol coagulation. This work discusses the conditions for the occurrence of coagulation, and considers the experience of conducting an experiment to achieve the goal. During the experimental work, it was concluded that to determine the coagulation threshold of the sol, it is necessary to take reagents with high concentrations, both sol and electrolytes. Since it is known that Mo-V blue is aggregation stable.

Thus, in the course of work, a sol Mo-V blue ratio [Mo]:[V] = 90:10 was synthesized using ascorbic acid as a reducing agent. The low content of ash and electrolyte, especially in the case of stable systems, such as molybdenum-vanadium blue, had to initiate reduced coagulation.

To achieve significant effects, high temperatures or changes in environmental conditions (pH, temperature) are necessary. For sol Mo-V, the constants of fast coagulation and half-time of coagulation were determined for KNO_3: $k_b = 5 \cdot 10^{-4}$ m^3/particle•s and Θ = 1563 s, for KCl: kb = $4 \cdot 10^{-4}$ m^3/particle·s and Θ = 1000 s.

Keywords. Sol, coagulation, electrolytes, polyoxometalates, aggregation stability

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Аннотация. Молибден-ванадий көгілдір түсті ерітінділер – олардың бірегей оптикалық және химиялық қасиеттерімен танымал коллоидтық дисперсиялар, оларды катализде, сенсорикада және материалтануда маңызды етеді. Мақалада золь коагуляциясының шегі және калий нитраты, калий хлориді және кальций нитраты сияқты электролиттердің золь коагуляциясына әсері қарастырылды. Бұл жұмыста коагуляцияның пайда болу жағдайлары қарастырылады, сонымен қатар мақсатқа жету үшін эксперименттер жүргізу тәжірибесі жасалды. Тәжірибелік жұмыс барысында зольдің коагуляция шегін анықтау үшін зольдің де, электролиттердің де концентрациясы жоғары реагенттерді алу керек деген Mo-V көктің агрегативті тұрақты екені белгілі болды.

Сондықтан, жұмыс барысында тотықсыздандырғыш ретінде аскорбин қышқылын қолданып, [Mo]:[V] = 90:10 қатынасы бар Mo-V көк золь синтезделді. Төмен күл мен электролит мөлшері, әсіресе молибден-ванадий көк сияқты тұрақты жүйелер жағдайында коагуляцияның төмендеуіне әкеледі.

Осындай маңызды әсерлерге қол жеткізу үшін жоғары температура немесе қоршаған орта жағдайларының өзгеруі (pH, температура) қажет. Mo-V зол үшін жылдам коагуляция константалары және коагуляцияның жартылай шығарылу кезеңі KNO_3 үшін анықталды: $k_b = 5 \cdot 10^{-4}$ м^3/бөлшек·с және Θ = 1563 с, KCl үшін: $k_b = 4 \cdot 10\text{-}4$ м^3/бөлшек · с және Θ = 1000 с.

Түйін сөздер. Золь, коагуляция, электролиттер, полиоксометалаттар, агрегациялық тұрақтылық

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ОПРЕДЕЛЕНИЕ ПОРОГОВ КОАГУЛЯЦИИ ЗОЛЕЙ МОЛИБДЕН-ВАНАДИЕВЫХ СИНЕЙ

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Аннотация. Золи молибденово-ванадиевого синего представляют собой коллоидные дисперсии, известные своими уникальными оптическими и химическими свойствами, что делает их важными в катализе, сенсорике и материаловедении. В статье рассматривается порог коагуляции золя и влияние электролитов, таких как нитрат калия, хлорид калия и нитрат кальция, на коагуляцию золя. В данной работе обсуждаются условия возникновения коагуляции, а также рассматривается опыт проведения эксперимента для достижения цели. В ходе экспериментальной работы был сделан вывод, что для определения порога коагуляции золя необходимо брать реагенты с высокими концентрациями, как золя, так и электролитов. Так как известно, что Mo-V синий агрегативно устойчив.

Таким образом, в ходе работы был синтезирован золь Mo-V синий с

соотношением [Mo]:[V] = 90:10 с использованием аскорбиновой кислоты в качестве восстановителя. Низкое содержание золы и электролита, особенно в случае стабильных систем, таких как молибден-ванадиевая синь, должно было инициировать пониженную коагуляцию.

Для достижения значительных эффектов необходимы высокие температуры или изменения условий окружающей среды (pH, температура). Для золя Mo-V были определены константы быстрой коагуляции и полупериод коагуляции для KNO_3: $k_b = 5•10^{-4}$ м^3/частица•с и Θ = 1563 с, для KCl: k_b = 4•10-4 м^3/частица•с и Θ = 1000 с.

Ключевые слова: золь, коагуляция, электролиты, полиоксометталаты, агрегативная устойчивость.

Introduction. Colloidal systems, such as molybdenum-vanadium blue salts, exhibit unique optical and physical properties that largely depend on their stability and aggregation behavior. The critical concentration of coagulation is the most important parameter determining the stability of these colloids, since it marks the threshold at which the Van der Waals attraction overcomes the stabilizing forces, which leads to irreversible aggregation (Berouaken, et al, 2020).

In this study, we examine the coagulation behavior of molybdenum-vanadium blue salts by systematically changing the salt concentration and tracking changes in the average hydrodynamic diameter over time. The initial slope of the kinetic curves is proportional to the coagulation rate, which is small at sufficiently low salt concentrations and increases with increasing salt concentration until it reaches its maximum in the fast coagulation mode (Chudnovskii, 2002).

Stabilization of colloidal systems can be achieved through various mechanisms such as charge stabilization or steric stabilization. For blue molybdenum-vanadium salts, stability is likely mediated by a combination of electrostatic and steric forces, which can be adjusted by the presence of certain ions or surfactants (Fei, et al, 2014). The critical concentration of coagulation is influenced by several factors, including the concentration and distribution of colloidal components, the salt content, and the dielectric properties of the medium. However, the stability of these sols is very sensitive to changes in ionic strength, pH, and other environmental factors. Understanding the coagulation threshold — the minimum concentration of electrolyte required for rapid aggregation of colloidal particles — is important for monitoring their stability and optimizing their use (Navyashree, et al, 2018)

Blue molybdenum-vanadium sols are colloidal dispersions formed by reducing the ions of molybdate and vanadate. These sols exhibit unique optical and catalytic properties, which makes them interesting for materials science and chemistry. However, their stability is crucial for practical applications, as coagulation or aggregation can lead to loss of functionality. Understanding the coagulation thresholds under different conditions provides insight into the factors affecting sol stability and allows precise control of their behavior.

Preparation of sols. Blue molybdenum-vanadium salts were synthesized by reducing

a solution of potassium nitrite and calcium nitrate. Analytical KNO_3 and KCl were used as electrolytes to study coagulation thresholds. Before conducting an experiment on the effect of electrolytes on Mo-V blue, sol was synthesized and salt solutions with a volume of 100 ml, a concentration of 0.3 mol/l KNO_3, 0.2 mol/l $Ca(NO_3)_2$ were prepared and electrolyte solutions with a volume of 50 ml, a concentration of 2 mol/l of KNO_3 and KCl. To do this, the mass of salt was determined by the formula:

$$m = n \cdot M$$

g – where m is the mass of the substance,

M – is the molar mass of the substance,

n – is the amount of the substance. From the equation it is necessary to find the amount of substance n according to the formula:

$$n = C \cdot V$$

where C – is the concentration of the solution,

V – is the volume of the solution.

The pH of the sol was determined before the experiment. Water was prepared with the same pH solution as the sol for dilution. To determine the pH of solutions, the device must be calibrated with a buffer solution with pH = 4.01 and 6.86. Before each pH meter measurement, the device was rinsed with distilled water so that the device would get rid of salt residue from previous measurements. Next, first release the electrode into a buffer solution with a pH of 4.01, then into a buffer solution with a pH = 6.86. After calibrating the device, the pH of the molybdenum-vanadium blue sol medium can be measured. Next, hydrochloric acid 2 M is needed to prepare acidified water.

To do this, immerse the electrode in distilled water, turn on the agitator, and gradually add hydrochloric acid to prepare the acidified water, then dilute the sol to the desired concentrations. The sol concentration is selected experimentally in such a way that when the absorption spectra are obtained, there is no shift of the maximum for Mo-V to blue (738 nm) (Jiwen, et al. 2015).

Materials and Methods. Based on data from previous studies, it is known that the MoV Salt has insufficient charge. Based on this, it was necessary to select suitable electrolytes for the experiment. K^+, Na^+, and Ca^{2+} were selected from the proposed series of cations. Thus, as electrolytes It was assumed that sol with a dispersed phase concentration of 0.035% undergoes coagulation at an electrolyte concentration of less than 0.3 mol/l. For the initial study of the coagulation threshold, the following electrolytes were selected: KNO_3 with a concentration of 0.3 mol/l and $Ca(NO_3)_2$ with a concentration of 0.2.

The volume of sol with a concentration of 0.035 % content is 10 ml. The proportions of acidified water and electrolyte used in the experiment are shown in Table 1.

Table 1. Compositions of sol samples for determining coagulation thresholds

№	1	2	3	4	5	6	7	8	9	10
Volume of water (acidified), ml	10,0	9,0	8,5	8,0	7,5	7,0	6,5	6,0	5,5	5,0
Volume of electrolytes, ml	0	1,0	1,5	2,0	2,5	3,0	3,5	4	4,5	5,0

10 test tubes are filled with 10 ml of dilute sol with a concentration of sol and water in the volume indicated in the table. The electrolyte is injected into each tube 2 minutes before measuring the absorption spectrum. This experiment did not yield results, so it was decided to investigate the kinetics of coagulation and change the concentrations of electrolytes and sol. Thus, KCl and KNO_3 were chosen as electrolytes, since Ca^{2+} cations are prone to precipitating with molybdates and vanadates. The parameters for determining the kinetic dependence are presented in Table 2.

Table 2. Concentrations of coagulation reagents

Parameter	Meaning
$C_{mass.\%}$ Sol Mo-V blue	0,17
C (KNO_3), mol/l	0,8
C (KCl), mol/l	0,8

In this experiment, optical density was measured using a spectrophotometer over time with minimal confidence (502 nm). Based on the data obtained, the velocity constants were calculated using the Smolukhovsky equation (Liu, et al. 2015).

Table 3. Compositions of sol samples for determining coagulation thresholds for this experiment

Tube Number №	1	2	3	4	5	6	7	8	9	10
Volume of water (acidification), ml	9	8,8	8,5	8,0	7,5	7,0	6,5	6,0	5,5	5,0
The volume of the el-ta, ml	0	0,2	0,5	1,0	1,5	2,0	2,5	3,0	3,5	4,0

For the experiment, the samples were prepared as follows: 0.5 ml of concentrated sol and water were added to 10 test tubes in the volume indicated in Table 3. The total volume of the solution in each tube was 9.5 ml. The concentrations and compositions of the electrolytes were matched in accordance with the experimental provisions.

In this experimental work, an electrolyte and acidified water were introduced into samples with molybdenum vanadium blues at a constant concentration. After mixing all the reagents, it is necessary to wait 10 minutes for KCl, 15 minutes for KNO_3. Immediately after the expiration of the time, the optical density was measured at a minimum of absorption for molybdenum-vanadium blues.

Results and Discussion. When using electrolytes of $Ca(NO_3)_2$ with a concentration of 0.2 M and KNO_3 with a concentration of 0.3 M, the data presented in Figures 1-4 were obtained.

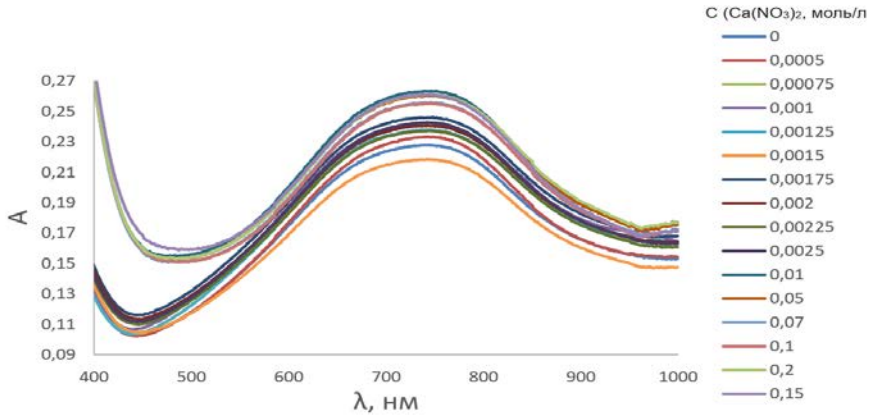

Figure 1. Effect of $Ca(NO_3)_2$ electrolyte concentration on the absorption spectra of MoV blue

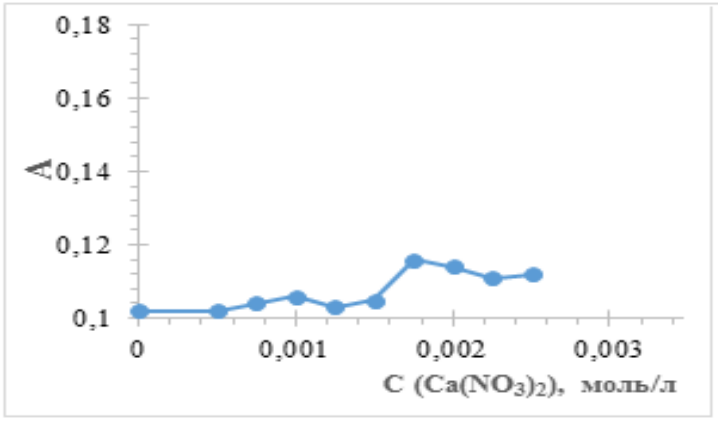

Figure 2. Dependence of the change in optical density (502 nm) on the concentration of electrolyte $Ca(NO_3)_2$

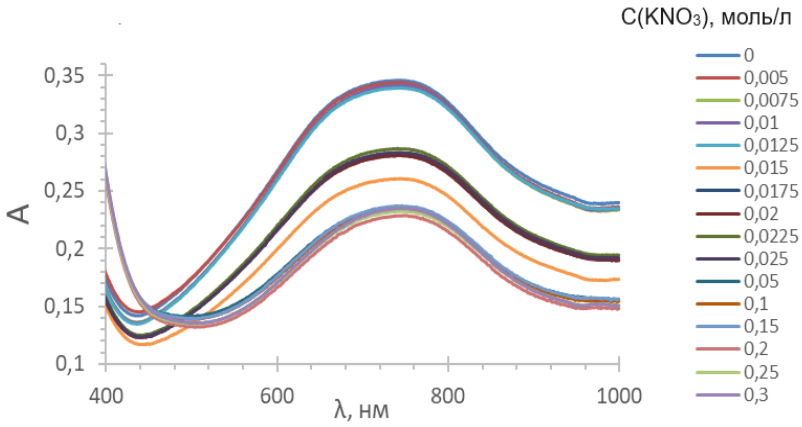

Figure 3. The influence of the concentration of electrolyte KNO_3 on the absorption spectrum of Mo-V blue

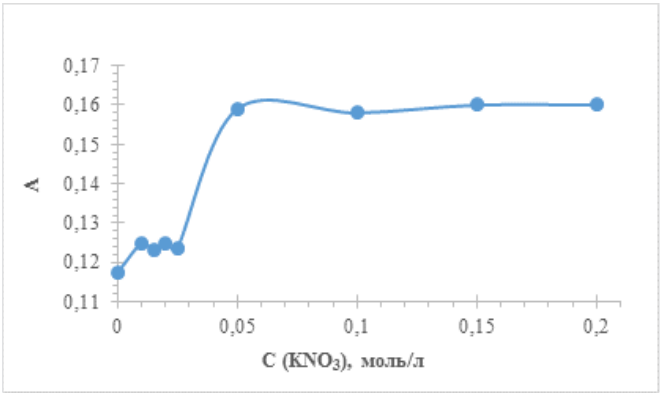

Figure 4. Dependence of the change in optical density in the region of the absorption minimum (502 nm) on the concentration of electrolyte KNO_3

According to the obtained results, it is possible to notice that the coagulation of the sol does not occur at low concentrations of electrolytes. Coagulation does not occur in the first case (Figure 1,2) because calcium nitrate does not affect the coagulation of the Mo-V sol, since a chemical reaction can occur during interaction and a precipitate of molybdate or calcium vanadate is formed. In the second case (Figure 3,4), it is possible to notice that some signs of coagulation are present in the form of changes in optical density, but since the difference in values is small, we will not consider these values. The reason for this is the low concentration of sol and electrolyte, as well as the stability of molybdenum-vanadium blue dispersions (Rafique, et al. 2020).

When choosing the concentrations of sol and electrolyte, the concentrations of C sol (wt.%) = 0.035% and C (KNO_3) = 0.3 M were initially selected. For the second experiment, the concentration of C sol (wt.%) = 0.17% and the concentration of electrolytes 2 M of potassium nitrate and potassium chloride were taken. Further, by studying the kinetic dependence, the constant and half-coagulation time of the sol with KNO_3 electrolyte were determined:

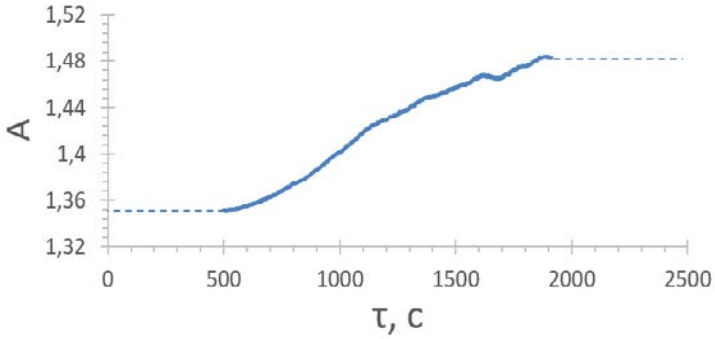

Figure 5. Kinetic dependence of the change in the absorption minimum of Mo-V blue (502 nm) with KNO_3

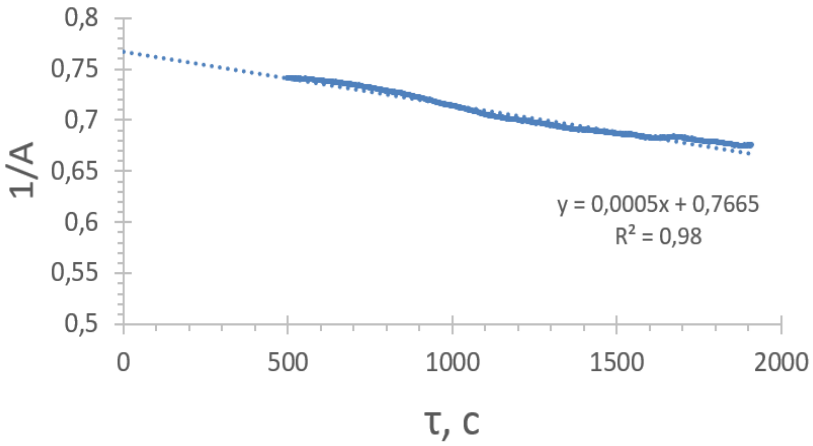

Figure 6. Linearization of the kinetic dependence Mo-V sinei with KNO_3

According to the graph, the constant of rapid coagulation and the time of half coagulation were determined: $k_b = 5 \cdot 10^{-4}$ m3/particle·s and Θ = 1563 s.

Then, after determining the time frame of the coagulation process, an experiment was conducted to determine the coagulation threshold. For this, absorption spectra of Mo-V blue with addition of different concentrations of KNO_3 were obtained (Figure 5-8).

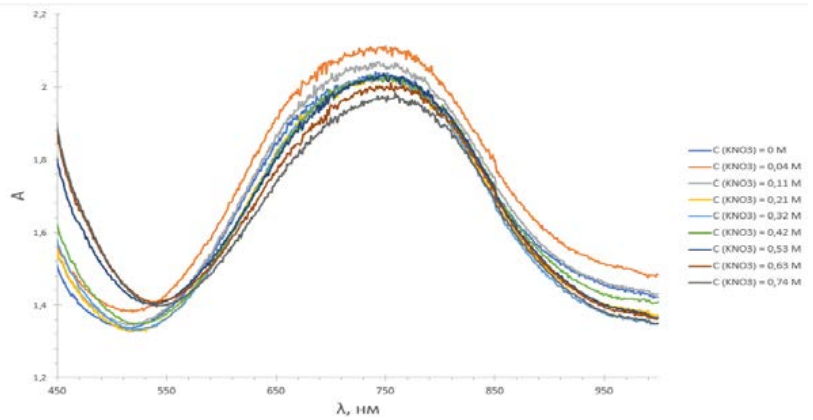

Figure 7. Changes in Mo-V blue absorption spectra when adding KNO_3

Coagulation threshold for sol Mo-V blue with electrolyte KNO_3 was $S_kKNO_3 = 0.53$ mol/l.

Next, the same experiment was conducted for KCl electrolyte, where the conditions of the experiment were identical. As can be seen from the data of coagulation kinetics (Figures 9, 10), the half-time of coagulation decreases.

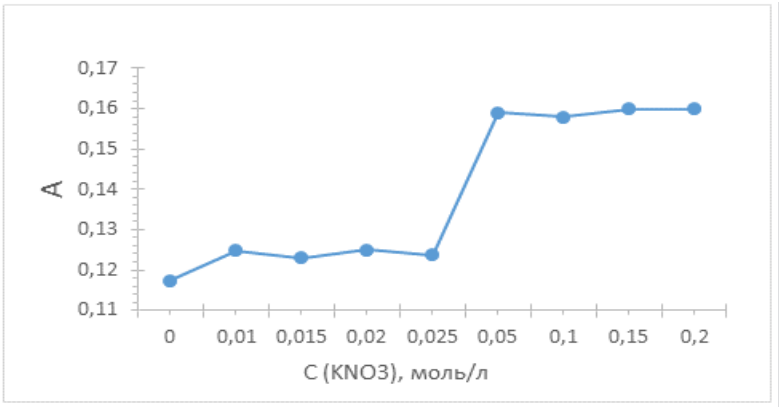

Figure 8. Dependence of optical density on Mo-V blue in the region of minimum absorption (502 nm) of fire concentration electrolyte (KNO_3)

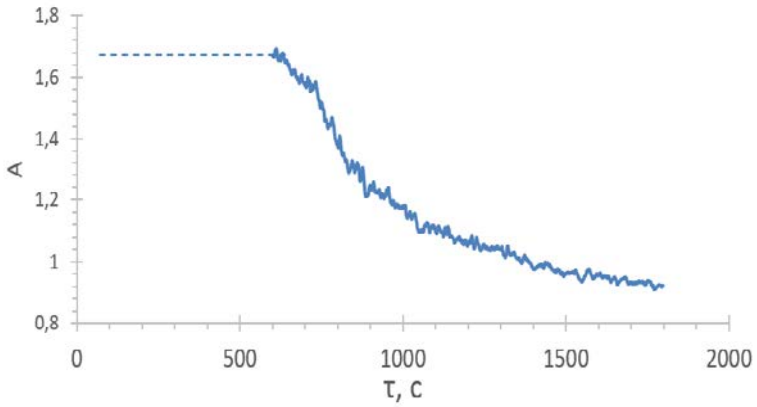

Figure 9. Kinetic dependence of the change in the absorption minimum of Mo-V blue (502 nm) with KCl

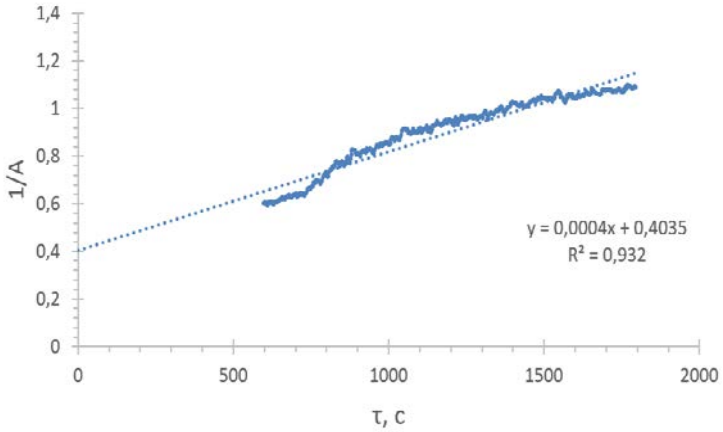

Figure 10. Linearization of the kinetic dependence for Mo-V blue with KCl

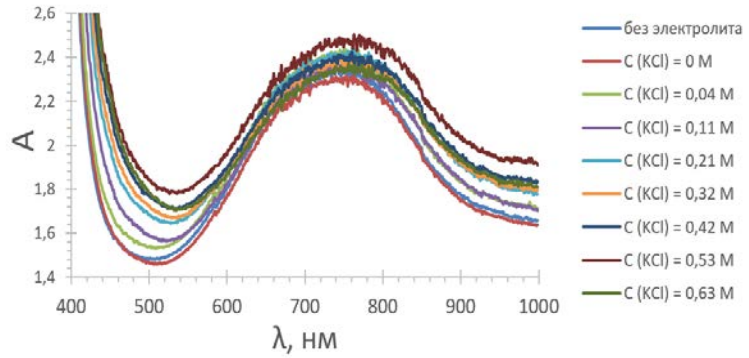

Figure 11. Changes in the absorption spectra of sols Mo-V blue with addition of KCl

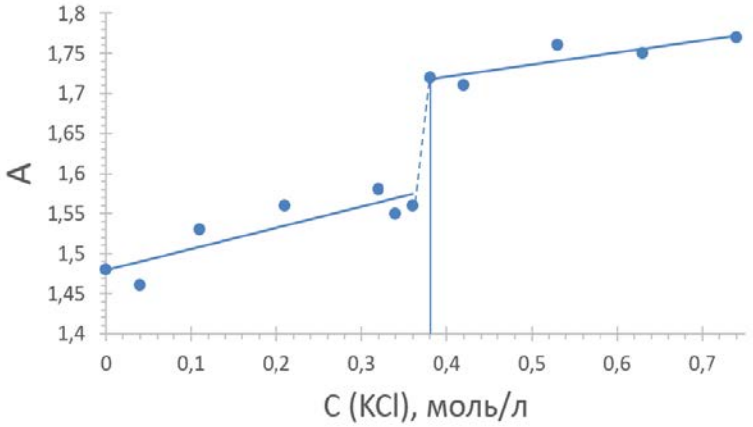

Figure 12. Dependence of the optical density of Mo-V blue in the region of the absorption minimum on the electrolyte concentration (KCl)

As a result of the linearization of the kinetic dependence, the following were obtained: the fast coagulation constant and the half-coagulation time for Mo-V blue using KCl as an electrolyte: $k_b = 4 \cdot 10^{-4}$ m^3/particle·s and $\Theta = 1000$ s.

Then, after determining the time frame of the coagulation process, an experiment was conducted to determine the coagulation threshold. For this, absorption spectra of Mo-V blue with addition of different concentrations of KCl were obtained (Figures 11, 12).

The coagulation threshold value for the studied electrolyte was 0.38 mol/l (Miras et al. 2012, Rand, et al. 2011, Toth, et al. 2019, Velmurugan, et al. 2019, Tan, et al. 2020).

Conclusion. Thus, in the course of work, a sol Mo-V blue ratio [Mo]:[V] = 90:10 was synthesized using ascorbic acid as a reducing agent. The low content of ash and electrolyte, especially in the case of stable systems, such as molybdenum-vanadium blue, had to initiate reduced coagulation. Chemical interaction of electrolytes with components of the sol (for example, the formation of precipitates) completely prevents coagulation (Камбалина, и др., 2014).

To achieve significant effects, high temperatures or changes in environmental conditions (pH, temperature) are necessary. For sol Mo-V, the constants of fast coagulation and half-time of coagulation were determined for KNO_3: $k_b = 5 \cdot 10^{-4}$ m^3/particle•s and Θ = 1563 s, for KCl: kb = $4 \cdot 10^{-4}$ m^3/particle·s and Θ = 1000 s. The coagulation thresholds for the Mo-V blue electrolytes with KNO_3 S_k = 0.52 mol/l and KCl Sk = 0.38 mol/l were also obtained. During the work, it was determined that the threshold of coagulation of the sol with the studied electrolytes is reached at a sol concentration of 0.17 mass% and at a concentration of electrolytes of at least 0.4 mol/l (Офицеров, и др., 2011, Савченко, и др., 2013).

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www:nauka-nanrk.kz
http://chemistry-technology.kz/index.php/en/arhiv
ISSN 2518-1491 (Online), ISSN 2224-5286 (Print)

Директор отдела издания научных журналов НАН РК *А. Ботанқызы*
Редакторы: *Д.С. Аленов, Ж.Ш. Әден*
Верстка на компьютере *Г.Д. Жадырановой*

Подписано в печать 26.03.2025.
Формат 60x88$^{1}/_{8}$. Бумага офсетная. Печать – ризограф.
13,5 п.л. Заказ 1.

Национальная академия наук РК
050010, Алматы, ул. Шевченко, 28, т. 272-13-19