



**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 study investigates the technology of obtaining multifunctional, multicomponent complex-mixed organomineral fertilizers based on local mineral and secondary raw materials—namely vermiculite, biohumus, and phosphogypsum—combined with mineral salts (ammophos and urea). In the first series of experiments, the effect of varying phosphogypsum and vermiculite contents (from 6 to 28%) on the fertilizer properties was evaluated, while the second series analyzed the transition dynamics and ratios between the organic (biohumus) and mineral phases. Based on a comprehensive evaluation of agrochemical and physico-mechanical properties, samples No. 6 and No. 7 from the first series were selected as the optimal compositions. These optimal formulations exhibit high nutrient concentrations (total P_2O_5 11.2–12.3%, K_2O 2.42–2.75%, total nitrogen 10.2–14.3%), an adequate supply of calcium and sulfur derived from phosphogypsum (CaO 7.2–12.52%, SO_3 15.2–25.32%), and exceptionally high phosphorus bioavailability with citrate-soluble P_2O_5 degrees reaching 97.76–98.84%. Furthermore, these samples achieved maximum granule mechanical strength (2.4–2.6 MPa) and a high commercial yield of the 2–4 mm fraction (78–80%). SEM-EDS and TGA/DSC characterization confirmed that the optimal samples consist of a polymineral vermiculite aluminosilicate (Mg–Al–Si) matrix successfully modified with essential nutrients (P, K, Ca, Mg, S) and exhibit a multi-stage thermal decomposition behavior with structural stability maintained up to 550–600 °C. The synthesized organomineral fertilizers demonstrate great potential as



multifunctional, slow-release fertilizer-ameliorants for restoring soil fertility in the degraded and saline soils of the Aral Sea region.

Introduction

The development of multifunctional organomineral fertilizers based on local mineral and secondary raw materials is one of the priority directions for the rational use of natural resources of the Republic and for improving the fertility of the degraded, saline soils of the Aral Sea region. In the present work, complex organomineral fertilizers were obtained by granulating biohumus, phosphogypsum and vermiculite together with the mineral components ammophos and urea. Such compositions combine an organic and a mineral part and thereby provide complex (balanced) plant nutrition. Vermiculite — a hydrated magnesium-aluminum layered silicate — acts as a structure-forming and prolonging matrix, phosphogypsum serves as a source of calcium and sulfur and as an ameliorant, while biohumus supplies organic matter and biogenic components. The aim of the study was to examine the chemical and mineralogical composition of the obtained fertilizers, to establish the effect of the component ratio on their properties, and to select the optimal composition.

Materials and Methods

Biohumus, phosphogypsum and vermiculite were used together with commercial ammophos and urea (carbamide) as the source components. Two series of experiments were carried

out. In the first series (Table 1), the contents of biohumus, phosphogypsum, vermiculite, ammophos and urea were varied. In the second series (Table 2), the ratio between the organic (biohumus) and the mineral part was varied during the transition from a purely organic fertilizer to organomineral compositions with a high mineral content. The components were mixed in the corresponding mass ratios, homogenized, moistened with water and granulated.

In the obtained granules, the pH, total and citrate-soluble P_2O_5 , total CaO, K_2O , total SO_3 , organic matter and total nitrogen contents were determined by conventional methods, together with the granulometric composition and the mechanical strength of the granules. The microstructure, elemental composition and thermal behavior of the selected optimal samples were investigated by scanning electron microscopy with energy-dispersive X-ray spectroscopy (SEM-

EDS) and by combined thermogravimetric / differential scanning calorimetric analysis (TGA/DSC).

Results and Discussion

The composition together with the main physico-chemical and commercial characteristics of the obtained organomineral fertilizers of the first series is summarized in Table 1.



Table 1. Effect of the on the properties of the organomineral phosphogypsum and vermiculite content fertilizers

No.	Biohumus	Phosphogypsum	Vermiculite	Ammophos	Urea	pH	P ₂ O ₅ total, %	P ₂ O ₅ citrate-sol., %	CaO total, %	K ₂ O, %	SO ₃ total, %	Organic, %	N total, %	< 2 mm, %	2-4 mm, %	> 4 mm, %	Strength, MPa
1	42.5	42.5	6	6.4	2.6	5.1	2.75	96.68	21.35	0.12	31.8	19	2.4	21	63	16	1.3
2	58.12	19.38	9	9.6	3.9	5.3	4.6	94.88	10.32	0.35	21.32	25	4	18	70	22	1.2
3	32.5	32.5	14	14.94	6.06	4.8	6.9	95.35	13.25	0.41	29.32	15	5	12	75	13	1.4
4	41.25	13.75	18	19.2	7.8	5.3	9.6	97.56	8.2	1.01	18.35	21	5.6	11	73	16	1.3
5	14.25	42.75	17.2	18.34	7.46	4.1	9.7	98.98	18.4	1.02	32.8	5	6.4	18	68	14	1.9
6	20	20	24	25.6	10.4	4.6	11.2	97.76	12.52	2.42	25.32	8	14.3	9	80	11	2.6
7	22.5	7.5	28	29.88	12.12	5.5	12.3	98.84	7.2	2.75	15.2	11	10.2	8	78	14	2.4

As the vermiculite content increases from 6 to 28%, the content of total P₂O₅ rises from 2.75 to 12.3%, the K₂O content increases from 0.12 to 2.75%, and the total nitrogen content also grows, reaching 14.3% in sample No. 6. The mechanical strength of the granules increases up to 2.6 MPa. Vermiculite improves the structure formation of the granules, enhances their water-retention capacity, promotes the retention of ammonium and potassium ions, and increases the strength of the granules owing to its layered structure; in addition, it acts as a prolonging matrix for the nutrient elements.

With increasing phosphogypsum content, the CaO and SO₃ contents grow and a stabilization of pH within the range 4.1–5.5 is observed. Phosphogypsum is a source of calcium and sulfur; it improves the agrophysical properties, reduces alkalinity and promotes the aggregation of soil particles. The maximum SO₃ content is recorded for the mineral-rich compositions, which indicates a high sulfur-supplying capacity of the fertilizer.

All compositions are characterized by a high content of assimilable phosphorus: the degree of citrate-soluble P₂O₅ amounts to 94.88–98.98%. This

points to a high bioavailability of phosphorus, a good solubility of the phosphate phase and the efficiency of ammophos as a phosphorus source; the highest value (98.98%) is observed for sample No. 5.

The organic-matter content decreases as the share of biohumus is reduced. The bulk of the granules belongs to the 2–4 mm fraction, which meets the requirements for commercial granular fertilizers; the maximum yield of the target fraction (78–80%) is observed for samples No. 6 and No. 7. The granule strength grows with increasing vermiculite and mineral-phase content, the highest values being recorded for sample No. 6 (2.6 MPa) and sample No. 7 (2.4 MPa). A high strength reduces dust formation, increases the resistance to transportation and improves the storage stability of the granules.

In the second series of experiments (Table 2), the effect of the biohumus-to-mineral-part ratio was studied during the transition from an organic fertilizer to organomineral compositions with a high mineral content.

Table 2. Effect of the ratio between biohumus and the mineral part

No.	Organic: mineral ratio	Biohumus	Vermiculite	Ammophos	Urea	pH	P ₂ O ₅ total, %	P ₂ O ₅ citrate-sol., %	N total, %	K ₂ O, %	Organic, %	Strength, MPa
1	100 : 0	100	0	0	0	7.0	1.5	86.37	1.2	0	41	0



2	75 : 25	75	10	10.67	4.33	6.2	3.8	91.56	4	0.42	32	0
3	50 : 50	50	20	21.34	8.66	5.4	7.8	98.98	6.8	1.42	21	0.5
4	25 : 75	25	30	32	13	5.1	12.1	97.76	8.6	1.72	10.6	0.9

As the mineral component increases, the pH decreases from 7.0 to 5.1; this acidification is associated with the presence of ammophos, the hydrolysis of ammonium compounds and the reduction of the organic phase. The pH range of 5.1–6.2 is favorable for most agricultural crops. The total P₂O₅ content increases from 1.5 to 12.1%, while the share of citrate-soluble phosphorus simultaneously rises (86.37–97.76%); thus the mineral component substantially raises both the concentration of phosphorus and its availability to plants. The nitrogen content increases from 1.2 to 8.6% owing

to the larger proportion of urea and ammophos, the K₂O content grows from 0 to 1.72% owing to the introduction of vermiculite, and the organic-matter content regularly decreases from 41 to 10.6%. The mechanical strength increases from 0 to 0.9 MPa, since the mineral components improve granulation, increase the granule density and reduce deformation. The compositions with organic-to-mineral ratios of 50:50 and 25:75 are characterized by the most alanced agrochemical properties and a high availability of nutrients.

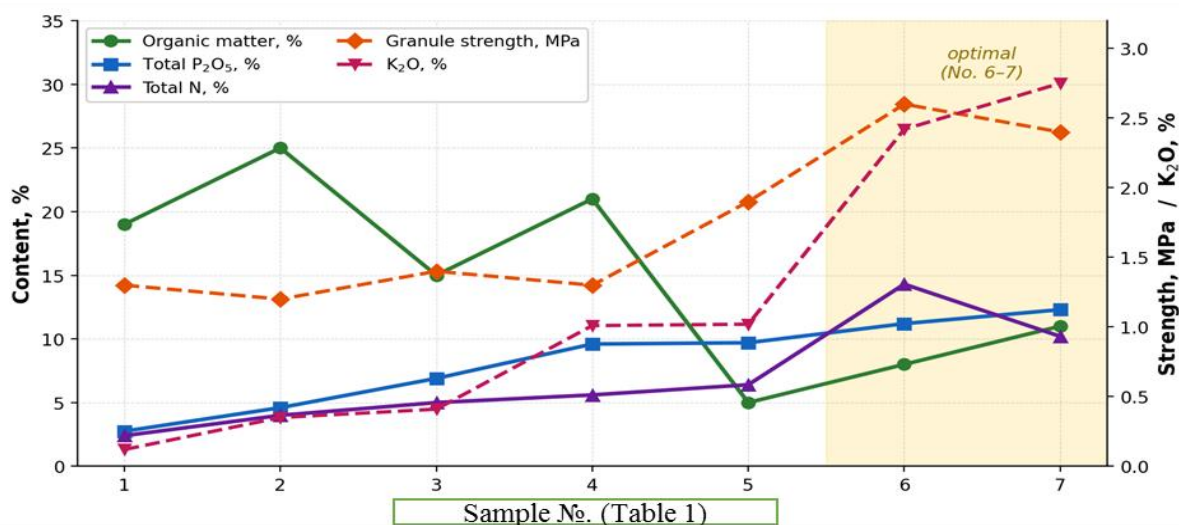


Fig. 1. Graphical analysis of the correlation between the organic and the mineral phases (Table 1): inverse relationship between the organic-matter content and the contents of available nutrients (P₂O₅, N, K₂O) and the granule strength.

Selection of the Optimal Composition

A comparative evaluation of the whole series (Table 1) shows that samples No. 6 and No. 7 — containing the highest vermiculite contents (24 and 28%, respectively) — provide the most favorable combination of agrochemical and physico-mechanical properties and were therefore selected as the optimal variants. Their advantages are as follows:



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-the highest total P_2O_5 content (11.2 and 12.3%) combined with a very high degree of citrate-soluble (assimilable) phosphorus (97.76 and 98.84%), which ensures maximum availability of phosphorus to plants;

-the highest K_2O contents in the series (2.42 and 2.75%) and high total nitrogen contents (14.3 and 10.2%), provided by the combined action of vermiculite, ammophos and urea;

-a sufficient supply of calcium and sulfur from phosphogypsum (CaO 12.52 and 7.2%; SO_3 25.32 and 15.2%), which is important for the chemical amelioration of saline and sodic soils, where Ca^{2+} displaces Na^+ from the soil-absorbing complex;

-the maximum mechanical strength of the granules (2.6 and 2.4 MPa) and the highest yield of the commercial 2–4 mm fraction (80 and 78%), which reduce dust formation and improve transportation and storage stability;

-a favorable, moderately acidic reaction of the medium (pH 4.6 and 5.5), suitable for most crops.

Thus, samples No. 6 and No. 7 combine a high content of available phosphorus, potassium and nitrogen, an adequate supply of calcium and sulfur,

and the best physico-mechanical characteristics. These two compositions were therefore selected for detailed microstructural, elemental and thermal characterization.

Microstructural, Elemental and Thermal Characterization of the Optimal Samples

Sample No. 6. The microstructure and elemental composition of sample No. 6 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 organomineral matrix, while the EDS layered map confirms a uniform distribution of the elements. According to the map-sum spectrum, the material has a predominantly aluminosilicate nature: the main elements are oxygen (46.3%), nitrogen (14.3%), sulfur (7.3%), calcium (6.9%), silicon (5.5%) and phosphorus (5.1%), together with magnesium (3.3%), iron (3.1%), aluminum (2.9%) and potassium (2.3%). The Mg–Al–Si system is typical of the layered silicates of the vermiculite group, while the elevated nitrogen, sulfur, calcium, phosphorus and potassium contents confirm the fertilizing functionality of the material.

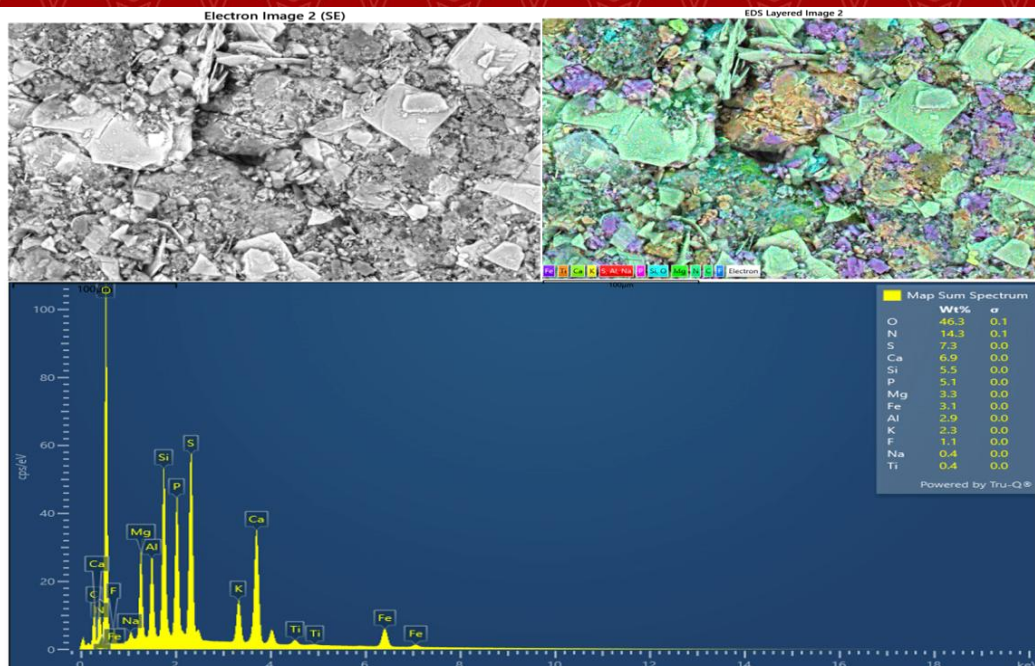


Fig. 2. SEM-EDS analysis of the vermiculite-containing fertilizer (Table 1, sample No. 6): secondary-electron image, EDS layered map and map-sum spectrum.

The combined TGA/DSC analysis of sample No. 6 (Fig. 3) reveals a multi-stage thermal decomposition. The first stage (145–266 °C) is accompanied by a mass loss of 22.88% and is caused by the removal of adsorbed and interlayer water and volatile components. The second stage (266–531 °C, mass loss 20.69%) corresponds to the main

destruction of the organic matrix. The third stage (560–810 °C, mass loss 10.03%) is associated with the decomposition of thermally stable inorganic and carbonaceous structures. The DSC curve shows thermal effects at 138.8, 240.3, 376, 662 and 803 °C, reflecting successive dehydration, thermal destruction and high-temperature phase transformations; the residual mass at 1000 °C is about 46%, which indicates a high proportion of a heat-resistant residue.

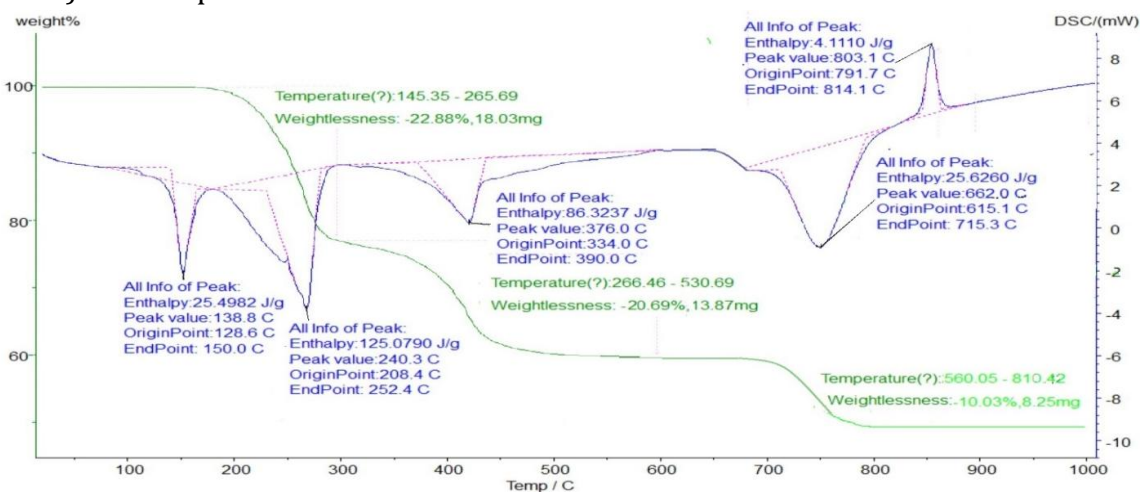


Fig. 3. TGA/DSC analysis of the vermiculite-containing fertilizer (Table 1, sample No. 6).

Sample No. 7. The EDS analysis of sample No. 7 (Fig. 3.4.6) revealed a complex multicomponent mineral composition typical of aluminosilicate agromineral materials. The matrix is dominated by oxygen (43.0 wt.%) and silicon (17.0 wt.%), which confirms the predominance of a silicate matrix and the presence of vermiculite-group minerals; the significant magnesium (4.2%) and aluminum (3.9%) contents are also consistent with the composition of magnesium-type layered hydromica aluminosilicates. The relatively high carbon content (10.2%) is related to the

organic components of the fertilizer, while phosphorus (7.9%), sulfur (7.7%), calcium (4.6%) and potassium (4.0%) represent the agronomically significant elements, probably present as phosphate and sulfate compounds and as exchangeable cations of vermiculite. Iron (4.4%) and trace amounts of fluorine, barium and sodium are attributed to the aluminosilicate structure and to natural mineral impurities of the raw material. These results confirm that the sample is a polymineral vermiculite-containing material with a pronounced silicate base and a content of biogenic macroelements (P, K, Ca, Mg, S) that determines its potential efficiency as a complex mineral fertilizer and soil-improving additive.

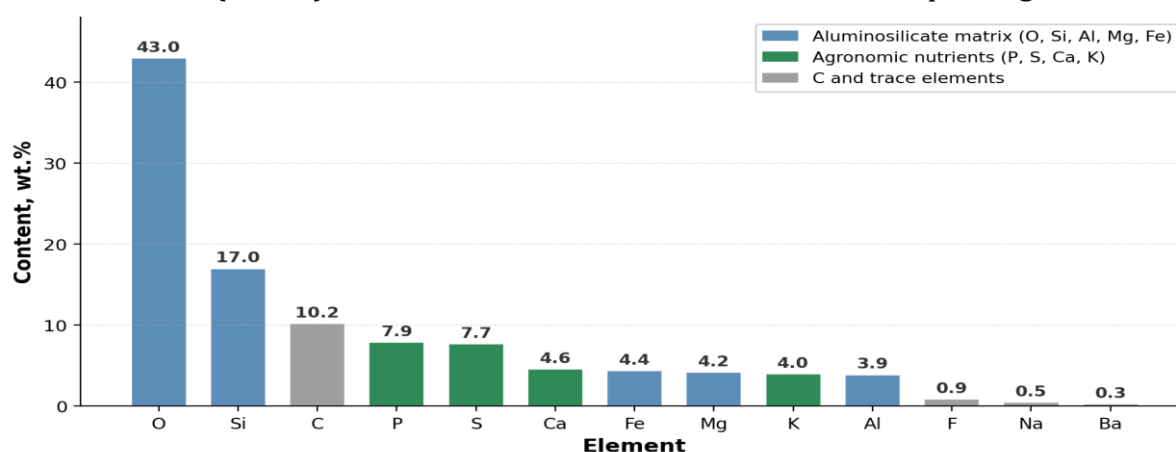


Fig. 4. Elemental composition (EDS data) of the vermiculite-containing fertilizer (Table 1, sample No. 7).

The combined TGA/DSC analysis of sample No. 7 (Fig. 5) likewise shows a multi-stage thermal behavior. In the range 89–273 °C a first mass loss of about 25.88% is observed, accompanied by endothermic effects at 141.2 and 245.0 °C and caused by the removal of physically adsorbed and interlayer water of vermiculite and by the dehydration of hydrated compounds. In the range 274–533 °C a second stage (mass loss 13.15%)

with an endothermic effect at 382 °C is associated with the removal of more firmly bound crystallization water, partial dehydroxylation of the silicate structure and the thermal decomposition of organic components. In the high-temperature region 563–816 °C a third stage (mass loss 12.03%) with an endothermic effect at 652 °C corresponds to further dehydroxylation and structural rearrangement of the aluminosilicate matrix, while a pronounced exothermic peak at about 801 °C is related to the crystallization of

new phases, densification of the structure and the formation of heat-resistant silicate compounds. The results show that the vermiculite-containing fertilizer is characterized by a multi-

stage mechanism of thermal transformations and retains a relative structural stability up to about 550–600 °C.

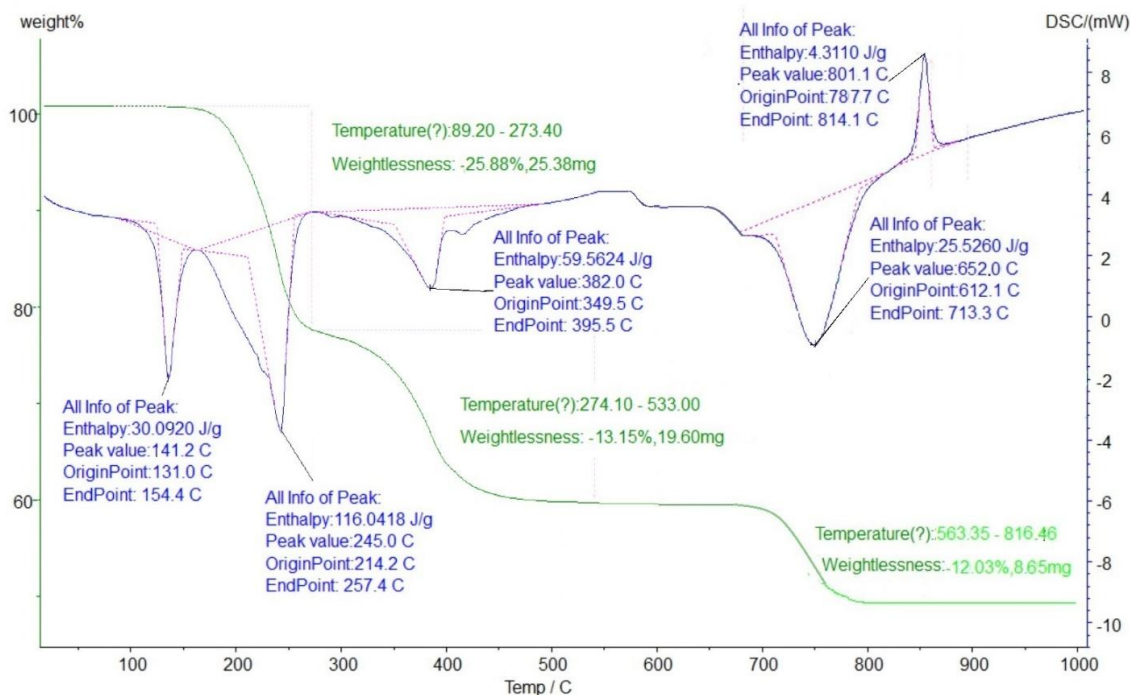


Fig. 5. TGA/DSC analysis of the vermiculite-containing fertilizer (Table 3.4.2, sample No. 7).

Conclusion

Complex organomineral fertilizers with satisfactory agrochemical and physico-mechanical properties were obtained by granulating biohumus, phosphogypsum and vermiculite together with ammophos and urea. It was established that an increase in the vermiculite and mineral-component content promotes a rise in the contents of available forms of phosphorus, potassium, sulfur and nitrogen, as well as an increase in the mechanical strength and in the yield of the commercial 2–4 mm fraction, while the organic-matter content and the pH decrease. On the basis of a comprehensive comparison of these

characteristics, samples No. 6 and No. 7 of Table 1 (24 and 28% vermiculite) were selected as the optimal compositions, combining the highest contents of available P₂O₅, K₂O and nitrogen, an adequate calcium and sulfur supply, and the maximum granule strength. SEM-EDS and TGA/DSC characterization confirmed that these samples represent polymineral vermiculite-containing materials with an aluminosilicate (Mg–Al–Si) base modified with phosphorus, sulfur, potassium and calcium, and with a multi-stage thermal behavior and structural stability up to about 550–600 °C. Such materials can be regarded as promising multifunctional, slow-release organomineral fertilizer-ameliorants for the saline soils of the Aral Sea region.



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