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Methods for Obtaining Phosphorus-Containing Fertilizers Based on Industrial Waste

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Abstract: This article discusses the method of obtaining phosphorus-containing components from cottrel dust from the industrial wastes of the New-Jambul phosphorus plant. Accumulated industrial waste heavily pollutes the environment and has a direct impact on all living things. Therefore, their processing is of special interest for the state and grant programs have been allocated in order to obtain new valuable substances. In order to solve these problems, a number of experimental works have been carried out to study the chemical and mineralogical composition and chemical structures during the heat treatment of cottrel dust—the waste of phosphorus production. The optimal parameters of the process of obtaining mono-calcium phosphate from cottrel dust were determined and the process of crystallization of mono-calcium phosphate was studied. A method has been developed for obtaining a phosphorus-containing fertilizer based on cottrel dust from the industrial waste of the New-Jambul phosphorus plant by means of sulfuric acid solutions. The advantage of the resulting phosphorus-containing fertilizer is that it has a high solubility and digestibility of phosphorus plants. They are also high in phosphorus-containing substances that ensure the growth and yield of agricultural plants. The developed method for obtaining phosphorus-containing fertilizers is aimed at reducing the accumulated industrial waste, which in turn allows you to regulate and improve the environmental situation in the region.

Keywords: industrial waste; fertilizers; cottrel dust; sulfuric acid; phosphate production

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1. Introduction

The development of industry around the world has made the problem of integrated recycling of industrial waste and attracting substandard mineral resources into production relevant. In recent years in Kazakhstan, there have been no fully integrated methods of processing of accumulated industrial waste. Therefore, their processing is of special interest to the state and grant programs have been allocated in order to obtain new valuable substances.

According to the results of analytical studies and experts in the field of mineral resources and metallurgy, it was determined that more than 30 billion tons of industrial waste have accumulated in the country, a significant part of which is toxic. Analysts and experts emphasize that now industrial waste should be considered as a raw material base. Industrial waste is very long and slow in terms of technological and material composition, even with the use of advanced recycling methods. Despite the accumulated waste, the New-Jambul phosphorus plant (NJPP) for the production of phosphorus and phosphorus-containing fertilizers discharges industrial waste into the environment [1,2].

In the territory of the branch of the NJFP "Kazphosphate" located in Taraz city—the mineral fertilizers plant—more than 10 million tons of waste from the production of phosphoric acid have accumulated. The accumulated waste is partially processed to obtain various commercial products for use in the form of fertilizers in agriculture [3,4]. Studies by scientists and manufacturers as well as laboratory experiments show that these industrial wastes are quite valuable secondary raw materials for the production of phosphate and complex phosphorus-potassium containing mixed fertilizers. Cottrel dust is a waste product of phosphate production in New-Jambul. Cottrel dust is formed as a result of high-temperature processing of phosphate ore. In this process, the reaction gas that leaves the phosphorus furnace contains up to 200 g/m^3 of particulate matter in the form of dust. The dispersed composition of cottrel dust is particles with a size of less than 0.5–12.0 micrometers—90%, and a small fraction of 20.0–200.0 micrometers—10%. These particles are captured by the special part in the filters then discharged into the storage area (Figure 1). As a result of thermal treatment of phosphate ore, about 150–160 kg of cottrel dust is formed per 1 ton of yellow phosphorus at the phosphorus plant [5–7].

Figure 1. Industrial waste of the New-Jambul phosphorus plant.

Accumulated industrial waste heavily pollutes the environment and has a direct impact on all living things. The accumulation of waste in landfills increases the level of pollution of the atmosphere, soil, groundwater, and surface water, disrupts the functioning of ecosystems, and damages agriculture and construction. In addition, landfill gas emissions have a negative impact on climate change. Pollution of the environment by industrial wastes, due to their ability to have a negative impact on the biosphere, is a global environmental and hygienic problem of our time.

To date, many methods of processing this waste have been developed, but these methods are not fully improved, so this problem is still relevant.

By developing this methodology, we can recycle the accumulated waste of phosphorus production and obtain a phosphorus-containing component—a fertilizer to increase crop yields. The relevance of the scientific work lies in the fact that accumulated phosphorus waste creates crisis problems in the environment. That is why their recycling and disposal with the subsequent receipt of commercial products is of particular interest [8,9].

The purpose of scientific work is to substantiate scientific and technological methods of processing industrial waste with the subsequent production of phosphorus-containing mineral fertilizers with a large number of major components to increase the yield of crops. The developed methodology is aimed at reducing accumulated industrial waste [9,10].

2. Materials and Methods

The object of the research is a cottrel dust and monocalcium calcium phosphate produced by the decomposition of cottrel dust with sulfuric acid solution. For the processing of cottrel dust under experimental conditions, the test methods were chosen using a Jeol JSM-6490l V scanning electron microscope (SEM), an IR Fourier spectrometer (Zhimadzu IR Prestige-21, Shimadzu, Kyoto, Japan), and a Q-1500 derivatograph.

2.1. Instrumental Research Methods

The chemical composition and micrograph of Cottrel dust was determined using a Microsoft Analysis Report (Figure 2, Table 1).

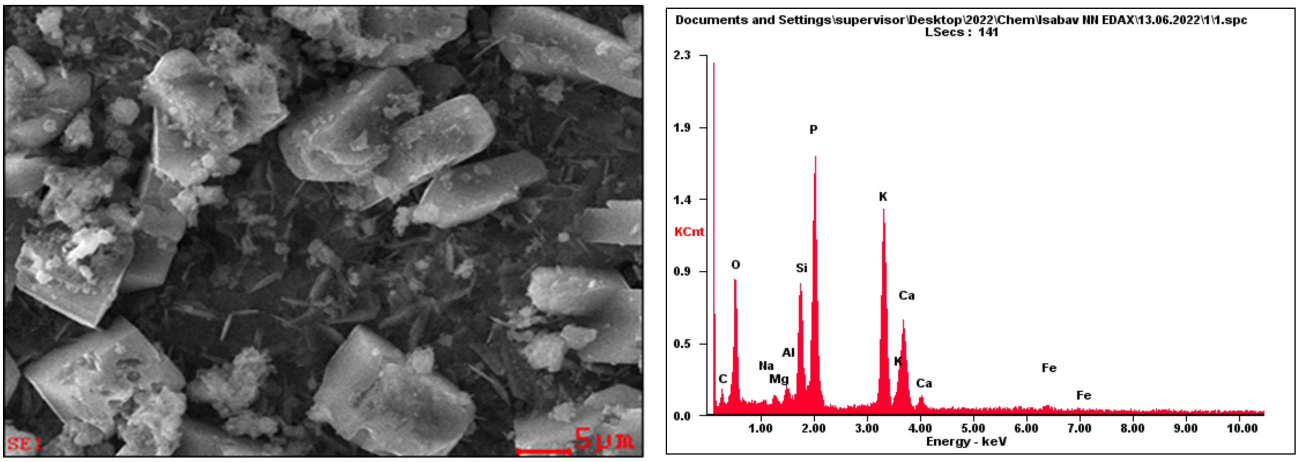

Figure 2. The micrograph of cottrel dust.

Table 1. Chemical composition of cottrel dust.

Composition	Share,%	Oxides	Converted to Oxides, %	Composition	Share, %	Oxides	Converted to Oxides, %
O	42.1	-	-	K	5.91	K_2O	7.12
Na	0.84	Na_2O	1.13	Ca	6.35	CaO	8.89
Mg	0.91	MgO	1.51	Fe	0.58	Fe_2O_3	0.82
Al	1.05	Al_2O_3	1.98	Zn	0.55	ZnO	0.68
Si	7.31	SiO_2	15.6	C	17.6	CO_2	-
P	13.4	P_2O_5	30.7	F	2.99	-	-

It follows from Figure 2 that the surface of the cottrel dust is characterized by imperfect cohesion of minerals with the formation of cracks and chips. The main mineralogical phases of the sample are silicophosphates and silicofluorophosphates, which have colorless lamellar crystal structures. Small inclusions of potassium phosphates are characterized by soldered crystals of small elongated irregularly shaped plates. An accumulation of fine-grained chain structures of calcium silicate crystals is observed around phosphate minerals. Intermediate spaces and cracks are filled with carbonaceous phase.

According to the results (Table 1) of instrumental studies, it was found that the chemical composition of cottrel dust contains metals such as Na, Mg, Al, K, Ca, Fe, and Zn. These metals are light and in small amounts. Moreover, the necessary element phosphorus (P) is 13.4%. This phosphorus content is enough to process this waste, then obtain mineral fertilizers.

In addition to elemental analysis, chemical analysis for the total and assimilable form of phosphorus in cottrel dust was carried out. Chemical analysis was performed using 0.2 M Trilon B buffer solutions and 2% citric acid. On the basis of chemical analysis in the composition of cottrel dust, $P_2O_{5(total)}$) —30.7%, $P_2O_{5\ (assimilable)}$—17.2% and there are light alkali and earth metals. According to the results of two studies, it is sufficient to use cottrel dust for mineral fertilizers [11–13].

To study the chemical composition of the studied cottrel dust during heat treatment, a differential thermal analysis was performed in the Q-1500 Derivatograph device. The derivatogram (DTA) of cottrel dust is shown in Figure 3.

Figure 3. Derivatogram (DTA) of cottrel dust.

The DTA curve in Figure 3 is characterized by low-intensity endoeffects at 533 K, 853 K, 1053 K, and 1073 K. The first endoeffect at 533 K characterizes the removal of surface and hydrated moisture. At 853 K, the residual crystallohydrate moisture is removed. The non-intensive endoeffect at 1053–1073 K indicates the decarbonization of magnesium-containing impurity compounds and the latter indicates the decomposition of calcium carbonates. There are also three consecutive exoeffects at 863 K, 1108 K, and 1148. The first exoeffect is characterized by the oxidation of insignificant free phosphorus. Two second exoeffects are characteristic of the decomposition of phosphorous compounds [14].

IR spectral analysis of cottrel dust (Figure 4) was performed on a Fourier infrared spectrometer in order to determine the functional groups. Figure 4 shows the IR spectrum of cottrel dust, from which it follows that [14,15]:

- absorption spectra with wavelengths of 1800–1600 (1705.07 and 1597.06) inherent in the presence of silicate substances fit Si-O-Si and Si-O-C chemical bonds in cottrel dust, as well as in the range of 1100–950 (1026.13 and 1002.98) are inherent in the phosphorus compounds;
- intense fluctuations in the range of 910.40, which are inherent in the phosphorus-containing compounds by the P-F group;
- less intense fluctuations in the range of 821.68–694.37, which are inherent in the phosphorus-containing substances by the P = S group;
- spectra in this interval (644.22–559.36) are inherent in the compounds Al^{3+}, Fe^{3+}, Mg^{2+}, Fe^{+2} in the valence state Si-O-Al, Si-O-Fe, and Si-O.

2.2. Methodology of the Experiment

The process of Cottrel dust decomposition was studied under laboratory conditions using the following methodology: a sample of Cottrel dust was decomposed in a thermostatically controlled glass reactor with a volume of 250 mL, filling ratio 0.6, with a solution of sulfuric acid with a concentration of 20% at temperatures of 363 K [16].

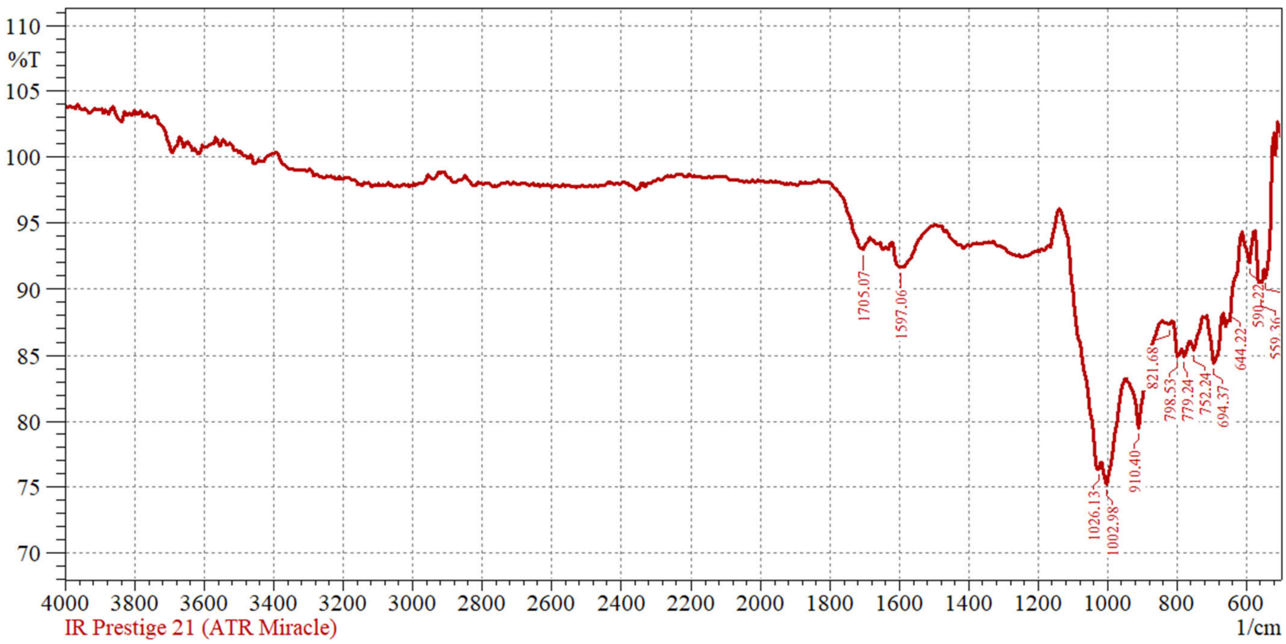

Figure 4. IR spectrum of cottrel dust.

Based on the results of the analysis, we calculated the completeness index of the process, the degree of P_2O_5 extraction into the solution, or the decomposition coefficient according to Formula (1):

$$K_p = 1 - m_{(dried)} \times (P_2O_{5(total)})^{dried} - (P_2O_{5(water)})^{dried} / m_{(c.d)} \times (P_2O_{5(total)})^{c.d} \quad (1)$$

where, $m_{(dried)}$—mass of the dried insoluble sediment, g; $m_{(c.d)}$—is the mass of cottrel dust taken for decomposition, g; $(P_2O_{5(total)})^{dried}$ and $(P_2O_{5(water)})^{dried}$ content in the dry sludge, respectively $P_2O_{5(total)}$ and $P_2O_{5(water)}$ %. $P_2O_{5(total)})^{c.d}$—content of total P_2O_5 in the cottrel dust, %. The sludge washing coefficient was determined according to the Formula (2):

$$K(_{washing)} = 1 - P_2O_{5(water)} \times (m_{(dried)} / m_{(c.d)}) / P_2O_{5(total)})^{c.d} \times K_p \quad (2)$$

The filtration productivity of the insoluble residue was calculated according to Formula (3):

$$P_{(productivity)} = m / F \times t \quad (3)$$

P—productivity; m—mass of dried insoluble residue, g; F—filter surface area, m^2; t—filtering time, h.

The main stages of processing of Cottrel dust by the liquid-phase method in laboratory conditions are the crystallization of the production monocalcium phosphate and its separation from the solution. The crystallization of monocalcium phosphate occurs from solutions and monocalcium phosphate in phosphoric acid; close to saturation. Consequently, the indicators of the crystallization process will depend only on the degree of saturation of the solution with monocalcium phosphate [17,18].

Cottrel dust (DTIR) was used to study the crystallization process of monocalcium phosphate. Under experimental conditions, the degree of its decomposition was 82.8%, the content of P_2O_5 in the sample was 30.7%. The decomposition of cottrel dust was carried out at a temperature of 363 K for 90 minutes with 20% sulfuric acid containing 11.42% phosphorus. The chemical reaction of this process can be specified in this way:

$$Ca_5(PO_4)_3F + 3.5H_2SO_4 + 2.5H_2O = 1.5Ca(H_2PO_4)_2 \times H_2O + 3.5CaSO_4 \times H_2O + HF \quad (4)$$

After decomposition, an insoluble residue was separated from the suspension, and the mother liquor was cooled to isolate mono-calcium phosphate crystals. During spontaneous polythermal crystallization of the salt, the temperature decreased to 313 K, which was then kept constant throughout the process.

From Figure 5 it follows that with an increase in time to 90 minutes, the yield of monocalcium phosphate increases. After 90 minutes, the product yield does not change. The optimal process time is 120 minutes.

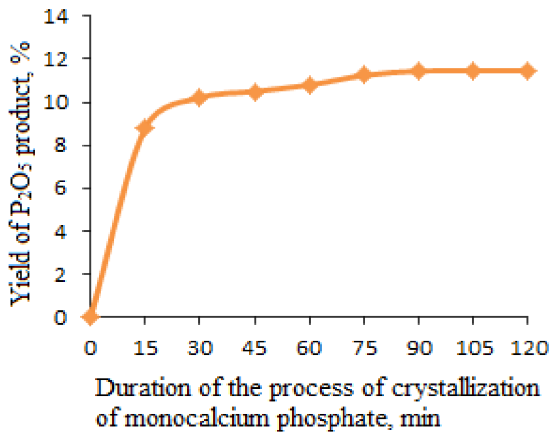

Figure 5. Dependence of the yield of P_2O_5 with the product on the duration of the crystallization process.

It was found that with the duration of the crystallization process of 60–90 min, the process speed decreases and does not change significantly in the next 90–120 min. At the same time, respectively, there is an increase in the yield of MCP and P_2O_5 with the product. During 60–90 min, the bulk of the crystals is released, after which the increase in the mass of the precipitate becomes insignificant, since by the end of the phase transformation, the system is approaching a state of thermodynamic equilibrium [18,19].

3. Results and Discussion

Thermodynamic regularities of the process of decomposition of cottrel dust using sulfuric acid in the temperature range 333–363 K were carried out with the help of HSC-6 Chemistry programs [20]. The entropy (ΔS), enthalpy (ΔH), and Gibbs energy (ΔG) of the process were studied on the basis of this research. The results are shown in Table 2.

Table 2. Thermodynamic indicators in the process of decomposition of Cottrel dust.

Temperature, K	ΔH, kJ/Mole	ΔS, J/(mol·K)	ΔG, kJ/Mole
333	−399,29	−321,2	−292,28
338	−401,25	−327,0	−290,66
343	−403,19	−332,7	−289,01
348	−405,09	−338,2	−287,33
353	−406,96	−343,5	−285,63
358	−408,80	−348,7	−283,89
363	−410,61	−353,7	−282,14

According to the results of thermodynamic studies (Table 2), it was found that with an increase in temperature in the range of 333–363 K, the enthalpy value increases from −399.29 to −410.61 kJ/mole, entropy from −321.2 to 353.7 J/mol × K and the Gibbs energy values are from −292.29 to 282.14 kJ/mole. The minus value of the Gibbs energy indicates that a chemical reaction occurs between the cottrel dust and sulfuric acid [21,22].

The kinetic regularities of the process of obtaining monocalcium phosphate on the basis of Cottrel dust have been studied. Experimental data on the process of obtaining phosphorus compounds were processed according to the Pavlyuchenko Equation (5), and the kinetic laws of the process were determined, with the calculation of the values of the "apparent" activation energy (Equation (6)).

$$1 - (1 - \alpha)^{1/3} = K \times \tau^{1/2} \tag{5}$$

$$K = A_0 \times e^{E/RT} \tag{6}$$

where, E is the "apparent" activation energy, kJ /mol; R is a constant value of −8.314, and T is the process temperature. The degree of decomposition of cottrel dust depending on temperature and time during decomposition is shown in Figure 6.

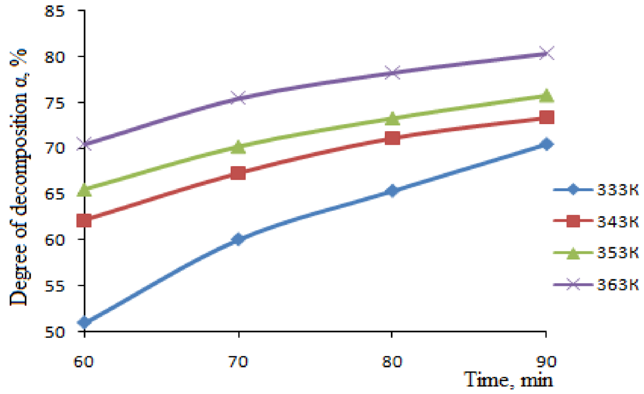

Figure 6. Degree of decomposition of Cottrel dust depending on temperature and time.

Using the Pavlyuchenko equation in determining the constant of the rate of the chemical reaction and the energy of "apparent" activation during the decomposition of cottrel dust with sulfuric acid solutions. Table 3 shows the results of processing experimental data.

Figure 7 shows a graph of the dependence $1 – (1 – \alpha)^{1/3}$ from $\sqrt{\tau}$.

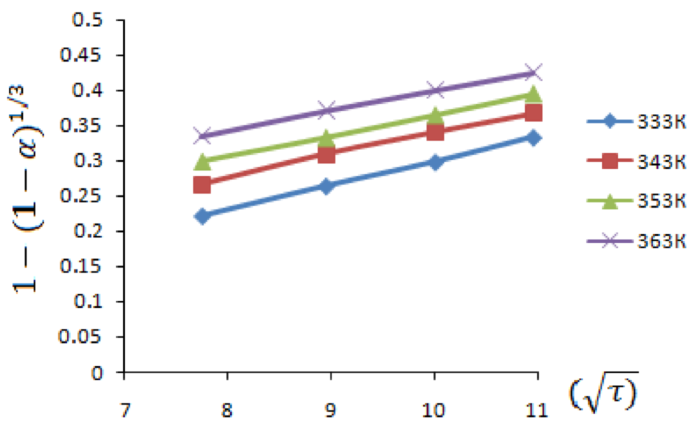

Figure 7. Dependencies between $1 - (1 - \alpha)^{1/3}$ from $\sqrt{\tau}$.

From Table 4 it follows that in the process with an increase in temperature from 333 K to 363 K, the rate constant of the chemical reaction increases from 0.0295 to 0.0401 and the activation energy of the process increase.

Table 3. Results of processing according to the Pavlyuchenko equation.

α	$1-\alpha$	$(1-\alpha)^{1/3}$	$1-(1-\alpha)^{1/3}$	τ, min	$\sqrt{\tau}$
		T = 333 K			
0.510	0.490	0.788	0.222	60	7.745
0.601	0.399	0.736	0.264	80	8.944
0.654	0.346	0.702	0.298	100	10.00
0.702	0.298	0.667	0.333	120	10.95
		T = 343 K			
0.622	0.378	0.723	0.277	60	7.745
0.683	0.317	0.681	0.319	80	8.944
0.701	0.299	0.659	0.341	100	10.00
0.733	0.267	0.643	0.367	120	10.95
		T = 353 K			
0.655	0.345	0.701	0.299	60	7.745
0.702	0.298	0.667	0.333	80	8.944
0.723	0.277	0.645	0.355	100	10.00
0.748	0.252	0.621	0.379	120	10.95
		T = 363 K			
0.705	0.295	0.665	0.335	60	7.745
0.762	0.238	0.619	0.381	80	8.944
0.783	0.217	0.600	0.400	100	10.00
0.804	0.196	0.580	0.420	120	10.95

Table 4. The relationship between the value of the velocity constant and the inverse temperature.

Constant Speeds	Numerical Value K	*lnK*	Temperature, K	*1/T*
$tg_{\varphi 1} = K_1$	0.0295	−1.530	333	0.0030
$tg_{\varphi 2} = K_2$	0.0355	−1.449	343	0.0029
$tg_{\varphi 3} = K_3$	0.0365	−1.436	353	0.0028
$tg_{\varphi 4} = K_4$	0.0401	−1.396	363	0.0027

The values of the "apparent" activation energy of the decomposition process of cottrel dust with a solution of sulfuric acid are determined. The value of the "apparent" energy of the activation of a chemical reaction is equal to equation = 5.94 kJ/mol. Based on the data obtained, it can be concluded that the chemical reaction of decomposition of cottrel dust with a solution of sulfuric acid proceeds in the diffusion region.

Methods of mathematical processing are considered in order to determine the reliability and reliability of the results of experimental work carried out in laboratory conditions. During experimental studies, it was determined that the degree of decomposition of cottrel dust is 82.8% with a solution of sulfuric acid concentration of 20%, at a temperature of 363 K. The equation of recording the degree of decomposition of cottrel dust based on a linear mathematical function is shown:

$$Z = 16.7213 + 0.4602 \times x + 0.2009y \quad (7)$$

Mathematical processing of experimental work on the decomposition of cottrel dust in a 20% solution of sulfuric acid was carried out [23]. The results of mathematical planning are shown in Figure 8.

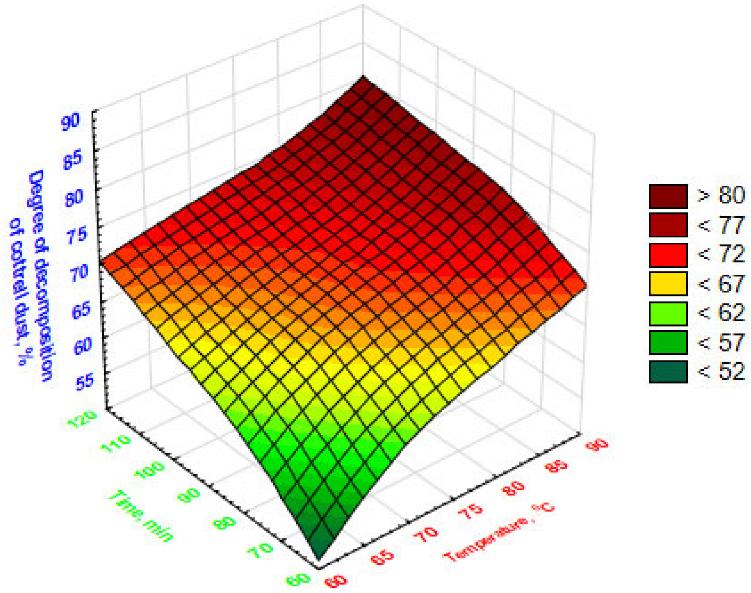

Figure 8. Dissolution rate of Cottrel dust with changes in temperature and time.

According to the results of mathematical planning of experiments from Figure 8, it follows that the dissolution of cottrel dust with changes in temperature and time increases. This is evidenced by the change and difference of the green square color to the red.

The chemical composition was revealed using scanning microscopy (JSM-6490lV, Jeol. Tokyo, Japan) on samples of a phosphorus-containing compound, monocalcium phosphate, extracted from cotton dust from the wastes of the New-Jambul phosphorus plant. The results of the scientific work are given in Table 5.

Table 5. Chemical composition of mono-calcium phosphate.

Compounds	P_2O_5 (Total)	P_2O_5 (Assimilable)	P_2O_5 (Water Solubility)	P	Ca	Mg	Fe	Al	S
Mono-calcium phosphate,%	25.42	22.43	2.99	11.1	7.98	0.04	0.6	0.25	0.08

According to the results (Table 5) of elemental analysis of monocalcium phosphate, it was determined that at a sulfuric acid solvent concentration of 20% and a temperature of 363 K, the phosphorus oxide yield is P_2O_5(total)—25.42% and P_2O_5(assimilable)—25.42%. Such a content of phosphorus obtained from waste can be used in medium fertile soils as a phosphorus-containing fertilizer.

In addition to phosphorus, monocalcium phosphate contains metals such as calcium, magnesium, iron, and aluminum. These metals do not affect the function of phosphorus. Some of the metals remained undissolved in the precipitate. The solubility of phosphorus in monocalcium phosphate was checked with 96% in Trilon B buffer solutions and 95–96 in 2% citric acid.

The filtration properties and moisture content of the sediment were determined by standard methods. The sediment and filtrate were analyzed for the content of P_2O_5 water and P_2O_5 assimilable by double titration. The yield of P_2O_5 was calculated by the content of P_2O_5 assimilable in the wet sediment, and not P_2O_5 of water, taking into account that in the finished product, P_2O_5 assimilable.

Based on the data in Figure 9, the formation of a crystal structure in the microstructural image of the formed calcium monophosphate is described. It was found that the compound contains calcium, phosphorus, and a small amount of magnesium, iron, and aluminum.

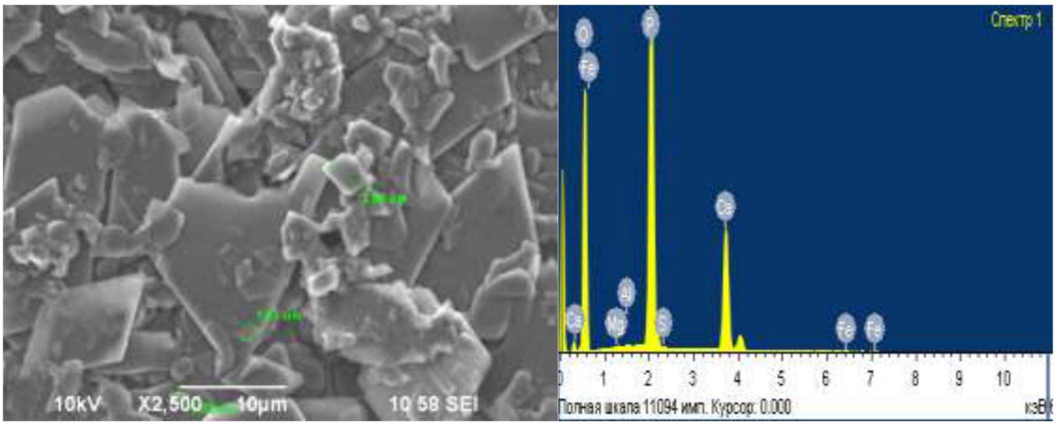

Figure 9. Microscopic image of mono-calcium phosphate.

It was found that the crystallization of monocalcium phosphate occurs within 60–90 min, then the rate of the process decreases and does not change significantly in the next 90–120 min. At the same time, respectively, there is an increase in the yield of monocalcium phosphate and P_2O_5 with the product. The optimal parameters taking a phosphorus-containing compound from cottrel dust were determined and the process of crystallization of monocalcium phosphate was studied depending on the duration of the process and the yield of P_2O_5 with the product [24,25].

During the study, it was determined that the obtained monocalcium phosphate has a total—25.42%, an assimilable—22.43% and a water solubility—2.99% form of phosphorus oxides. The share of phosphorus in monocalcium phosphate obtained is quite sufficient for its use as a phosphorus-containing component in the production of fertilizers [26].

Monocalcium phosphate is a fertilizer that contains a huge amount of minerals. They are a white or grayish powder, consisting of small granules, easily soluble in water. The high content of potassium and phosphorus ensures its efficient and high-quality use on soils with a low level of fertility. Fertilizer monocalcium phosphate is especially effective for growing root crops, as well as crops that are very sensitive to chlorine. Due to its complete water solubility, this product ensures the successful use of monocalcium phosphate in vegetable growing in open or protected ground, and even in top dressing with any drip irrigation.

Calcium monophosphate is a quick way to make up for the deficiency of phosphorus and calcium. The additive is used for garden, ornamental, and horticultural crops. Fertilizer refers to the complex. The use of calcium monophosphate helps increase yields. The developed method differs from the international standard methods in several respects: feedstock is industrial waste from phosphorus production; slightly concentrated sulfuric acid is used as a solvent; the optimal technological parameters for the production of monocalcium phosphate based on cottrel dust have been established; mathematical planning of the experiments was carried out; and this method reduces industrial waste and receives phosphorus-containing components.

The resulting phosphorus-containing (monocalcium phosphate) finished product based on cottrel dust has a high demand among potential users. The cost of the resulting product is cheaper due to the use of industrial waste and cheap sulfuric acid solvent, so it is economically advantageous.

4. Conclusions

Accumulated phosphorus waste creates crisis problems in the environment. That is why their recycling and disposal with the subsequent receipt of commercial products is of particular interest. Among industrial waste is cottrel dust, which requires a very complex and time-consuming approach during processing since there are different elements and a complex granular dispersed composition in the composition of dust.

Based on the conducted studies, the elemental and mineralogical compositions of cottrel dust were studied, and the structural features of this waste were also clarified. A differential thermal analysis was carried out to observe the changes in the initial raw materials that occur as a result of heat treatment.

The thermodynamic regularities of the process of obtaining monocalcium phosphate on the basis of cottrel dust with the use of sulfuric acid have been studied. The thermodynamic parameters entropy, enthalpy, and Gibbs energy are given. Based on the results of experimental work, mathematical processing 3D of experimental work on the decomposition of cottrel dust was carried out. The kinetic regularities of the process of obtaining monocalcium phosphate obtained by decomposition cottrel dust with the help of sulfuric acid were studied. The rate constant and the activation energy of the process were determined [26–28].

A method was developed for producing monocalcium phosphate, which has phosphorus in its composition derived from cottrel dust from the wastes of the New- Jambul phosphate plant using inorganic sulfuric acids. The advantage of the obtained phosphorus-containing mono-calcium phosphate is that it has a high solubility and assimilable of phosphorus to plants. They are also described by a great deal of phosphorus-containing substances that ensure the growth and yield of agricultural plants.

The developed method for obtaining phosphorus-containing fertilizers is aimed at reducing the accumulated industrial waste, which in turn allows you to regulate and improve the environmental situation in the region.

Author Contributions: Conceptualization: B.I.; and B.Z.; Methodology: A.K.; and B.S.; Validation: B.S.; and N.I.; Formal analysis: B.Z.; S.K.; and A.A.; Investigation: A.K.; S.K. and N.I.; Resources: B.Z.; Data curation: B.S.; Writing — original draft: B.I.; Writing — review & editing: B.I.; Visualization: A.K.; and N.I.; Supervision: S.K.; B.S.; and A.A.; Funding acquisition: B.I. All authors have read and agreed to the published version of the manuscript.

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Data Availability Statement: The data used to support the findings of this study are included within the article.

Conflicts of Interest: The authors declare no conflict of interest.

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