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Article

Morphological Studies of Chlorine-Free Potassium Fertilizers

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Abstract: This article analyzes methods for producing potassium nitrate from local raw materials and provides a scientific basis for them. The technological parameters of the conversion reaction involving potassium chloride and magnesium nitrate—such as temperature, concentration of the reaction mass, molar ratio, mixing rate, and process duration—were studied for their effect on product formation. In addition, the potassium nitrate obtained from the synthesis was analyzed using modern physicochemical methods, including crystal structure, solubility, moisture content, and purity. The study identified the optimal technological conditions that ensure the maximum yield of potassium nitrate.

Keywords: conversion, potassium chloride, magnesium nitrate, multicomponent water–salt system, crystallization, temperature, filtration

1. Introduction

One of the main directions of economic development in the Republic of Uzbekistan is the utilization of natural resources, their integrated use, and the creation of competitive, import-substituting products based on local raw materials [1].

Developing a highly efficient technology for producing potassium nitrate from local raw materials in Uzbekistan is a pressing task, as it enables meeting domestic market demand and increasing the country's export potential [2].

Currently, there is a rapidly growing global demand in agriculture for environmentally safe and highly efficient mineral fertilizers. In particular, potassium fertilizers that do not contain chloride ions are of great importance for agricultural crops. This is because chloride can negatively affect many crop types, deteriorate the agrochemical properties of the soil, and impact the quality of the produce. For this reason, the production of complex NK and NPK mineral fertilizers using chloride-free potassium sources is one of the most promising directions in the modern agro-industrial market [3]

From this perspective, developing technologies for producing chloride-free potassium fertilizers, including complex NK and NPK fertilizers, based on local raw material sources is crucial for ensuring the sustainable development of the country's agriculture, increasing the volume of import-substituting products, and enhancing the competitiveness of the agro-industrial complex [4].

2. Materials and Methods

The process of obtaining potassium nitrate involves several consecutive technological stages. In the first stage, an ion-exchange reaction occurs between potassium chloride and magnesium nitrate, resulting in the formation of potassium nitrate in the solution. This process is carried out under specific temperature and concentration conditions to ensure complete reaction and maximize product yield [5].

In the next stage, the resulting reaction mass — the pulp — is separated using mechanical and physicochemical methods. During this stage, magnesium chloride, which forms as a by-product, is removed from the solution, playing an important role in ensuring the purity of potassium nitrate in subsequent processes.

Potassium nitrate is then obtained from the separated solution through a crystallization process. By controlling the size and morphology of the formed crystals, the product achieves high agronomic and chemical purity [6].

In the final stage, the separated potassium nitrate crystals undergo a drying process, reducing the moisture content to standard levels. The drying process enhances the product's storage stability and prepares it for further use or packaging [7].

Studying the crystallization rate within a specific temperature range and determining the K_2O yield depending on key technological parameters are important directions of the research. The obtained potassium nitrate and intermediate solutions were analyzed using commonly accepted methods [8].

The following physicochemical analysis methods were used in the research: scanning electron microscopy, infrared spectroscopy, thermoanalytical methods, and X-ray phase analysis.

The morphology and microstructure of the samples were studied using a scanning electron microscope, SEM – EVO MA 10 (Carl Zeiss, Germany). The local elemental composition of the powders was determined using an energy-dispersive X-ray analyzer (EDX, Oxford Instruments) [9].

3. Results and Discussion

Analysis of the research results showed that during the stepwise cooling of the solutions obtained after the conversion process, the formation of Potassium nitrate crystals was observed in the system, resulting in the formation of a suspension consisting of crystals and a liquid phase. This phenomenon directly affects the technological efficiency of the crystallization and subsequent filtration stages in the process of potassium nitrate separation. In order to determine the effect of crystal content on the crystallization intensity and the main technological parameters of the filtration process during potassium nitrate production, experiments were carried out at different stoichiometric ratios. In particular, the $KCl/Mg(NO_3)_2$ ratios were selected as 1:0.8, 1:1, and 1:1.2. These ratios made it possible to more thoroughly analyze the completeness of the reaction, the composition of the solution, and the crystal formation process.

The conversion process was carried out in the temperature range of 80–90 °C, with reaction times set at 10 and 15 minutes. These parameters were evaluated as key factors influencing the reaction rate, the degree of ion exchange, and the subsequent crystallization ability of the solution. After completion of the reaction, the solutions were cooled under specified conditions, and the crystallization process was conducted at 10–20 °C [10].

The experimental results showed that a decrease in crystallization temperature led to an intensified formation of Potassium nitrate crystals, an increase in crystal yield, and changes in their morphological characteristics. It was also noted that the amount of formed crystals significantly affected the filtration rate, filtration resistance, and the purity of the separated product.

Thus, optimization of the cooling conditions of the conversion solutions, as well as the proper selection of raw material ratios and temperature–time parameters, was found to be crucial for improving the efficiency of the crystallization and filtration stages in the production of potassium nitrate.

According to the results of the conducted experiments, the liquid-to-solid phase ratio (L:S) varies within the range of 3,7–7,94 depending on the technological conditions of crystallization. At

the same time, the filtration rate does not have a significant effect on the duration of the exchange decomposition reaction or on the K_2O yield.

As shown in Table 1, the moisture content of the product after filtration decreases from 17,6% to 9,6% and from 12,3% to 3,54%. The highest degree of solution clarification is observed at a temperature of 10 °C. When the temperature exceeds 20 °C, the potassium yield in the potassium chloride : magnesium nitrate ratio remains almost unchanged.

Under these parameters, the filtration rate of the suspension is relatively high, reaching 732–1051 kg/m²·h.As a result, the chlorine content in the dry, unwashed product decreases from 2–3% to 1.7–1.8%. This can be explained by the formation of large prismatic crystals with dimensions e–h–b = 1.13 × 0.3 × 0.1 mm (Figure 1).

Analysis of the provided microscopic images clearly reveals the morphological characteristics of Potassium nitrate crystals. Specifically, the crystals are predominantly elongated, prismatic, and needle-like in shape, reflecting the characteristic crystal structure of potassium nitrate. The orientation of the crystals in various directions, as well as the observation that many of them are interconnected or form small aggregates, indicates that crystal growth during the crystallization process occurred in both parallel and oblique directions [11].

Table 1. Effect of Technological Parameters on the Production of Potassium nitrate by the Conversion Method.

№	KCl/ $Mg(NO_3)$	Conversion temperature	Conversion time	Crystallization temperature	Crystallization time	Filtration rate $V_ф$	Moisture content of the solid phase%	Liquid-to-Solid ratio L:S	K_2O yield %
1	1;0,8	80	10	10	15	1051	3,5	5,3;1	35,55
2			10	20	10	837	3,9	5,79;1	41,91
3		90	15	10	15	667,7	5,6	7,78;1	30,31
4			15	20	10	732	5,23	7,94;1	30,26
5	1;1	80	10	10	15	1086,3	17,6	5,34;1	46,9
6			15	20	10	942,2	11,5	7,1;1	49,1
7		90	15	10	15	895	13,7	3,7;1	45,37
8			15	20	10	1031	9,6	5;1	44,98
9	1;1,2	80	10	10	15	817	12,3	4,4;1	49,59
10			10	20	10	1093	3,87	5;1	45,37
11		90	15	10	15	1866	3,54	5,2;1	50,06
12			15	20	10	791	9,4	5,6;1	50,70

Microscopic analysis of the potassium nitrate crystals showed well-defined edges and relatively smooth surfaces, indicating stable temperature and concentration during crystallization.

The crystals were polydisperse, with some large elongated and others smaller, and some exhibiting internal zoning and growth lines, reflecting stepwise precipitation and changing solution saturation. Crystals were mostly transparent with few mechanical inclusions, confirming high chemical purity suitable for further processing and agrochemical use [12].

4x 10x

Figure 1. Micrographs of Potassium nitrate crystals (×4 and ×10) obtained from the samples prepared according to Table 1

Optical microscopy revealed predominantly needle-like, elongated prismatic crystals, with lengths ranging from 6.75 μm to 14.28 μm, suggesting numerous nuclei formation during the initial crystallization stage followed by limited growth.

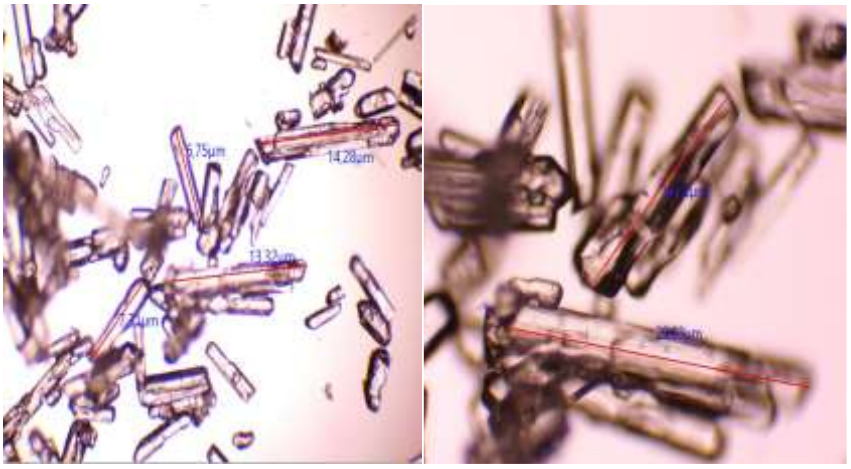

Figure 2. Effect of technological parameters on the particle size distribution of Potassium nitrate crystals after crystallization at a KCl:$NaNO_3$ ratio of 1:1 and a temperature of 10 °C [13].

In the second microphotograph, relatively large and well-formed crystals are observed, with lengths ranging from 18,72 μm to 29,83 μm. This indicates that in the later stages of crystallization, the number of nuclei decreased, allowing the existing crystals to grow freely. Additionally, some crystals are seen to have adhered to each other (agglomeration), which may suggest a higher degree of supersaturation in the solution [14].

The variation in crystal sizes and differences in morphology confirm that technological parameters—specifically, solution concentration, cooling rate, stirring intensity, and crystallization time—are directly related to crystal formation. By optimizing these factors, the size and shape of Potassium nitrate crystals can be purposefully controlled.

Overall, the microscopic analysis results indicate that the obtained potassium nitrate crystals are morphologically stable, have controllable sizes, and possess high quality. This further supports their effective use in agriculture as a chlorine-free potassium fertilizer.

4. Conclusion

Based on the experimental results, the obtained potassium nitrate (KNO_3) fully meets the requirements for use as an effective chlorine-free potassium fertilizer in agriculture. The potassium nitrate content of the product is 98–99%, confirming its high purity. Additionally, the levels of foreign ions and impurities are below permissible limits, ensuring that the K^+ ions are efficiently absorbed by plants at a rate of 97–98% [15].

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