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STUDY OF THE PROCESS OF PRODUCING COMPLEX NITROGEN-PHOSPHORUS FERTILIZERS BY PROCESSING LOW-GRADE LOCAL PHOSPHORITES

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On a global scale, the production of highly efficient mineral fertilizers is of great strategic importance for increasing agricultural productivity and maintaining soil fertility. In this regard, improving the technology for producing phosphorus-containing and complex fertilizers based on local raw materials is one of the pressing challenges facing the chemical industry. At the same time, due to the annual depletion of conventional high-grade phosphate resources, increasing attention is being paid to the utilization of relatively low-grade, complex-composition, and low-quality phosphate ores in the production of phosphorus-containing fertilizers [1, 2].

The processing of low-grade phosphate raw materials (~20% P_2O_5) into extraction phosphoric acid (EPA) is economically unviable due to excessive reagent consumption, a high concentration of impurities, and significant operating costs. Consequently, the utilization of such ore remains limited and is primarily confined to the production of ordinary superphosphate. However, conventional superphosphate is characterized by a low nutrient content (9–11% P_2O_5 and 1–2% N), which significantly reduces the economic efficiency of its production and increases the unit costs associated with the logistics, packaging, and storage of the finished product [3].

The challenge of incorporating the ultra-low-grade Shorsu phosphate raw material (Uzbekistan) into industrial production is primarily associated with its extremely low content of the target component (2–8% P_2O_5). The investigated ore sample contains 6.87% P_2O_5 and a significant proportion of magnesium–calcium impurities, which makes its direct utilization impractical. The development of an innovative technological process for processing this material provides a solution to the important challenge of producing concentrated complex fertilizers from locally available substandard raw materials.

In this study, a technological approach based on the decomposition of Shorsu phosphorites using extraction phosphoric acid obtained from washed calcined phosphorite concentrate (WCPC) from the Central Kyzylkum region and pre-activated with ammonium nitrate (NH_4NO_3) was investigated.

The activation processes of extraction phosphoric acid (EPA) in the presence of ammonium nitrate have been studied in detail previously. Activation of EPA (19.50% P_2O_5) was carried out by adding ammonium nitrate at a rate of 1.5% of the acid mass,

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which makes it possible to intensify the decomposition of substandard phosphate ore. The use of activated EPA is aimed at enhancing the decomposition of low-grade phosphate raw materials. Subsequent ammonization and drying of the resulting acidic phosphate slurry enable optimization of the chemical composition of the final product, thereby increasing the nitrogen content while maintaining a high proportion of assimilable phosphate forms.

The study aims to determine the optimal technological conditions for processing ultra-low-grade Shorsu phosphate raw materials using activated phosphoric acid. Particular attention is given to investigating the effects of the ratio of the initial components, the ammonization and drying conditions of the phosphate slurry, on the degree of P_2O_5 assimilability and the quality characteristics of the resulting sulfur-containing complex calcium- and magnesium-containing fertilizer.

As the phosphate raw material, crushed Shorsu phosphate ore (particle size <0.25 mm) with the following composition (wt.%) was used: P_2O_5 = 6.87; CaO = 19.60; MgO = 4.20; CO_2 = 13.63; 1.97 = 1.97; and impurities of Fe, Al, and S. The ore was decomposed using dihydrate extraction phosphoric acid (EPA) containing 19.50% P_2O_5, 0.28% CaO, 0.85% MgO, and 2.67% SO_3, obtained from a Central Kyzylkum phosphate concentrate. Prior to the thermochemical decomposition process, the EPA was preliminarily activated by adding dry industrial-grade ammonium nitrate (NH_4NO_3).

The process for producing a nitrogen–phosphorus complex fertilizer containing the macronutrients calcium and magnesium was carried out under laboratory conditions using a method analogous to the continuous production process for double superphosphate. The experimental procedure included the following stages: acid activation, decomposition of the phosphorite, ammonization of the phosphate suspension, evaporation of the ammonized slurry, followed by drying and granulation through mixing with recycled material (recycle).

The decomposition of Shorsu phosphorite with extraction phosphoric acid activated by ammonium nitrate was carried out at a phosphorite-to-EPA mass ratio (calculated on the basis of 100% P_2O_5) of 100:(80–100), corresponding to 100–120% of the stoichiometric acid requirement for the formation of monocalcium phosphate. The decomposition process was conducted at 80 °C for 90–120 minutes. To enrich the product with nitrogen, the acidic superphosphate suspension obtained after the decomposition of the secondary phosphate with activated EPA was neutralized with gaseous ammonia (NH_3) until a pH of 2.5–3.5 was reached.

The resulting ammonized suspension is evaporated until the residual moisture content reaches 20–25%. A portion of the evaporated slurry is then dried at 100–105 °C for use as recycle material. It is subsequently mixed with the remaining portion of the slurry until the moisture content of the mixture reaches 8–10%. The resulting material is granulated and dried at 100–105 °C. The granules of the final product are screened, and the commercial fraction with the specified particle size (2–4 mm) is collected. The coarse fraction (particle size >4 mm) is crushed and mixed with the powder fraction (particle size <1 mm) generated during screening; this fine fraction is returned to the process as recycle material.

The efficiency of phosphate conversion into plant-available and water-soluble forms was evaluated based on the degree of conversion of phosphates into the

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respective available and water-soluble forms ($K_{av.}$ and K_w.s., respectively), calculated using the following equations:

$$K_{a.v.} = \frac{P_2O_{5a.v.}}{P_2O_{5total.}} \times 100, \qquad K_{w.s.} = \frac{P_2O_{5w.s.}}{P_2O_{5total.}} \times 100$$

where: $P_2O_{5av.}$ and P_2O_5w.s. represent the contents of plant-available P_2O_5 and water-soluble P_2O_5 in the product, respectively; P_2O_5total represents the total P_2O_5 content in the fertilizer.

When activated extraction phosphoric acid (EPA) containing 19.50% P_2O_5 was used at a phosphorite-to-EPA mass ratio of 100:80, the degree of phosphate conversion into the plant-available form ($K_{av.}$) reached 95.90% after decomposition for 1.5–2.0 hours (90–120 minutes).

During neutralization of the suspension obtained at a phosphorite-to-EPA mass ratio (calculated on the basis of 100% P_2O_5) of 100:80 with gaseous ammonia, certain reverse chemical transformations associated with phosphorus retrogradation (conversion of phosphates into non-assimilable forms) were observed. As the medium of the suspension changed with increasing pH, the readily assimilable monocalcium and dicalcium phosphates were partially converted into sparingly soluble tricalcium phosphate, resulting in a decrease in the proportion of assimilable phosphates. During the ammonization of the acidic suspension, the pH increased from 3.2 to 3.5, while the P_2O_5 content increased from 19.26 to 19.98%. As a result of retrogradation, 2.01% of the assimilable phosphates was lost, causing the $K_{av.}$ value to decrease from 95.90 to 93.89%. At the same time, the proportion of water-soluble phosphates, K_w.s., also decreased markedly from 74.97 to 68.92%.

An increase in the EPA application rate has a positive effect on the degree of phosphorite decomposition. When the acid dosage is increased by 1.25 times relative to the stoichiometric amount, i.e., when the phosphorite-to-EPA mass ratio is increased to 100:100, the $K_{av.}$ value in the acidic pulp increases to 97.57%, while the K_w.s. value reaches 84.38%.Upon neutralization of the acidic suspension with gaseous ammonia, with an initial pH of 2.4, until a pH of 3.1 is reached, an increase in the contents of P_2O_5total from 19.81 to 20.86%, $P_2O_{5av.}$ from 19.33 to 20.12%, and P_2O_5w.s. from 16.75 to 17.09% is observed. Despite this increase, the ratio of assimilable phosphates to their total amount ($K_{av.}$) decreases from 97.57 to 96.45%, while the proportion of water-soluble phosphates (K_w.s.) decreases from 84.38 to 81.93%.

The suspensions obtained at the decomposition stage of natural phosphorite with activated extraction phosphoric acid (EPA) contained approximately 52–56% water. Subsequently, the slurry was evaporated to reduce its water content to 20–25%. Upon adding dry fertilizer as recycle material, the moisture content was further reduced to 8–10%, after which the resulting mass was granulated and dried at 100–105 °C.

During the drying stage of the acidic and ammonized slurries obtained by decomposing Shorsu phosphorites with activated extraction phosphoric acid (EPA), the chemical equilibrium within the system undergoes further changes.

During the drying of phosphate slurries, further decomposition of the residual phosphorite contained in the slurry may occur. As a result, depending on the amount of free acid remaining in the slurry, the proportion of plant-available P_2O_5 may increase slightly. When acidic and ammonized slurries obtained at a phosphorite-to-EPA ratio of 100:80 were dried at 100–105 °C, the $K_{av.}$ values of the resulting

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products reached 96.04% and 93.92%, respectively. Compared with the corresponding values for the initial slurries, these increases amounted to only 0.14% and 0.03%, respectively. The free P_2O_5 content in the resulting acidic and ammonized products was 1.76% and 0.41%, respectively.

Despite the fact that, following the ammonization and subsequent drying of the suspension obtained by decomposing natural phosphorite with activated extraction phosphoric acid (EPA) at a phosphorite-to-EPA mass ratio (calculated on the basis of 100% P_2O_5) of 100:80, the degree of phosphate conversion into the plant-available form ($K_{av.}$) reaches 93.92%, this value remains below the established quality requirements. Therefore, the production of ammonized fertilizer under these conditions was considered technologically impractical.

In contrast, when the suspension obtained at the same phosphorite-to-EPA mass ratio (calculated on the basis of 100% P_2O_5) of 100:80 was dried without preliminary ammonization, the $K_{av.}$ value of the resulting product was 96.04%. At the same time, the free P_2O_5 content in the product (1.76%) met the established technological requirements. Therefore, under these conditions, direct drying of the acidic suspension provides a higher proportion of assimilable phosphates compared with the method involving neutralization followed by drying.

Thus, the acidic product obtained by decomposing Shorsu phosphorites with extraction phosphoric acid (EPA) activated by ammonium nitrate at a phosphorite-to-EPA mass ratio (calculated on the basis of 100% P_2O_5) of 100:80, followed by evaporation of the suspension, granulation, and drying, and having the following chemical composition (wt.%): P_2O_5total = 41.40; P_2O_5avail. = 39.76; P_2O_5w.s. = 28.15; free P_2O_5 = 1.76; K_2O = 0.36; CaO = 8.24; MgO = 5.02; N = 0.92; H_2O = 2.21; and other components, meets the established technological requirements. The proportion of assimilable phosphates in the product, calculated as (P_2O_5avail./P_2O_5total) × 100, is 96.04%, while the proportion of water-soluble phosphates, calculated as (P_2O_5w.s./P_2O_5total) × 100, is 68.0%.

Increasing the dosage of activated extraction phosphoric acid (EPA) to 1.25 times the stoichiometric amount [at a phosphorite-to-EPA mass ratio (calculated on the basis of 100% P_2O_5) of 100:100] during the decomposition of natural phosphorite makes it possible to increase the $K_{av.}$ value of the resulting product by 2.48%, reaching 98.52%. However, the free P_2O_5 content in the final product is 8.42%, which exceeds the established limit and, consequently, restricts the applicability of this product.

In this case, i.e., at a phosphorite-to-EPA mass ratio (calculated on the basis of 100% P_2O_5) of 100:100, the most optimal results are achieved by neutralizing the resulting acidic pulp with gaseous ammonia. Following ammonization, the $K_{av.}$ value of the pulp is 96.45%, while during subsequent drying, this value increases slightly to 96.61%. At the same time, the free P_2O_5 content in the final product decreases to 3.57%. The calcium- and magnesium-containing nitrogen–phosphorus fertilizer obtained under these optimal conditions demonstrates a favorable combination of high phosphate assimilability and reduced residual free acidity.

The fertilizer obtained under these conditions has the following chemical composition (wt.%): P_2O_5total = 43.63; P_2O_5avail. = 42.15; P_2O_5w.s. = 34.24; P_2O_5free = 3.57; K_2O = 0.41; CaO = 6.46; MgO = 4.54; N = 2.85; and H_2O = 3.22. The proportion of assimilable phosphates in the product, calculated as

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(P_2O_5avail./P_2O_5total) × 100, reaches 96.61%, while the proportion of water-soluble phosphates, calculated as (P_2O_5w.s./P_2O_5total) × 100, is 78.48%.

The optimal technological parameters for producing a complex calcium- and magnesium-containing nitrogen–phosphorus fertilizer based on the decomposition of ultra-low-grade Shorsu phosphorites with activated phosphoric acid were established as follows: the initial concentration of activated extraction phosphoric acid (EPA) was approximately 19% P_2O_5; the EPA dosage for decomposition of the raw material was 125% of the stoichiometric amount [phosphorite-to-EPA mass ratio (calculated on the basis of 100% P_2O_5) = 100:100]; the temperature and duration of the decomposition process were 80 °C and 90–120 minutes, respectively; the pH for neutralization of the resulting suspension with gaseous ammonia was 2.5–3.0; and the slurry was dried and granulated at 100–105 °C.

Thus, the developed method of chemical processing enables the efficient utilization of substandard Shorsu phosphate raw materials to produce a concentrated multicomponent fertilizer balanced in terms of macronutrients (N, P) and secondary nutrients (Ca, Mg).

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