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STUDY OF THE PROCESS OF OBTAINING CARBON-HUMIC BENTONITE FERTILIZERS

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Abstract

Mixing brown coal from the Angren deposit, oxidised in an optimal mode with a mixture of nitric and sulphuric acids, with bentonite (from the Azkamar and Navbahor deposits) is a promising method for obtaining highly effective carbon-humic bentonite fertilizer. With a ratio of organic coal to bentonite from the Azkamar deposit of 100:10, a carbon-humic bentonite fertilizer was obtained containing 3.14% nitrogen, 73.20% organic matter, 54.92% humic acids, with a granule strength of 2.28 MPa and a moisture content of 5.2%, which does not cake and retains its friability even at higher moisture levels.

Keywords: *coal, bentonite, humic acids, carbon-humic bentonite fertilizer, granule strength*

Introduction

About 50% of the world's food production relies on synthetic fertilizer application, hence to meet food production, its use has been increased tremendously during the last 4 to 5 decades (FAO, 2021). Inorganic fertilizers are frequently used to increase soil fertility and crop yield. However, they are not a permanent solution to tackle soil infertility. The long-term application of mineral fertilizers into soils with a low percentage of organic compounds accelerates the mineralization of soil organic matter (SOM) and disrupts the natural metabolic processes of soil organisms. This leads to a reduction in the SOM content, nutritional imbalance, and soil acidification. Therefore, natural amendments, such as bentonite and its combination with organics, could be useful tools to maintain or increase the SOM in a sustainable way (Hernández, T. et al., 2016).

Bentonite is classified as an agronomic ore. Abroad, bentonites and preparations made from them are used in agriculture: as fillers in pesticides to combat agricultural pests; as additives to sandy and other low-fertility soils to improve their agronomic properties; as diluents and accumulators of mineral fertilizers to reduce their harmful effects on soil biocenoses, to prevent the leaching of fertilizers from the soil and, as a result, to prevent the contamination of groundwater with mineral salts; in the production of liquid complex fertilizers as suspending and stabilising agents (Mi J. et al., 2020; Umar W. et al., 2022; Eslek Koyuncu D. D. et al., 2024)

Bentonite is recommended for use on light (sandy and sandy loam) soils. It improves their water-physical and thermal properties, increases maximum moisture capacity, and reduces the water permeability of sandy soils. As a result, the living conditions of soil microorganisms and the nutrition of cultivated plants are improved. The high sorption capacity of bentonite prevents the leaching of fertilizers from the soil (Mi J. et al., 2020). Bentonite clay composite has also been evaluated as a tool for the remediation of heavy metal-contaminated soil (Myasnikov, S. K. et al., 2016).

Environmental studies by American scientists have shown that bentonite prevents the spread of phytopathogenic microorganisms in the soil. The use of bentonite provides high economic efficiency in agriculture (Agafonov E. V., Khovansky M. V., 2014). About 200 deposits and occurrences of bentonite clays have been identified in Uzbekistan, with estimated reserves of more than 2 billion tonnes (Avliyakulov A. et al., 2003).

Research method

We decided to combine the high agrochemical properties of carbon-humic compounds (oxidised carbon) with the equally high agrochemical properties of bentonite clays to obtain carbon-humic bentonite fertilizers.

In our work, we used bentonite clays from three deposits in Uzbekistan: Azkamar, Navbahor and Lagon. Their composition is shown in Table 1.

Table 1. *Chemical composition of bentonites*

No.	Bentonite deposit	CO_2	SiO_2	TiO_2	Al_2O_3	Fe_2O_3	MgO	MnO	CaO	Na_2O	K_2O	P_2O_5	SO_3	H_2O
1.	Azkamar	3.41	50.34	0.73	15.21	5.67	2.3	0.03	4.76	2.31	2.36	0.13	1.48	5.42
2.	Navbahor	9.35	46.06	0.39	8.78	3	4.33	0.27	12.2	0.75	1.05	0.77	1.39	6
3.	Lagon	2.97	49.73	0.73	14.74	5.57	4.45	0.02	2.26	2.15	4.75	0.1	0.57	3.8

To obtain oxidised coal, a representative sample of BOMSSH grade coal fine from the Angren deposit, which, after drying to an air-dry state and grinding in a ball mill to a size of 0.25 mm, had the following composition (wt.%): moisture 14.1; ash 13.7; organic matter 72.2; humic acids 4.1% of organic mass. Nitric acid was taken from Maxam-Chirchik JSC with a concentration of 59%. The initial sulphuric acid had a concentration of 93%. The oxidation process was carried out at a nitric acid concentration of 5 to 30%, a temperature of 40 to 80 $^{\circ}$ C, a duration of 2 hours and a weight ratio of coal: HNO3 equal to 1:2. Sulphuric acid was added to nitric acid in such an amount that its concentration in the nitric acid solution was between 2.5 and 20%. The experiments were carried out in a glass cylindrical reactor equipped with a thermostatic jacket and a screw stirrer. The acid mixture was poured into the reactor, the set temperature was set, the stirrer was turned on, and a sample of brown coal was loaded. At the end of the process, the reaction mass was separated into liquid and solid phases. The solid phase was washed with distilled water from acids to a neutral reaction, dried to an air-dry state, and its ash content, moisture content, organic matter, and yield of humic and fulvic acids were determined. The ash content was determined according to GOST 11022–75, the moisture content according to GOST 11014–70, the organic matter according to the difference between the ash and the sum of the percentage contents of ash and moisture, and the yield of humic acids according to GOST 9517–76 (Zhumanova M. O. et al. 2009).

Nitrogen oxides released during the oxidation of brown coal with a mixture of nitric and sulphuric acids were identified. Using a water jet pump, they were passed through absorbers with a 0.5 N NaOH solution. All solutions were collected together, brought to the 500 ml mark, from where an aliquot was taken to determine nitrogen according to Kjeldahl (Pozin M. E., 1980).

Results and discussion

After oxidising brown coal from the Angren deposit with a mixture of nitric and sulphuric acids, the resulting mass was separated into solid and liquid phases by filtration. The liquid phase was used to dilute the initial 59% nitric acid. The wet solid phase was ammoniated to a pH of 3.5–4.0.

Initially, we studied the process of producing granular carbon-humic fertilizers by adding finely ground bentonite from various deposits to the wet solid phase of oxidised coal without washing. The mass ratio of the organic part of oxidised coal to bentonite clay (OU : BG) varied in the range of 100: (3–15). The suspension was granulated in a porcelain cup by rolling. The product was then dried in a thermostat at 80 °C. The chemical composition of the obtained carbon-humic fertilizers with the addition of bentonite clays is given in Table 2.

Table 2. *Chemical composition of carbon-humic bentonite fertilizers obtained from oxidised coal without washing*

Ratio of organic carbon to bentonite	N, %	Organic matter, %	Humic acids, %	Moisture, %
When using Lagon bentonite				
100: 3	6.22	75.53	56.81	5.33
100: 7	5.98	66.25	53.07	5.18
100: 10	5.75	63.23	51.59	5.26
100: 15	5.39	60.17	48.61	5.64
When using Navbahor bentonite				
100: 3	6.32	70.57	56.17	5.22
100: 7	5.95	66.94	53.21	5.58
100: 10	5.76	64.77	51.43	5.50
100: 15	5.53	59.06	48.19	5.47
When using Azkamar bentonite				
100: 3	6.23	75.22	55.58	5.74
100: 7	5.87	71.73	53.95	5.48
100: 10	5.63	69.05	51.92	5.56
100: 15	5.39	65.81	48.78	5.44

However, the nitrogen content in the fertilizer is high. Therefore, in order to reduce the nitrogen content in fertilizers, processes for obtaining organic fertilizers were investigated by twice washing oxidised coal with water at a weight ratio of organic coal to H_2O of 1:0.28, then adding bentonite to the wet product and granulating it by rolling.

The wet product was then dried. The composition of the fertilizers is shown in Tables 3.

Table 3. *Chemical composition of carbon-humic bentonite fertilizers obtained on the basis of twice-washed solid phase products of coal oxidation*

Ratio of organic carbon to bentonite	N, %	Organic matter, %	Humic acids, %	Moisture, %
When using Navbahor bentonite				
100: 5	5.35	69.96	52.73	5.71
100: 10	5.00	66.41	50.10	5.26
100: 15	4.74	60.15	47.01	5.60
When using Azkamar bentonite				
100: 5	5.23	75.69	56.30	5.71
100: 10	4.90	72.88	53.03	5.22
100: 15	4.76	68.05	50.74	5.37

In the composition of the carbon-humic bentonite fertilizers obtained by double washing of the solid phase of oxidised coal, the reduction in nitrogen content compared to the unwashed variant is only about 1%. Therefore, to achieve a significant reduction in nitrogen content, the coal oxidation products were subjected to triple washing.

The triple washing of the oxidation products was carried out periodically. The filtration of the nitric acid coal pulp was carried out on a Buchner funnel under vacuum.

For the first washing of the oxidation products, a mixture of HNO3 washing solutions with a concentration of 21.3% obtained during the second and third washing stages was used. The mixture of filtrates obtained after separation of the oxidation product and the first stage of washing with a content of 27.1% HNO3 is sent to the technological cycle for dilution of the initial HNO3. The oxidised part of the coal after threefold washing was mixed with bentonite. The weight ratio of the organic part of coal to bentonite is 100:(3–15). Next, the resulting wet product was granulated, dried, and the chemical composition and strength of the granules were determined. The results are shown in Table 4.

Table 4. *Chemical composition and strength of carbon-humic bentonite fertilizer granules obtained from triple-washed solid phase coal oxidation products*

Ratio of organic carbon to bentonite	N, %	Organic matter, %	Humic acids, %	Moisture, %	Granule strength, MPa
When using Navbahor bentonite					
100:5	3. 65	71.60	54.03	5.06	2.01
100:10	3.16	67.07	50.48	5.15	2.34
100:15	3.08	62.96	47.04	5.19	2.59
When using Azkamar bentonite					
100:5	3.75	76.53	57.61	5.01	2.09
100:10	3.14	73. 20	54.92	5.20	2.28
100:15	3.02	69.45	52.01	5.18	2.65

The data shows that the nitrogen content decreased from 5.63 to 3.14%, while the organic matter content in the fertilizer was 73.20% and the humic acid content was 54.92%. The strength of the granules reached 2.28 MPa.

Conclusion

Based on laboratory studies and a series of experiments conducted on a large-scale laboratory setup, the main technological parameters of the process have been developed.

Main technological parameters of the process.

Particle size of crushed coal, mm –0,25––0,5; coal moisture content –12–15%; nitric acid concentration-30%; sulphuric acid concentration-92,5–93,0%; sulphuric acid concentration in 30% nitric acid –5%; weight ratio of coal (organic part): HNO3 (mg) acid –1: 2; oxidation temperature, °C-35–40; oxidation duration –120 min; concentration of HNO3 formed after the first washing-27,4%; mass ratio of organic part of coal: H_2O at the second and third washing stages –1: 0,2; neutralisation temperature, °C– 60; pH value of ammoniated coal pulp –3,5–4,0; mass ratio of organic coal component: bentonite – 100: 10; mixing time, min –15; temperature of furnace gases at the inlet to the drying drum, °C -.400–500; gas temperature at the outlet of the drying drum, °C – 85–100; product temperature, °C- 80–90. Fertilizer composition, %: Total N-3,14; organic matter –73,20; humic acids- 54,92; total nutrients, (N+organic matter) –76,34.

Thus, by oxidising brown coal from the Angren deposit with nitric acid in the presence of sulphuric acid, followed by triple washing of the solid phase of the oxidised coal and mixing with bentonite clays, drying and granulation, it is possible to obtain granular carbon-humic bentonite fertilizers with a high humic substance content.

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