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Investigation of surface tension and contact angles for effective polymer binders based on epoxy oligomers and active diluents

Angelina D. Bresskaya[1,@], Dmitry A. Trofimov[1], Igor D. Simonov-Emel'yanov[1], Sergey I. Shalgunov[2], Vladimir I. Sokolov[2]

[1]*MIREA – Russian Technological University (M.V. Lomonosov Institute of Fine Chemical Technologies), Moscow, 119571 Russia*
[2]*NPO Stekloplastic, Andreevka, Moscow oblast, 141551 Russia*
[@]*Corresponding author, e-mail:* angelinabresskaya@gmail.com

Objectives. *This study focused on the quantification of the surface tension and the static and dynamic contact angles of epoxy oligomers, active diluents, and their mixtures of various compositions at different temperatures. The active diluents were aliphatic compounds based on glycidyl ethers, namely laproxides and a laprolate of different structure, functionality, molecular weight, and viscosity. Moreover, the preparation of effective polymer binders (matrices) for composites was explored.*
Methods. *In this study, the epoxy oligomers ED-20 and DER-330, laproxides 201B, DEG-1, E-181, and 703, laprolate 301, and their mixtures in various compositions were investigated. Their surface tension and the static and dynamic contact angles were determined by the Wilhelmy plate and ring methods on a semiautomatic tensiometer at different temperatures (20–60°C). The static contact angle was measured on a thin aluminum borosilicate glass plate, and the dynamic contact angles were determined using an installation for measuring surface tension developed by NPO Stekloplastik.*
Results. *The surface tension and static and dynamic contact angles were obtained for all epoxy oligomers and active diluents, as well as for their mixtures at 20–60°C. For binders based on systems of epoxy oligomers and active diluents, the impregnation rate of fiber reinforcement was also calculated. The introduction of laproxides or laprolates into the epoxy oligomers led to a decrease in surface tension and contact angles, while the increase in temperature increased the impregnation rate by 10–20 times.*
Conclusions. *The temperature increase from 20 to 60°C resulted in a decrease in the surface tension of mixed systems of epoxy oligomers and active diluents by almost two times. In addition, the contact angles changed by only 4°–7°, while the impregnation was significantly improved and the corresponding rate increased by 10–20 times.*

Keywords: *epoxy oligomers, active diluents, glycidyl ether-based aliphatic compounds, laproxides, laprolate, surface tension, static and dynamic contact angles, impregnation of fiber reinforcement.*

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Исследование поверхностного натяжения и углов смачивания для создания эффективных полимерных связующих на основе эпоксидных олигомеров с активными разбавителями

А.Д. Бресская[1,@], Д.А. Трофимов[1], И.Д. Симонов-Емельянов[1], С.И. Шалгунов[2], В.И. Соколов[2]

[1]*МИРЭА – Российский технологический университет (Институт тонких химических технологий имени М.В. Ломоносова), Москва, 119571 Россия*
[2]*АО «НПО Стеклопластик», Московская область, Андреевка, 141551 Россия*
[@]*Автор для переписки, e-mail:* angelinabresskaya@gmail.com

Цели. *Определение поверхностного натяжения, статического и динамического углов смачивания при разных температурах для эпоксидных олигомеров марок ЭД-20 и DER-330; для активных разбавителей – алифатических соединений на основе глицидиловых эфиров – Лапроксидов и Лапролата разной структуры, функциональности, молекулярной массы, вязкости; а также для систем, состоящих из эпоксидных олигомеров и активных разбавителей разного состава. Также целью являлось создание эффективных полимерных связующих (матриц) для композитов.*
Методы. *В качестве объектов исследования использовали эпоксидные олигомеры марок ЭД-20 и DER-330; активные разбавители – Лапроксиды (201Б, ДЭГ-1, Э-181, 703) и Лапролат 301; а также системы эпоксидный олигомер + Лапроксид (Лапролат) разных составов. Поверхностное натяжение, статический и динамический углы смачивания определяли методом Вильгельми и методом отрыва кольца на полуавтоматическом тензиометре при разных температурах (20–60 °С). Статический угол смачивания измеряли на тонкой пластине из алюмоборосиликатного стекла. Значения динамического угла смачивания определяли на установке для измерения поверхностного натяжения, разработанной АО «НПО Стеклопластик».*
Результаты. *Определены значения поверхностного натяжения, углов статического и динамического смачивания для эпоксидных олигомеров ЭД-20 и DER-330, Лапроксидов 201Б, ДЭГ-1, Э-181, 703 и Лапролата 301, а также для смешанных систем при температурах от 20 до 60 °С. Рассчитаны скорости пропитки армирующих волокнистых наполнителей эффективными связующими на основе смешанных систем. Показано, что при введении в эпоксидные олигомеры Лапроксидов (Лапролата), поверхностное натяжение снижается, углы смачивания уменьшаются, температура повышается, в результате чего скорость пропитки возрастает в 10–20 раз.*
Выводы. *Повышение температуры от 20 до 60 °С приводит к снижению поверхностного натяжения систем, состоящих из эпоксидных олигомеров и активных разбавителей, практически в 2 раза. Углы смачивания изменяются всего на 4°–7°, существенно улучшается качество пропитки, скорость пропитки увеличивается в 10–20 раз.*

Ключевые слова: *эпоксидные олигомеры, активные разбавители, алифатические соединения на основе глицидиловых эфиров, Лапроксиды, Лапролат, поверхностное натяжение, статический и динамический углы смачивания, пропитка армирующих волокнистых наполнителей.*

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INTRODUCTION

During the production of fiberglass for several purposes, various modifiers are introduced to regulate the physical, chemical, technological, and operational characteristics of epoxy binders [1, 2]. The use of glycidyl ester-based aliphatic compounds that contain epoxy groups as modifiers of epoxy oligomers (EO) is of particular interest. These compounds are also known as laproxides and laprolates[1] and can be efficiently combined with EO [3, 4]. More specifically, laproxides and laprolates act as active diluents (AD) and are embedded in the three-dimensional structure of the epoxy polymer during curing [5, 6], thus eliminating the solvent removal (drying) stage in the fiberglass manufacturing. Their effect on the kinetics of bulk shrinkage and stress during curing, as well as on the physical properties, deformation behavior, and molecular structure of epoxy matrices has already been investigated [7–10].

Although it is known that determining the composition of epoxy binders and the impregnation rate of reinforcing fiber fillers requires additional data on the contact angles and surface tension [11], there are no relevant literature reports on the surface tension and static contact angle of various AD surfaces and systems that are based on an epoxy oligomer and an AD (EO–AD). Moreover, data on the dynamic contact angle of polymer epoxy binders, EO, AD and EO–AD systems are also not available in the literature.

Therefore, the aim of this work is to study the surface tension (σ) and the static (θ_{st}) and dynamic (θ_{dyn}) contact angles of laproxides, laprolates, and EO–AD systems of different compositions at temperatures 20–60°C for the preparation of effective polymer binders based on the EO ED-20 and DER-330 and various AD with high impregnation rate of reinforcing fiberglass systems.

MATERIALS AND METHODS

For this study, EO ED-20 (*Ya.M. Sverdlov plant*, Russia) and DER-330 (*DOW Chemicals*, CAS Number 25036-25-3/1330-20-7 (100-41-4), USA), laproxides 201B (L-201B), DEG-1 (L-DEG-1), E-181 (L-E-181), and 703 (L-703) with different structures, functionalities, molecular weights, and viscosities, and laprolate 301 (LT-301) (*V.S. Lebedev NPP Makromer*, Russia) were used. Their main characteristics are provided in Table 1. Moreover, a series of EO–laproxide (or laprolate) systems of different compositions were used.

Table 1. Characteristics of EO and AD

Brand of EO or AD[2]	M_w, g/mol	ρ, g/cm^3	Functionality *N*, units	Epoxy content, wt %	Viscocity η at 25°C, mPa·s
Epoxy oligomer DER-330	340	1.16	2	23.2–24.4	5–7*
Epoxy oligomer ED-20 (GOST 10587-84[3])	410	1.17	2	20.0–22.5	20–25*
Laproxide 201 B (TU 2225-037-10488057-2007)	130	1.01	1	≥25.0	≤2.5
Laproxide DEG-1 (TU 2225-053-10488057-2010)	218	1.02	2	≥24.0	≤70
Laproxide E-181 (TU 2225-058-10488057-2010)	222.5	1.25	2	25.0–30.0	≤80
Laproxide 703 (TU 2226-029-10488057-98)	434	1.09	3	13.6–16.5	90–160
Laprolate 301 (TU 2226-303-10488057-94)	230	1.04	3	~2.5	≤30

* viscosity in Pa·s.

[1] Catalogue of products. NPP Makromer. Available from: http://www.macromer.ru/product/him/komponenty-dlya-lakokrasochnoj-promyshlennosti/aktivnye-razbaviteli-marki-laproksid/ (Accessed May 26, 2019) (in Russ.).
[2] Catalogue of products. NPP Makromer. Available from: http://www.macromer.ru/product/him/komponenty-dlya-lakokrasochnoj-promyshlennosti/aktivnye-razbaviteli-marki-laproksid/ (Accessed May 26, 2019) (in Russ.).
[3] GOST 10587-84. Uncured epoxy resins. Specifications (amended). Moscow: Standard Publishing, 1989 (in Russ.).

The surface tension (σ) of the EO, laproxides, laprolate, and EO–AD systems was determined by the Wilhelmy and ring separation methods using a semiautomatic tensiometer at different temperatures according to GOST R 50003-92 (ISO 304-85)[4]. The static contact angle (θ_{st}) was measured according to GOST 7934.2-74[5]. A thin plate of aluminum borosilicate glass was used as contact surface, which fully corresponded to the composition of glass fibers used in the production of fiberglass. Furthermore, the dynamic contact angles (θ_{dyn}) for the EO, AD, and EO–AD systems were calculated from the surface tension measurement data that were obtained using an installation developed by *NPO Stekloplastik* [12].

RESULTS AND DISCUSSION

The main aim of this study was to determine the surface tension (σ) and the static (θ_{st}) and dynamic (θ_{dyn}) contact angles for the indicated EO and AD (Table 2) and their mixed systems. It is worth noting that the θ_{dyn} values of the analyzed substances were identified for the first time and allowed the calculation of the impregnation rate of fibrous fillers, packages, and frames during a dynamic movement of the polymer binder, e.g., by infusion.

It is clear from Table 2 that the physical and chemical characteristics (σ, θ_{st}, and θ_{dyn}) of the indicated laproxides with various structures, LT-301, and the EO ED-20 and DER-330 were significantly different. Thus, these distinct variations were exploited to prepare a series of EO–AD systems with different compositions and characteristics and optimize the impregnation process of fibrous fillers within a wide range.

According to the σ values obtained after combining different EO–AD systems (Table 3), an increase in the content of AD to 40 vol % led to a significant decrease in the surface tension value of DER-330 and ED-20 by 1.5–4 times, i.e., from 37.2 and 43.0 mN/m, respectively, to a range of 11.6–25.6 mN/m depending on the AD brand. The greatest reduction was achieved for the DER-330–L-201B and ED-20–L-201B systems when 40 vol % L-201B was added, mainly due to the low surface tension of L-201B. However, the introduction of 40 vol % L-DEG-1 in each EO led to a less significant decrease in surface tension, while L-181 and L-703 led to a reduction in the surface tension of ED-20 and DER-330 by about 1.5 times. Instead, the introduction of LT-301 (40 vol %) in the EO was more effective, as the σ value of the corresponding ED-20–AD and DER-330–AD systems was reduced to 14.5 and 13.1 mN/m, respectively.

It should also be noted that reducing the surface tension can improve the impregnation of fiber fillers with an EO–AD-based polymer binder. Thus, the dependence of the surface tension on the AD content for the ED-20–AD systems and a semilogarithmic version of this dependence were plotted as depicted in Fig. 1. Based on Fig. 1a, the most effective reduction in the EO σ value was achieved when 25–40 vol % AD was added. However, it is known that the introduction of more than 20 vol % of a laproxide or a laprolate in EO is impractical, since there is a sharp decrease in the glass transition temperature of cured epoxy systems [8].

Furthermore, the linear dependence of lnσ on the AD content (Fig. 1b) allowed the application of the semilogarithmic additivity rule:

$$\ln\sigma = \varphi_{EO}\ln\sigma_{EO} + \varphi_{AD}\ln\sigma_{AD}, \quad (1)$$

where φ_{EO} and φ_{AD} are the volume fractions of the EO and AD contents, respectively, in the EO–AD system. Thus, the compositions of the EO–AD

Table 2. Surface tension and contact angles for the EO and AD at 20°C

Parameters	Epoxy oligomers		Laproxides				Laprolate
	DER-330	ED-20	L-201B	L-DEG-1	L-181	L-703	LT-301
σ, mN/m	37.2	43.0	9.4	18.7	14.5	13.8	10.0
θ_{st}, °	37.0	38.0	7.0	21.0	20.0	18.0	11.0
θ_{dyn}, °	57.8	58.7	27.0	39.8	36.5	33.0	31.4

[4] GOST P 50003-92 (ISO 304-85). Surface active agents. Determination of surface tension by drawing up liquid films. Moscow: Standard Publishing; 1992 (in Russ.).

[5] GOST 7934.2-74 Watch oils. Method for the determination of regional wetting angle (amended). Collection of Standards. Moscow: Standartinform; 2006 (in Russ.).

Table 3. Surface tension of the studied EO–AD systems at 20°C*

AD content, vol %	Surface tension (σ), mN/m				
	L-201B	**L-DEG-1**	**L-181**	**L-703**	**LT-301**
10	24.0/16.0	34.7/24.8	31.3/29.1	28.3/22.4	26.4/19.5
20	16.0/13.3	29.8/21.9	26.0/27.6	21.2/18.2	19.5/14.5
30	13.1/12.8	26.0/21.0	22.5/26.8	18.0/15.9	16.3/13.7
40	11.6/12.0	23.4/20.7	20.0/25.6	16.0/13.8	14.5/13.1

*The numerator in the σ values indicates the σ value obtained for the ED-20–AD systems, and the denominator indicates the σ value obtained for the DER-330–AD systems.

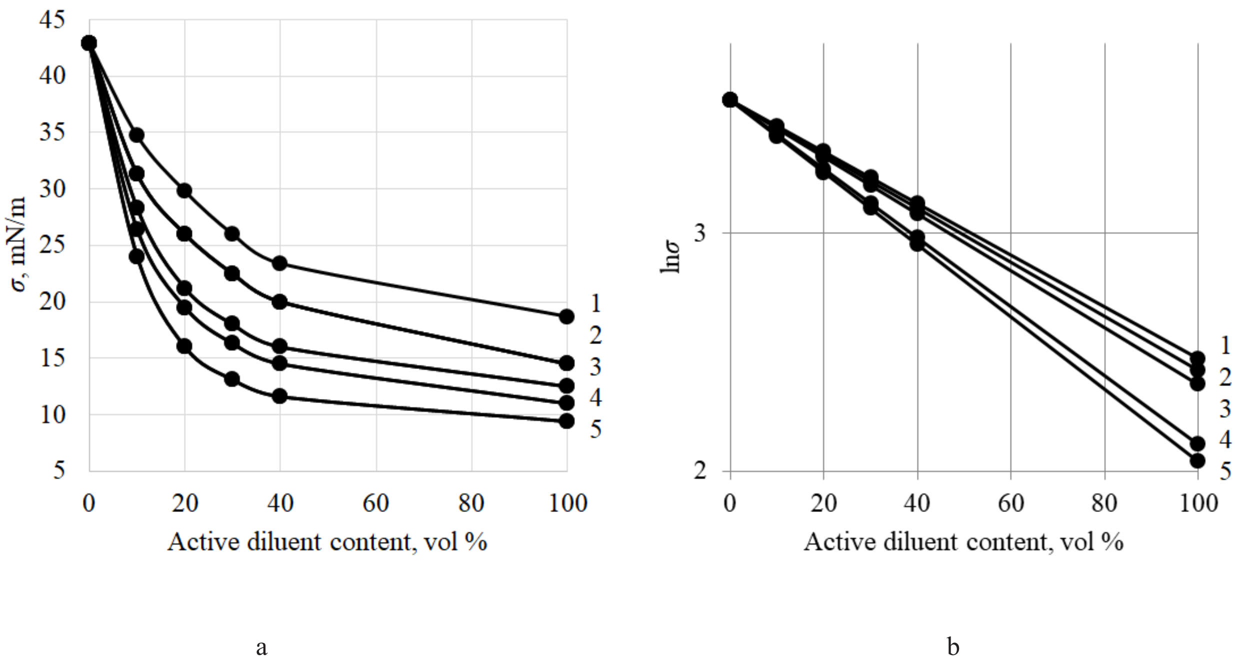


Fig. 1. Dependence of the (a) σ and (b) lnσ parameters of the ED-20–AD systems on the content of 1) L-E-181, 2) L-DEG-1, 3) LT-301, 4) L-703, and 5) L-201B at 20°C.

systems could be calculated based on Eq. (1) with a given value of surface tension.

In addition to the reduction of the σ value, the decrease in θ_{st} could also improve the impregnation of fiber fillers with an EO–AD-based polymer binder. According to the data obtained for the EO-20–AD and DER-330–AD systems with different AD contents (Table 4), the initial θ_{st} values (Table 2) of EO-20 and DER-330 were significantly reduced.

The results of Table 4 were further confirmed by exploring the dependence of θ_{st} and lnθ_{st} on the AD content for ED-20–AD systems. In particular, based on Fig. 2a, the introduction of different AD contents in ED-20 reduced the static contact angle by a range of 7°–21°, while the most effective decrease (by 3.5 times) was achieved for the ED-20–L-201B system, when 40 vol % L-201B was used.

Moreover, the linear dependence of lnθ_{st} on the AD content (Fig. 2b) allowed the application of the semilogarithmic additivity rule, which could be also used to calculate the composition of the EO–AD systems with a given θst value, as with Eq. (1):

$$\ln\theta_{st} = \varphi_{EO}\ln\theta_{EO}^{st} + \varphi_{AD}\ln\theta_{AD}^{st}, \quad (2)$$

where φ_{EO} and φ_{AD} are the volume fractions of the EO and AD contents, respectively, in the EO–AD system.

Another important parameter for the investigated substances is the dynamic contact angle (θ_{dyn}), which can significantly affect their impregnation in reinforcing systems. The θ_{dyn} values obtained for the EO–AD systems with different AD contents (Table 5) indicated that the introduction of various AD in the EO can decrease the θ_{dyn} value.

These results were further confirmed by the investigation of the dependence of θ_{dyn} and $\ln\theta_{dyn}$ on the AD content of the ED-20–AD systems (Fig. 3). However, the introduction of the laproxides and laprolate in EO reduced the θ_{dyn} value only by 15°–20° (Fig. 3a).

As for the other factors, the linear dependence of $\ln\theta_{dyn}$ on the AD content (Fig. 3b) allowed the application of the semilogarithmic additivity rule:

Table 4. Static contact angle (θ_{st}) of the studied EO–AD systems at 20°C*

AD content, vol %	Static contact angle θ_{st}, °				
	L-201B	L-DEG-1	L-181	L-703	LT-301
10	27/27	32/32	32/31	31/31	30/30
20	20/20	28/28	27/27	26/26	24/24
30	14/14	25/25	24/24	23/23	20/18
40	11/11	23/23	22/22	21/21	15/17

*The numerator in the θ_{st} values indicates the θ_{st} value obtained for the ED-20–AD systems, and the denominator indicates the θ_{st} value obtained for the DER-330–AD systems.

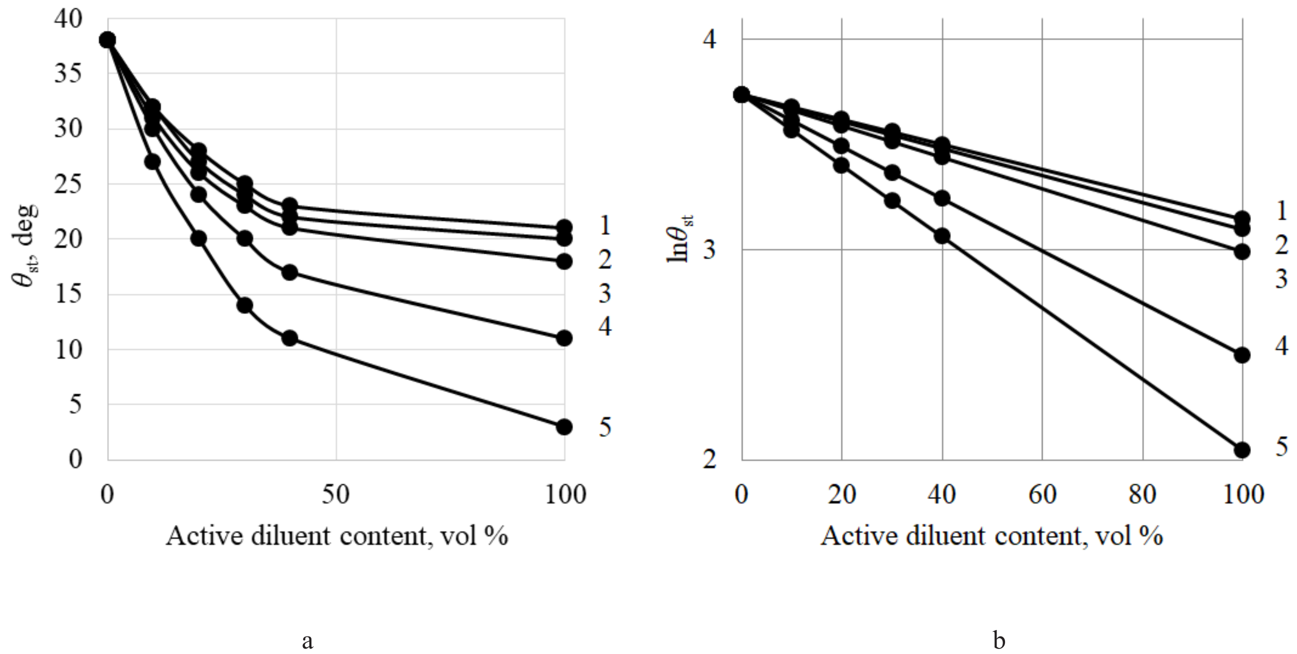


Fig. 2. Dependence of the (a) θ_{st} and (b) $\ln\theta_{st}$ parameters of the ED-20–AD systems on the content of 1) L-DEG-1, 2) L-E-181; 3) L-703, 4) LT-301, and 5) L-201B at 20°C.

$$\ln \theta_{dyn} = \varphi_{EO} \ln \theta_{EO}^{dyn} + \varphi_{AD} \ln \theta_{AD}^{dyn} \qquad (3)$$

In summary, based on the obtained results, the effectiveness of the studied AD substances on the σ, θ_{st}, and θ_{dyn} values of the EO–AD systems increased in the order L-DEG-1 → L-E-181 → L-703 → LT-301 → L-201B.

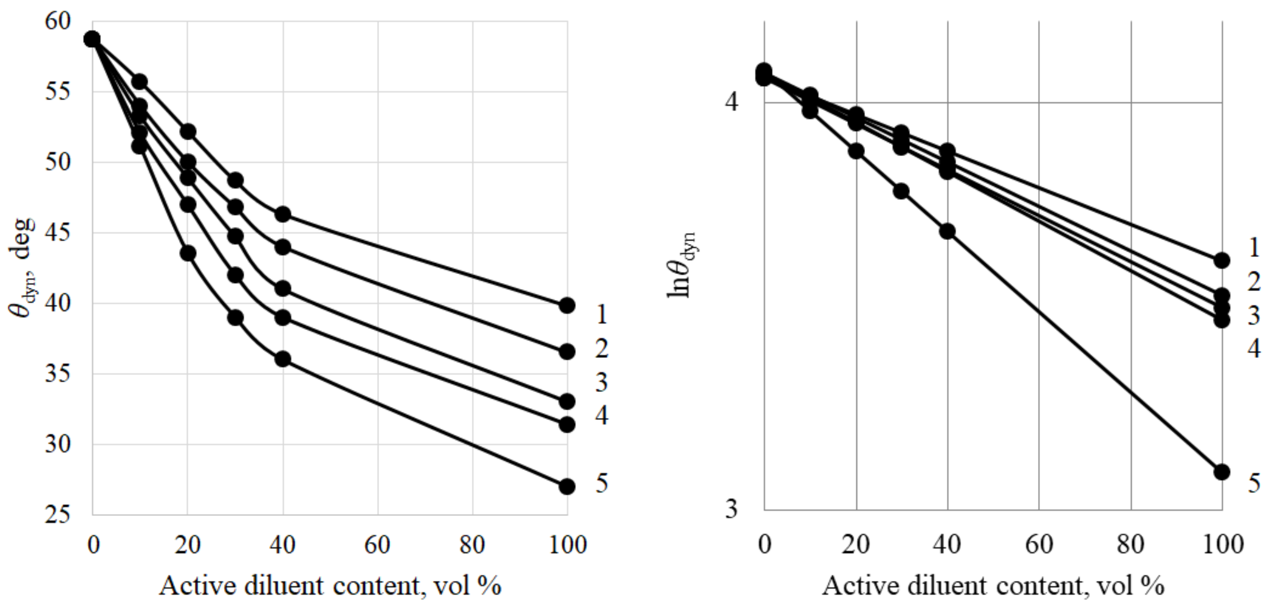
Furthermore, the molecular mobility of EO and AD increased with increasing temperature, leading to changes in the physical and chemical characteristics (σ, θ_{st}, and θ_{dyn}) of EO, laproxides, laprolate, and EO–AD systems. Thus, these values were measured for the ED-20–AD system at 20 and 60°C with 20 vol % AD content (Table 6). These temperatures were selected taking into account the technological impregnation modes of fibrous fillers with epoxy binders [11].

An increase in temperature from 20 to 60°C led to a decrease in the surface tension of the studied EO–AD systems by 4–14 mN/m. However, the θ_{st} and θ_{dyn} parameters were not significantly affected by the temperature increase, as they were reduced by only 2°–7°.

Table 5. Dynamic contact angle (θ_{dyn}) of the studied EO–AD systems at 20°C*

AD content, vol %	Dynamic contact angle θ_{dyn}, °				
	L-201B	L-DEG-1	L-181	L-703	LT-301
10	51.1/50.0	55.7/55.0	54.0/54.0	53.2/53.0	52.0/52.0
20	43.5/43.0	52.2/51.5	50.0/49.9	48.9/48.6	47.0/46.6
30	39.0/38.0	48.7/48.0	46.8/46.0	44.7/44.2	42.0/41.3
40	36.0/35.0	46.3/45.4	44.0/43.1	41.0/40.4	39.0/38.0

*The numerator in the θ_{dyn} values indicates the θ_{dyn} value obtained for the ED-20–AD systems, and the denominator indicates the θ_{dyn} value obtained for the DER-330–AD systems.



a b

Fig. 3. Dependence of (a) θ_{dyn} and (b) $\ln\theta_{dyn}$ for the ED-20–AD systems on the content of 1) L-DEG-1, 2) L-E-181, 3) L-703, 4) LT-301, and 5) L-201B at 20°C.

Table 6. Surface tension and contact angles of the ED-20–AD systems at 20 and 60°C*

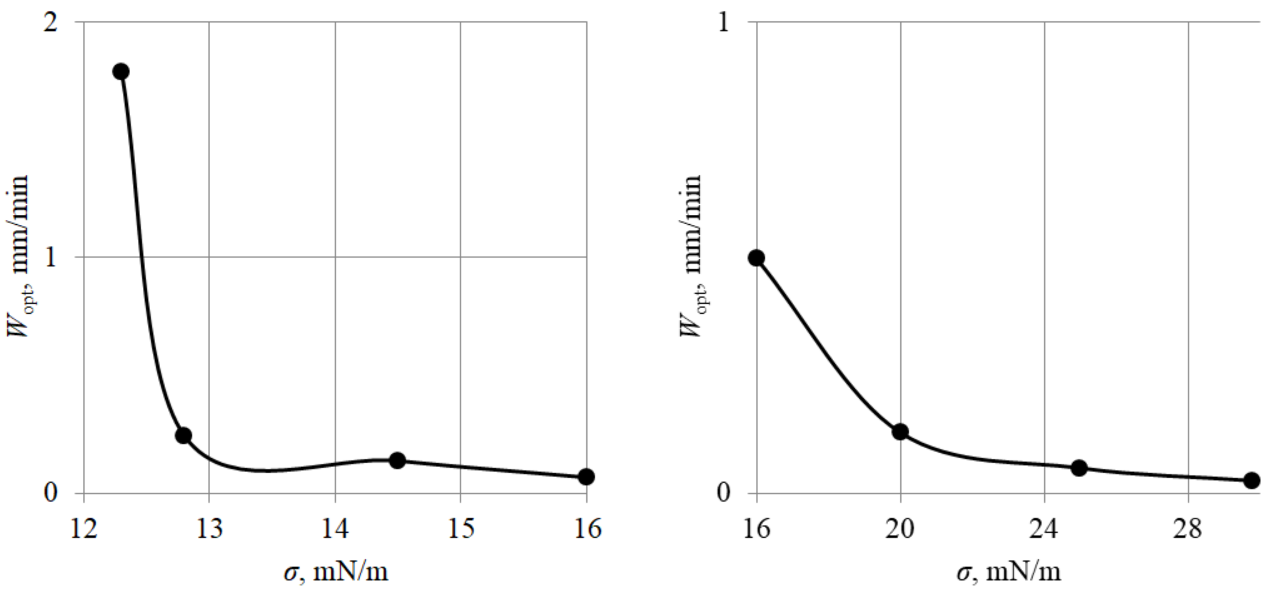
Parameters	ED-20–AD				
	L-201B	L-DEG-1	L-181	L-703	LT-301
σ, mN/m	16.0/12.3	29.8/16.0	26.0/15.0	21.2/13.8	19.5/11.8
θ_{st}, °	20/18	28/24	27/24	26/23	24/21
θ_{dyn}, °	43.5/39.7	52.2/47.5	50.0/46.6	48.9/42.7	47.0/40.0

*The numerators and denominators indicate the obtained values at 20 and 60°C, respectively.

To evaluate the effectiveness of AD in the preparation of new epoxy binders using EO–AD systems for the impregnation of reinforcing fillers used in the production of fiberglass, the optimal impregnation rate was calculated using the prediction method for the impregnation rate of frame fillers with epoxy binders during injection molding. The calculation formula [Eq. (4)] of the optimal impregnation rate (W_{opt} in m/s) involved the Deryagin criterion (*De*), which establishes a relationship between W_{opt} and the parameters of the polymer binder, i.e., σ, η, θ_{st}, and θ_{dyn}. Therefore, the optimal impregnation rate of a glass fiber filler with an EO–AD-based polymer binder could be calculated using the formula:

$$De = W_{opt} \times \eta / \sigma = 0.045 \times \left(\frac{\theta_{dyn} - \theta_{st}}{180 - \theta_{dyn}} \right)^{\frac{2}{1-\theta_{st}/180}} = 0.045 \times \left(\frac{60 - \theta_{st}}{120} \right)^{\frac{2}{1-\theta_{st}/180}} \quad (4)$$

The θ_{dyn} parameter was assumed to be 60°, as it should not exceed this value when impregnating the fibrous filler [11]. Moreover, from the graphs in Figs. 4 and 5, it is clear that the impregnation rate of the ED-20–L-201B and ED-20–L-DEG-1 systems was highly dependent on surface tension



a b

Fig. 4. Dependence of W_{opt} on surface tension for the (a) ED-20–L-201B and (b) ED-20–L-DEG-1 systems with an AD content of 20 vol % and known η and θ values.

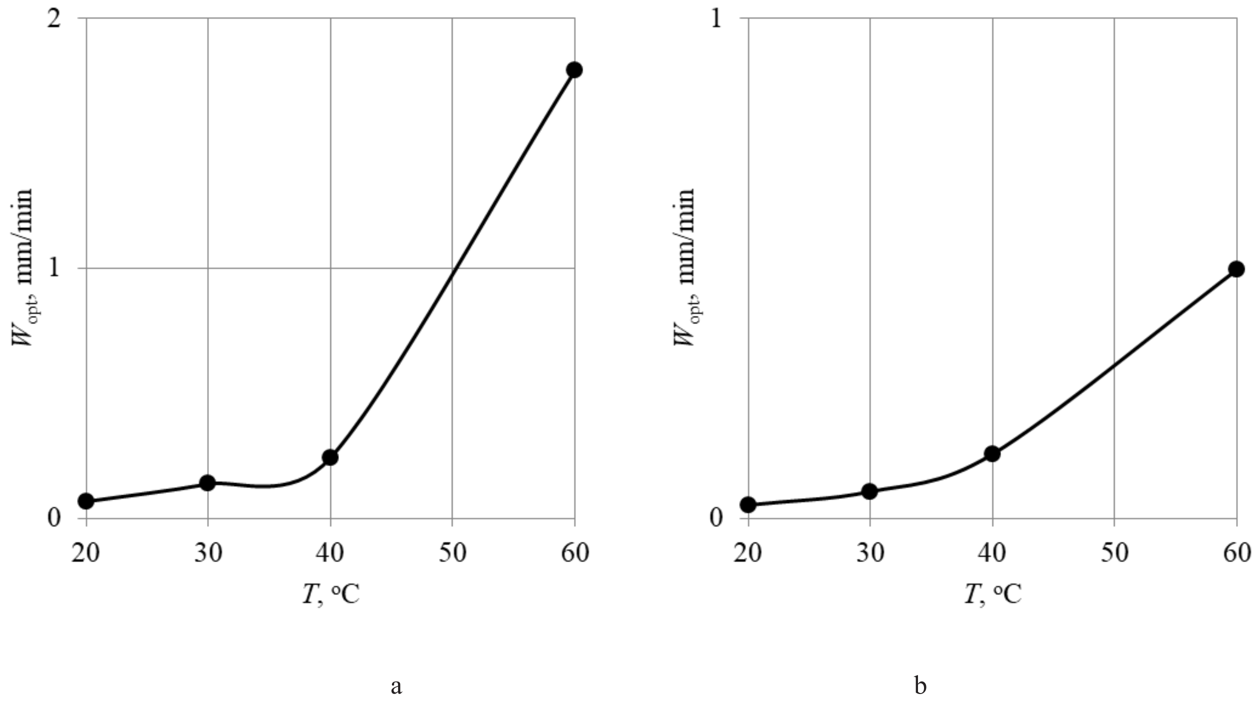


Fig. 5. Dependence of W_{opt} on the temperature for the (a) ED-20–L-201B and (b) ED-20–L-DEG-1 with an AD content of 20 vol % and η values of 0.03–0.9 and 0.9–2.4, respectively.

and temperature. In particular, the impregnation rate was significantly reduced as σ increased, whereas the temperature increase from 20 to 60°C increased the optimal W_{opt} by 10–20 times for optimal binder compositions. Moreover, according to the data of Table 6 and Fig. 5, it was revealed that the surface tension decreased at a higher temperature, which in turn led to an increase in the optimal impregnation rate.

Thus, it was concluded that increasing the temperature of optimal binder compositions based on EO–AD systems using active diluents of different nature, functionality, and molecular weight, allows the regulation of the technological characteristics of polymer binders, i.e., the surface tension, while increasing the impregnation rate of reinforcing systems by 10–20 times.

CONCLUSIONS

The surface tension and the static and dynamic contact angles of the EO, laproxides, laprolate, and EO–AD systems were obtained. These data allowed the variation of the generated systems' properties within a wide range and the regulation of the compositions and technological properties of epoxy binders and the impregnation parameters of fibrous fillers. Increasing the impregnation temperature from 20 to 60°C reduced the surface tension of the EO–AD systems by about two times, thus significantly improving the impregnation quality of fiber and frame fillers and increasing the impregnation speed by 10–20 times.

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About the authors:

Angelina D. Bresskaya, Master Student, Department of Chemistry and Technology of Plastic Processing and Polymer Composites, M.V. Lomonosov Institute of Fine Chemical Technologies, MIREA – Russian Technological University (86, Vernadskogo pr., Moscow, 119571, Russia). E-mail: angelinabresskaya@gmail.com. https://orcid.org/0000-0002-4941-2036

Dmitry A. Trofimov, Postgraduate Student, Department of Chemistry and Technology of Plastic Processing and Polymer Composites, M.V. Lomonosov Institute of Fine Chemical Technologies, MIREA – Russian Technological University (86, Vernadskogo pr., Moscow, 119571, Russia). E-mail: trofimov_d_a95@mail.ru. https://orcid.org/0000-0002-8458-3690

Igor D. Simonov-Emel'yanov, Dr. of Sci. (Engineering), Professor, Head of the Department of Chemistry and Technology of Plastic Processing and Polymer Composites, M.V. Lomonosov Institute of Fine Chemical Technologies, MIREA – Russian Technological University (86, Vernadskogo pr., Moscow, 119571, Russia). E-mail: simonov@mitht.ru. Scopus Author ID 6603181099

Sergey I. Shalgunov, Cand. of Sci. (Chemistry), Director of VNIISPV, NPO Stekloplastic (3-A, Andreevka, Solnechnogorsk district, Moscow oblast, 141551, Russia). E-mail: shsi@aha.ru. https://orcid.org/0000-0001-5893-6811

Vladimir I. Sokolov, Deputy Director of VNIISPV, NPO Stekloplastic (3-A, Andreevka, Solnechnogorsk district, Moscow oblast, 141551, Russia). E-mail: v.sokolov45@mail.ru. https://orcid.org/0000-0002-3104-3770

Об авторах:

Бресская Ангелина Дмитриевна, магистрант кафедры «Химия и технология переработки пластмасс и полимерных композитов» Института тонких химических технологий им. М.В. Ломоносова ФГБОУ ВО «МИРЭА – Российский технологический университет» (119571, Россия, Москва, пр-т Вернадского, д. 86). E-mail: angelinabresskaya@gmail.com. https://orcid.org/0000-0002-4941-2036

Трофимов Дмитрий Александрович, аспирант кафедры «Химия и технология переработки пластмасс и полимерных композитов» Института тонких химических технологий им. М.В. Ломоносова ФГБОУ ВО «МИРЭА – Российский технологический университет» (119571, Россия, Москва, пр-т Вернадского, д. 86). E-mail: trofimov_d_a95@mail.ru. https://orcid.org/0000-0002-8458-3690

Симонов-Емельянов Игорь Дмитриевич, профессор, доктор технических наук, заведующий кафедрой «Химия и технология переработки пластмасс и полимерных композитов» Института тонких химических технологий им. М.В. Ломоносова ФГБОУ ВО «МИРЭА – Российский технологический университет» (119571, Россия, Москва, пр-т Вернадского, д. 86). E-mail: simonov@mitht.ru. Scopus Author ID 6603181099

Шалгунов Сергей Иосифович, кандидат химических наук, директор ВНИИ стеклопластиков и стекловолокна (ВНИИСПВ) АО «НПО Стеклопластик» (141551, Россия, Московская область, Солнечногорский р-н, пос. Андреевка, стр. 3-А). E-mail: shsi@aha.ru. https://orcid.org/0000-0001-5893-6811

Соколов Владимир Иванович, заместитель директора ВНИИ стеклопластиков и стекловолокна (ВНИИСПВ) АО «НПО Стеклопластик» (141551, Россия, Московская область, Солнечногорский р-н, пос. Андреевка, стр. 3-А). E-mail: v.sokolov45@mail.ru. https://orcid.org/0000-0002-3104-3770

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