

THE STUDY OF THE INFLUENCE OF CHEMICAL NATURE OF FUNCTIONAL GROUPS IN OLIGOMERIC AND LOW-MOLECULAR MODIFIERS ON THE RHEOLOGICAL PROPERTIES OF THE EPOXY OLIGOMER

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Abstract. The influence of chemical nature of modifier functional groups on the level of intermolecular interactions in the system “epoxy oligomer – modifier”, as well as the structure formation and dynamic viscosity of epoxy oligomer has been studied in detail. Modifying additives in low concentrations contribute to an increase in the degree of structure formation of the epoxy system by increasing the intermolecular interaction between the associates of the epoxydiane oligomer. It was established that the strength of the formed coagulation structures depends both on the compatibility parameter of the modifiers and ED-20, and on their intermolecular interaction energy.

Keywords: epoxy oligomer, modifying additive, dynamic viscosity, intermolecular interaction, computer modeling, compatibility parameter.

1. Introduction

It is known that the increased viscosity of epoxy oligomers is determined by the level of intermolecular interactions between molecules in such polar groups as hydroxyls (the possibility of hydrogen bonds formation), epoxy and simple ethers (dipole-dipole interactions), as well as benzene nuclei (π - π interaction) [1, 2]. Therefore, epoxydian oligomers are highly viscous associated liquids [3]. The viscosity of epoxy oligomer binders determines the timing of their application and distribution on the surface, as well as reinforcing fillers impregnation. The reduction of viscosity ensures high-quality impregnation of fillers with binders and obtaining materials and coatings with a uniform structure, which affects the

performance properties. In this regard, the regulation of the epoxy binder viscosity is one of the most important stages in the production of epoxy polymeric materials. Moreover, the initial viscosity and the structure of the liquid epoxy oligomer significantly affect the kinetics of the solidification process and the subsequent formation of topological and supramolecular structures in epoxy polymers.

Modifying additives are low viscosity oligomers and plasticizers of various chemical nature containing epoxy, cyclocarbonate, complex ether, and other polar groups. They are widely used to improve the complex of technological properties of epoxy binders [4-6]. Adding polar groups of different nature to the liquid epoxy oligomer under these conditions should lead to a change in the level of intermolecular interaction, structural organization and, ultimately, dynamic viscosity. It was previously established [7-9] that in the epoxy systems studied, the structuring processes are determined by the interactions between the functional groups of oligomers that represent the active centers of Lewis and Bronsted.

Therefore, the purpose of this work is to provide a more detailed study of how the chemical nature of functional groups in modifiers influences the level of intermolecular interactions in the system “epoxy oligomer - modifier”, as well as the structure formation and dynamic viscosity of epoxy oligomer.

2. Experimental

This research studied an epoxy oligomer ED-20 ($M = 419$ g/mol). As the modifying additives were used such reactive oligomers as triglycidyl ether of oligoxypropylenetriol Laproxide-503 ($M = 481$ g / mol), tricyclocarbonate propyl ether of oligoxypropylenetriol Laprolate-803 ($M = 854$ g/mol) and dibutylphthalate (DBP) plasticizer ($M = 278$ g/mol). The amount of added

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modifying additives were 100 vol % relative to ED-20 oligomer.

Determination of intermolecular interactions in the epoxy oligomer with modifying additives was carried out by the semi-empirical calculation of PM3 [10] using the HyperChem software package for computer simulation. Three-dimensional models of intermolecular complexes were constructed. The following characteristics such as intermolecular distance, dipole moments, enthalpy of formation ΔH_{form} and the energy of interaction E_{int} of individual compounds and intermolecular complexes were obtained. The interaction energy in ED-20 + modifier complexes (AB) was determined by the difference between the heat of formation in the AB complex (ΔH_{formAB}) and the heat of formation in ED-20 (A), (ΔH_{formA}) and the modifier B (ΔH_{formB}):



$$E_{int} = \Delta H_{formAB} - (\Delta H_{formA} + \Delta H_{formB}) \quad (2)$$

To obtain data on the degree of intermolecular interactions in the “oligomer-modifier” system and the concentration dependences of the largest and smallest Newtonian viscosities, the viscosity was calculated by the additivity rule and the Bedaux method.

It is known that there is a logarithmic additivity of viscosity in a homogeneous mixture [11]:

$$\lg \eta_{mixture} = \varphi_1 \cdot \lg \eta_1 + (1 - \varphi_1) \cdot \lg \eta_2 \quad (3)$$

where φ_1 , η_1 and η_2 are the volume fraction and viscosity of the corresponding components of the polymer matrix, respectively, vol %.

The calculation of the viscosity of the non-filled biphasic mixtures was carried out by the Bedaux equation [12]:

$$\eta = \eta_1 \left\{ 1 + 2.5x_2 \frac{\eta_2 - \eta_1}{\eta_2 + 1.5\eta_1} \times \left[1 - x_2 \frac{\eta_2 - \eta_1}{\eta_2 + 1.5\eta_1} \right]^{-1} \right\} \quad (4)$$

where η_1 and η_2 are the viscosity of the continuous and dispersed phases of the mixture, Pa·s; x_2 is the volume fraction of the dispersed phase, vol %.

Dynamic viscosity of the initial components in compositions is defined experimentally at 293 K: 8.5 Pa·s for ED-20; 1.4–1.3 Pa·s for Laproxide-503; 2.9–2.1 Pa·s for Laprolate-803 and 0.19 Pa·s for dibutyl phthalate (DBP).

Evaluation of the contribution of each component of the “epoxy oligomer-modifier” mixture to the effectiveness of intermolecular interactions and the structure nature was carried out by the flow curves using Hoppler viscosimeter at the temperature of 293 K under the shearing stress up to 1800 Pa.

3. Results and Discussions

For a preliminary evaluation of the intermolecular interactions in the “oligomer-oligomeric” and “modifier-modifier” systems we calculated three-dimensional solubility parameters in the framework of the general solubility parameter, which can be represented as the sum of the values of three separate parameters [13]:

$$\delta^2 = \frac{(\Delta E_d + \Delta E_p + \Delta E_h)}{V_M} = \delta_d^2 + \delta_p^2 + \delta_h^2 = \delta_p^2 + \delta_a^2 \quad (5)$$

$$\delta_a^2 = \delta_p^2 + \delta_h^2 \quad (6)$$

where ΔE_d , ΔE_p and ΔE_h are energies that take into account dispersion, polar (dipole-dipole and induction) interactions, as well as interactions in the hydrogen bonds, kJ/mol; δ_d , δ_p and δ_h are the corresponding solubility parameters, $(\text{MJ}/\text{m}^3)^{1/2}$; δ_a^2 is an associative solubility parameter, $(\text{MJ}/\text{m}^3)^{1/2}$; V_M is a molar volume, m^3/mol .

Table 1 shows the calculated data based on the solubility parameters of the modifiers and the epoxy oligomer. It is shown that an epoxy oligomer has the greatest value of the three-dimensional parameter $\delta = 21.2 (\text{MJ}/\text{m}^3)^{1/2}$, and dispersion component makes a bigger contribution, despite the fact that the oligomers have a whole set of polar groups in their structure capable of dipole-dipole and specific interactions.

From the presented data, it follows that among the modifiers studied, the values of the solubility parameters increase from DBP containing a complex etheric polar group, to Laproxide-503 and Laprolate-803 containing 3 epoxy and 3 cyclocarbonate groups, respectively. Moreover, the dipole-dipole (δ_p) and hydrogen bonds interactions (δ_h), which determine the size of the associative parameter (δ_a), contribute to the value of the three-dimensional solubility parameter.

Table 1

Solubility parameters of an epoxy oligomer and modifying additives

Oligomer, modifiers	Dispersion component δ_d , $(\text{MJ}/\text{m}^3)^{1/2}$	Polar component δ_p , $(\text{MJ}/\text{m}^3)^{1/2}$	Hydrogen bonds interaction, $(\text{MJ}/\text{m}^3)^{1/2}$	Solubility parameter, $(\text{MJ}/\text{m}^3)^{1/2}$
ED-20	19.79	5.06	5.21	21.1
Laproxide-503	17.40	7.46	7.46	20.4
Laprolate-803	16.06	10.93	7.99	21.0
Dibutyl phthalate	16.28	9.06	5.57	19.2

The presented data show that the values of the solubility parameters of Laprolate-803 and ED-20 are the closest comparing with DBP and Laproxide-503. At the same time, it is known that the parameters δ characterize only the energy expended on separating the modifier molecules from each other; they reflect the interaction energy only between these molecules and are not always the measure of the interaction energy between the modifier and the epoxy oligomer [14].

The nature of the intermolecular interactions of ED-20 and modifying additives was studied using computer simulation by the method of the semiempirical calculation PM3 in the HyperChem program. As a result of the computer simulation, the next parameters were obtained: the intermolecular distance and dipole moments, the enthalpies of the formation ΔH_{form} and the interaction energy E_{int} of individual compounds and intermolecular complexes, which are presented in Table 2. Three-dimensional models of ED-20 molecules interacting are presented in Fig. 1 and models of ED-20 and modifiers molecules interacting are presented in Figs. 2-4. Symbols of atoms in molecular models are presented in Fig. 5.

Fig. 1 shows that the epoxy oligomer molecules are oriented by the epoxy group of one molecule towards the hydroxyl group of another molecule. The distance between the atoms of Oxygen and Hydrogen is 7.5 Å, and the energy of interaction of the molecules ED-20 is 65.58 kJ/mol. This energy value is typical of the hydrogen bonds interaction and all types of Van der Waals interactions, which usually lead to the association of molecules and to the growth of viscosity. Figs. 2-4 show computer models of molecular complexes between ED-20

molecules and the modifying additive molecule, as well as between two ED-20 molecules and one modifier molecule.

It can be seen from these figures that the molecules of Laprolate-803 and Laproxide-503 with their functional end groups containing proton-receptor oxygen atoms are oriented to the hydroxyl group of ED-20, and the interaction energy is 38.16 and 17.27 kJ/mol, respectively (Table 2). These quantities are close to the energies of hydrogen bonds. Such a difference in the values of the interaction energies is most likely due to a higher value of the dipole moment of the carbonate group (4.6 D) in the chain of the Laprolate-803 molecule when compared with the dipole moment of the epoxy group (1.8 D) in the chain of the Laproxide-503.

An aromatic ring in the DBP molecule is oriented toward the epoxy group of a single ED-20 molecule, and a butyl radical in the DBP molecule is oriented toward a hydroxyl group of an ED-20 molecule. The interaction energy of the bimolecular complex ED-20 + DBP is -8.23 kJ/mol, and the energy of the interaction of the three-molecular complex ED-20+DBP+ED-20 decreases almost by half and its value is -3.39 kJ/mol. Besides, the interaction energy of the three-molecular complex with Laprolate-803 and Lamroxide-503 also decreases, but in this case to the less extent due to three functional and flexible hydroxypropylene units in the structure.

Taking into account the obtained results of calculations of the three-dimensional solubility parameter and computer simulation of the intermolecular interactions degree in the "oligomer-modifier" system, it can be noted that Laprolate-803 is the most compatible with ED-20, while DBP is the least compatible.

Table 2

Computer simulation parameters of molecular complexes

Molecular complex	ΔH_{form} , kJ/mol	E_{int} , kJ/mol	The distance between the molecules, Å	Dipole moment μ , D
ED-20	-473.97			2.76
ED-20 + ED-20	-882.35	65.6	7.5	5.51
Laproxide-503	-1721.3			5.47
Laproxide-503 + Laproxide-503	-3443.5	0.1	9.7	
ED-20 + Laproxide-503	-2178.0	17.27	9.5	4.59
ED-20 + ED-20 + Laproxide-503	-2685.23	-7.19		7.81
Laprolate-803	-3153.31			4.68
Laprolate-803+ Laprolate-803	-6306.26	0.36	8.4	
ED-20 + Laprolate-803	-3589.12	38.16	9.0	7.41
ED-20 + ED-20+ Laprolate-803	-4085.78	-22.69		9.95
DBP	-892.64			2.65
DBP + DBP	-1785.70	-0.42	6.1	
ED-20+ DBP	-1374.80	-8.23	7.8	4.92
ED-20 + ED-20+ DBP	-1851.36	-3.39		7.18

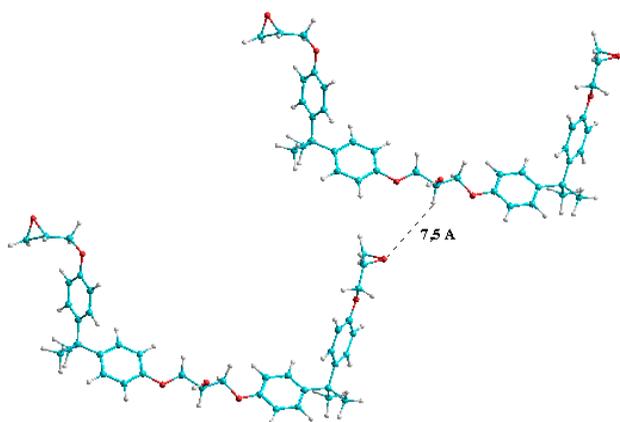


Fig. 1. Geometry of ED-20 molecules

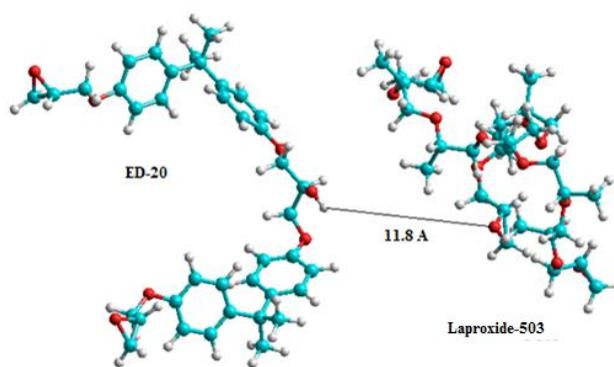


Fig. 2. Geometric optimization of the molecular complex of ED-20 and Laproxide-503

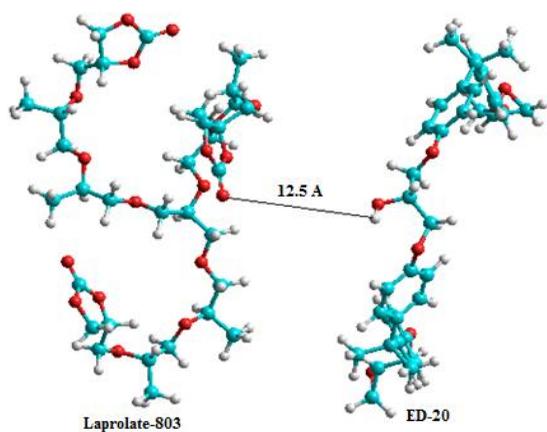


Fig. 3. Geometric optimization of the molecular complex of ED-20 and Laprolate-803

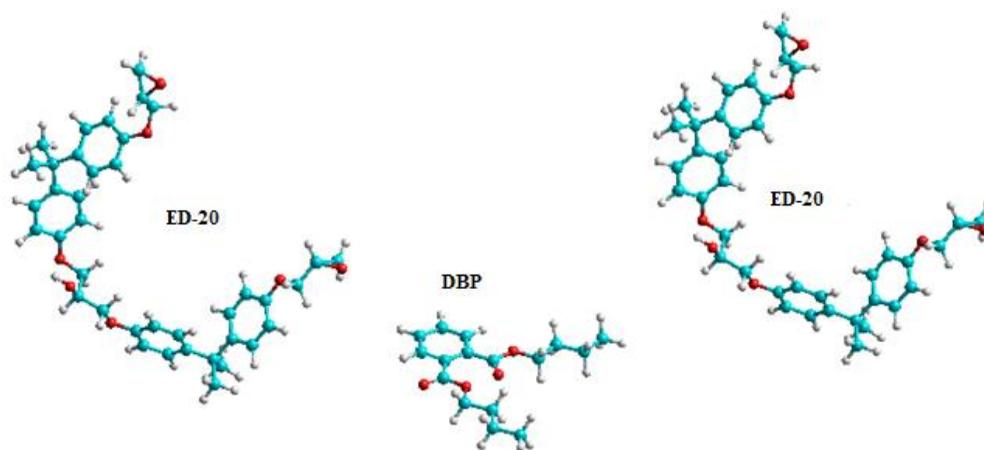


Fig. 4. Geometric optimization of the molecular complex of ED-20 and DBP

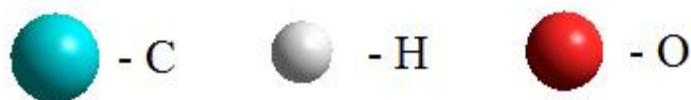


Fig. 5. Atomic conventions

For the experimental evaluation of the intermolecular interactions degree in the “oligomer-modifier” system, the rheological properties of binary mixtures were studied. The comparison of the concentration dependences of the largest Newtonian viscosities received as experimental and the data calculated by the rule of logarithmic additivity and the Bedaux method were presented.

The experimental results of the dynamic viscosity dependency on shear stresses of the epoxy oligomer ED-20 and the modifying additives Laproxide-503 and Laprolate-803, as well as mixtures with ED-20, are shown in Fig. 6.

Rheological curves allowed to determine such structural-rheological characteristics as the maximum viscosity at the initial moment of flow (η_0) with the minimally destroyed structure and the viscosity of the

“destroyed” structure at the Newtonian flow (η_n), anomaly of viscosity (η_0/η_n), the flow limit (τ_0), the shear stress at which the Newtonian flow (τ_n) begins. The obtained results are presented in Table 3

The data obtained show that the systems studied are structured liquids with the non-Newtonian character of the flow. Obtained values of the viscosity anomaly and the absence of the area with the greatest Newtonian viscosity on flow curves mean that the pure oligomers are liquids with a low degree and can be attributed to pseudoplastics. The modifying oligomers Laproxide-503 and Laprolate-803 are characterized by a significantly lower strength of the structure, compared with the epoxy oligomer, the structural viscosity range of which extends to 2800 Pa on the flow curve. These data are in good agreement with the previously calculated interaction energy between bimolecular complexes.

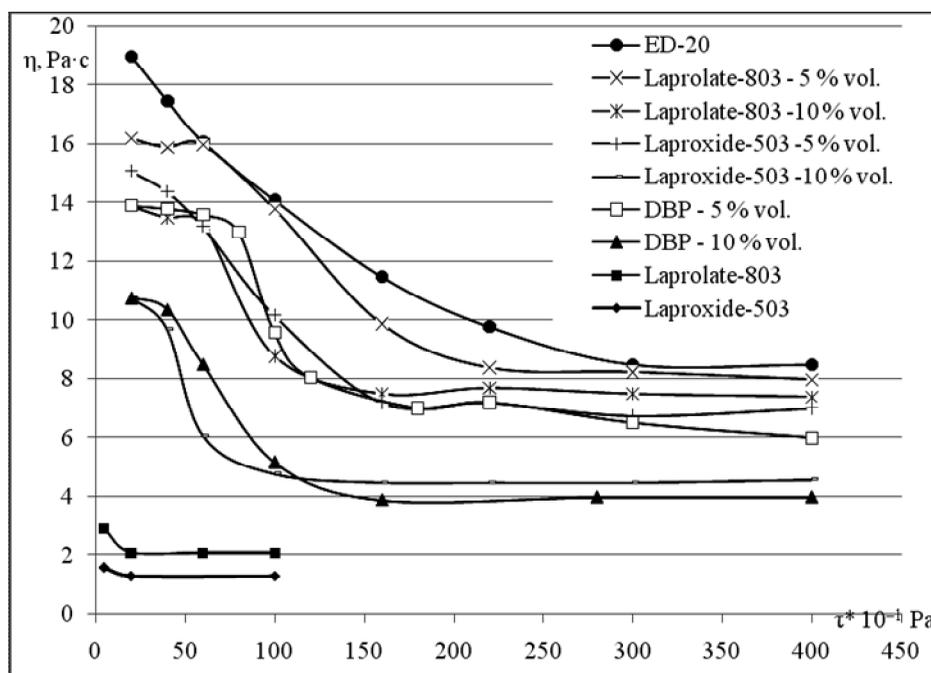


Fig. 6. The flow curves of ED-20 and compositions with modifying additives

Table 3

Structural-rheological characteristics of epoxy compositions

Composition, vol %	τ_0 , Pa	τ_n , Pa	η_0 , Pa·s	η_n , Pa·s	η_0/η_n
ED-20 – 100 % vol	200	2800	19.0	8.5	2.24
ED -20 + Laproxide -503 5 % vol	350	1600	15.1	6.5	2.32
ED -20 + Laproxide -503 10 % vol	290	750	10.8	4.5	2.4
ED -20 + Laprolate -803 5 % vol	600	2000	16.0	8.2	1.95
ED -20 + Laprolate -803 10 % vol	600	1600	13.8	7.8	1.76
ED -20 + DBP 5 % vol	800	1250	14.0	7.0	2.0
ED -20 + DBP 10 % vol	500	1200	10.5	4.0	2.63
Laprolate - 803 – 100 % vol	20	200	2.9	2.1	1.3
Laproxide - 503 – 100 % vol	20	100	1.4	1.3	1.1

The obtained results give grounds to assume that the epoxy oligomer is a strongly associative fluid. The decrease of viscosity with the growth of displacement stress at the initial stage can occur due to the weakening of the bonds between the molecule associations. Higher shear stresses can lead to the breakage of bones between associations and to their destruction.

Adding Laproxide-503, Laprolate-803 and DBP to ED-20 does not change the format of the flow curves, but there are areas with the highest Newtonian viscosity in the area with low values of shear stresses. There is a narrowing and displacement of the effective structural viscosity area towards the low values of shear stresses, especially in the presence of Laproxide-503 and DBP. In the case of adding 5 vol % of Laproplate-803 and DBP, the degree of viscosity anomaly decreases; in the case of Laproxide-503 it increases (Table 3). It is noteworthy that when adding 10 vol % of Laproxide-503 to ED-20, an area characterized by the smallest Newtonian flow appears at lower shear stresses (~ 1000 Pa) than in the presence of the same amount of DBP (~ 1600 Pa) under equal initial viscosities (η_0) of both modifiers. This factor may be a priority when we choose modifiers to make the fiber fillers soaking easier.

Fig. 6 also depicts that after adding Laprolate-803 to ED-20 the flow curve shows the area with the greatest Newtonian viscosity, which indicates the formation of rapidly restoring sufficiently strong spatial structures ($\tau_0 = 60\text{--}800$ Pa). It can be due to strong intermolecular interactions after formation of hydrogen bonds of Laproplate-803 with ED-20. This circumstance, coupled with a relatively small value τ_n , is valuable in the selection of modifying additives for the development of epoxy coatings. Despite the fact that Laproxide-503 also forms

hydrogen bonds with ED-20, those bonds are not that strong (Table 2) and the spatial structure is weak. Stronger spatial structure is formed in the case of DBP addition, but, probably, in another way. If we proceed from the concept of the associative structure of the epoxy oligomer [3], the low-molecular DBP, being incompatible with ED-20, in a small content predominantly lies in the inter-structure space of the oligomer. Thereby, the DBP provides the mobility of structural formation by weak Van-der-Waals interactions. It helps to create sufficiently stable spatial grid under conditions of low shearing stress. The spatial structure is destroyed and the viscosity of the composition decreases sharply when the displacement stress increases to 800 Pa (Table 3). This is a common feature of the poorly compatible systems.

Figs. 7 and 8 represent the experiment results of the concentration dependences at maximum and minimum Newtonian viscosity for mixtures of ED-20 with modifying additives.

Fig. 7 shows that the concentration dependence of the maximum viscosity of the ED-20+Laproxide-503 (5 vol %) mixture is in the area of the additive curve values. An increase of Laproxide-503 content leads to falls into the region below the additive dependence. This indicates the tendency to reduce the epoxy oligomer molecules association with an increase of the Laproxide-503 content due to the formation of intermolecular complexes.

The viscosity concentration dependences for mixtures of ED-20 with Laprolate-803 clearly fall into the region below the values of additive dependencies, which indicates a decrease of the self-association degree of epoxy oligomer molecules and Laprolate-803 molecules as a result of the formation of intermolecular complexes ED-20+Laprolate-803.

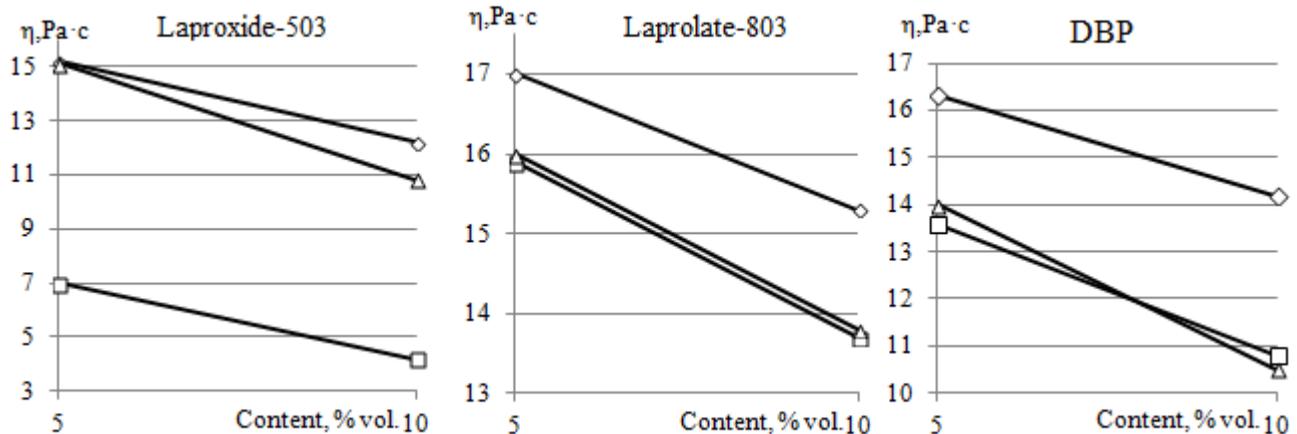


Fig. 7. Maximum viscosity concentration dependences for mixtures ED-20 with modifying additives

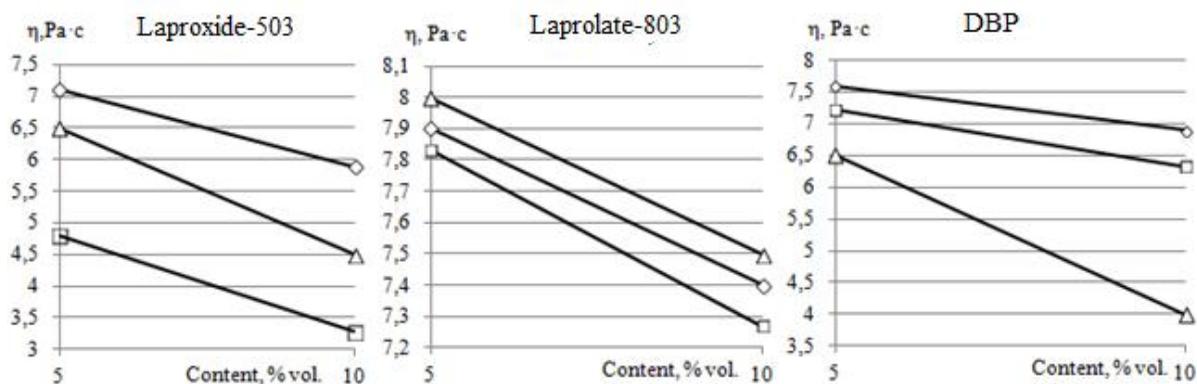


Fig. 8. Newtonian viscosity concentration for mixtures ED-20 with modifying additives

It is also evident that the viscosity concentration dependences for the ED-20+DBP mixture are located in the curve region for two-phase mixtures and with the increase in the DBP content the viscosity concentration dependences go to the region below the values typical for two-phase systems. This may indicate an epoxy oligomer and dibutyl phthalate compatibility deterioration with an increase of DBP concentration.

Rheological properties experiment on the “oligomer-modifier” mixture in the area of the minimum Newtonian viscosity (Fig. 8) showed that the viscosity concentration dependence of the ED-20+Laprolate-803 mixture is above the additive dependence. This indicates the specific interactions of the functional groups of the mixture components and the formation of strong intermolecular complexes under strong deformation. Concentration viscosity dependences for ED-20+Laproxide-503 and ED-20+DBP mixtures are below the values of additive curves. At the same time, with the increase of the Laproxide-503 content and, especially, DBP by more than 5%, the negative deviation from additive curve also increases. This may indicate a decrease of the intermolecular energy of the epoxy oligomer complexes with DBP and Laproxide-503, as compared to Laroprolate-803.

4. Conclusions

The solubility parameters of ED-20 and modifying additives were calculated. The nature of intermolecular interactions of ED-20 with modifying additives has been studied by the computer simulation. Experimental studies of dynamic viscosity of ED-20, modifying additives and mixtures were conducted. A comparison of the concentration dependences of the largest and Newtonian viscosities by experimental and calculated data was carried out.

Structural-rheological characteristics and calculated energy of interaction between molecules of epoxy oligomer showed that due to the strong hydrogen bond,

ED-20 is a strongly associate non-Newtonian fluid, with a low degree of structuring. Its relatively high structural strength is caused by the forces of associative intermolecular interaction. The initial area of effective structural viscosity is determined by weak intermolecular bonds between the oligomer associates.

It was shown that the small concentration of studied additives contributes to an increase of the structure formation degree of the epoxy system due to an increase in intermolecular interaction between the epoxy oligomer associations. It was established that the strength of the formed coagulation structures depends on both the solubility parameter of modifiers and ED-20 and their energy of intermolecular interaction.

The decrease of the modified epoxy system structural strength is caused by an increase in the fraction of the formed less stable ED-20+modifier heterocomplexes, in comparison with the strength of the complex ED-20+ED-20. This reduction is even greater, the smaller the energy of the formed complexes ED-20+modifier intermolecular interactions.

The received consistent patterns of oligomer modifiers Laproxide-503 and Laroprolate-803 influence on the structural-rheological characteristics of the epoxy oligomer can be used for the rational recipes of binders, for use as impregnating compositions or thick-layer coatings.

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ДОСЛІДЖЕННЯ ВПЛИВУ ХІМІЧНОЇ ПРИРОДИ ФУНКЦІОНАЛЬНИХ ГРУП ОЛІГОМЕРНИХ ТА НИЗЬКОМОЛЕКУЛЯРНИХ МОДИФІКАТОРІВ НА РЕОЛОГІЧНІ ВЛАСТИВОСТІ ЕПОКСИДІАНОВОГО ОЛІГОМЕРУ

Анотація. Вивчено вплив хімічної природи функціональних груп модифікаторів на рівень міжмолекулярних взаємодій в системі “епоксидний олігомер – модифікатор”, а також на структуроутворення і динамічну в'язкість епоксидного олігомеру. Модифікуючі добавки в невеликій концентрації сприяють збільшенню ступеня структуроутворення епоксидної системи внаслідок збільшення міжмолекулярної взаємодії між асоціатами епоксидіанового олігомеру. Встановлено, що міцність утворених коагуляційних структур залежить як від параметру сумісності модифікаторів з ЕД-20, так і від їх енергії міжмолекулярної взаємодії.

Ключові слова: епоксидолігомер, модифікуючий додаток, динамічна в'язкість, міжмолекулярна взаємодія, комп'ютерне моделювання, параметр сумісності.