TECH SCIENCE

ISSN 3030-3702

TEXNIKA FANLARINING DOLZARB MASALALARI

TOPICAL ISSUES OF TECHNICAL SCIENCES

№ 5 (3) 2025

TECHSCIENCE.UZ

№ 5 (3)-2025

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TOPICAL ISSUES OF TECHNICAL SCIENCES

TOSHKENT-2025

OAK Ro'yxati

Mazkur jurnal O'zbekiston Respublikasi Oliy ta'lim, fan va innovatsiyalar vazirligi huzuridagi Oliy attestatsiya komissiyasi Rayosatining 2025-yil 8-maydagi 370-son qarori bilan texnika fanlari bo'yicha ilmiy darajalar yuzasidan dissertatsiyalar asosiy natijalarini chop etish tavsiya etilgan ilmiy nashrlar ro'yxatiga kiritilgan.

Muassislar: "SCIENCEPROBLEMS TEAM" mas'uliyati cheklangan jamiyati;
Jizzax politexnika insituti.

TECHSCIENCE.UZ- TEXNIKA FANLARINING DOLZARB MASALALARI elektron jurnali 15.09.2023-yilda 130343-sonli guvohnoma bilan davlat ro'yxatidan o'tkazilgan.

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Toshkent shahri, Yakkasaroy tumani, Kichik Beshyog'och ko'chasi, 70/10-uy.
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TEXNIKA FANLARINING DOLZARB MASALALARI
3-jild, 5-son (Avgust, 2025). -119 bet.

MUNDARIJA

TECH SCIENCE.UZ
Texnika fanlarining dolzarb masalalari
Topical Issues of Technical Sciences
2025-yil | 3-jild | 5-son
ISSN (onlayn): 3030-3702

INVESTIGATION OF THE PURIFICATION PROCESS OF PLATINUM POWDER FROM IMPURITIES

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Annotation. This article presents the results of research on the purification of platinum powder from impurities. The optimal technological parameters were determined based on the results of removing palladium and other non-ferrous metals from the powder by leaching. A new, cost-effective technology for the purification of platinum powder from impurities has been developed. The chemical composition of the purified powder is provided, and formic acid was selected as a reducing agent for palladium oxide prior to its subsequent dissolution in a nitric acid solution. The results of all the tests conducted are presented, the optimal technological parameters for the purification of platinum powder were determined and a new technological scheme for the recovery of platinum powder was developed.

Keywords: electrolyte, chemical properties, ammonium chloride, platinum, refining, reduction, palladium, precipitation, aqua regia, solution, filtration, washing..

PLATINA KUKUNINI QOʻSHIMCHALARDAN TOZALASHNING TADQIQOTLARI

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Annotatsiya. Ushbu maqolada platina kukunini qoʻshimchalardan tozalash boʻyicha tadqiqot natijalari keltirilgan. Palladiy va boshqa rangli metallarni eritish yoʻli bilan kukundan tozalash natijalariga asoslanib optimal texnologik parametrlar tanlandi. Platina kukunini qoʻshimchalardan tozalashning yangi, iqtisodiy jihatdan samarali texnologiyasi ishlab chiqildi. Tozalangan kukunning kimyoviy tarkibi keltirildi va palladiy oksidini azot kislotasi eritmasida keyinchalik eritish uchun formiat kislotasi qaytaruvchi vosita sifatida tanlandi. Oʻtkazilgan barcha sinov natijalari keltirilib, platina kukunini tozalash uchun optimal texnologik parametrlar tanlandi hamda platina kukunini ajratib olishning yangi texnologik sxemasi ishlab chiqildi.

Kalit soʻzlar: elektrolit, kimyoviy xossalar, ammoniy xlorid, platina, tozalash, qaytarish, palladiy, choʻktirish, shox arogʻi, eritma, filtrlash, yuvish.

DOI: https://doi.org/10.47390/ts-v3i5y2025N12

INTRODUCTION

Platinum in the composition of ore is present in the following minerals: Ptbi2 insisuits - occurs in the form of inclusions of the chalcopyrite and in fragments with acctic. Forms of grains are incorrect, dimensions up to 6 microns. Chemical composition, wt. %: Pt-28.0-29.91; Bi-62,96-65,1; Pd-2,16-4.8; Te-4.97; Sn-2.1 [1]. In Uzbekistan, a powerful mineral resource base was created, which is the basis for the development of the country's economy, which is today more than 1,800 deposits and about 1,000 promising manifestations of minerals, 118 species of mineral raw materials, of which 65 are developed [2].

The ores of the Kalmakyr deposit contain MPG in the redistribution from 0.03 to 0.5 g / t, the light platinum is dominated, platinum is present. Among the light platinoids prevails Pd. Sulphide minerals of the copper-molybdenum deposit serve as carriers of Pd. The highest content of Pd (3.5 g / t), Pt (0.7 g / t) and Os187 (3.2 g / t) are mounted in molybdenites [3].

According to the foreign raw material potential of the IPY testifies to the opposite - there are great opportunities to increase production of platinoids in South Africa and increasing palladium production in the United States and Canada (for 20-40 tons every year until 2024). This is a certain extent means that Russia can be fastened in the global MPG market, where the Russian Federation is a leading partner [4].

Currently, the demand for metals of the platinum group increases, therefore the purpose of this work is to develop a cost-effective technology of selective extract and cleaning platinum powder from impurities.

Experimental part. For the provision of experimental studies, dumping technological solutions of the gold and silver and silver of the copper smelter JSC Almalyk MMC were chosen as objects of research. The results of experimental tests were obtained based on the methods of photocolorimetry, potentiometry and IR Fourier spectroscopy, the results of atomic absorption, granulometric and x-ray phase analysis were given, technological testing was carried out according to the results of enlarged laboratory experiments and technological research, semi-industrial and experimental tests. Laboratory tests During the study, platinum content is determined mainly in the liquid phase. When platinum elements are in solution or may be translated into the solution, an atomic absorption method is used. Etode analysis method requires a simpler spectral and recording equipment than emission analysis. The optimal region of the measured concentrations of platinum metals (mkg / ml): platinum 10-100, palladium 15-100, Rhodium 10-200, Iridium 100-2000, Osmium 20-200. The error of the atomic-absorption method of determination in the optimal region of the measured concentrations is 1.0-0.2%, i.e. lower than in the case of emission analysis [5].

Platinum is well soluble in the "Tsarist Vodka" solution, in the initial electrolyte platinum is present as complex $H_2[PtCl_6]$[[6]. Based on the audible semi-industrial experiments, a new technology was developed to extract platinum from gold affinity solutions. The developed technology is from the following operations:

Platinium-diluted with sulfuric acid in the presence of thiourea

- Filtration of precipitate

Palladium and Palladium Filter

- Planning and drying platinum sediment
- Platinum precipitate

-When abrasion

- purity of the resulting powder from impurities

After calcination and abrasion, platinum powder was obtained by a platinum content in it 86.2%. According to the results of the tests, it was determined that the platinum integrated connection during the calcination was not fully decomposed. After that, several laboratory tests were carried out in order to determine the optimal temperature for the full decomposition of the platinum complex. Platinum precipitate was calcined at temperatures of 600-700-800-900-1000 ° with decomposition to elementary platinum.

Laboratory experiments on calcination were carried out in a muffle furnace at different temperatures. The calcination of the substance was performed gradually increasing the temperature, and in order to avoid losses, the tigli was covered with a lid. If the calcined substance contains organic components, at first with weak heating, the organic part was burned so that the flame was not formed. This operation was carried out in an open crucible, and after charging the substance was covered with a cap with a lid. The decomposition of the complex connection proceeds as follows:

$$([PtSC(NH_2)_2]SO_4) + O_2 \rightarrow Pt + SO_2 + CO_2 + N_2 + 2H_2O \qquad (1)$$

As a result of the calculated experiments, platinum powders of different colors and different masses were obtained. The results of analyzes for these powders are reduced to Table 1.

Table 1. Results of experiments on the decomposition of the Pt sediment

№	**T calcining, °C**	**Calcination time, h**	**Mass of the resulting powder (the initial mass of the sample 50 g)**	**Degree of decomposition Pt sediment,%**
1.	600	0,5	38,0	69,8

2.	650	0,5	36,6	72,5
3.	700	1,0	35,6	73,2
4.	750	1,0	35,2	75,5
5.	800	1,0	29,5	89,1
6.	850	1,2	29,8	90,0
7.	900	1,25	29,1	90,2
8.	950	1,25	26,7	99,99
9.	1000	1,25	26,8	99,99

Laboratory experiments have shown that high-temperature firing makes it possible to completely decompose platinum to a metal state with the formation of a purest powder. The calcination was carried out at different temperatures and with different time durations in the muffle furnace. The effect of temperature to the degree of decomposition of platinum complexes is shown in the following figure. (Fig. 1)

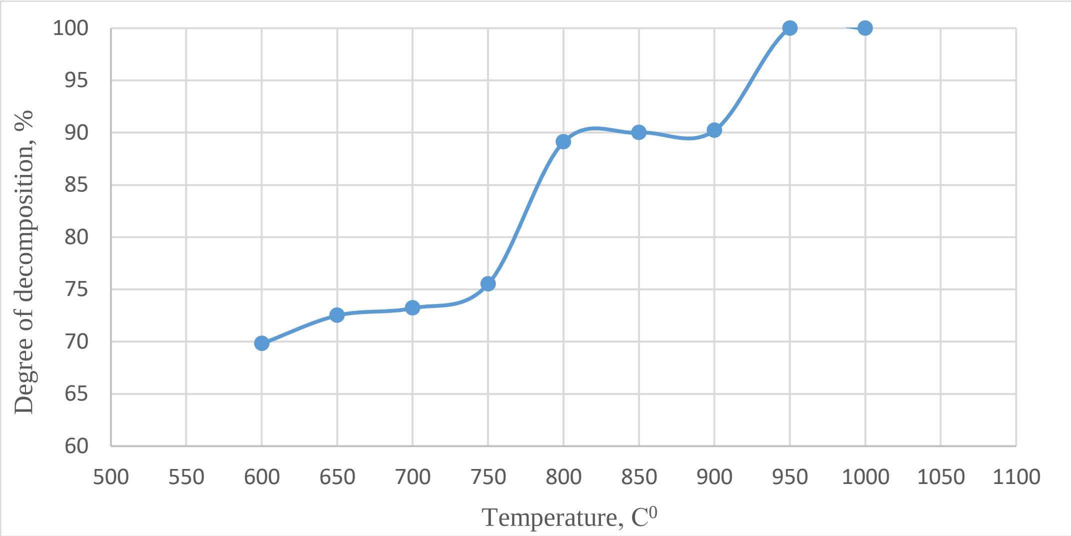

FIGURE 1. Dependence of the degree of decomposition of platinum complexes from temperature change

As a result of the calculated experiments, the planned result was achieved, the platinum comprehensive compound was completely decomposed. The obtained platinum after fixing powders are handed over to the analysis to determine the chemical composition. According to the results of spectral analysis, it was determined that, in the composition of the platinum powder there are 1-1.5% palladium and up to 0.1-1.0% of other non-ferrous metals.

Before starting work, a little about the physicochemical properties of Paldia. Palladium is the most chemically active from platinum metals. Does not react with water, diluted with acids, alkalis, ammonia solution. Reacts with hot concentrated sulfur and nitric acids, in contrast to other platinum metals. It can be translated into a solution by anodic dissolution in hydrochloric acid [7].

As it was above, palladium said in contrast to platinum is well dissolved in nitric and sulfuric acids, as well as most non-ferrous metals and their oxides that may be present in the composition of the platinum powder [8]. It is based on these properties that the technology for cleaning powder was developed by leaching. On the developed technology, several laboratory experiments were carried out. To clean the platinum powder made of paldia and other non-ferrous metals by leaching, sulfuric acid solutions of different concentrations were tested. Sulfuric acid solutions poured into heat-resistant glasses and warm up to 70 ° C on the electric stove. Then in each glass in the same amount (T: G = 1: 4) added a crude platinum powder. The results of the experiments are shown in Table number 2.

Table 2. Results of experiments on palladium leaching and non-ferrous metals from platinum powder

Observe number	Concentration H_2SO_4, %	Before leaching		After leaching	
		Pd content in powder,%	Σ The content of non-ferrous metals (CuFeNi and other) in powder,%	Pd content in powder,%	Σ The content of non-ferrous metals (CuFeNi and other) in powder,%
1.	20	1,31	0,63	1,24	0,26
2.	25	1,23	0,61	1,20	0,11
3.	30	1,25	0,58	1,10	0,05
4.	35	1,25	0,55	1,06	0,02
5.	40	1,28	0,74	1,05	0,01

Based on the results of the research results, it was revealed that the effects of sulfuric acid on non-ferrous metals and palladium are different, depending on the concentration. The effects of sulfuric acid on non-ferrous metals and palladium at different concentrations are shown in Figure 2.

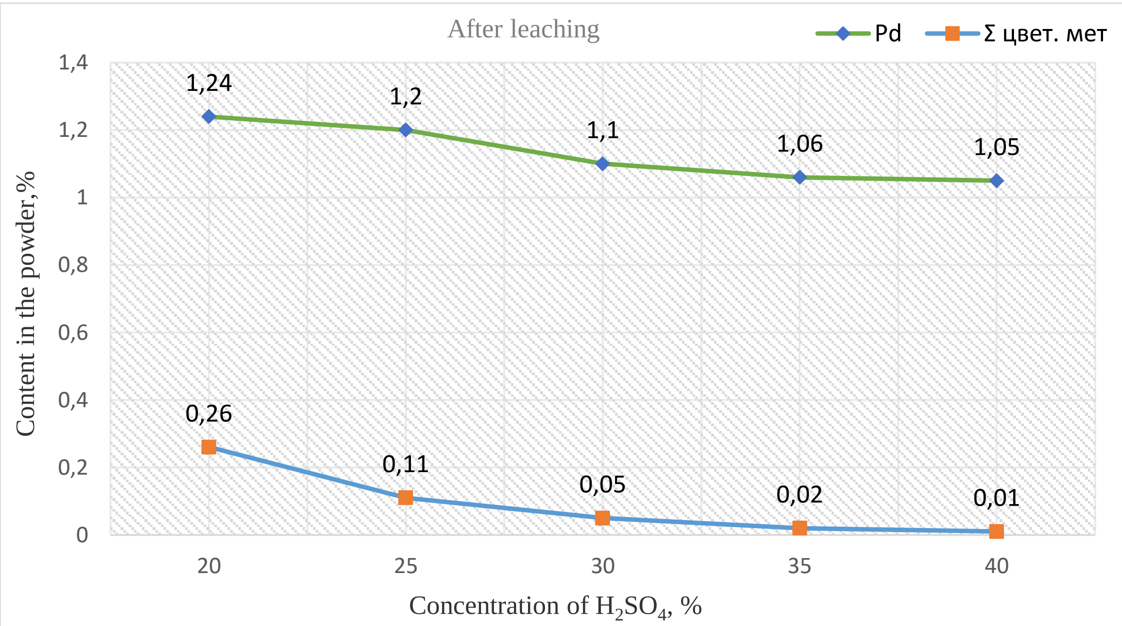

FIGURE 2. Impact of sulfuric acid for non-ferrous metals and palladium at different concentrations.

Experiments on the purification of platinum powder with a hydrometallurgical method with the same technological parameters were carried out, but the solutions of nitric acid of different concentrations were applied as a solvent.

Table 3. Results of experiments on leaching palladium and non-ferrous metals from platinum powder

Observe	Concentration	Before leaching	After leaching

number	H_2SO_4, %	Pd content in powder,%	Σ The content of non-ferrous metals (CuFeNi and other) in powder,%	Pd content in powder,%	Σ The content of non-ferrous metals (CuFeNi and other) in powder,%
1.	20	1,31	0,63	0,86	0,05
2.	25	1,23	0,61	0,80	0,04
3.	30	1,25	0,58	0,69	0,025
4.	35	1,25	0,55	0,61	0,014
5.	40	1,28	0,74	0,58	0,01

Normally, the total content of metals of the platinum group (PD, RH, IR, RU) in the composition of the paid powder should not exceed 0.05% and non-ferrous metals 0.025%. The effect of nitric acid on non-ferrous metals and palladium at different concentrations is shown in Figure 3.

The set of experiments on cleaning the platinum powder with a hydrometallurgical way was to no avail. As a result of the experiments, a sulfuric acid was used as a cleansing reagent, a platinum powder was purified from 12% palladium and from 96.4% non-ferrous metals. As a result of experiments, a nitric acid, platinum powder was purified from 51.2% palladium and from 98.4% of non-ferrous metals as a purifier reagent.

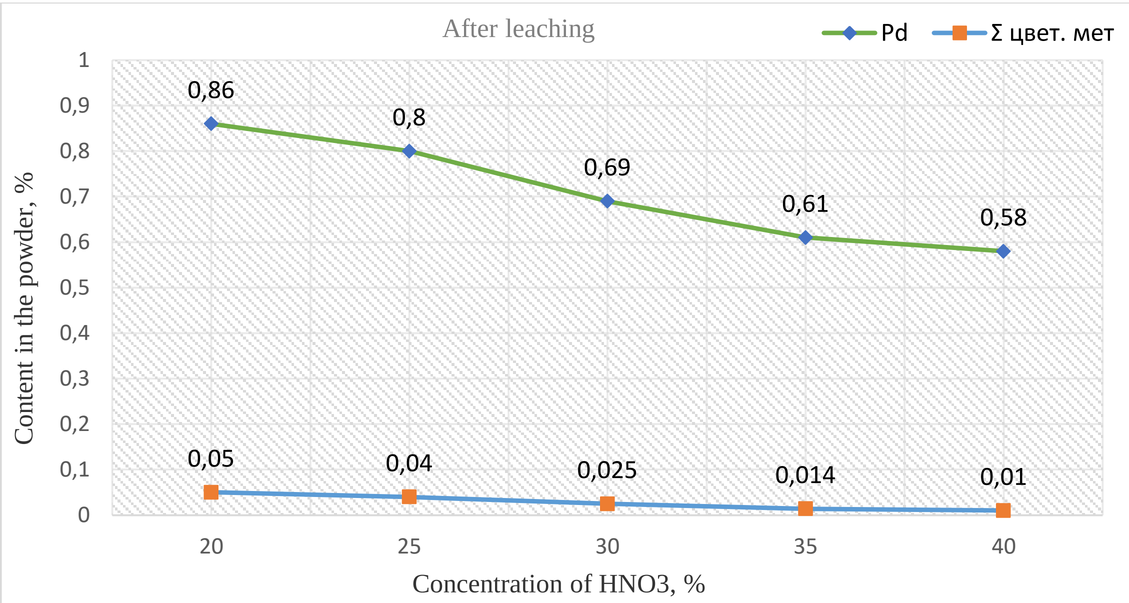

FIGURE 3. Impact of nitric acid for non-ferrous metals and palladium at different concentrations

After that, the phase analysis of the purified powder was carried out as a result of which it was revealed that the main part of the undisturbed palladium in the form of oxide. In order to modernize the powder cleaning technology, palladium oxidation parameters were studied. According to palladium powder, when heated in air is stable to ~ 300 ° C and above 850 ° C; In the range of 300 ... 850 ° C, it fills due to the formation on the surface of the PDO palladium oxide film [9].

Different reducing agents were used to restore palladium that oxidized during the calcination to the elementary state, since palladium oxide is not dissolved in inorganic acids even in royal vodka.

According to theoretical data, formic acid is chosen as a reducing agent. Formic acid, similar to aldehydes and, in contrast to other carboxylic acids, is easily oxidized by oxidizing agents [10].

To restore palladium oxide in the composition of the platinum powder, a 30% solution of formic acid was prepared. The heat-resistant glass poured a cooked solution of formic acid and heated to 50 °C on an electric stove. Next, in an equivalent number of portions, a platinum powder was added to a glass for an hour. The restoration of palladium oxide proceeds to the following reactions:

$$PdO+HCOOH=Pd+H_2O+CO_2 \qquad (2)$$

After the reduction process was washed with bi distilled water and dried platinum powder. Then there were still laboratory experiments on purifying powder by leaching by different nitric acid solutions. Analyzed duplicate powders in the same quantities taken to perform experiments. The data of the experiments are shown in Table No. 4.

Observe number	Concentration H_2SO_4, %	Before leaching			Observe number		
		Pd content in powder, %	Σ The content of non-ferrous metals (CuFeNi and other) in powder,%	Pt purity degree, %	Pd content in powder, %	ΣThe content of non-ferrous metals (CuFeNi and other) in powder,%	Pt purity degree, %
1.	20	1,31	0,63	98,2	0,17	0,05	99,74
2.	25	1,23	0,61	98,0	0,11	0,046	99,80
3.	30	1,25	0,58	98,11	0,08	0,04	99,84
4.	35	1,25	0,55	97,96	0,06	0,012	99,91
5.	40	1,28	0,74	98,08	0,06	0,01	99,92

According to the results of the experiments, it can be said that when cleaning the platinum powder it is important to know in the form of which compound contains impurities. The effect of nitric acid on the impurity is shown in Figures 4 and 5.

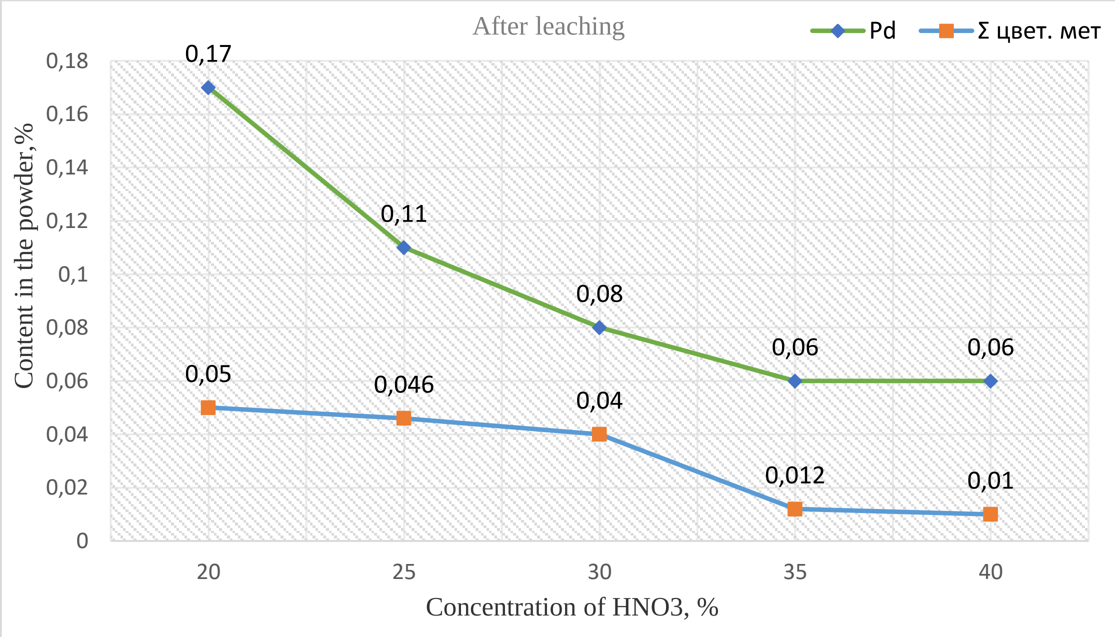

FIGURE 4. The dependence of impurities in powder from the concentration of nitric acid after washing

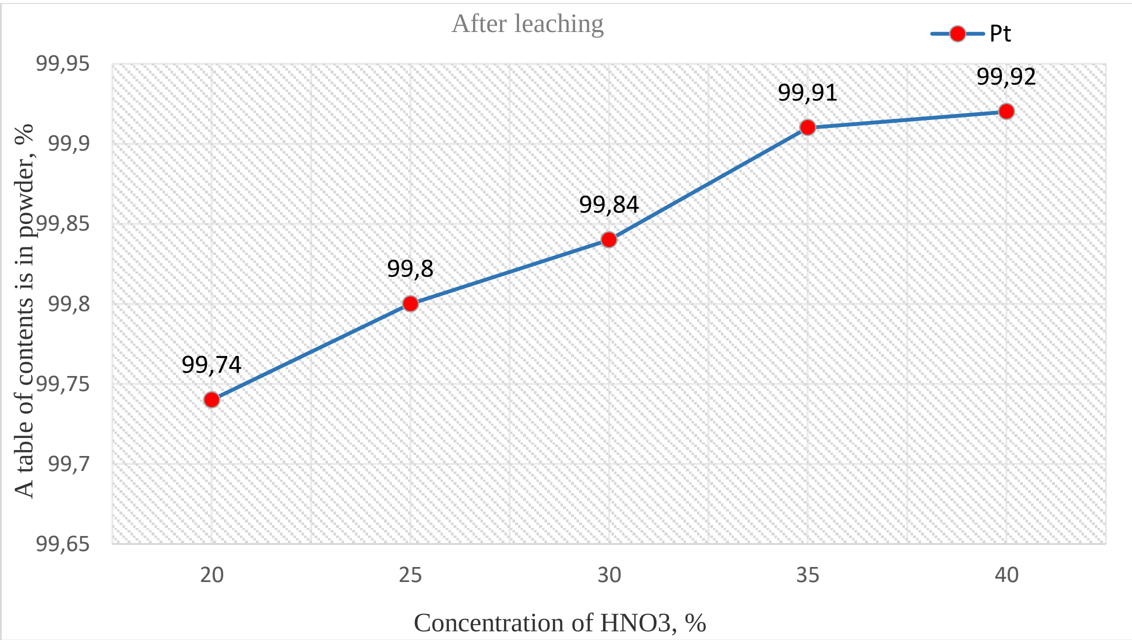

FIGURE 5. Dependence of the degree of purity of the concentration after washing

As a result of the study, a new, efficient, economically favorable and downtime technology on platinum powder affine was developed. Cleaning from impurities for this technology is carried out as follows. When obtaining platinum, selective precipitators and the obtained precipitation (NH_4Cl, ($[PtSC(NH_2)_2]SO_4$))are calcined at high temperatures [11]. After the calcination is obtained

The proposed technology can solve the following tasks:

1. Simplify the technology of cleaning platinum powder. This technology consists of 5 operations with a duration of cycles 4-6 hours.

2. The use of this technology allows saving reagents and other costs.

3. Preparation of platinum in the form of a powder with a mass fraction of platinum at least 99.90%.

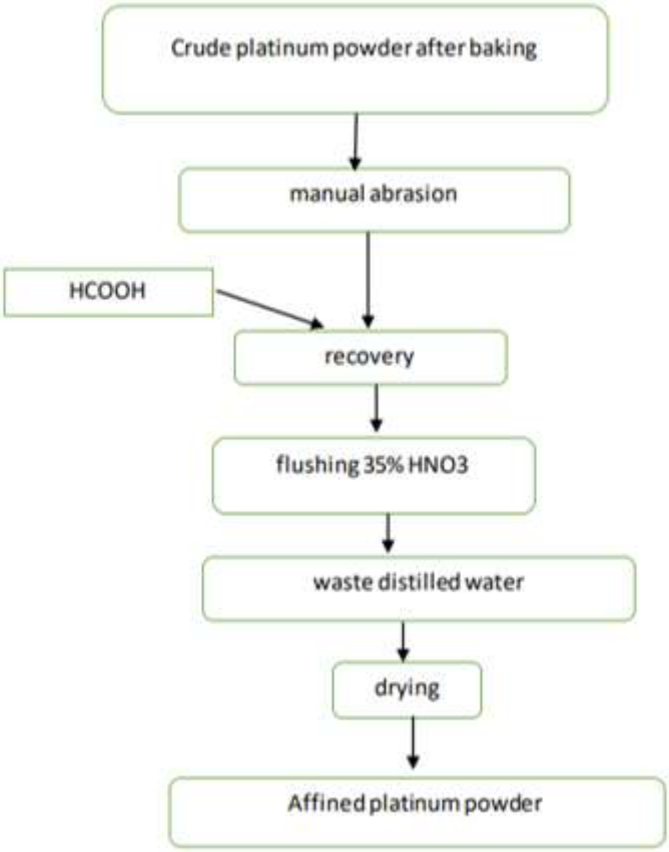

FIGURE 6. Technological scheme Cleaning platinum powder from impurities

CONCLUSION

Based on the results of the research results, it can be said that the recommended technology allows you to get a platinum powder of high purity and with significantly less energy costs. In global production, in order to purify from impurities, platinum powder is dissolved in royal vodka and precipitated and this cycle is repeated 2-3 times. In this technology, the operation was excluded the operation of dissolving platinum powder in the royal vodka.

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ISSN: 3030-3702 (Online)
САЙТ: https://techscience.uz

TECHSCIENCE.UZ

TEXNIKA FANLARINING DOLZARB MASALALARI

№ 5 (3)-2025

TOPICAL ISSUES OF TECHNICAL SCIENCES

TECHSCIENCE.UZ- TEXNIKA FANLARINING DOLZARB MASALALARI elektron jurnali 15.09.2023-yilda 130345-sonli guvohnoma bilan davlat roʻyxatidan oʻtkazilgan.
Muassislar: "SCIENCEPROBLEMS TEAM" mas'uliyati cheklangan jamiyati;
Jizzax politexnika insituti.

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