Original articles

Organic fouling of anion-exchange and bipolar membranes during the separation of amino acid and sucrose by electrodialysis

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Abstract

The article presents a study of the behaviour of the MA-41 anion-exchange membrane and MB-2 bipolar membrane during the electrodialysis of a solution containing tyrosine and sucrose. It establishes changes in current-voltage, transport, and structural characteristics of ion-exchange membranes. The study of the evolution of membrane characteristics during a prolonged contact with solutions containing an aromatic amino acid and disaccharide is aimed at providing a deeper understanding of and finding solutions to the problem of organic fouling of membranes, which complicates the electromembrane separation of components of the solution during microbiological synthesis of amino acids.

It was found that the fluxes of tyrosine and sucrose through the MA-41 membrane measured after its operation during 50-hour electrodialysis reach higher values than during the first hours of operation after the system reaches a steady state. However, it was noted that when the membrane continues to be used, the flux of components through the MA-41 membrane decreases. What is more, this change is pronounced with a high current density.

This decrease in mass transport, an increased voltage drop on the MB-2 and MA-41 membranes, and lower values for the effective OH⁻ ion transport number for the MA-41 membrane are associated with the phenomenon of organic fouling confirmed by revealed structural changes in the ion-exchange material, which become significant after a prolonged contact (more than 60 hours) with a mixed solution of tyrosine and sucrose. These changes are associated with the accumulation of an amino acid and its oxidation product, 3,4-dihydroxyphenylalanine, in the membrane phase, as well as with a decrease in the content of sucrose absorbed by the membrane.

Keywords: Electrodialysis, Separation, Ion-exchange membrane, Tyrosine, Sucrose, Organic fouling

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1. Introduction

Amino acids in the form of pure compounds are in great demand in the food, medical, pharmaceutical, and biotechnology industries. They are used as nutrient additives, such as seasonings and flavourings, growth feed supplements, and pharmaceutical preparations to treat various diseases. The demand for amino acids continues to grow. Currently, L-amino acids are produced by microorganisms (by fermentation). However, their processing and extraction from fermentation broths can be challenging and inefficient [1, 2].

Membrane technologies offer a promising solution to the extraction of amino acids. Their implementation is attractive due to the fact that there is a wide range of materials that can be used to produce membranes with certain required properties and there are various ways of controlling the parameters of membrane processes. Electromembrane technologies use ion-exchange membranes and the electrical potential gradient as the driving force of mass transport of charged particles. These technologies have such important advantages as the possibility of separating components with different charges, they require no reagents, are environmentally friendly, and relatively inexpensive [3–5]. Electrodialysis with ion-exchange membranes provides a tool for the separation of electrolytes and non-electrolytes, desalination, and concentration of electrolyte solutions. This method is often used in food industry for the deionisation of various intermediates and food products and for demineralisation of wastewater, sea and brackish water, desalination of tobacco leaf extract, extraction of lactic acid and amino acids from the juice of grass silage, etc. [6–12]. Bipolar membrane electrodialysis is used to obtain acids and bases from salts, to perform other chemical transformations, to separate electrolytes and non-electrolytes, to deoxidise certain foods, to separate mixtures of organic and inorganic acids, and to produce highly purified water [13–15]. Some works consider the separation of amino acids and their isolation from impurities remaining after synthesis, mineral salts and carbohydrates [16–19].

Ion-exchange membranes are modern materials and have a wide range of applications [20–23]. To effectively use bipolar and monopolar membranes for amino acids extraction from solutions, it is important to understand changes in the structural, transport, and current-voltage characteristics of these polymer materials since when ion-exchange membranes come into contact with organic substances, there might appear undesirable side interactions.

The long-term use of ion-exchange materials in solutions of amino acids and a number of other organic substances can complicate electromembrane processes due to organic fouling, which causes changes in the electrochemical, transport, and structural characteristics of membranes [24–26]. Biological fouling is also a possibility [27].
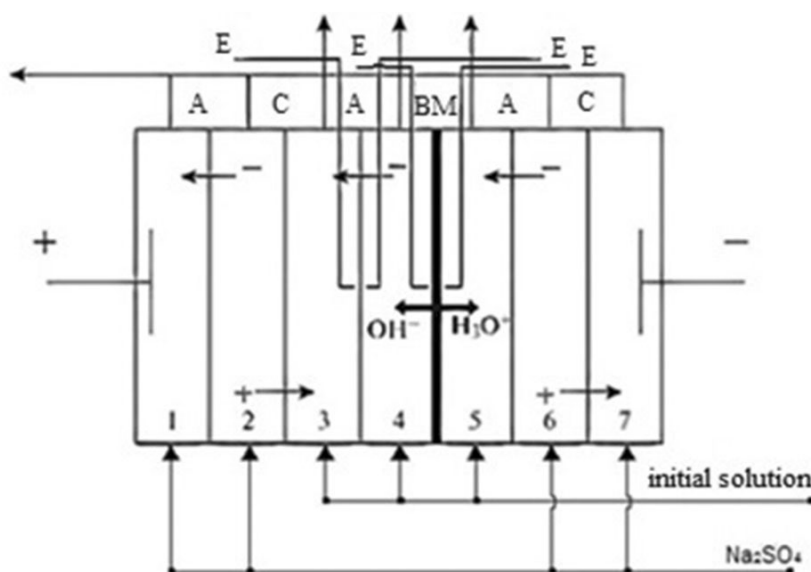
The study of the properties of membranes used in electromembrane systems for a long time is very important since currently there is not enough data on the characteristics of ion-exchange membranes that come in durable contact with aromatic amino acid solutions.

Therefore, the purpose of this study was to assess changes in the current-voltage, transport, and structural characteristics of anion-exchange and bipolar membranes during prolonged electrodialysis of a solution of an aromatic amino acid (tyrosine) and disaccharide (sucrose).
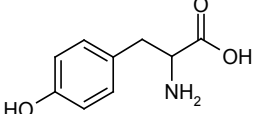
2. Experimental

Changes in the current-voltage and transport characteristics of ion-exchange membranes were studied during a prolonged electrodialysis of model solutions of an aromatic amino acid, i.e. tyrosine (Sigma-Aldrich, Burlington, USA) (C = 0.0025M) and sucrose (Sigma-Aldrich, Burlington, USA) (C = 0.02M) in a seven-compartment laboratory apparatus (Fig. 1) for 60 hours. We used the L-optical isomer of tyrosine, an amino acid that contains a para-hydroxyphenylmethyl side radical in its structure. Some physicochemical properties of tyrosine are given in Table 1.

Electrodialysis compartment 4 had an anion-exchange and bipolar membrane. The anion-exchange layer of the bipolar membrane faced section 4. The working area of the membranes was 20 cm^2. The height of the apparatus was 20 cm, the intermembrane distance was 10 mm, and the linear fluid flow rate was 0.05 cm·s^{-1}. The anode

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Fig. 1. Seven-compartment electrodialysis cell with alternating cation (C)- and anion (A)-exchange membranes, BM – bipolar ion-exchange membrane, '+' – cation, '–'– anion, E – Pt electrodes

Table 1. Physicochemical properties of the amino acid studied [28–30]

Amino Acid	Structure	pI	pK			Molecular Weight	Solubility, g/100 mL H_2O, 25 °C	Side Radical Volume, nm^3
			pK_1	pK_2	pK_3			
Tyrosine (Tyr)		5.63	2.20	9.11	10.07	181.19	0.045	0.1388

was made of platinum, while the cathode was made of stainless steel.

We used an MA-41 heterogeneous anion-exchange membrane, an MK-40 cation-exchange membrane, and an MB-2 bipolar membrane manufactured by LLC *Shchekinoazot*. These heterogeneous membranes have a styrene-divinylbenzene matrix. The structure of the MA-41 anion-exchange membrane includes quaternary ammonium base groups, whereas the MK-40 membrane has sulpho groups. The MB-2 bipolar membrane is manufactured using the MA-41 and MK-40 membranes [31].

A mixed solution of tyrosine (Tyr) and sucrose (Suc) entered compartments 3, 4, and 5 of the electrodialysis apparatus. A sodium sulphate solution (C = 0.1M) (ZAO *Vekton*, Saint Petersburg, Russia) was supplied to compartments 1, 2, 6, and 7 of the apparatus. The analysis was performed for solutions flowing from sections 3, 4.

The changes in current-voltage characteristics (CVC) of bipolar and anion-exchange membranes during electrodialysis were detected using a V7-26 voltmeter. Platinum electrodes were placed near the studied membrane surfaces at a distance of 0.2 mm on each side.

The UV spectroscopy was used for the quantitative determination of Tyr [32], while Suc was analysed by a photometric method based on the sucrose oxidation in acidic medium with potassium dichromate to CO_2 applying the calibration curve procedure [33].

The substance flux through the ion-exchange membrane was calculated according to formula (1) [22]:

$$J = C \cdot V \cdot \tau^{-1} \cdot S^{-1}, \tag{1}$$

where J is the flux through the ion-exchange membrane, $mol \cdot cm^{-2} \cdot s^{-1}$; C is the concentration of the solution, $mol \cdot dm^{-3}$; V is the sample volume, dm^3; τ is the sampling time, s; and S is the working area of the ion-exchange membrane, cm^2.

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The effective hydroxyl ion transport number was determined by equation (2) [22]:

$$T = z \cdot F \cdot J \cdot i^{-1}, \qquad (2)$$

where T is the effective transport number; F is the Faraday constant, Cl·mol^{-1}; z is the ion charge; i is the current density, A·cm^{-2}; and J is the flux of OH$^-$ ions, mol·cm^{-2}·s^{-1}.

The pH of the studied solutions was measured using an I-160MI electronic pH-meter which had been calibrated by buffer solutions (pH = 1.65, pH = 9.18). The pH of the solutions supplied to compartment 4 was within 5.5–5.9, which was close to the isoelectric point of tyrosine.

The structural changes in the ion-exchange membranes which came in contact with solutions containing an aromatic amino acid were investigated by IR spectroscopy. The membrane samples were pre-dried at 50°C, crushed, and compressed with KBr into tablets at a ratio of 1 to 100. The spectra were obtained using a Vertex 70 IR-Fourier spectrometer, Bruker Optik GmbH (Germany).

3. Results and discussion

To assess the effect of the interactions of an aromatic amino acid, sucrose, and ion-exchange material under the action of the electrical potential gradient, we studied the changes in transport characteristics of the MA-41 membrane during the prolonged bipolar membrane electrodialysis of a solution containing tyrosine and sucrose (Fig. 2a, b). Figure 2 shows the dependences of the component fluxes through the MA-41 membrane on the current density for the "pristine" membrane after the steady state was reached and for the samples of membranes after they had been used for 50 and 60 hours without washing.

At the initial section of the obtained dependencies, there was a gradual increase in tyrosine flux with increasing current density followed by a "plateau" at a current density of $i = 1.0$–2.3 mA·cm^{-2}. Then, the amino acid mass transport continued to grow with a higher current density (Fig. 2a). The absence of decreasing fluxes through the MA-41 membrane in the area of the "barrier effect" observed in a system with alternating monopolar membranes was due to the intensive generation of hydrogen and hydroxyl ions by the bipolar membrane, which led to the conversion of tyrosine bipolar ions into anions in the compartment 4 even with a low current. At a higher current density, there was an increase in the slope angle of the considered dependencies $J - i$, which is associated with an increased electrical convection contributions and an increased effect of facilitated electromigration [34, 35]. There was a slight increase in the disaccharide flux with an increase in current density (Fig. 2b) since sucrose

a   b
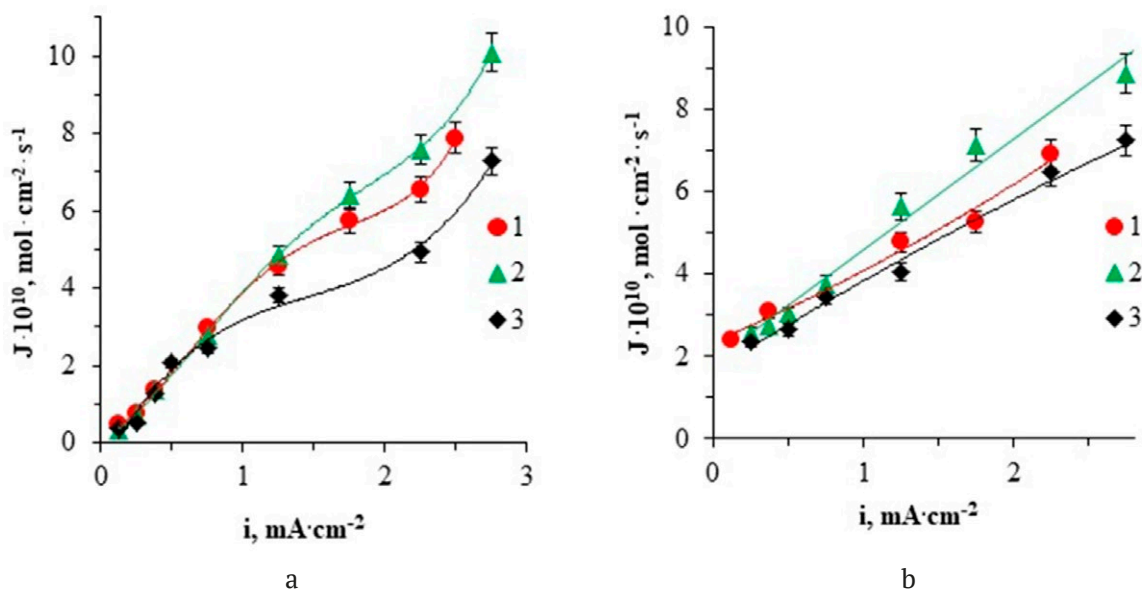
Fig. 2. Dependence of Tyr (a) and Suc (b) fluxes through the MA-41 membrane on the current density in electrodialysis of Tyr + Suc solution: *1* – membrane after establishment of a steady state ("pristine" membrane), *2* – membrane after 50 hours of operation, *3* – membrane after 60 hours of operation

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is a weak electrolyte with an acid dissociation constant of about 10^{-13} [30].

These dependencies mean that the tyrosine and sucrose fluxes through the anion-exchange membrane, which has been used for 50 hours, reach higher values as compared to the "pristine" membrane sample (Fig. 2a, b). These changes are due to the mass transport of quite large hydrated organic substances which changes the porosity of the membrane: pores of a larger radius appear in the membrane as compared to a "new" membrane [36].

However, if membranes continued to be used with a high current density, the fluxes of these components through the MA-41 membrane decreased. The dependences of the amino acid flux through the MA-41 membrane on the electrodialysis duration with different values of current density are shown in Fig. 3. It was obvious that with a high current density, the tyrosine flow through the membrane, which had been used for a long time in a solution containing an amino acid and disaccharide, started to decrease after 50 hours of operation.

This may be due to the gradual organic fouling of ion-exchange membranes that were in contact with a solution of tyrosine and sucrose during the electrodialysis.

We also recorded changes in the current-voltage characteristics of ion-exchange membranes during a prolonged electrodialysis of the Tyr + Suc solution. There was an increased voltage drop for both MA-41 and MB-2 membranes after 50 hours of operation. CVCs for MB-2 membranes at different operating times are shown in Fig. 4a. The changes may be explained by the shielding of the functional groups of the studied membranes with amino acid and sucrose molecules that block the membrane pores which impede the reaction of water dissociation due
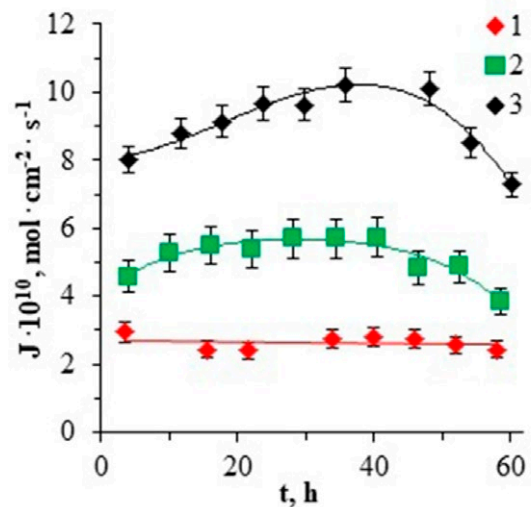
Fig. 3. Dependence of Tyr fluxes through the MA-41 membrane on time in electrodialysis of Tyr + Suc solution: *1* – at 0.75 mA·cm^{-2}, *2* – 1.25 mA·cm^{-2}, *3* – 2.75 mA·cm^{-2}

a

b
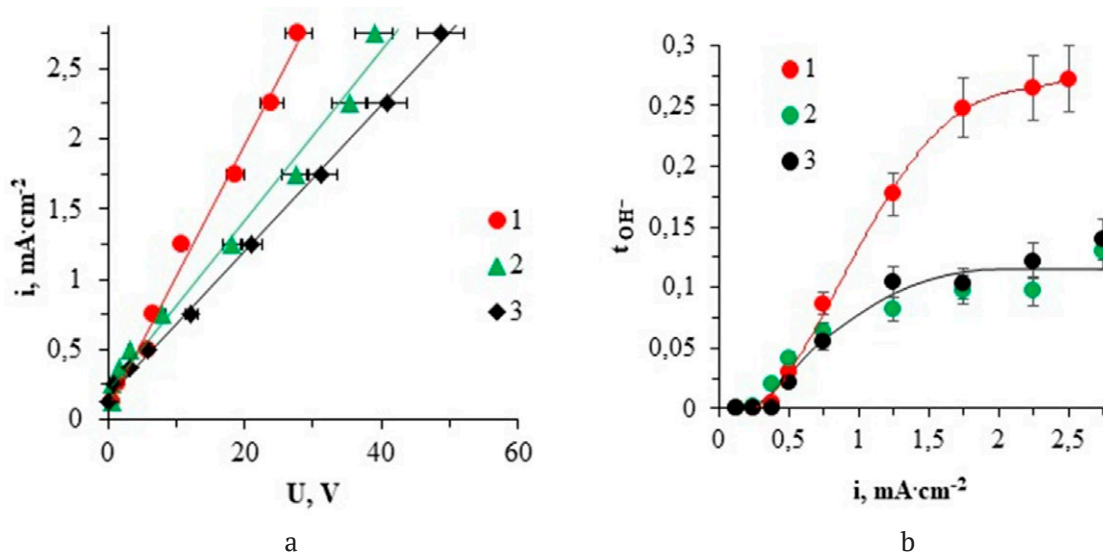
Fig. 4. Current-voltage characteristics of MB-2 (a) membranes and dependence of the effective transport numbers of OH- ions for the MA-41 membrane on the current density (b) in long-term electrodialysis of Tyr + Suc solution: *1* – «pristine» membrane, *2* – membrane after 50 hours of operation; *3* – membrane after 60 hours of operation

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to a decrease in the catalytic activity of the functional groups of the membranes in relation to the reaction of water dissociation. In case of the MA-41 anion-exchange membrane used for a long time during electrodialysis, this fact was confirmed by a decrease in the effective hydroxyl ion transport number (Fig. 4b).

The CVC of the MB-2 membrane (Fig. 4a) did not show the presence of three traditional sections characteristic of the MA-41 monopolar membrane [22]. This is due to the fact that when any current flows through the bipolar membrane, water molecules dissociate at the inner boundary separating the cation-exchange and anion-exchange parts of the membrane.

In addition, we recorded a change in the total cell voltage during a prolonged electrodialysis without washing (regeneration) of ion-exchange membranes. As the time of the membranes' contact with a solution containing an aromatic amino acid and a carbohydrate increased, the total voltage in the system increased at the same value of the current.

Figure 5 shows the recorded dependences of the pH of the solution in the compartment 4 on the operation time of the membranes. The obtained results show that after 50 hours of the membranes' operation without regeneration, lower pH values of the solution in the compartment 4 were achieved. This is due to a decrease in
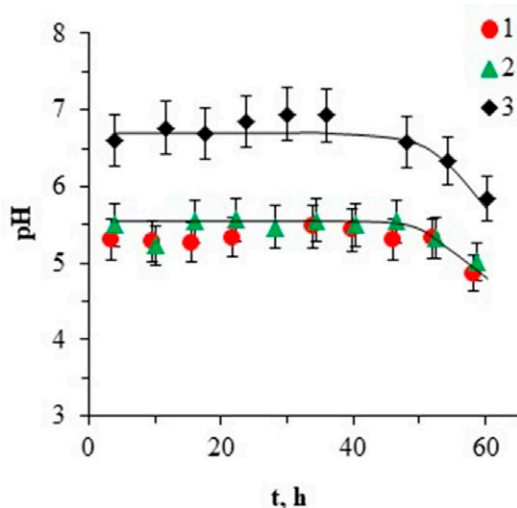
Fig. 5. Time dependence of pH for the compartment 4 output in long-term electrodialysis of Tyr + Suc solution: *1* – at 0.75 mA.cm^{-2}, *2* – 1.25 mA.cm^{-2}, *3* – 2.75 mA.cm^{-2}

the generation of hydroxyl ions by the MB-2 membrane over time and indicates fouling of the anion-exchange layer of the bipolar membrane.

The study revealed that the changes in the transport and current-voltage characteristics of the membranes were accompanied by changes in their structure. We studied samples of the MA-41 anion-exchange membrane, whose structure was identical to the anion-exchange layer of the MB-2 bipolar membrane: in the basic form, in the form that adsorbed the components from the Tyr + Suc solution, and the "fouled" membrane after its prolonged contact with the solution for 10 days.

On the IR spectra of the MA-41 membrane, the presence of tyrosine in the membrane phase after sorption from the Tyr + Suc solution was confirmed by the absorption bands at 1157 cm^{-1} (Tyr phenolate ion), 1573, and 1257 cm^{-1} (carboxylate ion). An increase in the intensity of these maxima after 10 days of the membrane's contact with a solution containing an amino acid and sucrose indicates the accumulation of tyrosine in the membrane phase. In addition, on the IR spectrum of the membrane which was in a prolonged contact with a Tyr + Suc solution for 10 days, there was a new peak of 1595 cm^{-1}, which may indicate the appearance of 3,4-dihydroxyphenylalanine, the product of tyrosine oxidation, in the membrane phase.

After the membrane adsorbed Suc from a mixed solution of amino acid and carbohydrate, there were bands on the spectra at 1635 cm^{-1} (C-O) and at 1307, 1257, and 1089 cm^{-1} (C-O-C group). In addition, the intensity of the band increased at 3425 cm^{-1} (OH group). These changes indicate the presence of disaccharide in the membrane phase. After 10 days of contact with the studied solution, on the IR spectrum, there was a decrease in the intensity of the bands at 1635 cm^{-1} (C-O), and 1307, 1257, and 1089 cm^{-1} (C-O-C group), which indirectly indicates a decrease in the content of disaccharide in the phase of the ion-exchange membrane.

4. Conclusions

The article presents the study of the behaviour of the MA-41 anion-exchange membrane and MB-2 bipolar membrane during the electrodialysis of a solution containing tyrosine and sucrose.

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The study revealed an increased voltage drop on the membranes during a prolonged electrodialysis. These changes can be due to the shielding of the functional groups of the MB-2 membrane with an amino acid and sucrose, as well as blocking of the membrane pores, which impede the reaction of water dissociation.

It was found that the flux of tyrosine and sucrose during electrodialysis reaches higher values when it passes through the MA-41 membrane, which has been used for 50 hours rather than through the "pristine" membrane. The transport characteristics of the membrane increase due to changes in the porosity and moisture content of the membrane during its contact with highly hydrated organic substances. However, further usage of membranes leads to a reduction in the flux of amino acids and sucrose through the MA-41 membrane with a high current density. A reduction in the mass transport of components through the MA-41 membrane, an increase in the resistance of the MB-2 membrane, as well as the detected decrease in the achieved values of the effective OH⁻ ion transport number for the studied membranes during their operation are associated with the phenomenon of organic fouling accompanied by detected structural changes that occur after a prolonged contact (over 50 hours) with a mixed solution of tyrosine and sucrose and that manifest themselves in the retention of an amino acid and its oxidation product, 3,4-dihydroxyphenylalanine, in the membrane phase, and in a decrease in the sucrose content in the membrane phase.

Contribution of the authors

A. Yu. Kharina – preparation of the review, discussion of the results, writing and editing the text. O. E. Charushina – conducting research, writing the text. T. V. Eliseeva – scientific guidance, concept of the study, discussion of the results.

Conflict of interests

The authors declare that they have no known competing financial interests or personal relationships that could have influenced the work reported in this paper.

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