

## THERMOACTIVATED CURRENT SPECTROSCOPY OF POLYETHYLENE TEREPHTHALATE FILMS IRRADIATED BY HEAVY XENON IONS

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The paper analyzes the electret state of polyethylene terephthalate (PET) films irradiated on the IC-100 cyclotron (JINR LAR) using thermostimulated spectroscopy methods. Thermally stimulated discharge currents (thermally stimulated depolarization) of PET film samples obtained at different technological stages of track membrane manufacturing are studied. The results of the analysis of thermally stimulated depolarization currents indicate a significant change in the spectra of the energy states of the electric charge accumulated during the polarization of PET films, during irradiation with accelerated heavy Xe ions, ultraviolet treatment, and alkaline etching of latent tracks. The parameters of relaxation processes (activation energy and effective frequency factor) occurring in track membranes are calculated using the Eyring method. A significant change in the energy state spectra of the electric charge accumulated during the polarization of PET films as a result of ion-track processes in them indicates the possibility of creating an electret state in PET films and track membranes irradiated with heavy ions on their basis.

**Keywords:** electret, track membrane, polyethylene terephthalate, thermal-stimulated discharge current, Eyring method

**Citation:** Gorokhovatsky Yu.A., Temnov D.E., Shabanova N.S., Nechaeff A.N., Rossouw A., Thermoactivated current spectroscopy of polyethylene terephthalate films irradiated by heavy xenon ions, St. Petersburg Polytechnical State University Journal. Physics and Mathematics. 14 (3) (2021) 8–20. DOI: 10.18721/JPM.14301

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## ТЕРМОАКТИВАЦИОННАЯ СПЕКТРОСКОПИЯ ПЛЕНОК ПОЛИЭТИЛЕНТЕРЕФТАЛАТА, ОБЛУЧЕННЫХ ТЯЖЕЛЫМИ ИОНАМИ КСЕНОНА

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Методом термостимулированной токовой спектроскопии в работе проанализировано электретное состояние в облученных на циклотроне пленках полиэтилентерефталата. Изучено поведение термостимулированных токов деполяризации в образцах пленок, полученных на разных технологических стадиях изготовления трековых мембран. Результаты исследования указывают



на существенное изменение спектров энергетических состояний электрического заряда, накопленного при поляризации пленок после их облучения ускоренными тяжелыми ионами ксенона, затем последующей ультрафиолетовой обработки и щелочного травления латентных треков. С помощью метода Эйринга рассчитаны параметры релаксационных процессов (энергия активации и эффективный частотный фактор), возникающих в трековых мембранах. Указанные существенные изменения спектров свидетельствуют о возможности создания электретного состояния в облученных пленках и трековых мембран на их основе.

**Ключевые слова:** электрет, трековая мембрана, полиэтилентерефталат, термостимулированный ток деполяризации, метод Эйринга

**Ссылка при цитировании:** Гороховатский Ю.А., Темнов Д.Э., Шабанова Н.С., Нечаев А.Н., Россоу А. Термоактивационная спектроскопия пленок полиэтилентерефталата, облученных тяжелыми ионами ксенона // Научно-технические ведомости СПбГПУ. Физико-математические науки. 2021. Т. 14. № 3. С. 8–20. DOI: 10.18721/JPM.14301

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## Introduction

The results of fundamental and applied studies, obtained recently using heavy ion beams with low and medium energies, are clear evidence that these approaches have good prospects for the high-tech industry. Analysis of fundamental physical processes characterizing particle interaction with a solid provides the basis for the practical applications of these processes for ion-beam modification of materials [1].

Accelerated heavy ion beams can serve as a unique tool for transforming the nano- and microstructure of solids, in particular, for obtaining track membranes [2 – 4, 7].

Fabricating polymer track membranes with heavy ion accelerators is one of the major applications of ion track technologies. Track membranes based on polyester films made of polyethylene terephthalate (PET) and polycarbonate (PC) are effectively used for ultra-purification of water, in biotechnology for producing vaccines and serums, in medicine as a key filtering element of plasma filters, in analytical chemistry and environmental sanitation control [10, 11].

Modern track membranes are mainly based on polyester films such as polyethylene terephthalate (PET) and polycarbonate. PET is a polymer that is a complex thermoplastic polyester of terephthalic acid and ethylene glycol. PET films that track membranes are produced from are composites whose structure includes both amorphous and crystalline phases. PET in amorphous state is characterized by mutual random arrangement of macromolecule chains with rare isolated formations of an ordered structure. The crystalline state of PET is characterized by crystallites that are bunched macromolecules appearing as folded stacks. The crystallite size in films is about 5 – 10 nm, and the degree of crystallinity is about 40 – 50%.

The structural and physicochemical characteristics of PET-based track membranes irradiated with heavy ions depend on the parameters of the ion beam, the chemical composition of the irradiated polymer film and its structure, as well as the methods for physicochemical processing of latent tracks in it. A heavy ion track forms in a polymer material through a complex physicochemical process. A high-energy multicharged ion passing through a PET film enters into a Coulomb interaction with its electron subsystem. Amorphization of PET and a decrease in the size of crystallites are observed due to irradiation with heavy ions. In addition, the destruction of PET macromolecules is accompanied by an increase in the number of carboxyl groups in the polymer [8, 9].

Tracks in PET are capable of selective chemical etching upon exposure to aqueous solutions of alkali metals. A chemical reaction of etchant interaction occurs in ester polymers, including PET, accompanied by breaking of ester bonds, elimination of ethylene glycol and terephthalate ion molecules with carboxyl and hydroxyl groups forming on the surface, which determine the negative elec-

tric charge of the material surface. Photo-oxidation of radiolysis products in tracks exposed to soft ultraviolet (UV) radiation in the presence of oxygen is used to increase the selectivity of track etching [4, 5, 12, 15].

Notably, PET films are also used for producing electrets with a stable electrical charge. An electret is a dielectric that maintains a polarized state for a long time after the external influence is removed, leading to polarization of this dielectric and generating a quasi-static electric field in the surrounding space. The electret state is inherent to varying degrees in all dielectrics, including PET.

Analyzing the literature, we have found that the methods of ion-track electret charging of polymer films are insufficiently understood. It can be assumed that the electret properties of polymer films change under bombardment with accelerated heavy ions, similar to the above-mentioned effects of UV irradiation and alkaline etching of tracks. The reason for this is that the morphology and chemical structure of the PET film change upon exposure to both ionizing radiation processes and highly alkaline thermal hydrolysis.

We used methods of thermally stimulated depolarization to consider changes in the properties of track membranes based on polyethylene terephthalate (PET) for the purpose of analyzing the effect of ion-track etching in PET on the variation in electret properties [17 – 21].

Thermally stimulated current spectroscopy is widely used to study the processes of electrical relaxation, in particular, the electret state in various dielectric materials [17 – 22]. One of the most popular methods of TSC spectroscopy used to study charge relaxation in electrets is the method of thermally stimulated discharge current, or thermally stimulated depolarization (TSDC). Measurement of thermally stimulated discharge currents is carried out in short-circuit mode (when the resistance of the external measuring circuit is much lower than that of the given dielectric). Analysis of the shape of the TSDC curve (temperature dependence of the initial section of the current peak, temperature position of the current maximum, etc.) allows determining the most probable activation energy of electrically active defects and their effective frequency factor based on experimental data (methods for processing TSDC data are described in detail in [21]).

The motivation behind the study was to understand how the properties of the charged surface of PET films and track membranes based on them change, in order to expand the range of their practical applications. Track membranes with electret properties can find further application in various types of transducers, for example, in sealing systems, filters, membranes, and in biomedical technologies.

### Materials and methods

Samples were prepared from Hostaphan RNK PET film 30  $\mu\text{m}$  thick by Mitsubishi Polyester Films (Germany). The samples were irradiated by  $^{132}\text{Xe}^{+26}$  ions with a 1.16 MeV beam at  $\pm 35^\circ$  angles sequentially on each side at the IC-100 cyclotron of the Flerov Laboratory of Nuclear Reactions (Joint Institute for Nuclear Research, Dubna, Moscow Region, Russia). The ion path length in PET was about 20  $\mu\text{m}$ . The surface pore density on each side was  $N = 4.5 \cdot 10^8 \text{ cm}^{-2}$ . Following cyclotron irradiation, all samples were exposed to UV radiation for 2 h on each side before chemical etching. UV irradiation was performed in air using LE-30 lamps (by Lisma, Saransk, Russia); the pure PET film was used as a filter and periodically replaced with a fresh one. UV radiation intensities measured with a TPK-PKM radiometer amounted to 3 – 4  $\text{W/m}^2$  in the UV-A range (315 – 400 nm) and 1  $\text{W/m}^2$  in the UV-B range (280 – 315 nm). The density of nano- and micropores on the surface was determined using a Hitachi SU8020 scanning electron microscope. Furthermore, we used the SEM microscope to examine the membrane cross-sections obtained via polymer matrix embrittlement by mild photooxidation.

In this study, we applied the TSDC method for analyzing different samples of PET films and PET-based track membranes.



Samples 1 – 6 were first irradiated with UV light; the exposure time was 2 h on each side of the film. They were then chemically etched in a thermostat at 80 °C with a concentration of sodium hydroxide (NaOH) solution equal to 1 mol/L for different periods of time: from 1 to 6 min.

Measurements of the pore diameter on the surface of the samples were carried out by SEM. The results are presented in Table 1.

A TSC-II setup from Setaram (France) was used to measure the thermally stimulated depolarization currents.

The measurement process included the following stages:

polarization of samples at a constant temperature  $T_p = 90$  °C in an electric field of 5 kV/mm for a time  $t_p = 0.5$  min;

cooling at a rate of 4 °C/min in an electric field until the temperature reached  $T_0 = 10$  °C;

exposition at a temperature  $T_0$  in the absence of an electric field for a time  $t_0 = 1$  min;

heating at a given rate (4, 7, or 9 K/min) to a temperature  $T_f = 130$  °C with simultaneous measurement of depolarization currents (with a Keithley 6517E electrometer).

All measurements were carried out in a vacuum chamber in a gaseous helium atmosphere in order to prevent access of oxygen to the samples.

Table 1

### Structural characteristics of irradiated PET films after etching in alkaline solutions

Sample No.	Etching time, min	Average pore diameter on the surface, nm	SEM data
1	1.0	25	No etchback
2	2.0	36	
3	3.0	57	
4	4.0	78	
5	5.0	160	Etchback
6	6.0	232	

Note. Samples 7, 8, 9 were pure films, not subjected to etching; sample 8 was exposed to UV rays, sample 9 to UV rays and xenon ions.

### Experimental results

We considered the samples obtained at different stages of producing track membranes (see the procedure in the previous section), comparing the results with the corresponding data on the pure PET polymer films.

Fig. 1 shows the TSDC curves for pure polymer film obtained at two different heating rates of the sample.

Several maxima associated with relaxation processes are clearly visible on the dependences presented, typically due to various molecular movements inside the polymer:

high-temperature  $\alpha$ -process (dipole-segmental) around 90 °C, caused by orientational rotations of the polar macromolecular units when segmental motion is possible, i.e., in a highly elastic state;

$\beta$ - and  $\gamma$ -processes (dipole-group) in the temperature ranges of 60 and 25 °C, respectively, due to the mobility of polar end and side groups of macromolecules (they can also appear in the glassy state).

A technique for finding the activation energy of these processes relies on different linear heating rates of the sample, subsequently using numerical solution methods abased on the Tikhonov – Arseniev regularization algorithms. From a mathematical standpoint, finding the required parameters,

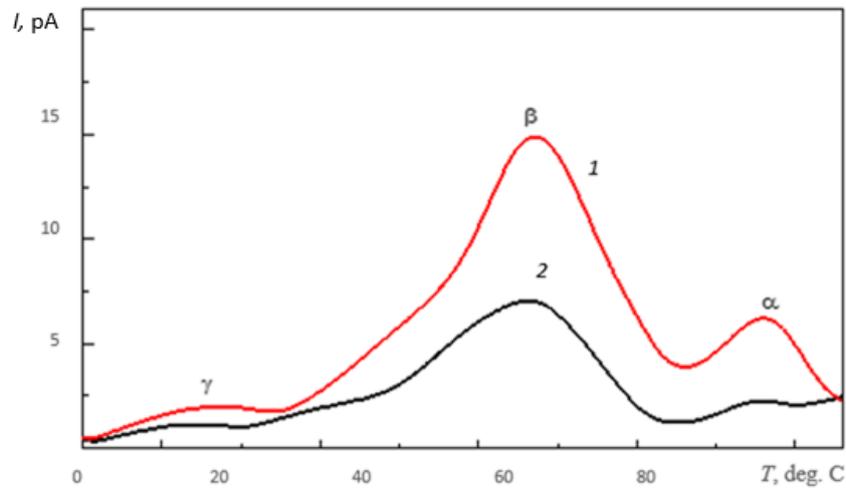


Fig. 1. TSD spectra of pure PET films at two heating rates of the samples: 9 K/min (1) and 7 K/min (2)

that is, the frequency factor  $\omega$  and the activation energy  $E$  of electrically active defects (EAD) from the experimental dependence means solving the following integral equation (1):

$$j(T) \sim \int_0^{\omega_m} d\omega \int_0^{E_m} G(E, \omega) \xi(E, \omega, T, \beta) dE, \quad (1)$$

where  $T$  is the sample temperature;  $\beta$  is its heating rate;  $G(E, \omega)$  is the EAD distribution function;  $\xi(E, \omega, T, \beta)$  is the integrand function whose specific form depends on the physical model incorporated in the theory describing the phenomenon of thermally stimulated depolarization.

Analysis of Eq. (1) confirms that this problem can only be solved if the value of the frequency factor  $\omega$  is known. However, in practice, this value is unknown in advance, yielding an ambiguous solution. This difficulty can be eliminated using a method proposed in [5], comparing the energy spectra numerically reconstructed from the TSDC curves measured for a given sample at two heating rates with other conditions being equal. If the value of the frequency factor is chosen correctly, then the shapes of energy spectra should not differ for different heating rates. Conversely, the difference in the reconstructed spectra for different heating rates means that a different value has to be selected as the frequency factor.

Thus, this technique makes it possible to determine the EAD parameters in the given material with a sufficient degree of reliability even when the relaxation processes observed are not monoenergetic, and the electrically active defects have a complex energy distribution.

Processing the TSDC spectra, we discovered that the activation energies of relaxation processes turned out to be equal (in eV): 1.24, 0.93, and 0.45; they also correspond to a decrease in the maximum temperature of the depolarization peak.

Irradiation of PET films with xenon ions and UV photons significantly increases the intensity of  $\alpha$ -relaxation, which implies an increase in the segmental mobility of polar macromolecular units in the irradiated samples (Fig. 2), which is apparently due to amorphization of the samples upon irradiation with ions [17].

Further etching of the films with the duration increased from 0.5 to 6 min considerably changes the TSDC spectrum both with respect to the temperature scale and to intensity (Fig. 3). It can be assumed that another process becomes most important for charge relaxation in this case, most likely associated with the first the formation and then the depopulation of charge traps on the track walls.

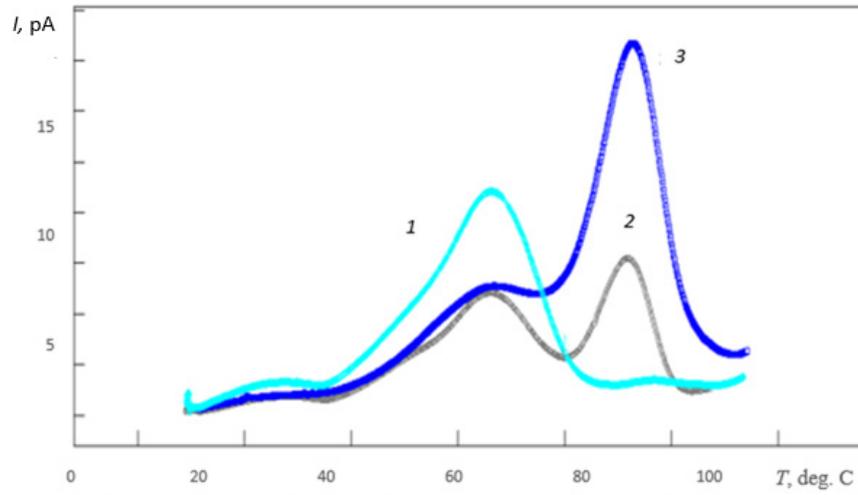


Fig. 2. TSDC spectra of pure PET films (1), irradiated with xenon ions (2) and then with UV photons (3) at a heating rate of 7 K/min

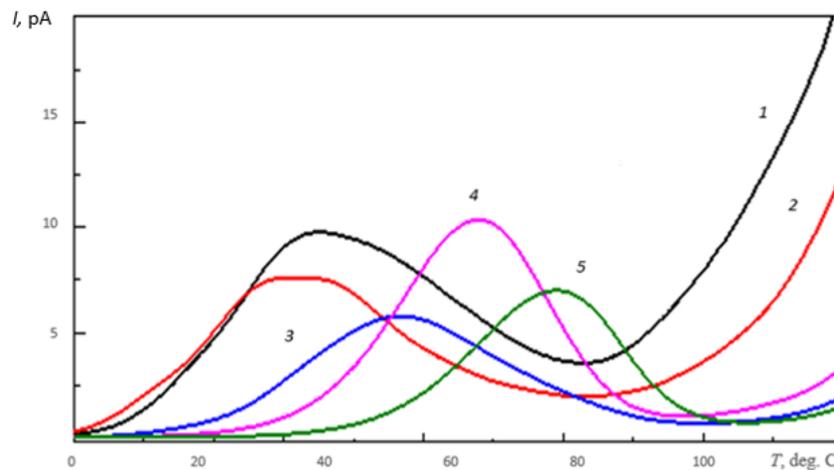


Fig. 3. TSDC spectra of PET films irradiated with xenon ions and UV photons upon subsequent chemical etching of samples 1 – 5 (curve numbers correspond to the sample numbers). Sample heating rate was 7 K/min

It can be seen from the data in Fig. 3 that the TSDC maximum shifts to the high-temperature region with an increase in the etching time, and its intensity remains approximately the same.

The parameters of this relaxation process were calculated for all etching times: the activation energy and effective frequency factor (Table 2); Eyring's method was used for this purpose [6].

It is known [4] that an increase in the etching time leads to an increase in the average diameter of the pores formed in track membranes. This is confirmed by the data given in Table 2.

Apparently, the dependence of the activation energy on the average pore diameter is practically linear (Fig. 4,*a*), and the corresponding dependence of the effective frequency factor bears a pronounced nonlinear character (Fig. 4,*b*).

A sharp increase in the frequency factor with increasing average pore diameter likely points towards the formation of through pores in PET films at an etching time sufficient for this phenomenon,

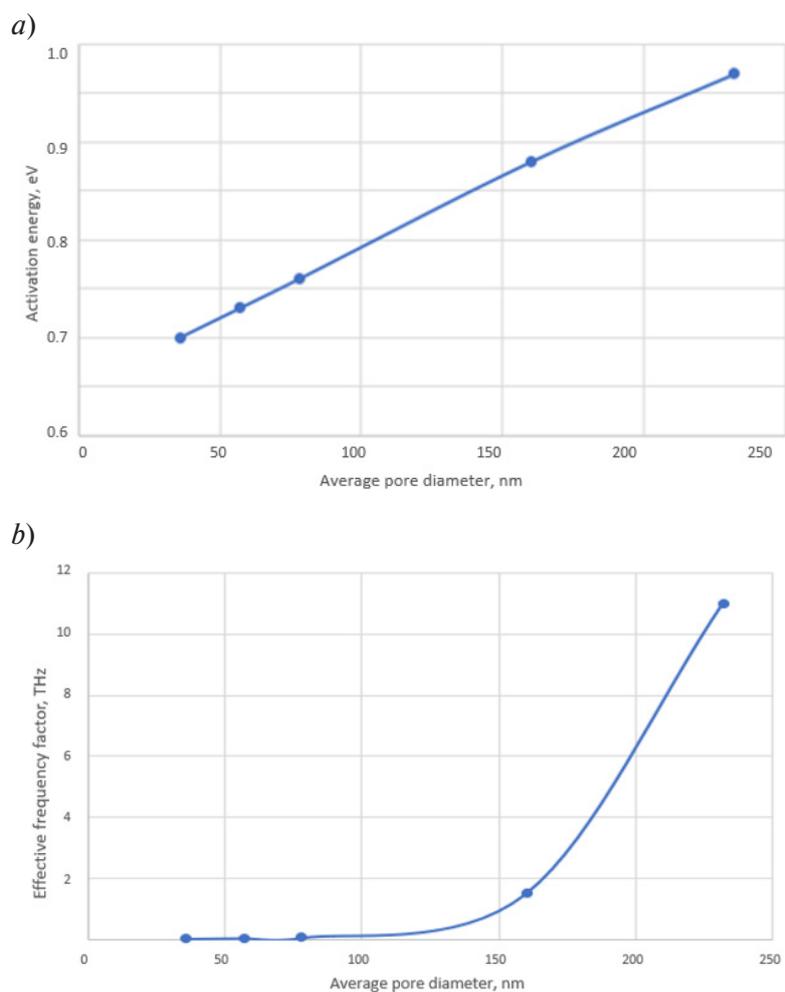


Fig. 4. Dependences of activation energy (a) and effective frequency factor (b) on average pore diameter with increasing etching time

as well as towards an increase in the free volume of the polymer. An increase in the activation energy suggests that progressively deeper charge carrier traps evolve with increased etching time.

Table 2

**Parameters of dipole-group relaxation process observed in PET films at different etching times**

Etching time, min	Mean diameter of pores, nm	Activation energy, eV	Effective frequency factor, $10^{10} \text{ s}^{-1}$
1	36	0.70	2.4
2	57	0.73	4.4
3	78	0.76	5.4
4	160	0.88	150
5	232	0.97	1100

The method of relaxation maps [7] was used to calculate the degree of disorder in the polymer system. With an increase in the etching time, it increases from 25.2 (this etching time is 1 min) to 36.6



(this time is 5 min), which means that the degree of disorder can serve as a characteristic of the total volume of the 'loose' polymer along the pore walls formed during etching.

### Conclusion

The method of thermally stimulated current spectroscopy has been applied for the first time to track membranes based on PET films. We have confirmed that the processes of ion track technology, such as irradiation with heavy  $^{132}\text{Xe}^{+26}$  ions, UV sensitization and etching of PET film tracks, have a major effect on the TSDC spectra. The obtained spectroscopic data were used to calculate the parameter values of the relaxers, as well as the degree of disorder of the polymer system for track membranes with different pore diameters. Several relaxation processes are clearly observed for the pure PET films, typically associated in the literature with various molecular movements inside the polymer; in particular, a high-temperature process (dipole-segmental) takes place around 90 °C, due to orientational rotations of the polar macromolecular units under the conditions when segmental motion is possible. Irradiation of PET films with xenon ions and UV photons considerably increases the intensity of  $\alpha$ -relaxation, which means an increase in segmental mobility in irradiated samples. Further etching of the films with an increase in the etching time significantly changes the TSDC spectrum both with respect to the temperature scale and the peak intensity. It can be assumed that another process becomes most important for charge relaxation in this case, most likely associated with the formation and subsequent depopulation of charge traps on the track walls. The maximum of the TSDC spectrum shifts to the high-temperature region with increasing etching time, while the dependence of the activation energy on the average pore diameter is almost linear, and the corresponding dependence of the effective frequency factor bears a pronounced nonlinear character.

The most likely reason for the sharp increase in the frequency factor is that through pores evolve in PET films given a sufficient etching time. An increase in the activation energy suggests that progressively deeper charge carrier traps appear with increased etching time. The results may indicate that the spectrum of energy states of the electric charge accumulated during polarization of such films changes considerably and that an electret state can be generated in such materials. The relaxation parameters of track membranes can be tailored in accordance with the structural peculiarities of such objects.

### Acknowledgment

The authors are grateful to the staff of the Center for Applied Physics at the Flerov Laboratory of Nuclear Reactions (Dubna, Moscow Region) for their assistance with our studies: O.M. Ivanov (irradiation of films at the IC-100 cyclotron), T.I. Mamonov (synthesis of membranes), N.V. Lizunov (SEM studies) and P.Yu. Apel (implementation of the experimental design and fruitful discussions of the experimental data).

The research was supported by the Ministry of Education of the Russian Federation as part of a state task (project No. FSZN-2020-0026).

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*Received 15.06.2021, accepted 22.06.2021.*

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*Статья поступила в редакцию 15.06.2021, принята к публикации 22.06.2021.*

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