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Influence of granule structure mineral fertilizers for their physical and chemical properties

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<p>Received: May 28, 2024 Peer-reviewed: June 28, 2024 Accepted: September 5, 2024</p>	<p>ABSTRACT</p> <p>In this article, maintaining the quality and commercial properties of mineral fertilizers during repeated transshipment and long-term transportation is one of the important issues. The purpose of the research work is to study the structure of granules of complex mixed fertilizers, as well as to find and develop methods for improving it at the production stage in order to improve the physical and mechanical properties of the finished product. In order to improve the granular structure of the mineral fertilizer, the static strength of the granules, the mass fraction of moisture, the chemical composition and structural shape of the granules were determined using an electron microscope. Using the X-ray microtomography method, experimental data on the internal structure of granules of complex mineral fertilizers were obtained and generalized for the first time, porosity, the nature of the distribution of pores and components of granules by volume were assessed. It has also been studied that the structure of granules and the nature of the pore distribution of fertilizers obtained according to different granulation schemes have fundamental differences, which is explained by the difference in the mechanisms of granule formation. It has been shown that when producing NP and NPS fertilizers using a reverse neutralization scheme, existing defects in the granule structure (cracks and pores) do not impair the physical and mechanical characteristics of the product, which makes it possible to recommend this scheme for use and thereby increase productivity by 20-25%.</p>
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Introduction

The manufacture of mineral fertilizers is an important area of the Kazakh agro complex, occupying a leading position in non-hydrocarbon, non-resource exports.

The country is experiencing an increase in the production of complex mixed mineral fertilizers for export.

The range of mineral fertilizers produced in the Republic of Kazakhstan today is changing in favor of complex NP, NP(S) and NPK fertilizers, which meets modern consumer preferences. Complex mixed fertilizers, as a rule, are more concentrated and require lower costs for transportation, storage and application compared to single-component fertilizers.

Improved quality of exported mineral fertilizers is a prerequisite for increasing export potential and an important factor in maintaining the competitiveness of Kazakhstani producers. Thus, the problem of preserving the quality of mineral fertilizers during numerous transshipments and long-term transportation is one of the key ones for the industry as a whole. In addition, by improving the physicochemical properties of mineral fertilizers, can significantly growth the efficiency of their use and avoid losses at the application stage [[1],[2]].

The consumer properties of granular mineral fertilizers - static strength of granules, caking, dustiness - are interrelated and depend on the substance constituents and building of the granules, which is largely determined by the technological parameters of the production process [[3], [4]].

Currently, modern non-destructive methods for studying the structure of solids, such as X-ray microtomography and scanning electron microscopy, have appeared and become available. The use of these methods allows us to study in detail the structure and distribution of chemical elements in granules of complex mineral fertilizers, as well as their relationship with physical and chemical properties [[5],[6]].

The importance of scientific work is to study the structure of granules of complex mixed fertilizers, as well as to search and develop methods for improving it at the production stage to improve the physical and mechanical feature of the eventual product.

Experimental part

Object of research. To study the properties of granular mineral fertilizers, the most popular brands of NP, NPS and NPK fertilizers produced by Kazphosphate LLP were selected. Sample preparation and sampling for experimental work was carried out in accordance with the established procedure [7].

Research methods. *Determination of the static strength of sample granules.* The static strength of sample granules was measured according to GOST 21560.2-82. The static strength of the granules was studied using a texture analyzer Text plus. The strength measurement range is from 0.1 to 10 MPa, with a relative error $\pm 4\%$ and a speed of movement of the work table from 0.8 to 1.0 mm/s.

Determination of mass fraction of water. The mass fraction of hygroscopic and total water was determined by drying in oven in accordance with [8].

Study of the structure of granules using X-ray microtomography. The study of the structure of fertilizer granules using X-ray microtomography was carried out using a SkyScan1172 device (Bruker), followed by computer analysis of the data obtained. Computer processing was carried out in the nRecon and CTan programs. nRecon converts shadow projections into tomographic slices. Analysis of tomographic sections was carried out in the CTan program to isolate X-ray contrast phases. The analysis is carried out over a certain volume in the shape of a sphere inscribed within the dimensions of a mineral fertilizer granule.

Study of the structure and chemical composition of granules using scanning electron microscopy. The building of the granules was researched by SEM using a TM3030 microscope (Hitachi, Japan), as well as on a JEOL JSM-JSM-6490I V scanning electron microscope SEM. The spread of chemical compounds over the surface of chipped granules was researched by X-ray using an EDS Quantax 70 (Bruker).

The discussion of the results

The structure of granules of complex fertilizers is determined, first of all, by the technology of their production: the method of granulation and drying, as well as the modes of the technological cycle and the method of introducing raw materials. In turn, the structure of granules affects the physicochemical and physicomechanical characteristics of granular fertilizers - static strength, caking, abrasion, dustiness [[9], [10]].

To assess the surface porosity of fertilizer granules, it is proposed to supplement the X-ray microtomography method by studying the distribution of carbon on chipped granules treated with conditioning additives based on mineral oil. The depth of penetration into the carbon granule, and thus the depth and approximate volume of surface pores. This is possible because the fertilizers under study contain no carbon [[11], [12]]. Distribution of phosphorus and carbon over the cleavage (blue color indicates phosphorus, red color indicates carbon). The obtained data are depict in Figure 1.

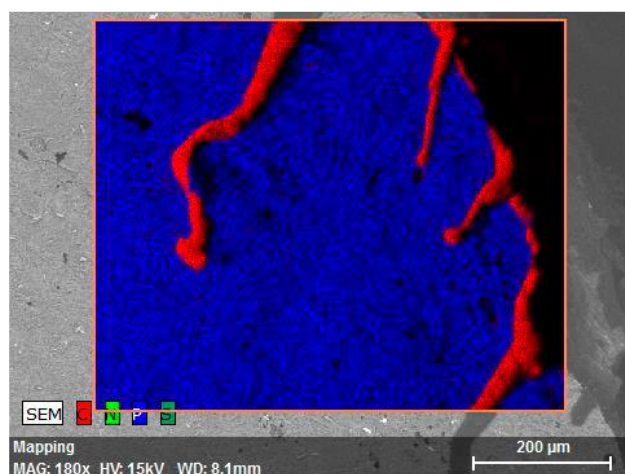
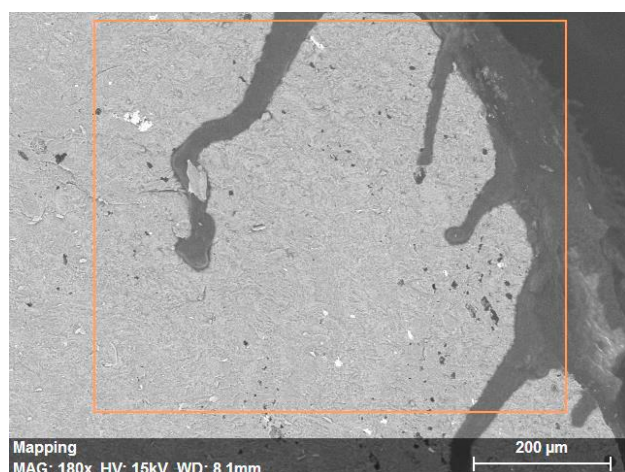


Figure 1 - Micrograph and distribution of chemical elements over a chipped granule

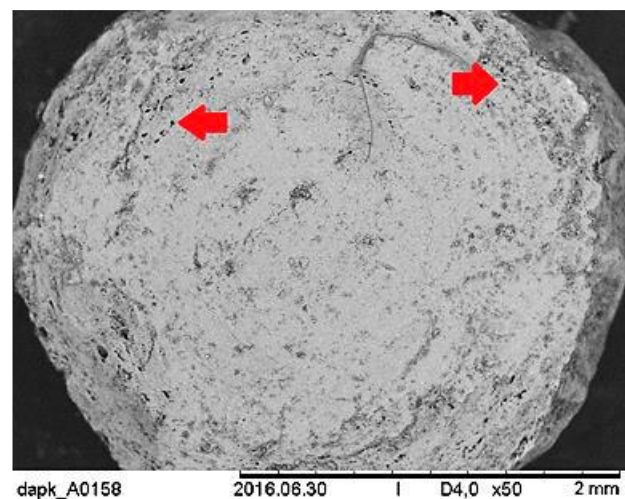


Figure 2 - Micrograph of chipped granule sample 1

Based on Figure 1, it is shown that the depth of individual surface pores can reach 400 μm. It should be noted that the presence of surface pores largely determines the hygroscopic characteristics of the product, and can also increase the consumption of conditioning additives. Using non-

destructive methods, a comparative analysis of the structure of granules and physical and mechanical properties of monoammonium phosphate samples (sample 1), obtained according to the scheme with one-stage neutralization and with reverse neutralization - sample 2, was carried out. The data obtained are depicted in Figures 2 and 3.

From Figure 2 it can be seen that the granules of sample 1 have a more uniform structure.

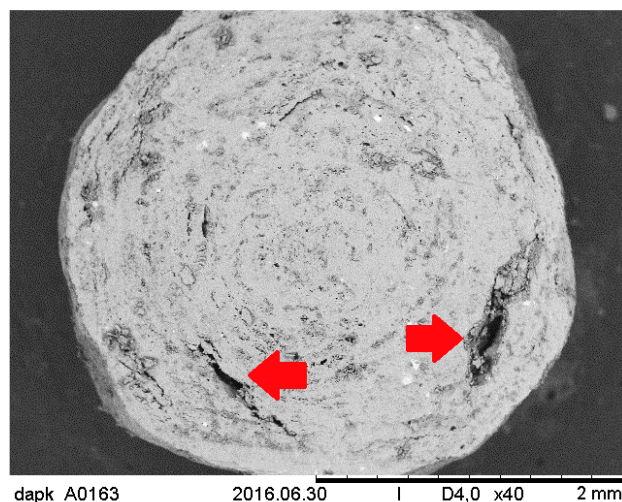


Figure 3 - Micrograph of chipped granule sample 2

From Figure 3 it follows that the granules of sample 2 contain individual large cracks and pores with a diameter of up to 100-150 μm. The overall porosity of the granules is also slightly higher compared to sample 1. This is due to the lower humidity and higher viscosity of the pulp during the neutralization process [[13], [14]].

The results obtained during processing with the energy-dispersive method using scanning electron microscopy JEOL JSM-JSM-6490I V) (JEOL, Japan) are depicted in Figure 4.

From Figure 4 it follows that the distribution of chemical elements over the chips of granules of the studied samples allowed us to establish that the distribution of nitrogen, phosphorus and sulfur in both cases is uniform over the volume of the granule [[15], [16]]. For the samples under study, the static strength of the granules was also determined.

Table 1 - Static strength and porosity of MAF granules

Product	Static strength of granules, MPa	Porosity, %
Sample 1	7.5±0.5	4.1±0.1
Sample 2	9.2±0.8	5.8±0.1

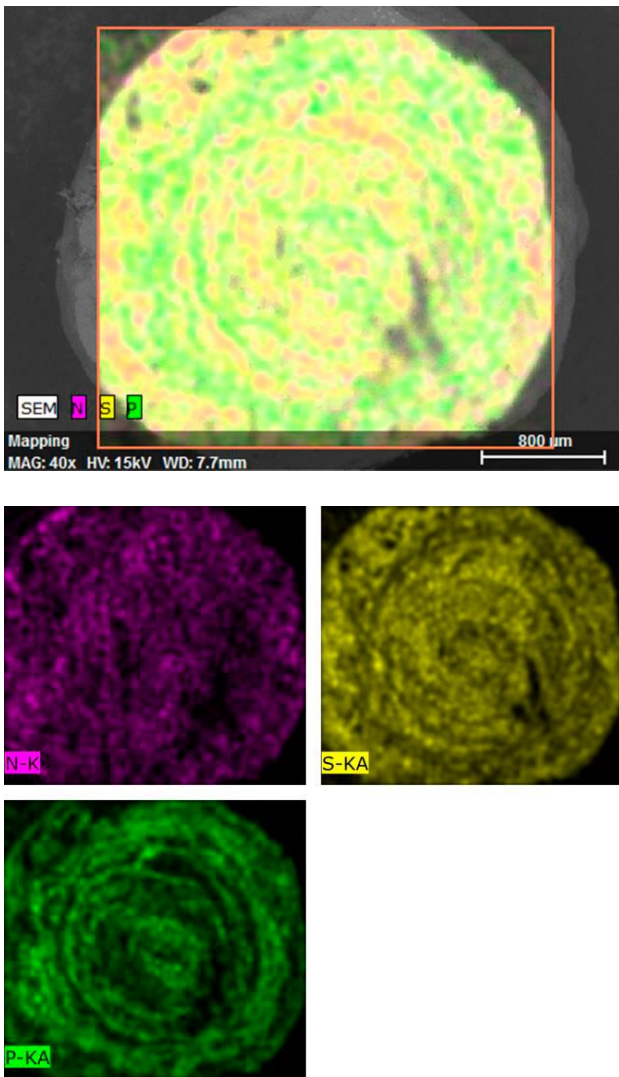


Figure 4 - Distribution of chemical elements over a chipped MAF granule

From Table 1 it follows that the static strength of the sample granules is satisfactory; for granules obtained using the “deoxidation” scheme, the static strength is slightly higher. Based on the results of the studies, it was determined that in the case of samples obtained with reverse neutralization, the existing structural defects (cracks and pores) do not have a significant effect on the strength of the granules. The distribution of the main elements - nitrogen, phosphorus, sulfur - is evenly distributed throughout the volume of the granules. All this allows us to recommend a scheme with reverse neutralization for use [[17], [18]].

Using non-destructive analysis methods, the structure of granules of a number of NPK and NP(S) samples was studied and a tomographic cross-section of an NPK fertilizer granule was presented [[19], [20]]. The maximum X-ray density on the

presented tomographic sections corresponds to white, and the minimum (air) to black.

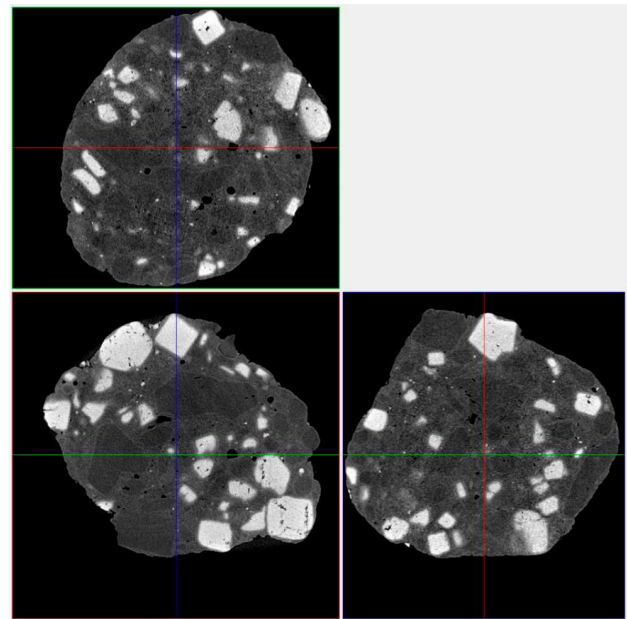


Figure 5 - Tomographic cross-section of an NPK fertilizer granule

Based on the data (Figure 5) obtained using microtomography, we can conclude that in samples of NPK and NPS fertilizers, crystals of ammonium sulfate and potassium chloride are distributed throughout the volume of the granules quite evenly and practically do not dissolve in the phosphate pulp (crystal boundaries clearly expressed), and the pores present in the granule are not associated with low wettability of KCl and $(NH_4)_2SO_4$. This is confirmed by analysis of granule chips using SEM.

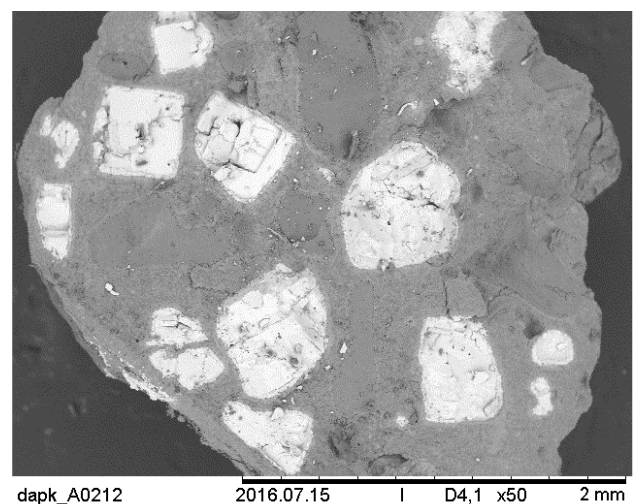


Figure 6 - Microphotograph of a chipped NPK fertilizer granule

From Figure 6 it follows that the structure of the granule is quite dense, the crystals of NPK fertilizers are evenly distributed throughout the volume of the granule.

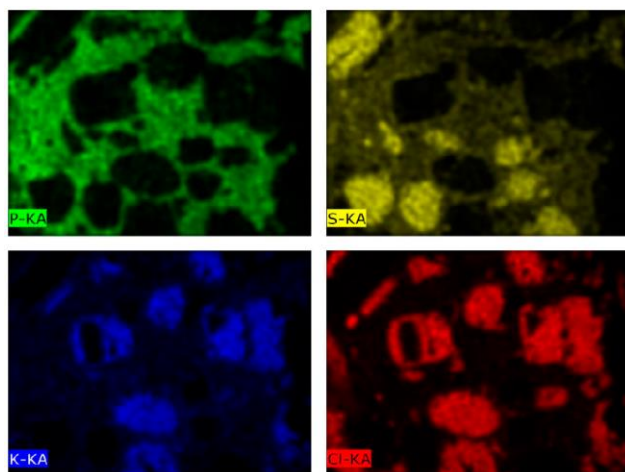
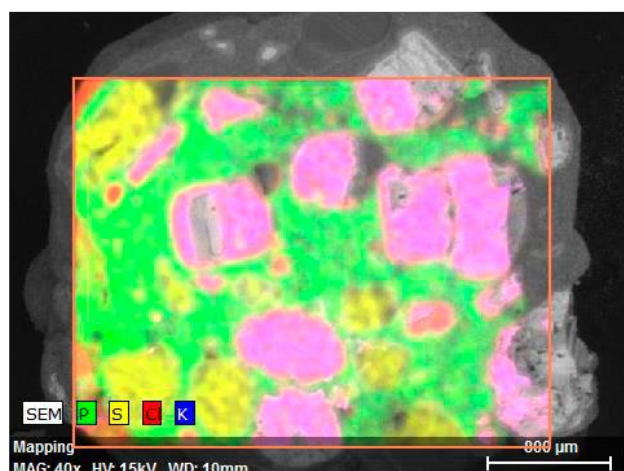


Figure 7 - Distribution of chemical elements across the grain of an NPK fertilizer granule

From Figure 7 it follows that the analysis of chips of granules of the fine fraction (1-2 mm) of complex NP(S) and NPK fertilizers confirms that the introduced KCl and $(\text{NH}_4)_2\text{SO}_4$ are well wetted by the phosphate solution.

Conclusions

Using the X-ray microtomography method, experimental data on the internal structure of granules of complex mineral fertilizers were obtained and generalized for the first time, porosity, the nature of the distribution of pores and components of granules by volume were assessed. It has been shown that the structure of granules and the nature of the pore distribution of fertilizers obtained according to different granulation schemes have fundamental differences, which is explained by the difference in the mechanisms of granule formation.

It was revealed that in granules of NPK and NP(S) fertilizers produced according to the AG-SB scheme, crystals of ammonium sulfate and potassium chloride are distributed evenly, and the pores present in the granules are not associated with the low wettability of the introduced KCl and $(\text{NH}_4)_2\text{SO}_4$.

It has been shown that when producing NP and NPS fertilizers using a reverse neutralization scheme, existing defects in the granule structure (cracks and pores) do not impair the physical and mechanical characteristics of the product, which makes it possible to recommend this scheme for use and thereby increase productivity by 20-25%.

CRedit author statement: **B.Smailov:** Conceptualization, formal analysis, investigation, data writing, original draft preparation, writing–review and editing. **O.Beisenbayev:** Data curation, writing draft preparation, methodology, **A. Anarbayev:** Resources, supervision. **B. Zakirov:** Investigation, visualization. **U. Aravind:** software, validation.

Conflicts of Interest. On behalf of all authors, the correspondent author declares that there is no conflict of interest.

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Минералды тыңайтқыштар түйіршіктерінің құрылымының олардың физика-химиялық қасиеттеріне әсері

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<p>Мақала келді: 28 мамыр 2024 Сараптамадан өтті: 28 маусым 2024 Қабылданды: 5 қыркүйек 2024</p>	<p>ТҮЙІНДЕМЕ Минералды тыңайтқыштарды қайта тиеу және ұзақ мерзімге тасымалдау кезінде олардың сапасы мен тауарлық қасиеттерін сақтау маңызды мәселелердің бірі болып табылады. Ғылыми-зерттеу жұмысының мақсаты – күрделі аралас тыңайтқыштардың түйіршіктерінің құрылымын зерттеу, сонымен қатар дайын өнімнің физика-механикалық қасиеттерін жақсарту мақсатында өндіріс сатысында оны жетілдіру әдістерін табу және әзірлеу. Минералды тыңайтқыштың түйіршікті құрылымын жақсарту мақсатында түйіршіктердің статикалық беріктігі, ылғалдың массалық үлесі, түйіршіктердің химиялық құрамы мен құрылымдық пішіні электронды микроскоптың көмегімен анықталды. Рентгендік микротомография әдісін қолдана отырып, күрделі минералды тыңайтқыштардың түйіршіктерінің ішкі құрылымы туралы тәжірибелік мәліметтер алынды және алғаш рет жинақталды, кеуектілігі, көлемі бойынша түйіршіктердің кеуектері мен компоненттерінің таралу сипатына баға берілді. Сондай-ақ түйіршіктердің құрылымы мен әртүрлі түйіршіктеу схемалары бойынша алынған тыңайтқыштардың кеуекті таралу сипатының принципті айырмашылықтары бар екендігі зерттелді, бұл түйіршіктердің түзілу механизмдерінің айырмашылығымен түсіндіріледі. Кері бейтараптандыру схемасын қолдана отырып, NP және NPS тыңайтқыштарын өндіру кезінде түйіршік құрылымындағы бар ақаулар (жарықтар мен кеуектер) өнімнің физикалық және механикалық сипаттамаларын өзгертпейтіні анықталды, бұл осы схеманы пайдалануға ұсынуға мүмкіндік береді және сол арқылы өнімділікті 20-25%-ға арттырады.</p>
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Влияние структуры гранул минеральных удобрений на их физико-химические свойства

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	<p>АННОТАЦИЯ Сохранение качественных и товарных свойств минеральных удобрений при многократной перевалке и длительной транспортировке является одним из важных вопросов. Цель научно-исследовательской работы – изучить структуру гранул комплексных смешанных удобрений, а также найти и разработать методы ее улучшения на этапе производства с целью улучшения физико-механических свойств готового продукта. С целью улучшения</p>
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<p>Поступила: 28 мая 2024 Рецензирование: 28 июня 2024 Принята в печать: 5 сентября 2024</p>	<p>зернистой структуры минерального удобрения с помощью электронного микроскопа определяли статическую прочность гранул, массовую долю влаги, химический состав и структурную форму гранул. С использованием способа рентгеновской микротомографии были определены и обобщены экспериментальные данные о внутренней структуре гранул сложных минеральных удобрений, оценена пористость, характеризующий распределения пор и соединений гранул по объему. Также изучено, что строение гранул и свойства пор удобрений, полученных по разным схемам грануляций, имеют принципиальные различия, что объясняется различием механизмов гранулообразования. Показано, что при получении NP и NPS удобрений по схеме обратной нейтрализации имеющиеся дефекты структуры гранул (трещины и поры) не ухудшают физико-механические характеристики продукта, что позволяет рекомендовать данную схему к использованию и тем самым повысить производительность на 20-25%. Установлено, что при производстве NP-и NPS-удобрений по установленной схеме с обратной нейтрализацией имеющиеся дефекты строения гранул не изменяет физико-механические характеристики готового продукта, что позволяет рекомендовать данную технологию к применению и тем самым повышает производительность на 20-25%.</p>
	<p>Ключевые слова: минеральное удобрение, прочность, структура, зерно, исследование.</p>
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