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RESEARCH ARTICLE

Rheological properties of phosphorus-containing oligoester(meth)acrylate for processing by vacuum infusion

Oleg O. Tuzhikov[1,✉], **Lyubov Yu. Donetskova**[1], **Semyon M. Solomakhin**[1], **Anna V. Nalesnaya**[1], **Ali Al-Hamzawi**[2], **Boris A. Buravov**[1], **Sergey V. Borisov**[1], **Oleg I. Tuzhikov**[1]

[1] *Volgograd State Technical University, Volgograd, 400005 Russia*

[2] *Al-Qadisiyah, Al-Diwaniyah, 58002 Iraq*

✉ *Corresponding author, e-mail: tuzhikovoleg@mail.ru*

Abstract

Objectives. To obtain polymer composite materials (PCM) with enhanced physicomechanical properties using the vacuum assisted resin transfer molding (VaRTM) method, binders must have a viscosity of up to 500 mPa·s. In some cases, this leads to restrictions on the use of certain materials or requires the use of temporary diluents. This is closely related to the deterioration of other required composite characteristics, such as increased flammability. Three phosphorus-containing oligoester(meth)acrylates PhOEM-1, PhOEM-2, and PhOEM-3 were synthesized with significant differences in viscosity characteristics in the series PhOEM-1 << PhOEM-2 << PhOEM-3. The polymer based on PhOEM-1 exhibits inferior physicomechanical properties despite having lower viscosity. Hence, the aim of the study was to investigate the viscosity characteristics of mixtures of methacrylate binders of the same nature but different structures and functionalities. This was done by studying the rheological properties of the original oligoester(meth)acrylates and their mixtures taken in various ratios. The method used was to optimize compositions via a simplex lattice (Scheffe's plan), in order to obtain PCM using the VaRTM technology.

Methods. The study of rheological properties of phosphorus-containing oligoester(meth)acrylates and their mixtures was conducted using the method of rotational viscometry on a Brookfield LVDV-II + Pro viscometer with a spindle 27 at different shear rates ranging from 0 to 70 s^{-1} and temperatures from 30 to 70°C. Rheological studies were also conducted on a Lamy Rheology GT300 PLUS (GEL TIMER) viscometer in the same range of shear rates and temperatures.

Results. It was established that the objects under investigation can be characterized by viscosity values ranging from 96 to 2137 mPa·s depending on the temperature. The nature of the viscous flow of phosphorus-containing oligoester(meth)acrylates and their mixtures is similar to that of Newtonian liquids only at certain shear rates. The effective activation energies of the viscous flow of binders and their mixtures were calculated, and the influence of temperature on the viscosity of binders was determined.

Conclusions. The study identified the features and nature of the flow curves of phosphorus-containing oligoester(meth)acrylate binders of the same nature but different structures and functionalities, as well as of their mixtures. The optimal composition ranges of three-component mixtures of phosphorus-containing oligoester(meth)acrylates for use in the VaRTM technological process in producing polymer composite materials within the temperature range of 30 to 70°C were defined. The optimal compositions and temperature conditions for obtaining polymer composite materials using the VaRTM technology were also identified. This enables the production of polymer products with complex geometric shapes and varying sizes.

Keywords

rheology, polymer composite materials, VaRTM technology, phosphorus-containing binders, mathematical planning

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НАУЧНАЯ СТАТЬЯ

Реологические свойства фосфорсодержащего олигоэфир(мет)акрилата для переработки методом вакуумной инфузии

О.О. Тужиков[1,✉], Л.Ю. Донецкова[1], С.М. Соломахин[1], А.В. Налесная[1], А. Аль-Хамзави[2], Б.А. Буравов[1], С.В. Борисов[1], О.И. Тужиков[1]

[1] *Волгоградский государственный технический университет, Волгоград, 400005 Россия*

[2] *Университет Аль-Кадисия, Эд-Дивания, 58002 Ирак*

✉ *Автор для переписки, e-mail: tuzhikovoleg@mail.ru*

Аннотация

Цели. Для получения полимерных композиционных материалов (ПКМ) с повышенным уровнем физико-механических свойств методом безавтоклавной технологии (vacuum assisted resin transfer molding, VaRTM) связующие должны обладать вязкостью до 500 мПа·с. В ряде случаев это приводит к ограничению по применению тех или иных материалов, либо требует использования временных разбавителей, что неразрывно связано с ухудшением других требуемых характеристик композита, например, увеличением горючести. Нами были синтезированы три фосфорсодержащих олигоэфир(мет)акрилата PhOEM-1, PhOEM-2, PhOEM-3, обладающих значительными отличиями по характеристикам вязкости в ряду PhOEM-1 << PhOEM-2 << PhOEM-3, при этом полимер на основе PhOEM-1, обладая меньшей вязкостью, характеризуется худшими физико-механическими свойствами. В связи с этим, целью исследования явилось изучение вязкостных характеристик смесей метакрилатных связующих одинаковой природы, разного строения и функциональности путем изучения реологических свойств исходных олигоэфир(мет)акрилатов и их смесей, взятых в различных соотношениях, с применением метода оптимизации составов по симплекс-решетке (плану Шеффе) для получения ПКМ по безавтоклавной (VaRTM) технологии.

Методы. Исследование реологических свойств фосфорсодержащих олигоэфир(мет)акрилатов и их смесей проводили методом ротационной вискозиметрии на вискозиметре Brookfield LVDV-II + Pro с использованием шпинделя 27 при скоростях сдвига в диапазоне от 0 до 70 $с^{-1}$ и температурах от 30 до 70°С. Параллельно проводили реологические исследования на вискозиметре Lamy Rheology GT300 PLUS (GEL TIMER) в том же диапазоне скоростей сдвига и температур.

Результаты. Установлено, что исследуемые объекты, в зависимости от температуры, характеризуются значениями вязкости от 96 до 2137 мПа·с. По характеру вязкого течения фосфорсодержащие олигоэфир(мет)акрилаты и их смеси ведут себя аналогично ньютоновским жидкостям только при определенных скоростях сдвига. Рассчитаны эффективные энергии активации вязкого течения связующих и их смесей, установлено влияние температуры на вязкость связующих.

Выводы. Определены особенности и характер кривых течения фосфорсодержащих олигоэфир(мет)акрилатных связующих одинаковой природы, разного строения и функциональности, а также их смесей. Установлены области оптимальных составов трехкомпонентных смесей фосфорсодержащих олигоэфир(мет)акрилатов для использования их в технологическом процессе VaRTM при получении ПКМ в диапазоне температур от 30 до 70°С. Определены оптимальные составы и температурные условия для получения ПКМ методом VaRTM, что позволяет получать полимерные изделия сложной геометрической формы и разного размера.

Ключевые слова

реология, полимерные композиционные материалы, VaRTM-технология, фосфорсодержащие олигоэфир(мет)акрилатные связующие, математическое планирование

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INTRODUCTION

Over the past decade, the market for polymer composite materials (PCM) based on thermosets has been growing rapidly. This is due to the unique properties of the polymers obtained: resistance to wear and chemicals, including in a wide temperature range, and the ability to process material using modern energy-saving and environmentally friendly methods, such as vacuum assisted resin transfer molding (VaRTM) [1–3].

In addition to a wide range of choice of binders for obtaining PCM, methods for their processing are also being developed. Examples include manual laying out, spraying, injection and autoclave molding. As a rule, the above methods for obtaining PCM are relatively expensive processes requiring simultaneously increased manufacturability and reduced production cost[1] [4, 5].

One promising technology in terms of cost and manufacturability is a relatively new non-autoclave technology: the vacuum infusion method VaRTM [6, 7]. However, when producing composites in this way, one of the most important technological characteristics limiting the use of the binder is its viscosity. The authors [8–11] found that the optimal viscosity of the binder for vacuum infusion should not exceed 500 mPa·s.

Colleagues previously synthesized and patented a phosphorus-containing trifunctional oligoester(meth)-acrylate (PhOEM-1) [12]. Later, we obtained and patented tetrafunctional oligoester(meth)acrylates containing spacers (PhOEM-2, PhOEM-3) [13, 14]. All of the oligoester(meth)acrylates under consideration are capable of curing in the presence of a peroxide initiator, while possessing different levels of initial viscosity and physical and mechanical properties. Synthesized tetrafunctional ester(meth)acrylates, unlike trifunctional ones, are characterized by relatively high viscosity values at temperatures up to 40°C. These temperatures limit their use under standard production conditions. We selected the above compounds, in order to study the rheological properties of their mixtures in the temperature range of 30–70°C.

Taking the above into account, the aim of this work is to study the features of the flow curves of phosphorus-containing oligoester(meth)acrylates and their mixtures at temperatures of 30–70°C, in order to determine the optimal compositions of three-component mixtures that satisfy the processes of obtaining PCMs by the VaRTM method.

EXPERIMENTAL

The objects of the study were phosphorus-containing polymerizable oligoester(meth)acrylates (PhOEM) with different molecular weights and relative unsaturation, with and without a spacer in the structure.

The compounds were synthesized according to the method described in [15].

The rheological properties of the binders and their mixtures were studied using rotational viscometry. For this purpose, a Brookfield LVDV-II + Pro spindle 27 viscometer (*Brookfield*, USA) and a Lamy Rheology GT300 PLUS (GEL TIMER) viscometer (*Lamy Rheology*, France) were used. The results obtained on the Lamy Rheology GT300 PLUS (GEL TIMER) viscometer showed similar trends in the change in the properties of the binders.

In order to determine the region of optimal compositions of oligoester(meth)acrylates, the STATISTICA program (*StatSoft*, Russia) was used. It implements a graphically oriented approach to the analysis of experimental data [16].

RESULTS AND DISCUSSION

Figures 1–3 presents the structures of the compounds studied and synthesized in accordance with work [15].

Generalized structural formulas are presented in Fig. 4.

Figure 4 shows the difference in the structure of the compounds: different numbers of unsaturated groups; and the presence or absence of a spacer (the fragment of the structure contains an insert between the reactive centers in one molecule) [15].

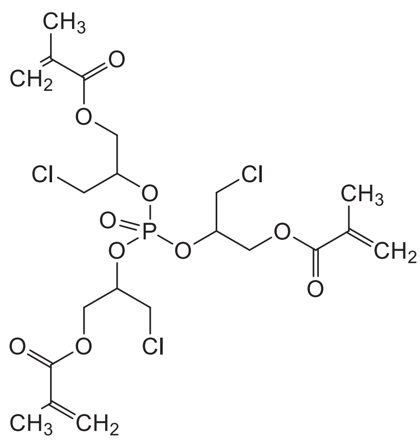

Fig. 1. Structure of PhOEM-1

[1] Veshkin EA. *Technologies of non-autoclave molding of low-porous polymer composite materials and large-sized structures made from them.* Diss. Cand. Sci. (Eng.). Moscow: VIAM; 2016. 146 p. (in Russ.).

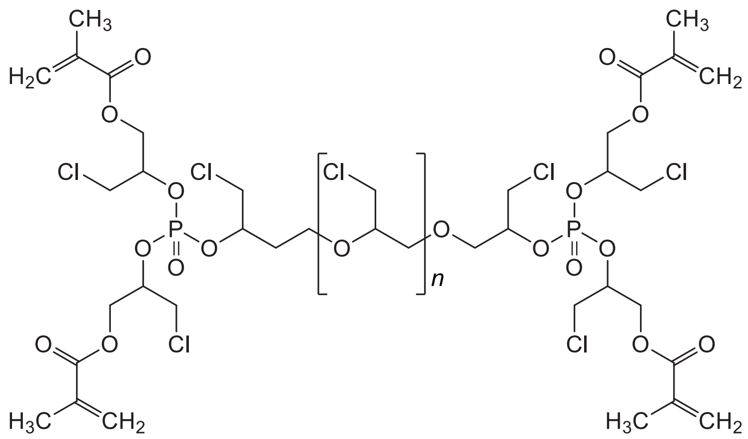

Fig. 2. Structure of PhOEM-2

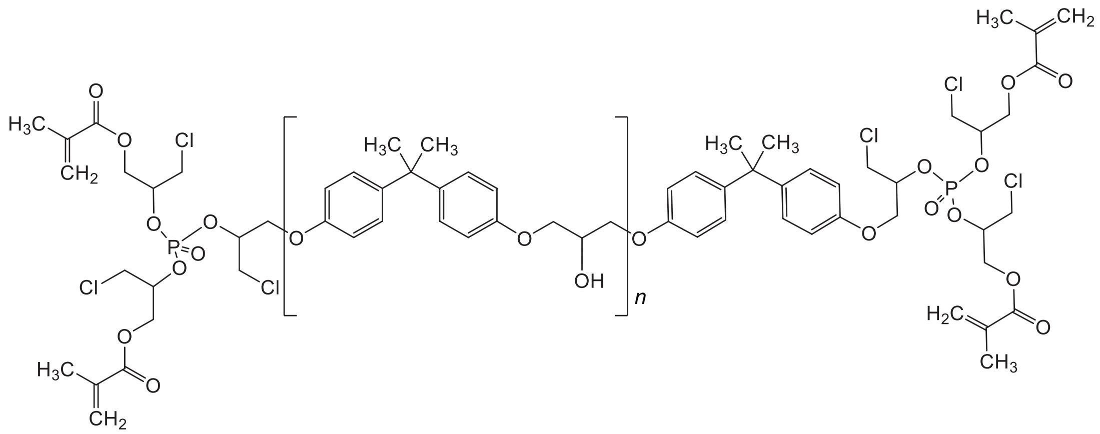

Fig. 3. Structure of PhOEM-3

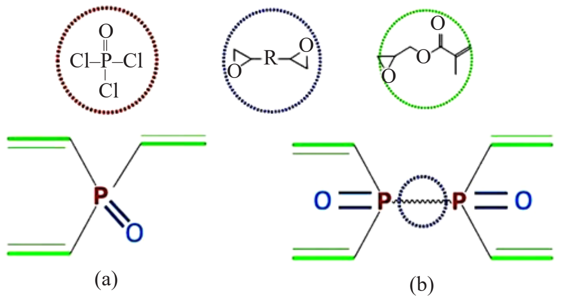

Fig. 4. Generalized structural chemical formulas of PhOEMs and of the starting reagents for their production:
(a) PhOEM-1 (without spacer);
(b) PhOEM-2 and PhOEM-3, with the different spacer structure

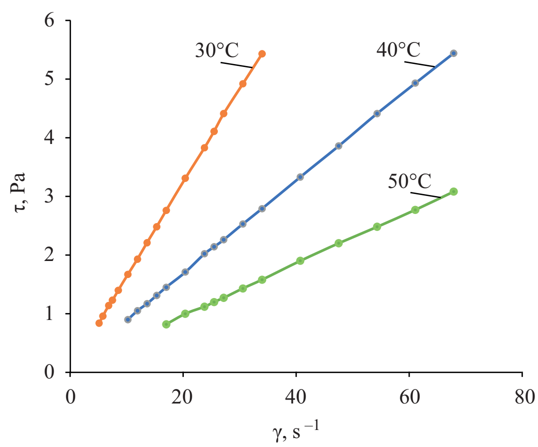

Fig. 5. Share rate versus shear stress for PhOEM-1 depending on temperature: τ is shear stress, Pa; γ is shear rate, s^{-1}

The viscosity of the synthesized compounds was studied at different shear rates and temperatures. The results of the studies of the rheological properties in the form of flow curves are presented in Figs. 5–7.

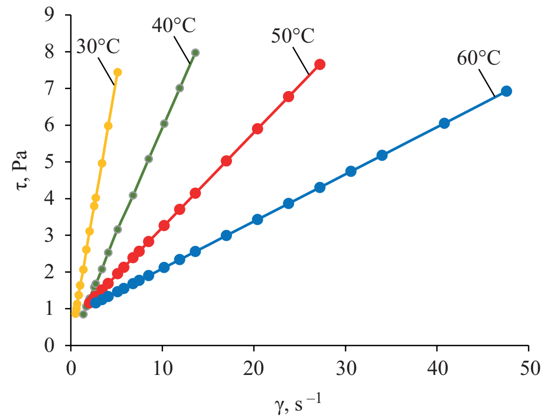

Fig. 6. Share rate versus shear stress for PhOEM-2 depending on temperature: τ is shear stress, Pa, γ is shear rate, s^{-1}

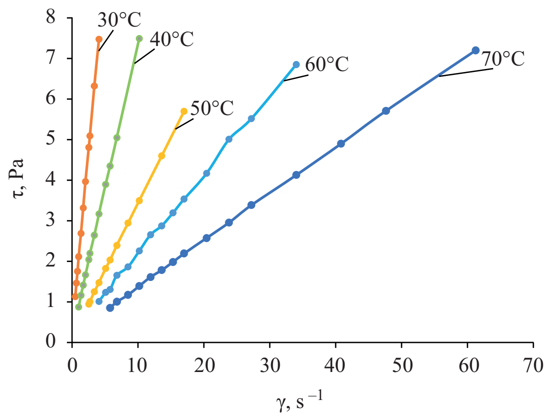

Fig. 7. Share rate versus shear stress for PhOEM-3 depending on temperature: τ is shear stress, Pa, γ is shear rate, s^{-1}

The study of the rheological properties of PhOEM-1 is limited to the temperature range of 30–50°C. This is due to the insufficient sensitivity of the device and the low viscosity of the oligomer at these temperatures. In this case, the developer of the device recommended using a spindle of a different size. However, measurements of liquid viscosity using different geometric spindle sizes are not recommended for comparative studies.

In order to compare the viscosity properties of the oligomers under study, we used a range of shear rates at which the liquids behaved similarly to Newtonian ones. As temperature increased, the study was continued at higher shear rates to ensure the specified conditions.

Based on the results obtained, the effective activation energies of the viscous flow of the objects under study were calculated using Eq. (1) [17].

$$E_a = \frac{R \cdot T_1 \cdot T_2}{T_1 - T_2} \lg \frac{\eta_2}{\eta_1}, \quad (1)$$

wherein E_a is the effective activation energy of viscous flow, J/mol; R is the gas constant, J/(K·mol); T_i is the test temperature, K; η_i is the viscosity, mPa·s.

The results obtained are presented in Fig. 8.

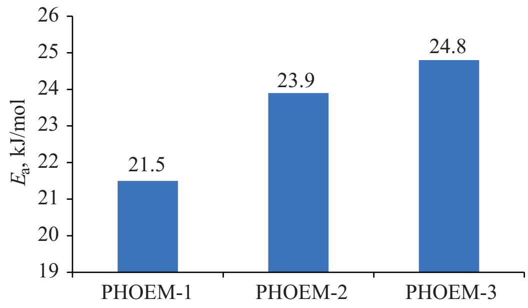

Fig. 8. Flow activation energy in PhOEM-1, PhOEM-2, PhOEM-3

The data presented shows that the effective activation energy of viscous flow depends on the compound structure (Fig. 4) and the presence of spacers of various structures in it. This must be taken into account when developing the process for obtaining PCM using VaRTM technology.

Studies were carried out using the mathematical planning method of an experiment using a simplex lattice (Scheffe's plan) with the construction of a fourth-order polynomial model [18]. The objective was to select the optimal compositions of oligoester(meth)acrylate mixtures which provide the necessary conditions for the technological criteria for vacuum infusion. The essence of the method consists in constructing a regression dependence of the mixture properties on the content of components. The use of this method makes it possible to establish the dependence of the properties of binders on their composition [19, 20].

The study area of the PhOEM-1, PhOEM-2, PhOEM-3 system includes mass fractions from 0 to 1. This study area is presented in the form of an equilateral Gibbs concentration triangle. Figure 9 shows the position of the experimental points of the compositions, i.e., the points of quantitative ratios of the dosages of the given compounds which form the basis of the experimental planning matrix table.

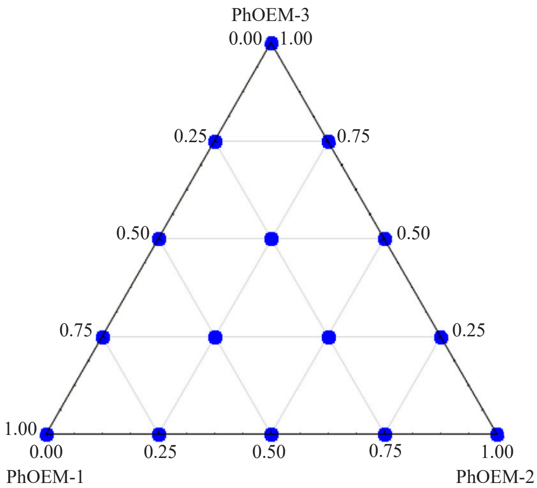

Fig. 9. Gibbs triangle showing concentration

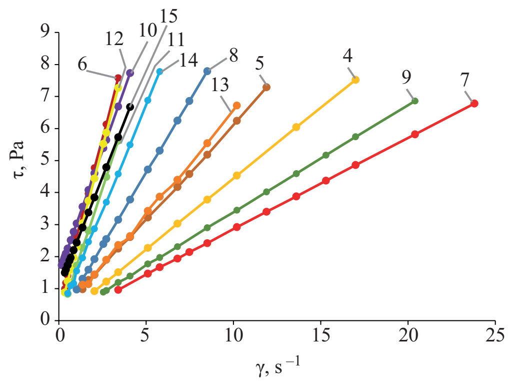

Fig. 10. Share rate versus shear stress for mixtures at a temperature of 30°C: τ is shear stress, Pa; γ is shear rate, s^{-1}

Figure 10 presents the results of the studies of the rheological properties of the binder mixtures in accordance with mathematical planning using Scheffe's plan (see Table) in the form of flow curves. As in the above study, regions of viscous flow were selected where the mixtures behaved similarly to Newtonian fluids.

The rheological dependencies presented in Fig. 10 show that the mixtures behave similarly to Newtonian fluids [21] and are satisfactorily described by Eq. (2):

$$\tau = \eta \cdot \gamma, \quad (2)$$

wherein η is the dynamic viscosity, Pa·s; τ is the shear stress, Pa; γ is the shear rate, s^{-1}.

Using the results obtained, we then calculated the values of the effective activation energy of viscous flow for the oligomer mixtures.

The ratio of components, viscosity values at temperatures of 30–70°C and the values of the effective activation energy of viscous flow for the mixtures studied are presented in the table.

In order to determine the region of optimal compositions of oligoester(meth)acrylates using a graphical method, the STATISTICA program was used. This program implements a graphical-oriented approach to the analysis of experimental data. Diagrams of the nature of the change in properties from the composition of phosphorus-containing binders are presented in Fig. 11.

Figure 11 shows that there are ranges of viscosity values at temperatures of 30 and 40°C, below which

Table. Planning matrix and corresponding viscosities and activation energy of viscous flow (10 rpm)

Composition number	PhOEM			Viscosity, mPa·s					E_a, kJ/mol
	1	2	3	30°C	40°C	50°C	60°C	70°C	
1	1	0	0	162	89	48	–	–	21.5
2	0	1	0	1460	612	434	401	373	23.9
3	0	0	1	1856	776	368	248	155	24.4
4	0.5	0.5	0	445	232	127	66	–	23.2
5	0.5	0	0.5	661	408	220	124	94	20.3
6	0	0.5	0.5	2231	1035	530	304	178	24.2
7	0.75	0.25	0	284	150	96	56	34	19.7
8	0.25	0.75	0	926	436	239	124	71	24.4
9	0.75	0	0.25	349	202	131	89	67	16.6
10	0.25	0	0.75	1987	984	661	506	363	16.6
11	0	0.75	0.25	1652	764	361	195	129	25.9
12	0	0.25	0.75	2137	932	438	255	148	25.8
13	0.5	0.25	0.25	694	347	195	114	74	21.9
14	0.25	0.5	0.25	1347	668	328	192	102	23.7
15	0.25	0.25	0.5	1699	905	530	312	227	20.6

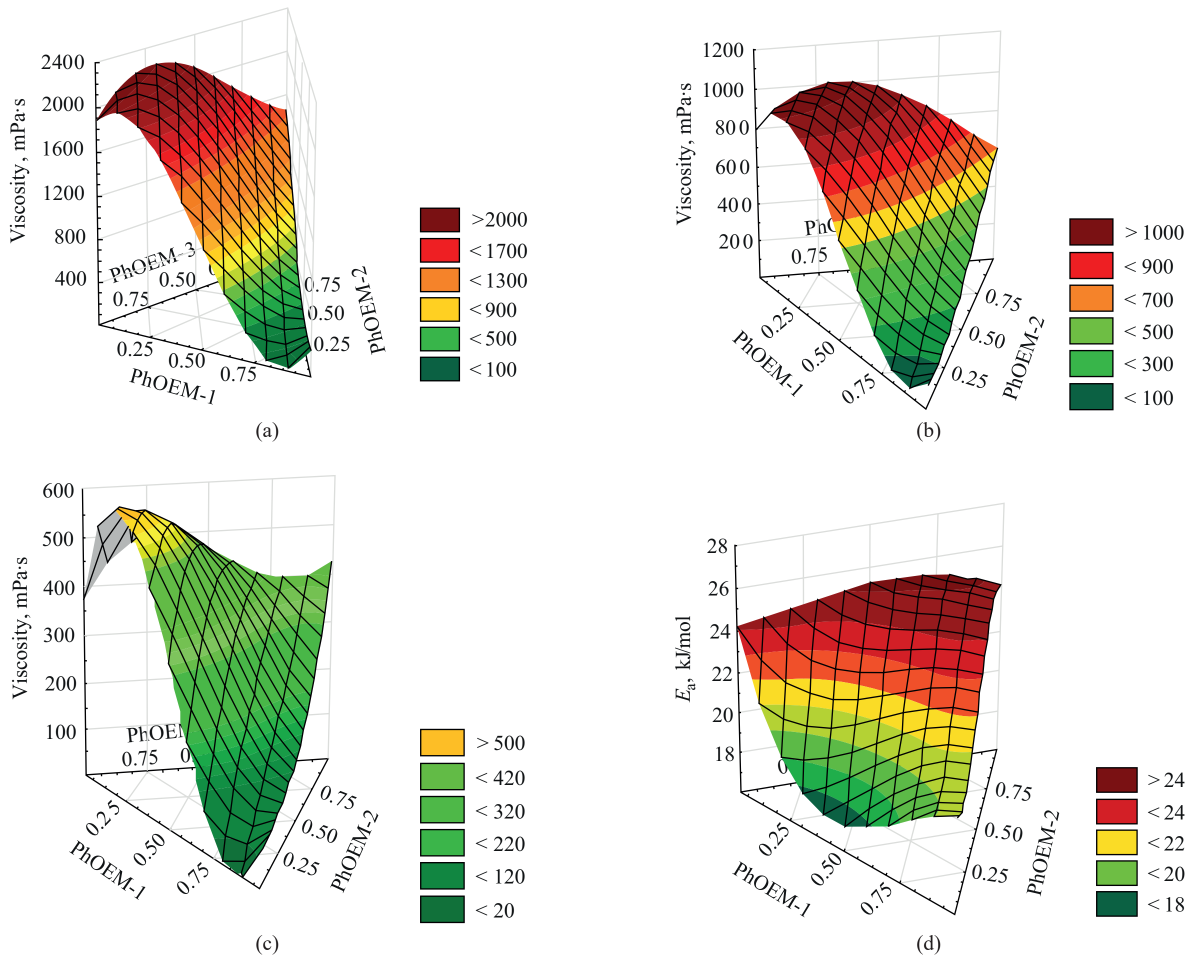

Fig. 11. Composition–property diagrams of mixtures at temperatures:
(a) 30°C, (b) 40°C, (c) 50°C, (d) effective E_a, kJ/mol

the ratio of mixture components is optimal. Note that at temperatures above 50°C, the initial binders and their mixtures meet the requirements for processing polymer materials using the VaRTM technology, since their viscosity values do not exceed 500 mPa·s.

The mixture PhOEM-1 : PhOEM-3 = 1 : 1 has the minimum value of the effective activation energy of viscous flow. The introduction of PhOEM-2 into this mixture leads to an increase in the effective activation energy of viscous flow. This is probably due to the intermolecular interaction of the oligomers and their thermodynamic compatibility.

CONCLUSIONS

The paper studies the features of flow curves of phosphorus-containing oligoester(meth)acrylates and their mixtures. The areas of optimal compositions of three-component mixtures for their use in the VaRTM process for producing PCMs at temperatures from 30 to 70°C were determined.

The difference in the obtained flow curves of methacrylates was determined by the molecular weight of the compounds and their structure, as well as the presence of a spacer in the structure. The flow curves become linear at certain shear rates, above which, within the studied range of rates, the behavior of the studied liquids is similar to a Newtonian fluid.

Compounds with a spacer (PhOEM-2, PhOEM-3) can be characterized by significantly higher viscosity values of 1460 and 1856 mPa·s at 30°C, which is two or more times higher than the viscosity of PhOEM-1 (162 mPa·s). The same tendency remains at high temperatures.

It was established that at temperatures above 50°C, the viscosity of both the initial components and their mixtures in any ratio meets the conditions of the VaRTM technology for obtaining PCM with improved physical and mechanical properties.

By using the composition–viscosity and composition–activation energy diagrams together, the region of optimal technological conditions (temperature, pressure difference) for obtaining PCM by the vacuum infusion method can be determined.

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Authors’ contributions

O.O. Tuzhikov—research idea, consultation on experimental procedures, writing the article.

L.Yu. Donetskova—experiment execution, data analysis, data processing.

S.M. Solomakhin—experiment execution, data analysis, data processing.

A.V. Nalesnaya—literature analysis, formalization of the reference list.

A. Al-Hamzawi—consultation on experimental procedures.

B.A. Buravov—experimental data processing, writing the article.

S.V. Borisov—consultation on rheological properties of binders.

O.I. Tuzhikov—consultation on chemistry of phosphorus-containing compounds, as well as on planning, methodology, and implementation of research.

The authors declare no conflicts of interest.

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About the authors

Oleg O. Tuzhikov, Dr. Sci. (Eng.), Associate Professor, Head of the Department of General and Inorganic Chemistry, Volgograd State Technical University (28, pr. im. V.I. Lenina, Volgograd, 400005, Russia). E-mail: tuzhikovoleg@mail.ru. Scopus Author ID 12645529200, RSCI SPIN-code 8142-5915, https://orcid.org/0000-0001-6316-8896

Lyubov Yu. Donetskova, Master Degree, Engineer, Department of General and Inorganic Chemistry, Volgograd State Technical University (28, pr. im. V.I. Lenina, Volgograd, 400005, Russia). E-mail: lovedonetskova@mail.ru. RSCI SPIN-code 8843-1727, https://orcid.org/0009-0008-8085-5071

Semyon M. Solomakhin, Master Degree, Engineer, Department of General and Inorganic Chemistry, Volgograd State Technical University (28, pr. im. V.I. Lenina, Volgograd, 400005, Russia). E-mail: solomakhin-sim@mail.ru. https://orcid.org/0009-0007-5040-3683

Anna V. Nalesnaya, Cand. Sci. (Chem.), Associate Professor, Department of General and Inorganic Chemistry, Volgograd State Technical University (28, pr. im. V.I. Lenina, Volgograd, 400005, Russia). E-mail: nalesnaya@yandex.ru. RSCI SPIN-code 1669-1722, https://orcid.org/0009-0006-4083-129X

Ali Al-Khamzawi, Lecturer, Department of Chemical Engineering, College of Engineering, Al-Qadisiyah University (Al-Qadisiyah, Al-Diwaniyah, 58002, Iraq). E-mail: ali.alhamzawi80@gmail.com. RSCI SPIN-code 2551-0018, https://orcid.org/0000-0003-4491-494X

Boris A. Buravov, Cand. Sci. (Chem.), Associate Professor, Department of General and Inorganic Chemistry; Senior Researcher, Laboratory of Polymer, Composite and Hybrid Functional Materials, Volgograd State Technical University (28, pr. im. V.I. Lenina, Volgograd, 400005, Russia). E-mail: byravov@ya.ru. Scopus Author ID 57972246000, RSCI SPIN-code 6730-5763, https://orcid.org/0000-0001-9039-571X

Sergey V. Borisov, Cand. Sci. (Eng.), Associate Professor, Department of Chemistry and Processing Technology of Elastomers; Senior Researcher, Laboratory of Polymer, Composite and Hybrid Functional Materials, Volgograd State Technical University (28, pr. im. V.I. Lenina, Volgograd, 400005, Russia). E-mail: borisov.volgograd@yandex.ru. Scopus Author ID 57193435253, RSCI SPIN-code 4774-4238, https://orcid.org/0000-0003-4400-0822

Oleg I. Tuzhikov, Dr. Sci. (Chem.), Professor, Department of Technology of Macromolecular and Fibrous Materials, Volgograd State Technical University (28, pr. im. V.I. Lenina, Volgograd, 400005, Russia). E-mail: tuzhikov_oi@vstu.ru. Scopus Author ID 6507272270, RSCI SPIN-code 7255-0330, https://orcid.org/0000-0003-1893-2861

Об авторах

Тужиков Олег Олегович, д.т.н., доцент, заведующий кафедрой общей и неорганической химии, ФГБОУ ВО «Волгоградский государственный технический университет» (400005, Россия, Волгоград, пр-т им. В.И. Ленина, д. 28). E-mail: tuzhikovoleg@mail.ru. Scopus Author ID 12645529200, SPIN-код РИНЦ 8142-5915, https://orcid.org/0000-0001-6316-8896

Донецкова Любовь Юрьевна, магистр, инженер кафедры общей и неорганической химии, ФГБОУ ВО «Волгоградский государственный технический университет» (400005, Россия, Волгоград, пр-т им. В.И. Ленина, д. 28). E-mail: lovedonetskova@mail.ru. SPIN-код РИНЦ 8843-1727, https://orcid.org/0009-0008-8085-5071

Соломахин Семён Михайлович, магистр, инженер кафедры общей и неорганической химии, ФГБОУ ВО «Волгоградский государственный технический университет» (400005, Россия, Волгоград, пр-т им. В.И. Ленина, д. 28). E-mail: solomakhin-sim@mail.ru. https://orcid.org/0009-0007-5040-3683

Налесная Анна Владимировна, к.х.н., доцент кафедры общей и неорганической химии, ФГБОУ ВО «Волгоградский государственный технический университет» (400005, Россия, Волгоград, пр-т им. В.И. Ленина, д. 28). E-mail: nalesnaya@yandex.ru. SPIN-код РИНЦ 1669-1722, https://orcid.org/0009-0006-4083-129X

Аль-Хамзави Али, преподаватель кафедры химической технологии, технический факультет, Университет Аль-Кадисия (58002, Ирак, Эд-Дивания, Аль-Джамаа, Сунния ул.). E-mail: ali.alhamzawi80@gmail.com. SPIN-код РИНЦ 2551-0018, https://orcid.org/0000-0003-4491-494X

Буравов Борис Андреевич, к.х.н., доцент кафедры общей и неорганической химии; старший научный сотрудник лаборатории полимерных, композитных и гибридных функциональных материалов, ФГБОУ ВО «Волгоградский государственный технический университет» (400005, Россия, Волгоград, пр-т им. В.И. Ленина, д. 28). E-mail: byravov@ya.ru. Scopus Author ID 57972246000, SPIN-код РИНЦ 6730-5763, https://orcid.org/0000-0001-9039-571X

Борисов Сергей Владимирович, к.т.н., доцент кафедры химии и технологии переработки эластомеров; старший научный сотрудник лаборатории полимерных, композитных и гибридных функциональных материалов, ФГБОУ ВО «Волгоградский государственный технический университет» (400005, Россия, Волгоград, пр-т им. В.И. Ленина, д. 28). E-mail: borisov.volgograd@yandex.ru. Scopus Author ID 57193435253, SPIN-код РИНЦ 4774-4238, https://orcid.org/0000-0003-4400-0822

Тужиков Олег Иванович, д.х.н., профессор кафедры технологии высокомолекулярных и волокнистых материалов, ФГБОУ ВО «Волгоградский государственный технический университет» (400005, Россия, Волгоград, пр-т им. В.И. Ленина, д. 28). E-mail: tuzhikov_oi@vstu.ru. Scopus Author ID 6507272270, SPIN-код РИНЦ 7255-0330, https://orcid.org/0000-0003-1893-2861

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