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STUDY OF THE CHEMICAL AND MINERALOGICAL COMPOSITION OF THE COMPLEX-MIXED FERTILIZERS OBTAINED ON THE BASIS OF VERMICULITE AND SELECTION OF THE OPTIMAL COMPOSITION

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ABSTRACT

This paper investigates the possibility of obtaining multicomponent complex mixed fertilizers based on local mineral raw materials—Karakalpakstan vermiculite—and fertilizer salts (ammophos and urea). The vermiculite content in the fertilizer was varied at 40, 50, and 60%, while the ratio of nutrient components was adjusted within the range of N: P_2O_5= 2:1 to 1:2 to synthesize 15 distinct samples using a pan (disc) granulator. The chemical composition, pH, granulometric distribution, and mechanical strength of the resulting granules were systematically evaluated. Based on a comprehensive evaluation of agrochemical and physico-mechanical properties, sample No. 4 was selected as the optimal composition. This sample—containing 40% vermiculite at an ammophos:urea mass ratio of 42.67 : 17.33 (N:P_2O_5 = 1:1.5)—exhibits a high total P_2O_5 content (17.392%), the highest total CaO content (8.978%), maximum granule mechanical strength (3.2 MPa), and a high yield of the commercially demanded 2–4 mm fraction (73%). SEM-EDS characterization of the optimal sample confirmed that the material retains its original vermiculite Mg–Al–Si matrix while being successfully modified with essential nutrient components (N, P, K). The developed formulation shows great potential as a multifunctional, slow-release fertilizer-ameliorant suitable for the chemical reclamation of saline and sodic soils in the Aral Sea region.

Introduction

The rational involvement of local mineral raw materials in the production of multicomponent fertilizers is one of the priority directions for developing the agrochemical base of the Republic. Vermiculite — a hydrated magnesium–aluminum–iron layered silicate that is widely distributed in Karakalpakstan — possesses a high cation-exchange capacity, a developed layered structure and a pronounced water-retention capacity, which makes it an attractive carrier and structure-forming component for slow-release complex fertilizers. The present work examines the possibility of obtaining complex vermiculite-containing fertilizers by granulating mineral salts in a pan (disc) granulator, and evaluates their chemical composition, granulometric distribution and mechanical strength in order to select the optimal composition.

Materials and Methods

Commercial ammophos and urea (carbamide) produced by the chemical enterprises of the Republic were used as the source nutrient components. Prior to granulation, the granulated mineral fertilizers were ground in a porcelain mortar to a powdered state. The powdered components were then mixed with vermiculite at various mass ratios of vermiculite : ammophos : urea, as listed in Table 1. The mixtures were homogenized thoroughly and moistened with water in an amount of 8–17.7 wt.% of the total mass of the mixture, after which the prepared mass was granulated in a pan granulator.

The resulting complex vermiculite-containing granules were comparable in appearance to standard, industrially produced granular fertilizers. The obtained samples were subjected to chemical analysis with determination of nitrogen (N), phosphorus (expressed as P_2O_5), potassium (expressed as K_2O) and sulfate ions (SO_4^{2-}) by conventional methods. The mechanical strength of the granules and their granulometric composition were also determined. The microstructure and elemental composition of the selected optimal sample were examined by scanning electron microscopy with energy-dispersive X-ray spectroscopy (SEM-EDS).

Results and Discussion

The composition together with the main physico-chemical and commercial characteristics of the obtained fertilizers are summarized in Table 1. The vermiculite content was varied at 40, 50 and 60%, while the ratio of nutrient components was changed within the range $N:P_2O_5$ = 2:1 to 1:2.

Table 1. *Composition of the complex-mixed fertilizers obtained on the basis of Karakalpakstan vermiculites*

No.	Vermiculite, %	Ammophos	Urea	$N:P_2O_5$	pH	P_2O_5 total, %	P_2O_5 water-sol., %	CaO total, %	CaO water-sol., %	< 2 mm, %	2–4 mm, %	> 4 mm, %	Strength, MPa
1	40	21.9	38.1	2:1	5.8	7.835	4.181	3.180	0.517	17	65	18	2.1
2	40	26.78	33.22	1.5:1	5.6	9.251	4.706	4.078	0.585	6	77	17	2.3
3	40	34.5	25.5	1:1	5.2	15.507	6.414	7.847	0.867	14	71	15	2.7
4	40	42.67	17.33	1:1.5	4.9	17.392	7.675	8.978	1.211	9	73	18	3.2
5	40	47.6	12.4	1:2	4.4	18.555	8.498	3.578	1.287	7	75	18	3.2
6	50	18.25	31.75	2:1	6.0	8.395	60	2.782	1.124	17	65	18	1.8
7	50	22.31	27.69	1.5:1	5.7	10.262	61	2.924	1.562	6	77	17	2.0
8	50	28.75	21.25	1:1	5.4	13.225	63	5.624	2.245	14	71	15	2.1
9	50	35.55	14.45	1:1.5	5.1	16.353	65	6.224	2.865	9	73	18	2.2
10	50	39.67	10.33	1:2	5.0	18.248	65	8.325	3.542	7	75	18	2.4
11	60	14.6	25.4	2:1	6.2	6.221	5.396	2.152	0.796	23	55	22	1.5
12	60	17.85	22.15	1.5:1	6.1	8.156	5.954	1.855	0.657	21	60	19	1.7
13	60	23	17	1:1	5.7	10.865	6.536	1.481	0.586	7	68	25	1.9

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14	60	28.45	11.55	1:1.5	5.4	11.607	6.935	1.657	0.518	12	77	11	2.0
15	60	31.73	8.27	1:2	5.5	13.497	7.809	1.393	0.451	8	83	9	2.1

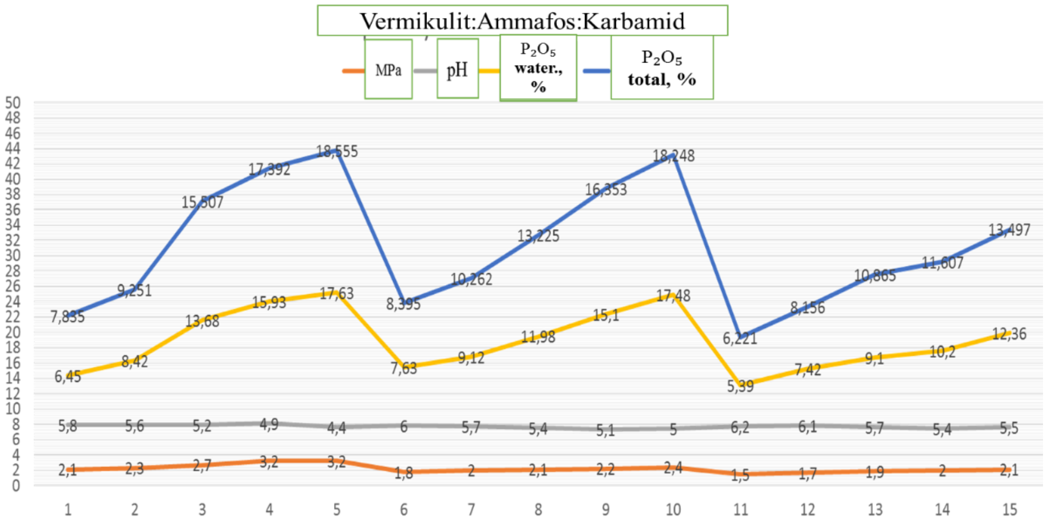

Fig. 1. Graphical interpretation of the results obtained for the complex vermiculite-containing fertilizers (vermiculite : ammophos : urea series).

The obtained results show that an increase in the ammophos fraction in the composition is accompanied by an increase in both total and water-soluble phosphorus. Thus, at a vermiculite content of 40% and on going from the ratio N:P_2O_5 = 2:1 to 1:2, the total P_2O_5 content increases from 7.835 to 18.555%, while water-soluble P_2O_5 rises from 4.181 to 8.498%. A similar tendency is observed for the samples containing 50 and 60% vermiculite.

Variation of the component composition also affects the acidity of the medium. The pH values of the studied samples lie in the range 4.4–6.2. As the ammophos fraction increases, a certain decrease in pH is observed, which is associated with the physiological acidity of the phosphorus-containing components.

The total and water-soluble CaO content varies depending on the ratio of the initial components. For the samples containing 40 and 50% vermiculite, an increase in the ammophos fraction promotes a rise in the calcium content, whereas at 60% vermiculite the CaO values remain comparatively low. This is attributed to the diluting effect of vermiculite and the corresponding decrease in the concentration of mineral components in the system.

Granulometric analysis showed that the bulk of the granules belongs to the 2–4 mm fraction, which is the most technologically demanded size for fertilizer application. The content of this fraction amounts to 55–83%. The highest yield of the commercial fraction is observed for the samples with an elevated vermiculite content (60%) and the ratio N:P_2O_5 = 1:2, where the proportion of 2–4 mm granules reaches 83%.

The granule strength varies within 1.5–3.2 MPa and depends both on the vermiculite content and on the ratio of the mineral components. The highest mechanical strength is characteristic of the samples with 40% vermiculite and an elevated ammophos content (samples No. 4 and No. 5), where the strength reaches 3.2 MPa. An increase in the vermiculite fraction to 60% leads to a certain decrease in the strength of the granules owing to the higher porosity of the composition.

Selection of the Optimal Composition

A comparative evaluation of the entire series of samples (Table 3.4.1) makes it possible to select sample No. 4 as the optimal composition. This sample — containing 40% vermiculite at a mass ratio ammophos : urea = 42.67 : 17.33 ($N:P_2O_5$ = 1:1.5) — combines the most favorable balance of agrochemical and physico-mechanical properties:

– a high content of total (17.392%) and water-soluble (7.675%) P_2O_5, close to the maximum values recorded for the whole series;

– the highest total CaO content among all the studied compositions (8.978%), which is particularly valuable for the chemical amelioration of the saline and sodic soils of the Aral Sea region, where exchangeable Ca^{2+} displaces Na^+ from the soil-absorbing complex;

– the maximum mechanical strength of the granules (3.2 MPa), ensuring resistance to attrition during storage, transportation and field application;

– a high yield of the commercial 2–4 mm fraction (73%);

– a moderately acidic reaction of the medium (pH 4.9), which is less aggressive than that of sample No. 5 while providing comparable phosphorus availability.

Although sample No. 5 exhibits a marginally higher P_2O_5 content (18.555%) at the same strength level (3.2 MPa), it is characterized by a substantially lower total CaO content (3.578% versus 8.978%) and a more pronounced acidity (pH 4.4). Therefore, sample No. 4 provides the most balanced combination of nutrient supply, calcium content and mechanical stability, and was selected as the optimal variant for further microstructural and mineralogical characterization.

Microstructural and Mineralogical Characterization of the Optimal Sample

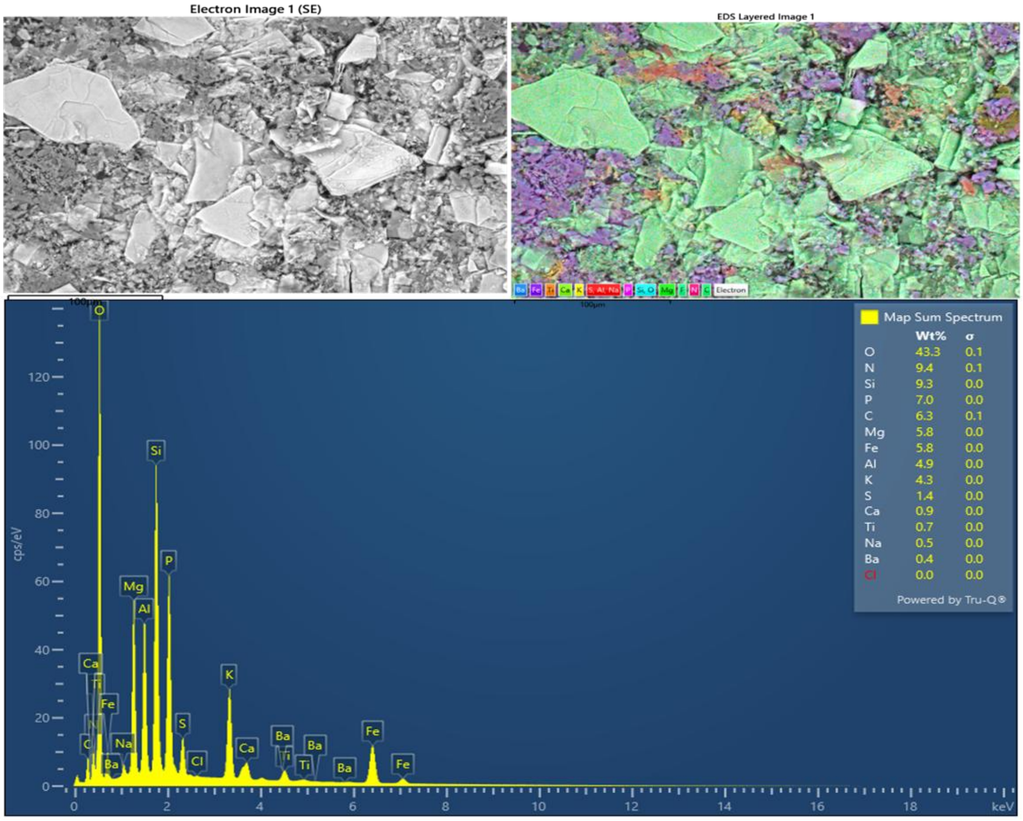

Fig. 2. SEM-EDS analysis of the optimal vermiculite-containing fertilizer (Table 1, sample No. 4): secondary-electron image, EDS layered map and map-sum spectrum.

The microstructure and elemental composition of the optimal sample (No. 4) were studied by SEM-EDS (Fig. 2). The secondary-electron image reveals the characteristic lamellar, plate-like particles of vermiculite embedded in a finely dispersed mineral matrix, while the EDS layered map confirms a uniform distribution of the principal elements. According to the map-sum spectrum, the material is based on a magnesium–aluminosilicate matrix, the main elements being oxygen (43.3%), nitrogen (9.4%), silicon (9.3%), phosphorus (7.0%), magnesium (5.8%), iron (5.8%), aluminum (4.9%) and potassium (4.3%), with minor amounts of sulfur, calcium, titanium, sodium and barium.

The presence of the Mg–Al–Si system confirms the retention of the vermiculite phase, while the elevated phosphorus, nitrogen and potassium contents indicate successful modification of the mineral with nutrient components. The combination of the layered silicate matrix with the introduced nutrients endows the material with a potential for the prolonged (slow) release of plant nutrients, which allows it to be regarded as a promising multifunctional mineral fertilizer.

Conclusion

Complex vermiculite-containing fertilizers with satisfactory agrochemical and physico-mechanical properties were obtained by granulating ammophos and urea with Karakalpakstan vermiculite in a pan granulator. The composition of the initial mixture was shown to govern the phosphorus and calcium contents, the acidity, the granulometric distribution and the mechanical strength of the product. On the basis of a comprehensive comparison of these characteristics, the composition containing 40% vermiculite at the ratio $N:P_2O_5$ = 1:1.5 (sample No. 4) was selected as optimal, combining a near-maximum phosphorus content, the highest calcium content and the maximum granule strength. SEM-EDS characterization confirmed that this sample retains the vermiculite Mg–Al–Si matrix modified with phosphorus, nitrogen and potassium, making it a promising multifunctional, slow-release fertilizer-ameliorant for the saline soils of the Aral Sea region.

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