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# DEVELOPMENT OF A PROCESS FOR THE PRODUCTION OF HARD SOLID NANOPHERS (FE-CO) FROM COMPLEX BLACK SLATE SOURCE

**Abstract.** The paper presents the results of developing a method for producing nanopowder ***(Fe-Co)*** in the form of a solid solution by precipitation from vanadium-containing raw materials of the hydrometallurgical plant Balausa Firm LLP. Solutions obtained by leaching black shale ores with an iron content of up to 4-6% to obtain iron-containing powders were studied. X-ray phase analysis has established that the initial iron content of 2 to 6% is sufficient for applying the scheme for extracting iron sulfate and obtaining powders based on it from solutions obtained by leaching black shale ores. By the results of electron microscopy analysis, was established that the size of crystallites in the resulting powders ***(Fe-Co)*** is 30-50 nm and that of their agglomerates is 100-200 nm.
***Key words:*** *black shale ores, vanadium-containing solutions, leaching, precipitation, iron-cobalt solid solution.*

## Күрделі қара сақтас шикізатынан қатты ерітімдердің (Fe-Co) наноұнтақтарын алу әдісін әзірлеу

**Аңдатпа.** Мақалада «Балауса Фирма» ЖШС гидрометаллургиялық зауытының құрамында ванадий бар шикізаттан тұндыру арқылы қатты ерітінді түріндегі наноұнтақ ***(Fe-Co)*** алу әдісін әзірлеу нәтижелері берілген. Құрамында темірі бар ұнтақтарды алу үшін құрамында темірі 4-6%-ға дейінгі қара тақтатас кендерін шаймалау арқылы алынған ерітінділер зерттелді. Рентгендік фазалық талдау темір сульфатын алу және оның негізіндегі ұнтақтарды қара тақтатас кендерін шаймалау арқылы алынған ерітінділерден алу схемасын қолдану үшін бастапқы темірдің 2-ден 6%-ға дейін жеткілікті екенін анықтады.Электрондық микроскопияның көмегімен алынған ұнтақтардың ***(Fe-Co)*** құрамындағы кристаллиттер мөлшері 30-50 нм, ал олардың агломераттары 100-200 нм болатыны анықталды.
***Түйінді сөздер:*** *қара тақтатас кендері, құрамында ванадий бар ерітінділер, шаймалау, тұндыру, темір-кобальт қатты ерітіндісі.*

## Разработка способа получения нанопорошков твердых растворов (Fe-Co) из комплексного черносланцевого сырья

**Аннотация.** В статье приведены результаты разработки способа получения нанопорошка ***(Fe-Co)*** в виде твердого раствора методом осаждения из ванадийсодержащего сырья гидрометаллургического завода ТОО «Фирма «Балауса». Исследовались растворы, полученные при выщелачивании черносланцевых руд с содержанием железа до 4-6% для получения железосодержащих порошков. Рентгенофазовым анализом установлено, что исходное содержание железа от 2 до 6% является достаточным для применения схемы извлечения сульфата железа и получения порошков на его основе из растворов, полученных при выщелачивании черносланцевых руд. Методом электронной микроскопии установлено, что размеры кристаллитов в полученных порошках ***(Fe-Co)*** составляют 30-50 нм и их агломератов 100-200 нм.
***Ключевые слова:*** *черносланцевые руды, ванадийсодержащие растворы, выщелачивание, осаждение, твердый раствор железо-кобальт.*

## Introduction

Over the past few years, there has been a rapid development of the metal products market around the world. The average annual growth rate of metal products production is about 10-12%. This is mainly due to the development of production in Asian countries, and, above all, in China. Simultaneously with the growth in consumption of rolled steel and other metallurgical products, the demand for special alloy steels and alloying elements, one of which is vanadium, has increased. About 87% of vanadium from total production is used in ferrous metallurgy as an effective alloying additive in the production of steels of various grades. The raw material from which vanadium is extracted is interesting and promising from the point of view of complex processing. The main raw material for the extraction of vanadium in Kazakhstan is black shale ores of complex composition and double resistance. It is promising to obtain iron-containing products from this type of raw material in the form of reduced iron and iron nanostructured powders [1].

One of the main methods of vanadium extraction from black shale ores is sintering [1]. High temperatures activate chemical reactions and convert vanadium into an easily soluble form. The optimum temperature for sintering with soda is about 900-1000 °C and with sodium chloride about 700-800 °C. In industrial applications of the process, the ratio of soda or sodium chloride to ore is an important parameter. The optimum ratio of soda to ore is about 1:1. If the amount of soda or sodium chloride is too high, insoluble compounds may be formed and the efficiency of the process may be reduced [2, 3].

Leaching methods are also widely used for vanadium recovery. Sulfuric acid can be used for vanadium leaching at high temperatures and long-time intervals. In a study [4] from black shale ores, vanadium recovery in solution was 85%. Scientists are working on improving vanadium leaching technologies using sulfuric acid. One of the new developments is the use of sulfuric acid and hydrogen peroxide [5]. In this experiment, the extraction of vanadium into solution was 92%. The use of sulfuric acid and sodium chloride is also practiced [6], according to the authors' data, because of vanadium leaching the extraction into solution amounted to 90%. In general, the use of concentrated sulfuric acid at high temperatures and longtime intervals can achieve high extraction of vanadium into solution. The technology of heap sulfuric acid leaching of black shale ores allows to extract vanadium from ores with low concentration and obtain a satisfactory degree of extraction. Work [7-9] provides data on the degree of extraction of vanadium under different experimental conditions, for example, at a concentration of sulfuric acid 2% extracted about 45% of vanadium. World data confirm the effectiveness of sulfuric acid leaching for the recovery of metals, including vanadium, from black shale ores.

One of the most common methods of sorptive extraction of vanadium from sulfate productive leaching solutions of black shale ore is based on the use of anionite [10]. The combination of extractants based on ketones and other extractants was also investigated. It was shown that at optimum values of pH and extraction duration it was possible to extract about 94% of vanadium without significant co-extraction of other metals. However, it is also of interest to obtain by-products of vanadium extraction, such products are the production of iron powders, because the solutions from leaching are rich in iron [9, 11].

The aim of the research is to obtain nanopowders of iron-cobalt solid solution ***(Fe-Co)*** from vanadium leaching solutions. Such powders can be used in the production of magnetic carriers, as permanent magnets, in the production of sensors, in medicine and biology (carriers of drugs for delivery to the desired target organ using a magnetic field, magnetic resonance imaging, etc.) and for magnetochemical separation of radioactive and hazardous waste from nuclear power.

**Materials and methods of research**

**Starting materials**

The objects of the study were representative samples of vanadium-containing raw materials of the hydrometallurgical plant «Firma «Balausa» LLP, which were analyzed by chemical, X-ray phase, X-ray spectral, mineralogical and thermogravimetric methods of analysis.

Chemical analysis showed that polymetallic vanadium bearing ores contain %: $V_2O_5$ – 0,89; $MoO_3$ – 0,036; $U_3O_8$ – 0,018; Rare-earth elements – 0,069; $TiO_2$ – 0,250; $WO_3$ – 0,036; $Al_2O_3$ – 3,79; $Fe_2O_3$ – 2,07; $K_2O$ – 1,18; $CaO$ – 0,383; $SiO_2$ – 72,54; $Na_2O$ – 0.0732; $P_2O_5$ –1,150, $C$ – 17,891.

The initial raw material for production of iron-containing powders will be solutions obtained during leaching of black shale ores with iron content up to 6%. The composition of black shale ore leaching solutions is as follows, g/dm$^3$: $Fe^{2+}$ – 1,6; $Fe^{3+}$– 6,4; $Al$ – 9,05; $V_{gen}$ – 2,78; $Mo$ – 0,044; $U$ – 0,019; Rare-earth elements – 0,075; $K$ – 2,4, all elements are in solution as sulfates.

**Research Methods**

Iron sulfate was isolated from the solution by the method of chemical precipitation, then iron sulfate was prepared in the form of $FeSO_4$-$7H_2O$. This compound is well soluble in water. To obtain a solid solution of nanosized powder ***(Fe-Co)***, the following is carried out: a solution of salts of iron sulfate heptahydrate and cobalt chloride hexahydrate was prepared, then heated and precipitated with alkali metals in the form of iron and cobalt hydroxides with continuous stirring, during stirring precipitation was carried out by introducing 22-28 g of dry sodium hydroxide into the solution of metal salts, after which 22-42 ml of a solution of 67 wt. % hydrazine hydrate and cobalt hydroxide was added to the solution. % hydrazine hydrate and incubated for 3-18 minutes. Iron sulfate and cobalt chloride hydrates were used as iron and cobalt salts. Before precipitation, the solution of iron and cobalt salts was heated to 82-92 °C. Solutions of iron and cobalt salts were prepared in the following ratio, wt. %: iron 72-33, cobalt 32-72. The technique offers the possibility of obtaining nanosized monophase powder of ***Fe-Co*** solid solution by reduction of metal salts from aqueous solutions under certain conditions. This technique is fast and hardware uncomplicated without additional input of equipment in the technological process, while highly energy-efficient process. Electron-microscopic studies were carried out on the analyzer JEOL ISM – 25S 3, + 70-100 microns, magnification x1500.

X-ray phase and X-ray diffraction analysis was performed on (DRON-3 machine, Broker Advance D8).

Thermal analysis was performed on an analyzer combined with a mass spectrometer NETZSCH STA 449 PC/PG QMS.

**Results and their discussion**

The method for producing the powder ***(Fe-Co)*** included preparing a stock solution containing 2.9873 g of iron sulfate, 5.6550 g of $CoCl_2$ hexahydrate and 75 mL of $H_2O$. This was followed by stirring with a mechanical stirrer (30-35 rpm). The prepared solution of ***Fe*** and ***Co*** salts was heated in a desiccator to 80 °C. Then 25 g of dry sodium hydroxide was added under constant stirring to precipitate ***Fe*** and ***Co*** metal hydroxides. After stirring for 10-15 sec, 25 mL of hydrazine hydrate solution was added to the mixture, having previously

*Table 1*

***Preparation results and dispersibility of iron-cobalt nanoscale powder***

*Кесте 1*

***Наноөлшемді темір-кобальт ұнтағын өндіру және дисперстілігінің нәтижелері***

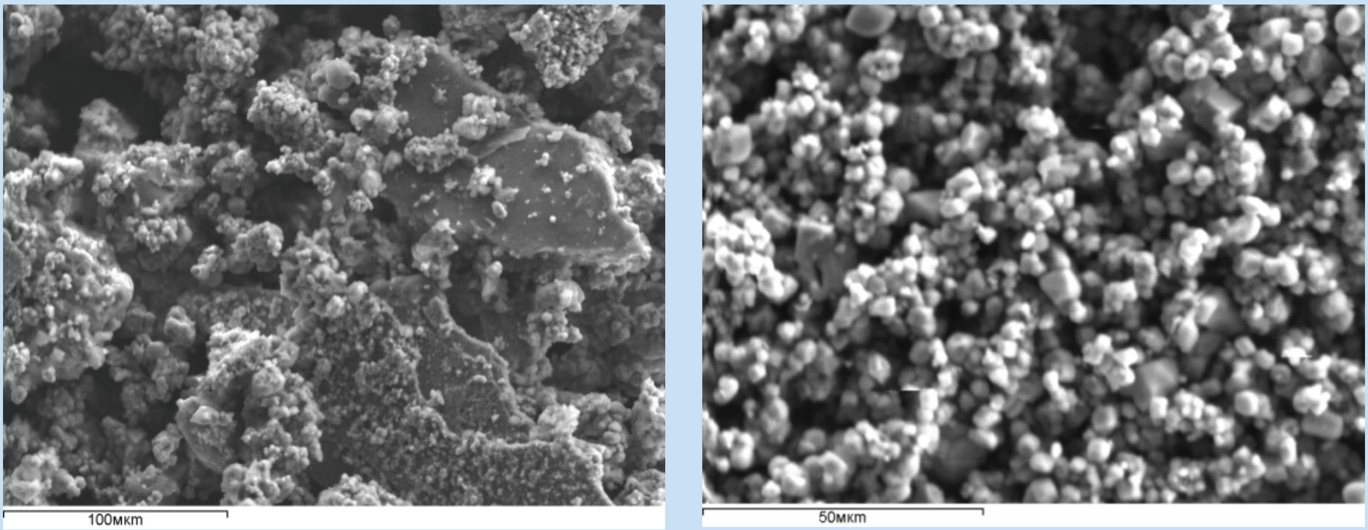
*Таблица 1*

***Результаты получения и дисперсность наноразмерного порошка железо-кобальт***

| № | Composition of nano-powder, mass. % | Starting salts, y | | Reagent 1 | Reagent 2 | Reagent 3 | T Recovery, °C | Dispersibility, nm |
|---|---|---|---|---|---|---|---|---|
| | | m1 $FeSO_4$* $7H_2O$ | m2 $CoCl_2$* $6H_2O$ | $H_2O$, ml | $NaOH$, g | $N_2H_4$, ml | | |
| 1 | ***30Fe-70Co*** | 2,9988 | 5,6451 | 75 | 25 | 25 | 80 | 30-50 |
| 2 | ***30Fe-70Co*** | 2,9988 | 5,6451 | 90 | 25 | 50 | 80 | 30-50 |
| 3 | ***40Fe-60Co*** | 3,9885 | 4,8752 | 90 | 20 | 20 | 80 | 30-50 |
| 4 | ***40Fe-60Co*** | 3,9885 | 4,8752 | 60 | 25 | 40 | 80 | 30-50 |
| 5 | ***50Fe-50Co*** | 4,9785 | 4,0296 | 80 | 25 | 20 | 80 | 30-50 |
| 6 | ***50Fe-50Co*** | 4,9785 | 4,0296 | 60 | 20 | 40 | 80 | 30-50 |
| 7 | ***60Fe-40Co*** | 5,9823 | 3,2136 | 85 | 15 | 15 | 80 | 30-50 |
| 8 | ***60Fe-40Co*** | 5,9823 | 3,213 | 80 | 25 | 20 | 80 | 30-50 |
| 9 | ***70Fe-30Co*** | 6,9703 | 2,4163 | 50 | 35 | 25 | 90 | 30-50 |
| 10 | ***70Fe-30Co*** | 6,9703 | 2,4163 | 85 | 20 | 15 | 90 | 30-50 |

stopped heating the reaction mixture. Under constant stirring for 10 min throughout the volume, the formation of iron-cobalt nanodispersed particles was recorded. After settling the obtained mixture was filtered. The resulting black precipitate was washed with distilled water and then isopropyl alcohol to remove the volatile by-products of the reaction. Next, the nanosized powder ***(Fe-Co)*** was dried under low vacuum (10-2 mmHg) at 40-50 degrees Celsius for one hour. The storage of powders was carried out in a bouquet without access to air, which was placed in an exicator with $P_2O_5$. It was found that in the volume of the mixture uniformly formed nanodispersed particles of ***Fe*** and ***Co***, the formation of which was recorded visually. Such uniform formation of nanoparticles in the volume of the obtained mixture stimulates the reduction of dispersibility of ***Fe-Co*** powders. The results of the studies of the proposed method for obtaining solid solutions of nanosized iron-cobalt powder in the concentration range ***Fe(100-x) Co(x)***, where x is from 30 to 70%, are introduced in Table 1.

The sizes of nanoparticles were determined by electron microscopic spectroscopy (JEOL JSM 6390 scanning electron microscope). It was determined that in the whole range of compositions of ***Fe-Co*** nanosized powders the particle sizes vary very slightly: the size of crystallites 30-50 nm and their agglomerates 100-200 nm, Figure 1.




*a) 100 mkm* *b) 50 mkm*

**Figure 1. Results of electron microscopic spectroscopy of *Fe-Co* powder.**
**Сурет 1. *Fe-Co* ұнтағының электронды микроскопиялық спектроскопиясының нәтижелері.**
**Рис. 1. Результаты электронно-микроскопической спектроскопии порошка *Fe-Co.***

## Conclusions

The method of obtaining nanopowder ***(Fe-Co)*** in the form of solid solution by precipitation method from vanadium-containing raw materials – vanadium hydrometallurgical solutions has been developed. The method of iron sulfate precipitation and the sequence of operations for obtaining iron sulfate heptahydrate and cobalt chloride hexahydrate to obtain iron-cobalt nanopowder in the form of solid solution were developed. By the method of electron microscopy it was found that the size of crystallites in the obtained powders ***(Fe-Co)*** is 30-50 nm and their agglomerates 100-200 nm**.**

By the method of X-ray phase and X-ray structural analysis it is established that in the mixture of cobalt content 35-65 wt. % ***Co*** (iron 65-35 wt. % ***Fe***) compositions are monophase, the structure contains metallic phases. By the method of thermal analysis combined with QMS mass spectrometer it was established that the ***Fe-Co*** powder is a solid solution, the decomposition mechanism of which corresponds to the decomposition of the structure of similar compounds.

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