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RESEARCH ARTICLE

Concentration of heavy metal ions from aqueous media under dynamic conditions using a composite sorbent based on chitosan and silica

Victoria A. Gabrin[1,✉], **Tatiana E. Nikiforova**[1], **Vladimir A. Kozlov**[1], **Pavel B. Razgovorov**[2]
[1] *Ivanovo State University of Chemistry and Technology, Ivanovo, 153000 Russia*
[2] *Yaroslavl State Technical University, Yaroslavl, 150023 Russia*
✉ *Corresponding author, e-mail: gabrinvictoria@gmail.com*

Abstract

Objectives. The study set out to investigate the sorption, toxicological, and regeneration properties of a composite sorbent based on chitosan hydrogel and unsuspended silicon dioxide (chitosan–colloidal silica), which manifest themselves under dynamic conditions of purification of aqueous solutions, as a means of removing heavy metal ions.

Methods. The total dynamic exchange capacity of a chitosan–colloidal silica composite sorbent was evaluated under dynamic sorption conditions by passing solutions containing Zn(II), Cd(II), Cu(II), and Cr(III) ions having a concentration of 240–251 mg/L through a fixed sorption bed. The method for determining acute toxicity using daphnia (*Daphnia magna* Straus) is based on the direct calculation of the mortality of daphnia exposed to toxic substances contained in the test aqueous extract in comparison with a reference culture in samples that do not contain toxic substances. The regeneration ability of the sorbent was assessed by counting the number of sorption–desorption cycles using 0.1 M NaOH and 0.1 M $NaHCO_3$ eluents, as well as aqueous solutions of H_2O_2 (1 and 3%).

Results. The effectiveness of the chitosan–colloidal silica composite sorbent in the process of dynamic purification of aqueous media to remove Cu(II), Zn(II), Cd(II), and Cr(III) ions was established. After determining the times of ion breakthrough and saturation of the developed sorbent, its dynamic exchange capacity was calculated by processing the kinetic curves of sorption of heavy metal ions under dynamic conditions. The results of regeneration of the sorbent were presented in the context of the possibility of its reuse. It is shown that the sorbent can withstand up to five sorption–desorption cycles while maintaining a level copper cation extraction above 90%.

Conclusions. Analysis of the kinetic curves demonstrated that the driving force behind the removal of heavy metals from aqueous media by means of the obtained sorbent is the external diffusion mass transfer of ions from the mobile phase of the solution. Biotesting of samples showed that the developed chitosan-based sorbent does not have acute toxicity.

Keywords
heavy metals, adsorption, composites, chitosan, biotesting, regeneration

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НАУЧНАЯ СТАТЬЯ

Концентрирование ионов тяжелых металлов из водных сред в динамических условиях композиционным сорбентом на основе хитозана и диоксида кремния

В.А. Габрин[1,✉], Т.Е. Никифорова[1], В.А. Козлов[1], П.Б. Разговоров[2]
[1] *Ивановский государственный химико-технологический университет, Иваново, 153000 Россия*
[2] *Ярославский государственный технический университет, Ярославль, 155023 Россия*
✉ *Автор для переписки, e-mail: gabrinvictoria@gmail.com*

Аннотация

Цели. Изучение сорбционных, токсикологических и регенерационных свойств композиционного сорбента на основе гидрогеля хитозана и несуспендированного диоксида кремния («хитозан–коллоидный кремнезем»), проявляющихся в динамических условиях очистки водных растворов от ионов тяжелых металлов.

Методы. Полную динамическую обменную емкость композиционного сорбента «хитозан–коллоидный кремнезем» оценивали в условиях динамической сорбции, пропуская через неподвижный сорбционный слой растворы, содержащие ионы Zn(II), Cd(II), Cu(II) и Cr(III) с концентрацией 240–251 мг/л. Метод определения острой токсичности с использованием дафний (*Daphnia magna* Straus) основан на прямом счете процента смертности дафний при воздействии токсических веществ, присутствующих в исследуемой водной вытяжке, по сравнению с контрольной культурой в пробах, не содержащих токсических веществ. Оценку регенерационной способности сорбента определяли фиксированием количества циклов сорбции–десорбции с использованием элюентов — 0.1 M NaOH, 0.1 M $NaHCO_3$, а также водных растворов H_2O_2 (1 и 3%).

Результаты. Установлена эффективность работы композиционного сорбента «хитозан–коллоидный кремнезем» в процессе динамической очистки водных сред от ионов Cu(II), Zn(II), Cd(II) и Cr(III). Определены времена проскока ионов и насыщения разработанного сорбента и рассчитана его динамическая обменная емкость путем обработки кинетических кривых сорбции ионов тяжелых металлов, снятых при осуществлении сорбции в динамических условиях. Представлены результаты регенерации и возможность повторного использования сорбента, который способен выдерживать до пяти циклов сорбции–десорбции с сохранением степени извлечения катионов меди выше 90%.

Выводы. Анализ кинетических кривых показал, что движущей силой динамической очистки водных сред от тяжелых металлов полученным сорбентом является внешнедиффузионный массоперенос ионов из подвижной фазы раствора. Биотестирование образцов показало, что разработанный сорбент на основе хитозана не обладает острой токсичностью.

Ключевые слова
адсорбция, тяжелые металлы, хитозан, композиты, биотестирование, регенерация

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INTRODUCTION

The need to improve the quality of water, representing a major technological and life resource, continues to present challenges [1–3]. Current improvements in wastewater treatment methods are mainly achieved by improving previously introduced methods, including those based on ion exchange-, electrolysis-, membrane separation-, ultrafiltration-, reverse osmosis-, catalysis-, sedimentation-, coagulation- (flocculation), and adsorption approaches [4–6]. Due to its low energy intensity and high efficiency, the last of these methods is considered optimal for the removal of hazardous pollutants, including heavy metals [7–9]. Adsorption water treatment methods use a variety of materials: silica gel, activated carbon, resins, clays, and polymers. In terms of their economic feasibility and environmental significance, carbohydrate biopolymers (cellulose, alginate, and chitosan) have received increased attention as efficient alternative adsorbents over the past twenty years. This is mainly due to their key advantages—natural abundance, environmental friendliness, biodegradability, ease of modification, and relatively low production cost [10]. Among the abovementioned carbohydrate biopolymers, chitosan is the most environmentally friendly due to its improved target sorption characteristics [11].

The main advantages of chitosan-based adsorbents are their high absorption capacity for heavy and rare earth metal ions. However, a key disadvantage of chitosan is its insolubility in aqueous solutions with pH < 6.5. In this regard, the development of chitosan-containing sorbents that are stable in acidic media was studied in numerous works. Attempts to solve the problem include the use of crosslinking agents: glutaraldehyde, epichlorohydrin, ethylene glycol diglycidyl ether, etc. [12].

The possibility of using chitosan adsorbents to obtain products of any shape (in the form of powder, microspheres, fibers, or membranes) dramatically expands its potential for wastewater treatment and in modern medicine. Chitosan can be used as a matrix to obtain composite sorbents, representing relatively inexpensive and effective materials with a developed surface. The fillers of this matrix can be zeolites, diatomite, silica, titania, montmorillonite, cellulose, etc. Although the introduction of such fillers into the chitosan matrix does not always lead to a significant increase in sorption capacity, the development of the scientific foundations and technology for creating new composite chitosan-containing sorbents based on its structural and mechanical characteristics represents a very promising direction in the development of industrial ecology [1, 7, 13].

The present work set out to investigate the sorption, toxicological, and regeneration properties of a composite sorbent based on chitosan hydrogel and unsuspended silicon dioxide (“chitosan–colloidal silica”), manifested under dynamic conditions of purification of aqueous solutions, as a means of removing heavy metal ions.

EXPERIMENTAL

Materials

Chitosan (*Bioprogress*, Russia; degree of deacetylation, 88%; molar mass, 220 kDa); epichlorohydrin (*Sigma-Aldrich*, USA; >98.0%); colloidal silica (silicon dioxide) (*Ekokremnii*, Russia; Kovelos 35/05; r = 3–5 μm); sulfates of copper ($CuSO_4 \cdot 5H_2O$); zinc ($ZnSO_4 \cdot 7H_2O$); cadmium ($CdSO_4 \cdot 8H_2O$); chromium ($Cr_2(SO_4)_3 \cdot 6H_2O$); sodium hydroxide; acetic acid (50 vol %); sodium bicarbonate; hydrogen peroxide (aqueous) (*LenReaktiv*, Russia). These reagents were used without additional purification.

Along with sorbent regeneration efficiency and acute toxicity of aqueous extracts, the dynamic sorption characteristics of a chitosan–colloidal silica composite sorbent obtained according to the previously published procedure were studied [13]. The obtained physicochemical and structural-mechanical characteristics, as well as the static sorption of copper ions, agree with the previous results [13].

Equipment and research methods

The total dynamic exchange capacity (TDEC) of the chitosan–colloidal silica composite sorbent was assessed under dynamic sorption conditions by passing solutions containing Zn(II), Cd(II), Cu(II), and Cr(III) ions at concentrations of 240–251 mg·L^{-1} through a fixed sorption bed. In a dynamic experiment, the sorbent (0.2 g on chitosan basis) was placed in a laboratory column in the sorption plant (Fig. 1). Every 10 min, the metal salt solution passing through the column was collected to determine its cation concentration with a 210 VGP atomic absorption spectrometer (*Buck Scientific*, USA).

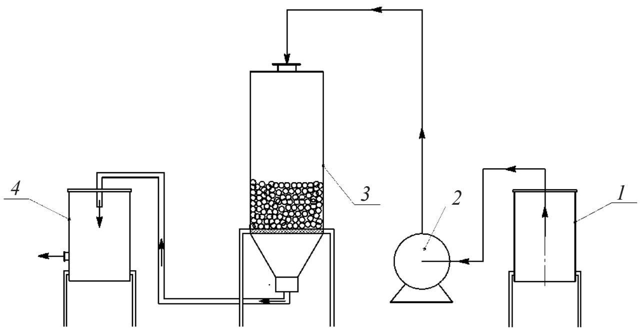

Fig. 1. Plant for studying dynamic sorption characteristics: (*1*) container with metal salt solution (electrolyte), (*2*) pump, (*3*) fixed-bed column with a sorbent, and (*4*) receiving container

The TDEC value was determined by analyzing the obtained dynamic curves by calculating the area above the output sorption curve:

$$S = C_{\text{in}} \times \Delta\tau_1 + \frac{C_{\text{in}} \times \Delta\tau_2}{2}, \quad (1)$$

$$\text{TDEC} = \frac{S \times Q}{m_{\text{sorb}}}, \quad (2)$$

where Q is the flow rate of the solution, L·s^{-1}; m_{sorb} is the mass of the sorbent placed in the ion-exchange column, g; $\Delta\tau_1$ and $\Delta\tau_2$ are the times of breakthrough and saturation, respectively, s; and C_{in} is the concentration of copper ions at the inlet, mg·L^{-1}.

Determination of the acute toxicity of the sorption material

The acute toxicity of the aqueous extract was determined using daphnia (*Daphnia magna* Straus) as a test object.

The method is based on direct calculation of the mortality percentage of daphnia exposed to toxic substances contained in the studied aqueous extract in comparison with a reference culture in the form of samples that do not contain toxic substances.

The acute toxicity A (%) of the aqueous extract was determined by the formula

$$A = \frac{X_{ref} - X_{test}}{X_{ref}} \times 100, \quad (3)$$

where X_{ref} is the number of surviving daphnia in the reference culture and X_{test} is the number of surviving daphnia in the water extract.

The preparation of cultivation water and dilutions of aqueous extracts for biotesting, and the biotesting itself were carried out according to the published method [14] for water samples obtained after contact with the studied modified sorbent. The biotesting was performed as follows. After growing daphnia cultures in a climatostat maintaining a 12-h photoperiod at room temperature (25 ± 0.1°C), ten age-synchronized daphnia cultures were placed for 1 day into each of the test tubes with test solutions pre-aerated for 30 min. The experimental conditions corresponded to the conditions for daphnia cultivation: T = (20 ± 2)°C, pH 7.0–8.2, and time 48 h.

Determination of the number of sorption–desorption cycles

In order to evaluate the possibility of reusing a composite sorbent without losing its effectiveness against heavy metal ions, desorption-based approaches can be used. Desorption of heavy metal ions was carried out using the suitable eluents 0.1 M NaOH and 0.1 M $NaHCO_3$, as well as aqueous solutions of H_2O_2 (1 and 3%). The spent sorbent containing 0.1 g of dry chitosan was placed in 10 mL of a desorbing eluent solution and kept for 10 min. Following desorption, the chitosan–colloidal silica sorbent was washed with distilled water. When determining the number of sorption–desorption cycles, the sorbent regenerated in this way was reused to extract Cu(II) ions.

Regeneration efficiency was selected as a characteristic parameter of the recovery of spent sorbent. The regeneration efficiency RE (%) of the sorbent was calculated by the formula

$$RE = \frac{A_r}{A_0} \times 100, \quad (4)$$

where A_r and A_0 are the sorption after regeneration and the initial sorption of the sorbent, mg·g^{-1}, respectively.

RESULTS AND DISCUSSION

The chitosan–colloidal silica composite sorbent was tested under dynamic conditions of sorption of various heavy metals (Fig. 2). The ion breakthrough time and sorbent saturation time were determined along with the TDEC (Table 1). In the experiment, the height of the packed sorbent bed ($5 \cdot 10^{-2}$ m) and the diameter of the column ($4 \cdot 10^{-2}$ m) remained unchanged. When calculating the total dynamic exchange capacity, the mass of the sorbent and the time consumption of the model wastewater solution were taken into account.

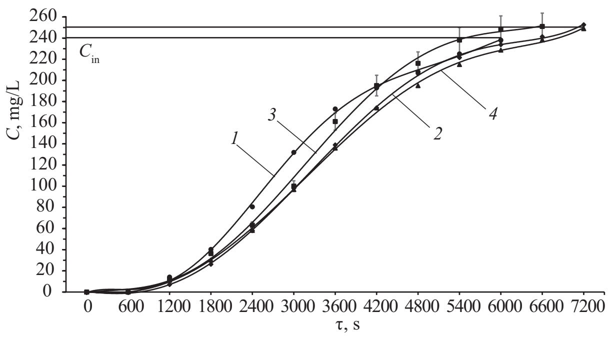

Fig. 2. Kinetic curves of sorption of (*1*) Cu(II), (*2*) Cd(II), (*3*) Zn(II), and (*4*) Cr(III) under dynamic conditions by the chitosan–colloidal silica sorbent at pH 5.9, T = 298 K, m_{sorb} = 0.2 g, and Q = 0.15 L·h^{-1}

The shape of the kinetic curves of sorption under dynamic conditions suggests that the sorption of Cu(II), Cd(II), Zn(II), and Cr(III) ions predominantly occurs by the external diffusion mechanism of mass transfer of ions

Table 1. TDEC of the sorbent and variables determined under dynamic conditions

Metal	C_{in}, mg·L^{-1}	m_{sorb}, h	τ_1, s	τ_2, s	TDEC	
					mg·g^{-1}	mol·kg^{-1}
Cu(II)	240	0.2	900	5400	180	2.8
Cd(II)	250		1050	6200	216	2.0
Zn(II)	251		1000	5800	204	3.0
Cr(III)	248		1100	6300	219	4.2

Table 2. Acute toxicity of aqueous extract after contact with the chitosan–colloidal silica sorbent

Sorbent	Dilution ratio (aqueous extract content), %	Number of surviving daphnia		Mortality of daphnia in test, %
		Test	Reference	
Biotesting time: 48 h				
Chitosan–silica composite	10 (10)	10	10	0
	3 (33.3)	10	10	0
	1 (89)	10	10	0

from the mobile phase of the solution to the fixed sorbent bed. This may be due to the value of the crystallographic radius of the ion. Under both dynamic and static sorption conditions, sorbate–sorbate interactions are not excluded, leading to the formation of electrostatically bound ionic clusters. In a constant flow of liquid, such clusters do not significantly penetrate into the bulk phase through pore channels, but are mainly retained on the surface [15].

Further dynamic experiments will be aimed at identifying optimal process parameters and increasing the efficiency of using the chitosan–colloidal silica sorbent. Taking into account the increased sorption (sorption capacity) of the resulting material under static conditions, this primarily concerns the dosage of the sorbent and the dimensions of the column (8–10 mol/kg) [13].

Biotesting of the aqueous extract demonstrated the absence of an acute toxic effect at $A \leq 10\%$ (Table 2), whereas in the case of $A \geq 50\%$, the previously shown acute toxic effect on living organisms is confirmed [14].

The biotesting of the water extract showed that, in the tested water after 48 h, the mortality of daphnia was less than 10%, which indicates the nontoxicity of the sorbent.

Figure 3 illustrates the change in the efficiency of regeneration of the chitosan–colloidal silica sorbent with an increasing number of sorption–desorption cycles using different eluents.

As noted earlier, a very important characteristic of sorbents is their regeneration ability. The developed chitosan–colloidal silica sorbent can maintain its efficient extraction of copper ions after five sorption–desorption cycles. The most efficient eluent is an H_2O_2 solution; here, the efficiency of using a 1% solution is comparable to that of a 3% solution. After five sorption–desorption cycles, the sorption capacity can be restored by more than 90%. When using sodium bicarbonate, the reduction

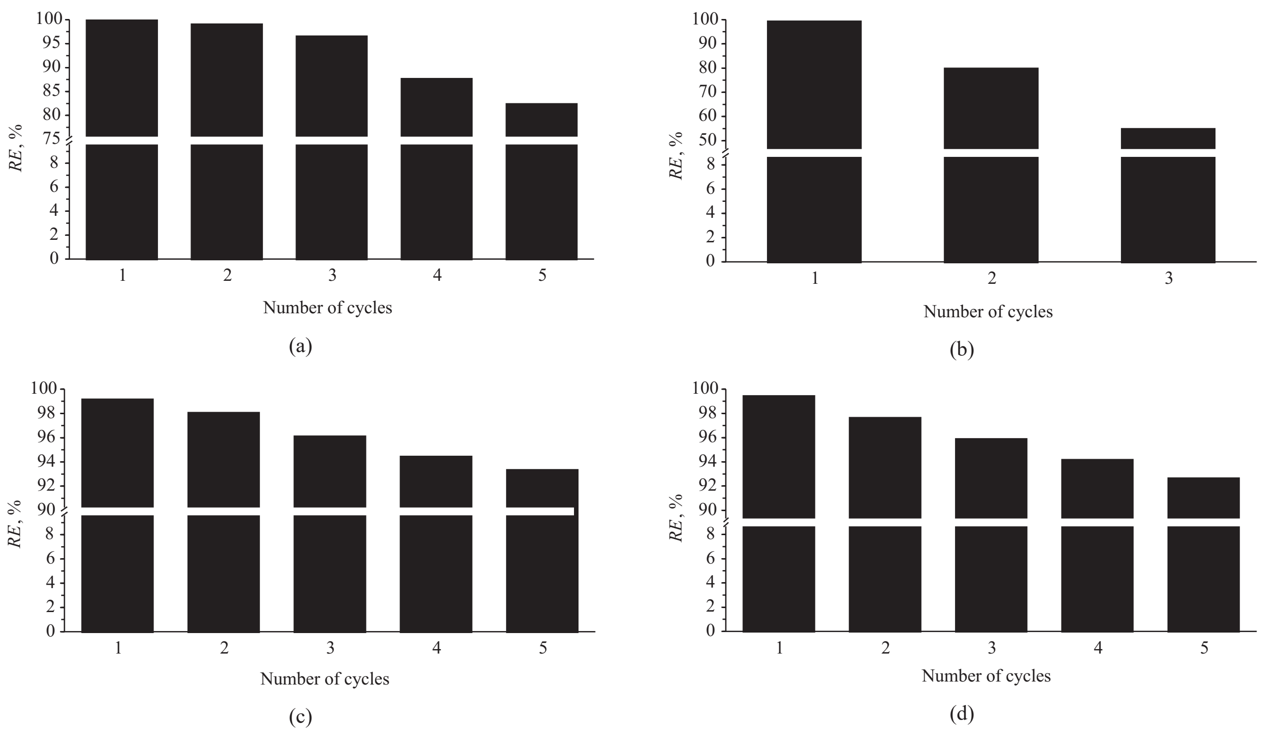

Fig. 3. Change in the efficiency of regeneration of the chitosan–colloidal silica sorbent with increasing number of sorption–desorption cycles. Eluents: (a) 0.1 M NaOH, (b) 0.1 M $NaHCO_3$, (c) 3% H_2O_2, and (d) 1% H_2O_2

Table 3. Effect of the contact time of the spent sorbent with the eluent on the efficiency of sorbent regeneration and its mass loss in the first sorption–desorption cycle

0.1 M NaOH			0.1 M $NaHCO_3$			3% H_2O_2			1% H_2O_2		
Contact time, min	Sorbent mass loss, %	*RE*, %	Contact time, min	Sorbent mass loss, %	*RE*, %	Contact time, min	Sorbent mass loss, %	*RE*, %	Contact time, min	Sorbent mass loss, %	RE, %
$V_{solution}$ = 10 mL											
1	0	15	1	0	10	1	0	2	1	0	1
3	1	45	3	0	30	3	0	20	3	0	25
5	2	70	5	0	59	5	0	80	5	0	86
10	5	97	10	0.5	92	10	0	97	10	0	98
15	8	96	15	2	93	15	1	95	15	0.5	97

in regeneration efficiency to 47% after three cycles makes further testing impractical. The optimal contact time of the sorbent and eluent was determined to be 10 min (Table 3).

A further increase in the contact time between the sorbent and the eluent did not provide more efficient regeneration. In addition, an increase in regeneration time was accompanied by a significant (up to 8% in the first cycle) sorbent mass loss in comparison with the initial mass of the sorbent.

Thus, crosslinked chitosan granules bulk-modified with colloidal silica, which have valuable properties in terms of permitting their reuse, can serve as an economical and effective sorbent for the extraction of heavy metal ions from aqueous media.

CONCLUSIONS

Dynamic parameters of the extraction of heavy metal ions by the chitosan–colloidal silica composite sorbent were obtained. It is shown that the column sorption of Cu(II), Cd(II), Zn(II), and Cr(III) ions from aqueous media predominately occurs by the external diffusion mechanism of mass transfer of ions from the mobile phase of the solution to the fixed bed of the sorbent. The conditions for the regeneration of the spent composite sorbent are identified. It is determined that the use of a 1% hydrogen peroxide solution as a reducing eluent allows the obtained sorbent to be used in five sorption–desorption cycles while maintaining the degree of extraction of metal ions above 90%. The biotesting method established the absence of acute toxicity for living organisms of the aqueous extract of the solution in contact with the sorbent. In the future, the developed sorbent based on chitosan and silicon dioxide will be tested under conditions of purification of aqueous media similar to industrial conditions in order to evaluate the quality of purification of aqueous solutions for removing heavy metal ions used in electroplating and hydrocarbon processing (oil refining). This will help to improve effectiveness of natural water protection systems and reduce anthropogenic impacts on the environment.

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Authors' contributions

V.A. Gabrin—formulating the aim of the study, planning and conducting experiments, processing experimental data, and writing the text of the article.

T.E. Nikiforova—correcting the aim of the study, research methodology, discussion of the results, and editing the content of the article.

V.A. Kozlov—correcting the research methodology, scientific consulting.

P.B. Razgovorov—general management and discussion of the results.

The authors declare no conflicts of interest.

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About the authors

Victoria A. Gabrin, Postgraduate Student, Department of Food Technology and Biotechnology, Ivanovo State University of Chemistry and Technology (ISUCT) (7, Sheremetevskii pr., Ivanovo, 153000, Russia). E-mail: gabrinvictoria@gmail.com. Scopus Author ID 58398403100, RSCI SPIN-code 6197-2268, https://orcid.org/0000-0002-1712-9121

Tatiana E. Nikiforova, Dr. Sci. (Chem.), Associate Professor, Department of Food Technology and Biotechnology, Ivanovo State University of Chemistry and Technology (ISUCT) (7, Sheremetevskii pr., Ivanovo, 153000, Russia). E-mail: tatianaenik@mail.ru. Scopus Author ID 7006694263, ResearcherID N-1026-2017, RSCI SPIN-code 4080-7735, https://orcid.org/0000-0002-0817-9258

Vladimir A. Kozlov, Dr. Sci. (Chem.), Full Professor, Department of Chemistry and Technology of Macromolecular Compounds, Ivanovo State University of Chemistry and Technology (ISUCT) (7, Sheremetevskii pr., Ivanovo, 153000, Russia). E-mail: kozlov@isuct.ru. Scopus Author ID 57196498026, RSCI SPIN-code 4805-4551, https://orcid.org/0000-0002-1034-4339

Pavel B. Razgovorov, Dr. Sci. (Eng.), Professor, Head of the Department for the Organization of Research and Intellectual Activities, Yaroslavl State Technical University (YSTU) (88, Moskovskii pr., Yaroslavl, 150023, Russia). E-mail: razgovorovpb@ystu.ru. Scopus Author ID 17344647700, RSCI SPIN-code 2735-1209, https://orcid.org/0000-0003-0514-5770

Об авторах

Габрин Виктория Александровна, аспирант кафедры технологии пищевых продуктов и биотехнологии, ФГБОУ ВО «Ивановский государственный химико-технологический университет» (ИГХТУ) (153000, Россия, г. Иваново, Шереметевский пр-т, д. 7). E-mail: gabrinvictoria@gmail.com. Scopus Author ID 58398403100, SPIN-код РИНЦ 6197-2268, https://orcid.org/0000-0002-1712-9121

Никифорова Татьяна Евгеньевна, д.х.н., доцент кафедры технологии пищевых продуктов и биотехнологии ФГБОУ ВО «Ивановский государственный химико-технологический университет» (ИГХТУ) (153000, Россия, г. Иваново, Шереметевский пр-т, д. 7). E-mail: tatianaenik@mail.ru. Scopus Author ID 7006694263, ResearcherID N-1026-2017, SPIN-код РИНЦ 4080-7735, https://orcid.org/0000-0002-0817-9258

Козлов Владимир Александрович, д.х.н., профессор кафедры химии и технологии высокомолекулярных соединений ФГБОУ ВО «Ивановский государственный химико-технологический университет» (ИГХТУ) (153000, Россия, г. Иваново, Шереметевский пр-т, д. 7). E-mail: kozlov@isuct.ru. Scopus Author ID 57196498026, SPIN-код РИНЦ 4805-4551, https://orcid.org/0000-0002-1034-4339

Разговоров Павел Борисович, д.т.н., профессор, начальник управления организации научно-исследовательской и интеллектуальной деятельности ФГБОУ ВО «Ярославский государственный технический университет» (ЯГТУ) (150023, Россия, г. Ярославль, Московский пр-т, д. 88). E-mail: razgovorovpb@ystu.ru. Scopus Author ID 17344647700, SPIN-код РИНЦ 2735-1209, https://orcid.org/0000-0003-0514-5770

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