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Article

Recovery of Zinc Oxide from Industrial Zinc-Bearing Wastes

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Abstract: This study investigates the regeneration of spent zinc catalysts containing 71–76% zinc oxide and their conversion into zinc oxide through nitric acid processing. The effects of key technological parameters on zinc recovery were examined. Optimal conditions for zinc extraction were identified as an acid dosage of 120% of the stoichiometric requirement, a nitric acid concentration of 30%, a leaching time of 3 hours, and a temperature of 95°C. The resulting zinc nitrate solution was further processed by precipitation using an 18% sodium carbonate solution at a temperature of 65–70°C and a pH range of 7,9–8,2, with a reaction time of 45 minutes, leading to the formation of zinc hydroxide carbonate. The obtained precipitate was then dried at 120°C for 2 hours and subsequently calcined at 500°C for 5 hours to produce zinc oxide.

Keywords: Nitric acid, zinc nitric acid, sodium carbonate, zinc hydroxide carbonate, zinc oxide, precipitation, temperature, pH, time, physical and chemical properties.

Introduction

In line with the development strategy of the Republic of Uzbekistan, which emphasizes the modernization and diversification of industry through the transition to advanced technologies and the expansion of high value-added products based on deep processing of local raw materials [1], the recycling of spent catalysts has become increasingly important. Evaluating their performance in comparison with global counterparts and enhancing their export potential are key priorities.

The national development concept also focuses on elevating the industrial sector to a new qualitative level by intensifying production processes through the efficient utilization and deep processing of secondary raw materials, as well as by promoting the manufacture of new types of products.

Literature Review

A review of previous experimental studies shows that the chemical-thermal activation approach enables the complete dissolution of ZnO into solution in the form of zinc nitrate, followed by its precipitation as a carbonate compound. Subsequent thermal decomposition of zinc carbonate results in the formation of highly active, porous, and lightweight zinc oxide. The adsorbent produced from chemically and thermally activated ZnO exhibits a sulfur capacity of up to 29% and a specific surface area of 78 m^2/g [2].

During processing, zinc nitrate is obtained along with the formation of an insoluble dark residue, while small amounts of H_2S and nitrogen oxides are released depending on the concentration of nitric acid used. Dissolution was carried out using nitric acid solutions with concentrations of 30% and 56%. Experimental studies were conducted to determine the optimal conditions for precipitating zinc from the nitrate solution using ammonium carbonate. The precipitation process was performed both with heating up to 80°C and without heating, using a saturated ammonium carbonate solution. As a result, a white zinc carbonate precipitate was formed.[3]

To obtain zinc oxide, a sequence of operations was carried out, including washing the zinc carbonate to remove ammonium nitrate and zinc sulfate impurities, drying at 100°C, and calcination at 500°C. During the calcination stage, zinc carbonate undergoes thermal decomposition, resulting in the formation of ZnO [4].

Research methods

Among the available methods for zinc oxide production under the conditions of our Republic, the nitric acid route combined with precipitation is considered the most suitable. Accordingly, this study focuses on obtaining zinc oxide from spent zinc catalysts through nitric acid leaching followed by precipitation.[5][6]

Experimental investigations were carried out using spent zinc catalysts containing 71–76% ZnO. The decomposition process was performed in a 500 ml reactor equipped with an electric stirrer, a reflux condenser, and a dropping funnel, and placed in a water bath. The reaction mixture was continuously stirred and heated via the water bath. Nitric acid was introduced through one of the reactor necks, while the reflux condenser was installed in another to maintain controlled conditions.[7]

The leach solutions obtained were analyzed for zinc content using standard chemical analytical methods [8].

Zinc hydroxide carbonate was then precipitated from the zinc nitrate solution using a sodium carbonate solution. The precipitation process was carried out by simultaneously introducing zinc nitrate and sodium carbonate solutions from separate funnels under constant stirring and heating. The pH of the system was maintained within the range of 7,9–8,2 to ensure complete precipitation, and the process duration was 45 minutes.[9]

Following precipitation, the zinc hydroxide carbonate was separated from the sodium nitrate mother liquor and washed with distilled water to remove residual nitrate ions.[10]

Finally, the obtained precipitate was subjected to drying and calcination in a porcelain crucible placed in a muffle furnace at a specified temperature. After thermal treatment, the product was cooled in a desiccator for a predetermined period.[11]

Results and discussion

The effect of nitric acid concentration in the range of 20–30% was investigated under constant operating conditions, including an acid dosage of 120% of the stoichiometric requirement, a temperature of 95°C, and a leaching time of 3 hours. The results demonstrating the influence of acid concentration on the extent of zinc extraction from the spent zinc catalyst are presented in Table 1.[12]

Table 1. Influence of nitric acid concentration on zinc extraction efficiency.

№	HNO_3, %	Chemical composition, mass %					Extraction ZnO, %
		ZnO	MgO	Al_2O_3	CaO	SO_3	

1	20	6,64	0,78	0,08	0,06	0,62	65,58
2	30	13,14	1,11	0,12	0,08	1,22	91,28

The experimental results showed that the highest zinc extraction efficiency, reaching 91.28%, was obtained at a nitric acid concentration of 30%.[13]

For the subsequent precipitation of zinc hydroxide carbonate from the resulting zinc nitrate solution, it is essential to evaluate the physicochemical properties of the intermediate products. The precipitation process was carried out using sodium carbonate at a pH range of 7.9–8.2, a temperature of 65–70°C, and a reaction time of 40–45 minutes. The analytical results of zinc hydroxide carbonate formation are presented in Table 2.[14]

Table 2. Impact of ph on the degree of precipitation

pH	Salt composition of the liquid phase, mass %					Degree of precipitation, %
	$ZnCO_3$	$Zn(OH)_2$	$Zn(NO_3)_2$	$NaNO_3$	HNO_3	
7,9	3,438	2,725	0,127	18,845	-	98,79
8,0	3,312	2,603	-	19,267	-	99,93
8,2	3,261	2,581	-	19,693	-	99,96

An increase in pH leads to a higher precipitation efficiency. Raising the pH from 7,9 to 8,2 at a temperature of 70°C improves the precipitation yield of zinc hydroxide carbonate from 98,79% to 99,96%.

In addition, the influence of temperature and process duration on the precipitation degree of zinc hydroxide carbonate from zinc nitrate solution using sodium carbonate was investigated at different temperatures. The corresponding results are presented in Table 3.

Table 3. Influence of temperature and time on precipitation efficiency.

№	Temperature, °C	Salt composition of the liquid phase, mass %				Degree of precipitation, %
		$ZnCO_3$	$Zn(OH)_2$	$Zn(NO_3)_2$	NH_4NO_3	
1	60	3,287	2,603	1,410	8,702	85,68
2	70	3,289	2,609	0,003	10,159	99,97

An increase in temperature from 60°C to 70°C significantly enhances the precipitation efficiency of zinc hydroxide carbonate, raising it from 85,68% to 99,97% within 45 minutes at a pH range of 7,9–8,2.

Prior to calcination, the obtained zinc hydroxide carbonate was subjected to a drying stage. The drying process was carried out at temperatures between 50°C and 120°C for a duration of 2 hours. The corresponding results are presented in Table 4.

Table 4. Effect of temperature on moisture reduction during drying.

№	t,°C	Weight loss, %	Content of components, mass %		
			$ZnCO_3$	$Zn(OH)_2$	moisture
1	100	46,23	46,13	36,55	14,85
2	110	50,83	50,49	39,97	6,85
3	120	55,46	53,97	42,75	-

Increasing the drying temperature from 100°C to 120°C leads to a noticeable rise in moisture removal from zinc hydroxide carbonate, with the moisture loss increasing from 46,23% to 55,46%.

Calcination experiments for zinc hydroxide carbonate were conducted within a temperature range of 150–550°C for a duration of 5 hours. The corresponding results are presented in Table 5.

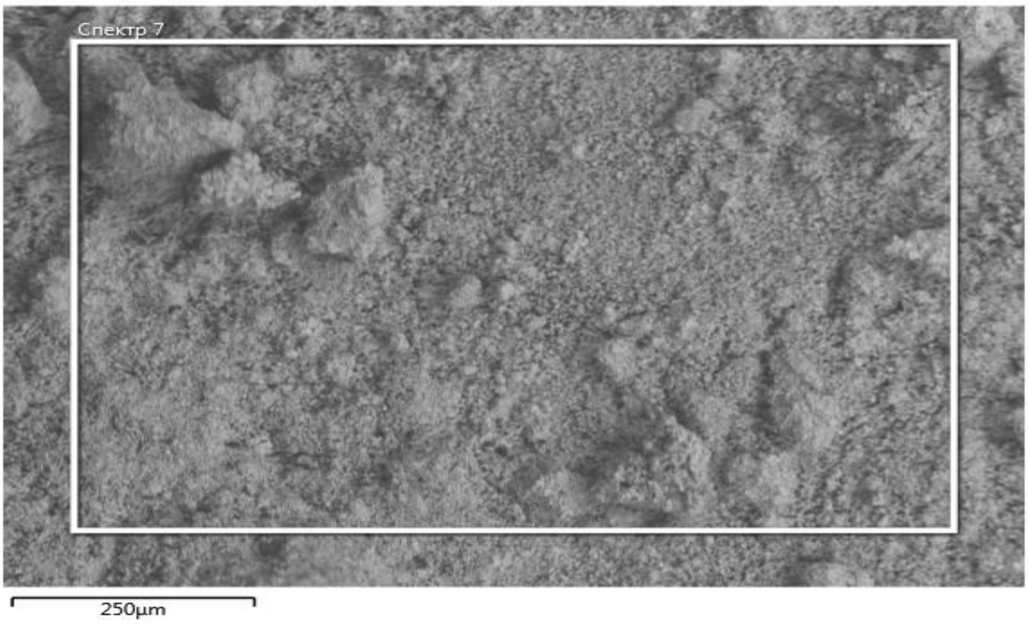
Table 5. Influence of calcination temperature on zinc oxide formation.

№	t,°C	Weight loss, %	Content of components, mass %		
			$ZnCO_3$	$Zn(OH)_2$	ZnO
1	450	22,97	14,02	-	81,74
2	500	26,79	-	-	95,56

As the temperature increases from 450°C to 500°C, zinc carbonate undergoes progressive thermal decomposition, resulting in its complete conversion into zinc oxide.

Figure 1 shows an scanning electron microscopy image of zinc oxide, accompanied by energy-dispersive X-ray analysis used to determine the quantitative composition of the elements present.

The results indicate that the dominant elements in the sample are zinc, oxygen, and sulfur. Their respective contents are 79,9%, 19,9%, and 0,2%.



Element	Zn	O	S	Summa:
Weight, %	79,9	19,9	0,2	100,00

Sigma Weight, %	0,86	0,84	0,27

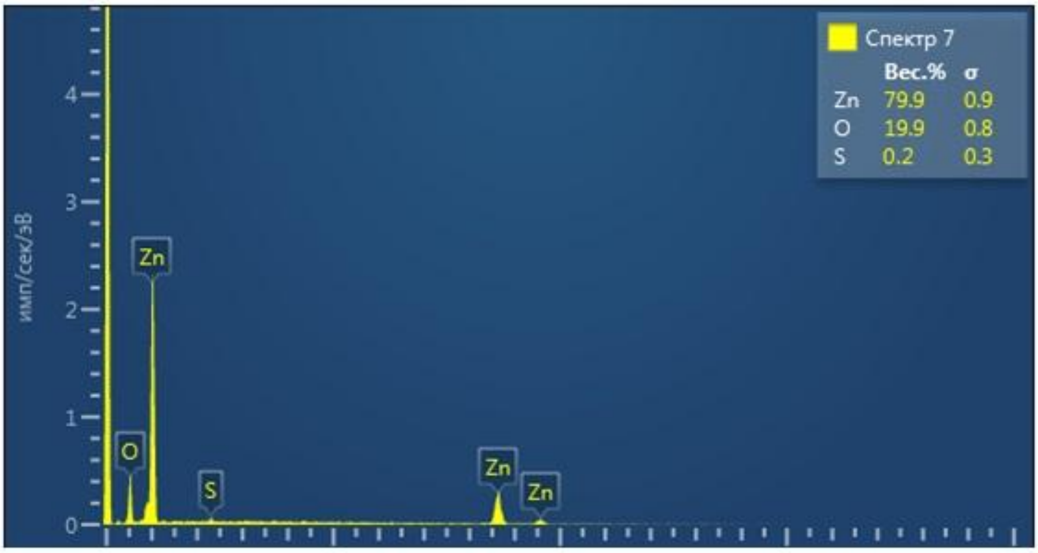


Рис. 1. Scanning electron microscopy image of zinc oxide.

Conclusions and recommendations. The experimental results on nitric acid leaching of spent zinc catalysts confirm the feasibility of producing zinc nitrate solutions. The optimal process conditions were determined to be a nitric acid concentration of 30%, an acid dosage of 120% of the stoichiometric requirement, and a leaching duration of 3 hours at a temperature range of 90–95°C. Under these conditions, the zinc extraction efficiency reaches 91,28%.

Laboratory studies on the precipitation of basic zinc hydroxide carbonate using sodium carbonate also demonstrated favorable outcomes. The optimal precipitation parameters were identified as a pH range of 7,9–8,2, a temperature of 70°C, and a process duration of 40–45 minutes, resulting in a precipitation efficiency of up to 99,97%.[15]

The obtained zinc hydroxide carbonate was subjected to drying prior to calcination at a temperature of 120°C for 2 hours, followed by calcination at 500°C for 5 hours to produce zinc oxide.

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