The process of decomposition of tricalcium phosphate by phosphoric acid with partial replacement of P_2O_5 to sulfuric acid

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Abstract. The article covers the decomposition of tricalcium phosphate with thermal phosphoric acid with a concentration of 20, 30 and 40% P_2O_5 with the replacement of 10, 20 and 30% P_2O_5 with sulfuric acid and it has been established that, regardless of the content of ammonium nitrate, magnesium sulfate or their combined presence in phosphoric acid, with an increase in the proportion of sulfuric acid, the content of both total and aqueous forms of SO_3 in the acidic pulp increases. The highest results of the decomposition coefficient were obtained when phosphoric acid was evaporated to a content of 32.22-25.77% P_2O_5 and 5.54-9.76% SO_3. The SO_3 content of both total and aqueous forms in dried products increases with increasing proportion of sulfuric acid. When the proportion of sulfuric acid increases to 20%, the degree of decomposition increases to 93.45% and to 99.55% when replacing 30% P_2O_5 with sulfuric acid.

1 Introduction

Due to the depletion of rich deposits of phosphate raw materials worldwide, a high attention is paid to the development of technology for concentrated phosphorus fertilizers involving low-grade and high-magnesium phosphorites in the production [1]. In this regard, providing the agro-industrial complex with the necessary plant protection products, stimulants of plant growth and development, mineral and organic-mineral fertilizers in a wide range with different ratios of the main macroelements such as nitrogen, phosphorus, potassium, calcium, magnesium, sulfur is an important direction in increasing productivity agricultural plants [2].

In this regard, substantiation of scientific and technical solutions for the development of technologies for single and concentrated phosphorus fertilizers with a water-soluble form of sulfates is important. In order to provide agricultural production with them, it is necessary to justify a number of existing scientific solutions, establishing optimal technological parameters for the partial replacement of phosphoric acid with sulfuric acid, the degree of ammonization of acidic pulp, at which the maximum content of water-soluble sulfates is

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observed, and developing a technology for producing concentrated nitrogen-phosphorus fertilizers with a water-soluble form of sulfates.

Concentrated WPPA is the basis for the production of double superphosphate. WPPA is made from high-quality raw materials, is well evaporated to a concentration of 54% P_2O_5 and is used to produce double superphosphates. WPPA, obtained from magnesium-containing raw materials, for example, from phosphorites of Karatau, Kizylkum and Kingisepp deposits, due to the content of magnesium impurities in it, thickens at a concentration of 37-38% P_2O_5. It is argued that the presence of more than 0.3% MgO in phosphate raw materials causes difficulties in concentrating WPPA due to the formation of insoluble magnesium phosphate, leading to the formation of sludge. Other authors believe that the thickening of magnesium-containing WPPA, evaporated to a concentration of 37-38% P_2O_5, occurs due to the formation of MgF_2 and SiO_2 as a result of the hydrolysis of $MgSiF_6$.

By studying the properties of real solutions of EPA from Karatau phosphorites depending on the ratio of impurity compounds of magnesium, iron, aluminum, sulfuric acid and others, it was established that the thickening of WPPA as it concentrates occurs due to the presence of magnesium in the acid in the form of magnesium sulfate hydrates. WPPA (21% P_2O_5) from Karatau phosphorites contains 1.5-2.0% MgO, i.e., 4.5-6.0% $MgSO_4$ or 9.0-12.0% $MgSO_4 \cdot 7H_2O$ in dissolved form, and when the concentration increases to 37-38% P_2O_5, the content of this salt reaches 16-21%. In this case, the solution is oversaturated with magnesium sulfate hydrates. When cooled, they crystallize and associate water molecules (this property is inherent in all crystalline hydrates), as a result of which the solution thickens. Other impurities present (calcium sulfate, iron and aluminum phosphates, sodium and potassium silicofluorides), especially sulfuric acid, enhance the thickening process.

In order to eliminate thickening, i.e., to improve the rheological properties, a chemical reagent ammonium nitrate was used, and the solubility of magnesium compounds in a solution of this reagent was studied [3-5].

2 Methods

In order to theoretically substantiate the processing of phosphorites into single phosphorus fertilizers with phosphoric acid, with partial replacement of P_2O_5 phosphoric acid with sulfuric acid, studies were carried out on the decomposition of tricalcium phosphate with thermal phosphoric acid containing 20% P_2O_5 with the replacement of 10, 20 and 30% P_2O_5 with sulfuric acid at a total stoichiometric normal acid.

Research of the decomposition process of tricalcium phosphate was also carried out with more concentrated thermal phosphoric acid, and replacing 10, 20 and 30% P_2O_5 with H_2SO_4 with a concentration of 91.75% in a laboratory installation. The laboratory setup consisted of a reactor equipped with a screw stirrer with an electric motor and placed in a water thermostat. The temperature in the thermostat was maintained using a contact thermometer. The temperature in all experiments was constant, and it was equal to 80 °C and the process duration was 2 hours.

After reaching the set temperature, the calculated amount of tricalcium phosphate was dosed. For a certain period of time, pulp samples were taken for analysis to determine the content of various forms of P_2O_5, CaO, SO_3 and calculation of the decomposition coefficient of tricalcium phosphate, the extraction coefficients of CaO and SO_3 into the liquid phase.

3 Results and discussion

Research on the decomposition of tricalcium phosphate 20% into P_2O_5 by thermal phosphoric acid with partial replacement of P_2O_5 by sulfuric acid were carried out with phosphoric acid containing 1% magnesium sulfate. The need for these studies is justified by the fact that phosphorites of Karatau and Central Kyzylkum contain magnesium, and WPPA based on them contains magnesium sulfate, which significantly affects the activity and rheological properties of the acid. The results obtained are shown in Table 1.

Adding 1% $MgSO_4$ to the original phosphoric acid containing 20% P_2O_5 and replacing 10% P_2O_5 with sulfuric acid leads to a decrease in P_2O_5 to 16.25%, which contains 4.58% SO_3.

Evaporation of these acids allows obtaining phosphoric acid containing 24.38-19.50% P_2O_5 and 5.08-8.28% SO_3, as well as 32.50-26.00% P_2O_5 and 5.59-9.85% SO_3.

When decomposing tricalcium phosphate with thermal phosphoric acid, with an initial concentration of 16.25% P_2O_5 and containing 4.58% SO_3, the content of P_2O_{5total} is 21.12%, $P_2O_{5assim,}$ is 20.01% and P_2O_{5ws} is 18.39%. The ratio of assimilated forms to the total form increases from 94.74% to 95.87% and 98.27% and aqueous from 87.07% to 90.03% and 91.85%, respectively. Accordingly, the decomposition coefficient of tricalcium phosphate increases from 85.30% to 89.27% and 96.44% [6-10].

When replacing 10% P_2O_5 phosphoric acid with H_2SO_4, the content of SO_{3total} in the pulp is 3.83% and SO_{3aq} is 1.16%. An increase in the rate of sulfuric acid leads to an increase in the content of the total form of SO_3 to 4.69% when replacing 20% P_2O_5 with H_2SO_4 and to 5.57% when replacing 30%. At the same time, the content of the water-soluble form of SO_3 is 1.28% and 1.13%.

When using more concentrated phosphoric acid, the content of the total form of SO_3 is 3.92-7.01% and aqueous is 1.15-1.44%. Increasing the concentration of H_3PO_4 and replacing 10-30% of P_2O_5 with H_2SO_4, the extraction coefficient of SO_3 into the liquid phase decreases and amounts to from 30.29-20.29% to 29.34-20.63% and to 30.42-20.54%.

Table 1. The effect of partial replacement of phosphoric acid containing magnesium sulfate with sulfuric acid on the chemical composition of the product.

No.	Indicators	Content of components in pulp, mass. %								
		When replacing H_3PO_4 with H_2SO_4. %								
		10	20	30	10	20	30	10	20	30
1.	Initial concentration of P_2O_5 in WPPA, %	16.25	14.65	13.00	24.38	21.98	19.50	32.50	29.30	26.00
2.	Initial concentration of SO_3 in WPPA, %	4.58	5.63	6.70	5.08	6.66	8.28	5.59	7.69	9.85
3.	Content in pulp:									
	P_2O_5 (total.), %	21.12	19.85	18.53	29.27	28.29	25.64	36.26	34.02	31.72
	P_2O_5 (assim.), %	20.01	19.03	18.21	28.04	27.29	25.04	35.90	33.74	31.52
	P_2O_5 (aq.s), %	18.39	17.87	17.02	25.58	25.58	23.62	32.95	31.53	30.27
	SO_3 (total), %	3.83	4.69	5.57	3.92	5.12	6.35	4.01	5.49	7.01
	SO_3 (aq.s.), %	1.16	1.28	1.13	1.15	1.41	1.31	1.22	1.44	1.44
	CaO (total), %	8.93	9.04	9.13	12.36	14.35	16.49	15.32	15.48	15.64
	CaO (aq.s.), %	2.26	2.06	1.45	3.07	3.03	2.67	3.92	3.37	2.52

	MgO %	1.49	1.48	1.48	1.37	1.37	1.37	1.28	1.27	1.27
	Humidity, %	51.7	52.3	53.0	37.3	38.2	39.1	25.0	26.1	27.3
4.	(P_2O_5 assim.: P_2O_5total.)×100, %	94.7	95.9	98.3	95.8	96.5	97.7	99.1	99.2	99.4
5.	(P_2O_5aq.s.:P_2O_5total)×100, %	87.1	90.0	91.8	87.4	90.4	92.1	90.9	92.7	95.4
6.	Kp according P2O5assim, %	85.3	89.3	96.4	88.2	90.8	95.2	97.2	97.9	98.7
7.	Kextr. according CaO in aqueous solution, %	25.3	22.8	15.9	24.8	21.1	16.2	25.6	21.8	16.1
8.	Degree of SO_3 extraction into aqueous solution, %	30.3	27.3	20.3	29.3	27.5	20.6	30.4	26.2	20.5

Table 2 shows the results of the drying process of tricalcium phosphate decomposition products with 20% thermal phosphoric acid containing 1% magnesium sulfate, replacing 10%, 20% and 30% P_2O_5 with sulfuric acid.

The pulps were dried in a drying oven at a temperature of 105 °C until the moisture content was less than 3%.

The table shows that during the drying process, due to the removal of moisture, the content of the total form of P_2O_5 increases to 42.87%, when using acid 16.25% P_2O_5 with a content of 4.58% SO_3 and decreases to 40.86% for acid 14.65% P_2O_5 and 5.63% SO_3 and up to 38.70% for acid 13.00% P_2O_5 and 6.70% SO_3. The content of the assimilated form is 40.70%, the aqueous form is 37.45-35.62%.

At the same time, the ratio of the assimilated form to the total form increases from 94.95% when replacing 10% P_2O_5 with sulfur to 96.06% when replacing 20% P_2O_5 and to 98.55% when replacing 30% P_2O_5.

The ratio of the water-soluble form to the total form is, respectively, 87.35%, 90.75% and 92.04%. The decomposition coefficient is 85.86%, 89.76% and 97.04% when replacing 10%, 20% and 30% P_2O_5 with sulfuric acid.

The use of more concentrated phosphoric acid with the replacement of 10, 20 and 30% P_2O_5 with sulfuric acid allows obtaining a product with a higher degree of decomposition. When decomposing tricalcium phosphate with phosphoric acid containing 24.38% P_2O_5 and 5.08% SO_3, the degree of decomposition is 89.30%, and with acid 32.50% P_2O_5 and 5.59% SO_3, it is 98.60%.

When the proportion of sulfuric acid increases to 20%, the degree of decomposition increases to 92.39% and to 98.90% when replacing 20% P_2O_5 with sulfuric acid. The highest results of the decomposition coefficient were obtained when phosphoric acid was evaporated to a content of 32.50-26.00% P_2O_5 and 5.59-9.85% SO_3. The SO_3 content of both total and aqueous forms in dried products increases with increasing proportion of sulfuric acid. When using extraction phosphoric acid activated with ammonium nitrate, it contains magnesium sulfate as a concomitant component of phosphorites of Karatau and Central Kyzylkum.

Table 2. The effect of partial replacement of phosphoric acid containing magnesium sulfate with sulfuric acid on the chemical composition of the product.

No.	**Indicators**	**Content of components, mass. %**								
		When replacing H_3PO_4 with H_2SO_4, %								
		10	**20**	**30**	**10**	**20**	**30**	**10**	**20**	**30**
1.	Initial concentration of P_2O_5 in WPPA, %	16.25	14.65	13.00	24.38	21.98	19.50	32.50	29.30	26.00
2.	Initial concentration of SO_3 in WPPA, %	4.58	5.63	6.70	5.08	6.66	8.28	5.59	7.69	9.85

3.	Content in product:									
	P_2O_5 (total.), %	42.87	40.86	38.70	45.29	44.86	41.21	47.24	45.67	42.99
	P_2O_5 (assim.), %	40.70	39.25	38.14	43.56	43.55	40.36	47.00	45.48	42.86
	P_2O_5 (aq.s), %	37.45	37.08	35.62	39.99	40.78	38.20	43.11	42.43	41.07
	SO_3 (total), %	7.77	9.65	11.63	6.07	8.12	10.21	5.22	7.37	9.50
	SO_3 (aq.s.), %	2.37	2.65	2.38	1.81	2.26	2.21	1.62	1.97	1.98
	CaO (total), %	18.12	18.61	19.07	19.13	22.75	26.50	19.96	20.78	21.15
	CaO (aq.s.), %	4.70	4.28	3.10	4.80	4.75	4.52	5.26	4.67	3.58
	MgO %	3.02	3.16	3.09	2.12	2.17	2.20	1.67	1.70	1.72
	Humidity, %	2.05	1.88	1.85	3.05	2.04	2.19	2.33	0.85	1.48
4.	(P_2O_5assim.:P_2O_5total.)×100, %	94.95	96.06	98.55	96.18	97.07	97.94	99.50	99.58	99.70
5.	(P_2O_5aq.s.:P_2O_5total) ×100, %	87.35	90.75	92.04	88.30	90.90	92.70	91.25	92.90	95.53
6.	Kp according P_2O_5assim, %	85.86	89.76	97.04	89.30	92.39	95.79	98.60	98.90	99.39
7.	Kextr. according CaO in aqueous solution, %	25.94	23.00	16.26	25.09	20.88	17.06	26.35	22.47	16.93
8.	Degree of SO_3 extraction into aqueous solution, %	30.50	27.46	20.46	29.82	27.83	21.64	31.03	26.73	20.84

Therefore, further research on the decomposition of tricalcium phosphate with phosphoric acid with partial replacement of P_2O_5 with sulfuric acid were carried out with 20% thermal phosphoric acid containing 1% each of ammonium nitrate and magnesium sulfate. The results obtained are shown in Table 3.

Further research on the decomposition of tricalcium phosphate with phosphoric acid with partial replacement of P_2O_5 with sulfuric acid were carried out with 20% thermal phosphoric acid containing ammonium nitrate (1%) and magnesium sulfate (1%).

Evaporation of these acids allows obtaining phosphoric acid containing 24.17-19.33% P_2O_5 and 5.04-8.21% SO_3, as well as 32.22-25.77% P_2O_5 and 5.54-9.79% SO_3.

When decomposing tricalcium phosphate with thermal phosphoric acid with an initial concentration of 16.10% P_2O_5 and containing 4.54% SO_3, the content of P_2O5_{total} is 20.95% P_2O_5aq.s 19.89%, P_2O_5assim. 18.42%. With an increase in the amount of replacement of P_2O_5 with sulfuric acid, the ratio of assimilated forms to the total form increases from 94.94% to 96.14% and 98.86% and aqueous from 87.92% to 90.86% and 92.55%, respectively. The decomposition rate of tricalcium phosphate increases from 85.83% to 89.97% and 97.67%.

When using more concentrated phosphoric acid, the content of the total form of SO_3 is 3.90-6.96% and aqueous 1.34-1.54%. SO_3 extraction coefficient into the liquid phase, with increasing proportion of H_2SO_4 decreases and amounts to 35.26-22.24% and 34.36-22.19% and 35.43-22.13%.

Table 3. The effect of partial replacement of phosphoric acid containing ammonium nitrate and magnesium sulfate with sulfuric acid on the chemical composition of the pulp.

No.	**Indicators**	**Content of components in pulp, mass. %**								
		When replacing H3PO4 with H2SO4, %								
		10	**20**	**30**	**10**	**20**	**30**	**10**	**20**	**30**
1.	Initial concentration of P_2O_5 in WPPA, %	16.10	14.52	12.89	24.17	21.78	19.33	32.22	29.04	25.77
2.	Initial concentration of SO_3 in WPPA, %	4.54	5.58	6.64	5.04	6.60	8.21	5.54	7.63	9.76

3.	Content in pulp:									
	P_2O_5 (total.), %	20.95	19.70	18.40	29.07	29.29	25.47	36.04	33.80	31.50
	P_2O_5 (assim.), %	19.89	18.94	18.19	27.94	28.27	24.94	35.74	33.58	31.35
	P_2O_5 (aq.s), %	18.42	17.90	17.03	25.64	26.76	23.66	32.96	31.60	30.30
	SO_3 (total), %	3.90	4.66	5.53	3.90	5.09	6.31	3.98	5.46	6.96
	SO_3 (aq.s.), %	1.34	1.36	1.23	1.34	1.50	1.40	1.41	1.55	1.54
	CaO (total), %	8.85	8.96	9.07	12.28	12.42	10.62	15.22	15.38	15.51
	CaO (aq.s.), %	2.68	2.25	1.86	3.71	3.20	2.24	4.62	3.91	3.39
	MgO %	1.48	1.48	1.47	1.37	1.36	1.36	1.27	1.27	1.26
	N %	0.29	0.29	0.29	0.27	0.27	0.27	0.25	0.25	0.25
	Humidity, %	51.27	51.86	52.45	36.98	37.88	38.77	24.74	25.88	27.06
4.	(P_2O_5assim:P_2O_5total.)× 100, %	94.94	96.14	98.86	96.11	96.52	97.92	99.17	99.35	99.52
5.	(P_2O_5aq.s.:P_2O_5total) ×100, %	87.92	90.86	92.55	88.20	91.36	92.89	91.45	93.49	96.19
6.	Kp according P_2O_5assim, %	85.83	89.97	97.67	89.11	90.96	95.75	97.68	98.31	99.02
7.	Kextr. according CaO in aqueous solution, %	30.28	25.11	20.51	30.21	25.76	21.09	30.36	25.42	21.86
8.	Degree of SO_3 extraction into aqueous solution, %	35.26	29.18	22.24	34.36	29.47	22.19	35.43	28.39	22.13

Table 4 shows the results of the process of drying the decomposition products of tricalcium phosphate with 20% thermal phosphoric acid containing 1% ammonium nitrate and magnesium sulfate, replacing 10%, 20% and 30% P_2O_5 with sulfuric acid.

The pulps were dried in a drying oven at a temperature of 105 °C until the moisture content was less than 3%. The table shows that during the drying process, due to the removal of moisture, the content of the total form of P_2O_5 increases to 41.28% when using acid 16.10% P_2O_5 with a content of 4.54% SO_3 and decreases to 39.35% P_2O_5 for acid 14.52% P_2O_5 and 5.58% SO_3 and up to 37.63% P_2O_5 for acid 12.89% P_2O_5 and 6.64% SO_3.

The content of the assimilated form is 39.52%, the aqueous form is 36.42-35.08%. At the same time, the ratio of the assimilated form to the total form increases from 95.74% when replacing 10% P_2O_5 with sulfur to 96.90% when replacing 20% P_2O_5 and to 99.70% when replacing 30% P_2O_5. The ratio of the water-soluble form to the total form is, respectively, 88.23%, 91.26% and 93.22%.

The decomposition coefficient is 88.07%, 91.94% and 99.39% when replacing 10%, 20% and 30% P_2O_5 with sulfuric acid. The use of more concentrated phosphoric acid with the replacement of 10, 20 and 30% P_2O_5 with sulfuric acid allows obtaining a product with a higher degree of decomposition.

When decomposing tricalcium phosphate with phosphoric acid containing 24.17% P_2O_5 and 5.04% SO_3, the degree of decomposition is 91.20%, and with acid 32.22% P_2O_5 and 5.54% SO_3, it is 98.04%.

The highest results of the decomposition coefficient were obtained when phosphoric acid was evaporated to a content of 32.22-25.77% P_2O_5 and 5.54-9.76% SO_3. The SO_3 content of both total and aqueous forms in dried products increases with increasing proportion of sulfuric acid.

Table 4. The effect of partial replacement of phosphoric acid containing ammonium nitrate and magnesium sulfate with sulfuric acid on the chemical composition of the product.

No.	Indicators	Content of components, mass. %								
		When replacing H3PO4 with H2SO4, %								
		10	**20**	**30**	**10**	**20**	**30**	**10**	**20**	**30**
1.	Initial concentration of P_2O_5 in WPPA, %	16.10	14.52	12.89	24.17	21.78	19.33	32.22	29.04	25.77
2.	Initial concentration of SO_3 in WPPA, %	4.54	5.58	6.64	5.04	6.60	8.21	5.54	7.63	9.76
3.	Content in product:									
	P_2O_5 (total.), %	41.28	39.35	37.63	45.17	45.69	41.60	46.94	44.52	41.77
	P_2O_5 (assim.), %	39.52	38.13	37.51	43.75	44.54	41.06	46.61	44.29	41.68
	P_2O_5 (aq.s), %	36.42	35.91	35.08	40.17	42.17	39.18	43.30	41.97	40.62
	SO_3 (total), %	7.49	9.31	11.31	6.06	7.94	10.07	5.18	7.19	9.23
	SO_3 (aq.s.), %	3.02	3.06	2.87	2.32	2.56	2.70	1.88	2.18	2.34
	CaO (total), %	17.44	17.90	18.55	19.08	19.38	16.95	19.82	20.26	20.57
	CaO (aq.s.), %	6.14	5.07	4.37	6.64	5.56	4.25	6.47	5.75	4.95
	MgO %	2.94	2.96	3.01	2.13	2.12	2.17	1.65	1.67	1.67
	N %	0.57	0.57	0.57	0.42	0.42	0.42	0.32	0.32	0.32
	Humidity, %	3.98	3.85	2.75	2.08	3.09	2.27	1.97	2.37	3.28
4.	(P_2O_5assim:P_2O_5total.)×100, %	95.74	96.90	99.70	96.86	97.48	98.70	99.30	99.48	99.78
5.	(P_2O_5aq.s.:P_2O_5total)×100, %	88.23	91.26	93.22	88.93	92.29	94.18	92.25	94.27	97.25
6.	Kp according P_2O_5assim, %	88.07	91.94	99.39	91.20	93.45	97.34	98.04	98.65	99.55
7.	Kextr. according CaO in aqueous solution, %	35.21	28.32	23.56	34.80	28.69	25.07	32.64	28.38	24.06
8.	Degree of SO_3 extraction into aqueous solution, %	40.32	32.87	25.36	38.28	32.24	26.81	36.29	30.32	25.35

4 Conclusion

It has been established that the highest results of the decomposition coefficient were obtained when phosphoric acid was evaporated to a content of 32.22-25.77% P_2O_5 and 5.54-9.76% SO_3. The SO_3 content of both total and aqueous forms in dried products increases with increasing proportion of sulfuric acid. The proportion of the water-soluble form of SO_3 relative to the total form when replacing 10% P_2O_5 with sulfur changes from 40.32% to 36.29%, when replacing 20% from 32.87% to 30.32%, and when replacing 30% from 25.36% to 25.35%. When the portion of sulfuric acid increases to 20%, the degree of decomposition increases to 93.45% and to 99.55% when replacing 30% P_2O_5 with sulfuric acid.

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