

KINETICS OF LOW MOLECULAR SUBSTANCES SORPTION BY THE POLYMER MIXTURES

Mykhailo Bratyshak Jr^{1,✉}, Natalia Chopyk¹, Victoria Zemke¹

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Abstract. The sorption capacity of polymer blends was analyzed on the basis of adsorption kinetics. The study was carried out in an adsorption-active medium with different degrees of deformation. Heptane and butanol were used as adsorption-active media. The influence of the chemical nature of the adsorbate on the sorption process was studied. The free volume fractions for each sample of the composition were calculated. The ability of materials to absorb different amounts of substances allows us to identify the behavior peculiarities of the interfacial regions of different chemical compositions.

Keywords: adsorption, free volume, diffusion, crystalline polymers, polyolefins, deformation degree, low molecular weight substances.

1. Introduction

The study of the interaction of polymers with low molecular weight liquids is of great importance in their processing and operation in various types of liquid media. At the same time, the permeability of low molecular weight liquids is not least affected by external stresses and deformation of the polymer sample. In the process of solvent sorption by polymers, a sharp delineation of regions is often observed. The appearance of a transition layer on the interface between the phases of components is one of the main features of composite materials based on polymer blends. This layer significantly determines the physico-chemical and mechanical properties of polymer blends as a whole^{1–3}.

The mechanism of boundary layers formation in the polymer blends and their structures are not yet well understood. The combination of polymer deformation with adsorption of liquid low molecular substances opens up new possibilities in the study of phase-boundaries in polymer mixtures. As known, sorption in crystalline

polymers and internal mass transfer occurs only in the amorphous phase, namely, at the phase boundary and depends on the free volume⁴. The main task was to study the diffusion kinetics of low molecular weight substances in deformed samples of crystalline polymers blends.

2. Experimental

2.1. Materials

The following materials were used for the study.

DFDA 7047 ultra-high molecular weight polyethylene (UHMWPE): density, $\rho = 920\div 950 \text{ kg/m}^3$; melt flow index, MFI = 0.024 g/10 min; melting point, m.p. = 408 K; tensile yield strength is 18 MPa; relative elongation during stretching is 100 %.

Low-density polyethylene (LDPE) of 15803-020 brand: $\rho = 900\div 939 \text{ kg/m}^3$; MFI = 1.97 g/10 min; m.p. = 378–398 K; tensile yield strength is 6.8–13.7 MPa; relative elongation during stretching is 160 %.

High-density polyethylene (HDPE) of 21008-075 brand: $\rho = 948\div 959 \text{ kg/m}^3$; MFI = 6.4 g/10 min; m.p. = 403–418 K; tensile yield strength is 19–26 MPa; relative elongation during stretching is 105 %.

Polypropylene (PP) of 21060 brand: $\rho = 900\div 910 \text{ kg/m}^3$; MFI = 12.52 g/10 min; m.p. = 433–446 K; tensile yield strength is 27–35 MPa; relative elongation during stretching is 18 %.

Butanol (C₄H₉OH) is an oily, colorless liquid with a characteristic smell of safflower oil and the following properties: molar weight is 74.12 g/mol; density at 293 K is 0.810 g/cm³; index of refraction at 293 K is 1.4000; boiling point is 390.4 K; flash point is 307 K; freezing point is 158 K; the mass fraction of water is slightly more than 0.03 %.

Heptane (C₇H₁₆) is a colorless, transparent liquid with a pleasant smell and the following properties: molar weight is 116.201 g/mol; density at 293 K is 0.820 g/cm³; index of refraction at 293 K is 1.4240; boiling point is 449 K.

¹ Lviv Polytechnic National University, 12, S. Bandery str., 79013, Lviv, Ukraine

✉ mbratyshak@gmail.com

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2.2. Preparation of Mixtures

The polymer mixture for testing was obtained by melting the previously obtained mechanical mixture in a plunger injection cylinder of a vertical type at $T = 463$ K for 15 min. The next stage was squeezing the melt through a nozzle with a diameter of 2.095 ± 0.005 mm and a length of 7 mm. The resulting cooled bar was crushed using a rotary knife device. Then, the granulated mixture was loaded into the Kuasy 25×32 II/1 molding machine (Germany). To obtain a more homogeneous system the mixing was carried out in the plasticizer of the molding machine in the zones of the cylinder at temperatures of 463, 473, and 493 K for 90 s. Then the samples – blades – were obtained by pressing in the mold at $t_m = 323\text{--}343$ K.

The samples were crushed and put into a plunger injection cylinder. Then the resulting melt was squeezed out through a nozzle of $d = 2.095 \pm 0.005$ mm at $T = 463$ K for 15 min and samples were obtained in the form of rods.

2.3. The Method of Determining the Adsorption Kinetics of Solvents by Polymer Mixtures

Determination of adsorption kinetics by polymers was carried out at different degrees of elongation of the sample using the P 4–40 test machine (Germany), the main characteristics of which are: range of elongation during stretching I – from 0 to 250 mm/min, II – from 0 to 50 mm/min, velocity is 3–900 mm/min, force is 4 – 40 kN. For the tests, rods made of the corresponding material were used at a temperature of 463 K.

Rods 80 mm long and 3–4 mm in diameter were placed in an elastic sheath fixed in the bottom of the sample at a distance of 15–20 mm with a tight winding (Fig. 1).

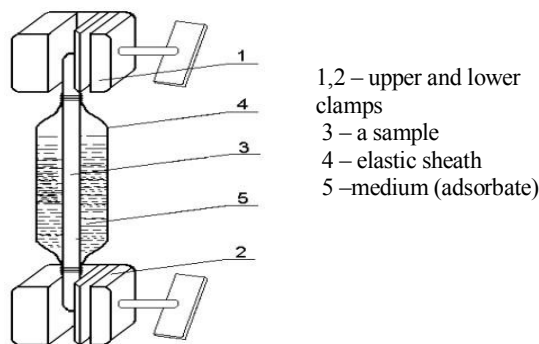


Fig. 1. Schematic representation of the deforming part of the device for determination the adsorption kinetics of samples in the adsorbate medium

After setting the required operating mode (plate speed and load), the sample was placed in the clamps of

the tensile machine and the adsorbate was poured into the elastic sheath. After that, the upper part of the elastic sheath was fixed with a thread. The study was carried out at four elongations (10, 15, 30, 80 mm), keeping the sample at each strain value for 3–5 min. After releasing the sample from the clamps and the elastic sheath, it was placed in the same environment and the mass gain was determined over four hours. Solvents of different polarity (butanol, $\varepsilon_{but.} = 17.1$, and heptane, $\varepsilon_{hep.} = 1.88$) were used to determine adsorption.^{2,5}

Experimental data on the kinetics of sample mass change were presented as a function of the relative increase in the sample mass m/m_∞ versus time, where m is the amount of substance sorbed by the sample with a radius r during time t , and m_∞ is the limit value of m at $t \rightarrow \infty$. For m_∞ we took the value m , which corresponded to the equilibrium sorption of the system. The amount of free volume in polymer blends was estimated by the degree of swelling⁶:

$$V = \frac{V_T - V_O}{V_T} \cdot 100\%, \quad (1)$$

where V_T and V_O are the valid and occupied specific volume of the medium in the polymer.

3. Results and Discussion

The study of the diffusion process in polymers was carried out based on the sorption method⁷, according to the procedure described in Subsection 2.3.

The analysis of the polymer sorption in an adsorption-active medium during deformation is based on the absorption kinetics in non-polar (heptane) and polar (butanol) liquids. When choosing an adsorption-active medium, we were guided by the fact that the nature of the liquid and its viscosity (heptane is less viscous than butanol) should determine the level of sorption of the polymer body. Thus, the low molecular weight liquids selected in this way should help to understand the structure of polymer blends and, probably, in the future, to estimate the energy of interfaces and the structure of interphase boundaries, as well as to calculate the relatively free volume in the blend.

The mechanism of low-molecular-weight liquids (LMLs) penetration into the polymer system can be explained by the so-called “hole” model, which is described by the molecular-kinetic notion of the translational movement of the diffusing part. It is divided into the mechanism of activation diffusion and submicrocapillary flow, carried out through micropores, cracks, and other cavities in the polymer, around which additional vacancies are formed. Vacancies make it possible to reduce internal

energy without requiring additional energy expenditures as far as the free volume of the polymer and liquid medium is continuously redistributed between various areas of these media⁶.

To interpret the experimental data of diffusion in polymers, the following equations^{8,9} have been used:

$$D \cong A \cdot \exp\left(-\frac{\gamma \cdot V_c \cdot V_T}{V_T \cdot (V_T - V_o)}\right), \quad (2)$$

where A is the pre-exponential factor; $\frac{\gamma \cdot V_c}{V_T} = B$;

$\left(\frac{V_T - V_o}{V_T}\right) = f$ is a share of the free volume of the medium;

$V_T - V_o = V$ is a free volume⁶; γ is an overlap factor introduced to take into account the available microcavity for different adsorbent molecules; V_T and V_o are the valid and occupied specific volume of the medium at the temperature T . Since A and B are constant, but as far as their data are unknown, then

$$\ln \frac{D^*}{D_s^*} = B \cdot \left(\frac{1}{f_s} - \frac{1}{f}\right) \quad (3)$$

where D_s^* and f_s are parameters characterizing the standard state of the adsorbing medium, which are chosen randomly. Diffusion in a polymer medium can be interpreted by the energy distribution of the free volume determined by Boltzmann statistics¹⁰.

In Figs. 2–4 the kinetics of heptane and butanol sorption by polymer mixtures are presented, respectively. Adsorption was studied using the compositions UHMWPE : additive = 50 : 50 % (w/w). As can be seen from the graphs, the compositions have different times for reaching the equilibrium sorption level. In addition, each of the compositions has a clearly different maximum sorption value.

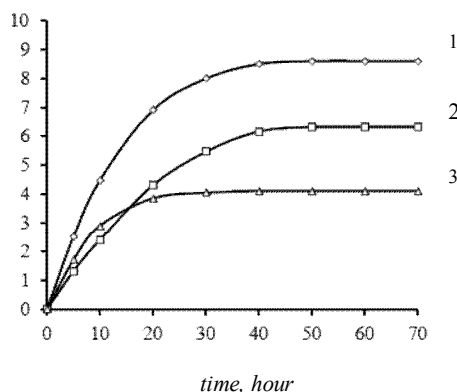


Fig. 2. Kinetics of heptane sorption by polymer mixtures at an elongation of 10 mm (1.12 %). Polymer systems: 1–UHMWPE:LDPE; 2– UHMWPE:HDPE; 3– UHMWPE:PP

Diffusion in crystalline polymers, shortly loaded in a medium to stresses below the limit of forced elasticity, is self-accelerating over a long period of time (up to 20 h). As can be seen, regardless of the particles' nature, whether of the same nature (PE – PE) or different (PE – PP), the same regularity is observed¹¹. The sorption capacity of PE mixtures is slightly higher than that of PP mixtures in heptane (Fig. 2), while in butanol the opposite dependence is observed (Figs. 3 and 4), which can be explained by both structural changes in the matrix and the nature of the adsorbate.

The rapid growth of sorption by polymer mixtures at the initial stages is caused by filling of cavities, microcracks, channels, after which the sorption capacity decreases sharply.

$$m/m_{\infty}, \% \quad (4)$$

When the saturation state is reached, the decrease in NMR sorption occurs due to a decrease in stress when the crack volume (growth rate) becomes smaller than the migration rate of the medium into the material volume, and the change in strength is also the result of the medium penetration at the boundaries of supramolecular formations and the interaction of liquid molecules with polymer macromolecules.

Despite the high crystallinity degree, which is associated with greater order and density of the structure, from the point of view of transport processes, the body acquires capillary-porous properties. Thus, the initial samples already have structures with areas of high transport speed of low molecular weight substances^{8,11}.

It should be noted, that, in general, the butanol adsorption by polyolefin mixtures is much lower than heptane adsorption. Consequently, it is confirmed that the chemical nature of the adsorbate plays a major role in the sorption process.

At the same time, when butanol is used as a sorbate, a clear dependence of the increase in the sorption capacity of the mixtures compared to the original polymers is observed. This confirms the assumption of the phase boundary role in the formation of “vacancies” into which butanol diffuses as an “inactive” solvent with low energy of intermolecular interaction with non-polar polyolefins^{11, 12}. Consequently, the total volume of the phase boundary in the mixtures increases, as well as the free volume.

The increase of heptane sorption by the mixtures, in addition to interphase interaction, may be a consequence of the higher energy of intermolecular interaction with polymers which structure is similar to that of the solvent.

For a direct correlation between diffusion and the supramolecular structure of a polymer body, we can use the density corresponding to the configuration of the free

volume, that is, we use the density as a structural characteristic of the polymer. Density, as an integral value

of the structural characteristic – a fraction of free volume, can be directly combined with mass transfer constants¹⁰.

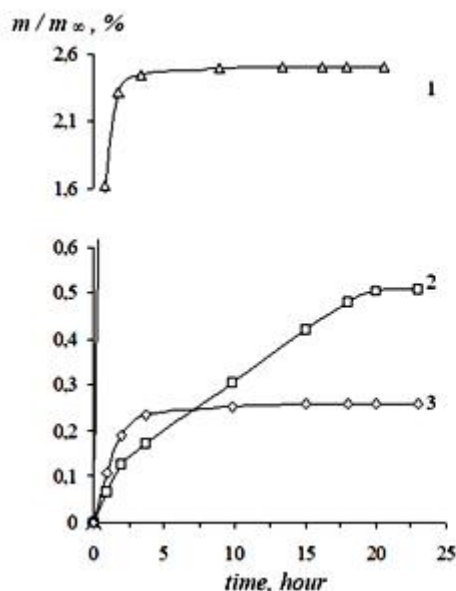


Fig. 3. Kinetics of sorption of polymer blends in butanol at elongation by 10 mm (1.12 %). Polymer systems: 1 – UHMWPE:PP; 2 – UHMWPE:HDPE; 3 – UHMWPE:LDPE

Therefore, research on the sorption capacity indicate that the change in the polymer structure is caused by the influence of the phase boundary on the permeability of the low molecular weight liquid (LMWL). Acceleration of the penetration process of LMWL into the polymer system in a stressed state is associated with the growth of material defects at the phase boundary, and as a result, the increase in the volume of vacancies^{4,12}.

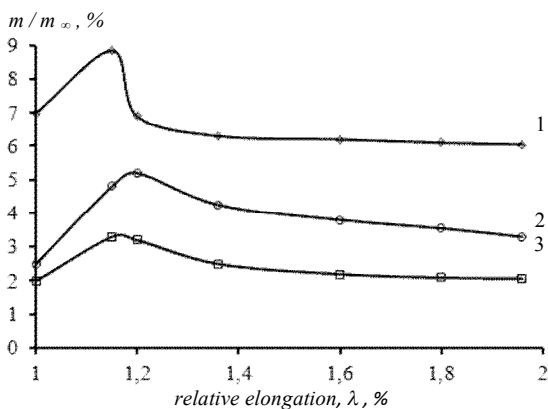


Fig. 5. Dependence of heptane sorption on the deformation degree of the initial polymers. Polymers: 1 – LDPE; 2 – UHMWPE; 3 – HDPE

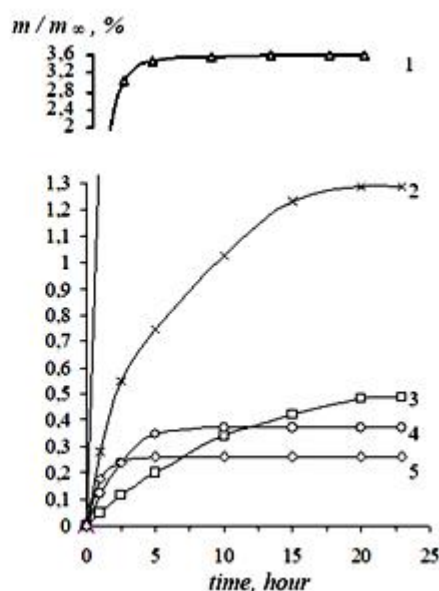


Fig. 4. Kinetics of sorption of polymer blends in butanol at elongation by 15 mm (1.19 %). Polymer systems: 1 – UHMWPE:PP; 2 – UHMWPE:LDPE; 3 – UHMWPE:HDPE; 4 – UHMWPE; 5 – LDPE

In our opinion, the stretching of samples in the medium within the limits of elastic (reversible) deformation can be the most informative when comparing the adsorption capacity of different compositions, both for assessing the homogeneity and compatibility of the studied polymers with UHMWPE, and for possible intermolecular interactions during their blending.

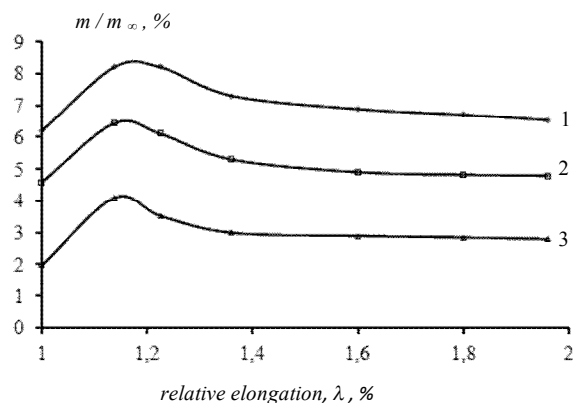


Fig. 6. Dependence of heptane sorption on the deformation degree of the samples. Polymer systems: 1 – UHMWPE:LDPE; 2 – UHMWPE:HDPE; 3 – UHMWPE:PP

Figs. 5 and 6 show the dependence of the relative increase in the weight of polymer samples on the value of relative elongation after deformation and one-hour stay in the adsorbate medium. The calculated values of the elongation degree λ are 1.12, 1.19, 1.36, and 1.96 %. It can be seen that the maximum sorption is observed for samples deformed at $\lambda = 1.12$ and 1.19 mm, after which, as the deformation increases, the value of relative increase in sorption reduces, which is agreed with the literature data.

This can be explained by the fact that the much longer UHMWPE macromolecules form more defective sites at the crystallite interface during crystallization. The relatively high sorption by low-density polyethylene supports the same view, as LDPE is known to have a lower degree of crystallinity due to lateral branching.

Fig. 6 presents the dependence of heptane sorption by polymer mixtures on the degree of deformation. Blends of UHMWPE : LDPE and UHMWPE : HDPE (Fig. 6, curves 1, 2) possess significantly higher sorption than pure UHMWPE (Fig. 5, curve 2).

Interestingly, the UHMWPE : LDPE mixture at the initial stages of deformation has the same sorption as the original LDPE (Fig. 5, curve 1). The values of initial and maximum sorption differ not much, in favor of pure LDPE at the same character of sorption from $\lambda = 0$ to $\lambda = 1.19$. Blends of UHMWPE : PP have the lowest absorption value among all tested samples (curve 3, Fig. 6) and in terms of its value is close to pure HDPE (Fig. 5, curve 3).

At the initial stages of deformation, the applied stress causes an increase in the free volume of the material promoting the acceleration of relaxation processes, which facilitates structural rearrangement in the material. During the stretching in an adsorption-active medium, the polymer undergoes a peculiar structural transition from a fragile structure to a more compact one. That is, a jumpy change in dependencies is accompanied by a transition from an amorphous to a crystalline state^{13, 14}.

As a result of this transition, the polymer acquires a characteristic structure consisting of densely packed aggregates – fibrils – oriented in the direction of the stretching axis, which makes it similar to a polymer deformed in air with the formation of a neck. In this regard, a sample deformed to high elongation, although reducing its transverse dimensions, contains a significant amount of microvoids.

Thus, the process of polymer stretching is accompanied by the formation of a kind of colloidal solution that fills the volume of microcracks and in which the dispersed phase is long and very thin fibrillated aggregates of oriented macromolecules. The dispersed

medium in the described case is molecules of an adsorption-active medium^{13, 15}. From this point of view, it is easy to explain the above-noted loss of part of the liquid captured by the polymer during its transition from a fragile structure to a more compact one. The loss of part of the dispersed medium is a phenomenon mainly inherent for the colloidal systems. It is explained by the fact that during the gel formation between the elements of the structure, a relatively small number of contacts is formed, which does not correspond to the extremely dense state of the system. Then, as a result of rearrangements of structural elements caused by their thermal movement, the number of contacts increases, which constantly leads to the compression of the gel and the squeezing out of the dispersed medium.

Obviously, for the captured liquid to be released into the surrounding space in the process of reducing sorption, it is necessary to provide its viscous flow through the porous polymer structure. The transport properties of the adsorption-active medium significantly affect the transition kinetics from a fragile structure to a more compact one. That is why the maximum on the dependence curves of the trapped liquid amount on the deformation degree shifts to the area of higher elongations. The described phenomenon has a kinetic nature and caused by the complications having a liquid of a certain viscosity when it flows through very narrow pores in microcracks^{13, 16}.

In Figs. 7 and 8 the dependences of the relative increase in polymer sample weight in butanol depending on the deformation degree have been presented.

It is noticeable that the total amount of sample sorption in butanol (a polar substance) is much smaller than in heptane (a non-polar substance that is similar in structure to polyethylene). The maximum value of sorption is observed for the same samples in the same intervals as in heptane, which indicates the same nature of absorption at the initial stages of deformation. It should be noted that the highest value of sorption is observed for a blend of ultrahigh molecular weight polyethylene with polypropylene, apparently due to the specific behavior of the given composition, which is manifested in distinct fibrillization.

According to our observations, the deformation of the samples in contact with liquid leads to more intensive formation of microcracks than when deformed in air. This is evidenced by a significant difference in the weight increase of samples deformed in liquid (Figs. 5–8) compared to those deformed in air.

In addition, the samples deformed in air have a larger value of relative deformation during destruction,

which causes changes in the activation volume and, accordingly, the speed of the diffusion process^{12, 16}.

A decrease in the sorption strength of polymer samples, that is, a change in mechanical characteristics, can be considered as the implementation of elementary

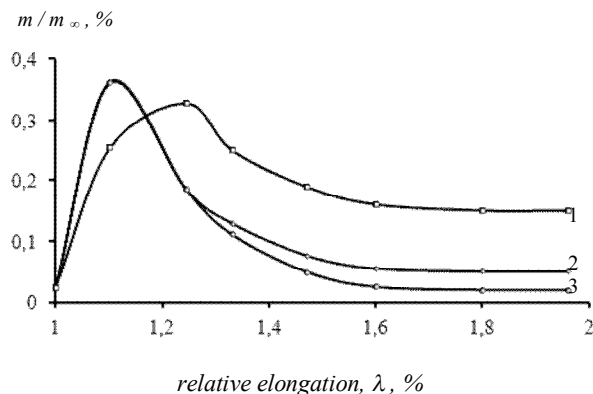


Fig. 7. Dependence of butanol sorption on deformation degree of the initial polymers. Polymers: 1 – LDPE; 2 – UHMWPE; 3 – HDPE

acts of destruction and reconstruction of bonds involving adsorbed-active atoms.

The obtained results are represented in Table, which shows that the highest sorption capacity is possessed by the blends placed in a heptane medium.

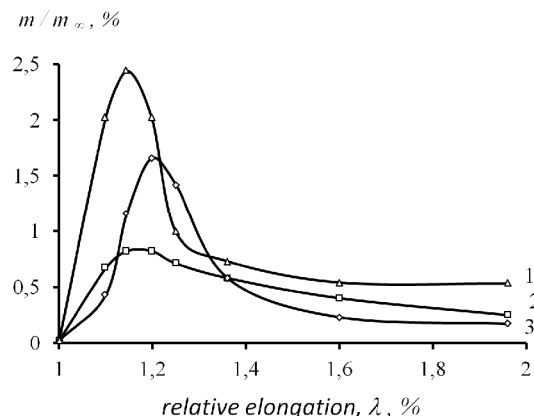


Fig. 8. Dependence of butanol sorption on the deformation degree of samples. Polymer systems: 1 – UHMWPE:PP; 2 – UHMWPE:HDPE; 3 – UHMWPE:LDPE

Table. Sorption of low molecular weight liquids by UHMWPE-based polymer blends

elongation, mm blends	Heptane					Butanol				
	0	10	15	30	80	0	10	15	30	80
LDPE	0.5	8	6.5	broken	–	0.02	0.38	0.25	0.24	0.23
HDPE	0.4	2.4	3.0	7.8	8.5	0.019	0.35	broken	–	–
PP	0.45	13.4	broken	–	–	0.007	0.17	broken	–	–
UHMWPE	0.2	3.0	4.8	4.2	broken	0.01	0.24	0.35	0.58	broken
UHMWPE : LDPE	0.3	8.5	8.0	7.1	5.7	0.012	0.25	1.3	0.33	0.68
UHMWPE : HDPE	0.27	6.3	6.4	8.5	7.0	0.025	0.51	0.48	0.14	broken
UHMWPE : PP	0.51	4.0	3.4	broken	–	0.1	2.45	3.5	broken	–

* The ratio of components in a 1:1 blend

The transfer of the sorbent mainly occurs along the boundary of the spherulites, *i. e.*, the volume between the spherulite formations (free volume) is more permeable than the inner surface of the spherulites. When stretching, due to the heterogeneity of the structure, a significant concentration of stresses occurs in the weakest place. Macroscopically, this leads to inhomogeneity of deformation in the volume of the crystalline polymer as a whole and manifests itself in the appearance of a neck at some point. This kind of local deformation at the stress concentrator can easily occur due to the slowness of relaxation processes in crystalline polymers. In such a stress concentrator, the macromolecules are locally oriented relative to each other.¹⁵

Based on the dependence of the diffusion process in polymers proposed above (Eq. 2), the calculated values

of the fraction of free volume (f) for the initial UHMWPE polymer is 0.81 %, for polymer additives: LDPE – 1.6 %, HDPE – 1.4 %, PP – 0.5 %. During the butanol sorption for UHMWPE : LDPE, UHMWPE : HDPE, and UHMWPE : PP blends with 50:50 weight ratio the fraction of free volume is 0.92 %, 1.7 %, and 5.12 %, respectively. Large f values were obtained during the sorption of heptane by polymers, which indicates the swelling of the samples under study¹⁶.

Diffusion properties in crystalline polymer mixtures are similarly based on the permeability of low molecular weight substances to the porous medium⁷:

$$D = D_{am} / \tau \cdot \beta_s, \quad (4)$$

where τ is the deterioration factor of the diffusion path of the diffusing molecule, $\tau = 1 + (1 - \varphi_{am}) \cdot \chi^{-1}$ or $\tau \cong \varphi_{am}^{-n}$;

β_s is a parameter taking into account the limitations in the segmental mobility of macromolecules in the amorphous state areas of the polymer; D_{am} is the diffusion coefficient in an amorphous polymer or an amorphous analog; φ_{am} is the content of amorphous regions; n is constant (for PP $n = 1.61$, PE $n = 1.25$); χ is crystal shape factor.

In crystalline blends, transport consists of two processes: diffusion in the amorphous part and Knudsen-type diffusion through crystallite defects^{7, 17}. Based on the given data, we can conclude about the complex nature of the interphase areas in the mixtures of the researched polymers. The sorption capacity during the deformation of polymer blends in an adsorption-active medium depends on the rearrangement and alignment of the height of the crystal folds. Thus, this explains the loosening in the crystal body due to the formation of microcavities, the packing density of the original chains and defects.

4. Conclusions

Studies have shown that the change in the structure of the polymer is caused by the influence of the phase boundary on the permeability of a low molecular weight liquid.

The growth of the phase boundary in the blends has a clear dependence on the composition content, based on which it is possible to estimate their ability to form a homogeneous mixture.

It was determined that mixtures of ultrahigh molecular weight polyethylene with polypropylene possess the highest value of sorption capacity.

The values of the free volume fraction for the starting polymers and their mixtures were calculated. It was found that the sorption capacity in the heptane medium depends on the value of the free volume.

The study of the kinetics of diffusion of low molecular weight substances in deformed samples of polymer mixtures allows us to reveal the peculiarities of the behaviour of interfacial regions of different chemical structures.

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КІНЕТИКА СОРБЦІЇ НИЗЬКОМОЛЕКУЛЯРНИХ РЕЧОВИН ПОЛІМЕРНИМИ СУМІШАМИ

Анотація. На основі кінетики поглинання проаналізовано сорбційну здатність сумішей полімерів. Дослідження здійснено в адсорбційно активному середовищі з різним ступенем деформації. Функцію адсорбційно активного середовища виконували гептан і бутанол. Розраховано частки вільного об'єму для кожного зрзкма. Здатність матеріалів адсорбувати різну кількість речовин дає змогу виявити особливості поведінки міжфазних областей різної хімічної будови.

Ключові слова: адсорбція, вільний об'єм, дифузія, кристалічні полімери, поліолефіни, ступінь деформації, низькомолекулярні речовини.