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RESEARCH ON THE SEPARATION OF ZINC FROM METALLURGY WASTE WITH A MIXTURE OF AMMONIA AND AMMONIUM SALTS

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Abstract: To involve the dust of the "Bekabad" metallurgical combine in the production of complex NPK fertilizers, studies were conducted on several factors such as the duration of processes, the ratio of solid and liquid substances S:L, and temperature in the extraction of zinc from the dust with molar ratios of ammonia and ammonium salts. In this study, an ammonia-ammonium chloride mixture was selected as the most suitable liquidant to achieve high zinc leaching efficiency. Research on the extraction of zinc from waste was carried out in a glass reactor equipped with a paddle stirrer and placed in a thermostatic vessel. The amount of zinc in the filtrate solution was determined by the atomic adsorption method, as well as by the complexonometric titration method with the value of pH=10, using the black Erichrom-T indicator. The effect of Q: S ratio on dust condition in 20^{0}C temperature, 60 minute duration and 3M NH_3:3M NH_4Cl molar ratio solutions was investigated. As can be seen from the results in Table 1, when the S:L ratio is 1:5, the release of zinc into the solution is 81,2 %. When S:L=1:20, the rate of zinc separation is significantly reduced by 54,4 %. Thus, in the extraction of zinc from the dust of "Bekabad" metallurgical combine with NH_3: NH_4Cl ratio, molar ratio of 3M NH_3 : 3M NH_4Cl, Q:S = 1:5, temperature of 60^{0}C and process 180 minutes are the optimal conditions. From the optimization of the extraction process, it can be concluded that by increasing the factors such as temperature, time and NH_3: NH_4Cl ratio to a defined amount, the extraction rate of zinc can be maximized.

Keywords: microelement, ammonia, ammonium chloride, ammonium carbonate, ammonium sulfate, molar ratio, temperature, S:L ratio, time.

Introduction. There are three large metallurgical plants and three nitrogen fertilizer manufacturing enterprises in Uzbekistan. The metallurgical industry generates secondary sources of raw materials in the form of technical waste. Metallurgical plants in Uzbekistan produce zinc waste, Fergana, Chirchik and Navoi nitrogen fertilizer production plants annually generate a large amount of spent industrial catalyst waste containing copper, zinc, nickel, etc. Industrial waste and waste catalysts can be used as sources of trace elements.

At present, the demand for nitrogenous, phosphorous, and potassium fertilizers as well as micronutrient fertilizers is increasing. Production of mineral fertilizers using various wastes and products of non-ferrous metallurgy containing microelements, spent catalysts, sour wastewater and other types of byproducts is one of the solutions for the production of fertilizers containing microelements [1].

In recent years, many studies have shown that the use of NPK fertilizers, which contain trace elements zinc, manganese and copper, is one of the important factors in increasing productivity [2-4]. A significant increase in grain yield due to the combined use of zinc micronutrient with NPK fertilizers shows the importance of micronutrient fertilizers in plant breeding [5, 9]. In many studies, an increase in the growth and yield of plants such as corn, barley, alfalfa, cotton and potatoes was observed through the application of zinc fertilizers [10].

Today, the problem of waste is becoming one of the most urgent environmental issues worldwide. Energy, non-ferrous and ferrous metallurgy, chemical industry and

construction industry facilities are among the main sources of waste and environmental pollution. In the process of beneficiation and smelting of metals, wastes are generated, and their disposal has become one of the most urgent environmental problems today. This is because metallurgical waste contains large amounts of toxic chemicals. Therefore, tons of waste from metallurgical production, billions of them, are an important environmental problem. Due to the presence of lead, cadmium, arsenic, chromium and other metals in metallurgical dust particles, the generation of metallurgical waste causes a great amount of damage to the environment. Therefore, in the chemical and metallurgical industry, there is a growing interest in the extraction of microelements from secondary wastes such as ash, slag, chimney dust, mud, residue containing microelements [11].

Black metallurgical dust contains iron and zinc in the form of oxide, and the amount of zinc in the dust is much lower than the amount of iron. At the same time, if iron purification steps are not included, further processing processes will be difficult [12].

Through the method of hydrometallurgy, it is possible to dissolve primary intermediate secondary products and transfer valuable components to solution in the form of various compounds [13]. The effects of various detergents such as sulfuric acid, hydrochloric acid, ammonia, and sodium hydroxide have been studied in the separation of industrial secondary products and waste [14,15].

Methods. Waste of "Bekabad" Metallurgical Combine (O'zmetkombinat JSC) for research on the production of NPK fertilizers containing zinc: composition (wt., %): ZnO – 17,8%, Fe_2O_3 – 40,6%, CaO – 4,34%, SiO_2 – 4,01%, K_2O – 2,21%, MnO – 1,87%, SO_3 – 1,42%, PbO – 1,15% were used. Gaseous ammonia (GOST 6221-90; CAS №7664-41-7), ammonium sulfate (GOST 3769-78; to analyze (AR), 99%, CAS 7783-20-2), and ammonium chloride (chemically clean, GOST 3773-72, CAS 12125-02-9, Russia) substances were used to extract zinc from the waste of O'zmetkombinat JSC, which is the object of the research.

Research on the extraction of zinc from waste was carried out in a glass reactor equipped with a paddle stirrer and placed in a thermostatic vessel. The amount of zinc in the filtrate solution was determined by the atomic adsorption method, as well as by the complexonometric titration method with the value of pH=10, using the black Erichrom-T indicator.

Results. In many literatures, it is noted that zinc leaching efficiency is high when metallurgical waste is washed by hydrometallurgical method using sulfuric acid and oxalic acid. It was also stated that many elements such as manganese, copper, cadmium, lead, and iron dissolve in the solution, so removing these elements from the process does not benefit economic efficiency. Research on the extraction of zinc from waste was carried out in a glass reactor equipped with a paddle stirrer and placed in a thermostatic vessel. To involve the zinc contained in the wastes of the "Bekabad" metallurgical complex for the production of complex NPK fertilizers, the duration of the extraction processes of zinc through aqueous solutions of ammonia and ammonium salts was studied at different S:L ratios, at different temperatures and at different time intervals. Ammonia-ammonium

chloride mixture was selected as the most suitable liquidant to achieve high zinc leaching efficiency. The obtained results are presented in Table 1.

For the extraction of zinc from industrial intermediate products and wastes, studies were conducted on the increase in the extraction efficiency of zinc with the formation of the $[Zn(NH_3)_4]^{+2}$ complex by using ammonia together with various ammonium salts (Figure 1).

Complex chemical reactions leading to the formation of complex zinc compounds occur in the presence of ammonia and ammonium chloride during washing, as a result of the interaction between Zn^{2+} ions, Cl^- and NH_3 ligands depending on the pH value. Below are the proportions of Zn(II) in the formation of a complex compound with a certain number of ligands, depending on the pH medium.

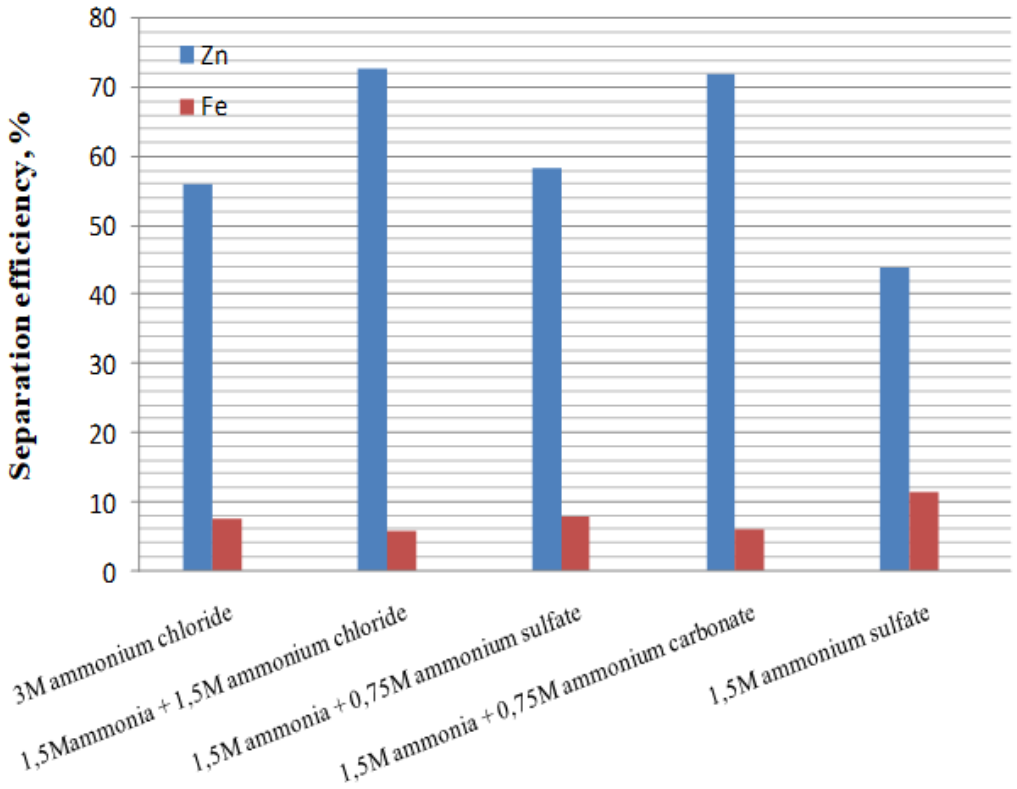

Figure 1. The use of various ammonium salts in the separation of zinc from the wastes of "Bekabad" metallurgical combine

Four chloro complexes interact with Zn (II) ions in solution in an environment with a pH value below 5.5. In the pH range from 5.7 to 7.5, Zn (II) is in the form of the sparingly soluble $[Zn_5(OH)_8]Cl_2$ hydro complex, and the amine complex is formed in small amounts in the form of $[Zn(NH_3)_3]^{2+}$. It proceeds with the formation of $[Zn(NH_3)_4]^{2+}$ complex in the pH environment from 8.0 to 11.0.

Table 1. Effect of NH_3 and NH_4Cl ratio, Q:S ratio, temperature and process duration on the extraction of zinc and iron from the dust of "Bekabad" metallurgical combine.

Factors	Separation of zinc, %	Separation of iron, %	Research conditions
Effect of NH_3 and NH_4Cl ratios			
1,5M NH_3:1,5M NH_4Cl	70,3	9,7	20⁰C temperature, S:L=1:5, duration 60 minutes
2,5M NH_3:2,5M NH_4Cl	72,3	8,3	
2,5M NH_3:3M NH_4Cl	73,7	8,5	
1,5M NH_3:3M NH_4Cl	76,2	8,8	
2M NH_3:3,5M NH_4Cl	77,9	8,6	
3M NH_3:3M NH_4Cl	80,9	7,9	
3,5M NH_3:3,5M NH_4Cl	78,6	7,5	
Effect of S:L			
1:5	81,2	7,9	20⁰C temperature, a solution in the ratio of 3M NH_3:3M NH_4Cl, duration 60 minutes
1:6	77,8	8,5	
1:8	71,5	9,2	
1:10	65,3	9,7	
1:15	57,8	10,8	
1:20	54,4	11,2	
Effect of temperature			
20°C	81,2	7,9	a solution in the ratio of 3M NH_3:3M NH_4Cl, S:L=1:5, duration 60 minutes
30⁰C	82,5	7,2	
40⁰C	83,9	6,7	
50⁰C	86,2	4,9	
60⁰C	90,6	3,8	
70⁰C	89,5	3,9	
Effect of duration of exposure			
60 minute	90,6	3,8	a solution in the ratio of 3M NH_3:3M NH_4Cl, S:L=1:5, 60⁰C temperature
90 minute	91,3	3,5	
120 minute	92,8	3,1	
150 minute	93,9	2,9	
180 minute	95,6	2,5	
210 minute	94,8	2,7	

This method is one of the less selective methods for zinc extraction. Metallurgical waste 3M NH_4Cl, 1.5M NH_3:1.5M NH_4Cl, 1.5M NH_3:0.75M $(NH_4)_2SO_4$, 1.5M NH_3:0.75M $(NH_4)_2CO_3$, 1.5M $(NH_4)_2SO_4$ solutions were used. It was expressed that the separation efficiency of zinc increases in the order $(NH_4)_2SO_4$<$(NH_4)_2CO_3$<NH_4Cl.

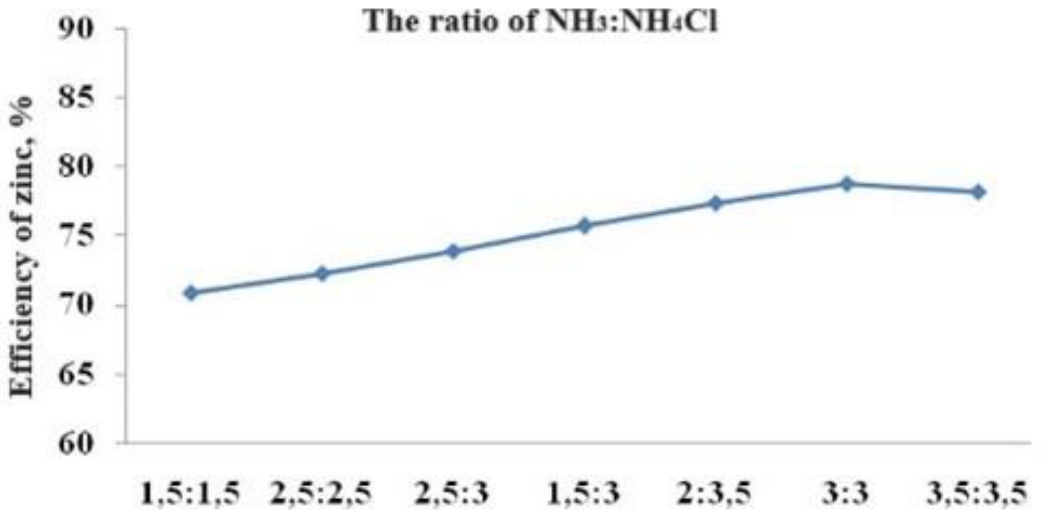

Figure 2. The effect of solutions based on NH_3:NH_4Cl molar ratios on the extraction of zinc from the dust of "Bekabad" metallurgical combine.

It was also noted that NH_3:NH_4Cl and NH_3:$(NH_4)_2CO_3$ system mixtures are promising in the efficiency of zinc separation from metallurgical waste.

The influence of NH_3:NH_4Cl molar ratios on zinc extraction was studied under conditions of mixing 60 minutes and S:L=1:5 (Table 1, Figure 2).

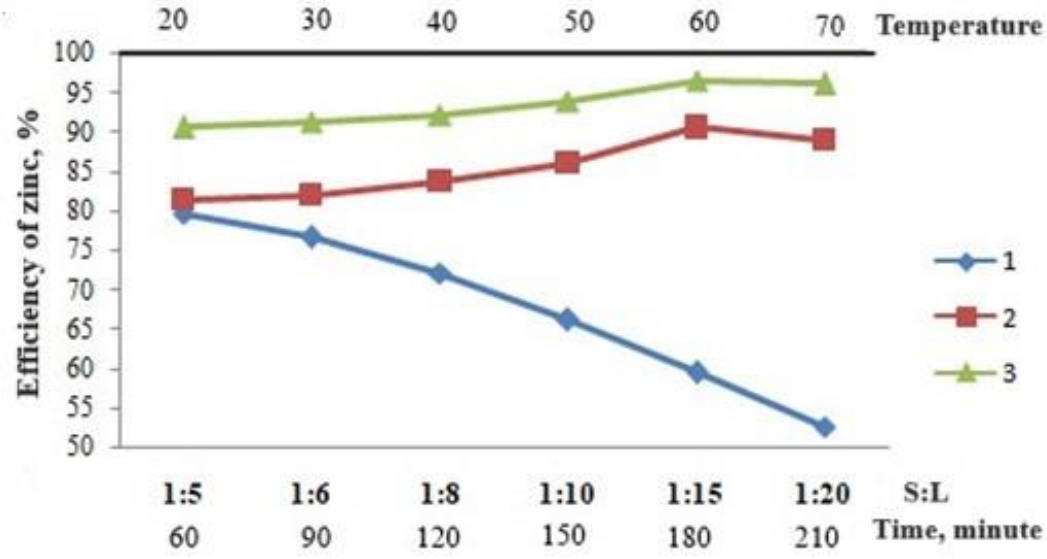

Figure 3. Factors influencing the extraction of zinc from the dust of the "Bekabad" metallurgical complex with solutions based on NH_3:NH_4Cl ratios: 1 - S:L effect; 2 - temperature effect; 3 - the effect of time.

The values of zinc dissolution in the solution are as follows: 1.5M NH_3 : 1.5M NH_4Cl 70.3%, 2.5M NH_3 : 2.5M NH_4Cl 72.3%, 2.5M NH_3 : 3M NH_4Cl 73.7%, 1.5M NH_3 : 3M NH_4Cl

is 76.2%, 2M NH_3 : 3.5M NH_4Cl is 77.9%, 3M NH_3 : 3M NH_4Cl is 80.9%, 3.5M NH_3 : 3.5M NH_4Cl is 78.6% (Figure 2).

During the study, increasing the total concentration of ammonia up to 3M increased the rate of zinc release, and further increasing the concentration above the optimal value was noted to have no significant effect on zinc leaching. Based on the results of our research and the information from the studied literature, the molar ratio of 3M NH_3 : 3M NH_4Cl was chosen as optimal.

The effect of Q: S ratio on dust condition in 20^0C temperature, 60 minute duration and 3M NH_3:3M NH_4Cl molar ratio solutions was investigated. As can be seen from the results in Table 1, when the S:L ratio is 1:5, the release of zinc into the solution is 81,2 %. When S:L=1:20, the rate of zinc separation is significantly reduced by 54,4 %.

Conclusion. Thus, in the extraction of zinc from the dust of "Bekabad" metallurgical combine with NH_3: NH_4Cl ratio, molar ratio of 3M NH_3 : 3M NH_4Cl, Q:S=1:5, temperature of 60^0C and process 180 minutes are the optimal conditions. From the optimization of the extraction process, it can be concluded that by increasing the factors such as temperature, time and NH_3: NH_4Cl ratio to a defined amount, the extraction rate of zinc can be maximized.

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