

## SOFT ORGANOMODIFICATION OF BENTONITE WITH OXYPHOSPHONATE AND ITS APPLICATION AS A PROLONGED-LASTING GROWTH STIMULATOR

*B.Y. Kapar*<sup>1,2</sup>, *T.Y. Zharkynbek*<sup>1,2\*</sup>, *B.B. Tyussyupova*<sup>2</sup>, *D.M.-K. Ibraimova*<sup>2</sup>,  
*S.M. Tazhibayeva*<sup>2</sup>, *V.K. Yu*<sup>1</sup>

<sup>1</sup>*A.B. Bekturov Institute of Chemical Sciences JSC, Almaty, Kazakhstan*

<sup>2</sup>*Al-Farabi Kazakh National University, Almaty, Kazakhstan*

\**Corresponding author e-mail: tolganay.zharkynbek@gmail.com*

**Abstract.** *Introduction.* Organomodified clay materials are of significant interest as functional systems for agriculture due to their ability to provide the sustained release of biologically active substances. Natural bentonite, with its high specific surface area and ion-exchange capacity, serves as an excellent matrix for the immobilization of organic growth stimulants. *The purpose* of this study is to develop a low-energy, efficient method for the soft organomodification of bentonite using a specific oxyphosphonate (Kaz-6) and to evaluate its effectiveness as a prolonged-action growth stimulator for wheat. *The proposed method* involves the pre-dispersion of bentonite in water at 40°C, followed by drying at 50°C. The prepared mineral matrix was then treated with low-concentration aqueous solutions of dimethyl(4-hydroxy-1-(2-ethoxyethyl)piperidin-4-yl)phosphonate (Kaz-6) in the concentration range of 10<sup>-2</sup>–10<sup>-6</sup> wt % at room temperature. The structure of the resulting composite was characterized using IR spectroscopy. It was found that the developed approach significantly reduces the process temperature and duration compared to existing analogs, while using substantially lower concentrations of the organic component. Biological trials showed that the organomodified bentonite exhibits pronounced growth-promoting activity toward wheat (varieties “Almaken” and “Zhenis”). The most significant stimulatory effect was observed at concentrations of 10<sup>-5</sup>–10<sup>-6</sup> wt. %, where the composite outperformed both the control group and the free (unbound) oxyphosphonate. The results indicate that the immobilization of Kaz-6 on bentonite ensures prolonged action and increased bioavailability of the active component. The developed organomineral composite can be recommended as a promising growth-promoting agent for sustainable agricultural applications.

**Key words:** bentonite, organomodification, oxyphosphonate, immobilization, growth stimulator, wheat, sustained release.

<i>Kapar Bayan Yerbolkyzy</i>	<i>Master student, Engineer; E-mail: kaparb13@gmail.com</i>
<i>Zharkynbek Tolganay Yerkinkyzy</i>	<i>Master of Engineering and Technology, Junior Researcher; E-mail: tolganay.zharkynbek@gmail.com</i>
<i>Tyussyupova Bakyt Baimuratovna</i>	<i>Candidate of Chemical Sciences, Associate Professor; E-mail: baimuratovna78@mail.ru</i>
<i>Ibraimova Dana Mykty-Kereyevna</i>	<i>Candidate of Chemical Sciences, Senior Lecturer; E-mail: dmk_82@mail.ru</i>
<i>Tazhibayeva Sagdat Mederbekovna</i>	<i>Doctor of Chemical Sciences, Professor; E-mail: sagdattazhibayeva2018@gmail.com</i>
<i>Yu Valentina Konstantinovna</i>	<i>Doctor of Chemical Sciences, Full Professor; E-mail: yu_yk@mail.ru</i>

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## 1. Introduction

Natural clay minerals, particularly bentonite, are widely used in various fields due to their high specific surface area, pronounced sorption properties, and high ion exchange capacity. These characteristics stem from the layered montmorillonite-type structure, which enables the intercalation and fixation of various organic and inorganic compounds [1].

In recent years, the use of bentonite in agriculture as a functional carrier of biologically active substances has attracted particular interest. It has been shown that the application of bentonite to soil improves its physicochemical properties, increases its water-holding capacity, and enhances the availability of nutrients to plants [2]. Furthermore, bentonite is capable of adsorbing organic compounds and ensuring their gradual release, providing a prolonged effect.

One effective approach to improving the functional properties of bentonite is its organomodification. Modern modification methods include the use of surfactants, polymers, and organic acids, which significantly alter the surface characteristics of the material and enhance its sorption activity [1,3]. However, most known approaches are characterized by high concentrations of modifying agents, elevated temperatures, and long processing times, which limits their practical application.

In this regard, a pressing task is the development of gentle and cost-effective methods for organomodification of bentonite using low concentrations of functional organic compounds. Of particular interest are oxyphosphonates, which possess high complexing capacity, hydrophilicity, and potential biological activity [4]. The introduction of phosphorus-containing groups into organomineral systems helps to enhance their coordination and adsorption properties [5].

Furthermore, modern research shows that modified bentonite compositions can improve soil fertility, stabilize organic component, and stimulate crop growth [2,6]. This area is particularly relevant for Kazakhstan due to the widespread use of bentonite clays and the need to increase crop yields in arid climates [7].

Previous studies have demonstrated that organomodified clay materials serve as effective carriers for agrochemicals and controlled-release systems, minimizing the loss of active substances and improving their utilization efficiency [8–10].

In particular, it has been shown that the intercalation of organic molecules into the interlayer space of montmorillonite promotes the formation of stable hybrid structures with controlled desorption kinetics [11].

Modern research also demonstrates that functionalization of the bentonite surface with phosphorus-containing compounds increases its affinity for biologically active molecules and improves their retention in the soil system [12,13]. An important factor is the use of low concentrations of modifying agents, which helps avoid aggregation and preserve the active surface of the mineral [14].

In addition, the use of nanostructured organo-mineral compositions is considered as a promising direction in agrochemistry, aimed at creating environmentally friendly and resource-saving technologies [15–17].

The aim of this work is to develop a method for obtaining organomodified bentonite using Kaz-6 under mild conditions, as well as to study its growth-stimulating activity in relation to wheat.

## 2. Experimental part

### Materials

Natural bentonite from the Tagan deposit (Kazakhstan) was used in the study. Before use, the bentonite was pre-treated (dispersed and dried). Dimethyl(4-hydroxy-1-(2-ethoxyethyl)piperidin-4-yl)phosphonate (Kaz-6) was prepared previously by a known method and used as a modifying agent [18]. Distilled water was used to prepare the solutions. All reagents were used without further purification.

### Preparation of organomodified bentonite

Bentonite was pre-treated as follows: the original bentonite was dispersed in distilled water at 40°C until a homogeneous suspension was obtained. The resulting suspension was dried at 50°C for 3 hours, after which the dried material was ground to a powder.

Organomodification was carried out by treating prepared bentonite with Kaz-6 aqueous solutions with various concentrations ( $10^{-2}$ – $10^{-6}$  wt. %). For this, 1 g of bentonite was mixed with 50 mL of the modifier solution and kept at room temperature for 40 minutes with occasional stirring.

After completion of the process, the mixture was filtered and the resulting solid product was dried at 50°C for 2 hours. As a result, organomodified bentonite was obtained in powder form.

### IR spectroscopy

The IR spectra were recorded in the 4000–400  $\text{cm}^{-1}$  range using a Nicolet 5700 IR spectrometer, using thin-films.

Spectral analysis was performed to confirm the immobilization of the organic component on the bentonite surface. Particular attention was paid to the bands corresponding to the stretching vibrations of the P=O ( $\approx 1230$ – $1250 \text{ cm}^{-1}$ ), P–O–C ( $\approx 1000$ – $1050 \text{ cm}^{-1}$ ) groups, as well as O–H and N–H bonds.

### Biological testing

The growth-promoting activity of organomodified bentonite was studied on the seeds of the “Almaken” and “Zhenis” wheat varieties. The experiments were conducted under laboratory conditions at Al-Farabi Kazakh National University at an average temperature of  $22 \pm 2 \text{ }^\circ\text{C}$  and a relative humidity of  $50 \pm 5\%$ .

The seeds were treated with suspensions of organomodified bentonite containing Kaz-6 at concentrations of  $10^{-2}$ – $10^{-6}$  wt. %. Distilled water and solutions of free phosphonate at appropriate concentrations were used as controls.

The growth-stimulating effect was assessed based on shoot length on days 2 and 14. A series of parallel experiments ( $n = 3$ – $5$ ) were conducted for each treatment, and the results were expressed as mean values.

### 3. Results and Discussion

A comparative analysis of the developed method is presented in Table 1. Compared to the method described in [19], the proposed approach is characterized by a simpler process flow. Direct immobilization of organic oxyphosphonate on a mineral matrix eliminates the need for suspension preparation, multiple washings, and centrifugation. This results in reduced synthesis time and reduced reagent consumption while maintaining the effectiveness of the modification.

**Table 1** – Comparison of conditions for obtaining organomodified bentonite (developed method and prototype)

Concentration of Organic Component, wt. %	Temperature, °C				Time, h			
	Soaking	Drying (pre-treatment)	Aging	Drying	Soaking	Drying (pre-treatment)	Aging	Drying
Dimethyl (4-hydroxy-1-(2-ethoxyethyl)piperidin-4-yl) phosphonate								
10 <sup>-2</sup>	40	50	Room temp.	50	12	3	2/3 (40 min)	2
10 <sup>-3</sup>	40	50	Room temp.	50	12	3	2/3 (40 min)	2
10 <sup>-4</sup>	40	50	Room temp.	50	12	3	2/3 (40 min)	2
10 <sup>-5</sup>	40	50	Room temp.	50	12	3	2/3 (40 min)	2
10 <sup>-6</sup>	40	50	Room temp.	50	12	3	2/3 (40 min)	2
Aminoacetic acid (prototype)								
5.0-5.5	–	110	Room temp.	70	24	6	24	4

Specifically, the processing temperature was reduced from 110 to 50°C, and the holding time was reduced from 24 hours to 40 minutes. Furthermore, the total duration of the preparation and modification stages was more than halved.

A significant advantage of the developed approach is the use of significantly lower concentrations of the organic component (10<sup>-2</sup>–10<sup>-6</sup> wt. %), whereas the prototype uses concentrations of approximately 5 wt. %. Reducing the modifier concentration not only reduces the cost of the process but also promotes a more uniform distribution of organic matter on the bentonite surface.

The IR spectra of organomodified bentonite demonstrate the appearance and/or strengthening of bands characteristic of the oxyphosphonate fragment (Table 2). In particular, bands of the P=O group stretching vibrations are observed in the region of ~1200 cm<sup>-1</sup> and the characteristic P–O–C bands appear as prominent peaks at 1150–1030 cm<sup>-1</sup>. Simultaneously, changes are recorded in the region of broad O–H bands (~3200–3600 cm<sup>-1</sup>), indicating the interaction of the organic component with the hydroxyl groups of the clay mineral surface.

The strong retention of Kaz-6 in the bentonite matrix is likely due to the formation of hydrogen and coordination interactions between the phosphonate groups of the modifier and the active sites of the clay mineral. Although specific

studies of desorption kinetics, binding strength, and the effect of pH on the release of the active component were not conducted in this study, the results of 14-day biological tests indicate the prolonged action of the composite and the effective retention of the modifier in the mineral matrix. A detailed study of the release kinetics and pH-dependent behavior of the system is of significant practical interest and will be the subject of further research.

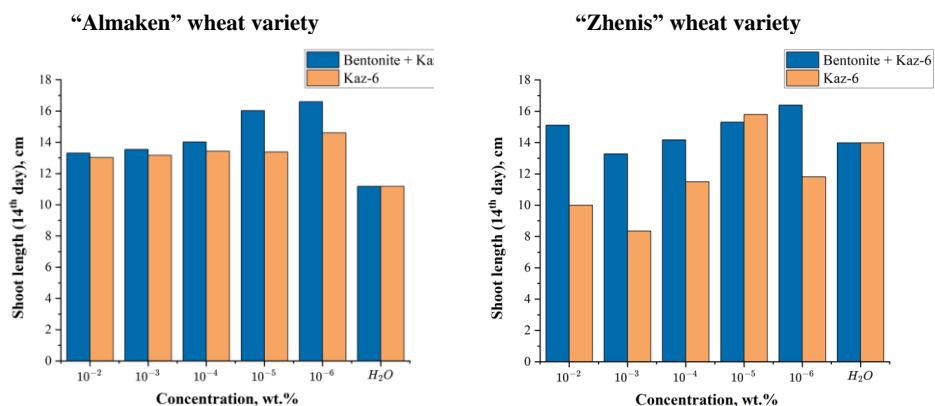
The results of biological tests are presented in Figure 1. The obtained data indicate a pronounced growth-stimulating activity of organomodified bentonite.

Compared to the control ( $H_2O$ ), a significant increase in shoot length was observed on both the 2<sup>nd</sup> and 14<sup>th</sup> days. The most pronounced effect was achieved at concentrations of  $10^{-5}$ – $10^{-6}$  wt. %, where seedling length exceeded control values by an average of 20–40%.

It is important to note that organomodified bentonite exhibits higher activity compared to free oxyphosphonate in similar concentrations. This confirms the effectiveness of immobilization of the active substance on the mineral matrix (Figure 2).

**Table 2** – IR spectra data of bentonite, oxyphosphonate and obtained composite

Functional group	Bentonite, $cm^{-1}$	Oxyphosphonate, $cm^{-1}$	Composite, $cm^{-1}$
v O–H	3628, 3438	3269	3631, 3442, 3347
v C–H <sub>alkyl</sub>	2922, 2853	2957, 2931, 2878, 2822, 2773	2977, 2756
v P=O	–	1225	~1200 (overlaps with Si–O)
v P–O–C / P–O	–	1150–1030	1150–1030 (overlaps with Si–O)
v Si–O	~1008	–	~1009
v Al–OH	916	–	916
$\delta$ CH <sub>2</sub> и CH <sub>3</sub>	–	1500–1300 (intense)	1500–1300 (weak)
$\delta$ Si–O–Al	754, 694	–	768, 700
$\delta$ P–O, Si–O, Al–O	541, 469	565, 492, 429	519, 489, 424



**Figure 1** – The effect of organomodified bentonite on the growth of wheat seedlings of the “Almaken” and “Zhenis” varieties.

An important aspect of using the developed organomineral complexes is assessing their environmental safety and phytotoxicity. During a 14-day observation period, no signs of growth inhibition, chlorosis, or necrotic changes were detected in seedlings of the “Almaken” and “Zhenis” wheat varieties at concentrations ranging from  $10^{-2}$  to  $10^{-6}$  wt.%. Active development of the root system and aboveground parts of the plants indicates the absence of phytotoxic effects and confirms the high biocompatibility of the Kaz-6-bentonite composite at the concentrations studied.

Visual observation of the experimental samples on the 11<sup>th</sup> day (Figure 3) correlates with the quantitative morphometric data. Wheat seedlings of the “Almaken” and “Zhenis” varieties treated with the bentonite-Kaz-6 composite at a concentration of  $10^{-6}$  wt. % show visibly more robust development of the vegetative mass compared to both the distilled water control ( $H_2O$ ) and the free oxyphosphonate.

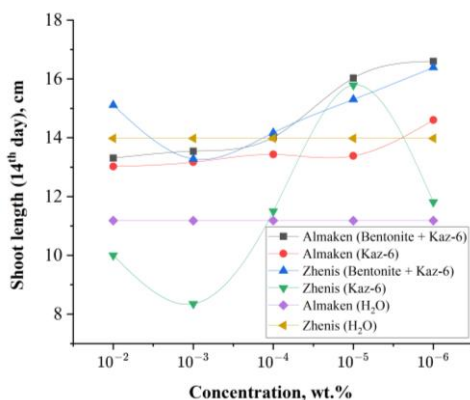


Figure 2 – Dependence of sprout length on modifier concentration



“Almaken” wheat variety



“Zhenis” wheat variety

Figure 3 – Visual observation of the experimental shoots on the 11<sup>th</sup> day

The results obtained allow us to propose a probable mechanism for the formation of organomodified bentonite and its biological activity. Immobilization of oxyphosphonate on the bentonite surface is apparently achieved through:

- coordination interaction between the phosphoryl group (P=O) and surface hydroxyl (Si–OH) of bentonite;
- hydrogen bonds involving O–H and N–H groups;
- possible ion adsorption in the interlayer spaces of montmorillonite.

This interaction leads to the formation of a stable organomineral system in which the oxyphosphonate is fixed on the surface and in the interlayer space of bentonite.

Unlike the free compound, immobilized oxyphosphonate is released gradually, providing a prolonged effect. This explains the higher effectiveness of organomodified bentonite at low concentrations ( $10^{-5}$ – $10^{-6}$  wt. %).

Additionally, bentonite acts as a carrier, improving the distribution of the active ingredient and promoting its retention in the plant root zone. The combination of the mineral's sorption properties and the biological activity of the oxyphosphonate results in a synergistic effect.

#### 4. Conclusion

In this study, we developed an effective method for the mild organomodification of bentonite using Kaz-6 as a modifying agent. It was demonstrated that the proposed approach allows the process to be carried out under mild conditions (40–50 °C) with a significant reduction in processing time compared to known methods.

It was established that the use of low concentrations of oxyphosphonate ( $10^{-2}$ – $10^{-6}$  wt. %) ensures effective immobilization of the organic component on the bentonite surface. IR spectroscopy data confirm the formation of an organomineral system due to the interaction of phosphorus-containing functional groups with the active sites of the clay mineral.

Biological testing results showed that the resulting organomodified bentonite exhibits pronounced growth-promoting activity against the “Almaken” and “Zhenis” wheat varieties. The greatest effect is observed at low modifier concentrations ( $10^{-5}$ – $10^{-6}$  wt. %), indicating a prolonged action and increased efficiency of the active substance.

It was found that immobilization of oxyphosphonate on bentonite provides higher biological activity compared to the free compound, which is associated with its gradual release and more uniform distribution in the system.

Thus, the proposed organomodified bentonite is a promising functional material combining the properties of a mineral carrier and a biologically active component. The practical significance of this work lies in the possibility of creating effective, prolonged-release growth-promoting preparations with reduced active ingredient consumption and a simplified production technology. The developed approach can be used to create new agrochemical compositions adapted to the conditions of arid regions, including Kazakhstan.

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## БЕНТОНИТТИ ОКСИФОСФОНАТПЕН ЖҰМСАҚ ОРГАНОМОДИФИКАЦИЯЛАУ ЖӘНЕ ОНЫ ҰЗАҚ УАҚЫТ ӘСЕР ЕТЕТІН ӨСУ ЫНТАЛАНДЫРҒЫШЫ РЕТІНДЕ ҚОЛДАНУ

*Б.Е.Капар*<sup>1,2</sup>, *Т.Е.Жарқынбек*<sup>1,2\*</sup>, *Б.Б.Тюсюпова*<sup>2</sup>, *Д.М.-К.Ибраимова*<sup>2</sup>,  
*С.М.Тажиебаева*<sup>2</sup>, *В.К.Ю*<sup>1</sup>

<sup>1</sup> *Ә.Б. Бектұров атындағы Химия ғылымдары институты АҚ, Алматы, Қазақстан*

<sup>2</sup> *Әл-Фараби атындағы Қазақ ұлттық университеті, Алматы, Қазақстан*

**Түйіндеме.** *Кіріспе.* Органомодификацияланған сазды материалдар биологиялық белсенді заттардың бақыланатын бөлінуін қамтамасыз ету қабілетіне байланысты ауыл шаруашылығына арналған функционалды жүйелер ретінде үлкен қызығушылық тудырады. Жоғары меншікті беттік ауданы мен ион алмасу қабілеті бар табиғи бентонит органикалық өсу ынталандырғышын иммобилизациялау үшін тамаша матрица болып табылады. *Бұл жұмыстың мақсаты* – ерекше оксифосфонатты (Каз-6) қолдана отырып, бентонитті жұмсақ органоиммобилизациялаудың энергия тиімді әдісін жасау және оның бидайдың өсу ынталандырғышы ретіндегі тиімділігін бағалау. *Ұсынылған әдіс* бентонитті 40 °С температурада суда алдын ала диспергирлеуді, содан кейін 50 °С температурада кептіруді қамтиды. Дайындалған минералды матрица бөлме температурасында 10<sup>-2</sup>–10<sup>-6</sup> мас. % концентрация ауқымында диметил(4-гидрокси-1-(2-этоксипиридин-4-ил)фосфонаттың (Каз-6) сулы ерітінділерімен өңделді. Алынған композиттің құрылымы ИҚ-спектроскопия әдісімен сипатталды. Өзірленген тәсіл органикалық компоненттің айтарлықтай төмен концентрациясын қолдана отырып, белгілі аналогтармен салыстырғанда процестің температурасы мен ұзақтығын айтарлықтай төмендетуге мүмкіндік беретіні анықталды. Биологиялық сынақтар органоиммобилизацияланған бентониттің бидайға («Алмакен» және «Жеңіс» сорттары) қатысты айқын өсуді ынталандырушы белсенділікті көрсететінін дәлелдеді. Ең жоғары тиімділік 10<sup>-5</sup>–10<sup>-6</sup> мас. % концентрациясында байқалды, мұнда композит бақылау тобынан және еркін оксифосфонаттан асып түсті. Нәтижелер Каз-6-ны бентонитке иммобилизациялау белсенді компоненттің ұзақ әсер етуін және жоғары биоәтімділігін қамтамасыз ететінін көрсетеді. Өзірленген органоиммобилизацияланған композит ауыл шаруашылығын тұрақты дамыту үшін перспективалы өсу стимуляторы ретінде ұсынылуы мүмкін.

**Түйін сөздер:** бентонит, органоиммобилизация, оксифосфонат, иммобилизация, өсу ынталандырғышы, бидай, біртіндеп босату.

<i>Капар Баян Ерболқызы</i>	<i>Магистрант, Инженер</i>
<i>Жарқынбек Толғанай Еркінқызы</i>	<i>Техника және технология магистрі, кіші ғылыми қызметкер</i>
<i>Тюсюпова Бахыт Баймуратовна</i>	<i>Химия ғылымдарының кандидаты, қауымдастырылған профессор</i>
<i>Ибраимова Дана Мықты-Кереевна</i>	<i>Химия ғылымдарының кандидаты, аға оқытушы</i>
<i>Тажиебаева Сағдат Медербековна</i>	<i>Химия ғылымдарының докторы, профессор</i>
<i>Ю Валентина Константиновна</i>	<i>Химия ғылымдарының докторы, профессор</i>

## МЯГКАЯ ОРГАНОМОДИФИКАЦИЯ БЕНТОНИТА ОКСИФОСФОНАТОМ И ЕГО ПРИМЕНЕНИЕ КАК ПРОЛОНГИРОВАННОГО РОСТСТИМУЛЯТОРА

**Б.Е.Капар<sup>1,2</sup>, Т.Е.Жаркынбек<sup>1,2\*</sup>, Б.Б.Тюсюпова<sup>2</sup>, Д.М.-К.Ибраимова<sup>2</sup>,  
С.М.Тажибоева<sup>2</sup>, В.К.Ю<sup>1</sup>**

<sup>1</sup>АО Институт химических наук им. А.Б. Бектурова, Алматы, Казахстан

<sup>2</sup>Казахский национальный университет им. аль-Фараби, Алматы, Казахстан

**Резюме.** *Введение.* Органомодифицированные глинистые материалы представляют значительный интерес как функциональные системы для сельского хозяйства благодаря их способности к контролируемому высвобождению биологически активных веществ. Природный бентонит, обладающий высокой удельной поверхностью и ионообменной емкостью, является отличной матрицей для иммобилизации органических стимуляторов роста. *Целью* данной работы является разработка энергоэффективного метода мягкой органомодификации бентонита с использованием специфического оксифосфоната (Каз-6) и оценка его эффективности в качестве стимулятора роста пшеницы пролонгированного действия. *Предложенный метод* включает предварительное диспергирование бентонита в воде при 40°C с последующей сушкой при 50°C. Подготовленную минеральную матрицу обрабатывали водными растворами диметил(4-гидрокси-1-(2-этоксипиридин-4-ил)фосфоната (Каз-6) в диапазоне концентраций 10<sup>-2</sup>–10<sup>-6</sup> мас. % при комнатной температуре. Структура полученного композита охарактеризована методом ИК-спектроскопии. Установлено, что разработанный подход позволяет существенно снизить температуру и продолжительность процесса по сравнению с известными аналогами при использовании значительно меньших концентраций органического компонента. Биологические испытания показали, что органомодифицированный бентонит проявляет выраженную ростстимулирующую активность по отношению к пшенице (сорта «Алмакен» и «Женис»). Наиболее значимый эффект наблюдался при концентрациях 10<sup>-5</sup>–10<sup>-6</sup> мас. %, где композит превзошел показатели контроля и свободного оксифосфоната. Результаты указывают на то, что иммобилизация Каз-6 на бентоните обеспечивает пролонгированное действие и повышенную биодоступность активного компонента. Разработанный органоинеральный композит может быть рекомендован как перспективный стимулятор роста для устойчивого развития сельского хозяйства.

**Ключевые слова:** бентонит, органомодификация, оксифосфонат, иммобилизация, стимулятор роста, пшеница, пролонгированное высвобождение.

<i>Капар Баян Ерболқызы</i>	<i>Магистрант, инженер</i>
<i>Жаркынбек Толганай Еркінқызы</i>	<i>Магистр техники и технологии, младший научный сотрудник</i>
<i>Тюсюпова Бахыт Ибраимова</i>	<i>Кандидат химических наук, ассоциированный профессор</i>
<i>Дана Мыкты-Кереевна Баймуратовна</i>	<i>Кандидат химических наук, старший преподаватель</i>
<i>Тажибоева Сагдат Медербекевна</i>	<i>Доктор химических наук, профессор</i>
<i>Ю Валентина Константиновна</i>	<i>Доктор химических наук, профессор</i>

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