



SPENT CATALYSTS AS A SOURCE OF MICRO- AND MACROELEMENTS FOR LIQUID FERTILIZERS

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ABSTRACT

This article discusses the possibilities of obtaining water-soluble liquid fertilizers enriched with microelements by acid processing of spent catalysts of, widely used in the nitrogen industry. Studies have been conducted on the effect of nitric acid concentration (in the range of 20–50%) on the degree of extraction of macro- and microelements. Physicochemical and rheological properties of the obtained solutions have been determined. The results demonstrate the prospects for developing an energy-efficient technology for processing in spent catalyst waste using local raw materials, which is relevant in the environmental conditions of the Republic of India.

KEYWORDS: Macro and microelements, system, spent catalyst, liquid fertilizers, nitric acid, processing

INTRODUCTION

At present, the problem of organizing the production of water-soluble complex fertilizers remains unresolved in country, since technologies for their production from local raw materials have not yet been developed in the conditions of the republic. In this regard, the creation of our own production of such fertilizers is a relevant and promising task. One of the key objectives of the study is to determine the role of soil-forming processes, identify the deficiency or excess of microelements and develop recommendations for the effective use of micronutrients. Also, based on the data obtained, it will be possible to adjust micronutrient maps for planning fertilizers and conducting further research in agriculture.

Copper is found in all organs and all phases of cotton growing, when growing it on different soils. Copper micro fertilizers for cotton are used with sufficient provision of the optimal amount of basic fertilizers (N, P, K), in areas where the content of copper available to plants in the arable horizon is less than 0.6 mg / kg of soil. The role of zinc in the ontogenesis of cotton in the conditions of irrigated agriculture in Central Asia has not been sufficiently elucidated. The forms and types of zinc bonds in a living organism are varied. Manganese influences the course of oxidation-reduction reactions in plants: in ammonia, plants behave as an oxidizer, and in nitrate, as a reducing agent.

The physiological role of molybdenum in plants is associated with its participation in dense metabolism,

primarily in enzymatic reduction to ammonia and in the synthesis of proteins and amino acids. In recent years, data have been obtained on the positive effect of molybdenum on phosphorus metabolism in plants. The introduction of molybdenum not only sharply increases the amount of organic phosphorus, but also leads to a qualitative change in protein. In order to provide a physicochemical justification for the process of obtaining microelements, the physicochemical characteristics of spent catalysts used in the nitrogen industry, the accumulation volumes of which reach hundreds of tons, were studied before the stage of decomposition with mineral acids. The energy dispersive spectra of these catalysts are shown in Figure 1. Before use, the spent catalysts were crushed to a particle size of less than 0.05 mm.

METHODOLOGY

To obtain liquid microelement-containing fertilizers, solutions were used that were obtained by nitric acid decomposition of spent catalysts formed during the production of the nitrogen industry. The process of acid processing of catalysts was carried out as follows: in laboratory conditions, nitric acid solutions with concentrations of 20%, 30%, 40% and 50% were used to process 10 g of catalyst waste.

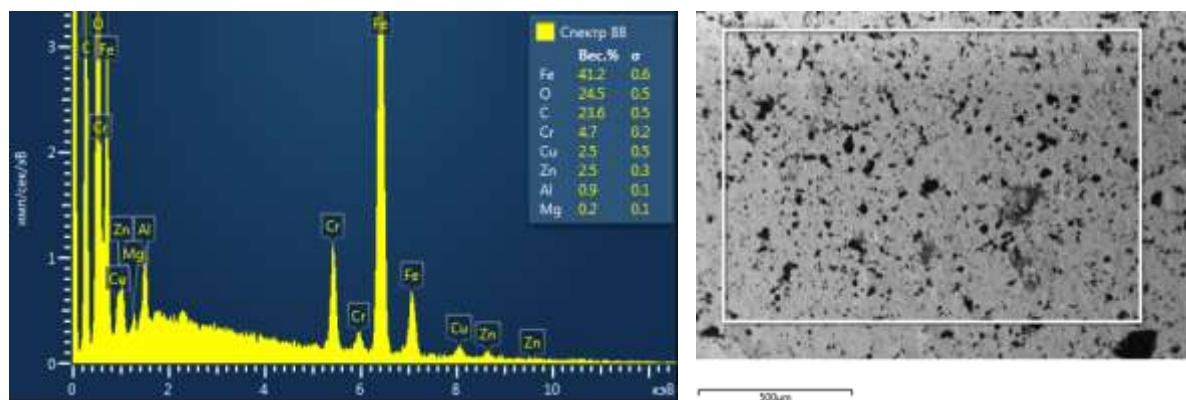
The process of decomposition of the crushed catalyst (to a particle size of 0.05 mm) was carried out on a laboratory set- up consisting of a tubular glass reactor with a stirrer equipped with an electric wire and placed in a water thermostat. The temperature in the thermostat was maintained using a thermometer and an electronic relay and was constant, amounting to 45 °C. The rotation speed of the stirrer was regulated within 250–300 rpm.

The interaction process of the components lasted for 30 minutes. Upon completion of the decomposition, the reaction mass was separated into liquid and solid phases by filtration. The reactor contents were filtered through a Buchner funnel using a Bunsen flask, under a vacuum of 0.60 mm Hg and one layer of “white ribbon” filter paper. The filters were pre-weighed. The sediment remaining on the filter was washed with 10 g of water at a given temperature and dried with the filter at 100 °C until a constant weight was achieved.

RESULTS AND DISCUSSION

A comparative analysis of the results of energy-dispersive spectral research of spent catalysts showed that their elemental composition has a significant diversity. For example, in the spent catalyst of the brand, the copper and zinc content is 2.52% and 2.47%, respectively (Table 1).

The results of mass spectrometric analysis presented in Table 3 also confirm that the spent catalysts contain a wide range of macro- and microelements that are necessary for the production of liquid complex fertilizers with microelements. After decomposition with nitric acid, the liquid phase was analyzed for elemental composition, which is given in Table 2. As follows from Table 2, the liquid phase has in its composition and contains: P-242.0, K-478.0, Ca-1207, Mg-580 mcg/l macro elements and B-50.0, Cu-28000, Zn-19100, Co-68.0, Mn-214 and Fe-545605 mcg/l microelements, respectively.

Figure 1. Energy dispersive spectrum of the spent initial catalyst of the brand**Table 1.** Elemental composition of the spent initial catalyst brand

Element	Weight.%	Sigma Weight%	Name of the standard
C	23.58	0.53	With Vit
O	24.47	0.46	SiO ₂
Mg	0.25	0.08	MgO
Al	0.87	0.09	Al ₂ O ₃
Cr	4.69	0.21	Cr
Fe	41.15	0.57	Fe
Cu	2.52	0.48	Cu
Zn	2.47	0.35	Zn
Sum:	100.00		

Table 2. Mass spectrometric analysis of the initial spent catalysts

Spent catalysts	Macroelements, mcg/l						Microelements, mcg/l				
	P	K	Ca	Mg	Cu	Zn	B	Mn	Fe	Co	Mo
	242.0	478.0	1207	580	28000	19100	50.0	4300	545605	68.0	214

We then investigated the influence of process parameters on the process of nitric acid decomposition of spent catalyst waste (Table 3).

Table 3. Study of the influence of technological parameters on the process of nitric acid decomposition of spent catalyst waste

No.	Ratio HNO ₃	Concentration HNO ₃ , %	Filtration rate, kg/m ² *		W: T ratio	Humidity, %
			h main fil- trate	wash fil- trate		
Waste catalyst brand SHIFT MAX 210						
1	1:10	20	457.08	47.12	12.18:1.0	29.11
2	1:10	30	378.70	69.70	11.62:1.0	26.64
3	1:10	40	305.45	57.44	12.20:1.0	27.63
4	1:10	50	128.5	69.50	10.62:1.0	28.74

Table 4. Mass spectrometric analysis liquid phase of spent catalyst after decomposition with nitric acid

Acid concen- tration ,	Macroelements, mcg/l						Microelements, mcg/l				
	P	K	Ca	Mg	Cu	Zn	B	Mn	Fe	Co	Mo
20	1200	94000	320000	53000	2600	2100	56.0	55000	280000	4900	720
30	1300	60000	290000	54000	2900	2300	53.0	66000	350000	6300	530
40	1400	46000	260000	49000	2900	2300	43.0	69000	390000	4900	400
50	1400	58000	270000	49000	3100	2500	38.0	72000	400000	5500	420

The table presents the results of the analysis of the macro- and microelement composition of solutions obtained by decomposing nitrogen industry catalysts with nitric acid of different concentrations (20%, 30%, 40%, 50%). (Table 4)

Increasing the concentration of nitric acid contributes to:

- Increasing the content of phosphorus, iron, manganese and some other elements in the solution;
- At the same time, the solubility of potassium, calcium, boron and molybdenum deteriorates.
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At a certain concentration of acid (approximately 40–50%), the process of release of many elements stabilizes, or their content changes insignificantly.

Table 5. Rheological properties of the obtained solutions of spent catalysts after ammonization before filtration

No.	Density before filtration, g/cm ³				Viscosity before filtration, mm ² /s				pH
	10	20	30	45	10	20	30	45	
1	1.21596	1.21227	1.20545	1.19859	1.7167	1.4503	1.2727	1.2283	2.11
2	1.19318	1.18624	1.18182	1.17694	2.5455	2.2199	2.0127	1.8351	4.69
3	1.20563	1.20005	1.19563	1.18905	2.2791	2.0127	1.7759	1.6575	6.01
4	1.19800	1.19440	1.18699	1.18485	2.4271	2.3087	2.0719	1.9535	7.45

Studies have shown that with an increase in the temperature of the medium from 10 to 45 °C, the density and viscosity of the solution decrease in direct proportion, i.e. 1.19800–1.18485 g/cm³ and 2.4271–1.9535 mm²/s, respectively. (Table 5).

CONCLUSION

As a result of the conducted research, it was established that spent catalysts of the nitrogen industry of the brand are a valuable source of macro- and microelements necessary for the production of liquid complex fertilizers. The conducted studies have shown that the spent catalysts contain a large number of useful elements (iron, copper, zinc, etc.), which can be used to obtain liquid fertilizers. Decomposition of catalysts with nitric acid of different concentrations (20–50%) revealed that with increasing acid concentration, the content of phosphorus, iron and manganese in the solution increases, but the solubility of potassium, calcium, boron and molybdenum decreases; at an acid concentration of 40–50%, the composition of the solutions stabilizes. Rheological studies showed that with an increase in temperature from 10 to 45 °C, the density and viscosity of the solutions decrease, which improves the filtration condition.

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