

# Studies of the physico-chemical properties of humic simple superphosphate obtained on the basis of Central Kyzylkum phosphorites and oxidized coal

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**Abstract.** This article presents the results of the physical and chemical properties of humic simple superphosphate obtained by decomposition of the ordinary phosphorous flour of the Central Kyzylkum with sulfuric acid and the addition of oxidized coal from the Angren deposit with hydrogen peroxide. The distinctive characteristics of humic simple superphosphate are shown in comparison with simple superphosphate obtained without the addition of oxidized coal.

## 1. Introduction

World farming experience and numerous agrochemical tests have shown that humic substances perform important functions in maintaining soil fertility, increasing the beneficial effects of mineral fertilizers and obtaining a high and high-quality crop from agricultural lands. Therefore, the receipt and use of humic fertilizers containing various nutrients is a criterion for increasing the productivity of agriculture.

The use of only mineral or organic fertilizers has both positive and negative effects on plant development. Organic fertilizers improve the physical and biological activity of the soil, but they have a relatively low nutrient content, so more is required for plant growth. Mineral fertilizers contain higher concentrations of nutrients that are directly available to plants. However, the use of only mineral fertilizers leads to the degradation of soil humic substances and a decrease in soil fertility. Only an integrated nutrient management system is an alternative system for the sustainable and cost-effective management of soil fertility through the combined application of mineral and organic fertilizers [1].

In [2], the results of studying the effect of organic and mineral fertilizers on the humus state of soddy-podzolic soil are presented. It is shown that the most significant positive balance of humus was noted when using 80 t/ha of organic fertilizer and a full dose of NPK. When using three doses of mineral fertilizers, a negative balance of humus in the soil (-0.63-0.85 t/ha) was established, the use of two doses of organic fertilizer contributed to a positive balance of humus (0.38-1.32 t/ha). The study of the fractional composition of humus showed that organic and mineral fertilizers, when used together, increased the content of the most valuable fraction of humic acids from 2.7 to 5.4%.

In [3], in order to assess the minimization of the application of mineral nitrogen fertilizers due to humic acid and organic sources, the effect of humic acids and organic substances in combination with mineral fertilizers was studied. The use of humic acids and organic fertilizers reduced the content of nitrates, increased the yield and recorded higher values, although two times less nitrogen fertilizer was used compared to the control.

The content of humic substances is one of the most important soil indicators, on which the agronomic properties of soils depend. Humic substances remove the negative effect on the plant of many harmful substances, an overdose of

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mineral fertilizers, salinity, heavy metals, in addition, the formation of soils and its fertility is associated with humic substances [4].

The use of humic substances together with mineral fertilizers improves the agrochemical properties of soils and preserves its fertility. Humic acids with mineral soil particles form an organomineral complex particle capable of absorbing moisture and dissolved substances. Humic substances bind mineral particles of the soil, and influence the formation of a water-resistant loose cloddy-granular structure, which increases the moisture-absorbing and agrochemical properties of soils. Plant nutrients in such soils are not chemically bound by soil minerals, are not washed out by water, and are in an accessible form for plants [5].

There is evidence that in soils, due to the occurrence of chemical reactions from the applied phosphorus fertilizers, the available forms of phosphorus for plants turn into an indigestible form, and as a result, the effectiveness of phosphorus fertilizers decreases, while the degree of use of phosphorus from fertilizers is on average 20-25%. Of all the factors considered, the greatest decrease in the availability of phosphorus for plants is associated with the interaction of phosphates with cations. Assimilated forms of phosphorus entering the soil with mineral fertilizers, especially in slightly acidic and slightly alkaline soils, as a result of chemical interaction with calcium and magnesium ions, are converted into  $\text{CaHPO}_4 \cdot 2\text{H}_2\text{O}$  and  $\text{MgHPO}_4$ , and in an acidic environment into poorly soluble  $\text{AlPO}_4$ ,  $\text{Al}(\text{OH})_3\text{PO}_4$ ,  $\text{FePO}_4$ ,  $\text{Fe}_2(\text{OH})_3\text{PO}_4$  [6].

In [7], it is shown that humic fertilizers have a higher agrochemical efficiency and properties that increase the assimilable forms of phosphorus, in addition, they contain physiologically active substances that create a loose structure, increase the surface that affects the retention of moisture, as well as nutrients.

In [8], it is shown that humic fertilizers have a complex effect, increasing soil fertility, increase the amount of assimilable phosphorus, inhibit phosphorus retrogradation, improve phosphorus nutrition of plants, improve soil structure, shorten the vegetative period by improving protein metabolism, suppress the accumulation of nitrates, enhance the development plant root system.

Currently, in many countries of the world, humic substances have received legal confirmation in the field of practical solutions to environmental problems. The multifunctionality of humic substances provides them with a dominant role in improving soil fertility. Hydrophilicity and molecular structure enable humic substances to structure soils, regulate air and water regimes. The interaction of  $\text{Fe}^{3+}$ ,  $\text{Cu}^{2+}$ ,  $\text{Zn}^{2+}$ ,  $\text{Ca}^{2+}$ ,  $\text{Sr}^{2+}$  ions with humic substances has been scientifically substantiated. Also substantiated, the interaction of salt forms of humic substances with ions of polyvalent metals. On the basis of humic substances in the Republic of Belarus, more than ten new materials for environmental protection have been organized and used, namely: sorbents of heavy metal ions; humic ameliorants of soils contaminated with heavy metals; humic ameliorants of sandy soils, ameliorants of saline soils for the purpose of their green arrangement, for solving problems of environmental protection based on humic substances [9].

The work [10] provides data that preparations based on humic acids are very effective for landscaping on sandy, arid and saline soils, as well as in disturbed areas for human economic activity. And these drugs have been comprehensively tested in Bahrain, Jordan, Qatar and the United Arab Emirates. Almost unlimited possibilities of using humic preparations for reclamation of the territory, disturbed by the economic activity of people, are discovered.

It is known that [11] heavy metals are among the most widespread environmental pollutants, and they are one of the most hazardous in terms of toxicity. Among heavy metals, cadmium, copper, zinc, and lead are considered harmful to plants, which enter the environment with man-made emissions. To solve this problem, one should consider obtaining detoxifiers based on humic substances that interact with toxic substances and block their accessibility to plants while improving the physical properties of soils. In [12], the detoxifying ability of humic substances with respect to heavy metals was studied. The degree of detoxification of heavy metals with the use of humic substances was determined.

In [13], the preparation of humic acids as detoxifying agents with respect to Cd (II), Pb (II), Cu (II) is given. The resulting humic products had a stimulating effect on the yield of lettuce, in addition, the use of humic preparations in tests with metals showed a significant decrease in the toxic effects of heavy metals, in the presence of samples of humic products at a concentration of 50 mg/l, the yield of dry lettuce biomass, which under the influence of Cd (II) decreased to 44.4% of the control, increased from 71 to 82%. The reducing effect of the negative effect of humic fertilizers was also revealed in experiments with Pb (II), Cu (II).

The work [14] shows the effectiveness of the use of a humic preparation obtained on the basis of peat in the cultivation of potatoes. This drug had a positive effect on the growth processes and quality indicators of potatoes. In addition, the manifestation of late blight and *Alternaria* infections were noted at a later date, compared with the control. The use of which made it possible to obtain the highest yield of 24.5 t/ha, which exceeded the control by 11.13%.

The work [15] presents the results of a field experiment studying the effect of mineral fertilizers with humic acids on yield, content and absorption of nutrients, as well as on the efficiency of nutrient use by potatoes. The use of humic fertilizer in the recommended dose increased the yield of tubers by 9.3% compared to mineral fertilizer. The use of a humic product increased the efficiency of using nitrogen, phosphorus, and potassium by 16.4%, 9.3%, and 18.3%, respectively, compared with mineral fertilizer.

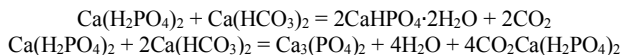
The paper [16] presents the results of studies of complex fertilizers of humus nature, obtained on the basis of brown coals from the deposits of the South Ural brown coal basin, having the following composition (wt.%): total content of organic substances 66-77;  $N_{tot}$  - 5.8;  $P_2O_5$  - 0.01;  $K_2O$  - 0.31, pH - 7-9. When introduced the typical carbonate into black soil, it contributed to an increase in the amount of nitrate and ammonia forms of nitrogen and mobile phosphorus, exchangeable potassium in the soil. The effect of fertilizer in the year of application to the soil and in the aftereffect was stronger than that of manure. The effect of this fertilizer was most clearly manifested at minimal doses, the mass fraction of grain in the total productivity of spring wheat plants increased by 90%.

One of the non-traditional sources for obtaining humic fertilizers and various humates can be peat, brown coal and other organic substances of a humic nature. It is known that in nature, along with coals that are good in terms of fuel, there are a significant amount of weathered coals with a reduced calorific value. The presence of up to 40% or more of humic acid in weathered coals makes it possible to use them as a raw material for the production of humic fertilizers. It is from such coals that research on the production of humic fertilizers and production, as well as their use in agriculture, is currently increasing in many countries of the world [17].

It is known that phosphorus, along with such elements as nitrogen and potassium, is an important element for plants. Metabolic processes in the plant organism occur in the presence of phosphorus, it serves as a source of energy. Phosphorus is an integral part of DNA and RNA and other significant substances in the plant organism. With a lack of phosphorus in the soil, plant growth slows down, leaf blades change shape and color, leaves fall prematurely, dark spots appear on the lower leaves of plants. And they begin to die off, the growth of vegetable plants stops, as a result of this, the plants begin to bush, the roots of the plants die, which is why the trees often fall. Plants cease to reduce air pollution and dustiness, cease to reduce the concentration of harmful gases in the air, plants cease to release phytoncides, which have the ability to kill pathogenic bacteria harmful to humans or inhibit their development [18].

The problem of phosphorus in agriculture has been of interest since the formation of agricultural chemistry as a science. This is due to the following factors. First, the total content of this element in the vast majority of soils is lower than that of other nutrients. Secondly, due to the chemical nature of the element, along with a low content, it is characterized by very low mobility in the soil, that is, low availability for plants. Thirdly, the reserves of phosphate raw materials for the production of fertilizers are very limited, and some of them are practically unsuitable for industrial processing, for obtaining water-soluble phosphate fertilizers. Sustaining food production for a growing global population requires a combination of phosphorus management approaches that not only extend the life of the remaining non-renewable phosphate reserves, but include improved efficiency and a sustainable phosphorus cycle. Solutions include improving the efficiency of fertilizer application on agricultural land along with a better understanding of the phosphorus cycle in soil-plant systems and the interactions between soil physics, chemistry and biology in combination with plant traits [19,20].

At present, the most common phosphorus-containing fertilizer in the world is simple superphosphate. It is mainly water-soluble calcium monophosphate  $Ca(H_2PO_4)_2$ . It is obtained by decomposition of phosphate raw materials with sulfuric acid. When introduced into neutral soils saturated with bases, it relatively quickly turns into  $CaHPO_4$  and  $Ca_3(PO_4)_2$ .



Under such conditions, the appearance of hydroxyapatite or fluorapatite, which are indigestible by plants, is possible in soils. In acidic soils rich in sesquioxides, poorly soluble, hardly assimilable phosphorus compounds can form. For this reason, the use of phosphate generally does not exceed 25%. As a result, the consumption of water-soluble phosphate fertilizers is much greater than the actually required amount of phosphorus [21].

Increasing the effectiveness of phosphate fertilizers can be achieved by adding organic fertilizers, they improve the mobility of phosphorus in the soil solution. During the decomposition of organic substances in the soil, various organic acids are formed, which bind with many polyvalent cations, such as  $Ca^{2+}$ ,  $Fe^{2+}$ ,  $Al^{3+}$ , as a result, phosphorus is released into the soil solution and ultimately absorbed by plants [22].

As already noted, humic substances that are part of fertilizers and contained in the soil contribute to better absorption of the main nutrients by plants. They prevent the retrogradation of assimilable phosphates, contain microelements, physiological and growth active substances, form a loose structure, create favorable conditions for the life of

microorganisms, stimulate the growth and development of plants. All this makes it possible to significantly reduce the rate of application of nutrients, obtain a high and high-quality yield, and also preserve soil fertility [5,23].

**Table 1.** Chemical composition of Central Kyzylkum phosphorites

Types of phosphoric raw material	Content of components, wt. %								$\frac{P_2O_{5\text{assim}}}{P_2O_{5\text{tot}}} \times 100$ , %	CaO : P <sub>2</sub> O <sub>5</sub>
	P <sub>2</sub> O <sub>5</sub>	CaO	MgO	Fe <sub>2</sub> O <sub>3</sub>	Al <sub>2</sub> O <sub>3</sub>	F	CO <sub>2</sub>	H <sub>2</sub> O		
WBC	27,26	53,36	0,61	0,51	1,30	1,04	2,41	5,95	12,24	1,96
WDC	18,22	47,28	0,99	0,60	1,18	1,41	14,90	6,81	17,80	2,60
OPF	17,18	46,14	1,73	1,02	1,20	1,25	16,00	7,69	18,50	2,69

In this regard, we studied the processes of obtaining humic simple superphosphate based on the decomposition of phosphorites of the Central Kyzylkum with sulfuric acid in various norms and oxidized coal with hydrogen peroxide, depending on the weight ratios of the initial components.

In the studies, the products of the Kyzylkum phosphorite complex were used, namely ordinary phosphorous flour (OPF), washed dried concentrate (WDC) and washed burnt concentrate (WBC), the compositions of which are shown in Table 1. Sulfuric acid with a concentration of 92% was used to activate phosphate raw materials. Oxidized coal with hydrogen peroxide, obtained by oxidation of brown coal from the Angren deposit with hydrogen peroxide, was used as an organic component. Oxidized coal has the following composition (wt.%): moisture 48.2; ash 10.35; organic 41.45; humic acid (HA) 53.64 on organic matter. The processes for obtaining oxidized coal based on the brown coal of the Angren deposit and the optimal conditions are given in [24].

As a result of the research, it was shown that the addition of oxidized coal to the acidic superphosphate mass before ammonization and drying does not lead to a decrease, as usually occurs when superphosphate is obtained by decomposition of phosphorites with sulfuric acid, followed by ammoniation and drying, but to a significant increase in the relative content of assimilable forms of P<sub>2</sub>O<sub>5</sub>. The optimal norm (60%) of sulfuric acid and the weight ratio of phosphorite to oxidized coal (1 : 0.25) were found. Highly efficient humic superphosphates have been obtained from all types of raw materials, even as poor as ordinary phosphate flour [25–30].

It is known that physical and chemical properties are one of the most important characteristics of fertilizers. Preservation during storage, transportation and application to the soil constitute a set of properties of fertilizers. Hygroscopicity, caking, strength and flowability of fertilizers are important physical indicators, because they predetermine the conditions for storage, transportation and direct use of fertilizers. In connection with the above, the physicochemical properties of fertilizers have been studied.

## 2. Materials and Methods

For the study, the initial phosphorous flour, the chemical composition of which is given in Table 1, and three samples of fertilizers were selected: simple superphosphate obtained without the addition of oxidized coal (sample 1); humic simple superphosphate obtained on the basis of phosphorite flour and oxidized brown coal at sulfuric acid rates of 60 and 70% of the stoichiometry for the formation of monocalcium phosphate and the mass ratio of phosphorite to oxidized coal is 1 : 0.25 (sample 2 and 3, respectively). The product of decomposition of phosphorous flour with sulfuric acid at a rate of 100% of the stoichiometry for the formation of monocalcium phosphate was used as a simple superphosphate. The chemical composition of fertilizers is given in table 2.

Figures 1-3 show radiographs of the original phosphoric flour, simple superphosphate and radiographs of humic superphosphates obtained at two different above-mentioned rates. X-ray analysis was carried out on a computer-controlled XRD-6100 apparatus. The phases were identified using domestic catalogs and ASTM [31–33].

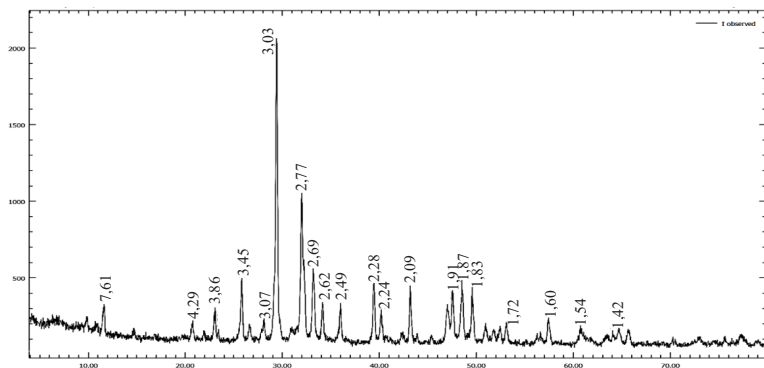
## 3. Results and Discussion

On the X-ray pattern of phosphorite flour (Figure 1.) diffraction bands 2.69; 2.62; 2.28; 2.24; 1.83; 1.72Å° belong to fluorocarbonate apatite. The presence of calcite is confirmed by interplanar distances of 3.86; 3.03; 2.49; 2.09; 1.91; 1.87; 1.60 Å°, dolomite - 1.54 Å°, gypsum - 7.61; 3.07; 2.77; 1.42 Å°, tricalcium phosphate - 3.45 Å°. Bands 4.29; 1.54 Å° indicate the presence of an insoluble residue, quartz, in the phosphorite flour. On the X-ray pattern of simple superphosphate (Figure 2.) that was obtained by the decomposition of phosphorite flour at a high rate of sulfuric acid, diffraction maxima appear: monocalcium phosphate - 7.57; 3.80; 2.79; 2.21 Å°; dicalcium phosphate - 3.50; 2.69 Å°, tricalcium phosphate - 2.69 Å°, ammonium dihydroorthophosphate 3.75 Å°, ammonium sulfate 4.33 Å°, gypsum 4.28; 2.87 Å°. On the radiographs (Figure 3) of humic superphosphates obtained by decomposition of

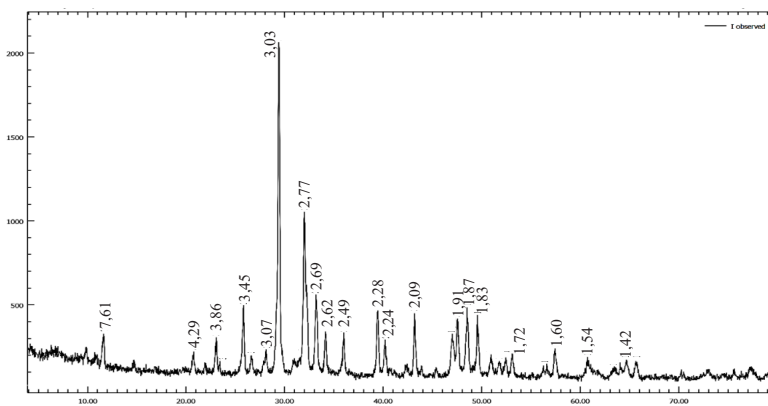
phosphate raw materials at lower rates of sulfuric acid, but with the addition of oxidized coal, diffraction maxima also appear: monocalcium phosphate - 2.90; 1.83; 1.65 A°, dicalcium phosphate - 5.80; 2.80; 1.75 A°, Mg (H<sub>2</sub>PO<sub>4</sub>)<sub>2</sub> - 5.31 A°, ammonium dihydroorthophosphate 5.31 A°, ammonium sulfate 4.33 A°, gypsum 3.49; 3.07 A°, quartz - 2.85 A°.

**Table 2.** The composition of simple superphosphate and humic superphosphates obtained on the basis of oxidized coal with hydrogen peroxide and ordinary phosphoric flour

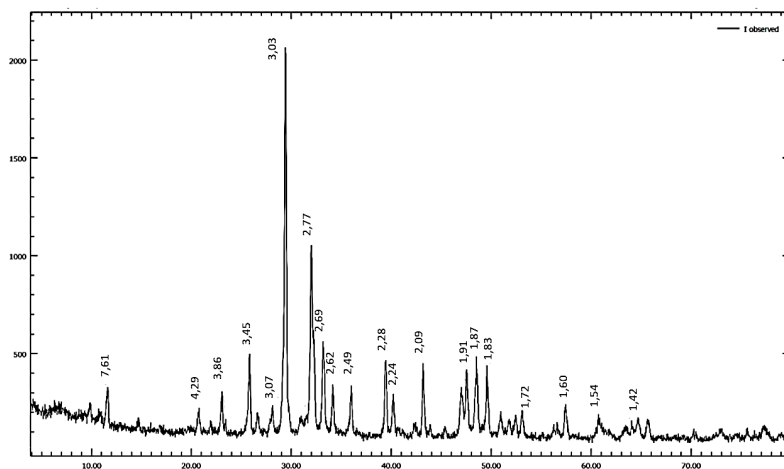
coal: OPF ratio	Moisture %	P <sub>2</sub> O <sub>5</sub> tot., %	P <sub>2</sub> O <sub>5</sub> assim. lim. %	P <sub>2</sub> O <sub>5</sub> assim. Tr.B., %	CaO tot., %	$\frac{P_2O_{5assim.}}{P_2O_{5tot.}}$ , SO <sub>3</sub> , tot %	N, tot %	Org. mat., %	HA %	
Norm of H <sub>2</sub> SO <sub>4</sub> from stoichiometry to Ca(H <sub>2</sub> PO <sub>4</sub> ) <sub>2</sub> formations, % 100										
1: 0	1,43	11,24	9,86	8,82	30,91	87,72	39,67	3,91	0	0
Norm of H <sub>2</sub> SO <sub>4</sub> from stoichiometry to Ca(H <sub>2</sub> PO <sub>4</sub> ) <sub>2</sub> formations, % 60										
1: 0,25	1,84	10,62	7,67	6,63	29,06	72,22	19,41	1,89	10,46	4,71
Norm of H <sub>2</sub> SO <sub>4</sub> from stoichiometry to Ca(H <sub>2</sub> PO <sub>4</sub> ) <sub>2</sub> formations, % 70										
1: 0,25	1,94	10,35	7,83	6,82	25,44	76,31	23,18	2,38	12,08	5,44



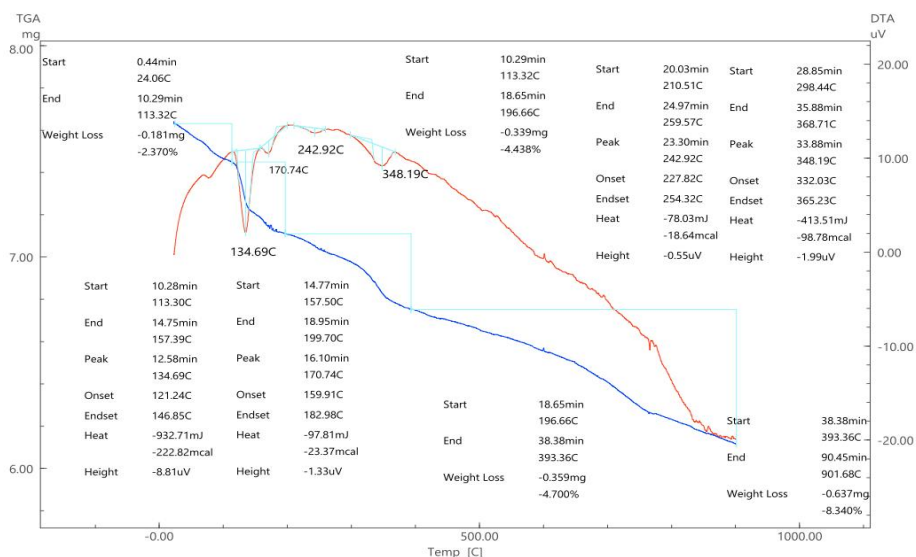
**Fig. 1.** X-ray pattern of phosphoric flour



**Fig. 2.** X-ray pattern of simple superphosphate



**Fig. 3.** X-ray pattern of humic simple superphosphate



**Fig. 4.** Derivatogram of simple superphosphate

Figures 4 and 5 show the DTA of simple superphosphate and humic simple superphosphate, which consist of two curves. DTA (Figure 4) shows that the decomposition of simple superphosphate mainly occurs in 4 temperature ranges: 24.06-113.32; 113.32-196.66; 196.66-393.36; 393.36-901.68°C, at which weight loss occurs, respectively: 2.37; 4.438; 4.7; 8.34%. In two endoeffects of 24.06-113.32°C and 113.32-196.66°C, a decrease in mass occurs by 2.37 and 4.438%, in which sorbed and crystalline moisture is lost. Effects in the temperature range of 196.66-393.36 and 393.36-901.68°C accompanied by a weight loss of 4.7 and 8.34% correspond to the dehydration of phosphate compounds. DTA of humic simple superphosphate (Figure 5) shows that the decomposition of humic simple superphosphate mainly occurs in 3 temperature ranges: 21.81-194.31; 194.31-703.51; 702.7-901.73°C at which following weight losses occur: 5.482; 15.957; and 25.253%, respectively. The first endoeffect lies in the range of 21.81-194.31°C and is accompanied by a decrease in mass by 5.482%, at which sorbed and crystalline moisture is lost. In the range of 194.31-703.51°C, organic substances are split, leading to the formation of an additional amount of low molecular weight compounds that are removed from the system. In the range of 702.7-901.73°C, deep

oxidation of organic substances and dehydration of phosphate compounds occur. Table 3 shows the decomposition temperatures of simple superphosphate and humic simple superphosphate. It can be seen from it that in humic simple superphosphate obtained on the basis of oxidized brown coal and phosphate raw materials and sulfuric acid, the decomposition temperature is much lower than the decomposition temperature of simple superphosphate. This indicates that the addition of oxidized carbon to the acidic superphosphate mass before ammonization and drying leads to the formation of organomineral compounds that reduce decomposition temperatures.

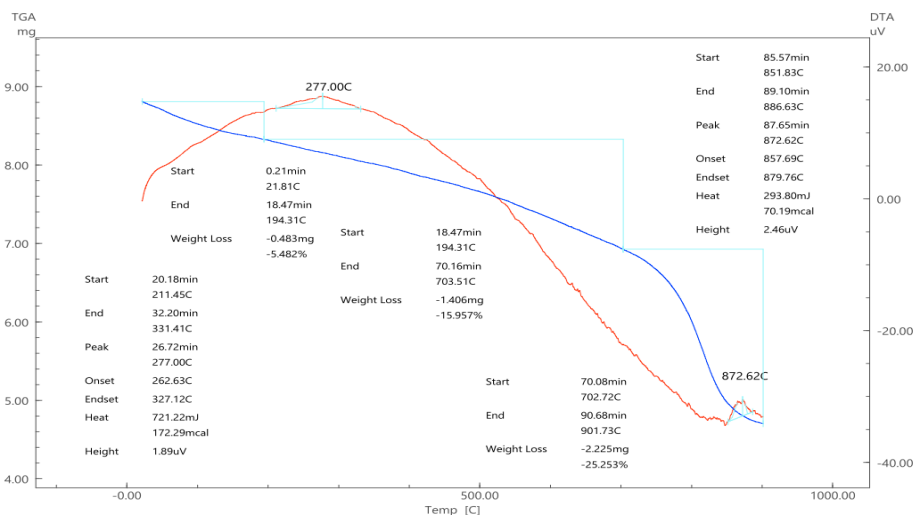


Fig. 5. Derivatogram of humic simple superphosphate

Table 3. DTA results of simple superphosphate and humic simple superphosphate

Temperature, °C	Weight loss, mg.		Weight loss, %	
	Simple superphosphate (7,638 mр)	Humic simple superphosphate (8,811 mр)	Simple superphosphate	Humic simple superphosphate
100	0,17	0,29	2,27	3,295
200	0,53	0,5	6,97	5,66
300	0,66	0,7	8,69	7,98
400	0,89	0,92	11,67	10,4
500	0,98	1,15	12,86	13,1
600	1,072	1,5	14,04	16,93
700	1,23	1,88	16,1	21,31
800	1,41	2,8	18,4	31,81
900	1,52	4,11	19,95	46,63

#### 4. Conclusions

Thus, the process of obtaining humic simple superphosphate by decomposition of phosphorites of the Central Kyzylkum: OPF, WDC and WBC with sulfuric acid and the addition of oxidized brown coal with hydrogen peroxide was studied. It has been established that the addition of oxidized coal to the acidic superphosphate mass before ammonization and drying does not lead to a decrease, as usually occurs in the production of simple superphosphate by decomposition of phosphorites with sulfuric acid, followed by ammonization and drying, but to a significant increase in the relative content of assimilable P<sub>2</sub>O<sub>5</sub> forms. The optimal norm (70%) of sulfuric acid and the weight ratio of phosphorite to oxidized coal (1:0.25) were found. The commercial properties of fertilizers were determined:

hygroscopicity, moisture capacity, caking, strength of granules. X-ray and thermogravimetric analysis of the obtained fertilizers were performed. Fertilizers do not cake, even with a high moisture content they retain complete friability, their characteristics are not inferior to the most common phosphate fertilizers.

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