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STRUCTURAL PARAMETERS OF SPATIALLY CROSS-LINKED COPOLYMERS OF POLYETHYLENE GLYCOL MALEATE AND ACRYLIC ACID

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Abstract. *Introduction.* The development of polymer binders for construction and composite materials requires purposeful control over the structure of spatially cross-linked polymer networks, since the internal structural parameters largely determine the mechanical, sorption, and performance characteristics of materials. In this context, the quantitative evaluation of crosslink density and the number of junctions in polymer networks as a function of system composition represents an important scientific task. *Methodology.* In this study, spatially cross-linked copolymers based on polyethylene glycol maleate modified with acrylic acid were synthesized by a "cold" radical curing method at various mass ratios of the components. The structure of the obtained materials was confirmed by Fourier transform infrared (FTIR) spectroscopy. Quantitative assessment of the structural parameters of the polymer network was performed using equilibrium swelling data of the copolymers in water. The crosslink density, number of network junctions, and average molecular weight between junctions were calculated using the Flory–Rehner equation. *Results of the study.* It was established that an increase in the polyester component content in the investigated systems leads to a decrease in the swelling degree and to the formation of a denser spatially cross-linked structure, characterized by an increase in crosslink density and a reduction in the molecular weight between network junctions. The obtained results demonstrate the possibility of purposeful control over the structural parameters and properties of polyester binders by varying the system composition, thereby opening prospects for their application in construction and composite materials.

Keywords: unsaturated polyester; "cold" curing; spatially cross-linked polymers; swelling; crosslink density; Flory–Rehner equation; molecular weight between crosslinks

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1. Introduction

Currently, when creating polymer binders for construction and composite materials, special attention is paid to the study and targeted regulation of the structure of spatially cross-linked polymer networks, since the parameters of the structural organisation of the network largely determine the durability and performance characteristics of materials [1]. For thermosetting and network polymer systems, key characteristics of the internal structure include crosslink density and molecular weight between nodes, which affect mechanical strength, deformation behaviour, sorption properties, and resistance to moisture and temperature [2].

In polyester systems based on unsaturated polyesters and reactive monomers, the curing process is accompanied by the formation of a three-dimensional polymer network with nodes that unite macromolecular chains into a single spatial structure. The number of such nodes is one of the main quantitative characteristics of the degree of cross-linking and the level of structural organisation of the material [3]. An increase in node density usually leads to an increase in rigidity and a decrease in chain mobility, while less dense networks are characterised by increased elasticity and swelling capacity [4].

For systems based on polyethylene glycol maleate (p-EGM) modified with acrylic acid (AA), the formation of a polymer network occurs as a result of radical curing involving double bonds of the polyester component and acrylic monomer. The mass ratio of the components determines the concentration of reactive groups and, accordingly, the number of nodes in the spatially cross-linked structure [5], which makes the composition of the system an effective tool for controlling its structural parameters.

Quantitative assessment of the number of nodes is an important stage of structural analysis, allowing us to move from describing the composition and experimentally observed properties to characterising the internal structure of the material [6]. One of the most informative methods of such assessment is the study of the equilibrium swelling of network polymers, in which the degree of swelling reflects the balance between the elastic forces of the polymer network and the thermodynamic interaction of the polymer with the solvent [7]. For spatially cross-linked systems, an increase in the number of nodes is accompanied by a decrease in the material's swelling capacity [1].

Based on the swelling data, the calculation of crosslink density using the Flory–Rehner equation, which links the parameters of the polymer network with the thermodynamic characteristics of the system, is widely used [8]. Despite its known limitations, this approach remains one of the most common in the study of networked polyester and acrylate materials [3]. Its application is particularly relevant for polyester binders used in construction, where an optimal combination of strength, water resistance and dimensional stability is required [2]. Analysis of p-EGM–AA systems across a wide range of compositions allows establishing the relationship between the formulation, structural parameters and operational properties of materials [5].

Thus, calculating the number of nodes in the p-EGM–AA polymer network based on swelling data is a valid structural analysis tool that allows linking the characteristics of the composition and internal structure with the functional characteristics of materials and creates a basis for further optimisation of polyester binders [3].

2. Experimental part

The research was conducted in four stages. At the first stage, the starting unsaturated polyester, polyethylene glycol maleate, was synthesized, and its average molecular weight (M_W) was determined by GPC and turbidimetry to be ~1232 Da.

In the second stage, using the 'cold' radical curing method of the initial p-EGM with acrylic acid at different mass ratios of the components, the following systems were obtained: p-EGM15 (p-EGM–AA 15:85 wt.%), p-EGM30 (p-EGM–AA 30:70 wt.%), p-EGM45 (p-EGM–AA 45:55 wt.%) and p-EGM60 (p-EGM–AA 60:40 wt.%). Benzoyl peroxide (BPO, initiator) and dimethylaniline (DMA, activator) were used as the initiating system in a mass ratio of 1.0:0.5 wt.%. Curing was carried out at a temperature of 293K without additional heat exposure [9,10]. The initial reaction mixtures were prepared by thoroughly mixing the components until a homogeneous system was obtained, after which the 'cold' curing initiation system was added and left at a set temperature until the formation of a spatially cross-linked structure was complete.

After curing, the cured samples were purified to remove unreacted monomers. Purification was carried out by sequential washing with dioxane and then distilled water. The composition of the cured polymers was determined by HPLC analysis of the resulting mother liquors in dioxane. After washing, the samples were dried at room temperature until a constant weight was achieved, which was monitored by repeated weighing.

The third stage of the study included determining the structure of the obtained systems based on p-EGM of various compositions by IR spectroscopy and determining their degree of swelling. The swelling study was carried out in distilled water. For each composition, weights of dry copolymer with a mass of ~3.0 g were used. The weights were placed in an excess of solvent and kept at a constant temperature until an equilibrium mass was established. The swelling equilibrium was considered to be reached when there was no change in the mass of the sample within the weighing error [11]. After reaching equilibrium, the samples were removed from the water, surface moisture was removed with filter paper, and they were weighed immediately.

The degree of swelling (S) of the copolymers was calculated using formula (1):

$$S = \frac{m_{swel} - m_{dry}}{m_{dry}} \quad (1)$$

where m_{swel} is the mass of the sample in a state of equilibrium swelling (after blotting the surface), g;

m_{dry} is the mass of the dry sample, g.

Each swelling measurement was performed in at least three parallel experiments. The deviation of mass values did not exceed ±2%, indicating good reproducibility of the results. The tables present the average values of the measured parameters.

At the final stage, calculations were made of the number of nodes (N) in the polymer network and the molecular mass (M_c) between them. For this purpose, to calculate the structural parameters of the polymer network, the experimentally determined value of the degree of swelling S, calculated as the relative increase in sample mass, was converted into the mass swelling coefficient Q_m according to formula (2):

$$Q_m = 1 + S \tag{2}$$

The volume fraction of the polymer in the swollen state φ_2 was determined taking into account the densities of the polymer and solvent according to equation (3):

$$\varphi_2 = \frac{1/\rho_{dry}}{1/\rho_{dry} + (Q_m - 1)/\rho_{solv}} \tag{3}$$

where ρ_{dry} is the density of the dry polymer, g/cm^3;

ρ_{solv} is the density of the solvent (for water, ρ_{solv} = 1 g/cm^3), g/cm^3.

The polymer density was assumed to be a constant value in accordance with generally accepted assumptions for calculations of spatially cross-linked polyester systems.

The assumption of a constant density of the dry polymer for all investigated compositions is based on the similarity of the chemical nature of the resulting cross-linked network structures and the absence of fundamentally different phase components in the system. Variation in the mass ratio of p-EGM and AA primarily leads to changes in crosslink density rather than to the formation of chemically heterogeneous materials.

The calculated density values for the different samples differ only slightly (Table 2), which confirms the validity of using an averaged approach. The potential error associated with this assumption does not exceed the experimental uncertainty in the determination of the swelling degree and does not affect the established qualitative trends in the variation of v_e and M_c.

The number of polymer network nodes (cross-link density) was calculated using the Flory–Rehner equation (4):

$$\nu_e = \frac{-\ln(1-\varphi_2) - \varphi_2 - \chi\varphi_2^2}{V_1(\varphi_2^{1/3} - \frac{\varphi_2}{2})} \quad (4)$$

where ν_e – concentration of network chains, mol/cm^3;

V_1 – molar volume of solvent (for water V_1=18 cm^3/mol);

χ – was selected based on literature data for polyether-acrylate systems in an aqueous medium [8].

Next, the concentration of cross-linked chains was converted into the number of polymer chain nodes per unit volume using the expression (5):

$$N = \nu_e \cdot N_A \quad (5)$$

where N_A – Avogadro's number.

The obtained values were used for quantitative comparison of the structural characteristics of copolymers of different compositions and analysis of the effect of the degree of swelling on the parameters of the spatially cross-linked network.

The following reagents (‘Sigma-Aldrich’) were used for the study: acrylic acid, benzoyl peroxide, dimethylaniline, dioxane. All reagents were of analytical grade and used without additional purification. The starting unsaturated polyester, p-EGM, was previously obtained by the polycondensation reaction of ethylene glycol with maleic anhydride using a standard method [11].

3. Results and discussion

The curing of the initial unsaturated polyester – p-EGM – was carried out using a binary ‘cold’ curing initiation system consisting of BPO and DMA (1.0:0.5 wt.%) at a temperature of 293K.

The actual composition of the obtained cured systems p-EGM15, p-EGM30, p-EGM45 and p-EGM60 was determined by HPLC. The results are presented in Table 1:

Table 1 – Dependence of the composition of copolymers and some of their parameters on the composition of the initial mixtures of p-EGM (M_1) with AA (M_2)

Composition of the initial polymer-monomer mixture, wt.%		Composition of cured systems, wt.%		Yield, %	Degree of swelling, S
M_1	M_2	m_1	m_2		
15.04	84.96	13.98	86.02	92.8	38.29±1.91
30.21	69.79	28.87	71.13	91.2	11.51±0.58
45.06	54.94	43.54	56.46	89.7	2.17±0.11
60.12	39.88	58.12	43.88	89.1	1.44±0.07

* The reported data represent average values; the relative error in the determination of the swelling degree does not exceed ±5%.

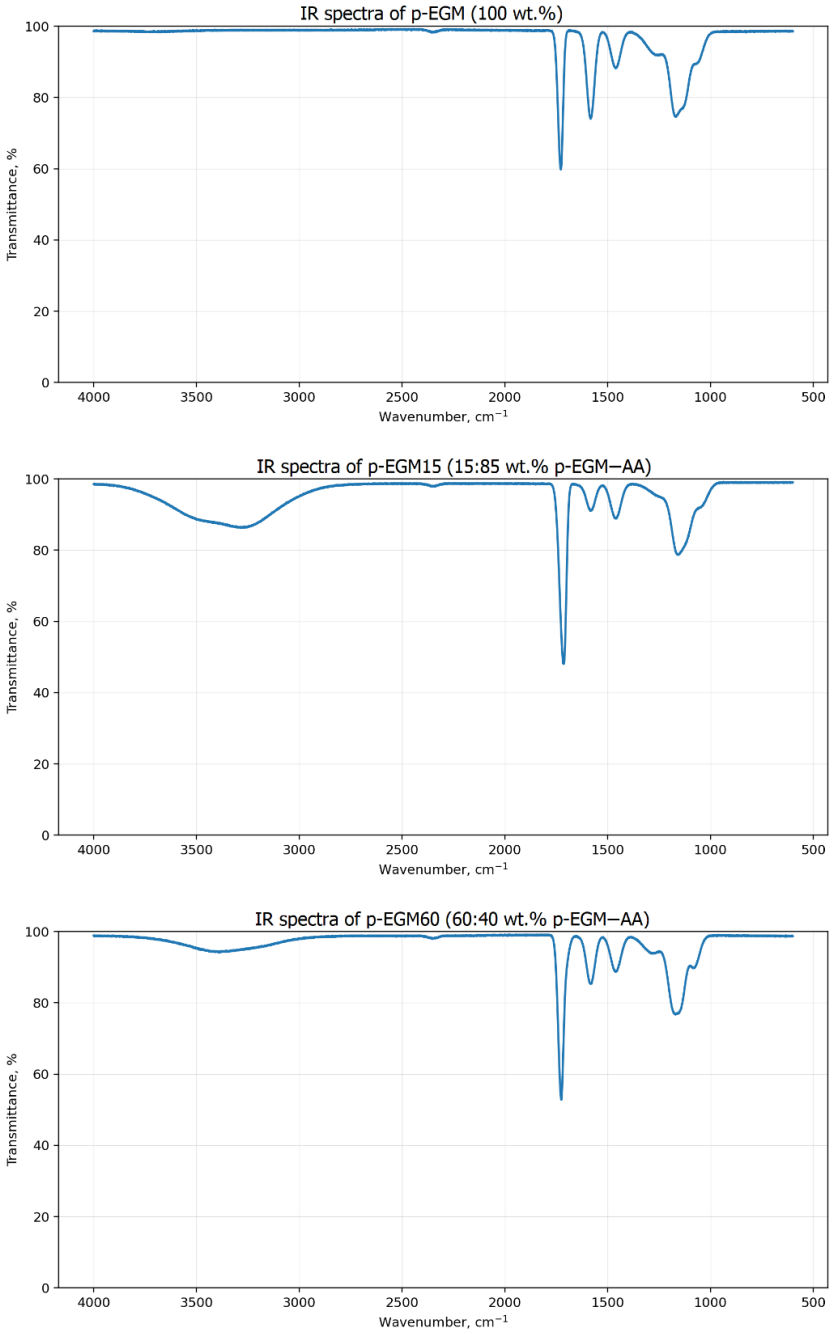

Figure 1 – IR spectra of p-EGM and copolymers based on it.

The data obtained, presented in Table 1, indicate a direct dependence of the cured product yield on the AA content. It is also worth noting that varying the AA content allows products with different physicochemical properties to be obtained, which makes them promising materials in various industries, both as effective polymer gel sorbents and as hydrophobic compounds used as polymer binders for the manufacture of building materials.

The structure of the synthesized compounds was studied using IR spectroscopy. The spectra of the initial p-EGM and p-EGM–AA copolymers of various compositions are characterized by a set of absorption bands indicating the formation of the corresponding polymer structures (Fig.1). For the initial p-EGM, an intense band of valence vibrations of carbonyl groups of ester fragments in the range of 1725–1730 cm^{-1} was recorded, as well as a band related to the vibrations of unsaturated –C=C– bonds of maleate units in the range 1575–1590 cm^{-1}. Deformation vibrations of methylene $–CH_2$ groups appear in the range 1455–1465 cm^{-1}. High-intensity bands in the range 1140–1160 cm^{-1} correspond to vibrations of ether –C–O–C– bonds of the polyester chain.

When an increased content of AA (copolymer p-EGM15) is introduced, a broad absorption band appears in the IR spectrum in the range of 3100–3600 cm^{-1}, caused by the contribution of valence vibrations of hydroxyl groups of carboxyl fragments involved in the formation of hydrogen bonds. A characteristic feature of the modification is an increase in the intensity of the carboxyl group –COOH band in the range 1710–1715 cm^{-1} compared to the initial p-EGM, which indicates an increase in the content of acrylic acid in the copolymer structure. At the same time, the band corresponding to the vibrations of unsaturated double bonds in the range 1575–1585 cm^{-1} is preserved, but its intensity is noticeably reduced, which indicates a partial copolymerization reaction involving –C=C– bonds [12].

Next, the structural parameters of spatially cross-linked copolymers of the p-EGM–AA system were calculated, which made it possible to quantitatively characterize the features of polymer network formation and establish the relationship between the composition, internal structure, and properties of the materials. The use of equilibrium swelling data in water in combination with the Flory–Rehner equation made it possible to move from a qualitative description of the behaviour of copolymers to a quantitative assessment of the density of cross-links and the molecular organization of the polymer matrix.

It should be noted that in the calculation of the crosslink density ν_e and the average molecular weight between network nodes M_c, the polymer–solvent interaction parameter χ was taken from literature data for polyester–acrylate systems in an aqueous medium [8]. It is well known that the value of χ can significantly influence the quantitative values of the calculated parameters, since it enters the Flory–Rehner equation in an exponential form.

Sensitivity analysis indicates that when χ is varied within reasonable limits (± 0.02–0.03), the absolute values of ν_e and M_c may change; however, the overall trend in the variation of structural parameters with changes in system composition

remains unchanged. Therefore, despite the possible uncertainty associated with the choice of χ, the observed relationships are robust and reliably reflect the effect of the component mass ratio on the spatial organization of the polymer network.

It should be taken into account that the Flory–Rehner equation is based on several simplifying assumptions, including an ideal network structure, uniform distribution of crosslink junctions, and the absence of structural defects such as loops, dangling chains, and local inhomogeneities in crosslink density. In real polyester–acrylate systems, deviations from the idealized model are possible due to microstructural heterogeneity and the statistical nature of radical copolymerization. Therefore, the calculated values of crosslink density and molecular weight between network junctions should be regarded as effective (estimated) parameters reflecting the averaged characteristics of the polymer network.

The experimental swelling values were reduced to a dimensionless quantity *S* (degree of swelling), reflecting the relative increase in sample mass compared to the dry state. Based on the value of S, the mass swelling coefficient Q_m was calculated, and taking into account the densities of dry copolymers, the volume fraction of the polymer in the equilibrium swollen state φ_2 was determined. Subsequent calculation of the crosslink density νe and the average molecular weight between the network nodes Mc allowed us to comprehensively characterize the structural parameters of the forming polymer network. The calculation results are presented in Table 2.

Table 2 – Structural parameters of the polymer network of copolymers of the p-EGM–AA system

Composition (wt.%) p-EGM–AA**	Q_m	ρ_{dry}, g/cm^3	φ_2	ν_e, mol/cm^3	M_c, g/mol
p-EGM15	39.29	1.2697±0.0635	0.020	$(4.90\pm0.25)\cdot10^{-6}$	$(2.59\pm0.13)\cdot10^{5}$
p-EGM30	12.51	1.2842±0.0642	0.063	$(4.40\pm0.22)\cdot10^{-5}$	$(2.92\pm0.15)\cdot10^{4}$
p-EGM45	3.17	1.3041±0.0652	0.261	$(1.18\pm0.06)\cdot10^{-3}$	$(1.11\pm0.06)\cdot10^{3}$
p-EGM60	2.44	1.3195±0.0660	0.345	$(2.58\pm0.13)\cdot10^{-3}$	$(5.11\pm0.26)\cdot10^{2}$

* The reported data represent average values; the relative uncertainty of the calculated structural parameters (ν_e and Mc) does not exceed ±5%.

** Since the numerical compositions of the cured products are quite close to those of the initial mixtures, it is customary to designate the resulting polymers according to the values of the initial compositions for clarity

Analysis of the data obtained shows that a change in the mass ratio of components in p-EGM–AA systems is accompanied by pronounced changes in the structural characteristics of the polymer network. Copolymers with a high AA content are characterized by high Q_m values and low values of the polymer volume fraction φ_2. This indicates the formation of a sparse spatially cross-linked structure in which macromolecular chains have high mobility and the number of polymer network nodes is small. Such systems are characterized by a significant

ability to sorb water, which is due to both the low density of cross-links and the presence of hydrophilic functional groups.

With an increase in the proportion of p-EGM in the composition of cured products, there is a regular increase in the volume fraction of the polymer in the equilibrium swollen state and a corresponding increase in the density of cross-links νe. For compositions with a higher polyester content, φ_2 values reach 0.26–0.35, and the crosslink density increases to about 10^{-3} mol/cm^3. This indicates the formation of a denser spatially cross-linked structure in which the mobility of macromolecular segments is significantly limited by the elastic forces of the polymer network. The decrease in the swelling ability of such systems is a direct consequence of an increase in the number of nodes and an increase in the elastic resistance of the network to solvent penetration.

Of particular interest is the analysis of the average molecular weight between nodes in the M_c network, which is an integral characteristic of the degree of structural organization of spatially cross-linked polymers. Calculation of M_c showed that as the density of cross-links increases, the value of this characteristic decreases by more than two orders of magnitude. For sparse networks with low node density, M_c values reach 10^5 g/mol, which corresponds to long and mobile macromolecular segments between cross-link nodes. At the same time, for denser networks, M_c decreases to values of the order of 10^2–10^3 g/mol, which indicates a significant shortening of the chain segments and an increase in the rigidity of the polymer matrix.

The obtained trends are consistent with modern concepts of the behavior of spatially cross-linked polymers: an increase in crosslink density leads to higher network elasticity, restricted segmental mobility of macromolecules, and a reduced swelling capacity. The calculation of the parameters ν_e and M_c makes it possible to quantitatively describe the internal organization of p-EGM–AA copolymers and to provide a well-founded interpretation of the changes in their sorption characteristics depending on composition.

Thus, the results of calculating structural parameters based on equilibrium swelling data demonstrate that varying the mass ratio of components in the p-EGM–AA system is an effective way to control the spatial organization of the polymer network. The data obtained provide a reliable basis for further analysis of the relationship between the operational characteristics of the copolymers obtained, and can also be used to justify their potential application as polyester binders for construction and composite materials.

In contrast to previously published studies on polyester network systems [3,5], the present work provides a comprehensive analysis of the effect of p-EGM–AA composition on the quantitative parameters of the network structure over a wide range of component ratios using an energy-efficient ‘cold’ curing method. This approach makes it possible to expand the understanding of the mechanisms of structural organization of polyester–acrylate networks and their regulation at the synthesis stage.

4. Conclusions

Based on the data on equilibrium swelling in water, the structural parameters of spatially cross-linked p-EGM–AA copolymers were calculated. It was found that a change in the mass ratio of the components leads to variations in the volume fraction of the polymer in the swollen state, the density of cross-links, and the molecular weight between the network nodes. It was shown that an increase in the proportion of the polyester component is accompanied by an increase in the density of cross-links and a decrease in the inter-node molecular weight, which indicates a densification of the spatially cross-linked structure. A relationship has been identified between the structural parameters of the polymer network and the swelling ability of copolymers, which is due to the balance of elastic and thermodynamic factors. The calculation of the number of nodes confirmed the informative value of the equilibrium swelling method for the quantitative analysis of the structure of polyester network systems and substantiated the prospects for the use of p-EGM–AA copolymers as polyester binders for construction and composite materials.

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ПОЛИЭТИЛЕНГЛИКОЛЬМАЛЕИНАТ ПЕН АКРИЛ ҚЫШҚЫЛЫНЫҢ КЕҢІСТІКТІК ТІГІЛГЕН СОПОЛИМЕРЛЕРІНІҢ ҚҰРЫЛЫМДЫҚ ПАРАМЕТРЛЕРІ

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Түйіндеме. *Кіріспе.* Құрылыс және композициялық материалдарға арналған полимерлі байланыстырғыштарды әзірлеу кеңістіктік тігілген полимерлік торлардың құрылымын мақсатты түрде басқаруды талап етеді, өйткені ішкі құрылым параметрлері материалдардың механикалық, сорбциялық және пайдалану сипаттамаларын айқындайды. Осыған байланысты жүйе құрамына тәуелді тігілулер тығыздығы мен полимерлік тор түйіндерінің санын сандық тұрғыдан бағалау өзекті ғылыми міндет болып табылады. *Әдістеме.* Жұмыста акрил қышқылымен модификацияланған полиэтиленгликольмалеинат негізіндегі кеңістіктік тігілген сополимерлер әртүрлі компоненттердің массалық қатынастары кезінде «суық» радикалды қатайту әдісімен алынды. Алынған материалдардың құрылымы инфрақызыл спектроскопия әдісімен расталды. Полимерлік тордың құрылымдық параметрлерін сандық бағалау үшін сополимерлердің суда тепе-теңдік ісіну деректері пайдаланылды. Тігілулер тығыздығы, тор түйіндерінің саны және түйіндер арасындағы орташа молекулалық масса Флори–Ренер теңдеуі негізінде есептелді. *Зерттеу нәтижелері.* Зерттелетін жүйелерде полиэфирлік компоненттің үлесінің артуы ісіну дәрежесінің төмендеуіне және тігілулер тығыздығының артуы мен тор түйіндері арасындағы молекулалық массаның кемуімен сипатталатын неғұрлым тығыз кеңістіктік тігілген құрылымның қалыптасуына әкелетіні анықталды. Алынған нәтижелер жүйе құрамын өзгерту арқылы полиэфирлік байланыстырғыштардың құрылымдық параметрлері мен қасиеттерін мақсатты түрде реттеуге болатынын көрсетеді және олардың құрылыс пен композициялық материалдарда қолданылу болашақтарын ашады.

Түйінді сөздер: қанықпаған полиэфир; «суық» қатайту; кеңістіктік тігілген полимерлер; ісіну; тігілулер тығыздығы; Флори–Ренер теңдеуі; түйіндер арасындағы молекулалық масса

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СТРУКТУРНЫЕ ПАРАМЕТРЫ ПРОСТРАНСТВЕННО-СШИТЫХ СОПОЛИМЕРОВ ПОЛИЭТИЛЕНГЛИКОЛЬМАЛЕИНАТА И АКРИЛОВОЙ КИСЛОТЫ

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Резюме. *Введение*. Разработка полимерных связующих для строительных и композиционных материалов требует целенаправленного управления структурой пространственно-сшитых полимерных сетей, поскольку параметры внутреннего строения в значительной степени определяют механические, сорбционные и эксплуатационные характеристики материалов. В этой связи актуальной задачей является количественная оценка плотности сшивок и количества узлов полимерной сети в зависимости от состава системы. *Методология.* В работе исследованы пространственно-сшитые сополимеры на основе полиэтиленгликольмалеината, модифицированного акриловой кислотой, полученные методом «холодного» радикального отверждения при различных массовых соотношениях компонентов. Структура полученных материалов была подтверждена методом ИК-спектроскопии. Для количественной оценки структурных параметров полимерной сети использованы данные равновесного набухания сополимеров в воде. Расчет плотности сшивок, количества узлов и средней молекулярной массы между узлами выполнен с применением уравнения Флори–Ренера. *Результаты исследования*. Установлено, что увеличение доли полиэфирного компонента в исследуемых системах приводит к снижению степени набухания и формированию более плотной пространственно-сшитой структуры, характеризующейся ростом плотности сшивок и уменьшением молекулярной массы между узлами сети. Полученные результаты демонстрируют возможность целенаправленного управления структурными параметрами и свойствами полиэфирных связующих путем варьирования состава, что открывает перспективы их применения в строительных и композиционных материалах.

Ключевые слова: ненасыщенный полиэфир, «холодное» отверждение, пространственно-сшитые полимеры; набухание; плотность сшивок; уравнение Флори–Ренера, межузловая молекулярная масса

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