COMBINED TECHNOLOGY FOR PRODUCING COMPOUND FERTILIZERS FROM LOW- GRADE PHOSPHORITES FROM CENTRAL KYZYLKUM

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Article History: **Received**: 18.06.2022 **Revised**: 17.07.2022 **Accepted**: 09.08.2022

Abstract: The question of the organisation of NPK fertilizers manufacture with the maintenance not less than 40 % of nutritious elements from phosphorites Central Kizikum concentrated is considered. In the developed flow sheet use of enriched and washed dried phosphorite containing 17-26 % P_2O_5 is provided. At low maintenance P_2O_5 in phosphorite raw materials it preliminary is exposed to acid enrichment. It was shown the possibility that nitrogen-phosphate-potassium fertilizers and nitro – ammophos - potassium can be prepared based on nitric and sulfuric acid processing low-grade phosphorite from Central Kyzyl Kum. The rough calculation of the consumption coefficients of obtaining nitro – ammophos - potassium and nitro-phosphate - potassium (mono- and dicalcium nitro-phosphate - potassium) at various CaO / P_2O_5 ratios in washed dried phosphorite with a content of 26% P_2O_5 was carried out. The triple fertilizers at least 40% nutrient elements are suitable for application in agriculture where their efficiency will have great significance in term of yield. The advantages of the developed scheme are suitable in terms of production of concentrated NPK fertilizer contenting at least 40% of nutrients, flexibility that makes it possible to obtain, except for NPK fertilizers, various technical phosphorus salts, large energy costs, possibility of using any kind of phosphate raw materials.

Keywords: phosphorite powder, washed dried phosconcentrated phosphogypsum, ammonization, nitrogen-ammophos-potassium, carbamide-ammophos-potassium, complex fertilizer.

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DOI: 10.31838/ecb/2022.11.06.002

INTRODUCTION

Currently, there is a growing demand on the world market for complex fertilizers containing nitrogen, phosphorus and potassium. The advantage of complex fertilizers is their high agrochemical efficiency, as well as a sharp reduction in the consumption of fuel and lubricants (by 2.5 times) due to the simultaneous introduction of basic nutrients into the soil, which reduces the cost of crops. For individual natural and economic regions, the range of complex fertilizers varies within five to eight brands. The leading role in the range belongs to fertilizers with a balanced ratio of nutrients (1: 1: 1). Such a brand can be represented by nitrogen-phosphate-potassium, nitrogen-ammophos, nitrogen-phosphate, as well as carbamide-ammophos-potassium. These fertilizers are used in various soil and climatic zones, especially in the non- black earth zone, where all types of fertilizers - nitrogen, phosphorus and potash - are effective. On light and sandy loam soils, fertilizers in a ratio of 1: 1: 1 are used in the spring before sowing under spring crops, potatoes, sugar beets, annual grasses, and on soils with a heavy texture, also in autumn under the fall [1, 2].

The number of brands of complex fertilizers produced by various US companies is large. This can be explained not only by the desire to have brands of fertilizers for various crops, but also by the production technology, as well as the quality of raw materials. The number of major brands in terms of the ratio of nutrients is relatively small. So, compound fertilizers are 80% represented by the ratio of nutrients 1: 1: 1; 1: 2: 2; 1: 4: 4; 1: 2: 0 and 1: 2: 3.

For different crops on soils with different supply of nutrients, the following ratios are recommended 1: 1: 1; 1.5: 1: 1; 1: 4: 4; 1: 1: 0; 1: 2.8: 0; 1: 1: 0 [2]. By changing the ratio of nutrients in nitrogen - phosphate - potassium, it is possible to meet the needs of a variety of crops cultivated on different soils. For example, in France, a technology was developed for obtaining 54 types of nitrogen - phosphate - potassium with a nitrogen content of 8 to 20%, P_2O_5 from 7 to 35% and K_2O up to 29%.

The practice of using NPK fertilizers has shown the sufficiency of a relatively small number of options for the ratio of nutrients. For example, for cotton, the ratio N: P_2O_5: K_2O, 1: 2: 0; 1: 0.5: 0; or 1: 1: 0; 1: 0.75: 0.5; for cereals-1: 2: 2; 1: 2: 1; or 1: 1: 1; for sugar beets -1: 2: 1; 1: 1.5: 1.5, etc [3-6]. Whereas for the yield of lemon and the quality of the fruit, NPK with nutrients is required: N = 220 kg ha^{-1}, P = 20 kg ha^{-1} and K = 310 kg ha^{-1} [7].

To obtain fertilizers with a balanced ratio of active ingredients, the neutralization of phosphoric acid with ammonia can be accompanied by the addition of other nitrogen-containing components to the reaction mixture - nitric acid, solutions or melts of ammonium and urea nitrate, etc. When combining ammonium phosphates with ammonium nitrate in such fertilizers, they are called nitrogen-ammophos N + P), and with carbamide – carbamide-ammophos (N + P); with the addition of potassium salts (KCI or K_2SO_4), triple fertilizers (N + P + K) are obtained – nitrogen-ammophos-potassium and carbamide-ammophos-potassium. All components of these fertilizers are readily soluble in water [1].

Granular nitroammophos and nitroammophos potassium, carboammophos and carboammophos potassium are highly concentrated ballastless fertilizers. The content of active substances in them may exceed 55%. There is ability to change easily the ratio of ammonium phosphates and other components - ammonium nitrate, carbamide, potassium salts - makes it possible to obtain these fertilizers with any given ratio of nutrients, and the use of acids of a sufficiently high concentration and melts reduces the energy costs for processing the neutralized mass into solid granular products. It turns out, for example, it is possible to combine the neutralization of acids with ammonia with drying the product, which is completely carried out by the heat of reaction without additional heat input from the outside [8].

The absence of calcium compounds in the reaction mass (when using extraction phosphoric acid, the CaO concentration does not exceed 1%) makes it possible to carry out fast and deep ammonization, since the retrogradation of phosphorus (formation of tricalcium phosphate) is excluded under these conditions. Therefore, ammonization can be carried out until all phosphorus is converted to diammonium phosphate to obtain, for example, diammonium-nitrogen-potassium [9].

Carbamide-ammophos and carbamide-ammophos-potassium can be obtained by neutralizing phosphoric acid with a carbamide-containing melt and mixing carbamide with a solution of monoammonium phosphate, followed by processing the resulting solution or melt into a granular product. In this case, partial hydrolysis of carbamide and dissociation of ammonium phosphate with the release of NH_3 can take place, which must be captured by phosphoric acid, which is sent for neutralization.

As known, the production of complex fertilizers by nitric acid decomposition of phosphates consists of two stages:

- obtaining nitric acid extract by decomposition of natural phosphate-apatite concentrate or phosphorite with nitric acid;
- processing of nitric acid extract into the final product. Nitric acid extract is obtained by the reaction:

$$Ca_5F(PO_4)_{3\,(s)}+10HNO_{3\,(aq)}=3H_3PO_{4(aq)}+5Ca(NO_3)_{2\,(aq)}+HF_{(g)}$$

It is a solution of phosphoric acid and calcium nitrate [10]. In addition, it contains the products of the interaction of raw material impurities with nitric acid. The liquid phase of the resulting pulp contains various amounts of HNO_3, H_3PO_4, $Ca(NO_3)_2$, Fe $(NO_3)_3$, $Mg(NO_3)_2$, $Al(NO_3)_3$, HF and H_2SiF_6; insoluble substances remain in the solid phase. Phosphates can be decomposed by a mixture of nitric acid with sulfuric or phosphoric acids, as well as with ammonium or potassium sulfate [1, 11-14].

The processing of the extract into final products consists in the neutralization of phosphoric acid with ammonia and followed by the releasing part of the calcium in the form of non-phosphate compounds, since the ratio of CaO / P_2O_5 in the fertilizer should be less than in the initial raw material. Depending on the method of leaching from the system or binding of a part of calcium, the following methods of processing nitric acid leaching and obtaining complex fertilizers are distinguished as follows:

- cooling and crystallization of calcium nitrate from solution;
- precipitation of excess calcium in the form of $CaCO_3$ by carbonization of ammoniated pulp (carbonate method);
- precipitation of excess calcium in the form of $CaSO_4$ with sulfuric acid (nitric-sulfuric acid method) or ammonium, sodium or potassium sulfates (sulfate method);
- the recovering calcium from the solution by these methods can be done by the process of nitric acid decomposition of phosphate. This makes it possible, under certain conditions, to carry out the process with a reduced consumption of nitric acid (with an incomplete norm of nitric acid calculated as CaO);
- introducing an additional amount of phosphoric acid (extraction or thermal) into the system to obtain the required ratio between CaO and P_2O_5.

Thus, neutralized pulp, obtained in one way or another, is mixed with potassium salt (if it was not introduced earlier during the preparation of the pulp) and dried in a mixture with the recycle of the finished product [1].

The aim of the work was to analyze the route of organizing the production of competitive products from the phosphorites of the Central Kyzyl Kum - a concentrated NPK fertilizer containing at least 40% of nutrients.

EXPERIMENTAL

There have been used two kind of Kyzyl Kum phosphorites in this study. Washed dried phosconcentrated contents (wt.%): 26.08 P_2O_5, 51.74 CaO, 0.89 MgO, 1.02 Al_2O_3, 0.31 Fe_2O_3, 9.95 CO_2, 1.59 SO_3, 3.41 F, 2.49 insoluble residue and phosphorite powder (low-grade phosphorite) (wt.%): 17.76 P_2O_5, 47.51 CaO, 1.79 MgO, 0.95 Al_2O_3, 0.73 Fe_2O_3, 17.02 CO_2, 3.27 SO_3, 2.01 F, 5.27 insoluble residue. JSC Kyzyl Kum phosphorite combine supplied these raw materials. Both washed dried phosconcentrated and low grade phosphorite were milled prior to particle size 0.25 mm. Nitric acid (57%) and sulfuric acid (94%) were purchased from Himreaktiv Ltd. Gaseous ammonia (99.9%) was purchased from Himlabpribor, Ltd.

RESULTS AND DISCUSSION

There have been investigated 4 routes to obtain concentrated NPK fertilizer, which include followings stages;

-washed dried concentrate is divided into two parts, one part is sent to obtain wet process phosphoric acid (WPA), and the second part is directed to obtain nitrogen-phosphorus pulp, prepared nitric acid pulp and WPA are mixed, ammonized, evaporated and potassium salt and urea are added before granulation (Fig. 1);

-low-grade phosphorite at an incomplete rate and solid-phase mode is decomposed by nitric acid and is repulped with solutions of ammonium and calcium nitrate, thereafter precipitation, brushite and solutions of ammonium and calcium nitrate are separated. The solution of ammonium and calcium nitrate (ACN) is converted to obtain chalk and a solution of ammonium nitrate. After evaporation of the solution, ammonium nitrate melt is obtained, one part of which is sent to obtain lime nitrate and the second part is sent to obtain NPK fertilizer with the addition of brushite and WPA obtained from brushite (Fig. 2);

-washed concentrate is decomposed by the nitric-sulfuric acid method to obtain brushite, when added to which ammonium nitrate (carbamide) and potassium salts are obtained dicalcium-containing nitrogen-phosphate potassium – route 3 (Table 1);

- the washed concentrate is decomposed by the nitric-sulfuric acid method to obtain monocalcium phosphate, when added to which ammonium nitrate (carbamide) and potassium salts are obtained monocalcium phosphate containing nitrogen-phosphate potassium –route 4) (Table 1);

Based on the analysis of these data, a unified generalized combined technological scheme for obtaining concentrated NPK fertilizer was proposed, which provides for the use of two types of phosphate raw materials of the Central Kyzyl Kum: unenriched and washed dried phosphorite and enriched phosphorite according to the method [6].

When processing washed dried phosphorite, the technological scheme consists of the following stages (Fig. 1):

1. Production of WPA.
2. Obtaining a nitric acid suspension.
3. Preparation of nitrophosphate solution.
4. Ammonization.
5. Pulp evaporation.
6. Drying and granulation in the presence of potassium chloride (sulfate) and, if necessary, urea

This technology makes it possible to obtain NPK - fertilizer in wide ranges of varying nutrients, taking into account the demands of the market.

When using raw phosphorite, it is preliminarily enriched by the nitric acid method [15].

The scheme consists of the following stages (Fig. 2):

1. Nitric acid decomposition of raw phosphorite in solid-phase mode.
2. Repulping and decantation with sludge separation.
3. Ammonization and filtration of nitric acid pulp with the recovering a chemical concentrate with a content of 30% P_2O_5 and a calcium module of 1.3.

One part of the chemical concentrate, leached at the third stage, goes to the process of obtaining WPA and the other part to the preparation of NPK fertilizer.

4. Carbonization and filtration of the mother liquor in the presence of ammonia with the removing a solid phase of calcium carbonate and 40-50% ammonium nitrate solution. Further calcium carbonate is mixed with ammonium nitrate melt to obtain lime-ammonium nitrate with a nitrogen content of 21%. The ammonium nitrate solution is evaporated to a concentration of 80-90% and sent to the stage of obtaining NPK-fertilizer.
5. Production of WPA pulp by decomposition of chemical concentrate with sulfuric acid followed by filtration. WPA is separated of phosphogypsum (PG) in a case of the filtration.
6. Preparation of nitrogen-phosphorus suspension by mixing ammoniated WPA, chemical concentrate and evaporated ammonium nitrate.

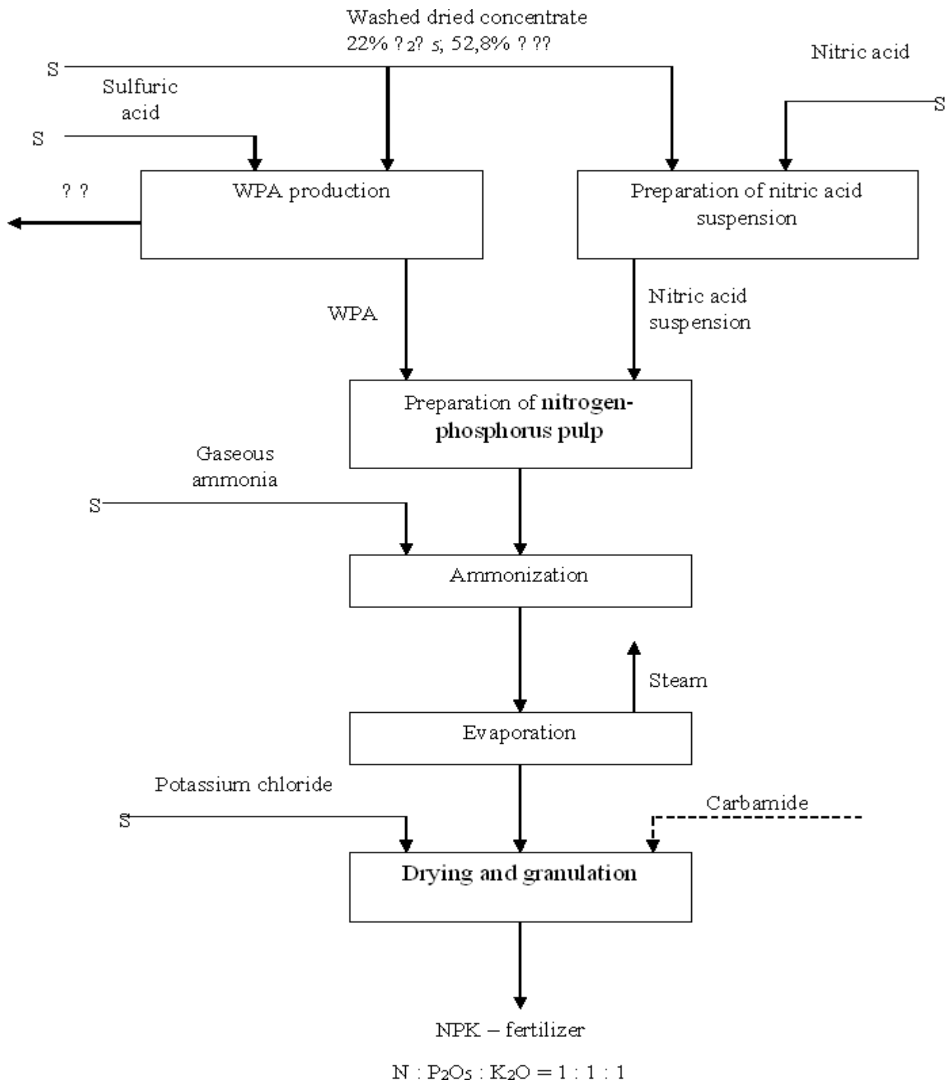

Figure 1. Basic technological scheme for the production of NPK fertilizers. (Route 1)

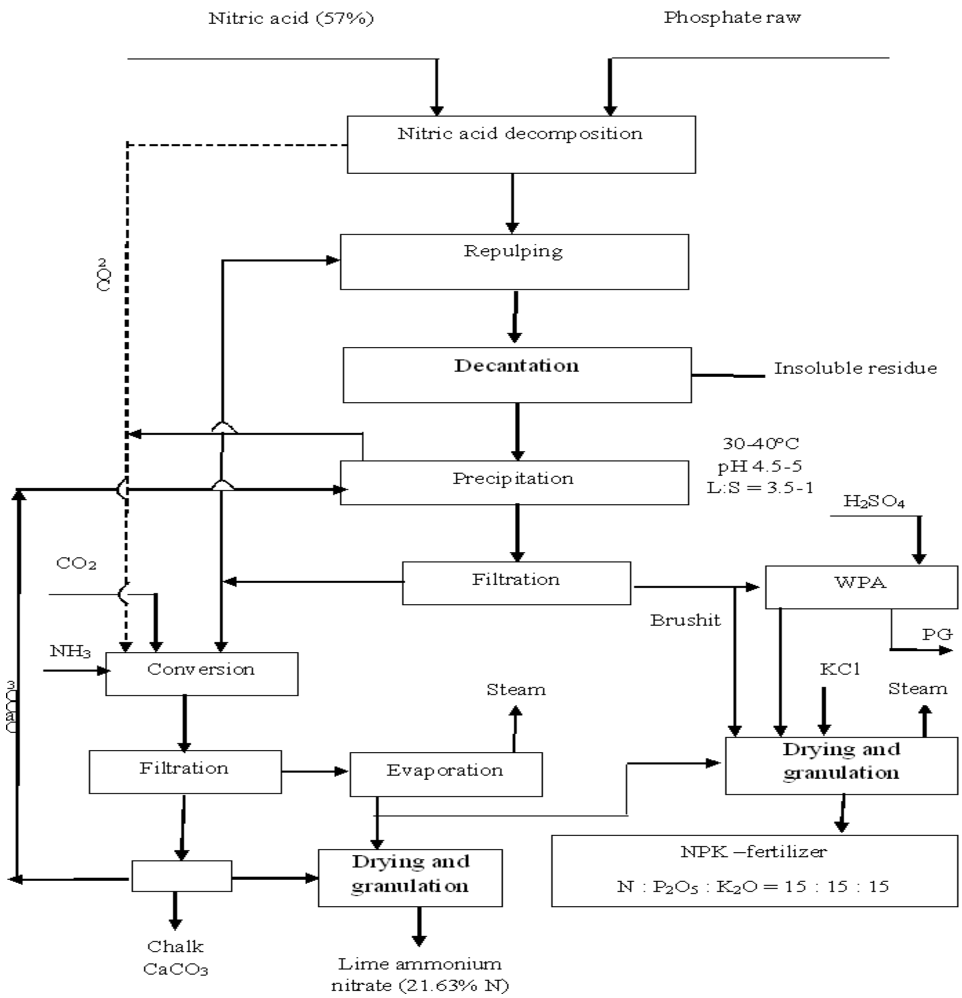

Figure 2. Basic technological scheme for the production of NPK fertilizers. (Route 2)

Table 1. Estimated consumption ratios per 1 ton of mixed fertilizer NPK = 1: 1: 1 (according to the sulfuric acid scheme)

№	Consumption	Washed concentrate	
		Nitric-sulfuric decomposition (route 3) ($CaHPO_4*2H_2O$)	Nitric-sulfuric decomposition (route 4) ($Ca(H_2PO_4)_2$)
1	Phosphates (22%P_2O_5, 52,8% CaO) - taking into account the degree of recovery (0.8)	0.66 0.73	0.68 0.75
2	Non-concentrated nitric acid (100%), t, (HNO_3 norm - 105%) - taking into account 2% losses	0.326 0.348	0.337 0.361
3	Sulfuric acid (100%) t, (H_2SO_4 norm - 105%) - taking into account 2% losses	0.407 0.436	0.527 0.564
4	Potassium chloride KCl (60%), kg - taking into account 2% losses	231.2 236.8	238.2 243.8
5	Ammonia (100%), kg - taking into account 5% losses	88.17 93.96	91.5 96.45

7	Conventional fuel, kg	-	-
8	Electricity, kW / h	-	-
9	Water, m^3	-	-
10	Waste: -Phosphogypsum - Steam condensate, t	0.715 -	0.926 -

7. Drying and granulation the suspension in the presence of potassium chloride to obtain NPK - fertilizer.

A generalized combined scheme for obtaining NPK-fertilizer from phosphorites of the Central Kyzyl Kum is presented in Fig. 3. This scheme can be used for both routes of obtaining NPK-fertilizer.

The advantages of the developed scheme are the followings:

1. Production of a competitive concentrated NPK fertilizer with a content of at least 40% of nutrients and their variation over a wide range.
2. Flexibility of the developed scheme, which makes it possible to obtain, except for NPK fertilizers, various technical phosphorus salts.
3. Large energy costs are not required, because most of the processes take place at normal temperature.
4. Possibility of using any kind of phosphate raw materials.
5. The scheme consists of reliably worked out units: production of WPA (JSC " Ammophos -Maxam"), nitric acid decomposition of low-grade phosphorites (JSC "Samarkandkime"), production of lime (phosphorite) - ammonium nitrate (tested at JSC "Navoiazot"), nitric acid enrichment phosphorite to obtain a chemical concentrate containing 30% P_2O_5 with a calcium module of 1.3, preparation of various NPK fertilizers composition (investigated and tested in the Tashkent chemical-technological Institute (TCTI) laboratory).

Table 2. Estimated calculation of the main technological indicators of NPK-fertilizer production and consumption factors per 1 ton of the amount of nutrient components

No	Name of NPK fertilizers	Nutrient ratio N: P_2O_5: K_2O	Brand fertilizers	The amount of nutritional components, %	Consumption coefficients of the initial components. t per 1 ton of the sum of nutrients				
					Phosphorite of Central Kyzyl Kum	HNO_3	H_2SO_4	KCl	Gaseous ammonia
1	Nitro-ammophos - potassium	1:1:1	16.7:16.7:16.7	50.1	1.51	0.58	1.33	0.55	0.24
2		1:1:1.5	14.6:14.6:21.91	51.1	1.29	0.49	1.19	0.71	0.20
3		1:1.5:1	14.8:22.3:14.8	51.9	2.27	0.49	2.08	0.55	0.26
4		1.5:1:1	20.11:13.4:13.4	46.9	1.29	0.82	1.19	0.47	0.29
5		1: 1.5:1.5	13.2:19.8:19.8	52.8	1.82	1.20	1.68	0.66	0.43
6		1: 0.75:0.5	20.8:15.6:10.4	46.8	1.52	1.99	1.40	0.37	0.63
7	(monocalcium phosphate containing) Nitro-ammophos - potassium	1:1:1	15.6:15.6:15.6	46.8	1.51	0.76	0.92	0.55	0.20
8		1:1:1.5	13.8:13.8:20.7	48.3	1.29	0.65	0.78	0.71	0.17
9		1:1.5:1	13.6:20.4:13.6	47.6	1.94	0.65	1.18	0.47	0.17
10		1.5:1:1	19.0:12.7:12.7	44.4	1.29	0.97	0.78	0.47	0.26
11		1: 1.5:1.5	10.6:15.9:15.9	42.4	1.70	0.57	1.05	0.93	0.15
12		1: 0.75:0.5	19.5:14.6:9.7	43.8	1.51	1.01	0.93	0.37	0.27
13	(dicalcium phosphate containing) Nitro-ammophos - potassium	1:1:1	13.9:13.9:13.9	43.2	1.51	1.32	0.46	0.56	0.20
14		1:1:1.5	12.9:12.9:19.4	45.2	1.29	0.64	0.39	0.70	0.17
15		1:1.5:1	12.3:18.5:12.3	43.1	1.27	0.75	0.69	0.54	0.20
16		1.5:1:1	17.9:11.95:11.95	41.7	1.29	0.97	0.39	0.47	0.26

Table 3. Estimated quantities (t) of raw materials per 1 ton of mixed NPK fertilizer -1: 1: 1 (according to the sulfuric acid scheme) at various CaO / P_2O_5 ratios in concentrates containing 22% P_2O_5

A ratio of CaO / P_2O_5	Preparation of nitric acid suspension	WPA production	Ammonization	Preparation of a potassium-containing solution	Amount of expenses

in phosphate raw	phosphate raw	at 100% rate	phosphate raw	H_2SO_4	PG	NH_3	KCl	phosphate raw	HNO_3
1.34	0.4	0.267	0.280	0.145	0.254	0.09	0.255	0.682	0.337
1.51	0.38	0.259	0.325	0.189	0.332	0.0911	0.255	0.682	0.338
1.87	0.29	0.267	0.394	0.284	0.499	0.0911	0.255	0.682	0.338
2.25	0.24	0.275	0.441	0.383	0.674	0.0911	0.255	0.682	0.338
2.72	0.20	0.265	0.484	0.506	0.891	0.0911	0.255	0.682	0.338

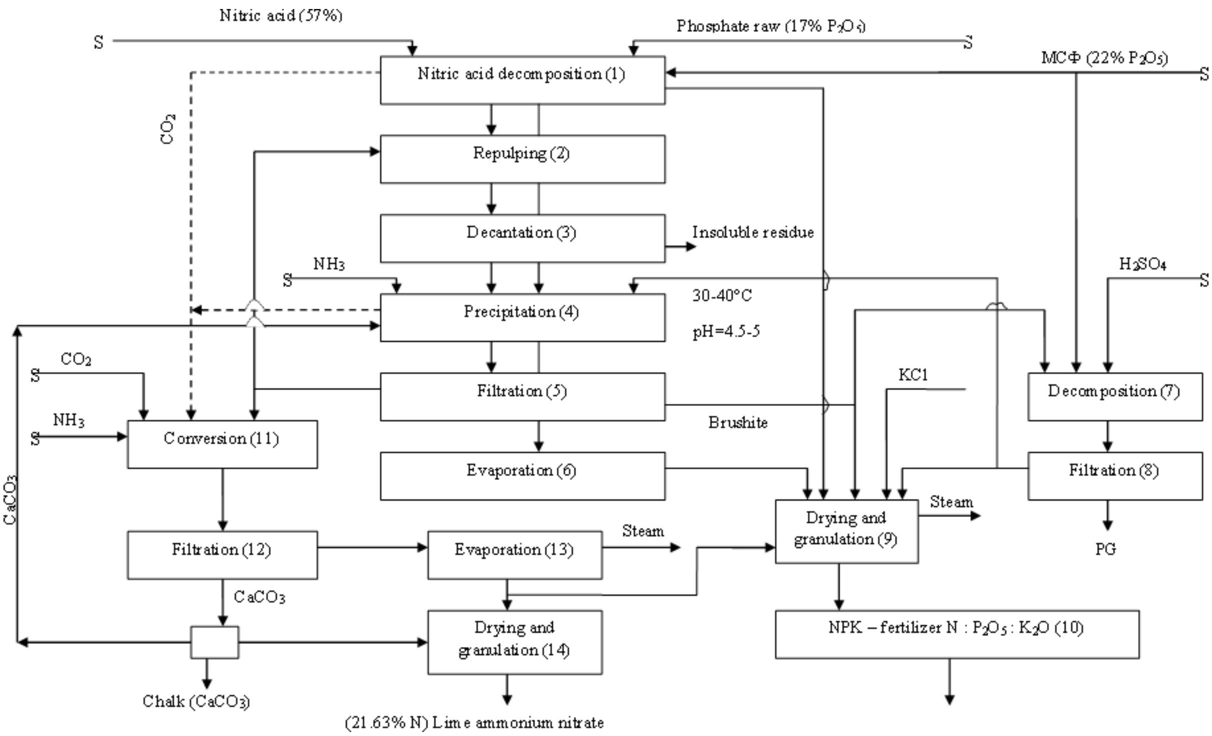

Figure 3. The production capacity of NPK-fertilizer from washed dried phosphorite is 250 thousand tons.

It should be noted that in the developed technology, 0.7-0.9 tons of phosphogypsum is obtained per 1 ton of production - a waste of WPA production, which can be used for the production of phosphogypsum-ammonium nitrate (Tables 2, 3).

Ammonization of nitric and phosphoric acids leads to the formation of ammonium nitrate melt and ammonium phosphate slurry. Granulating the latter in a mixture with potassium chloride and drying the resulting granules gives a nitrogen-ammophos- potassium.

According to TC 6-08-159-70, the sum of nutrients in the 1st grade product is 50%, 2nd grade - 44%. All nutrients in nitrogen -phosphate are in water-soluble form.

The production of nitro-ammophos-potassium consists of the following stages: obtaining a melt of ammonium nitrate and ammonium phosphates, granulation and drying. The smelt of ammonium nitrate is obtained by neutralizing nitric acid with ammonia and followed by evaporating the resulting solution.

In the developed technology, the phosphate raw material for the production of NPK fertilizers is washed phosphorite with a content of 22-24% P_2O_5 and 52.8% CaO, or phosphate raw materials with a content of 17-18% P_2O_5.

The production capacity of NPK-fertilizer from washed dried phosphorite is 250 thousand tons (Fig. 3).

To ensure the competitiveness of products, production should include the following stages:

In case of using phosphate raw materials with a content of 17% P_2O_5:

- nitric acid enrichment of phosphorite with further conversion of calcium nitrate solution with ammonia and carbon dioxide (1, 2, 3, 4 and 5);
- evaporation of ammonium nitrate solution to obtain calcium-ammonium nitrate (11, 12, 13);
- sulfuric acid decomposition of the chemical concentrate with the production of WPA (6, 7, 8);
- mixing WPA with a chemical concentrate, evaporated solution of ammonium nitrate and potassium chloride (5, 8, 13, 9, 10);
- drying and granulation of NPK fertilizers.

In the case of using a washed dried concentrate with a 22% P_2O_5 content:

- nitric acid decomposition of phosphate raw materials (1);
- sulfuric acid decomposition of phosphate raw materials with obtaining WPA (7, 8);

- ammonization of a mixture of WPA and nitric acid suspension (4);
- evaporation of the ammoniated suspension (6);
- drying and granulation of evaporated pulp in the presence of potassium salts (9).

To obtain ammonium phosphates, WPA with a concentration of at least 20% P_2O_5 and gaseous ammonia containing at least 99% NH_3 are used. The acid is ammoniated to pH = 4-4.5, and then in the granulator to pH = 8. In this case, the molar ratio of NH_3 / H_3PO_4 in the final slurry is 0.7.

CONCLUSION

Thus, it was shown the possibility that nitrogen-phosphate-potassium fertilizers and nitro – ammophos - potassium can be prepared based on nitric and sulfuric acid processing low grade phosphorite from Central Kyzyl Kum. The rough calculation of the consumption coefficients of obtaining nitro – ammophos - potassium and nitro-phosphate - potassium (mono- and dicalcium nitro-phosphate - potassium) at various CaO / P_2O_5 ratios in washed dried phosphorite with a content of 26% P_2O_5 was carried out. The triple fertilizers at least 40% nutrient elements are suitable for application in agriculture where their efficiency will have great significance in term of yield. The advantages of the developed scheme are suitable in terms of production of concentrated NPK fertilizer contenting at least 40% of nutrients, flexibility that makes it possible to obtain, except for NPK fertilizers, various technical phosphorus salts, large energy costs, possibility of using any kind of phosphate raw materials.

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