

ТЕХНОЛОГІЯ ВИСОКОМОЛЕКУЛЯРНИХ СПОЛУК І КОМПОЗИЦІЙНИХ МАТЕРІАЛІВ

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RESEARCH OF PROCESSES OF CHEMICAL PREPOSITION OF NICKEL AS A FILLER OF POLYMER HYDROGELS

<https://doi.org/10.23939/ctas2025.01.163>

The kinetic characteristics of the reaction of nickel precipitation by hypophosphites in the volume of the solution were studied, depending on the pH of the medium, temperature, oxidant content, nature and concentration of the activator, and the presence of a water-soluble polymer – polyvinylpyrrolidone (PVP). It was found that the presence of PVP in the solution affects the duration of the induction period and the rate of the reduction reaction, and the nature of the effect of PVP significantly depends on the pH of the medium. It has been proven that the use of previously obtained nickel hydrosols as a reduction activator makes it possible to significantly reduce the duration of the induction period of the reduction reaction at low temperatures.

Keywords: nickel, reduction, chemical deposition, kinetics, polyvinylpyrrolidone, hydrogels.

Introduction

Metal-filled hydrogels exhibit specific properties that can change depending on the moisture content, pressure, temperature and pH of the environment, which opens up new possibilities for their use [1–4]. A fundamentally new method of obtaining composite metal-filled polymer hydrogel materials is obtaining metal particles at the stage of polymer synthesis. This approach in the creation of composite hydrogels is currently relevant, as it eliminates many preparatory and final technological stages of the process of obtaining composites. The method is appealing from both practical and scientific perspectives because it enables the simultaneous formation of nano- and microscale metal particles during the polymer matrix formation, ensures better and more uniform distribution of these particles, and allows for the production of a material with isotropic properties. This work is one of the stages in the development of nickel-filled hydrogels based on copolymers of 2-hydroxyethyl methacrylate (HEMA) and polyvinylpyrrolidone (PVP) by combining the processes of polymerization and chemical reduction of metal ions. However, the combination of reduction and polymerization processes is impossible without know-

ledge of their kinetic patterns. In addition, taking into account the high reactivity of HEMA/PVP compositions in the presence of metal salts of varying oxidation state [4] and the significantly longer duration of nickel ion reduction processes [6], it is necessary to reduce the duration of the induction period as much as possible and increase the speed of the metal chemical deposition reaction. Therefore, knowledge of the kinetic characteristics of this process is an urgent task, the solution of which will make it possible to optimize the technology of obtaining polymer metal composites by polymerization with the simultaneous reduction of metal ions and to change the technological parameters within predetermined limits.

The choice of nickel as a filler is due to the special interest in it on the part of researchers, since composites based on it are promising as new materials characterized by magnetic, electrically conductive and catalytic properties. Currently, there are enough methods of obtaining metal-containing polymer materials [7]. Thanks to the simple technology of reduction from salts, the method of producing polymer composites by immobilizing nickel particles through chemical deposition in the pores of the

polymer has gained wide application. As a polymer matrix, for example, polypropylen, polyacrylates, cellulose, PVP copolymers [8–10] are used. But metal deposition in porous matrices does not always give the desired result in terms of technology. First of all, this is the possibility of filling only film materials. Secondly, it is difficult to obtain a uniform distribution of filler particles in the volume of the composite and, accordingly, isotropic properties. These disadvantages can be avoided by combining the processes of polymer matrix formation and deposition of metal particles.

However, during the chemical deposition of nickel powder, several challenges arise that require resolution. Thus, depending on the conditions of the chemical reduction reactions, the metal is not always obtained in the form of a powder, which is especially important during the combination of the reduction and polymerization processes. The obtained product is not always a pure metal [11]. In the case of reduction with phosphinates, an alloy of nickel and phosphorus is obtained, the ratio of which depends on the conditions of the reduction process [12].

Experimental information on the chemical reduction of nickel ions mainly concerns the preparation of various types of coatings and, practically, there is no information on the kinetic patterns of the chemical deposition of the metal in the volume of the solution.

The conditions for obtaining powders, in addition to the speed of reduction, will undoubtedly affect the composition, structure, dispersion of the powder, and, therefore, the properties of the filled composite and the technological mode of its production. On the other hand, certain temperature conditions are necessary for the chemical deposition of metals, which can be achieved thanks to the heat released during the copolymerization of HEMA with PVP [13].

The aim of this study was to investigate the kinetic patterns and determine the optimal conditions for the processes of chemical deposition of nickel in the solution volume.

Materials and research methods

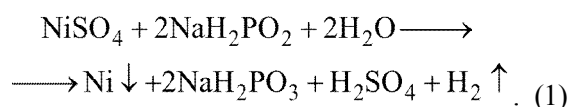
The reduction of nickel ions in an aqueous solution was carried out at temperatures ranging from 60 to 90 °C. Nickel sulfate ($\text{NiSO}_4 \cdot 7\text{H}_2\text{O}$) was used as an oxidizer, as a reducing agent –

sodium hypophosphite ($\text{NaH}_2\text{PO}_2 \cdot \text{H}_2\text{O}$). The redox system was obtained by adding nickel sulfate to water in a certain ratio (0,011–1,1 mol/L), sodium acetate (20 g/L) and sodium hypophosphite (0,024–2,4 mol/L). The pH value of the solution was adjusted with concentrated acetic acid and 25 % ammonia solution. To increase the activity of the reducing agent, a reduction activator was introduced into the redox system, in particular, a 2 % AgNO_3 solution. The kinetics of the chemical reduction reaction of nickel ions was evaluated using the volumetric method based on the volume of hydrogen released [14]. The kinetic patterns of the process were characterized by the duration of the induction period, the reaction rate, and the completion time, which were determined from the obtained curves of hydrogen evolution over time.

Results and discussion

Currently, nickel is reduced from aqueous solutions of its salts using hypophosphite, borohydride, boron-nitrogen reductants, and hydrazine [4].

The process of chemical deposition of nickel with hypophosphites is a redox reaction, the product of which is metallic nickel [4]:



It is known that the reduction of nickel ions with hypophosphites occurs intensively at temperatures of 90–95 °C in acidic or alkaline media [6]. However, at these temperatures, the rapid polymerization of HEMA/PVP compositions also takes place, which is an exothermic process. Due to the poor thermal conductivity of the polymer and the high rate of heat generation, thermal expansion and the development of internal stresses occur, leading to the formation of cracks within the sample [13]. The high polymerization rate and intense hydrogen evolution during the reduction of metal ions are the causes of material foaming or the formation of gas cavities (Fig. 1).

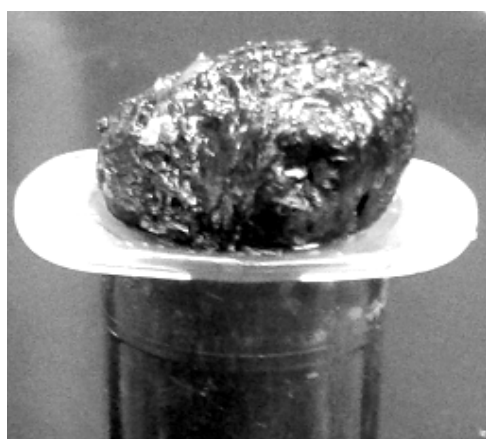
This is the reason why it is necessary to create such conditions that the polymerization and reduction reactions take place at much lower temperatures.

When solving specific practical problems, various kinetic characteristics of the chemical reduction of metal ions are of interest. In our case, where the task is to obtain composite metal hydrogels by combining the processes of polymer formation and

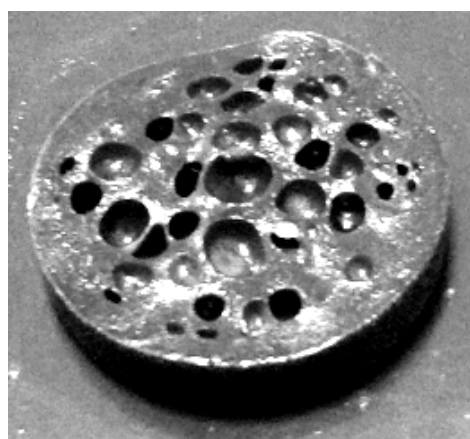
metal deposition, the important kinetic parameters of the reduction include the duration of the induction period, the reaction rate, and the time to completion. A significant factor is that during the process of chemical deposition of nickel, hydrogen is released in a stoichiometric ratio. Studies have shown that the cessation of hydrogen evolution is accompanied by a simultaneous loss of the color of the solution, which indicates the complete consumption of the oxidant (NiSO_4) and the completion of the reaction. Therefore, in this study, the kinetic parameters of the nickel ion reduction process were evaluated based on the volume of hydrogen released during the reaction. Of course, this method is not accurate for the quantitative determination of reduced nickel due to errors that arise during the experiments. However, parameters such as the duration of the induction period (τ_i , min), the reaction rate ($V_R^{\max}$, mmol/L·sec), and the time to completion (τ_{red} , min) can be determined with sufficiently high precision.

The induction period of the reduction reaction is the initial stage characterized by kinetic inhibition. During this period, stable solid-phase particles are formed in the solution, which subsequently exhibit autocatalytic effects. The duration of solid-phase particle formation, and thus the induction period, depends on numerous factors. In the case of combining the processes of polymerization and chemical metal deposition, it is important that the duration of the reduction induction period is shorter than the onset of the gel-effect stage of the polymerization reaction.

One of the conditions for carrying out a redox reaction to obtain a fine powder is its autocatalytic nature, which becomes possible under the introduction of activators into the system. A 2 % solution of AgNO_3 was used as an activator. As research results show, in the absence of an activator, the reaction either does not occur at all or proceeds at a very low rate (Table 1). With an increase in the activator content in the reaction medium, the reaction rate increases.



a



b

Fig. 1. The consequences of the high polymerization rate and metal ion reduction include:
a – foaming of the composition; b – formation of gas cavities

Table 1

Dependence of the kinetic parameters of the Ni^{2+} chemical reduction reaction on the content of the activator AgNO_3 ($T = 90^\circ\text{C}$; $\text{pH} = 4.5$; $[\text{NiSO}_4] = 0.55 \text{ mol/L}$)

No.	$[\text{AgNO}_3]$, g/L	$V_R^{\max}$, mmol/L·sec	τ_i , min	τ_{red} , min
1	0	0.001	-	-
2	0.25	0.390	31.1	152
3	0.50	0.524	25.2	101
4	1.00	0.627	20.4	76
5	1.25	0.660	19.1	70

$V_R^{\max}$ – the maximum rate of the Ni^{2+} reduction reaction, τ_i – the duration of the induction period, τ_{red} – the duration of the reduction process.

It is also worth noting a significant decrease in the induction period of the reaction and the duration of the reduction process. Opposite results were obtained in an alkaline environment. At an activator concentration of up to 0.25 g/L, the reduction process proceeds at a high rate.

With an increase in the concentration of the activator, metal is not formed; instead, a chemical reaction occurs without hydrogen release, resulting in the formation of a brown precipitate. However, unlike in an acidic environment, the use of a reduction activator in an alkaline medium allows the reaction to proceed at 60–70 °C. The results presented in Table 2 show that as the pH increases, the reaction rate increases, while the induction period and reaction completion time decrease. The increased reduction rate in an alkaline medium compared to an acidic one can be explained by the different phosphorus content in the surface layer of metal particles, which naturally leads to a decrease in the catalytic activity of Ni particles and, consequently, a slower growth rate.

According to the literature data [6], the lowest phosphorus content in the reduction product is characteristic of solutions with pH = 4.5–6.5 and pH > 10. During polymerization, it is generally not possible to maintain the pH of the medium within the required range. In this regard, for conducting the reduction reaction in acidic conditions, an initial pH of 4.0–4.5 was selected, while in alkaline conditions,

an initial pH of 7.0–7.5 was chosen, with the expectation of a pH decrease during the reaction.

The primary requirement for the composition of solutions used in the production of nickel coatings is to ensure a minimal reduction rate of metal ions and to prevent the formation of metal particles in the solution volume. At the same time, the concentration of the metal salt is relatively low, ranging from 0.08 to 0.13 mol/L [6]. Such an oxidant concentration in small volumes is insufficient to obtain a composite with the required metallic filler content and, consequently, the necessary properties. During metal deposition at the polymerization stage, it is not possible to adjust the concentration of the oxidant and reducer. Therefore, the content of the reduced metal in the polymer can only be regulated by the initial amounts of oxidant and reducer introduced into the starting composition.

As shown by the results presented in Fig. 2, an increase in the concentration of nickel ions in the reaction medium leads to a higher reduction rate and a shorter induction period. This pattern is observed in both acidic and alkaline environments. However, changes in the nickel salt concentration within the range of 0.0–0.05 mol/L have a negligible effect on the process rate.

It is well known that an increase in temperature leads to a higher reaction rate in most chemical processes. Therefore, it is logical to investigate how temperature affects the kinetic parameters of the reduction process in the selected redox system (Table 3).

Table 2

Dependence of the kinetic parameters of the chemical reduction reaction of Ni^{2+} on the solution pH
($T = 90\text{ }^{\circ}\text{C}$; $[\text{AgNO}_3] = 0.25\text{ g/L}$; $[\text{NiSO}_4] = 0.55\text{ mol/L}$)

No.	pH	$V_R^{\max}$, mmol/L·sec	τ_i , min	τ_{red} , min
1	4.5	0.038	31.0	160
2	5.5	0.393	21.3	86
3	6.0	0.500	18.0	60
4	7.5	0.698	13.2	23

Table 3

Effect of temperature on the kinetic parameters of the reduction process
(Solution composition: $[\text{NiSO}_4] = 0.55\text{ mol/L}$; $[\text{NaH}_2\text{PO}_2] = 1.2\text{ mol/L}$; $[\text{AgNO}_3] = 0.25\text{ g/L}$)

No.	Temperature, °C	$V_R^{\max}$, mmol/L·sec		τ_i , min		τ_{red} , min	
		pH = 4.5	pH = 7.5	pH = 4.5	pH = 7.5	pH = 4.5	pH = 7.5
1	60	0.005	0.92	90	53	430	83
2	70	0.013	3.85	74	25	320	39
3	80	0.066	7.63	55	15	240	27
4	90	0.389	12.32	30	12	165	23

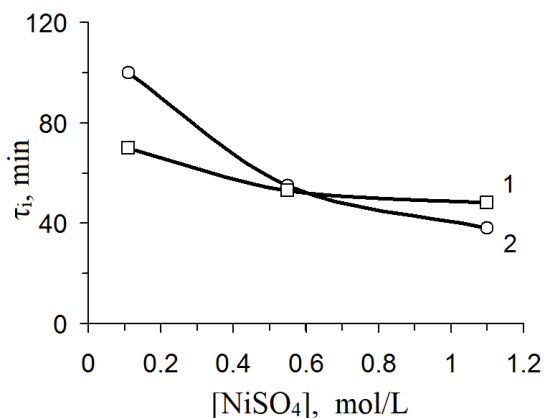
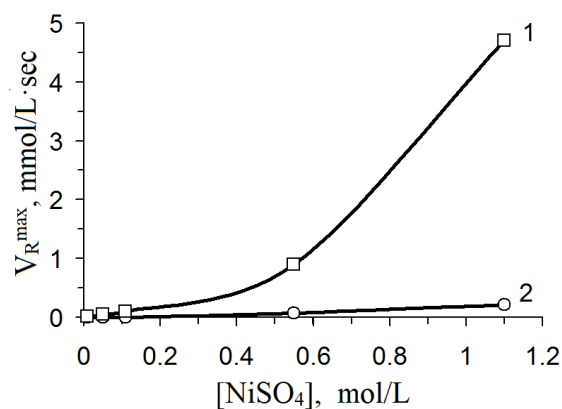


Fig. 2. Dependence of the maximum reduction rate of Ni^{2+} (a), the duration of the induction period (b), and the duration of Ni^{2+} reduction (c) on concentration.

NiSO_4 ($[\text{AgNO}_3] = 0.25 \text{ g/L}$):

1 – $T = 60^\circ\text{C}$, $\text{pH} = 7.5$;

2 – $T = 80^\circ\text{C}$, $\text{pH} = 4.5$

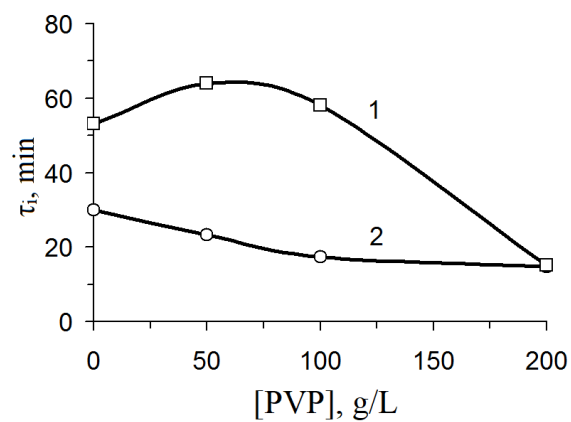
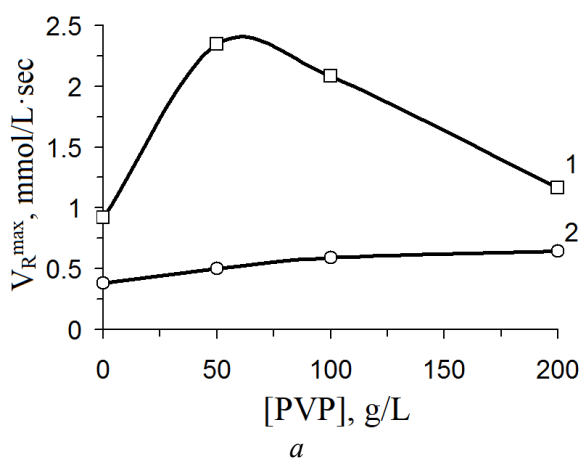
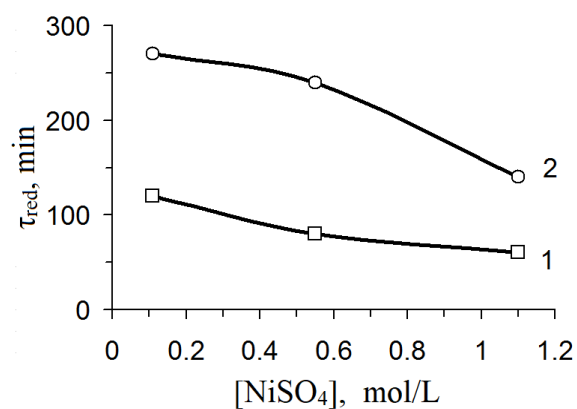
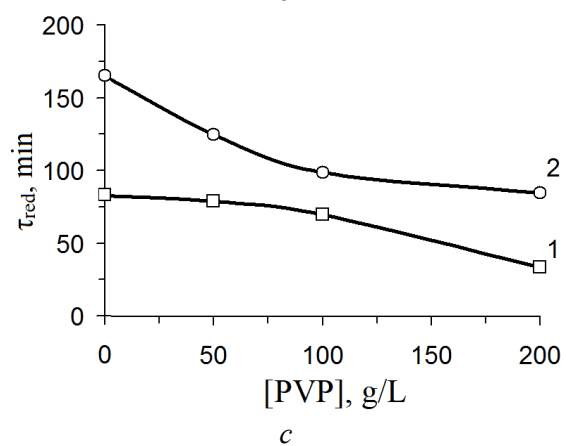


Fig. 3. Dependence of the maximum reduction rate of Ni^{2+} (a), the duration of the induction period (b), and the total reduction time of Ni^{2+} (c) on the PVP content in the redox system.

($[\text{NiSO}_4] = 0.55 \text{ mol/L}$; $[\text{AgNO}_3] = 0.25 \text{ g/L}$):

1 – $T = 60^\circ\text{C}$, $\text{pH} = 7.5$; 2 – $T = 90^\circ\text{C}$, $\text{pH} = 4.5$



It was found that in an alkaline medium, the effect of temperature on the kinetic parameters of the reduction process is more pronounced than in an acidic medium. Unlike in an alkaline environment, at 60 °C in an acidic medium, the reaction proceeds at a very slow rate, with a significantly longer induction period and overall reduction duration. At room temperature, nickel ion reduction does not occur from the studied solution composition, even under significantly increased oxidant concentration.

Currently, the literature provides sufficient experimental data on the study of metal reduction in the presence of water-soluble polymers, particularly PVP [15, 16]. In most cases, polyvinylpyrrolidone is used in small amounts as a stabilizer for metal particles. During the synthesis of composites based on pHEMA-gr-PVP copolymers, PVP serves as a component of the reaction mixture, participates in the copolymerization reaction, and is used in amounts of 10–50 parts by weight. Therefore, it was necessary to investigate how PVP, in quantities used for copolymerization, affects the kinetics of the reduction reaction. In both acidic and alkaline media, the introduction of PVP into the redox system has a minor effect on the reduction rate, although it shortens the induction period (Fig. 3).

In an alkaline medium, with a PVP concentration of 200 g/L in solution, the induction period lasts 14 minutes at a temperature of 60 °C. It is evident that due to its high complex-forming ability [17–19], PVP acts as an additional stabilizer in this case and promotes the formation of nucleation sites for reduction. Through complexation, nickel ions are attracted to and concentrated along the PVP chain, facilitating the formation of a greater number of active centers and preventing their aggregation. When studying the influence of various factors on the rate of the redox reaction, it was found that the process is always accompanied by an induction period – a stage in which metallic nickel particles with catalytic activity

are formed. During the synthesis of nickel powders in aqueous solutions, it was observed that the resulting powders exhibit a broad particle size distribution. The largest particles sediment within a few minutes. However, a fraction of metallic particles remains suspended in solution for 4–6 hours, indicating their nano- and ultradispersed size. It was therefore logical to investigate the possibility of activating the nickel deposition process using solutions containing nano- and ultradispersed metal particles – hydrosols – as reduction activators (Table 4).

Due to the use of pre-formed ultradispersed particles as an activator and changes in reduction conditions, the kinetic curve shifts significantly closer to the ordinate, the induction period decreases, and the reduction reaction proceeds at a high rate from the very beginning. In this case, the reaction spontaneously initiates on the surface of the introduced ultradispersed activator, and the subsequent increase in reaction rate is associated with the autocatalytic nature of the reduction process.

The obtained results made it possible to justify the optimal technological parameters for nickel powder production and, based on them, to determine the technological parameters for synthesizing composite metal hydrogels by combining polymerization and metal ion reduction processes. Using the established conditions, samples of nickel-filled composite materials based on pHEMA-gr-PVP copolymers were obtained. The presence of metallic nickel in the synthesized polymer samples is confirmed by their magnetic properties and coloration, which varies from gray to black depending on the composition, redox system content, and pH.

The main objectives of further research in this direction are to determine the influence of reduction conditions combined with polymerization on the particle size of the metallic filler, as well as the structure, physicomechanical, magnetic, and electrical properties of the obtained composite materials.

Table 4

Effect of temperature and activator type on the kinetic parameters of reduction
(Solution composition: $[\text{NiSO}_4] = 0.55 \text{ mol/L}$, $[\text{NaH}_2\text{PO}_2] = 1.2 \text{ mol/L}$; pH = 7.5)

No.	Activator	Temperature, °C	$V_R^{\max}$, mmol/L·sec	τ_i , min	τ_{red} , min
1	AgNO_3	60	0.92	53	83
2	Hydrosol Ni	60	0.18	21	136
3	Hydrosol Ni	70	0.50	13	106
4	Hydrosol Ni	80	1.29	7	52

Conclusions

Using the volumetric method, the kinetic characteristics of the reduction reaction of nickel ions with hypophosphites in solution were determined, depending on the solution composition and conditions. It was established that, in the absence of an activator, the reaction practically does not occur. A significant increase in the reduction rate is observed at nickel sulfate concentrations above 0.55 mol/L. In a slightly alkaline medium, the chemical deposition of metal is characterized by a high rate and a short induction period even at temperatures of 60–70 °C.

It was found that the presence of PVP in the solution affects both the duration of the induction period and the reduction reaction rate, with the nature of this influence strongly depending on the pH of the medium.

Thus, it was proven that the use of pre-formed nickel hydrosols as an activator significantly shortens the induction period of the reduction reaction at low temperatures.

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ДОСЛІДЖЕННЯ ПРОЦЕСІВ ХІМЧНОГО ОСАДЖЕННЯ НІКЕЛЮ ЯК НАПОВНЮВАЧА ПОЛІМЕРНИХ ГІДРОГЕЛІВ

Досліджено кінетичні характеристики реакції відновлення нікелю гіпофосфітами в об’ємі розчину залежно від рН середовища, температури, вмісту окисника, природи і концентрації активатора, присутності водорозчинного полімеру – полівінілпіролідону (ПВП). Виявлено, що присутність ПВП у розчині впливає на тривалість індукційного періоду та швидкість реакції відновлення, причому характер впливу ПВП значно залежить від рН середовища. Доведено, що використання як активатора відновлення попередньо одержаних гідрозолів нікелю дає можливість значно скоротити тривалість індукційного періоду реакції відновлення за низьких температур.

Ключові слова: нікель, відновлення, хімічне осадження, кінетика, полівінілпіролідон, гідрогелі.