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Conditions for the Development of Phase Components $K_2CaP_2O_7$, KCaP₃O₉ in Toxic Dust

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Zhumadilova, A.K., Madaliyeva, E.B., & Zhigitova, S.Z. (2021). Conditions for the development of phase components $K_2CaP_2O_7$, KCaP $_3O_9$ in toxic dust. *Scientific Horizons*, 24(3), 38-44. Abstract. The present study analyses the composition and main components of toxic dust. To develop and understand the methods of controlling the dust generation process, it is necessary to study the data that provide detailed information about the reaction mechanism. The results of studies of the phase composition of dust conducted in the laboratory and their comparison with the data obtained earlier by other authors allowed establishing a fairly reliable diagnosis of the phase composition of dust. The analysis revealed that the dust of various phosphorus plants comprises the same basic components, yet the chemical bonds between them differ. The purpose of this study, conducted in the research laboratory of the Zhambyl Branch of LLP "Kazphosphate" (NDFZ) is to investigate the possibility of using new toxic dust, as well as the toxic dust from storage tanks to obtain NPK fertilisers. The study comprises three stages of investigating the oxidation of elementary phosphorus with nitric acid, since elementary yellow phosphorus is dangerous for the environment. As a result of the 1st stage of the study, it was found that toxic dust oxidised with nitric acid cannot be used as a fertiliser, since a non-technological mass is generated, such as acid resin, which is not suitable for drying and granulation. To neutralise the acid reaction mass, it was decided to use an aqueous solution of ammonia, thereby increasing the nutrient content and obtaining a complex NPK fertiliser. In the course of the study, the authors found in the 2nd and 3nd stages of the experiment that to obtain a productsuitable for fertilisation, it is necessary to strictly control the content of elemental phosphorus in the initial toxic dust and adjust the consumption of nitric acid based on its results

Keywords: nitric acid, carbon, phosphorus, sample, temperature, experiment



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INTRODUCTION

The development of methods for controlling the dust generation process is impossible without information about the reaction mechanism. In general, it leads to the generation of individual dust components. Information about this is not found in the literature. Until recently, the scientific literature contained only scattered and incomplete data on the composition of dust [1]. The results of studies of the phase composition of dust conducted in the laboratory and their comparison with the data obtained earlier by other authors allowed establishing a fairly reliable diagnosis of the phase composition of dust. It was found that the dust of various phosphorus plants comprises the same basic components, and only the chemical bonds between them change. Such components are as follows: silicon dioxide, double calcium and potassium pyrophosphate, double calcium and potassium metaphosphate. The mass of the total content of these components in dust often reaches 30%. The average phase composition of the dust of various phosphorus plants is presented in Table 1 [5; 6].

Phase	Composition (% of weight)						
SiO ₂ (amorphous)							
K ₂ CaP ₂ O ₇							
KCaP ₃ O ₉	31						
C	20						
CaF ₂	38						
K ₂ SiF ₆							
КСІ							
Ca ₂ P ₂ O ₇	11						
KCaPO ₄							

To investigate the conditions for the development of double pyro-, meta-, potassium-, calcium phosphates, the authors of this study calculated the thermodynamic probability of the development of these salts from compounds K_2O , KPO_3 , CaO, P_2O_5 , which are the furnace gas components. The calculation results are presented in Table 2 [7].

Seq. No.	Reaction	∆N 298°C kcal/mol	ΔG -298°C kcal/mol	∆G 1773°C kcal/mol	
1	$K_2O + CaO + P_2O_5 \rightarrow K_2CaP_2O_7$	-187.0	-185.0	-159	
2	2 KPO ₃ + CaO \rightarrow K ₂ CaP ₂ O ₇	-45.0	-44.7	43.0	
3	$K_2O + 2CaO + 3P_2O_5 \rightarrow 2KCa(PO_3)_3$	-294.0	Χ*	_	
4	3 KPO ₃ + CaO \rightarrow KCa(PO ₃) ₃ + K ₂ O	+66.0	+67.0	_	
5	$KPO_x + CaO + P_2O_s \rightarrow KCa (PO_x)_x$	-76.0	-73.6	-	

Table 2. Thermodynamic characteristics of reactions between furnace gas components

Note: *X*^{*} – for compounds involved in the reaction, the calculation was not performed due to the lack of thermodynamic constants in the literature

Thus, from a thermodynamic standpoint, the reaction (reactions 1 and 3) of formation of double pyrophosphate and double metaphosphate from K_2O , CaO, P_2O_5 is possible in the furnace process. Reactions involving HPO₃ are less probable, and in the absence of P_2O_5 are not realised. According to the results of thermodynamic calculations, the study revealed the synthesis of these double compounds. Potassium metaphosphate KH_2PO_4 was pre-formed by heating according to a two-stage method: at t= 100°C, the water is evaporated, then the crucible is slowly cooled, and then heated to 325°C for 1 hour. The diagnosis of obtaining pure KPO₃ by infrared spectroscopy and X-ray phase was made.

The purpose of this study, conducted in the research laboratory of the Zhambyl Branch of LLP "Kazphosphate" (NDFZ) is to investigate the possibility of using freshly yielded toxic dust, as well as the toxic dust from storage tanks to obtain water-soluble complex (NPK) fertilisers.

THE REASONS FOR THE EMERGENCE OF RED PHOSPHORUS IN THE TOXIC DUST OF THE NDFZ (NEW ZHAMBYL PHOSPHORUS PLANT)

40

1000

900

800

700

600

500

400

300

200

100

0

The special composition of the dry residue of the toxic dust was observed at the NDFZ since the launch of the plant into operation. If the phosphorus content is expressed through P_2O_5 , then the sum of the constituent elements exceeded 100%. The studies performed in the laboratory of the Physico-Chemical Research Methods and Analyses (PCRMA) on several samples of elemental phosphorus Py (from benzene extract) and the total (distillation) type amount to 5%. Toxic dust from the NDFZ showed the difference between the definitions. Since it was assumed that the elements in toxic dust

are represented by red phosphorus, the derivatograms of the dry residue of the dust and the red phosphorus sold were removed [8; 9].

According to the derivatogram in Figure 1, the oxidation process of red technical phosphorus proceeds in several stages:

1. The onset of oxidation is observed at 240°C, which is manifested on the thermogram in the form of an exoeffect accompanied by an increase in the sample mass.

2. The second stage of oxidation begins at 350-360°C and is accompanied by an intensive increase in the sample mass.

3. The third stage of oxidation begins at 450-470°C and is accompanied by an intense increase in the sample mass.

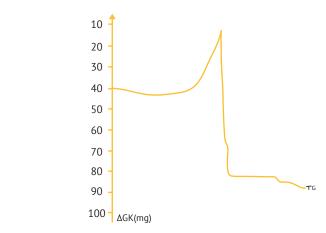


Figure 1. Derivatogram of technical red phosphorus

At temperatures above 500°C (after the end of the exoeffect), a sharp decrease in the mass of the sample begins, which corresponds to the generation of volatile forms of P_2O_5 (white vapour can be visually observed).

500

DTA

150

τ(min)

360

240

On the thermograms of the studied samples of toxic dust (Fig. 2), the oxidation process begins at 200-230°C and is accompanied by an intensive increase in the sample mass to a temperature of 400-420 °C.

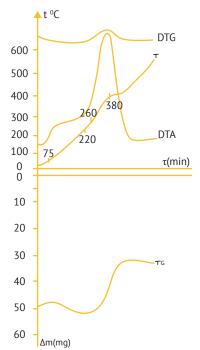


Figure 2. Dervitogram of the solid phase of toxic dust

During the experiments in the electrothermy laboratory in an inert medium, a light brown condensate was yielded, heated by the dry residue of the toxic dust to 280-350 °C. Like red phosphorus, it is stable in air and does not dissolve in organic solvents, but ignites at a temperature lower compared to red phosphorus and instantly discolours a weakly coloured solution of KMnO₄ (red phosphorus discolours when left to stand undisturbed). Upon combustion it gives off white smoke P_2O_5 , like red phosphorus. This suggests that the elementary phosphorus in the test sample has the form of a polymer with a less ordered structure than commercial red phosphorus (when the structure is regulated, the inertia of phosphorus increases) [10].

The study analysed the samples of Sholaktau phosphorite, Zhanatas shale, NDFZ agglomerate in pure form and with a mixture of coke. The light-brown plate on the first gas filter was found only in the agglomerate, while the coke mixture did not increase the yield. Spectral analysis of the coated part of the filter confirmed that the main composition of the light-brown sublimation is phosphorus. When heated up to 8000°C, the filters remained clean, the polymer form transforms into P4 and re-mains in the gas phase [11].

Upon developing a method for determining various forms of carbon in the agglomerate with the preservation of samples in nitrogen, in some cases, a decrease in carbon was detected at 5000°C. Conventionally, it was called "active carbon". It has been suggested that the agglomerate takes part in the low-temperature reduction of certain secondary phosphates generated upon the decomposition of phosphorites during carbon agglomeration. 3 samples with a relatively high total carbon content were tested (Table 3), but various carbon losses were observed at 5000°C, various carbon losses were observed in nitrogen, the third sample was selected in the absence of PO in the factory dust. Perhaps PO is not a consequence of low-temperature reduction of phosphites, for example, heavy metals, but a product of the decay of low-temperature phosphates, an intermediate form of reduction of ortho-, pyro-, metaforms.

Table 3. Comparative analysis of carbon losses in nitrogen at 5000°C									
Sample No.	Amount	The increase in the mass of the							
Sample No.	Total	N _{2 at} 500°C	filter compared to a blank, (g)						
1	1.13	0.62	0.0005						
2	1.06	0.36	0.0011						
3	1.47	1.24	n/a						

However, upon processing the agglomerate, it is inevitable that polymer phosphorus can be released into the toxic dust, and this is conditioned upon the action of residual carbon. Since P0 is oxidised in the liquid phase, oxidants must be introduced upon dust processing [12].

INVESTIGATION OF THE OXIDATION OF ELEMENTAL PHOSPHORUS BY NITRIC ACID IN LABORATORY CONDITIONS

The study comprised three stages of investigating the oxidation of elementary phosphorus with nitric acid, since elementary yellow phosphorus is dangerous for the environment:

 investigation of the possibility of yielding NPK fertilisers from freshly generated toxic dust and dust mixture from components stored for a long time (up to 20 years);

- study of the possibility of yielding NPK fertilisers from fresh toxic dust [13].

The method of investigation of the oxidation of simple phosphorus was as follows. The content of simple yellow and red phosphorus in toxic dust prepared with the ratio S:L=1:1 (S - solid, L - liquid phase) was identified according to the following methods. Upon constant stirring with a laboratory mixer at a speed

of 600 rpm, 56% nitric acid was poured into the dust placed in a porcelain glass in a thin stream and kept at a temperature of 65-700°C for 2 hours. By changing the ratio of toxic dust and nitric acid, complete oxidation of yellow phosphorus in the test sample (99.7%) was achieved (Table 4). The oxidation process of red phosphorus occurring under the action of nitric acid was observed mainly throughout the entire period of research until the emergence of lower phosphate forms [14].

As a result of the 1st stage of the study, it was found that toxic dust oxidised with nitric acid cannot be used as a fertiliser, since a non-technological mass is generated, such as acid resin, which is not suitable for drying and granulation. To neutralise the acid reaction mass, it was decided to use an aqueous solution of ammonia, thereby increasing the nutrient content and obtaining a complex NPK fertiliser. The method of conducting experiments to obtain NPK fertiliser from a mix of toxic dust and a new mixture of toxic dust was as follows. The authors of the study analysed the preformed, average, and sufficient amount of dust yielded from a disk from a depth of 1 m, and fresh dust with a ratio of S:L=1:1. The results of the analysis are presented in Table 5, columns 4, 5. 0.8 weight part of the toxic dust in the current formation and 1.1 weight part of 56% nitric acid were added to one weight part of the toxic dust from the mixer, while stirring constantly at a temperature of 60-70°C for 2 hours at a speed of 600 rpm in a laboratory mixer. Cellulose was neutralised with a concentrated ammonia solution to pH 6-7, dried, ground,

sieved through a 0.5 mm sieve, analysed to prevent nitrogen loss at 600°C. The results of the analysis are presented in Table 5, Figure 1.

Table 4. The results of the analysis of toxic dust													
	dust	oxic 0₃		Analysis of toxic dust after oxidation with HNO3									
Seq. No.	Analysed du	The ratio of toxic dust and HNO ₃	P yellow %	ortho-, poly- phosphate (P ₂ O ₅), %	ortho-, poly-, lower phosphates (P 20 5), %	P ₂ 0 ₃ , %	phospho-rus (P ₂ O ₅), %	P red, %	The degree of oxidation of yellow P, %				
1	Basic	_	0.3	9.8	11.2	1.1	12.3	0.5	-				
2	After oxidation	1:0.5	0.04	10.2	12.3	1.6	13.0	0.3	86.7				
3	-//-	1:0.8	0.01	10.3	12.5	1.7	13.0	0.2	96.7				
4	-//-	1:1.1	0.001	10.4	14.0	2.8	14.1	n/a	99.7				
5	-//-	1:1.3	0.001	10.4	13.7	2.6	13.5	n/a	99.7				

Table 5. The empirical data on yielding NPK fertilisers

		Analysis of the product on dry basis										
Scheme No.	H ₂ 0,%	P yellow, ppm	ortho-, poly- phosph. P ₂ O ₅ , %	Σ ortho-, poly-, lower. P ₂ O ₅ , %	P ₂ O ₃ , %	P ₂ 0 ₅ total, %	P red, %	P ₂ O ₅ absorb., %	P ₂ O ₅ water- soluble., %	CaO water- soluble, %	CaO total, %	N total, %
Scheme 1	11.1	170	22.5	25.1	2.0	25.2	0.04	20.0	9.9	0.4	7.1	8.5
Scheme 2	11.5	70	23.7	24.1	0.2	24.3	0.04	18.8	10.0	1.1	9.1	7.2
Scheme 3	12.6	50	21.3	21.7	0.3	22.1	0.17	16.5	8.6	0.2	10.2	7.5
Scheme 4	13.0	70	22.4	22.6	0.2	22.9	0.13	16.3	8.4	0.2	12.4	7.0
Scheme 5	12.2	40	22.4	22.8	0.3	23.2	0.17	16.2	7.9	0.4	12.5	6.8

To reduce the drying time and minimise the consumption of the ammonia solution, first 0.15 weight part, and then 0.3; 0.4; 0.6 weight parts of the phosphor are introduced into the mixture oxidised with nitric acid, while constantly stirred and heated to 60-700°C for 2 hours. The process was accompanied by intense, active gas generation. Then the cellulose was neutralised with an ammonia solution to pH 6-7 and dried at a temperature of 600°C. From the standpoint of the effectiveness of the neutralised reaction mass, the most optimal was the product obtained according to Scheme 4. It served as the basis for a set of statistical data with samples of toxic dust taken from the surface of the disk from different depths: 5 cm, 15 cm, 30 cm, 50 cm, 100 cm. The analysis results for toxic dust samples are presented in Table 4, columns 6-10, based on which the results of experiments on yielding NPK fertilisers are presented in Table 6.

Analysis of the data in Table 6 suggests that in this series of experiments, the oxidation process of elemental phosphorus turned out to be unsatisfactory. All samples had a characteristic smell of yellow phosphorus, the content of which in the samples was in the range of 150-840 ppm. Apparently, the samples taken from the samples were heterogeneous (the toxic dust in the storage was poorly averaged), and part of the nitric acid was spent on the oxidation of red phosphorus and lower phosphates in the lower layers of the storage [15].

Adjustment of nitric acid consumption was required. The basis for this series of experiments was again Scheme 4 of Table 5. The content of nitric acid – 1,7; 1,6; 1,4; 1,3, from the mass of toxic dust obtained from the storage. The experimental results are summarised in Table 7, diagrams 1-4. The analysis of the empirical data suggests that based on a toxic dust mixture and fresh toxic dust, it is possible to obtain a technological

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product with the characteristics provided in Table 7. The purpose of the next stage of the study was to investigate the composition of the resulting product without the addition of toxic dust in the storage of NPK fertilisers obtained from toxic dust generated at that time. For the experiment, average samples of toxic dust were prepared, the analysis results for which are presented in Table 4, column 11. The previous method served as the basis for this series of experiments, but all the toxic dust was preformed and the required amount of nitric acid was selected empirically, factoring in the large amount of red phosphorus in the source dust.

Table 6. The analysis results for samples of toxic dust taken from the storage surface at different depths

	Analysis of products on dry basis														
Level	H ₂ 0,%	P yellow, ppm	ortho-, poly- phos- phate (P ₂ O ₅), %	Σ ortho-, poly-, lower. P ₂ O ₅ , %	P ₂ O ₃ , %	P_2O_5 total, %	P red, %	P_2O_5 absorb., %	P ₂ O ₅ water-soluble, %	CaO water-soluble, %	CaO total, %	N total, %	N ammon., %	K ₂ O, % acid-degradable	Na ₂ O, % acid degradable
5 cm	10.0	230	11.6	25.5	10.8	25.2	-0.15	18.8	11.0	1.6	11.7	6.7	4.0	19.6	1.7
15 cm	9.0	220	23.7	25.0	1.0	25.4	0.15	18.0	11.3	1.7	10.9	6.7	3.0		
30 cm	5.0	840	23.5	27.0	2.7	31.6	2.0	19.2	12.1	1.9	11.0	7.6	3.1		
50 cm	8.8	150	25.4	27.9	1.9	30.9	1.3	18.0	11.1	1.6	11.0	7.2	3.2		
100 cm	14.6	360	25.4	27.0	1.2	29.6	1.1	19.0	10.3	1.4	10.9	7.5	3.4	19.4	1.7

 Table 7. Study results

Analysis of products on dry basis

Scheme No.	H ₂ 0,%	P yellow, ppm	ortho-, poly- phosphate (P ₂ O ₅), %	Σ ortho-, poly-, lower, (P ₂ O ₅), %	P ₂ O ₃ , %	P_2O_5 total, %	P red, %	P ₂ O ₅ absorb., %	P ₂ O ₅ soluble, %	CaO soluble, %	CaO total, %	N total, %	N ammon., %
1	2.0	40	21.9	23.2	1.0	23.2	n/a	17.1	11.0	1.5	9.8	10.0	4.6
2	2.6	40	20.0	20.8	0.6	20.8	n/a	16.1	9.0	1.1	9.9	12.3	4.1
3	4.0	40	19.9	20.8	0.7	20.5	n/a	16.2	9.1	1.3	10.2	11.2	4.6
4	3.7	40	21.0	21.8	0.6	21.9	n/a	16.9	8.7	1.2	10.3	11.2	4.3

CONCLUSIONS

For a complex fertiliser, a product with acceptable characteristics has been successfully obtained. According to the content of absorbing and water-soluble $P_2O_{5^3}$ total and ammonium nitrogen, it is similar to the product currently obtained from a mixture of storage dust and freshly generated toxic dust, but due to the considerable content of elemental red phosphorus in the initial toxic dust, the content of total and incomplete oxidised phosphates P_2O_5 is much higher. The general conclusion based on the empirical data found at stages II and III: to obtain a product suitable for fertilisation, it is necessary to strictly control the content of elemental phosphorus in the initial toxic dust and adjust the consumption of nitric acid based on its results.

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Дослідження умов утворення фазових компонентів К₂CaP₂O₇, KCaP₃O₉ котрельного пилу та «молока»

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Анотація. У статті проведено аналіз складу та основних компонентів котрельного пилу. Для розробки та розуміння методики управління процесом пилоутворення необхідно вивчити дані, що надають детальну інформацію про механізм реакції. Результати досліджень фазового складу пилу, проведених у лабораторії, і їх зіставлення з даними, отриманими раніше іншими авторами, дозволили встановити досить достовірний діагноз фазового складу пилу. Під час аналізу було встановлено, що пил різних фосфорних заводів складається з одних і тих же основних компонентів, тільки з'ясовується, що хімічні зв'язки між ними різні. Метою даної роботи, проведеної в дослідницькій лабораторії ДФ ТОВ «Казфосфат» (НДФЗ), є вивчення можливості використання нового котрельного молока і котрельного молока з накопичувачів для отримання NPK добрив. Дослідницька робота складалася з трьох етапів вивчення процесу окислення елементарного фосфору азотною кислотою, оскільки елементарний жовтий фосфор небезпечний для екології. У результаті 1-го етапу дослідження було встановлено, що котрельне молоко, окислене азотною кислотою, не може бути використано в якості добрива, оскільки утворюється нетехнологічна маса, така як кисла смола, яка не підходить для сушки і грануляції. Для нейтралізації кислотної реакційної маси вирішили використовувати водний розчин аміаку, тим самим збільшивши вміст поживних речовин і отримавши комплексне NPK добриво. У процесі дослідження авторами було встановлено на 2-му і 3-му етапах експерименту, що для отримання продукту, придатного для використання в якості добрива, необхідно строго контролювати вміст елементарного фосфору у вихідному котрельном молоці і за його результатами коригувати витрати азотної кислоти

Ключові слова: азотна кислота, вуглець, фосфор, зразок, температура, експеримент