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# Study of the Process of Producing Zinc Nitrate from Secondary Raw Materials

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***Abstract.*** *The results of studies on the production of zinc nitrate from waste zinc catalysts are presented. The waste zinc catalysts contains 74.55 % ZnO. The influence of technological factors on the degree of zinc extraction from the waste zinc catalysts by nitric acid has been investigated. The optimal parameters of the process have been determined. The maximum degree of zinc extraction is observed at an acid rate of 120%, and a nitric acid concentration of 30%. An increase in the duration of the process from 1 to 3 hours increases the degree of zinc extraction from 25.47% to 91.18% with other constant parameters. An increase in temperature from 55 to 95℃ contributes to an increase in the degree of zinc extraction to 91.18%.*

***Keywords:*** *Waste zinc catalysts, temperature, nitric acid, degree of extraction, time, zinc oxide.*

The concept of development of the Republic of Uzbekistan envisages the rise of industry to a qualitatively new level, further intensification of production based on deep processing of local raw materials, and the development of production of new types of products.

One of such raw materials is spent zinc adsorbent, the processing of which will allow obtaining a wide range of liquid, export-oriented and import-substituting products. Such types of products include zinc nitrate, zinc oxide, zinc white, adsorbents.

Zinc oxide adsorbents are intended for fine purification of natural gas from sulfur compounds. Spent adsorbents are replaced with fresh ones after saturation with sulfur. Hundreds of tons of zinc oxide adsorbents are required for a one-time loading of sulfur purification devices in the chemical industry. Adsorbents unloaded from devices are production waste [2, pp. 18324-18327].

Due to the lack of recycling, more than 1,000 tons of used zinc adsorbents of the GIAP-10 brand have accumulated at nitrogen industry enterprises in Uzbekistan.

A study of the composition of the waste showed that the main oxides included in the composition of the spent absorber GIAP-10 are: ZnO , S , C, MgO , in small quantities impurities of $Al_2O_3$ , CaO and others (Table 1)

**Table** 1. **Chemical composition of the original spent absorber GIAP-10**

| No. | Sample | Chemical composition, mass % | | | | | |
|---|---|---|---|---|---|---|---|
| | | **ZnO** | **S** | **WITH** | **MgO** | **Al2O3** | **CaO** |
| 1 | sample 1 | 74.55 | 11.83 | 6.37 | 5.78 | 0.61 | 0.43 |
| 2 | sample 2 | 76.71 | 11.23 | 5.91 | 5.02 | 0.55 | 0.41 |
| 3 | sample 3 | 78.83 | 10.07 | 4.88 | 4.94 | 0.57 | 0.49 |

The nitric acid method is the most suitable for the conditions of our Republic. Therefore, our research is aimed at obtaining zinc oxide from the spent absorber GIAP-10 by nitric acid leaching. In this case, the following reactions occur:

$ZnO + 2\ HNO_3 \rightarrow Zn(NO_3)_2 + \mathbf{H_2O}$

For laboratory research, tests were conducted on the dissolution of the spent absorber GIAP-10 of the composition (mass.%): ZnO – 74.55; S – 11.83; C – 6.37; MgO – 5.78; $Al_2O_3$ – 0.61; CaO – 0.43; pp – 0.14 in nitric acid in a setup consisting of: a reactor ( three-necked flask), an electric stirrer, a reflux condenser, a separatory funnel and an electric hotplate with a water bath.

In a reactor ( three-necked flask) of 500 ml, the spent absorber GIAP-10 was placed, then an electric stirrer, a reflux condenser, a long funnel were installed and the device was placed on a water bath. From a dropping funnel, with intensive stirring, $HNO_3$ was gradually added in recalculations for the substance, so that the temperature of the reaction mixture did not exceed. The reaction mixture was intensively stirred and heated by heating the water bath.

To reduce moisture loss, the hole in the reactor where nitric acid was fed was plugged. A reflux condenser was installed in the second hole.

The resulting leaching solutions were analyzed for zinc content using known chemical analysis methods. [5 , p. 103-106].

The influence of nitric acid concentration and rate, temperature and duration of the process on the degree of zinc extraction into nitric acid solutions was studied. Table 2 shows the data on the influence of nitric acid concentration on the degree of zinc extraction from the spent adsorbent.

The effect of acid concentration of 20-56% was studied with other parameters constant - nitric acid rate of 120% of stoichiometry, temperature of 95°C and duration of leaching process of 3 hours. It was found that the maximum degree of zinc extraction into solution of 91.18% is achieved with nitric acid concentration of 30%.

**Table 2. Effect of nitric acid concentration on the degree of zinc extraction from spent zinc adsorbents**

| No. | With HNO3, % | Chemical composition, mass % | | | | | Extraction rate ZnO, % |
|---|---|---|---|---|---|---|---|
| | | ZnO | MgO | $Al_2O_3$ | CaO | $SO_3$ | |
| 1 | 20 | 6.64 | 0.78 | 0.08 | 0.06 | 0.62 | 6 5.58 |
| 2 | 30 | 13,14 | 1.11 | 0.12 | 0.08 | 1.22 | 91.18 |
| 3 | 40 | 10.09 | 1.34 | 0.14 | 0.10 | 0.94 | 58,39 |
| 4 | 50 | 11.30 | 1.59 | 0.17 | 0.12 | 1.05 | 54.87 |
| 5 | 56 | 11.45 | 1.74 | 0.18 | 0.13 | 1.07 | 51.02 |

Table 3 shows the data on the effect of the nitric acid rate on the degree of zinc extraction in a 30 % nitric acid solution. It was found that the maximum degree of zinc extraction is achieved at a rate of 120 % or more.

**Table 3. Effect of nitric acid rate on zinc extraction degree**

| No. | Norm, % | Chemical composition, mass % | | | | | Extraction rate ZnO, % |
|---|---|---|---|---|---|---|---|
| | | ZnO | MgO | $Al_2O_3$ | CaO | $SO_3$ | |
| 1 | 80 | 8.25 | 1.55 | 0.16 | 0.12 | 0.77 | 41,12 |
| 2 | 90 | 11.03 | 1.41 | 0.15 | 0.11 | 1.03 | 60.39 |
| 3 | 100 | 12.31 | 1.30 | 0.14 | 0.10 | 1.15 | 73.40 |
| 4 | 110 | 12.78 | 1.20 | 0.13 | 0.09 | 1.19 | 82.49 |
| 5 | 120 | 13,14 | 1.11 | 0.12 | 0.08 | 1.22 | 91.18 |
| 6 | 130 | 12.32 | 1.04 | 0.11 | 0.08 | 1.15 | 91.62 |

Thus, if at the rate of 120% and constancy of other parameters the degree of extraction is 91.18%, then at the rate of 130% of stoichiometry it is 91.62%. Increasing the rate of acid reagent above 120% leads to an increase in the degree of zinc extraction by 0.34%, which is undesirable. The optimal acid rate is 120%.

Studies of the duration of the leaching process showed that increasing the leaching time from 1 to 5 hours with 30% nitric acid at an acid rate of 120% and a temperature of 95°C increases the degree of zinc extraction from spent zinc adsorbents from 25.57% to 91.47% (Table 4).

**Table 4. Effect of process duration on the degree of zinc extraction**

| No. | time, min | Chemical composition of the liquid phase, mass % | | | | Extraction rate, % |
|---|---|---|---|---|---|---|
| | | ZnO | MgO | $Al_2O_3$ | CaO | |
| 1 | 60 | 3.17 | 0.95 | 0.09 | 0.06 | 25.57 |
| 2 | 120 | 8.78 | 1.02 | 0.10 | 0.06 | 60.95 |
| 3 | 180 | 13,14 | 1.10 | 0.11 | 0.07 | 91.18 |
| 4 | 240 | 13.17 | 1,12 | 0.11 | 0.08 | 91.39 |
| 5 | 300 | 13.66 | 1.16 | 0.12 | 0.09 | 91.47 |

Temperature has a significant effect on the degree of zinc extraction from spent adsorbents (Table 5). Thus, at a leaching temperature of 55℃ the degree of extraction is 60.10 %, at 85℃ 83.67 % and at 95℃ reaches 91.18%. To achieve maximum zinc extraction from spent zinc adsorbents, it is necessary to increase the temperature above 95℃.

**Table 5. Effect of temperature on the degree of zinc extraction**

| No. | t, ℃ | Chemical composition of the liquid phase, mass % | | | | Extraction rate, % |
|---|---|---|---|---|---|---|
| | | ZnO | MgO | $Al_2O_3$ | CaO | |
| 1 | 55 | 8.65 | 1.11 | 0.12 | 0.08 | 60.10 |
| 2 | 65 | 9.71 | 1.11 | 0.12 | 0.08 | 67.45 |
| 3 | 75 | 10.81 | 1.11 | 0.12 | 0.08 | 75.06 |
| 4 | 85 | 12.05 | 1.10 | 0.12 | 0.07 | 83.67 |
| 5 | 95 | 13,14 | 1.10 | 0.11 | 0.07 | 91.18 |

Thus, the conducted studies on zinc leaching from spent zinc adsorbents with nitric acid have shown the possibility of obtaining zinc nitrate solutions. The optimal The parameters are nitric acid concentration of 30 %, standard of not less than 120 % of stoichiometry and process duration of not less than 3 hours at temperature of 90-95℃. In this case, the degree of zinc extraction is 91.18%.

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