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Ammonized Superphosphate and Liquid Nitrogen-Phosphorus Fertilizer Based on the Decomposition of Washed Dryed Concentrate with a Mixture of Sulfuric and Phosphoric Acids

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Authors' contributions

This work was carried out in collaboration among all authors. All authors read and approved the final manuscript.

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ABSTRACT

The process of obtaining ammoniated superphosphate and liquid NP-fertilizer by phosphorus-sulphuric acid processing of washed and burned phosphorus concentrate (WBPC) in one cycle were studied. It has been established that at ratios of 100 : 5÷10 : 15 the compositions of

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ammoniated superphosphate samples are characterized as follows (wt., %): $P_2O_{5total(t.)}$ - 20.60-24.85; P_2O_{5free} - absent; $P_2O_{5acceptable\ by\ citric\ acid(ac.c.a.)}$:$P_2O_{5t.}$ = 91.03-100; $P_2O_{5water.solubility(w.s.)}$. : $P_2O_{5t.}$ = 78.87-96.86; N - 4.20-4.79; pH - 2.95-3.17. The product is mainly from calcium sulfate di- and hemihydrate, mono-ammonium phosphate, mono- and dicalcium phosphate, quartz and activated fluorocarbonate apatite. A liquid NP fertilizer consists of 3.22-4.49% N, 16.92-17.75% P_2O_5, 2.18-2.86% CaO, 0.09-0.19% MgO, 0.5 -0.6% SO_3, 0.10-0.11% Fe_2O_3, 0.003-0.007% Al_2O_3, while $P_2O_{5ac.c.a.}$:$P_2O_{5t.}$ = 97.75-100% and $P_2O_{5w.s.}$: $P_2O_{5t.}$ = 96.93-100%. The material balance of obtaining ammoniated superphosphate and liquid NP-fertilizer in one cycle is calculated. It has been shown that the ammonization of phosphorus-sulfuric acid phosphate pulp results in the maximum precipitation of insoluble components in the form of complex salts, which ensures the production of a purified liquid complex fertilizer for drip irrigation.

Keywords: Ammonia; ammoniated superphosphate; liquid NP-fertilizer; material balance; mixture acids.

1. INTRODUCTION

The prospect for the development of agriculture is associated with increasing the yield of agricultural crops through the introduction of the latest soil cultivation technologies, the use of high-yielding varieties of industrial crops, and, of course, the integrated use of mineral fertilizers and plant protection products. One of the main factors determining plant productivity is the use of phosphorus-containing fertilizers.

Currently, the Kyzylum phosphorite complex produces washed burned phosphorus concentrate containing 26% P_2O_5 (WBPC -26) in the amount of 716 thousand tons per year. The production of ammophos from WBPC -26 has been mastered at Ammofos-Maxam JSC. The process is based on the sulfuric acid decomposition of phosphate raw materials in dihydrate method with the separation of phosphogypsum from extraction phosphoric acid (EPA), subsequent processing of the latter into the finished product. The optimal conditions for sulfuric acid extraction of WBPC -26 are: the norm of H_2SO_4 is 103%, the concentration of circulating phosphoric acid is 15% P_2O_5, the process temperature is 85°C and the liquid : solid ratio (L:S) = 3:1, at which indicators acceptable for the technology are achieved: $K_{decom.}$= 96.33%, $K_{extrac.}$= 95.04%; $K_{wash.}$= 97.27%; K_{yield} = 92.44%; the filtration rate is 1361 kg/m^2·hour, and the EPA concentration is 19.44% P_2O_5 [1]. Granulated ammophos obtained on the basis of this EPA contains (wt., %): $P_2O_{5t.}$ – 47.39; $P_2O_{5ac.c.a.}$ – 46.93; $P_2O_{5ac.EDTA}$ – 46.88; $P_2O_{5w.s.}$ – 42,77; $P_2O_{5ac.c.a.}$:$P_2O_{5t.}$ = 99.0; $P_2O_{5w.s.}$:$P_2O_{5t.}$=90.0; SO_3 – 6.02; CaO – 0,76; MgO – 1,27; Fe_2O_3 – 1,02; Al_2O_3 – 1,37; N – 12.05, and the strength of its granules is 5.4 MPa [2]. Due to the complexity of the enrichment technological scheme and high energy costs, the cost of WBPC -26 and ammophos based on it increases significantly. In addition, in the production of ammophos, the internal chemical energy of extraction phosphoric acid is not used. One of the ways to use poor raw materials in the production of concentrated phosphate fertilizers and reduce their cost is the technology for producing ammophosphate. In difference from ammophos, the consumption of sulfuric acid for the production of 1 ton of P_2O_5 in the form of ammophosphate is 10-15% lower, and the degree of use of phosphate raw materials is 1.0-1.5% higher. The process of its production is based on the decomposition of natural phosphates with a high (150-200%) norm of phosphoric acid, followed by neutralization of the remaining acidity with ammonia, granulation and drying of the product [3-5]. Wide agrochemical tests of ammophosphate have shown that it is not inferior in efficiency to ammophos and double superphosphate and can be used in various soil and climatic zones for all agricultural crops [6].

The phosphate component of ammophosphate is monoammonium phosphate, and as impurities dicalcium phosphate and basic calcium phosphates such as hydroxyl fluorapatite and unreacted phosphorites, as well as complex salts that precipitate into the solid phase during ammoniation of excess extraction phosphoric acid (EPA). In this case, we can provide a list of chemical compounds, the formation of which is possible when neutralizing extraction phosphoric acids with ammonia with different compositions and impurity contents [7]. Thus, in the pH range = 2.5-5.5 the following compounds are formed: $NH_4H_2PO_4$; $(NH_4)_2SO_4$; $MgHPO_4$;$MgNH_4PO_4·H_2O$;$Mg(Fe,Al)(NH_4)_2(HPO_4)_2F_3$;$Mg_3(NH_4)_2(HPO_4)_4·8H_2O$; $MgNH_4HFPO_4$; $(Fe,Al)NH_4(HPO_4)_2·0.5H_2O$; $(Fe,Al)NH_4HPO_4F_2$; SiO_2; $CaHPO_4$;

$Ca_5(PO_4)_3OH$. All phosphates, except hydroxyapatite, although insoluble in water, are citrate-soluble. Neutralization of EPA in the presence of ammonium fluoride or silicofluoride can lead to the formation of a number of fluorides of Fe, A1, Ca, Mg. In the absence of ammonium fluorides and silicofluorides, hydroxides are formed. They significantly degrade the quality of the finished product.

The essence of the method we propose is the decomposition of WBPC-26 with a mixture of phosphoric and sulfuric acids; partial ammoniation and separation of pulp into liquid and solid phases; granulation and drying of the solid phase to obtain enriched superphosphate; before ammoniation of the liquid phase with ammonia to obtain purified liquid nitrogen phosphorus fertilizer for drip application or a base solution for liquid NPK-fertilizer. Thus, in one technological cycle, two types of product can be obtained at the same time.

2. METHODS AND MATERIALS

For the experiments were used WBPC containing chemical composition (wt.,%): P_2O_5-25.77; CaO-52.70-; MgO-1.20; Fe_2O_3-0.63; Al_2O_3-1.15; $SO_{3t.}$ -2.67; CO_2-3.60; $CaO:P_2O_5$ = 2.05; insoluble reduce-4,49; extraction phosphoric acid (EPA) produced by Ammofos-Maxam JSC (wt.,%): P_2O_5-15.20; CaO-1.03; MgO-0.15; Fe_2O_3-0.43; Al_2O_3-0.08; $SO_{3t.}$ -1.92 and 93.5% H_2SO_4.

The physico-chemical and physico-mechanical properties of the raw materials have been determined. These properties include: bulk density, angle of repose, fluidity, dispersed composition, hygroscopicity and moisture capacity. These properties of the new phosphate raw material were determined using the methods described in [8, 9].

At an initial humidity of 0.27%, the free bulk density of WBPC-26 is 1.21 g/sm^3, and with compaction it is 1.52 g/sm^3. The smaller the angle of repose, the greater the mobility of the particles of the granular medium. The angle of repose of WBPC-26 is 28 degrees. Determination of fluidity through a funnel with a diameter of 4 mm showed that for WBPC-26 it is equal to 10 points. The hygroscopic point of WBPC-26 was determined by desiccator method. It was 51.5%. Its low value is explained by the presence of a hygroscopic substance (CaO) in the composition of WBPC-26.

The maximum moisture capacity of WBPC-26 is 4.0%, and at higher humidity this raw material loses its friability. In this regard, during storage and transportation it is necessary to protect it from moisture. The results of studying the dispersed composition of WBPC-26 are given in Table. 1. The table shows that the size class (+0.05) – (+0.315) mm accounts for the largest amount (90.5% of the total mass) of concentrate.

The phase composition of the WBPC-26 was performed by X-ray diffraction analysis using an XRD-6100 diffractometer (Shimadzu, manufactured in Japan). CuKα radiation was used (β-filter, Ni, tube current and voltage mode 30 mA, 30 kV) and a constant detector rotation speed of 4 deg/min, and the scanning angle varied from 4 to 80°. Interpretation and identification of mineral phases was carried out using international catalogs [10].

Table 1. Dispersion composition of washed and burned phosphorite concentrate

Size, mm	Fraction yield, mass. %
+ 3,0	0,2
+ 2,0	0,4
+ 1,0	0,6
+ 0,5	2,5
+ 0,315	3,4
+ 0,20	12,2
+ 0,16	13,2
+ 0,10	25,7
+ 0,05	36,0
- 0,05	5,8
Total	100

Fig. 1 shows an X-ray image of WBPC-26. The results of X-ray phase analysis show that its main minerals are fluorocarbonate apatite, calcium oxide and carbonate and quartz.

Diffraction peaks 1.72; 1.74; 1.78; 1.83; 1.92; 2.25; 2.62; 2.70; 2.79; 3.45; 4.04; 8.09A^o belongs to fluorocarbonate apatite. On radiographs, the presence of calcite is established by interplanar distances of 1.52; 1.60; 1.87; 1.91; 2.09; 2.28; 2.52; 3.03; 3.86 A^o. Peaks 2.13; 3.35; 4.23A^o belong to quartz.

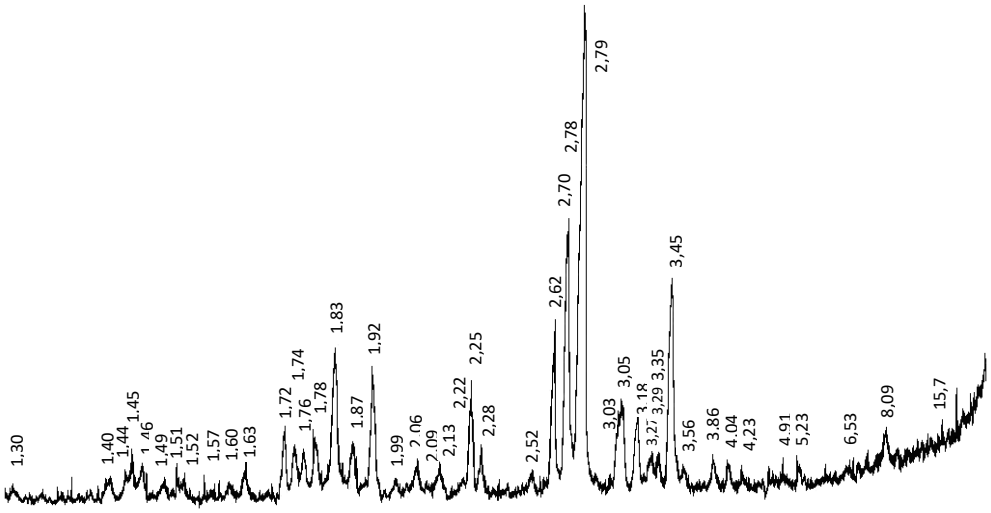

Fig. 1. X-ray image of washed and burned phosphorus concentrate.

2.1 Experiments

The experiments were carried out as follows: a certain amount of a mixture of acids was poured into a thermostated beaker and, with constant stirring with a stirrer, a sample of WBPC-26 was slowly dosed into it. In this case, the mass ratio of EPA : H_2SO_{4mng} :WBPC varied from 100 : 5 : 10 to 100 : 10 : 15. The duration of interaction of the components was 45 minutes at 70°C. Then the acidic phosphoric acid-gypsum pulps were neutralized with a 25% ammonia solution to pH = 3.0. After neutrality they were separated into liquid and solid phases by vacuum filtration. The sediment remaining on the filter was dried together with filter paper in an oven, first at 60°C, then at 90°C to constant weight.

The filtrate, that is, the liquid phase, is processed into liquid complex fertilizer. The products were analyzed for the content of various components using generally accepted methods [11]. Based on the results of chemical analysis of both products, the degree of distribution of components in solid and liquid phases was calculated.

3. RESULTS AND DISCUSSION

The degree of distribution of the components of the pulp partially ammoniated to pH = 3.0 into the liquid and solid phases is given in Table 2.

Thus, with a ratio of EPA: H_2SO_{4mhd}:WBPC = 100 : 5 : 10, the degree of transition of P_2O_5 into the solid phase is 28.14%, CaO - 80.59%, MgO - 75.88%, Fe_2O_3-80.57%, Al_2O_3-93.02%, SO_3-94.13% and N-26.65%. This suggests that the liquid phase is freed from insoluble impurities of di- and trivalent metals and fluorine compounds, and this creates the most favorable conditions for obtaining from it liquid complex fertilizers intended for drip application. At a mass ratio of EPA:WBPC = 100 : 15, with an increase in the mass fraction of H_2SO_{4mhd} from 5 to 10 g, the degree of precipitation of P_2O_5 into the solid phase increases from 36.67 to 38.17%, CaO from 78.19 to 83.03%, MgO from 61 .40 to 80.29%, SO_3 from 94.64 to 96.01% and N from 34.99 to 35.10%.

At the same time, Fe_2O_3 decreases from 85.87 to 85.11% and Al_2O_3 from 99.10 to 98.11%. The transition of di- and trivalent metals into the solid phase is explained by the fact that during ammoniation of phosphoric acid pulp, along with soluble ammonium phosphate, the formation of water-insoluble salts, citrate-soluble forms of dicalcium and dimagnesium phosphates and complex salts of sesquioxides occurs. The sulfuric acid present in EPA during the decomposition of WBPC-26 forms calcium sulfate dihydrate. Judging by the compositions of the liquid phases (Table 2), during ammoniation of phosphoric acid gypsum pulp, $CaSO_4 \cdot 2H_2O$ is partially converted into ammonium sulfate and dicalcium phosphate accordingly to the reaction:

$$NH_4H_2PO_4 + CaSO_4 + NH_3 \longrightarrow (NH_4)_2SO_4 + CaHPO_4$$

The resulting water-soluble $(NH_4)_2SO_4$ will give the liquid nitrogen-phosphorus fertilizer a new quality. The advantage of using $(NH_4)_2SO_4$ in

Table 2. Degree of distribution of ammoniated pulp components into liquid and solid phases

Mass ratio of EPA:H_2SO_{4mhd}: WBPC	Phases	Degree of distribution of components, wt.%						
		P_2O_5	CaO	MgO	Fe_2O_3	Al_2O_3	SO_3	N
100:5:10	Solid phase	28,14	80,59	75.88	80,57	93,02	94,13	26,65
	Liquid phase	71,86	19,41	24.12	19,43	6,98	5,87	73,35
100:5:15	Solid phase	36.67	78.19	61.40	85.87	99.10	94.64	34.99
	Liquid phase	63.33	21.81	38.60	14.13	0.90	5.36	65.01
100:7.5:15	Solid phase	37.02	81.25	68.32	85.54	98.22	95.82	34.81
	Liquid phase	62.98	18.75	31.68	14.46	1.78	4.18	65.19
100:10:15	Solid phase	38.17	83.03	80.29	85.11	98.11	96.01	35.10
	Liquid phase	61.83	16.97	19.71	14.89	1.89	3.99	64.90

Table 3. Composition of samples of ammoniated superphosphate (ASP) and liquid nitrogen-phosphorus fertilizer (LNP)

WPPA:H_2SO_{4mhd}: WPC	**100:5:10**		**100:5:15**		**100:7,5:15**		**100:10:15**	
Products	**SP**	**LP**	**SP**	**LP**	**SP**	**LP**	**SP**	**LP**
pH of 10 % product solution	3,07	-	3,17	-	3,16	-	3,05	-
$P_2O_{5t.}$	22.38	15.98	24.85	17.75	23.48	17.06	20.60	16.92
$P_2O_{5ac.c.a.}$	21.89	15.98	22.62	17.75	22.71	16.77	20.60	16.54
$P_2O_{5ac.EDTA}$	21.75	15.98	22.49	17.75	22.61	16.72	20.58	16.47
$P_2O_{5w.s.}$	20.62	15.95	19.60	17.75	21.40	16.67	19.96	16.40
P_2O_{5free}	отс.	1.03	-	1.51	-	1.44	-	1.14
$CaO_{t.}$	22.72	1.53	24.83	2.86	24.15	2.38	21.00	2.18
$CaO_{ac.c.a.}$	18.67	1.53	17.16	2.86	18.29	2.36	16.77	2.15
$CaO_{w.s.}$	14.21	1.51	11.37	2.86	13.73	2.12	12.39	1.71
MgO	0.9	0.08	0.72	0.19	0.75	0.15	0.75	0.09
Fe_2O_3	1.78	0.12	1.601	0.109	1.493	0.108	1.264	0.112
Al_2O_3	0.81	0.017	0.889	0.003	0.825	0.006	0.701	0.007
$SO_{3t.}$	26.39	0.46	21.54	0.504	26.91	0.502	28.51	0.600
N	3.99	3.07	4.20	3.22	4.54	3.69	4.79	4.49

WPPA:H_2SO_{4mhd}: WPC	100:5:10		100:5:15		100:7,5:15		100:10:15	
Products	**SP**	**LP**	**SP**	**LP**	**SP**	**LP**	**SP**	**LP**
$P_2O_{5ac.c.a.}$ /$P_2O_{5t.}$	97.81	100	91.03	100	96.72	98.3	100	97.75
$P_2O_{5\ ac.EDTA}$ /$P_2O_{5t.}$	97.18	100	90.5	100	96.29	98.01	99.9	97.34
$P_2O_{5w.s.}$ /$P_2O_{5t.}$	92.13	99.81	78.87	100	91.14	97.71	96.89	96.93
$CaO_{ac.c.a.}$/$CaO_{t.}$	82.17	100	69.11	100	75.73	99.16	79.86	98.62
$CaO_{w.s.}$/$CaO_{t.}$	62.54	98.69	45.79	100	56.85	89.07	59.00	78.44

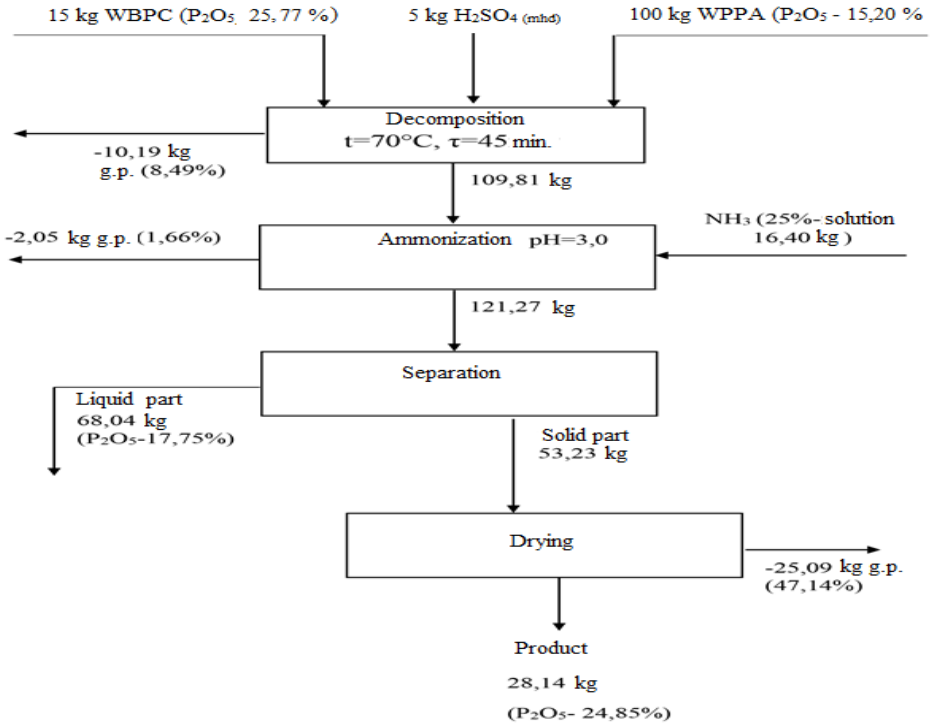

Fig. 2. Material balance of the process of producing ammoniated superphosphate and liquid NP- fertilizer in one cycle

liquid fertilizers is the presence of sulfur in it, which is part of proteins and amino acids during crop formation [12]. Sulfur prevents late blight and root rot. Academician D.N. Pryanishnikov put it in fourth place among the main nutritional elements for plants: nitrogen, phosphorus, potassium, sulfur, calcium, magnesium.

The compositions of dried samples of enriched superphosphate obtained from the solid part of the pulp and liquid fertilizer based on the filtrate is given in Table 3. Accordingly for requirements of State Stand. 5956-78, granulated superphosphate from apatite concentrate without additives must contain at least 19% $P_2O_{5ac.c.a.}$ and no more than 3.2% $P_2O_{5\ free}$. Thus, with the ratio EPA:H_2SO_{4mhd}:WBPC= 100:5:10, ammoniated superphosphate was obtained with the following composition (wt.,%): $P_2O_{5t.}$ – 22,38; P_2O_{5free} – absent; $P_2O_{5ac.c.a.}$: $P_2O_{5t.}$ = 97,81; $P_2O_{5ac.EDTA}$: $P_2O_{5t.}$ =97,18; $P_2O_{5w.s.}$:P_2O_{5t} = 92,13; N – 3,99; The pH of the product is 3.07. At ratios of 100: 5÷10:15, the compositions of the products are characterized as follows (wt.,%): $P_2O_{5t.}$ – 20.60-24.85; P_2O_{5free} - absent; $P_2O_{5ac.c.a.}$: $P_2O_{5t.}$ = 91.03-100; $P_2O_{5ac.EDTA}$: $P_2O_{5t.}$ =90.5-99.9; $P_2O_{5w.s.}$:P_2O_{5t} = 78.87-96.89; N – 4.20-4.79; The pH of the product is 2.95-3.17. With such a lower nitrogen content, these products can be effectively used as a single phosphorus fertilizer for fall plowing.

The phase composition of the products is represented by calcium sulfate, monoammonium phosphate, mono- and dicalcium phosphate, sesquioxide phosphates, quartz and activated fluorocarbonate apatite.

With what characteristics are products obtained from the liquid part of partially ammoniated phosphate pulp?

From Table 3 can be seen that with a ratio of EPA:H_2SO_{4mhd} :WBPC =100:5:10, the composition of the liquid part, which is also cleared of unwanted impurities, contains: 3.07% N, 15.98% P_2O_5, 1.53% CaO, 0. 08% MgO, 0.46% SO_3, 0.12% Fe_2O_3, 0.017% Al_2O_3, all of which are exclusively in water-soluble form. At ratios of 100: 5÷10: 15, the composition of liquid NP fertilizers consists of 3.22-4.49% N, 16.92-17.75% P_2O_5, 2.18-2.86% CaO, 0.09- 0.19% MgO, 0.5-0.6% SO_3, 0.10-0.11% Fe_2O_3, 0.003-0.007% Al_2O_3, while the composition of the suspension is significantly improved : $P_2O_{5ac.c.a.}$: $P_2O_{5t.}$ = 97,75-100% and $P_2O_{5w.s.}$: $P_2O_{5t.}$= 96,93-100%.

The material balance of ammoniated superphosphate and purified liquid NP- fertilizer in one cycle was calculated (Fig. 2).

Thus, using the proposed method it is possible to same time obtain two types of product. Ammoniated superphosphate for use under fall plowing and liquid NP fertilizer for drip irrigation. The suspension can also serve as a base solution for producing water-soluble liquid NPK-fertilizers.

4. CONCLUSION

The degree of distribution of components across phases was calculated, while the maximum precipitation of insoluble components in the form of complex salts was established. Ammoniated superphosphate containing 20.60-24.85% P_2O_5 and 4.0-4.8% N is obtained from the solid part of the pulp. The product consists mainly of di- and hemihydrate calcium sulfate, monoammonium phosphate, mono- and dicalcium phosphate, quartz and activated fluorocarbonate apatite. Purified liquid NP-fertilizer containing 3.1-4.5% N, 16.0-17.8% P_2O_5 was obtained from the liquid part of the pulp. They are recommended for drip application or as a base solution for producing liquid NPK-fertilizers.

COMPETING INTERESTS

Authors have declared that no competing interests exist.

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