Water insoluble part of ammophosphate based on decomposition of off-balance ore in neitralized phosphoric acid

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Водонерастворимая часть аммофосфата на основе разложения забалансовой руды нейтрализованной фосфорной кислотой

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Abstract: *the results of laboratory studies for ammophosphate slurry and its water insoluble based on interaction of mineral mass from Central Kyzylkum phosphorite with partially ammoniated wet-processing phosphoric acid depending on the weight ratio acid: phosphate raw (from 100 : 10 to 100 : 30) and pH of acid (from 1,2 to 2,5) has been given in this article. Expansion coefficient of phosphate raw material has been calculated. It has been revealed that lower mass ratio of acid:phosphate raw and pH of acid more all form of phosphorus in the ammophosphate slurry. The chemical composition of ammophosphate slurry and its water insoluble part have been determined 28,46-61,87% P_2O_5. The data of research have confirmed the activation of phosphate raw material during the phosphoris acid decomposition.*

Аннотация: *в статье приводятся результаты лабораторных исследований аммофосфатных пульп и ее водонерастворимой части на основе взаимодействия минерализованной массы Кызылкумских фосфоритов частично аммонизированной экстракционной фосфорной кислотой в зависимости от весового соотношения кислота : фоссырьё (от 100 : 10 до 100 : 30) и pH кислоты (от 1,2 до 2,5). Рассчитан коэффициент разложения фосфатного минерала. Выявлено, что чем меньше массовое соотношение кислота : фоссырьё и значение pH кислоты, тем больше содержание всех форм фосфора в аммофосфатной пульпе. На основе химического анализа определено, что водонерастворимая часть пульпы содержит 28,46-61,87% усвояемого P_2O_5. Данный результат исследований подтверждает активизацию фосфатного сырья при фосфорнокислотном его разложении.*

Keywords: *mineral mass, wet-processing phosphoric acid, ammonation, expansion coefficient, nitrogen-phosphate fertilizers, composition, water insoluble part.*
Ключевые слова: *минерализованная масса, экстракционная фосфорная кислота, аммонизация, коэффициент разложения, азотнофосфорное удобрение, состав, водонерастворимая часть.*

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Ammophosphate technology is one of issue acceptable solution of processing low-grade phosphorite. It is used that any kind of phosphate raw material for technology of ammophosphate. In addition, this fertilizer on composition, physico-chemical property and qualitative index is between ammophos and double superphosphate. As opposed to ammophos consumption of sulfuric acid for 1 ton of P_2O_5 production is than less 10-15% as ammophosphate and efficiency of phosphate raw material is more than 1.0-1.5%. It should be noted the according to ammophosphate technology phosphate raw material is decomposed by high norm of phosphoric acid at 150-300% of stoichiometry on generation of mono basic calcium phosphate ($Ca(H_2PO_4)_2$) subsequent ammonization of resulting acid intermediate by ammonia, then granulation and dry of final product. Therefore, phosphate component of ammophosphate is mono basic ammonium phosphate, dicalcium phosphate, mono basic calcium phosphate is a little, and there is high basic calcium phosphate as hydroxyl fluorine apatite.

Earlier [1] the ammophosphate process generation was carried out by us based on off-balance phosphorite ore from Central Kyzylkum containing (wt, %): 14.33 of $P_2O_{5total.}$; 43.66 of CaO; 14.70 of CO_2; 13.23 of insoluble residue and partially ammoniated wet-processing phosphoric acid (PAWPA), having pH = 1.2-2.5. The temperature of reaction mass supported at 65°C for 45 min. If ammophosphate slurry had pH no more than 3 the slurry was ammonized finally. After the ammonized slurry has been dried at 90-100 °C. As result show that resulting products on composition and properties does not rebate known type of fertilizer. $P_2O_{5total.}$ is in a range of 35.64-46.72%; $P_2O_{5accep.}$ on 2% of citric acid solution is 28.57-45.16%; $P_2O_{5water\text{-}solub.}$ 19.92-41.29%; nitrogen is 2.28-7.63%. It is interesting fact that after processing phosphate raw in partially ammonized WPA, what

changes are occur in the undecomposed part of phosphate mineral? Do whether flour carbonate apatite structure changes occur?, that is deformation of crystal lattice of phosphate mineral during the phosphoric acid decomposition. To discover replies for these questions we have carried out studies to determine the composition of ammophosphate slurry and water insoluble part on content of different form of P_2O_5 and CaO. In the beginning there were defined the composition of ammophosphate slurry and expansion coefficient (Kex) of the phosphate raw (PR) in the slurry. For that there were prepared the ammophosphate slurry maintained above and subjected on nitrogen, various form of phosphorus and calcium according to [2]. For determination of Kexp it was used $P_2O_{5accep.}$, value found on solubility in 0.2 M of Trilon B.

Kexp was calculated according to the following equation:

$$K_{раз.} = \left[1 - \frac{(P_2O_{5общ.} \cdot G_n - 100 \cdot \omega(P_2O_{5ЭФК}) - (P_2O_{5усв.} G_n - 100 \cdot \omega(P_2O_{5ЭФК})}{P_2O_{5ф} \cdot G_ф}\right] \cdot 100, \ \%$$

Where $P_2O_{5total.}$ and $P_2O_{5accep.}$ are total and acceptable form content of P_2O_5 in the slurry, %; G_n and $G_ф$ are weight of slurry and phosphorite applied, g; $\omega(P_2O_{5ЭФК})$ is mass fraction of P_2O_5 in the partially ammonized WPA, $P_2O_{5ф}$ is P_2O_5 content in the phosphorite, g.

Kexp is in the ranges 1.39 to 52.34% depending on the pH of WPA and weight ratio of PAWPA:PR.

As seen from findings that content of nitrogen, total, acceptable and water soluble form of P_2O_5 in the slurry depends from pH value PAWPA and weight ratio of PAWPA : PR. The more pH of PAWPA, the less content of all phosphorus form in the ammophosphate slurry. For example, when PAWPA : PR = 100 : 10 and rise of PAWPA pH from 1.2 to 2.5 leads to reduce $P_2O_{5total.}$ 21.37 to 19.16%; $P_2O_{5accep.}$ on citric acid from 20.92 to 17.92%; $P_2O_{5accep.}$ on Trilon B 20.64 to 17,61%; $P_2O_{5water\text{-}solub.}$ 19.11 to 15.59%; N 3.35 to 3.26%. Depending on the these values relative content of water and acceptable form of P_2O_5 on citric acid and Trilon B varied in the ranges of 81.37-89.42 and 93.53-96.58 and 91.91-96.63%, respectively. When all values of PAWPA pH increasing mass fraction of phosphate raw 100 : 10 to 100 : 30 decreases considerably of slurry humidity, nitrogen, acceptable and water soluble form of P_2O_5, but it is increased that all form of CaO. The water soluble form of P_2O_5 and CaO say about presence of mono basic ammonium phosphate and mono basic calcium phosphate in the fertilizer.

In order to discover the undecomposed part of ammophosphate slurry it was conducted that filtration process of the slurry in liquid and solid phases under vacuum indicated 550-600 mercury column. Further wet cake was washed by hot water to neutral reaction by indicator paper. Washed cake was dried at 105°C. As dried cake was weighed and analyzed for various form of phosphorus, nitrogen, calcium and CO_2.

The decarbonization degree (according to CO_2) depending on the pH of WPA and ratio of PAWPA: PR varied in the range of 54.23-91.75%. Moreover, the most interesting index defining water insoluble part of the slurry is relative acceptable form of P_2O_5. If starting mineralized mass contents 9.0% of P_2O_5 on citric acid 16.54% of P_2O_5 on Trilon B, respectively the water insoluble part of the slurry contents these values in the ranges 28.46-61.87% and 23.43-60.65%, respectively for citric acid and Trilon B. This phenomenon can be explained that water insoluble part of the phosphate raw is activated after treating, that is P_2O_5 in it transferred from unacceptable form into acceptable for plant form.

There is nitrogen in the solid phase can be explained presence complex salt of ferrum and aluminum in form of $NH_4(Fe,Al)_3H_8(PO_4)_6 \cdot 6H_2O$, $NH_4(Fe,Al)_3H_{14}(PO_4)_8 \cdot 4H_2O$ and $(NH_4)_2(Fe,Al)Mg(HPO_4)_2F_3$ that dependences from pH value under ammonization process [3-5]**.** It should be noted although these salts are water insoluble but they are soluble slightly in 2 % citric acid and 0.2 M solution of Trilon B.

Thus, these cakes can be used as fertilizer on soil is unsaturated by bases where has condition for step by step dissolution of undecomposed of fertilizer part.

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