OTIMIZAÇÃO DE UMA TECNOLOGIA DE PRODUÇÃO DE NP-FERTILIZANTES

OPTIMIZATION OF AN NP-FERTILIZER PRODUCTION TECHNOLOGY

ОПТИМИЗАЦИЯ ТЕХНОЛОГИИ ПОЛУЧЕНИЯ NP – УДОБРЕНИЯ

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RESUMO

O nitrato de amônio é o fertilizante de nitrogênio mais comum e eficaz no mundo e sua demanda diminuiu significativamente devido ao risco de incêndio e explosão. Para a indústria agrícola, os fertilizantes compostos são os mais preferíveis. Portanto, o desenvolvimento da tecnologia otimizada é uma tarefa urgente. Este estudo teve como objetivo desenvolver a tecnologia de fertilizantes NP com base na mistura da solução do primeiro estágio da evaporação da produção atual de nitrato de amônio e rocha fosfática moída para obter o produto alvo com propriedades aprimoradas para o consumidor e agroquímicas. Foram estabelecidos parâmetros operacionais ótimos da tecnologia para consumo específico de nitrato de amônio e rocha fosfática moída. Os resultados mostraram a regulação da razão dos elementos nutrientes N / P₂O₅ em formulações de fertilizantes como 23/5%; 21/6%; 19/7%; 13/10% e 11/11%. O método de modelagem de segunda ordem de planejamento rotativo Box-Hunter foi usado para obter os dados. A influência das variáveis independentes - o consumo específico de nitrato de amônio e rocha fosfática moída sobre o teor de nutrientes nos produtos-alvo - foi determinado. Foi estabelecido que sua mudança é acompanhada de dois processos opostos, ou seja, aumenta por um aumento no consumo específico de nitrato de amônio com uma diminuição simultânea no consumo específico de rocha fosfática moída e vice-versa; e diminui com uma diminuição no teor específico consumo de nitrato de amônio com um aumento simultâneo no consumo específico de rocha fosfática moída na mistura inicial. Uma equação de regressão adequada foi encontrada para a influência das variáveis independentes na proporção de nutrientes nos produtos. Foi demonstrado que, com as alterações correspondentes na mistura inicial de 0,36 t para 0,67t, e 0,54 t para 0,29 t nos produtos, as relações N / P₂O₅ aumentaram de 2,51 para 4,68. Além disso, é possível obter um conjunto completo de fertilizantes NP com propriedades agroquímicas e de consumo aprimoradas com um teor total de NP de 25% a 28% em sua composição.

Palavras-chave: *nitrato de amônio, rocha fosfática moída, nutrientes, nitrogênio, pentóxido de fósforo.*

ABSTRACT

Ammonium nitrate is the most common and useful nitrogen fertilizer globally, and its demand has decreased significantly due to its fire and explosion hazard. For the agricultural industry, compound fertilizers are most preferable. So, optimized technology's development is an urgent task. This study aimed to develop NP-fertilizers technology based on the solution mixture of the evaporation's first stage of the current production of ammonium nitrate and ground phosphate rock to obtain the target product with improved consumer and agrochemical properties. It was established optimal operating parameters of the technology for specific consumption of ammonium nitrate and ground phosphate rock. The results showed the ratio regulation of nutrient elements N/P₂O₅ in fertilizer formulations as 23/5%; 21/6%; 19/7%; 13/10%, and 11/11%. The method of rotatable planning-Box-Hunter second-order modeling was used to achieve the data. The independent variables' influence – specific consumption of ammonium nitrate and ground phosphate rock on the nutrients' content in the target products were determined. It has been established that two opposite processes accompany their change, that is, it increases by an increase in the specific consumption of ammonium nitrate with a simultaneous decrease in the specific consumption of ground phosphate rock, and vice versa; and it decreases with a decrease in the specific consumption of ammonium nitrate with a simultaneous increase in the particular consumption of ground phosphate rock in the initial mixture. An adequate regression equation was found for the independent variables' influence on the nutrients' ratios in products. It was shown that, with corresponding changes in the initial mixture

from 0.36 t to 0.67t and 0.54t to 0.29t in the products, the N/ P₂O₅ ratios increased from 2.51 to 4.68. Moreover, it is possible to obtain a whole set of NP-fertilizers with improved agrochemical and consumer properties, with a total NP content of 25% to 28% in their composition.

Keywords: ammonium nitrate, ground phosphate rock, nutrients, nitrogen, phosphorus pentoxide

АННОТАЦИЯ

В настоящее время, к аммиачной селитре спрос существенно снизился из – за ее огне и взрывоопасности, для сельскохозяйственной отрасли наиболее предпочтительными считаются сложные удобрения. Поэтому, разработка оптимизированной технологии получения таких удобрительных композиций представляет собой актуальную задачу. Целевые задачи исследований заключались: в разработке технологии NP – удобрений на основе смеси раствора первой ступени выпарки действующего производства аммиачной селитры и фосфоритной муки с получением целевого продукта с улучшенными потребительскими и агрохимическими свойствами, в установлении оптимальных режимных показателей технологии по удельным расходам аммиачной селитры и фосфоритной муки. Приведены результаты по регулированию соотношений питательных элементов N/P₂O₅ в удобрениях составов в % 23/5; 21/6; 19/7; 13/10; 11/11. Результаты лабораторно-опытно-производственных исследований обобщены с использованием метода рототабельного планирования - моделирования второго порядка Бокса-Хантера. Определено влияние независимых переменных - удельных расходов аммиачной селитры и фосфоритной муки на содержания питательных элементов в целевых продуктах. Установлено, что их изменение сопровождается двумя противоположными процессами: возрастает с увеличением удельного расхода аммиачной селитры при одновременном уменьшении удельного расхода фосфоритной муки, и наоборот, снижается с уменьшением удельного расхода аммиачной селитры при одновременном увеличении удельного расхода фосфоритной муки в исходной смеси. Найдено адекватное уравнение регрессии влияния независимых переменных на отношения питательных элементов в продуктах. Показано, что при соответствующих изменениях последних в исходной смеси от 0,36т до 0,67т и 0,54т до 0,29т в продуктах отношения N/P₂O₅ возрастают от 2,51 до 4,68. При этом возможно получение целого набора NP - удобрений с улучшенными агрохимическими и потребительскими свойствами, с суммарным содержанием NP в их составе от 25% до 28%.

Ключевые слова: аммиачная селитра, фосфоритная мука, питательные вещества, азот, пятиокись фосфора

1. INTRODUCTION:

Ammonium nitrate is the most common and effective nitrogen fertilizer globally (Chernyshev A.K. *et al.*, 2009; V. M. Olevskiy, 1978; Besterekov U. *et al.*, 2019; Besterekov U. *et al.*, 2018). It is used in agriculture for all types of crops and on any kind of soil.

However, in recent years, both consumers and producers of ammonium nitrate have encountered some difficulties and limitations due to its severe consumer shortcoming - fire and explosion hazard [J. Oxley *et al.*, 2002; N. Dechy *et al.*, 2004; Lavrov V.V. and Shvedov K.K., 2004; Taran A.L. *et al.*, 1991; Usmonov K.P. 2011]. Therefore, to obtain high and high-quality yields, it is necessary to apply balanced fertilizers, so-called complex fertilizers characterized by the optimal ratio of nitrogen, phosphorus, and other fertilizing elements, and have a high agrochemical value (K. Gorbovskiy *et al.*, 2017; Ilin V.A., 2006; Pavlova G.S., 2007; Madenov B.D. *et al.*, 2012; Ghiga R. *et al.*, 2008; Kurbaniyazov R.K. *et al.*, 2007; Levin B.V. and Sokolov A.N., 2004; Sagdullayev U.Kh., *et al.* 2007; Belova N.P. and

Ryabtseval. Yu. 2007; Dmitriyeva O.A. and Ovchinnikov V.M., 2007; Artemenko V.G. and Gunin V.V., 2010; Kurbaniyazov R.K., 2011; Abramov O.B. *et al.*, 2010; Rakcheyeva L.V. *et al.*, 2010; Sharipov T.V. *et al.*, 2011; Obrestad T. and Oksvik A., 2012; Ilyin V.A. *et al.*, 2014; Reimov A.M., 2012; Pak D.G. *et al.*, 2017; Alimov U.K. *et al.*, 2016; Madenov B.D. *et al.*, 2015; Madenov B.D. *et al.*, 2015).

Because of this, in recent years, there has been a great demand for fertilizers containing nitrogen and phosphorus. Therefore, numerous studies are being conducted on the addition of phosphorus-containing components into the composition of ammonium nitrate melt, which are natural minerals, as well as products or the technogenic phosphorus-containing wastes produced based on their industrial processing (Botirov B.B. and Beglov B.M., 2008; Usmonov K.P., 2013; Taran Yu.A. and Taran A.V., 2016; Namazov Sh.S., 2008; Kurbaniyazov R.K., 2008; Vorobeveva T.A. *et al.*, 2013; A.M. Reymov *et al.*, 2013; Bolysbek A.A. *et al.*, 2018; Madenov B.D. *et al.*, 2015; Madenov B.D. *et al.*, 2012; Taran A.L. *et al.*, 2016; Gorbovskiy K.G. *et al.*, 2019). The

purpose of this work was to investigate the possibility of obtaining NP-fertilizers of improved agrochemical properties with an adjustable nutrient ratio N/P_2O_5 based on a solution of ammonium nitrate and ground phosphate rock.

In the Republic of Kazakhstan, the only producer of ammonium nitrate with fertilizing purpose is "KazAzot" JSC (Aktau, Republic of Kazakhstan). Concerning solving those mentioned above especially urgent tasks for this production to improve agrochemical value and increase consumer qualities of ammonium nitrate, own raw material reserves of phosphate ores of the Republic of Kazakhstan can be successfully used (Directory of Kazakhstan deposits). Following the requirements specification of "KazAzot" JSC (Aktau, Kazakhstan) for establishing new opportunities for improving the agrochemical and consumer properties of ammonium nitrate the scientific team of the Department "Chemical Technology of Inorganic Substances" of M. Auezov South Kazakhstan State University (Shymkent, Kazakhstan) carried out a research. The research purpose was to obtain new NP-fertilizers considering a comprehensive analysis of their composition and properties. The initial components were the ammonium nitrate solution produced by "KazAzot" JSC according to GOST-2-2013 (This GOST 2-2013 applies to ammonium nitrate intended for agriculture, industry and export.), and Chilisay ground phosphorite rock (GPR-2 grade TS 930640000252-01-2011) containing 17% of P_2O_5 ("Temir-Service" LLP, Aktyubinsk, Kazakhstan). The initial solution contained 64-71% of ammonium nitrate and was obtained according to the traditional ammonium nitrate production technology on the first evaporation stage. The customer also agreed on the limiting contents of nitrogen and phosphorus pentoxide in the final target products, respectively, in the range of $11 \pm 23\%$ and $5 \pm 11\%$.

This study aimed to investigate the possibility of obtaining NP-fertilizers of improved agrochemical properties with an adjustable nutrient ratio N/P_2O_5 based on a solution of ammonium nitrate and ground phosphate rock.

2. MATERIALS AND METHODS:

The research was conducted in the laboratories of M. Auezov SKSU. Their results were tested in the trial plot of "KazAzot" JSC, which is currently producing the ammonium nitrate. Ammonium nitrate solution with a concentration of 64-71% of the known volume and temperature of $110-130^\circ C$ was mixed with the

calculated mass of ground phosphate rock. A modifying mineral additive was also added to the mixture in small quantities. The obtained suspension mixture was thoroughly mixed and fed to sprayers at the temperature of $120-130^\circ C$, and from there, it was sprayed into a granulator-drum, where it was dried by a drying agent in a direct-flow mode and granulated in compliance with the traditional technological operating parameters of the current production of ammonium nitrate (V. M. Olevskiy, 1978; Regulations of "KazAzot", 2012).

The analysis of composition and properties of the initial mixture and obtained samples NP-fertilizers were conducted according to the methods given in the normative documentation for fertilizer:

- the total nitrogen content in NP-fertilizer – according to GOST 30181.6-94 (Determination of total nitrogen in saltpeter and the resulting products was carried out by the titrimetric method GOST 30181.4-94. This method is based on the reduction of nitrate nitrogen to ammonium nitrogen by Devard's alloy in the presence of sodium hydroxide, followed by distillation of ammonia from the alkaline solution into a sulfuric acid solution and back titration of the excess acid with sodium hydroxide solution in the presence of a mixed indicator. The technique is designed to measure the amount of nitrogen in the range from 8% to 35%.);
- the P_2O_5 content of NP-fertilizer – according to GOST 20851.2-75 (The determination of the total P_2O_5 in the obtained products was carried out by the photocolometric method according to the GOST 20851.2-75. The technique is designed to measure the amount of P_2O_5 in the range from 3% to 50%. The method is based on a photometric change in phosphates with vanadium and molybdenum salts, which form compounds colored yellow. The optical density of the resulting compound is measured on a photocolometer.);
- the moisture content of the product – according to GOST 20851.4-75 (Determination of total moisture in ammonium nitrate, ground phosphate rock, and the resulting products was carried out by the drying method GOST 20851.4-75. This method is based on drying at a temperature of $105 - 110^\circ C$ to constant weight and subsequent weighing. The moisture content is calculated from the difference in sample weight before and after drying. The technique is intended for measuring hygroscopic and total moisture in terms of H_2O from 0.1% to 12% by drying in a drying oven.), using hydrometer Mettler Toledo;
- the strength of NP-fertilizer's granules – on the

device IPG-1M;

- pH of 10% solution of NP-fertilizer – on the device I-160 MI.

3. RESULTS AND DISCUSSION:

The results of experimental studies conducted under the laboratory-test-industrial conditions are presented in tables 1 and 2. Specific consumptions of the ammonium nitrate and ground phosphate rock were calculated in terms of 1 ton of the target product. At the initial stage of the research, the influence of the specific consumption of ammonium nitrate and ground phosphate rock and the nutrients contained in them – N and P_2O_5 on their ratio in the target products were studied. The results of these studies are shown in Figures 1 and 2.

As follows from the results obtained, the increase in the ratio of nutrient elements N/ P_2O_5 in the target products is accompanied by two different processes: the increase in the specific consumption of ammonium nitrate with the simultaneous decrease in the specific consumption of ground phosphate rock in them. In this regard, further studies were carried out by the method of rotatable design of second-order experiment (Box-Hunter method) (Akhnazarova S.A. and Kafarov B.V., 1985). As independent variables, the specific consumption of ammonium nitrate and ground phosphate rock was used. The optimization parameter was the ratio N/ P_2O_5 in the product. Table 3 shows the information on the variation areas of the independent variables, compiled based on the data from Table 1. Table 4 shows the matrix of experimental design and its results.

Based on the data of table 4 and (Inkov A.M. *et al.*, 2000) we obtained the regression equation of influence of ammonium nitrate and ground phosphate rock consumption in the initial mixture on the N/ P_2O_5 ratio both in coded and natural forms:

$$N/P_2O_5_{\text{cod}} = 1,758Z_1^2 - 9,951Z_1Z_2 + 7,615Z_1 + 10,688Z_2^2 - 10,227Z_2 + 2,683 \quad (1)$$

$$N/P_2O_5_{\text{nat}} = 2,683 + 7,615AN - 10,227GPR + 1,758AN^2 + 10,688GPR^2 - 9,951AN \cdot GPR \quad (2)$$

Assessment of the significance of the regression equations (1-2) was determined using the Student's test, but the adequacy of the equation by the Fisher criterion. Table 5 shows the experimental and calculated according to the equation (2) values of the nutrients' ratios in the samples of the obtained target NP-fertilizers were

compared.

The table value of Fisher's criterion is 6.59, and the calculated is 0.188 taking into account 5% of the experiment's errors. In this regard, $F_{\text{tabl}} > F_{\text{calc}}$ regression equation obtained is adequate. Based on equation (2) using the MathCAD program (Ochkov V.F., 2007) a volumetric image of response surface $N/P_2O_5_{\text{nat}} = f(AN, GPR)$ and its horizontal sections were constructed.

Figure 3 shows that by increasing the consumption of ammonium nitrate and the decrease in ground phosphate rock consumption, the ratio of nutrients in the target product increases. N/ P_2O_5 in the target product reaches its maximum level (4.68) with a consumption of ammonium nitrate of 0.67t and ground phosphate rock of 0.29t. Table 6 shows the values of technological parameters in the abc areas, in which the N/ P_2O_5 ratios vary from 2.5 to 4.68. From Figure 3 and Table 6 it follows that the optimal consumption parameters of ammonium nitrate and ground phosphate rock for the N/ P_2O_5 ratio in the target NP-fertilizer from 0.25 to 4.68 are located along with the lines ac (for AN) and cb (for GPR), i.e. AN can vary from 0.36t to 0.67t and GPR from 0.29t to 0.54t.

4. CONCLUSIONS:

Based on the results obtained by modeling the process of the influence of specific consumption of ammonium nitrate and ground phosphate rock on the ratios of nutrients (N/ P_2O_5) in target NP-products, the following conclusions can be made:

1. the specific consumption of ammonium nitrate and ground phosphate rock on the ratio of nutrients in the target product has the opposite influence: the increase in the particular use of ammonium nitrate and the corresponding decrease in the specific consumption of ground phosphate rock results the growth of the N/ P_2O_5 ratio in the target product, and vice versa, the reduction in the particular consumption of ammonium nitrate and the corresponding increase in the specific use of ground phosphate rock leads to the decrease of the N/ P_2O_5 ratio in the target product;
2. by the rise in the consumption of ammonium nitrate in the system under consideration from 0.36 t to 0.67 t with the corresponding decrease in the specific consumption of phosphate rock, the ratio of nutrients N/ P_2O_5 in the target NP-fertilizers increases from 2.51

to 4.68;

3. by the increase in the consumption of ground phosphate rock in the system under consideration from 0.29 t to 0.54 t with the corresponding decrease in the specific consumption of ammonium nitrate, the ratio of nutrients N/P₂O₅ in the target NP-fertilizers decreases from 4.68 to 2.51;
4. for the production of target N/P - fertilizers based on a mixture of a solution of ammonium nitrate of GOST 2-2013, the concentration of 64-71% and Chilisay ground phosphate rock of FM-2 grade TS 930640000252-01-2011 with a 17% content of P₂O₅, consumption of ammonium nitrate and ground phosphate rock in their mixture should be supported by ammonium nitrate in the range of 0.36t and 0.67t and in-ground phosphate rock – 0.54t and 0.29t. Moreover, in the composition of possible assortments of N/P-fertilizers obtained, the total content of nutrients will be: 28%; 27%; 26%; 25% or, respectively, in mass % N/P₂O₅ – 23/5; 21/6; 19/7; 18/7, which is quite convincing evidence of their high agrochemical value.

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Table 1. Specific consumptions of the initial components, contents, and ratios of nutrients in the target products

Samples of products obtained	Specific consumption, tonne		Content of the nutrients in the target product	The ratio of the nutrients in the target product
	Ammonium nitrate	Ground phosphate rock	N/ P ₂ O ₅ (%/%)	N/ P ₂ O ₅ (%/%)
1	0,320	0,647	11/11	1/1
2	0,378	0,588	13/10	1,3/1
3	0,552	0,412	19/7	2,71/1
4	0,610	0,353	21/6	3,5/1
5	0,668	0,294	23/5	4,6/1

Table 2. The basic physical and chemical properties of target products

Samples of products obtained	pH of 10% solution	The moisture of product (%)	Strength of product granules (N/g)	The ratio of nutrients in the product (N:P ₂ O ₅)	Granulometric composition of the target product, mass %	
					1-4 mm	2-4 mm
1	6,42	0,28	66,84	1/1	80-92	79-81
2	6,40	0,28	8,70	1,3/1	90-93	80-85
3	6,27	0,27	8,06	2,71/1	94-98	86-90
4	6,30	0,27	8,06	3,5/1	94-98	90-95
5	6,47	0,28	12,15	4,6/1	94-98	92-97

Table 3. Variation intervals of variables

Variation areas of the variables	Variables		
	In coded form	In natural form	
	X _i	Specific consumption of ammonium nitrate, t (AN)	Specific consumption of ground phosphate rock, t (GPR)
Basic level	0	0,494	0,470
Variation interval	Δ	0,123	0,125
Upper level	+1	0,617	0,595
Lower level	-1	0,371	0,345
Upper "stellar" arm	+1,414	0,668	0,647
Lower "stellar" arm	-1,414	0,320	0,294

Table 4. Planning matrix and research results on the impact of ammonium nitrate and ground phosphate rock in the initial mixture on the ratio of nutrients in the target products

No.	Variables				The ratio of N/ P ₂ O ₅ in fertilizer
	In coded form		In natural form		
	X ₁	X ₂	AN, t	GPR, t	
1	+1	+1	0,617	0,595	2,09
2	+1	-1	0,617	0,345	3,62
3	-1	+1	0,371	0,595	1,26
4	-1	-1	0,371	0,345	2,18
5	+1,414	0	0,668	0,470	3,02
6	-1,414	0	0,320	0,470	1,37
7	0	+1,414	0,494	0,647	1,54
8	0	-1,414	0,494	0,294	3,41
9	0	0	0,494	0,470	2,13
10	0	0	0,494	0,470	2,13
11	0	0	0,494	0,470	2,13
12	0	0	0,494	0,470	2,13
13	0	0	0,494	0,470	2,13

Table 5. Comparative information on the results of experimental and calculated values of the nutrients' ratios in samples of target NP – fertilizers

No.	N/P ₂ O ₅ experimental	N/P ₂ O ₅ calculated	% of errors
1	2,18	2,22	-1,84
2	3,62	3,67	-1,55
3	1,26	1,25	0,67
4	2,09	2,09	-0,35
5	3,02	2,98	1,16
6	1,37	1,35	0,91
7	1,54	1,55	-0,69
8	3,41	3,35	1,70
9	2,13	2,12	0,56
10	2,31	2,12	8,31
11	1,96	2,12	-8,06
12	2,04	2,12	-3,82
13	2,15	2,12	1,49

Table 6. Technological parameters in the abc areas boundary (according to Figure3)

Points in Figure 3	Specific consumption of ammonium nitrate, t	Specific consumption of ground phosphate rock, t	N/ P ₂ O ₅ ratio in the target product
a	0,36	0,29	2,51/1
b	0,67	0,54	2,51/1
c	0,67	0,29	4,68/1

Figure 1. The change in the ratio of nutrients depending on the content of nitrogen and phosphorus pentoxide in the target product

Figure 2. The change in the ratio of nutrients depending on the specific consumption of ammonium nitrate and ground phosphate rock in the target product

Figure 3. The influence of specific consumption of ammonium nitrate and ground phosphate rock on the ratio of nutrients (N/P₂O₅) in the target product. I – Volumetric image, II – Horizontal sections.