SYNTHESIS AND PROCESSING OF POLYMERS AND POLYMERIC COMPOSITES

СИНТЕЗ И ПЕРЕРАБОТКА ПОЛИМЕРОВ И КОМПОЗИТОВ НА ИХ ОСНОВЕ

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RESEARCH ARTICLE

High-performance slow-curing polyurea compositions

Sergei V. Romanov[1], Olga A. Botvinova[1], Evgenii A. Timakov[2], Dariya A. Rashchupkina[2], Yuri T. Panov[2],✉

[1] *Elast PU Production Company, Vladimir, 600026 Russia*
[2] *Alexander and Nikolay Stoletovs Vladimir State University, Vladimir, 600014 Russia*
✉ *Corresponding author, e-mail: tpp_vlgu@mail.ru*

Abstract

Objectives. *To improve the technology for obtaining polymer spray coatings based on polycarbodiimides (polyureas) by studying changes in the process and operational parameters due to the introduction of aspartic acid derivatives (AADs) into the composition.*
Methods. *The process of the production of sprayed and contact polyureas involves a number of difficulties, not least in terms of the cost of the components and high-pressure equipment. For this reason, mathematical modeling was used to optimize experimental design. The curing time of the composition was measured under conditions simulated to be close to actual. After thermostating and mixing Components A and B in predetermined ratios, the gelation time was measured to represent the curing time of the composition. The hardness of the material was determined by the Shore method according to GOST 24621-91. Tensile strength and relative elongation were determined according to a standard method (GOST 30436-96).*
Results. *The effect of three AADs on the properties of the finished polyurea was studied. It was found that the introduction of two of them (AAD-1 and AAD-2) into polyurea in an amount of up to 40 wt % produces slow-curing (>250 s) polyureas capable of manual application. The finished products have physical properties on par with machine-poured materials (breaking strength >73 MPa; tensile strength >23 MPa; elongation >500%). Compiled regression equations were used to construct graphs of equal levels showing the possible areas of directed modification of the studied compositions.*

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Conclusions. *AAD can be used as a modifying component for polyurea systems to obtain slow-curing polyureas with high performance properties, which can be purposefully controlled by mathematical modeling. The resulting products have commercial value due to their combination of valuable physical and mechanical properties.*

Keywords: *polyurea, curing time, polymer coatings, physical and mechanical properties, mathematical modeling*

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НАУЧНАЯ СТАТЬЯ

Медленные полимочевинные композиции с высокими эксплуатационными свойствами

С.В. Романов[1], О.А. Ботвинова[1], Е.А. Тимаков[2], Д.А. Ращупкина[2], Ю.Т. Панов[2,✉]

[1]Производственная компания «Эласт-ПУ», Владимир, 600026 Россия
[2]Владимирский государственный университет им. Александра Григорьевича и Николая Григорьевича Столетовых, Владимир, 600014 Россия
✉Автор для переписки, e-mail: tpp_vlgu@mail.ru

Аннотация

Цели. *Совершенствование технологии получения полимерных напыляемых покрытий на основе поликарбодиимидов (полимочевин), путем изучения закономерностей изменения технологических и эксплуатационных свойств при введении в состав композиции производных аспарагиновой кислоты (ПАК).*
Методы. *Процесс производства напыляемых и контактных полимочевин сопряжен с рядом трудностей, главной из которых является стоимость используемых компонентов и работа с высокопроизводительным оборудованием высокого давления, поэтому авторами было предложено использовать метод математического моделирования для оценки оптимального плана проведения эксперимента. Измерение времени жизни композиции проводили в условиях, приближенных к реальным условиям нанесения. Компоненты А и Б термостатировали, смешивали в заданных соотношениях. Затем замеряли время гелеобразования, которое считали временем жизни композиции. Твердость материала определяли по методу Шора по ГОСТ 24621-91. Прочность и относительное удлинение при растяжении определяли по стандартной методике (ГОСТ 30436-96).*

Результаты. *Исследовано влияние трех ПАК на свойства готовой полимочевины и показано, что введение в состав полимочевины двух из них (ПАК1 и ПАК2) в количестве до 40 мас. % дает возможность получить полимочевины со временем жизни >250 с, что позволяет перевести их в разряд «медленных», то есть способных наноситься вручную. Готовые продукты обладают физическими свойствами, аналогичными материалам, полученным методом машинной заливки (прочностью на разрыв >73 МПа, прочностью при растяжении >23 МПа и относительным удлинением >500%). Составлены уравнения регрессии, на основании которых построены графики равных уровней, показывающие возможные области направленной модификации исследуемых композиций.*

Выводы. *Применение ПАК в качестве модифицирующего компонента для полимочевинных систем позволяет получить «медленные» полимочевины с высокими эксплуатационными свойствами, которые можно целенаправленно регулировать, используя метод математического моделирования. Помимо прочего, полученные продукты несут в себе коммерческую ценность, так как обладают совокупностью нужных физико-механических свойств.*

Ключевые слова: *полимочевина, время жизни, полимерные покрытия, физико-механические свойства, математическое моделирование*

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INTRODUCTION

In order to compete in the production of high-performance finished coatings used as construction and finishing materials, it is necessary to combine operational simplicity with process rapidity. Polyurea coatings are widely used as electrical insulating, lining, and anti-corrosion materials. However, such coatings require the use of expensive high-pressure equipment operated by highly qualified personnel. While these difficulties do not present a major hurdle when applying coatings to large surfaces, they quickly become insurmountable if it is necessary to apply coatings to small surfaces, e.g., during repair work. For this reason, there is a demand for compositions having a curing time of more than 5 min, which can be manually mixed and applied. Preliminary experiments showed that the known methods of reducing the reaction rate (increasing the curing time of the system) can be used only with the simultaneous reduction in the performance properties of the finished coatings [1].

The use of aspartic acid derivatives (AADs) as the amine component for polyurea coatings has recently been proposed [2–4]. Thus, the present work set out to study the possibility of obtaining slow-curing high-performance AAD-based polyurea elastomers.

EXPERIMENTAL

Many studies have shown the potential of AADs as modifiers for the synthesis of polymers used to improve the physical and mechanical properties of finished products. Currently, a large number of AADs are produced on an industrial scale. This work used AADs synthesized by the interaction of unsaturated bisimides with aliphatic and aromatic diamines. Figure 1 and Table 1 present the AADs used in this work along with their general formula and reactivity.

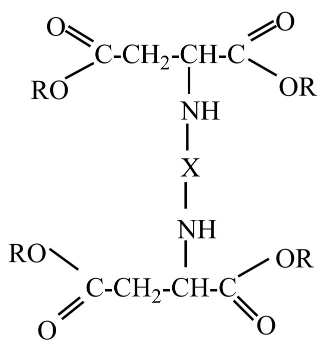
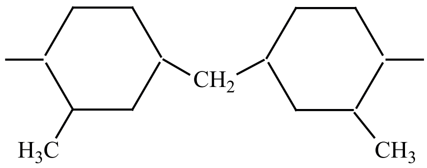
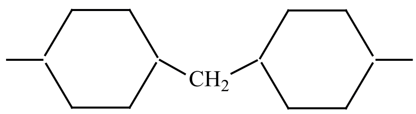
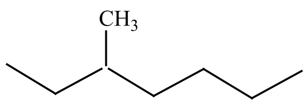


Fig. 1. General formula of an aspartic acid derivative (AAD).

As shown in Fig. 2, AADs react with isocyanates to form polyurea, which does not contain hydroxyl-containing components [3]:

The main components of the polyurethane–polyurea system are hydroxyl- and amine-containing substances (e.g., polyols and polyetheramines), which are collectively called component A of the system. Component B of the system comprises substances belonging to the class of isocyanates, which are responsible for the reaction of urethane formation.

Table 1. Structure of the main chain of AADs and their reactivity

Notation	Structure of main chain (X in Fig. 1)	Gelation time, h
AAD 1	Methylenebis(2-methylcyclohexane-4,1-diyl) 	8
AAD 2	Methylenedicyclohexane-4,1-diyl 	1
AAD 3	2-Methylpentane-1,5-diyl 	0.1

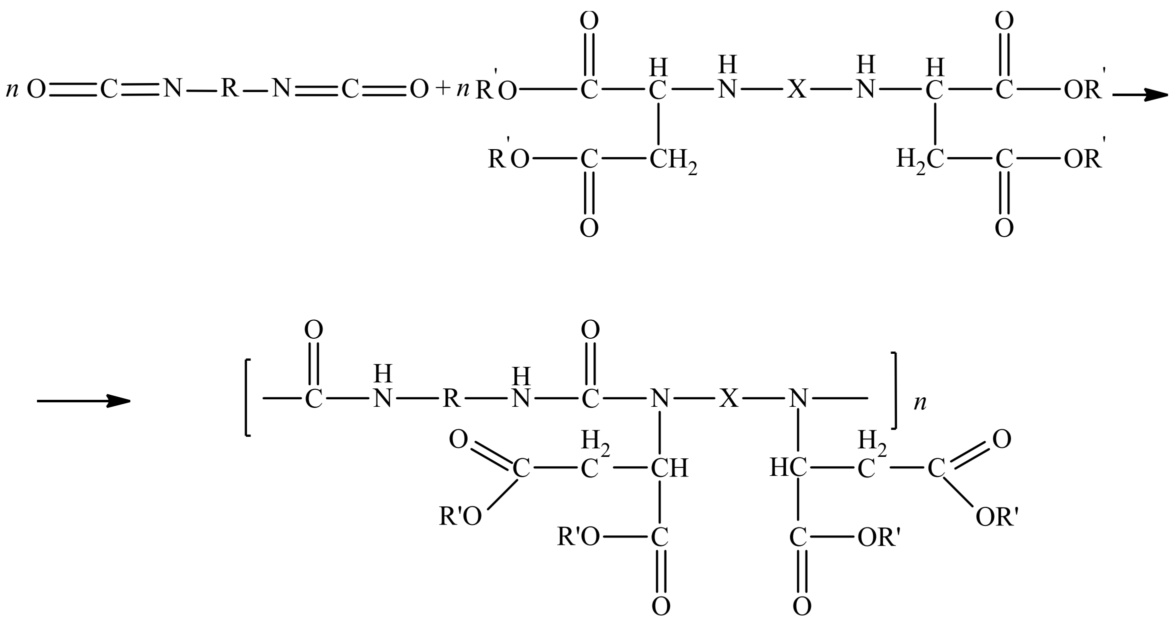


Fig. 2. Reaction to produce polyurea from AADs [3].

In the experiment, AADs were added to component A of the system in different ratios, provided that the index of the system would be in the range of 1.1–1.2.

The curing time of the composition was measured under conditions similar to the actual application conditions. Components A and B were thermostated and mixed in specified ratios. Then, the gelation time was measured, which represented the curing time of the composition. The hardness of the material was determined using the Shore method according to GOST 24621-91[1] with a TVR-A durometer (*VOSTOK-7*, Russia). Strength and relative tensile elongation were determined according to a standard method (GOST 11262-2017[2]) with an RKM 2.1 tensile testing machine (*Etalon-profit*, Russia). Table 2 presents the curing time and main performance properties of polyureas based on various AADs.

Table 2 shows that the addition of AAD-1 and AAD-2 makes the compositions capable of being applied manually. Moreover, the coatings based on AAD-2 are comparable to classical polyurea in hardness, and the coatings based on AAD-1 are superior to classical ones in elasticity. The addition of AADs in an amount of more than 40 wt % is unjustified since, in this case, the curing time exceeds several times the values convenient for work with them. Because of this, the performance characteristics of the finished coating are reduced, and the cost of the composition increases. In order to improve the properties of polyurea coatings, it was proposed to combine the two types of AADs.

Since studying the effect of the initial components in a polyurea composition is known to involve complex factors and expensive equipment operating at high pressure for long experimental times [5], it is most convenient to use mathematical modeling when taking such factors into account. While there are many methods of mathematical analysis for processing experimental data, the active experiment approach is useful for obtaining accurate data due to the possibility of processing several input factors simultaneously [6]. The active experiment approach was used in the present work to reduce the number of necessary experiments and visually represent the results of the analysis for further design of the synthesis of products that meet the specified requirements. Processing of experimental data to obtain a mathematical model was carried out the methods of classical regression and correlation analyses [7, 8].

The regression analysis method can be used if the following conditions are met [6, 8]:

1) the observation results are independent, normally distributed random variables;

2) the input variables are measured with a small error in comparison with the error in determining random variables and are nonrandom;

Table 2. Properties of coatings based on polyurea in the presence of various AAD

Notation	AAD content, wt %	Curing time, s	Shore A hardness, units	Tensile strength, MPa	Relative extension, %
AAD 1	20	850	65	18.5	580
	40	3600	58	15.0	615
AAD 2	20	250	75	23.4	531
	40	300	73	26.1	505
AAD 3	20	25	85	25.8	520
	40	50	92	26.5	390
Without AAD	0	10	78	22.1	550

[1] GOST 24621-91. State Standard of the USSR. Plastics and ebonite. Determination of indentation hardness by means of a durometer (Shore hardness). Moscow: Izdatelstvo standartov; 1992. URL: https://progost.com/gost/001.083.080/gost-24621-91/. Accessed November 08, 2023.

[2] GOST 11262-2017. Interstate Standard. Plastics. Tensile test method. Moscow: Standartinform; 2018. URL: https://docs.cntd.ru/document/1200158280. Accessed November 08, 2023.

3) the estimates of the variances of input parameters obtained under the same conditions are homogeneous.

To find analytical dependencies between the main performance characteristics (abrasion, tensile strength, relative elongation) and the contents of components of the composition, the experimental data were processed by the least squares method.

In the work, the mathematical description was made using a complete square polynomial

$$Y = b_0 + b_1 \cdot x_1 + b_2 \cdot x_2 + b_3 \cdot x_1 \cdot x_2 + b_4 \cdot x_1^2 + b_5 \cdot x_2^2,$$

where b_0, b_1, b_2, b_3, b_4, and b_5 are regression coefficients; x_1 and x_2 are dimensionless input variables calculated by the equation

$$x_j = \frac{X_j - X_{0j}}{h_j},$$

where x_j is the dimensionless jth input variable; X_j is the dimensional jth input variable; h_j is the variation range of the jth input variable, which is calculated by the equation

$$h_j = \frac{X_j^{\max} - X_j^{\min}}{2};$$

X_{0j} is the average value of the dimensional jth input variable, which is calculated by the equation:

$$X_{0j} = \frac{X_j^{\max} + X_j^{\min}}{2},$$

where $X_j^{\max}$ and $X_j^{\min}$ are the maximum and minimum values of the dimensional jth input variable, respectively.

In the work, a two-factor experiment was carried out: X_1 is the content of AAD-1 and X_2 is the content of free NCO groups, %. Table 3 presents the values of these factors for all levels of variation.

RESULTS AND DISCUSSION

The optimal design of experiment was developed using the MATLAB software (*MathWorks*, USA)[3]. Table 4 presents the extended design matrix and the experimental values of the properties of the composition.

To characterize these dependencies, a MATLAB program was developed, whose input data set comprises:

1) extended experimental design matrix, the dimension of which is determined by the number of input variables and the order of the regression equation (Table 4);

2) experimental values of output variables in accordance with the design matrix, which is written into the program in the form of a column vector;

3) ranges for each input variable.

The input data block comprises:

1) calculated values of the regression coefficients of the required equation;

2) table of comparison of experimental and calculated data using the obtained regression equation;

3) variance of the relative mean of the output variable;

Table 3. Input data for calculating the regression equation

Input variables / Levels of variation	Input factors			
	AAD 1 content in component A		Fraction of NCO-groups, %	
	X_1, wt %	x_1, dimensionless units	X_2, %	x_2, dimensionless units
Upper level	100	+1	15	+1
Lower level	0	−1	10	−1
Zero level	50	0	12.5	0
Variation step, h	50	–	2.5	–

[3] MATLAB. https://www.mathworks.com/. MathWorks, USA. Accessed November 08, 2023.

Table 4. Extended planning matrix in coded variables and property values

No. of experiment	x_0	x_1	x_2	$x_1 \times x_2$	x_2^2	x_1^2	Curing time, s	Tensile strength, MPa	Elongation at break, %
1	1	−1	1	−1	1	1	8	22	550
2	1	−1	−1	1	1	1	16	13	1050
3	1	−1	0	0	1	0	14	15	850
4	1	1	1	1	1	1	150	20	580
5	1	1	−1	−1	1	1	280	17	700
6	1	1	0	0	1	0	240	19	650
7	1	0	1	0	0	1	90	19	620
8	1	0	−1	0	0	1	260	16	720
9	1	0	0	0	0	0	210	17	680

4) residual variance;

5) Fisher's exact test;

6) graphic material, including the response surface obtained from the found regression equation and the isoline map.

The regression coefficients were calculated using the developed MATLAB program. The obtained regression equations are the following:

– for curing time,

$$Y1 = 200.44 + 105.33x_1 - 51.33x_2 + \\ + 30.5x_1x_2 + 68.67x_1^2 + 20.67x_2^2;$$

– for tensile strength,

$$Y2 = 16.78 + 1.0x_1 + 2.5x_2 + \\ + 1.5x_1x_2 + 0.33x_1^2 + 0.83x_2^2;$$

– for relative elongation at break,

$$Y3 = 688.89 - 86.67x_1 + 120x_2 + \\ + 95x_1x_2 + 56.67x_1^2 - 23.33x_2^2.$$

The adequacy of the equations was assessed using the Fisher's exact test. The adequacy estimate F_p was assessed with respect to the mean $S_{\bar{y}}^2$ of the output variable and the residual variance S_res^2:

$$F_\text{p} = \frac{S_{\bar{y}}^2}{S_\text{res}^2},$$

where the residual variance was found by the equation

$$S_\text{res}^2 = \frac{\sum_{i=1}^{N}(y_i - \bar{y}_i)^2}{N - K}.$$

The variance of the mean of the output variable was calculated by the formula

$$S_{\bar{y}}^2 = \frac{\sum_{i=1}^{N}(y_i - \bar{y})^2}{N - 1},$$

where N is the total number of experiments, K is the number of significant coefficients in the regression equation, and $\bar{y}$ are the means.

The calculated values of the Fisher's exact test are the following: strength is 191.1; relative elongation is 54.55; curing time is 32.15. The tabulated value

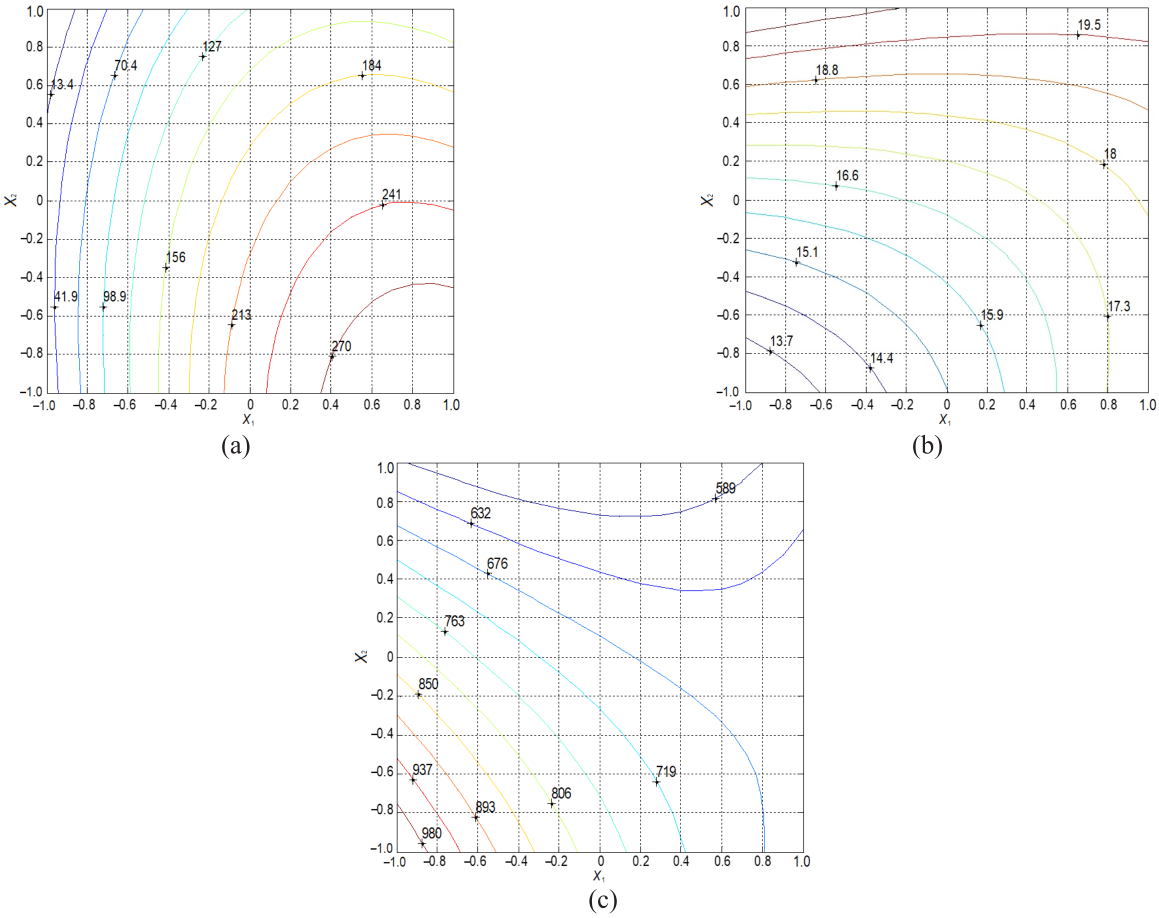


Fig. 3. Isoline maps of the main performance characteristics of polyurea coatings based on a mixture of AAD-1 and AAD-2: (a) for curing time, (b) for tensile strength, and (c) for relative elongation.

of Fisher's exact test under the corresponding conditions is 8.8 [7]. As can be seen, the calculated values of the Fisher's exact test exceed the tabulated value; consequently, the equations adequately describe the process.

Figure 3 presents the isoline map of the constructed regression functions for the selected factors.

The isoline maps depicted in Fig. 3 demonstrate the possibility of optimizing the process under study. The wide variation in the studied properties allows the composition developers to vary the performance parameters of the finished product depending on customer requirements. Importantly, throughout the studied range of properties, coatings of satisfactory quality were obtained.

CONCLUSIONS

This study showed that the use of AADs can increase the curing time to the 5 min required by the consumer. By introducing two of them (AAD-1 and AAD-2) in an amount of up to 40 wt %, it is possible to obtain polyurea compositions with a curing time >250 s, a breaking strength >73 MPa, tensile strength >23 MPa, and a relative elongation >500%.

According to the two-factor experimental design, regression equations were compiled for each of the studied properties. The adequacy of the equations was determined by the Fisher's exact test. Isoline maps constructed on the basis of the equations are used to form to model polyurea compositions over a wide range of performance properties in accordance with customer requirements.

Authors' contributions

S.V. Romanov – research idea, resource provision;

O.A. Botvinova – conducting research, analysis of the results;

E.A. Timakov – writing the text of the article, analysis of the results;

D.A. Rashchupkina – writing the text of the article;

Yu.T. Panov – supervision, scientific editing.

The authors declare no conflicts of interest.

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About the authors:

Sergei V. Romanov, Cand. Sci. (Eng.), Director of the Elast PU Production Company (21a, Gastello ul., Vladimir, 600026, Russia). E-mail: romanov-elastpu@mail.ru. Scopus Author ID 55809569400, RSCI SPIN-code 9952-4171, https://orcid.org/0000-0002-5152-4678

Olga A. Botvinova, Cand. Sci. (Eng.), Head of Research Laboratory, Elast PU Production Company (21a, Gastello ul., Vladimir, 600026, Russia). E-mail: ermolaeva_olya@inbox.ru. Scopus Author ID 56907022800, RSCI SPIN-code 3933-6884, https://orcid.org/0000-0003-0217-8446

Evgenii A. Timakov, Senior Lecturer, Chemical Technologies Department, Alexander and Nikolay Stoletovs Vladimir State University (87, Gor’kogo ul., Vladimir, 600026, Russia). E-mail: okakie@yandex.ru. Scopus Author ID 57219018403, RSCI SPIN-code 3178-1477, https://orcid.org/0000-0003-3312-0810

Dariya A. Rashchupkina, Master Student, Chemical Technologies Department, Alexander and Nikolay Stoletovs Vladimir State University (87, Gor'kogo ul., Vladimir, 600026, Russia). E-mail: dashulka010220002016@gmail.com. https://orcid.org/0000-0001-7724-6218

Yuri T. Panov, Dr. Sci. (Eng.), Professor, Head of the Chemical Technologies Department, Alexander and Nikolay Stoletovs Vladimir State University (87, Gor'kogo ul., Vladimir, 600026, Russia). E-mail: tpp_vlgu@mail.ru. Scopus Author ID 54789107500, RSCI SPIN-code 6961-3659, https://orcid.org/0000-0001-8528-1195

Об авторах:

Романов Сергей Викторович, к.т.н., директор ООО «Эласт-ПУ» (600026, Россия, Владимир, ул. Гастелло, д. 21а). E-mail: romanov-elastpu@mail.ru. Scopus Author ID 55809569400, SPIN-код РИНЦ 9952-4171, https://orcid.org/0000-0002-5152-4678

Ботвинова Ольга Андреевна, к.т.н., начальник исследовательской лаборатории ООО «Эласт-ПУ» (600026, Россия, Владимир, Гастелло, д. 21а). E-mail: ermolaeva_olya@inbox.ru. Scopus Author ID 56907022800, SPIN-код РИНЦ 3933-6884, https://orcid.org/0000-0003-0217-8446

Тимаков Евгений Александрович, старший преподаватель кафедры «Химические технологии», ФГБОУ ВО «Владимирский государственный университет им. Александра Григорьевича и Николая Григорьевича Столетовых» (600000, Россия, Владимир, ул. Горького, д. 87). E-mail: okakie@yandex.ru. Scopus Author ID 57219018403, SPIN-код РИНЦ 3178-1477, https://orcid.org/0000-0003-3312-0810

Ращупкина Дарья Андреевна, магистрант кафедры «Химические технологии», ФГБОУ ВО «Владимирский государственный университет им. Александра Григорьевича и Николая Григорьевича Столетовых» (600000, Россия, Владимир, ул. Горького, д. 87). E-mail: dashulka010220002016@gmail.com. https://orcid.org/0000-0001-7724-6218

Панов Юрий Терентьевич, д.т.н., профессор, заведующий кафедрой «Химические технологии», ФГБОУ ВО «Владимирский государственный университет им. Александра Григорьевича и Николая Григорьевича Столетовых» (600000, Россия, Владимир, ул. Горького, д. 87). E-mail: tpp_vlgu@mail.ru. Scopus Author ID 54789107500, SPIN-код РИНЦ 6961-3659, https://orcid.org/0000-0001-8528-1195

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