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RELAXATION OF ELECTRIC CHARGE IN POLYMER BLENDS BASED ON LOW-DENSITY POLYETHYLENE AND COPOLYMER OF ETHYLENE WITH VINYL ACETATE

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Abstract. This paper presents the results of a study of polymer films based on a blend of low-density (high-pressure) polyethylene (LDPE, HPP) with a copolymer of ethylene and vinyl acetate (EVA, sevilen). The use of thermal activation, infrared and dielectric spectroscopy methods made it possible to describe the electric charge relaxation processes in the polymer blends investigated. The data obtained suggested the presence of an α -relaxation process in the samples in the temperature range 250–280 K. An increased value of the activation energy of this process was also found in the LDPE/EVA samples compared to that in the LDPE one. This effect has been interpreted as the appearance of deeper traps of charge carriers in the blends. The dependences obtained by dielectric spectroscopy indicated the presence of hopping conductivity in the subjects of research.

Keywords: polyethylene, copolymer of ethylene with vinyl acetate, thermostimulated depolarization, thermo-activation spectroscopy

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Научная статья

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РЕЛАКСАЦИЯ ЭЛЕКТРИЧЕСКОГО ЗАРЯДА В ПОЛИМЕРНЫХ СМЕСЯХ НА ОСНОВЕ ПОЛИЭТИЛЕНА ВЫСОКОГО ДАВЛЕНИЯ И СОПОЛИМЕРА ЭТИЛЕНА С ВИНИЛАЦЕТАТОМ

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Аннотация. В статье представлены результаты исследования полимерных пленок на основе смеси полиэтилена высокого давления (низкой плотности) (ПЭВД) с сополимером этилена и винилацетата (СЭВА, сэвилен). Использование методов термоактивной, инфракрасной и диэлектрической спектроскопии позволило описать процессы релаксации электрического заряда в исследуемых полимерных смесях. Исходя из полученных данных, было сделано предположение о существовании в образцах процесса альфа-релаксации в области 250 – 280 К. Обнаружено повышенное значение энергии активации, соответствующей релаксационному процессу, в образцах ПЭВД/СЭВА, по сравнению с образцом ПЭВД. Этот эффект трактуется как появление в смесях более глубоких ловушек носителей заряда. Зависимости, полученные методом диэлектрической спектроскопии, свидетельствуют о наличии в объектах исследования прыжкового механизма проводимости.

Ключевые слова: полиэтилен, сополимер этилена с винилацетатом, термостимулированная деполяризация, термоактивная спектроскопия, дипольно-сегментальная релаксация

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Introduction

The electret effect is observed in dielectrics and consists in the ability of a material to maintain a polarized state for a long time in the absence of an external electric field [1]. Today, electrets are successfully used in various fields of science and technology. The most popular fields of application are electronics (microphones, electromechanical transducers, sensors), high-tech industry (electret filters), medicine (applicators, implants), etc. [1 – 3].

Low-density polyethylene (LDPE) is used to produce electrets because it has good dielectric properties and is inexpensive to produce. However, the disadvantage of polyethylene is the relatively short relaxation time of the electret state [1]. One of the most successful ways to improve the electret properties of polymers is the creation of composite materials by introducing chemical additives, foaming, mixing with solid dispersed fillers or with other polymers [4 – 7]. The method of combining one or several layers of polymer films is also promising [9]. These techniques make it possible to improve a set of properties of polymers, including electrical, mechanical and deformation characteristics.

Currently, the attention of researchers is attracted to a material called EVA, which is a copolymer of ethylene and vinyl acetate (EVA, SEVA, sevilen). The properties of EVA are determined by the vinyl acetate content. It is known that elasticity and adhesive properties of material are improved if it contains vinyl acetate. Improving elasticity has practical implications for electret material when it is used for active packaging, electret patches, and also as a component for stretchable bioelectronics [10, 11].

Earlier studies of the electret properties of EVA and its compositions with polyethylene [8, 9] did not provide a clear answer to the question of how the vinyl acetate content affects the dielectric properties and stability of the electret state. Nevertheless, it can be assumed that the EVA content has a positive effect on the electret properties of the composites.

In this connection, the purpose of our work was to identify the features of polarization processes in mixtures of LDPE and EVA using dielectric and thermal activation spectroscopy.

Research methods and materials

Blends of LDPE with EVA copolymer, differing in the ratio of components, were selected as samples for research. The film samples had a thickness of about 300 μm .

The blending of polymers was carried out on laboratory microrollers at a temperature of 130 ± 5 $^{\circ}\text{C}$ for 3 min. The films were prepared by pressing method at a temperature of 170 ± 5 $^{\circ}\text{C}$ for 5 min. Samples of blends of LDPE and EVA were provided by the Kazan National Research Technological University (Republic of Tatarstan, Russia).

The electrical properties were studied using thermal activation, infrared and dielectric spectroscopy methods.

Temperature dependences of thermostimulated depolarization (TSD) currents were recorded in the heating mode at a constant speed on a TSC-II installation (Setaram). Attenuated total internal reflection (ATR) spectra of the samples under study were recorded using an FSM 1202 Fourier spectrometer (an infrared range). Dielectric spectroscopy measurements were carried out using a Concept 41 installation (Novocontrol Technologies GmbH&Co).

Experimental results and discussion

The study of the frequency dependence of the imaginary part of the complex permittivity ϵ'' (the loss factor) revealed the existence of a dispersion maximum of this value in a wide temperature range (253 – 283 K). The shift of the ϵ'' maximum to the region of higher frequencies with increasing temperature was detected (Fig. 1). This shift of the maximum is typical of dipole-segmental losses (α -relaxation). It is observed in the mentioned graphs for nonpolar polymers and is bound up with orientational rotations of the polar units of the macromolecule [12]. Polyethylene contains a small number of polar groups (carbonyl and hydroxyl ones $>\text{C}=\text{O}$, $-\text{O}-\text{H}$, etc.). It also contains methyl groups $-\text{CH}_3$ and $>\text{C}=\text{C}<$ bonds, which have small dipole moments. A rise in the maximum on the frequency dependence curves obtained for a mixture of polymers was also observed with increasing temperature (Fig. 1, b). The maximum value of ϵ'' for the mixture is several times higher than that for the original polymer.

The relaxation process was also observed in the TSD spectra over the temperature range 273 – 283 K (Fig. 2). The course of the process does not depend on the percentage of blend components, and the maximum height escalates with increasing proportion of EVA. This behavior of the TSD peak can be explained by an increase in the number of relaxers in the polymer. It is also known from Ref. [13] that the height of the maximum related to α -relaxation on the frequency dependence of the loss factor ϵ'' is determined by the number of dipoles and their moments.

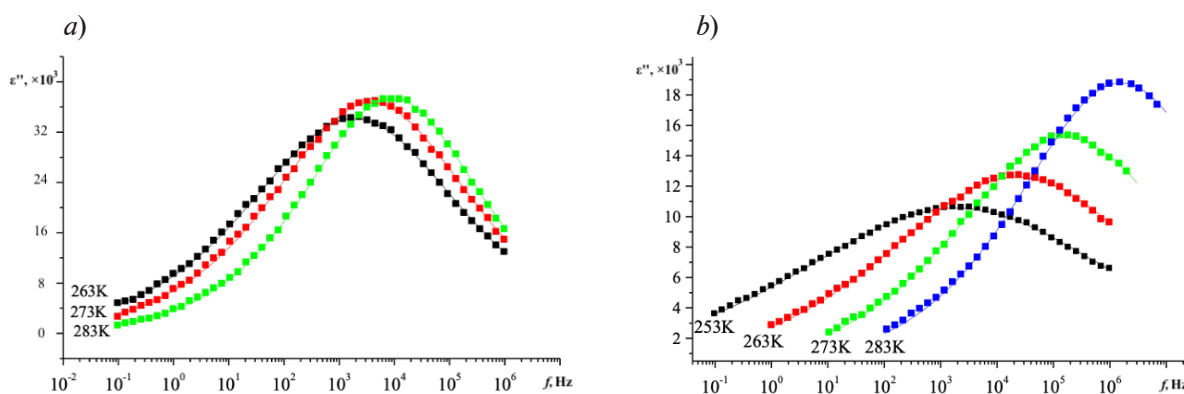


Fig. 1. Frequency dependences of the loss factor for LDPE (a) and LDPE+EVA (b) samples at different temperatures

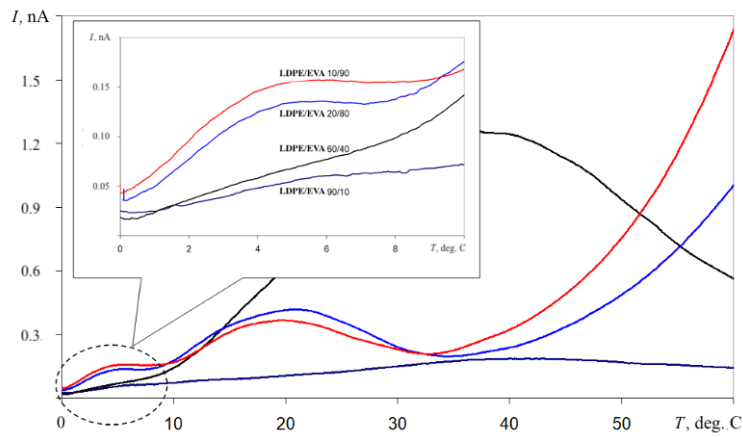


Fig. 2. Temperature dependences of TSD currents for various ratios of LDPE and EVA

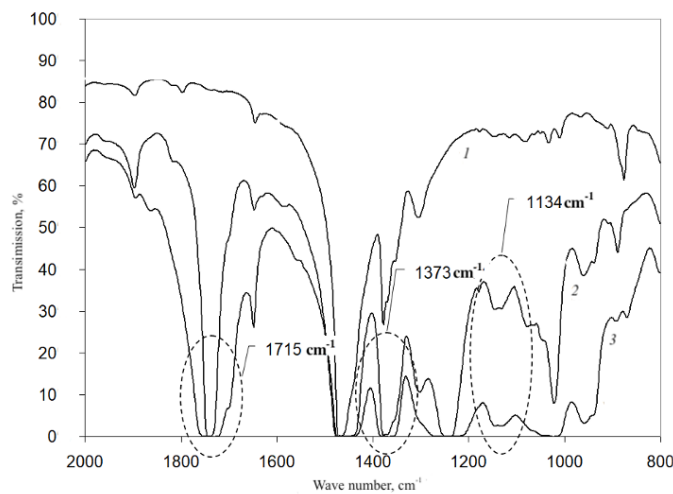


Fig. 3. Infrared transmission spectra for the samples of LDPE (1), LDPE/EVA 90/10 mixtures (2), and EVA (3)

Mixing the LDPE and EVA polymer materials led to the appearance of side polar groups $-\text{O}-(\text{C}=\text{O})-\text{CH}_3$ in the polymer chain. There are a lot of transmission bands in the IR spectra for EVA samples and LDPE/EVA mixtures (Fig. 3). For example, one can observe stretching vibrations of the $>\text{C}=\text{O}$ groups of polyvinyl acetate (1715 cm^{-1}), bending vibrations of the $-\text{CH}_3$ bonds of the acetyl group (1373 cm^{-1}), stretching vibrations of the $\sim\text{C}-\text{O}-\text{C}\sim$ ether groups in the composition of polyvinyl acetate (1134 cm^{-1}). Thus, the number of dipoles that contributed to dipole-segmental relaxation increased with increasing the proportion of EVA in the blend.

Complex dispersion dependences of dielectric parameters and features of the disordered structure of a polymer system suggest that the detected relaxation processes should be bound up with the existence of not only one relaxation time, but with a whole set of them.

The Havriliak – Negami (HN) model was used to study the features of the spectrum of relaxers. The Havriliak – Negami function is an empirical modification of the Debye relaxation model. Nevertheless, unlike the Debye model, the HN model explains the asymmetry and width of the dielectric dispersion curve. It also establishes a relation between the dispersion of permittivity in the polymer systems and relaxation parameters [14 –16]:

$$\varepsilon^*(\omega) = \varepsilon_\infty + \frac{\Delta\varepsilon}{\left[1 + (i\omega\tau)^{\alpha_{\text{HN}}}\right]^{\beta_{\text{HN}}}}, \quad (1)$$

where ϵ_∞ is the high-frequency limit of the real part of the dielectric constant; $\Delta\epsilon$ is the dielectric increment (the difference between the low-frequency limit and high-frequency one); $\omega = 2\pi f$; α_{HN} , β_{HN} are the shape parameters describing, respectively, the asymmetric and symmetric expansions of the relaxation time distribution function (RTDF); in this case $\alpha = 1.00$ is the Cole – Davidson distribution, and $\beta = 1.00$ is the Cole – Cole one.

The HN parameters for the studied relaxation processes were determined according to Eq. (1) using Winfit 3.3 software (Novocontrol Technologies GmbH). The obtained values of relaxation parameters are presented in Table. Taking into account the approximation error, we can conclude that a non-Debye oscillatory process (with a set of relaxation time values) obeying the Cole – Cole model over the studied ranges of frequency and temperature is observed in LDPE samples and LDPE/EVA blends.

Table
Temperature dependences of relaxation parameters for the polymer systems under study

| Polymer system | Average temperature, K | α_{max} , μs | β_{HN} |
|----------------|------------------------|--------------------------|--------------|
| LDPE | 263 | 0.8561 | 0.33200 |
| | 273 | 0.4185 | 0.35310 |
| | 283 | 0.1748 | 0.39110 |
| | 293 | 0.8419 | 0.35620 |
| LDPE/EVA | 253 | 0.8522 | 0.22790 |
| | 263 | 0.7116 | 0.27470 |
| | 273 | 0.9217 | 0.32050 |
| | 283 | 0.1016 | 0.34650 |

Footnote. Parameter $\beta_{HN} = 0.10000$ at all specified temperatures for the both systems.

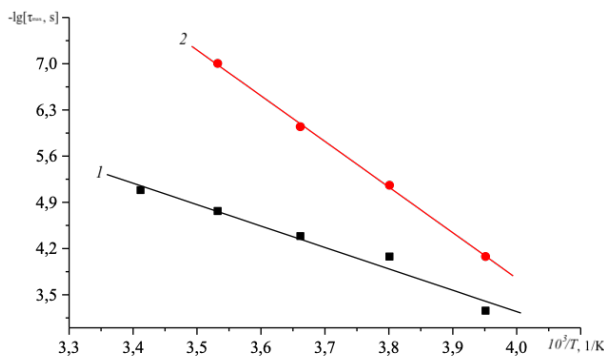


Fig. 4. Temperature dependences of the most probable relaxation time τ_{max} for the samples of LDPE (1) and LDPE/EVA (2); the found values of activation energies E_a are 0.69 eV (1) and 1.37 eV (2)

temperature [20]. This mechanism involves charge carriers jumping through a disordered structure and overcoming energy barriers. It should be noted that the fundamental parameter in this process is the energy required to complete the jump. It is also necessary to take into account the distributions of the energy barriers magnitudes.

The activation energy E_a was determined from the temperature dependence of the most probable relaxation time τ_{max} (Fig. 4) for the samples of the both systems. It turned out to be equal to 0.69 eV and 1.37 eV for the LDPE and LDPE/EVA samples, respectively. An approximately twofold increase in the E_a value for the blend samples may be bound up with the appearance of deeper traps in the energy spectrum. The appearance of these traps may be caused by two reasons. The first one is the rotation of the polar groups of the EVA molecules. The second one is the charge accumulation at the polymer boundary in the blends of LDPE and EVA.

The frequency dependence of the specific conductivity σ' for LDPE sample and the blend of two polymers is characterized by a growth in σ' with an increasing field frequency (Fig. 5). This corresponds to the power-law dependence in the form $\sigma'(\omega) \sim \omega^s$. In this case, the s value increases with increasing temperature (Fig. 6).

When s value becomes greater than 1, the movement of charge carriers is considered localized [17, 18]. These results can be explained within the theory of nonstationary hopping electrical conductivity in disordered systems [19].

In the system in question, there is a hopping mechanism of charge transfer at the indicated values of frequency and temperature. The signs of such a mechanism are the power law of conductivity dispersion and the characteristic change in exponent with

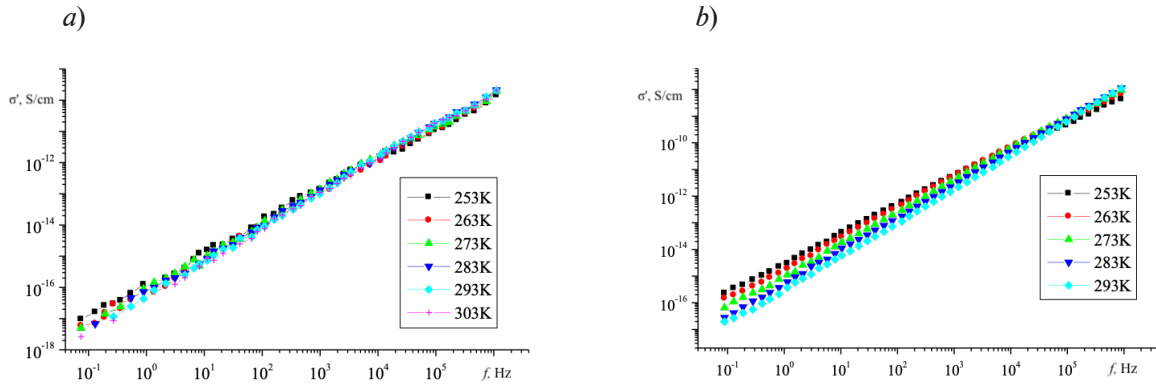


Fig. 5. Frequency dependences of specific conductivity for the samples of LDPE (a) and LDPE/EVA (b) at different temperatures

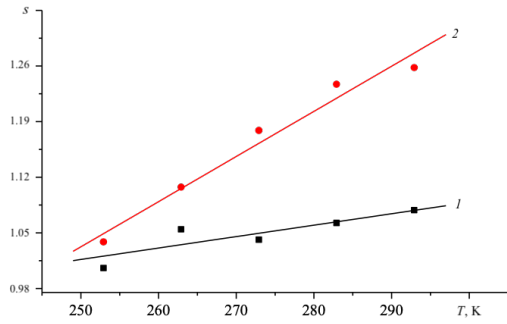


Fig. 6. Temperature dependences of the exponent for the samples of LDPE (1) and LDPE/EVA (2)

In the samples under study, such charge carriers can be negatively and positively charged hydrogen vacancies in the polymer chains [21, 22]. Such vacancies are created during the interaction between water molecules and its ions (H_3O^+ , OH^-) with hydrogen atoms, which are contained in the structural groups of $-CH_2-$, $>CH-$, $-CH_3$ chains. The EVA chains contain side polar groups $-O-(C=O)-CH_3$ and polyethylene molecules contain $>CH-$ groups.

Notice that the specific conductivity of LDPE/EVA blends is more than that of pure polyethylene. This can be explained by the fact that EVA has greater electrical conductivity than LDPE.

To explain the resulting linear frequency dependence of conductivity it is convenient to use the model proposed by the authors of Ref. [20]. According to this model, the frequency versus temperature characteristic of the AC conductivity $\sigma_{ac}(\omega, T)$ forms as a result of tunneling the center-to-center bipolaron transitions. This also assumes that there is no correlation between the potential barrier height and the jump distance.

For the case of the strong spatial correlations, when the pair correlation function

$$f(r) = Ae^{-\beta r} > 1,$$

the expression for the AC conductivity can be written as

$$\sigma_{ac}(\omega) = \frac{A}{48} \frac{e^4 \omega (\omega v_{ph})^{\beta/2\alpha}}{\cos(\beta/2\alpha) \alpha^4 \epsilon \epsilon_0} \left[\ln \left(\frac{v_{ph}}{\omega} \frac{2\alpha - \beta}{2\alpha + \beta} \right) \right]^3 N^2(E), \quad (2)$$

where ω is the cyclic frequency; v_{ph} is the phonon frequency (similar to that in a crystal); ϵ is the static dielectric constant; α^{-1} is the localization radius; r is the center-to-center jump distance; $N(E)$ is the concentration of centers in unit interval energies.

Eq. (2) explains the linear frequency dependence in the region of medium and high frequencies, taking into account that

$$s = 1 + \beta/2\alpha, \quad \beta = 1,6 \cdot 10^{-7} \text{ cm}^{-1}.$$

It can be concluded that the main contribution to dynamic conductivity is made by spatially correlated pairs of defects, for example, dangling bonds, etc.

Summary

Based on the results of the studies, it can be argued that the process of α -relaxation has been observed in blends of LDPE and EVA in the temperature range 250 – 280 K. The activation energies corresponding to this process in LDPE films and in the films of blends are 0.69 and 1.37 eV, respectively. The increased value of activation energy in the LDPE/EVA samples compared to the LDPE one can be explained by the appearance of deeper traps in the blends. These traps are bound up with the rotation of the polar groups in the EVA molecules or with the charge accumulation at the interface of two polymers.

The frequency dependence of the conductivity of the samples indicated the presence of hopping conductivity. Charge carriers involved in the conductivity can be negatively and positively charged hydrogen vacancies in polymer chains.

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