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| Research Article

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Purification of Ni(No3)2 •6h2o Obtained From Spent Giap-8

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Annotation

Currently, scientific research is being conducted in the world on the synthesis of catalysts with high strength, low hydraulic resistance and specific surface area. In this regard, special attention is paid to the creation technologies for the production of our own highly efficient and durable catalysts based on the processing of spent industrial catalysts ; development of technology for extracting nickel from spent industrial catalysts; technologies for obtaining high-strength reforming catalyst carriers and the scientific basis for obtaining a nickel-containing catalyst on a high-strength carrier.

In the process of nickel recovery, a partial reaction of nitric acid with the construction materials of the plant equipment occurs, and alkali metal impurities that settle on the catalyst during operation are transferred into the solution. As a result, the nickel nitrate solution becomes contaminated with impurities (Table 1).

Table 1

Fe	Co	Cu	Na	K	Ca	Mg	Zn	Cr
0,110	0,009	0,060	0,150	0,015	0,017	0.066	0.135	0.057

We have considered 2 options for purifying nickel nitrate solution:

Recrystallization method and precipitation method of nickel in the form of basic nickel carbonate. The expected result in terms of purity was not achieved by the recrystallization method, since during mass crystallization from a saturated solution the main component entrains impurities [1; p.173] .

Due to the good solubility of nickel carbonate hexahydrate in water, the separation of impurities by washing is impractical.

The solution of nickel nitrate hexahydrate was purified by converting nickel into basic nickel carbonate.

Having obtained the basic nickel carbonate of the required purity, it can be synthesized $Ni(NO_3)_2 \cdot 6H_2O$, meeting GOST requirements.

$$NiCO_3 * Ni(OH)_2 * H_2O + 4HNO_3 = 2Ni(NO_3)_2 + CO_2 + 3H_2O$$

In the work, a solution of $Ni(NO_3)_2$ obtained from crystalline nickel nitrate extracted from the spent GIAP-8 catalyst was used.

The concentration of impurities in nickel nitrate from the crystal hydrate $Ni(NO_3)_2 \cdot 6H_2O$ complies with GOST 4055-78.

Precipitation of basic nickel carbonate from a solution of nickel nitrate with a concentration of 170÷200 g/ ^{dm3} $Ni(NO_3)_2$ was carried out in various ways.

1. Precipitation with a saturated solution of ammonium carbonate salts, pouring solutions preheated to 40÷55℃ into a precipitator equipped with constant heating and stirring, at a pH value of 6.8÷7.2 with filtration of the precipitate and its subsequent drying at a temperature of 100÷110℃ to a constant weight.
2. Precipitation with a saturated solution of ammonium carbonate salts by adding nickel nitrate to it in a solution with constant stirring, without heating the pulp, bringing the pH value of the pulp to 7.5. The pulp was evaporated and dried to a constant weight at 100÷110℃.
3. Precipitation with a saturated solution of ammonium carbonate salts, pouring the solutions without preheating, separately with constant stirring, pH = 7.0 ÷ 7.5, without filtering the pulp with its evaporation and drying to constant weight at 100 ÷ 110 ℃.
4. Precipitation of a sodium carbonate solution by separately pouring solutions preheated to 70℃ into a precipitator , with constant stirring and heating of the pulp, at a pH value of 6.8÷7.2, followed by filtering the precipitate and drying it at 100÷110℃ to a constant weight.
5. Precipitation with sodium carbonate solution is similar to method p.4, at a pH value of 7.2÷7.5.

Nickel nitrate salts obtained by crystallization from a mixed solution do not have a constant composition, because they contain a large amount of moisture due to their high hygroscopicity. In addition, they contain a large amount of impurities (Table 2).

Talitsa 2. ***Impurity content in basic nickel carbonate obtained by precipitation from a solution of nickel nitrate salts***

No.	Method of obtaining	Chemical composition, mass%							
		Ni	Fe	Co	Cu	Na	K	Ca	Mg
1	GOST 4055-78	45-50	0,001	0.01	0.004	K + Na + Ca + Mg n.b. 0.15			
2	Precipitation from a heated solution	47.5	0.10	0.01	0.069	0.09	0,012	0,008	0.02
3	Precipitation without heating	48.0	0.11	0,011	0,060	0.46	0,021	0,008	0.05
4	Precipitation with sodium carbonate	47.7	0.055	0.01	0.061	0.66	0.022	0,007	0.03

Precipitation of impurities in the form of hydroxides during prolonged boiling of a nitric acid solution of nickel did not produce the expected effect.

After boiling a solution of nickel nitrate with a mass concentration of 65.5 g/dm3 , the mass concentration of iron in the solution decreased to 0.074 g/dm3 [5; pp . 264-266] .

When precipitating basic nickel carbonate with a saturated solution of ammonium carbonate salts with separation of the precipitate by filtration, a precipitate containing 19.14% carbonates was obtained. The filtrate after separation of the precipitate contained up to 5.1 g/m3 ^{of} nickel in the form of a soluble ammonia complex (Table 3).

Precipitation of basic nickel carbonate with a soda solution yielded a precipitate that was easily filtered and washed. The precipitate was ground after washing off nitrates and drying, the powder was light green, contained 48% nickel, up to 35% carbonates.

Table 3. ***Precipitation of basic nickel carbonate from nickel nitrate solution with solutions of soda and ammonium carbonate salts***

Mass concentration of nickel in a solution of nickel nitrate salts	Type of precipitator	Mass concentration of nickel in the filtrate	Mass fraction of nickel in the sediment	Mass fraction of nickel carbonates in the sediment	Bulk density of sediment	Note
g/ ^{dm3}	-	g/ ^{dm3}	%	%	kg/ ^{dm3}	
65.5	Ammonium carbonate solution	5.1	47.5	19.4		The precipitate was not filtered, the precipitate was calcined
65.5		-	24.05	15.2	0.52	
65.5		-	53.88	9.4	0.73	
55.5		-	47.7	36.3	0.51	
55.5	Soda solution	Ost	48.2	34.9	0.46	
55.5		Ost	47.7	34.7	0.45	
--	--	--	46.5	35.93	1.42	Indicators of the OKN sample from the KTZ workshop

Thus, $Ni(NO_3)_2 \cdot 6H_2O$ obtained from spent catalyst can be purified to the required standards by precipitation with a solution of soda ash.

Spent industrial catalysts are secondary raw materials for obtaining metal salts. The conducted studies on nickel extraction from spent catalysts with nitric acid solutions showed the possibility of nickel extraction from the catalyst by steam conversion of methane GIAP-8, R -67, GIP-16. The influence of the norm, concentration of nitric acid, temperature and duration of the nickel extraction process was studied.

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