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Nitrogen-phosphate Fertilizers Based on Activation of Phosphorite Powder with Partially Ammoniated Mix of Phosphoric and Sulphuric Acids

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Authors' contributions

This work was carried out in collaboration among all authors. Author AAR designed the study, performed the statistical analysis, wrote the protocol and managed the literature searches. Author SSN wrote the first draft of the manuscript. Author BES managed the analyses of the study and corrected this manuscript. All authors read and approved the final manuscript.

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ABSTRACT

In this article obtaining complex nitrogen-phosphate fertilizers based on activation of phosphorite powder with partially ammoniated mix of phosphoric and sulphuric acids have been studied. In order to reduce the foaming of the process, for the first time a partially ammoniated mix of acids was used. The optimal technological parameters for activation phosphorite powder with partially neutralized mixes of sulphuric and phosphoric acids are the followings: the mix of sulphuric and phosphoric acids with a ratio of SO_3:P_2O_5 = 1.2; pH of acids – 2.5; weight ratio of ammonium sulphate-phosphate slurry towards phosphorite powder is 100 : 20; temperature is 60°C; duration is 30 min. The composition nitrogen-phosphate fertilizers obtained in optimal condition contains (wt., %): N – 11.55; P_2O_{5total} -24.61; $P_2O_{5\text{acceptable by citric acid}}$ -21.66; $P_2O_{5\text{ acceptable by EDTA}}$ -20.24; $P_2O_{5\text{water-soluble}}$ -13.02; CaO_{total} -13.59; $CaO_{\text{acceptable by citric acid}}$ -11.43. In that condition, granulated products of nitrogen-phosphate can be produced with high strength. The advantage of offered promising technology concludes in reduction two times expenditure of the most expensive ammonia in comparison with ammonium sulphate production and an increase in gross domestic product.

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Keywords: Ordinary phosphorite powder; calcium module; mixture of phosphoric and sulphuric acids; nitrogen-phosphate fertilizers.

1. INTRODUCTION

Ordinary phosphorite powder from Central Kyzyl Kum contents (weight.%): 18.70 P_2O_5; 47.83 CaO; 15.3 CO_2; 1.24 Al_2O_3; 1.05 Fe_2O_3; 1.75 MgO; 2.00 F; 0.1 Cl; insoluble residue 6.04 and others 5.9. Calcium module, that is the ratio of CaO : P_2O_5 equal to 2.69 but relative content of the acceptable form of P_2O_5 towards to total form of P_2O_5 is 18.49%. Its dispersed composition is characterized by the following figures:

Class (- 0.5 + 0.315) mm – 1.5%; (- 0.315 + 0.16) mm – 6.3%; (-0.16 + 0.1) mm – 20.3%; (-0,1 + 0.063) mm – 38.3%; (-0.063 + 0.05) mm – 17.2% and classless 0.05 mm – 16.4%. Everything seems to indicate that it has 44.1% of fluorine apatite $Ca_5F(PO_4)_3$ and 34.8 % calcium carbonate $CaCO_3$.

This type of phosphate raw material is unserviceable for nitrogen, sulphuric and hydrochloric acids processing of it into concentrated phosphorous containing fertilizers. Since a large amount of this acid will waste into interaction with calcium carbonate only no reaching to fluorine apatite results large-tonnage wastes products such as calcium nitrate, calcium sulphate and calcium chloride respectively. While acid processing this type of high calcareous containing raw materials conducted plentiful foaming that leads to destroying whole technological process and reduces productive capacity. In case, the activation process of phosphorite powder with partially ammoniated wet-processing phosphoric acid (WPA) is the most available [1-4]. In addition, foaming is minimal phenomenon [5,6]. The composition nitrogen-phosphate fertilizers obtained from phosphorite powder (PP) in optimal condition WPA : PP = 100 : 15 – 35, where pH WPA = 1.2-2.0, is as follow: P_2O_{5total} 33.58-41.36%; $P_2O_{5acceptable\ by\ citric\ acid}$ 28.44 – 40.78%; P_2O_5 $_{acceptable\ by\ EDTA}$ 22.85 – 35.78%; $P_2O_{5water\text{-}soluble}$ 18.31 – 31.76%; N 2.73-5.16.

However, the reaction of phosphates from low-grade phosphorite from Kyzyl Kum with WPA takes place very slowly. At that by addition of some sulphuric acid into phosphoric acid can be intensified this process [7-9]. So, in [9] a basis of the investigation of phosphorite powder activation with WPA in the mixture sulphuric acid in a range of weight ratio of P_2O_5 in acid to P_2O_5 in raw materials from 1 : 0.3 to 1 : 1 (that is in the condition of the low norm of acid) and P_2O_5 in acid towards to H_2SO_4 (monohydrate) from 1 : 0.25 to 1 : 0.5 it was shown that addition of sulphuric acid decreases of total P_2O_5, but increases of acceptable and water-soluble content of P_2O_5 and CaO in products. Furthermore, the addition of sulphuric acid can involve in processing a lot of phosphate raw materials.

In patent [10] it was said that complete decomposition of carbonate compounds subsequently opening phosphate mineral and changing crystal structure undecomposed part of phosphate occurred in presence of large norm of sulphuric acid during the processing phosphate with phosphoric acid. As a result, formed in stage to ammonization of slurry and thermic processing on the drying process, in the product P_2O_5 transferred from an unacceptable form into acceptable one for plant.

Joint stock company (JSC) “Ammofos-Maxam” implemented in the industrial scale of novel nitrogen-phosphate fertilizers production called ammonium sulphate-phosphate by to ammonization of WPA and sulphuric acids [11]. Three brands (A, B, C) are produced depending upon the customer demands. Before that in laboratory condition mixtures’ properties of phosphoric and sulphuric acids were determined in a wide range of SO_3: P_2O_5 from 1.2 to 13.5, as well as products’ composition of their to ammonization [12,13].

In the present study, we have decided to use three mixtures WPA and sulphuric acids with a ratio of SO_3 : P_2O_5 = 1.2; 1.65; 2.6 and checked whether they will be activated or no after processing phosphorite by diverse pH.

2. MATERIALS AND METHODS

The mix of WPA produced by dehydrate method at JSC “Ammofos-Maxam” has the following composition (weight. %): 18.69 P_2O_5; 0.26 CaO; 0.64 MgO; 0.73 Al_2O_3; 0.46 Fe_2O_3; 2.72 SO_3; 1.02 F; 0.093 Cl, and sulphuric acid 95%.

Experiments were carried out on laboratory installation consisting of a tubular glass reactor equipped with a screw-type stirrer moved by engine. The required quantity of acid mix was placed in the reactor and ammoniated by ammonia gas to pH 2.5; 3.5; 4.5. After achievement of pH estimated amount of ordinary phosphorite powder was added into a partially ammoniated mix during intensive stirring (rotational velocity was 250-300 rpm). The temperature of reaction mass constituted was 60°C by contact thermometer. Feeding phosphorite powder realised gradually during 5-7 min. Then reaction mass was stirred in 30 min. Further, the obtained slurry was dried in thermostat at 90-100°C. pH of acid mix and the weight ratio of the ammoniated mix of acids (AMA) to phosphorite powder (PP) from 100 : 10 to 100-80 were as varied parameters. The granulation process of wet phosphate mass was performed during the dry process by intensive stir and balling. Next, dried the samples were subjected to chemical analyses on various content of P_2O_5 and CaO, as well as total N by technique [14]. The acceptable form of P_2O_5 was determined on solubility so in citric acid as in EDTA solution. Whereas acceptable form of CaO – only by citric acid. pH of the product was tested after hour shaking in 10% water solution.

3. RESULTS AND DISCUSSION

The strength of granules was tested on devise MIP-10-1 [15]. Results are summarized in Table 3 and Fig. 1 and 2.

As it is seen from table that activation phos phorite powder, that is transition of unacceptable form of P_2O_5 in it into acceptable when processing it by ammoniated mix of sulphuric and phosphoric acids is taken place. If relative content of acceptable form of P_2O_5 in initial phosphorite powder is 18.49% in the interaction of products with weight ratio of AMA:PP = 100:80 and for SO_3:P_2O_5 = 1.2 at pH = 2,5 it is 66.12%, at pH = 3.5 – 61.5% at pH = 4.5 – 61,72%. While for mix of acids with SO_3:P_2O_5 = 1.65 at pH = 2.5 – 64.35%, at pH = 4.5 – 61.12%. And for SO_3: P_2O_5 = 2.6 at pH = 2.5 – 55.43%, at pH = 3.5 – 54.71%, as well as at pH = 4.5 – 50.24%.

With the increasing amount of phosphorite powder from 10 to 80 and pH of mix from 2.5 to 4.5 in products content of total, acceptable and water-soluble form of P_2O_5 is reduced. In addition, with the increasing ratio of AMA: PP from 100 : 10 to 100: 80 in products content of nitrogen and acceptable form of CaO are decreased. However, with increasing content of sulphuric acid in the mix from SO_3 : P_2O_5 = 1.2 to 2.6 is reduced content of the total and water-soluble form of P_2O_5.

It is known that agrochemists appreciate rather phosphate fertilizers with a high content of the total and acceptable form of P_2O_5 and the relative content of water-soluble is no less than 50%. In our case, such kind of products can be obtained at SO_3:P_2O_5 = 1.2 with pH = 2.5 and 3.5 and the weight ratio of AMA:PP, equal to 100:20, while pH = 4.5 and at AMA:PP = 100:10. When pH = 2.5 composition of product in (weight.%) is as follow: 24.61 P_2O_{5total}; 11.55 N; $P_2O_{5acceptable\ by\ citric\ acid}$: P_2O_{5total} = 88.01; $P_2O_{5acceptable\ by\ EDTA}$: P_2O_{5total} = 82.24; $P_2O_{5water\text{-}soluble}$: P_2O_{5total} = 52.91; $CaO_{acceptable\ by\ citric\ acid}$: CaO_{total} = 84.11%; pH = 4.78.

Since when using the mix of acids with SO_3:P_2O_5 = 1.65 required composition can be obtained at pH of the mix equal to 2.5 and 4.5, but at a weight ratio of AMA:PP = 100 : 10. In case, at pH = 2.5 the product contents (weight.%): 20.94 P_2O_{5total}; 13.48 N; $P_2O_{5acceptable\ by\ citric\ acid}$: P_2O_{5total} = 95.32; $P_2O_{5acceptable\ by\ EDTA}$: P_2O_{5total} = 88.63; $P_2O_{5water\text{-}soluble}$: P_2O_{5total} = 63.80; $CaO_{acceptable\ by\ citric\ acid}$: CaO_{total} = 79.24%; pH = 4.10.

Whereas, when using the mix of acids with SO_3:P_2O_5 = 2.6 required composition of product can be obtained at pH = 2.5 and the weight ratio of AMA:PP = 100 : 10. The product contents (weight.%) : 17.22 $P_2O_{5total.}$; 13.13 N; $P_2O_{5acceptable\ by\ citric\ acid}$: P_2O_{5total} = 93.90; $P_2O_{5acceptable\ by\ EDTA}$: P_2O_{5total} = 87.17; $P_2O_{5water\text{-}soluble}$: P_2O_{5total} = 50.23; $CaO_{acceptable\ by\ citric\ acid}$: CaO_{total} = 97.09%; pH = 5.48.

It should be noted that optimal norm for activation phosphorite powder with partially neutralized mixes of sulphuric and phosphoric acids are as follow: the mix of sulphuric and phosphoric acids with a ratio of SO_3:P_2O_5 = 1.2; pH of acids – 2.5; weight ratio of ammonium sulphate-phosphate slurry towards phosphorite powder is 100 : 20; temperature is 60°C; duration is 30 min. In that condition, granulated products of nitrogen-phosphate can be produced with high strength of 3.2 MPa (Fig. 2). As it is seen from the figure that the strength of granule falls dramatically with changing the weight ratio of AMA:PP from 100 : 10 to 100 : 80. Moreover, strength of granules is reduced with increasing pH of acids from 2.5 to 4.5.

Fig. 3 illustrates below calculated material balance for nitrogen-phosphate fertilizers produc tion based on interaction phosphorite powder from Central Kyzyl Kum with a partially ammoniated mix of phosphoric and sulphuric acids above mentioned optimal conditions.

Table 1. Composition of products obtained by activation of ordinary phosphorite powder from Central Kyzyl Kum with ammonium sulphate-phosphate slurry at the ratio of SO_3/P_2O_5 = 1.2

Weight ratio of AMA:PP	pH 10% water solution of products	Chemical composition of dried products, %							$\frac{P_2O_{5acceptable}}{P_2O_{5total}}x100$ by citric acid, %	$\frac{P_2O_{5acceptable}}{P_2O_{5total}}x100$ by EDTA, %	$\frac{P_2O_{5water\text{-}soluble}}{P_2O_{5total}}x100$	$\frac{CaO_{acceptable}}{CaO_{total}}x100$ by citric acid, %
		N, %	$P_2O_{5\ total}$, %	CaO_{total}, %	P_2O_5 acceptable by citric acid, %	P_2O_5 acceptable by EDTA, %	$P_2O_{5\ water}$ soluble, %	CaO acceptable by citric acid, %				
Ammonium sulphate-phosphate slurry with pH = 2.5												
100:10	4,6	13,2	25,17	8,28	23,45	22,88	17,04	7,92	93,17	90,9	67,7	95,65
100:20	4,78	11,55	24,61	13,59	21,66	20,24	13,02	11,43	88,01	82,24	52,91	84,11
100:30	5,57	9,82	24,22	18,79	20,56	18,75	10,14	14,35	84,89	77,42	41,87	76,37
100:40	5,73	8,99	24,12	22,54	19,05	16,81	7,46	15,65	78,98	69,69	30,93	69,43
100:50	5,85	7,85	23,91	25,07	17,97	15,40	5,50	17,04	75,16	64,41	23,0	67,97
100:80	6,22	6,02	23,38	30,94	15,46	13,62	2,15	19,73	66,12	58,25	9,2	63,77
Ammonium sulphate-phosphate slurry with pH = 3.5												
100:10	4,74	13,42	24,84	7,91	22,73	22,22	16,34	7,48	91,51	89,45	65,78	94,56
100:20	5,32	11,74	24,42	13,48	21,19	19,87	12,5	10,71	86,77	81,37	51,19	79,45
100:30	5,8	10,12	24,2	18,37	19,79	18,12	9,54	13,77	81,78	74,88	39,42	74,96
100:40	5,87	9,1	24,10	22,35	18,54	16,35	7,27	15,08	76,93	67,84	30,17	67,47
100:50	5,92	7,63	23,9	24,96	17,29	14,83	5,36	16,14	72,34	62,05	22,43	64,66
100:80	6,29	5,4	23,22	30,06	14,28	13,17	2,13	17,54	61,5	56,72	9,17	58,35
Ammonium sulphate-phosphate slurry with pH = 4.5												
100:10	4,98	13,48	24,46	7,72	22,03	21,65	15,39	6,89	90,07	88,51	62,92	89,52
100:20	5,23	11,93	23,91	13,25	20,26	19,1	11,78	10,58	84,73	79,88	49,27	79,85
100:30	5,87	10,44	23,76	17,96	18,71	17,9	8,87	13,03	78,75	75,34	37,33	72,55
100:40	5,98	9,32	23,59	21,98	17,83	15,59	7,08	13,96	75,58	66,09	30,01	63,51
100:50	6,03	7,59	23,38	24,64	16,89	14,7	5,01	14,45	72,24	62,87	21,43	58,64
100:80	6,39	5,15	22,96	29,88	14,17	12,59	2,04	17,29	61,72	54,83	8,88	57,86

Table 2. Composition of products obtained by activation of ordinary phosphorite powder from Central Kyzyl Kum with ammonium sulphate-phosphate slurry at the ratio of SO_3/P_2O_5 = 1.65

Weight ratio of AMA:PP	pH 10% water solution of products	Chemical composition of dried products, %							$\frac{P_2O_{5acceptable}}{P_2O_{5total}}$ x100 by citric acid, %	$\frac{P_2O_{5acceptable}}{P_2O_{5total}}$ x100 by EDTA, %	$\frac{P_2O_{5water\text{-}soluble}}{P_2O_{5total}}$ x100	$\frac{CaO_{acceptable}}{CaO_{total}}$ x100 by citric acid, %
		N, %	$P_2O_{5\ total}$, %	CaO_{total}, %	P_2O_5 acceptable by citric acid, %	P_2O_5 acceptable by EDTA, %	$P_2O_{5\ water}$ soluble, %	CaO acceptable by citric acid, %				
Ammonium sulphate-phosphate slurry with pH = 2.5												
100:10	4,10	13,48	20,94	8,67	19,96	18,56	13,36	6,87	95,32	88,63	63,80	79,24
100:20	5,00	11,75	21,07	14,92	18,42	16,78	9,96	11,39	87,42	79,64	47,27	76,34
100:30	5,58	10,04	21,22	19,81	17,71	15,70	6,23	14,55	83,45	73,99	29,36	73,45
100:40	5,79	9,06	21,58	24,90	17,39	14,48	4,38	16,91	80,58	67,1	20,30	67,91
100:50	5,91	7,85	21,70	26,39	16,19	13,86	2,39	17,07	74,61	63,87	11,01	64,68
100:80	6,57	5,85	21,77	32,57	14,01	11,68	0,42	19,97	64,35	53,65	1,93	61,31
Ammonium sulphate-phosphate slurry with pH = 4.5												
100:10	5,15	13,49	20,75	8,61	19,4	17,51	12,68	6,94	93,49	84,38	61,11	80,60
100:20	6,03	12,11	20,88	14,81	18,17	15,92	8,67	11,27	87,02	76,25	41,52	76,1
100:30	6,17	10,85	20,91	19,41	17,41	14,45	5,86	13,82	83,26	69,11	28,02	71,2
100:40	6,19	9,28	21,18	23,04	16,16	13,95	4,57	15,21	76,30	65,86	21,58	66,02
100:50	6,4	7,70	21,3	26,73	15,29	13,05	2,56	16,92	71,78	61,27	12,02	63,3
100:80	6,65	5,56	21,5	32,41	13,14	11,15	0,54	19,99	61,12	51,86	2,51	61,68

Table 3. Composition of products obtained by activation of ordinary phosphorite powder from Central Kyzyl Kum with ammonium sulphate-phosphate slurry at the ratio of SO_3/P_2O_5 = 2.6

Weight ratio of AMA:PP	pH 10% water solution of products	Chemical composition of dried products, %							$\frac{P_2O_{5acceptable}}{P_2O_{5total}}x100$ by citric acid, %	$\frac{P_2O_{5acceptable}}{P_2O_{5total}}x100$ by EDTA, %	$\frac{P_2O_{5water\text{-}soluble}}{P_2O_{5total}}x100$	$\frac{CaO_{acceptable}}{CaO_{total}}x100$ by citric acid, %
		N, %	$P_2O_{5\ total}$, %	CaO_{total}, %	P_2O_5 acceptable by citric acid, %	P_2O_5 acceptable by EDTA, %	P_2O_5 water soluble, %	CaO acceptable by citric acid, %				
Ammonium sulphate-phosphate slurry with pH = 2.5												
100:10	5,48	13,13	17,22	10,32	16,17	15,01	8,65	10,02	93,90	87,17	50,23	97,09
100:20	5,75	11,69	17,93	16,99	15,26	13,92	5,43	16,07	85,11	77,64	30,28	94,58
100:30	6,40	9,35	18,42	21,49	14,5	12,71	3,01	19,13	78,72	69,0	16,34	89,02
100:40	6,52	8,54	18,78	27,41	13,62	11,72	1,42	18,70	72,52	62,41	7,56	68,22
100:50	6,68	7,30	19,22	28,68	13,28	11,16	0,77	18,76	69,09	58,06	4,01	65,41
100:80	6,99	5,60	19,07	33,97	10,57	9,46	0,44	21,69	55,43	49,61	2,31	63,85
Ammonium sulphate-phosphate slurry with pH = 3.5												
100:10	5,49	13,81	16,88	11,01	15,67	14,35	8,22	10,45	92,83	85,01	48,70	94,91
100:20	5,89	11,58	17,65	16,61	14,44	13,05	4,66	14,93	81,81	73,94	26,40	89,89
100:30	6,42	9,91	18,39	22,22	13,93	12,09	2,81	18,79	75,75	65,74	15,28	84,56
100:40	6,48	8,63	18,62	27,15	13,21	11,14	0,91	17,71	70,94	59,83	4,89	65,23
100:50	6,71	7,0	18,82	28,51	12,60	10,48	0,06	18,28	66,95	55,68	0,32	64,12
100:80	6,95	5,52	18,79	32,71	10,28	8,99	следы	21,20	54,71	47,84	-	62,89
Ammonium sulphate-phosphate slurry with pH = 4.5												
100:10	5,60	13,97	16,94	9,95	15,21	14,2	7,93	9,32	89,79	83,83	46,81	93,67
100:20	5,9	12,03	17,42	16,54	13,84	12,25	4,25	14,24	79,45	70,32	24,40	86,09
100:30	6,49	10,42	18,34	21,40	13,44	11,69	2,77	17,09	73,28	63,74	15,10	79,86
100:40	6,5	8,69	18,55	26,95	12,34	10,59	0,87	17,36	66,52	57,09	4,69	64,42
100:50	6,7	6,85	18,89	28,53	11,72	9,91	0,06	18,22	62,04	52,46	0,32	63,86
100:80	6,9	4,35	18,95	33,35	9,52	8,46	следы	20,06	50,24	44,64	-	60,15

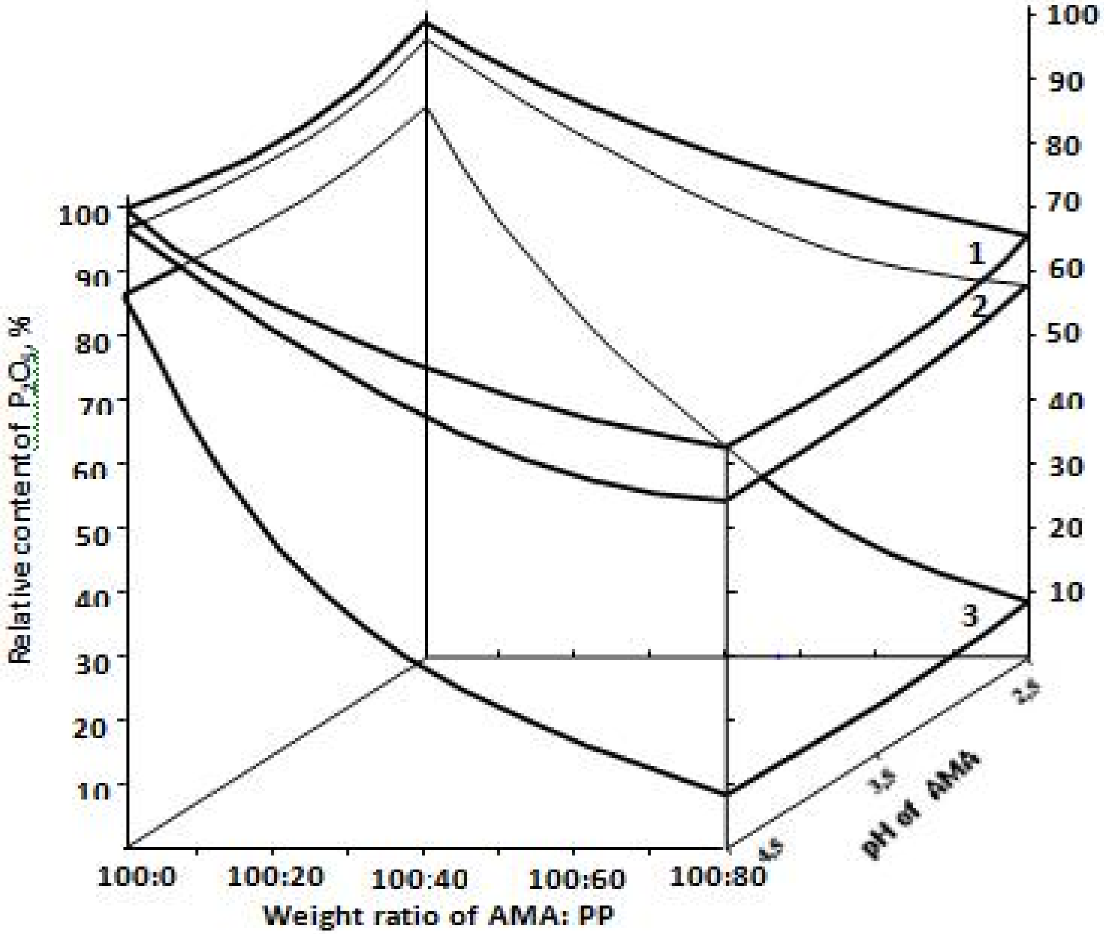

Fig. 1. Dependence of relative content of acceptable by citric acid (1), by EDTA (2) and water-soluble on the weight ratio the mix of acids:phosphorite powder and pH ammonium sulphate-phosphate slurry at SO_3/P_2O_5 = 1,2

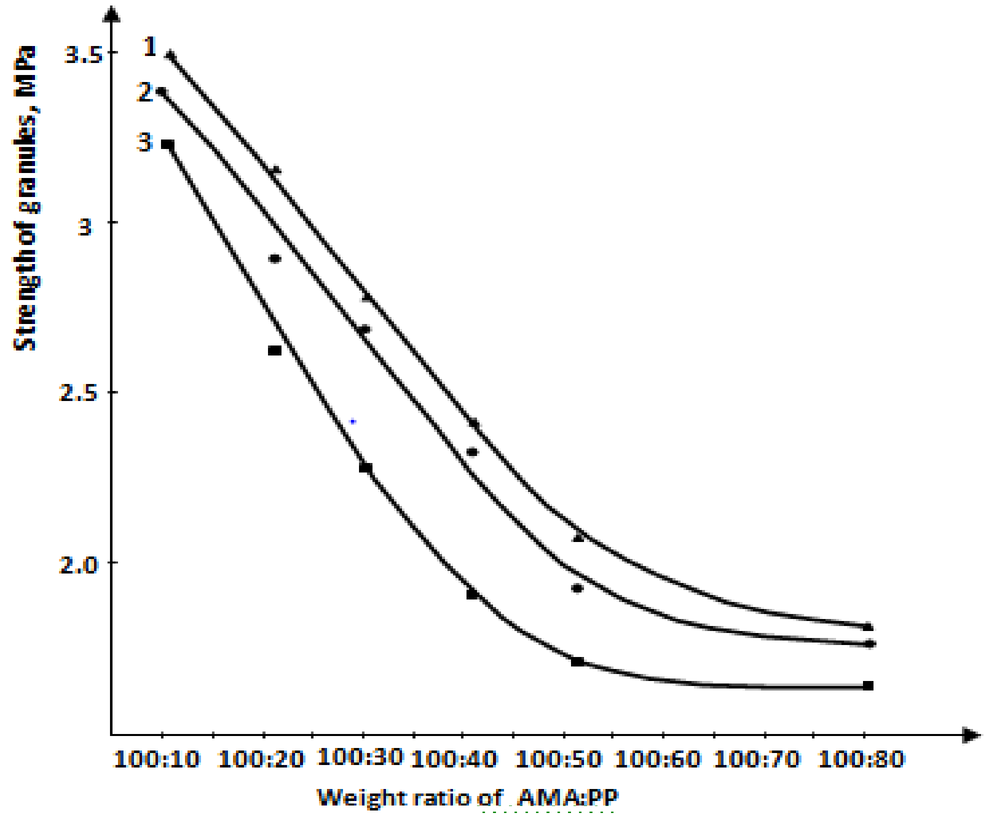

Fig. 2. Strength of product granules depending upon the weight ratio AMA: PP at pH of AMA slurry 1 – 2.5; 2 – 3.5; 3 – 4.5. The ratio of SO_3/P_2O_5 in the AMA – 1.2

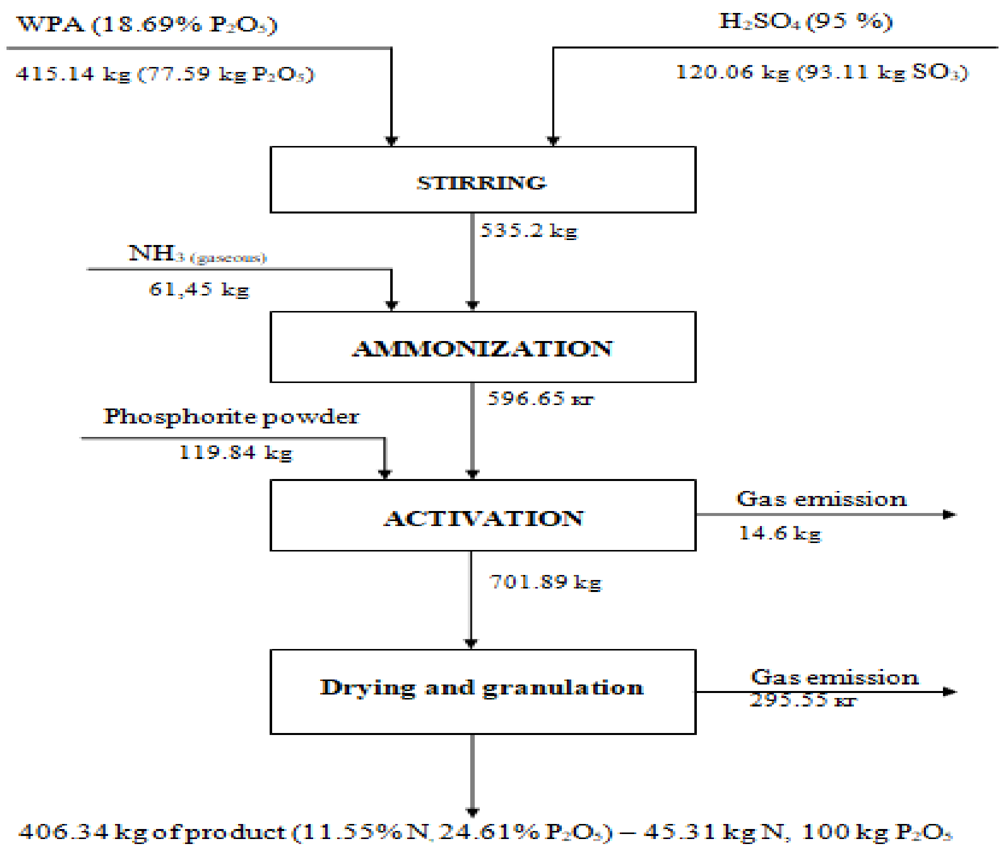

Fig. 3. Material balance of for obtaining nitrogen-phosphate fertilizer based on phosphorite powder from Central Kyzyl Kum with ammonium sulphate-phosphate slurry at SO_3/P_2O_5 = 1.2; AMA: PP = 100: 20 and pH=2.5

4. CONCLUSION

To sum up, the advantage of offered promising technology concludes in reduction two times expenditure of the most expensive ammonia in comparison with ammonium sulphate production and an increase in gross domestic product.

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COMPETING INTERESTS

Authors have declared that no competing interests exist.

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