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# Investigation of the Physical and Chemical Properties of Spent Zinc Catalysts and the Process of Processing with Nitric Acid

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**Abstract:** This article examines the physical and chemical properties of spent zinc catalysts and the process of processing 30% nitric acid. Used zinc catalysts contain at least 95% zinc oxide. The treatment process was carried out in 30% nitric acid in a cycle closed at a temperature of 85-950C, pH within 3-4, by mixing the solution for 5 hours. The mass fraction of Zn(NO3)2 in the solution obtained according to the above parameters is higher than 400 g/dm 3 , and the density of the liquid phase after decomposition varies from 1.312 gr/cm3 to 1.273 gr/cm3 , depending on the pH value of the medium and temperature. The viscosity under these conditions varies from 0.02 sPa to 0.008 sPa.

**Keywords:** physical and chemical properties, zinc oxide, nitric acid, sulfur, temperature, dissolution, filtration.

**Introduction.** The degree of zinc oxide extraction of 70-72% and the extraction time of more than 5 hours were experimentally established by nitric acid dissolution of crushed spent sulfur compounds absorber in a stationary layer.

The aim of this work is: to intensify the dissolution process, increase the degree of zinc oxide extraction and determine the distribution of sulfur between the phases;

## Results and their discussion.

### 1. Dissolution in a circulating liquid stream.

Tests on dissolving the spent absorbent in nitric acid were carried out on a setup consisting of a reactor, a reflux condenser, an ejector and a separatory funnel (Fig. 1)

The reactor has a built-in grid dividing it into two zones. A specified amount of spent granulated absorber GIAP-10 was loaded onto the grid in the upper zone and filled with water to the upper level of the absorber. Nitric acid with a mass fraction of 56% was supplied from a separatory funnel. Circulation was carried out using an ejector. The calculated amount of acid was supplied slowly over 2 hours. Due to the air supply to the ejector, the liquid from the lower part of the reactor rose to the upper part and was sprayed over the absorber layer. The temperature in the reactor was maintained at 80-90 $^{0}$C.

After dissolution was complete, the zinc nitrate solution was drained from the reactor and the insoluble residue was discharged. The content of zinc nitrate, zinc sulfate and nitric acid was determined in the zinc nitrate solution, previously filtered from graphite.

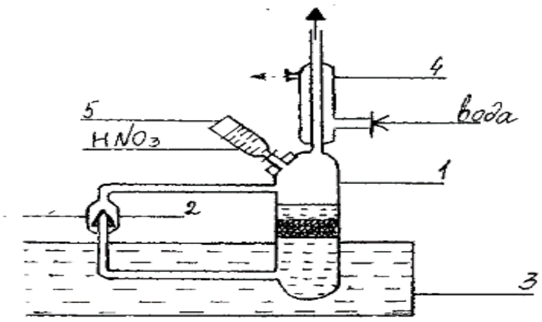

Fig. 1 Laboratory setup diagram

*1-Glass reactor; 2-ejector; 3-water bath; 4-refrigerator; 5-separatory funnel.*

Table 1 presents the results of experiments without condensation, with partial condensation and complete condensation of water and acid vapor.

In a series of experiments, the undissolved residue was reloaded into the reactor and spent absorbent granules were added.

When slowly dosing nitric acid into the reactor onto the circulating liquid layer, no violent reaction was observed, which is explained by the low concentration of the solution formed in the reactor. This made it possible to minimize the release of nitrogen oxides.

Condensation of vapors in a dephlegmator and reflux condenser made it possible to achieve a higher degree of extraction of zinc oxide into solution.

It should be noted that the removal of vapors from the reactor occurs from the circulation with the supply of air to the ejector.

From the data presented in Table 1 it is evident that the mass concentration of $Zn(NO_3)_2$ is from 343.8 to 433.3 g/dm$^3$ . Theoretically, from 100 g of spent GIAP-10 a solution with a mass concentration of 473 g/dm$^3$ should be formed.

From a comparison of the mass concentrations of $Zn(NO_3)_2$ it follows that with a decrease in vapor carryover, the degree of deviation of the mass concentration from the theoretical value is 6-15%.

The results of experiments No. 3, 4, 5 are considered together, since the undissolved portion of the raw material remained in the reactor. Experiment No. 3 left 30 g of wet residue, which dissolved in experiment No. 4 faster than the freshly loaded raw material. The larger amount of undissolved residue in experiment No. 4 can be explained by a lack of acid, since for 100 g of sample at a stoichiometric rate of 195.16 cm $^3$ we took 180 cm$^3$ of acid. Here, in addition to 100 g of sample, there is another 30 g of undissolved residue.

In experiment No. 5, 50 g of sample was added to 50 g of undissolved residue, the volume of acid did not change. The results of this experiment show that while maintaining the total calculated mass of the spent absorbent, the amount of undissolved residue decreases in subsequent cycles. After certain cycles, the amount of undissolved residue stabilizes (see the results of experiments 6-9).

The formation of zinc sulfate in the solution is noteworthy. During the rapid dissolution reaction, zinc sulfide forms mainly $H_2S$ , and part of the zinc sulfide passes into the solution in the form of zinc sulfate.

In contrast, when dissolved in a weakly concentrated acid, the main amount of ZnS passes into solution in the form of $ZnSO_4$. The data in Table 1 show that the mass concentration of $ZnSO_4$ is 47-83.66 g/dm$^3$.

Zinc sulfate, when precipitated with an ammonia-carbonate solution, forms ammonium sulfate according to the reaction

$$ZnSO_4+NH_4HCO_3+NH_4OH + H_2O \rightarrow ZnCO_3(ZnOH)_2+(NH_4)_2SO_4$$

When filtering and washing the precipitate, ammonium sulfate will pass into the filtrate and the product – ZnO will not be contaminated with sulfate ion.

The filtration of the zinc nitrate solution and the precipitation of basic zinc carbonate will depend on the mass concentration of free nitric acid in the solution. The more free acid in the solution, the more ammonium nitrate is formed during precipitation, which thickens the solution and complicates filtration and washing from ions $NO_3$.

Therefore, a high content of free nitric acid in the solution is not recommended. With a residual concentration of HNO $_3$ up to 1.5% by weight, the solution is well filtered on conventional filters.

**Results of dissolution of spent absorbent in circulating liquid flow t=90-96 $^0$ C; V $_{H2O}$ =290 cm $^3$; V $_{HNO3}$ =180 cm $^3$; t=2 hours**

| No. | Attachment g | $Zn(NO_3)$ solution is 2 $cm^3$ | Concentrate $Zn(NO_3)_2$ g/$dm^3$ | Mass of $Zn(NO_3)_2$ g | Con center $ZnSO_4$ g/ $^{dm3}$ | Mass of $ZnSO_4$ g | NHO Concentrate 3% | Non-solvent residue, g | Degree of extraction of ZnO, % |
|---|---|---|---|---|---|---|---|---|---|
| 1 | 100 | 250 | 343.8 | 85,995 | 47.04 | 11.74 | 2.58 | 45.0 | 45.25 |
| 2 | 100 | 340 | 374.6 | 127.36 | 58.97 | 20.05 | 2.49 | 42.0 | 68.4 |
| 3 | 100 | 360 | 417.3 | 150.22 | 65.35 | 23.53 | 0.78 | 30.0 | 91 |
| 4 | 100+remainder | 300 | 443.3 | 132.96 | 72,74 | 21.82 | 0.75 | 50.0 | |
| 5 | 50+remainder | 330 | 403.0 | 133,00 | 83.66 | 27.61 | 1.52 | 25.0 | |
| 6 | 100 | 370 | 426.74 | 157.9 | 38 | 14.06 | 0.84 | 25 | -//- |
| 7 | 25+75 | 350 | 436.22 | 152.68 | 49 | 17.15 | 0.93 | 20 | -//- |
| 8 | 20+80 | 350 | 414.88 | 145.21 | 46 | 16.10 | 0.93 | 18 | -//- |
| 9 | 18+82 | 350 | 393.54 | 137.74 | 35 | 12.25 | 0.91 | 30 | -//- |

2. *On the distribution of sulfur between solid, liquid and gaseous phases.*

During nitric acid dissolution of spent zinc absorbent of sulfur compounds, the mechanism of sulfur conversion can be represented as follows:

$$ZnS + 2HNO_3 \rightarrow Zn(NO_3)_2+H_2S \qquad (1)$$

$$ZnS + 2O_2 \rightarrow ZnSO_4 \qquad (2)$$

$$2ZnS + 3O_2 \rightarrow 2ZnO + 2SO_2 \qquad (3)$$

The release of sulfur compounds into the environment depends on the degree of reactions (1)-(3).

Therefore, the distribution of sulfur between the phases will determine the processing technology.

The experimental study was carried out in laboratory conditions by dissolving 300 g of spent GIAP-10 in 30% nitric acid at a temperature of 70-80 $^{0\,C}$ and capturing the resulting gases with a 10% sodium hydroxide solution.

The results of qualitative analysis showed that sulfur in the solution is in the form of ions $SO_4^{--}$. $SO_3^{--}$Traces of ions, $S^{--}$no ions.

Quantitative analyses have established the following:

- nitric acid solution of spent absorbent;

V = 1.180 $dm^3$, $C_{SO4}^{--}$= 91 g/$dm^3$, C $_{ZnSO4}$ = 436 g/$dm^3$

- solution for absorption of gases formed during dissolution:

V =0.183$dm^3$; With $_{NaS}$ =0; With $_{NaNO3}$ = 19.2 g/$dm^3$

- solid residue:

G -29g; With $_{ZnO}$ =0.24%; C $SO_4^{--}$=7.6%

Calculations of the material balance of sulfur in the absorber, in the solution in the gas and in the solid residue confirmed the results of the study. The discrepancy in the material balance was 7.7%.

Thus, sulfur from the spent absorber, when dissolved in 30% $HNO_3$ at 70-80$^0$C, remains completely in the solution and undissolved sediment. Dissolution in a circulating liquid flow intensifies the process of ZnO extraction , with a 90% degree of ZnO transition into solution achieved.

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