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EXTRACTION OF THE NICKEL MICROELEMENT FROM INDUSTRIAL SECONDARY PRODUCTS USING SULFURIC ACID

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Abstract: In this study, waste of hydrogenation catalysts of the "Pricat 9920" grade, used in the conversion of liquid vegetable oils into semi-solid products at IDEAL OIL JSC located in the Namangan region of the Republic of Uzbekistan, was utilized. To analyze the chemical composition of this hydrogenation catalyst waste, an average sample was collected and analyzed using a high-performance energy-dispersive X-ray fluorescence spectrometer equipped with a polarization system - Rigaku NEX CG EDXRF (Japan). In the territory of the Republic of Uzbekistan, Namangan region, sulfuric acid was used to extract nickel as a microelement from the spent hydrogenation catalyst of "IDEAL OIL" JSC. The effects of processing time, stirring rate using a magnetic stirrer, and the solid-to-liquid ratio of the waste with the solvent were systematically investigated. Taking all the above-mentioned parameters into account, it was determined that under the optimal technological conditions-20% sulfuric acid solution, temperature of 60 °C, solid-to-liquid ratio of 1:7, interaction time of 30 minutes, and a stirring speed of 800 rpm-it is possible to achieve a nickel recovery of 81.7% from the spent hydrogenation catalyst.

Keywords: microelements, sulfuric acid concentration, temperature, number of cycles, solid:liquid ratio, X-ray fluorescence spectrometer.

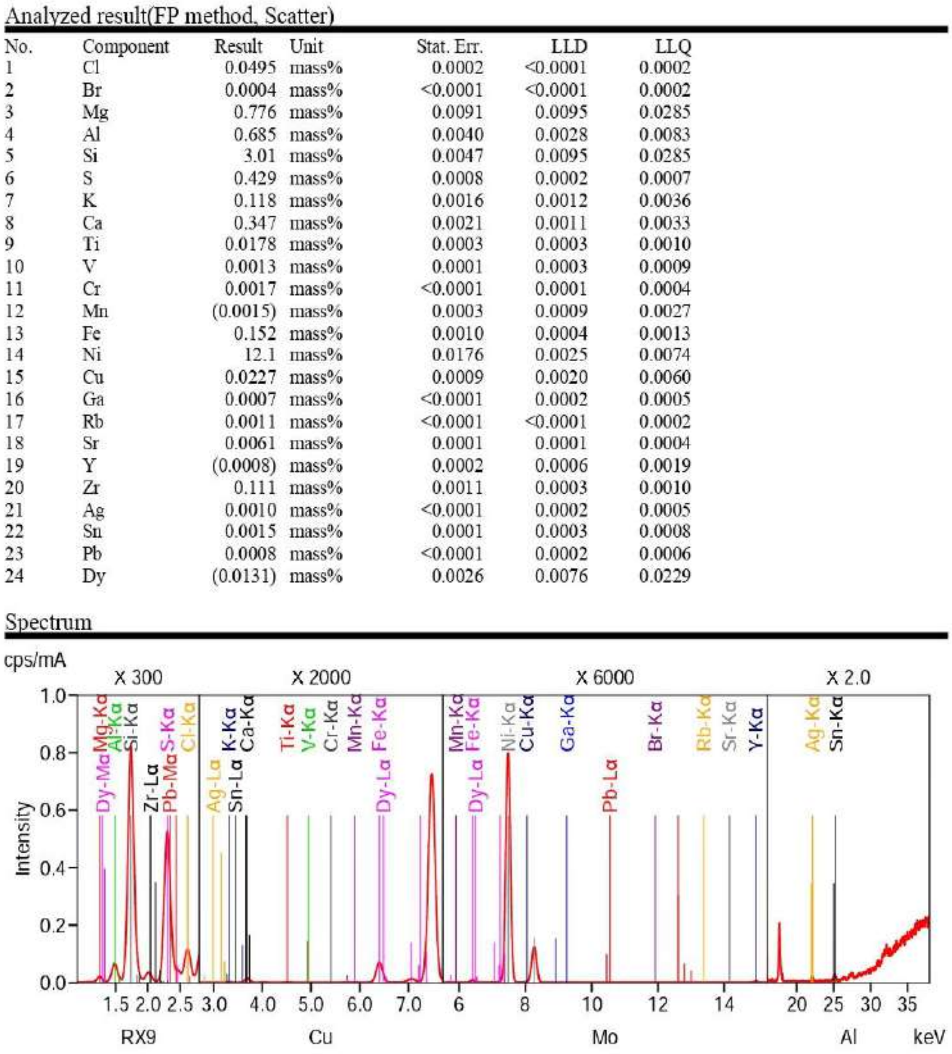
Introduction. With the increasing population on Earth, the demand of every individual for high-quality food to maintain a healthy and active life has become a persistent problem [1]. In this case, one of the most appropriate solutions is biofortification, which involves increasing the nutritional value of crops through the use of fertilizers [2]. Although traditionally used macroelement fertilizers positively affect crop growth, they cannot compensate for the role of microelements in performing essential life functions. The failure to replenish microelements in the soil, which are lost due to degradation or leaching, negatively affects not only plant life but also the health of humans and livestock that consume them [3]. It has been proven by scientists that a number of diseases in humans, such as anemia (iron, vitamin B12, and folate), anisocytosis (cell deformation, zinc and vitamin A), neutropenia and osteoporosis (copper and calcium), and hemolysis of red blood cells (selenium), arise as a result of microelement deficiency [4]. Insufficient intake of essential microelements such as selenium (Se), zinc (Zn), manganese (Mn), molybdenum (Mo), copper (Cu), boron (B), iron (Fe), nickel (Ni), and chlorine (Cl) leads to hidden hunger in the body [5].

Incorporating these microelements into traditionally used fertilizers and applying them as fertilizers provides solutions to a number of problems.

Extracting valuable microelements, which are preferable for use as fertilizers, from solid wastes that are secondary products of industry and production, and subsequently utilizing them, leads to an increase in the nutritional value of crops through fertilizers. At the same time, recycling wastes generated from commercial, industrial, agricultural, and mining activities eliminates the environmental hazards associated with them. Over the past few decades, the industrial sector has undergone rapid development, and the production process has begun to significantly demonstrate its negative impact on the environment. Failure to consider these generated products as recyclable secondary resources or their improper disposal leads to various problems. Unlike organic substances, solid wastes containing heavy metals do not undergo biological degradation; they can only change their oxidation state and may remain in a semi-decomposed state for many years [6]. Excessive accumulation of heavy metals and their salts in the soil negatively affects soil microorganisms [7], the plants growing there [8], and their functional activity [9]. Many heavy metals present at high concentrations are also potentially toxic to living organisms [10]. The use of industrial waste as a secondary resource mitigates its negative environmental impact, while utilizing the valuable microelements contained in it in appropriate amounts as fertilizer components makes a significant contribution to improving soil and crop productivity (the content of Ni microelement in soils for crop cultivation ranges from 3 to 1000 mg/kg-^{1} [11]) [12].

Various methods are used worldwide to extract such metals. In particular, Li and co-authors described two main methods for processing industrial solid wastes. The first method is direct leaching using acidic (such as H_2SO_4, HCl, and HNO_3) or alkaline (such as NaOH and Na_2CO_3) solutions [13, 14]. Jifeng Liao, Chunfa Liu Li, Jinxuy Li, Deshun Xu, Ye Zhong, and Bolar used an ammonium chloride solution, applying 2 M hydrochloric acid, 3 M ammonium chloride at specific molar ratios, and a liquid-to-solid ratio of 6:1 (ml/g) at 90°C for 90 minutes. As a result of their experiment, the amount of extracted nickel reached 87.7% [15]. A group of scientists including Luo, Zhai Mu, Huang, Xin, and Xu used low-temperature roasting, selective decomposition, and water leaching methods to extract nickel from low-grade nickel sulfide ores [16]. Scientists such as CW Nam, SP Barik, and KH Park developed a process for extracting Ni(II) from industrial solid wastes using sulfuric acid leaching depending on time, temperature, and H_2SO_4 concentration, followed by solution extraction, precipitation, and crystallization. In our study as well, the process is based on acid leaching (using different concentrations of H_2SO_4) of waste from the oil and fat industry followed by filtration, in which parameters such as time, temperature, and stirring rate were taken into account.

Materials and Methods. In this study, waste of hydrogenation catalysts of the “Pricat 9920” brand, used in the conversion of liquid vegetable oils into semi-solid products at “Uchqoʻrgʻon Yogʻ” JSC located in the Namangan region of the Republic of Uzbekistan, was utilized. To analyze the chemical composition of this hydrogenation catalyst waste, an average sample was collected and analyzed using a high-performance energy-dispersive X-ray fluorescence spectrometer - Rigaku NEX CG EDXRF (Japan), equipped with a polarization system. The obtained results are presented in Table 1 and Figure 1.



Analyzed result(FP method, Scatter)

No.	Component	Result	Unit	Stat. Err.	LLD	LLQ
1	Cl	0.0495	mass%	0.0002	<0.0001	0.0002
2	Br	0.0004	mass%	<0.0001	<0.0001	0.0002
3	Mg	0.776	mass%	0.0091	0.0095	0.0285
4	Al	0.685	mass%	0.0040	0.0028	0.0083
5	Si	3.01	mass%	0.0047	0.0095	0.0285
6	S	0.429	mass%	0.0008	0.0002	0.0007
7	K	0.118	mass%	0.0016	0.0012	0.0036
8	Ca	0.347	mass%	0.0021	0.0011	0.0033
9	Ti	0.0178	mass%	0.0003	0.0003	0.0010
10	V	0.0013	mass%	0.0001	0.0003	0.0009
11	Cr	0.0017	mass%	<0.0001	0.0001	0.0004
12	Mn	(0.0015)	mass%	0.0003	0.0009	0.0027
13	Fe	0.152	mass%	0.0010	0.0004	0.0013
14	Ni	12.1	mass%	0.0176	0.0025	0.0074
15	Cu	0.0227	mass%	0.0009	0.0020	0.0060
16	Ga	0.0007	mass%	<0.0001	0.0002	0.0005
17	Rb	0.0011	mass%	<0.0001	<0.0001	0.0002
18	Sr	0.0061	mass%	0.0001	0.0001	0.0004
19	Y	(0.0008)	mass%	0.0002	0.0006	0.0019
20	Zr	0.111	mass%	0.0011	0.0003	0.0010
21	Ag	0.0010	mass%	<0.0001	0.0002	0.0005
22	Sn	0.0015	mass%	0.0001	0.0003	0.0008
23	Pb	0.0008	mass%	<0.0001	0.0002	0.0006
24	Dy	(0.0131)	mass%	0.0026	0.0076	0.0229

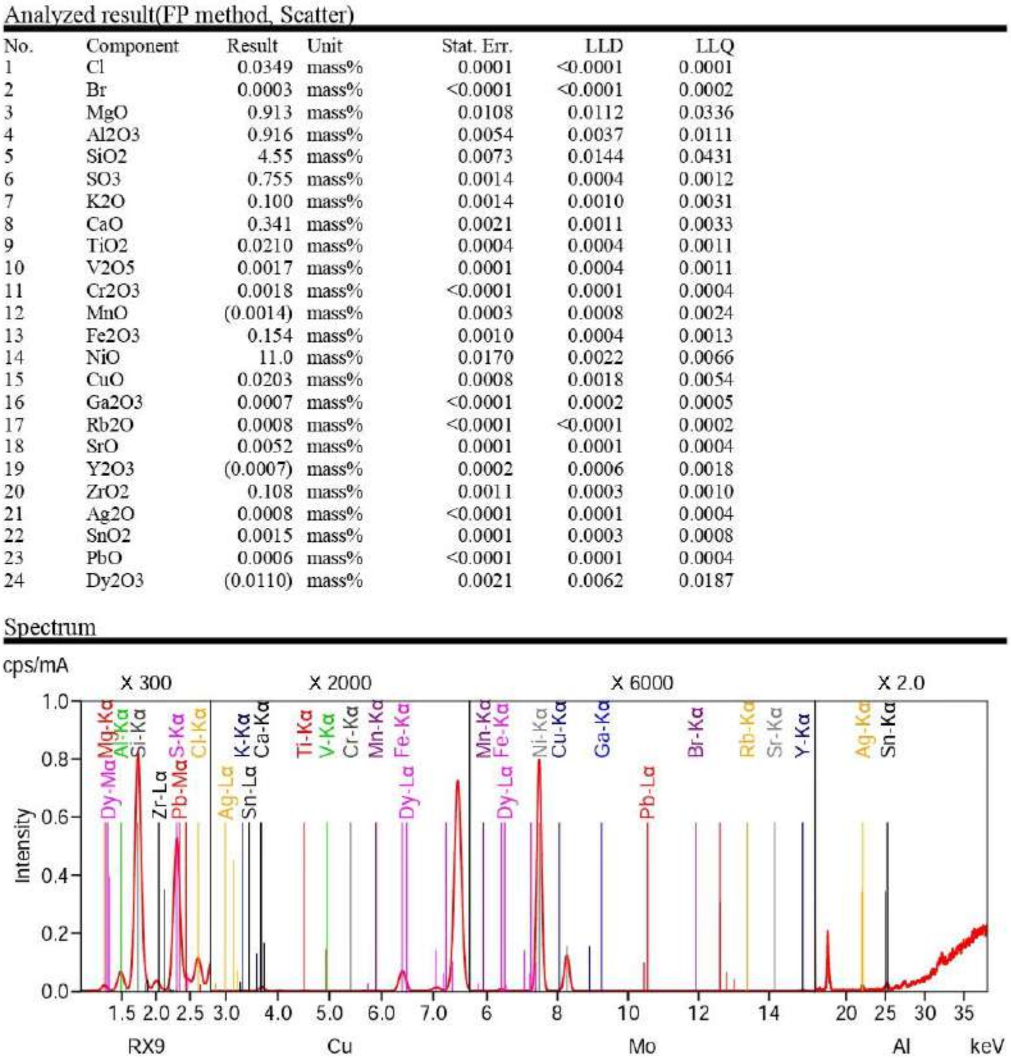
Spectrum

Figure 1. Elemental composition of spent hydrogenation catalyst

The data obtained show that the spent catalyst mainly contains nickel (12.1%) and silicon (3.01%). The analysis also noted the presence of aluminum, sulfur, and calcium in amounts well below the detection limit.

Table 1. Elemental chemical composition of hydrogenation catalyst waste from Uchkurgan Oil JSC.

№	Element	mass., %
1	Ni	12,1
2	Si	3,01
3	Al	0,68
4	S	0,42
5	Ca	0,34



Analyzed result(FP method, Scatter)

No.	Component	Result	Unit	Stat. Err.	LLD	LLQ
1	Cl	0.0349	mass%	0.0001	<0.0001	0.0001
2	Br	0.0003	mass%	<0.0001	<0.0001	0.0002
3	MgO	0.913	mass%	0.0108	0.0112	0.0336
4	Al2O3	0.916	mass%	0.0054	0.0037	0.0111
5	SiO2	4.55	mass%	0.0073	0.0144	0.0431
6	SO3	0.755	mass%	0.0014	0.0004	0.0012
7	K2O	0.100	mass%	0.0014	0.0010	0.0031
8	CaO	0.341	mass%	0.0021	0.0011	0.0033
9	TiO2	0.0210	mass%	0.0004	0.0004	0.0011
10	V2O5	0.0017	mass%	0.0001	0.0004	0.0011
11	Cr2O3	0.0018	mass%	<0.0001	0.0001	0.0004
12	MnO	(0.0014)	mass%	0.0003	0.0008	0.0024
13	Fe2O3	0.154	mass%	0.0010	0.0004	0.0013
14	NiO	11.0	mass%	0.0170	0.0022	0.0066
15	CuO	0.0203	mass%	0.0008	0.0018	0.0054
16	Ga2O3	0.0007	mass%	<0.0001	0.0002	0.0005
17	Rb2O	0.0008	mass%	<0.0001	<0.0001	0.0002
18	SrO	0.0052	mass%	0.0001	0.0001	0.0004
19	Y2O3	(0.0007)	mass%	0.0002	0.0006	0.0018
20	ZrO2	0.108	mass%	0.0011	0.0003	0.0010
21	Ag2O	0.0008	mass%	<0.0001	0.0001	0.0004
22	SnO2	0.0015	mass%	0.0001	0.0003	0.0008
23	PbO	0.0006	mass%	<0.0001	0.0001	0.0004
24	Dy2O3	(0.0110)	mass%	0.0021	0.0062	0.0187

Spectrum

Figure 2. Oxide composition of the spent hydrogenation catalyst

To extract the nickel microelement from the spent catalyst into the solution, waste with the following composition was used: NiO - 11.0%, SiO_2 - 4.55%, Al_2O_3 - 0.916%, SO_3 - 0.755%, CaO - 0.341%.

Figure 3. Ground state of the spent hydrogenation catalyst

The waste containing the spent catalyst was ground to a particle size of up to 500 microns and sieved to obtain the required fraction. During the experiment, studies were conducted to investigate the effects of technological parameters such as concentration, temperature, stirring rate, solid-to-liquid ratio, and mixing duration on the transfer of the nickel microelement from the waste into the solution.

Results. The effect of sulfuric acid concentration on the extraction of nickel from hydrogenation waste was studied under the conditions of a solid-to-liquid ratio of 1:3, temperature of 30 °C, mixing duration of 15 minutes, and a stirring rate of n = 500 rpm (Figure 4).

As the concentration of sulfuric acid increased from 3% to 20%, an increase in the degree of nickel extraction into the solution was observed. At sulfuric acid concentrations of 3, 5, 10, 15, and 20%, the nickel extraction degree increased to 10.6, 11.3, 13.5, 16.1, and 19.7%, respectively. However, with a further increase in sulfuric acid concentration from 25% to 30%, the degree of nickel transfer into the solution decreased from 17.5% to 16.2%. This can be explained by the fact that, with increasing sulfuric acid concentration, other elements present in very small amounts in the waste become activated, which leads to a lower extraction of nickel into the solution.

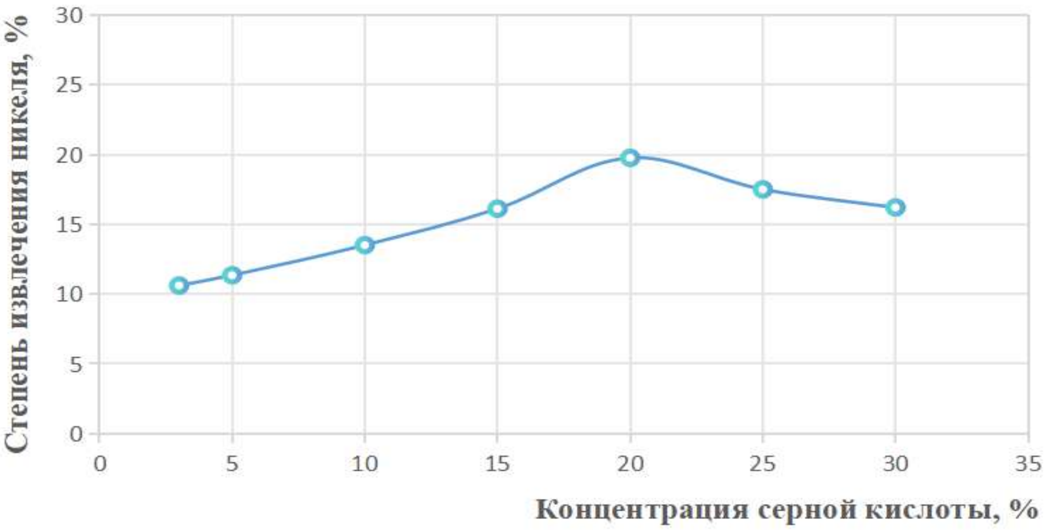


Figure 4. Effect of sulfuric acid concentration on the degree of nickel extraction (S:L = 1:3, temperature 30 °C, n = 500 rpm, τ = 15 min)

In subsequent studies, experiments were carried out using a sulfuric acid concentration of 20% under technological conditions of S:L=1:3, mixing duration of 15 minutes, and a stirring rate of 500 rpm. It was observed that when the temperature increased from 30 °C to 60 °C, the activation of reaction kinetics led to an increase in nickel extraction into the solution from 19.7% to 25.7%. However, when the temperature was further increased to 80 °C, the evaporation of water from the solution at high temperature caused a significant decrease in nickel extraction.

At a mixing duration of 15 minutes, stirring rate of 500 rpm, and the selected optimal conditions of 60 °C temperature and 20% sulfuric acid concentration, the effect of the solid-to-liquid ratio on the degree of nickel extraction was investigated. The results showed that when the solid-to-liquid ratio increased from 1:3 to 1:7, the nickel extraction increased from 25.7% to 41.5%, respectively.

Table 2. Effect of various factors on the degree of nickel extraction from hydrogenation catalyst waste using sulfuric acid solutions

Factors	Degree of nickel extraction, %	Experimental conditions
Effect of sulfuric acid concentration, %		
3	10,6	S:L = 1:3, interaction time 15 minutes, stirring rate 500 rpm, temperature 30 °C
5	11,3	
10	13,5	
15	16,1	
20	***19,7***	
25	17,5	
30	16,2	
Effect of temperature, °C		
30°C	19,7	Sulfuric acid concentration 20%, mixing time 15 minutes, mixing speed 500 rpm, Q:S=1:3
40°C	21,7	
50°C	22,6	
60°C	***25,7***	
70°C	23,5	
80°C	21,2	
Effect of solid:liquid ratio		
1:3	25,7	Sulfuric acid concentration 20%, mixing duration 15 minutes, stirring rate 500 rpm, temperature 60 °C
1:5	26,5	
1:7	***41,5***	
1:10	34,4	
1:15	19,2	
1:20	10,1	
Effect of mixing duration, minutes		
15	41,5	Sulfuric acid concentration 20%, S:L=1:7, mixing speed 500 rpm, temperature 60°C
30	***45,2***	
45	27,8	
60	15,1	
90	10,9	
120	7,01	
Effect of mixing speed, rpm		
500	45,2	Sulfuric acid concentration 20%, S:L=1:7, mixing duration 30 minutes, temperature 60°C
600	49,1	
700	68,6	

800	***81,7***
900	72,8
1000	63,5

However, since the selected object-the spent catalyst waste-was formed during the hydrogenation of vegetable oils, it contains a small amount of residual oil. At the same time, increasing the S:L ratio up to 1:20 leads to flotation of the nickel-containing waste on the solution surface due to the increased liquid phase, which disrupts the contact between sulfuric acid and the solid phase and results in a sharp decrease in the degree of nickel extraction.

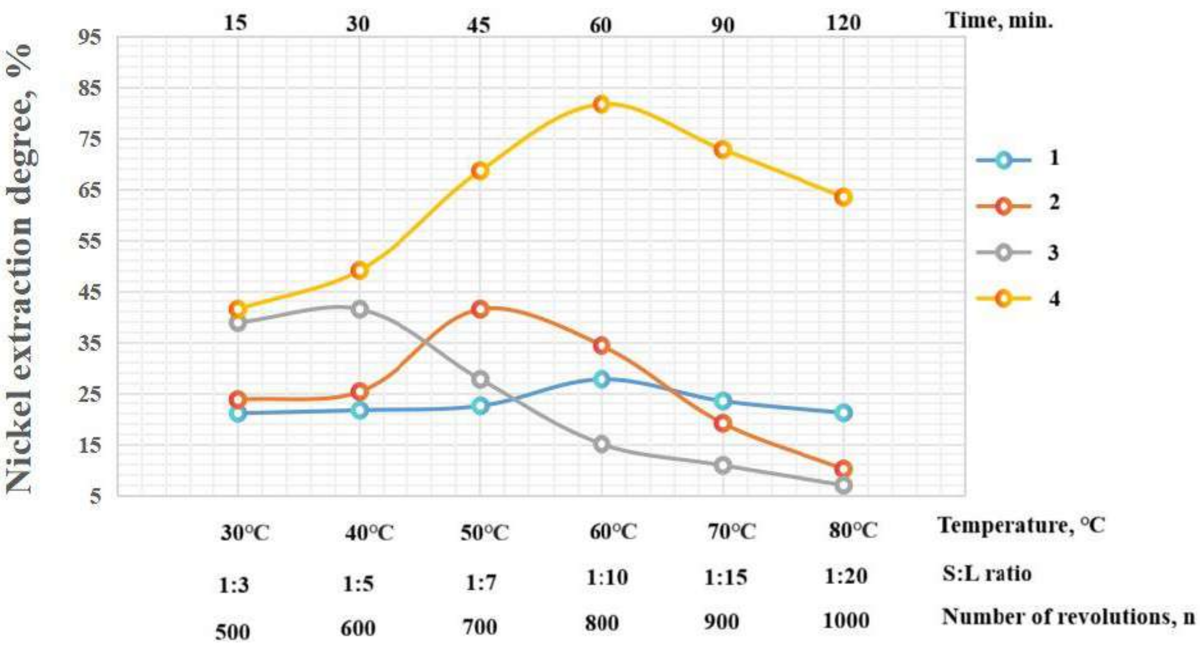


Figure 5. Effect of various factors on the degree of nickel extraction from spent hydrogenation catalyst using a 20% sulfuric acid solution: 1 - temperature; 2 - solid-to-liquid ratio; 3 - interaction time; 4 - stirring rate

Studies were also conducted on the effect of mixing duration on the degree of nickel extraction at a sulfuric acid concentration of 20%, S:L = 1:7, stirring rate of 500 rpm, and temperature of 60 °C. The results showed that when the duration increased from 15 to 30 minutes, the nickel extraction into the solution increased from 41.5% to 45.2%, respectively. However, when the mixing duration was further increased from 30 to 120 minutes, the extraction degree decreased by up to 7.01%.

In the final stage of the study on extracting nickel from spent catalyst waste using sulfuric acid, the effect of stirring rate on the degree of nickel extraction was investigated. The results showed that increasing the stirring rate from n = 500 rpm to n = 800 rpm significantly enhanced the reaction rate, leading to a sharp increase in nickel extraction from 45.2% to 81.7%. However, at a stirring rate of n = 1000 rpm, strong hydrodynamic effects between the solution and the solid phase caused intense splashing of the solution onto the reactor walls. This resulted in limited contact between the waste and the liquid phase, a reduction in the effective volume of the sulfuric acid solution, and consequently a decrease in the degree of nickel extraction.

Furthermore, under the optimal technological conditions - 20% sulfuric acid solution, temperature of 60 °C, solid-to-liquid ratio of 1:7, interaction time of 30 minutes, and a stirring rate of 800 rpm - it was possible to achieve 81.7% nickel extraction into the solution from the spent hydrogenation catalyst.

Figure 6. Nickel extraction into solutions of sulfuric acid at different concentrations.

Figure 7. Nickel extraction into a 20% sulfuric acid solution under optimal conditions.

As shown in Figures 6 and 7, when studies on nickel extraction from spent catalyst waste were carried out using sulfuric acid solutions of different concentrations, the formation of a green-colored solution confirming the presence of Ni^{2+} ions was observed. In addition, quantitative analysis data and visual results confirm that the maximum amount of nickel was transferred into the solution from the spent catalyst waste.

Conclusion. In conclusion, sulfuric acid was used to extract the nickel microelement from the waste of “IDEAL OIL” JSC located in the Namangan region of the Republic of Uzbekistan. In this process, sulfuric acid solutions of different concentrations were applied, the time regime was monitored, the stirring rate using a magnetic stirrer was taken into account, and the solid-to-liquid ratio of the waste with the solvent was also studied. Considering all the above-mentioned parameters, it was determined that under the optimal technological conditions - 20% sulfuric acid solution, temperature of 60 °C, solid-to-liquid ratio of 1:7, interaction time of 30 minutes, and a stirring rate of 800 rpm - it is possible to achieve 81.7% nickel extraction from the spent hydrogenation catalyst.

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CONTENTS

TECHNICAL SCIENCES: COTTON, TEXTILE AND LIGHT INDUSTRY

Parpiyeva N., Kayumov J., Parpiyev D., Tukhtasinov D., Rizayev D., Komilov M.
Rotational auto-oscillations of ribbed cylinders in a pneumatic pressure supply system **3**

Mirzaumidov A.
Integrated multi-track laser surface hardening of gears and rotating components: thermal field control and residual stress engineering **14**

Mirzaumidov A., Xabibullayev D.
Practical study of determining vibrations of 5LP machine in experimental research **20**

Kozokov S.
Determination of optimal parameters of an advanced device for cleaning cotton from large impurities based on a mathematical model **26**

Mamakhanova Z.
Biomechanical principles of sportswear design for kayak slalom athletes **38**

TECHNICAL SCIENCES: AGRICULTURE AND FOOD TECHNOLOGIES

Sattarov K., Khazratkulov J.
Use of non-traditional raw materials in the production of fish feed **46**

Akramova G.
Grain cold conditioning device and technological solution **52**

Ravshanov S., Abdullayeva F., Zaynobiddinov M.
Investigation of the chemical composition, structure, and functional–technological properties of the secondary product “gluten” from kokand spirit JSC **59**

Abdurahimov A., Tashmuratov A., Kuzibekov S., Ochilova S., Toshmuratov M.
Comprehensive assessment of linear dimensions, physical-mechanical and chemical properties of cotton seeds of foreign and local varieties **63**

Yakubjanova Y.
Milk-based refreshing beverages: classification, nutritional benefits and comparative advantages over other beverage types **69**

Aliyeva G., Kanoatov X.
Study of the efficiency of using cryoprotectors on the rheology of the test **73**

Abdurazzokova M., Raxmonova X.
Degradation of pectin and starch in sweet sorghum stem juice using enzymes 83

Ismanova A., Meliboyev M.
Physico-chemical analysis methods in combined drying of topinambur raw materials 87

Atamirzayeva S.
Investigation of additives in the composition of meat canned products based on taraxacum officinale Wigg. plant 93

Abdullayeva B.
Comprehensive assessment of quality and safety indicators of minced meat semi-finished products 100

Ikromov F., Ikromova Y., Xamdamov A.
The importance of using reverse osmosis in tomato paste production 105

CHEMICAL SCIENCES

Janaev M., Adilov R., Ergashev O.
Laws of micelle formation in aqueous solutions of azomethines based on monoethanolamine and acetaldehyde 111

Mukhammadjonov M., Rakhmatkarieva F., Oydinov M.
Hydrothermal synthesis of KA (LTA-type) zeolite from Angren kaolin: structural, morphological, and adsorption characterization 117

Abdukhamidova F., Ibragimova K., Khusenov A., Rakhmanberdiev G.
Nitro-carboxymethylinulin synthesis 128

Yusupova M., Mamadjonova M., Egamberdiev S., Abduvohidov I.
Study of the process of aminolysis of secondary polyethyleneterephalate with monoethanolamine without the participation of a catalyst and analysis of the obtained product 134

Eshonkhodzhaeva O., Mirzarakhmetova D.
Features of cationic pectin synthesis and properties 140

Urinboeva M., Abdikamalova A., Mamataliev N., Ismadiyorov A.
Synthesis of surfactants based on fatty acids and their spectral analysis 147

Urinboeva M., Abdikamalova A., Mamataliev N., Ismadiyorov A.
Adsorption activity of bentonite clays toward dyes 153

Hakimova Kh., Makhkamova D., Turayev Z.
Extraction of the nickel microelement from industrial secondary products using sulfuric acid 166

Ochilov G., Boymatov I., Ganiyeva N.
Adsorption properties of modified adsorbents for dyes 176

Shamuratova M., Giyasidinov A., Abdikamalova A., Eshmetov I.
Bonding of porous structure and soil moisture retention in modification surfactants and polymers 180

Giyasidinov A., Sultonov B., Dedaboyeva M., Aliyev O.
Optimal amounts and concentrations of calcium nitrate solution in the production of phosphate fertilizers 186

Shermatov A., Sherkuziyev D.
Optimization of acid decomposition of washed calcined phosphoconcentrate using a mixed secondary sulfuric–extraction phosphoric acid system: chemical and ftir investigation 195

Umirov F., Erkaev A., Kucharov B., Maxmudov R., Baxshilloyev N.
Production of magnesium chloride from magnesium-containing brines by the isothermal method at 25 °C based on the system $2Na^+, Mg^{2+} \parallel SO_4^{2-}, 2Cl^- – H_2O$ 202

Urinov A., Aslonov A.
Development of an effective anti-corrosion polymer composition for oil and gas trunk pipelines on a resource-saving basis 209

Khamidov R., Oydinov M., Abdulkhaev T.
Crystalline structure and spectroscopic analysis of LIA zeolite 216

TECHNICAL SCIENCES: MECHANICS AND MECHANICAL ENGINEERING

Azamov S.
Improvement of methods for increasing the energy efficiency indicators of an off-grid solar photovoltaic system 224

Kayumov U., Pardaeva Sh.
Operational characteristics of centrifugal pumps in the mining industry 231

Mamanazirov J., Mamatkulov Sh.
Investigating mxene material for evolution reaction in water splitting 240

Obidov A., Khudayberdiyeva D., Mirzaakhmedova D.
Experimental construction development of the device for cleaning cotton from small impurities 247

Sultanov D., Mamahonov A.
Assessment of the chemical and mineralogical properties of rocks from the mountainous areas of chortoq district, namangan region, based on xrf and ftir methods 257

Abduvakhidov M., Mirjalolzoda B., Umarov A.
Bending vibrations of flexible packet-type working bodies of technological machines 271

Abduvakhidov M., Mirjalolzoda B., Umarov A.
Theoretical study of bending vibrations of packet-type working bodies of technological machines with account of internal longitudinal forces **278**

ECONOMICAL SCIENCES

Ergashev A.
The impact of public–private partnership (PPP) mechanisms on enterprise competitiveness in the implementation of green technologies **283**