

**STERILIZATION INFLUENCE ON PET TRACK MEMBRANE PROPERTIES**

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**ВЛИЯНИЕ СТЕРИЛИЗАЦИИ НА СВОЙСТВА ТРЕКОВЫХ МЕМБРАН ИЗ  
ПОЛИЭТИЛЕНТЕРЕФТАЛАТА**

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***Abstract.** В работе представлены результаты исследований трековых мембран из полиэтилентерефталата после паровой стерилизации и стерилизации ионизирующим облучением. Исходя из полученных данных, можно считать, что стерилизация ионизирующим излучением радионуклидов  $Co^{60}$  является более предпочтительной, чем паровая стерилизация.*

Polyethylene terephthalate (PET) is a thermoplastic polymer of the polyesters class widely used in different scientific and industrial applications: electronic, food industry, chemical and truck-mounted industry, cryogenic technique and medicine. PET track membranes (TM) have a great opportunity to be used in ophthalmology as corneal implant for bullous keratopathy due to its required structure and bio compatibility [1]. All medical implants must be sterilized prior to operation. The steam or  $\gamma$ -rays sterilization can alter physical and chemical properties of polymer implant causing a change in its functionality, toxicity and safety [2, 3]. The aim of this study is to investigate the effect of sterilization on the properties of PET track membranes.

**Materials and investigation procedure**

Oriented PET films were irradiated with argon ion beam at maximum ion energy of 41 MeV in a specially designed vacuum chamber with a tape drive. Ions passing through the film create the latent tracks. Selective alkaline etching of the material in the latent tracks results in the formation of a pore system with cylindrical through holes having a typical symmetric structure in the initial film. Before etching, the film was irradiated with ultraviolet light for additional sensitization. Etching was carried out in a 1.5 N Na. Researched samples were track membranes with a 7  $\mu\text{m}$  of thickness, 0.4  $\mu\text{m}$  of pore's diameter,  $5 \times 10^8$  pores/ $\text{sm}^2$  of pores density.

Wetting angle measurement and surface energy were performed by KRÜSS Easy Drop DSA 20 at room temperature  $25 \pm 2^\circ\text{C}$ . For wetting angle measurement we used deionized water and glycerol. On the each samples we dealt with four drops of 3  $\mu\text{l}$ . The wetting angle and surface energy dynamics were analyzed 1, 3, 7, 14, 21 days after sterilization.

The sterilization was carried out in two ways. The first method was steam sterilization (temperature = 120°C, pressure = 0.11 MPa) and the second was  $\gamma$ -irradiation of  $\text{Co}^{60}$  radionuclide with 1 kGy dose using «Researcher» special devices for  $\gamma$ -irradiation (Russia).

The chemical analyses of track membranes after steam and  $\gamma$ -irradiation sterilization were carried out using Nicolet 5700 Fourier IR spectrometer.

**Results and discussion**

The table 1 illustrates results of surface roughness, wetting angle and surface energy measurement.

Table 1

Surface properties of the PET TM before and after sterilization

Sample	Ra, $\mu\text{m}$	Rms, $\mu\text{m}$	Wetting angle, $\theta^\circ$	Surface energy, $\text{mJ}/\text{m}^2$		
				dispersi ng	polarizi ng	full
Virgin 0.4 mkm pore's diameter	0.23	0.49	72.6	5.97	23.98	29.95
Steam sterilization 120°C	0.39	0.72	81.4	1.7	29.78	41.48
Sterilization by $\gamma$ -irradiation 1 kGr	0.26	0.45	68.7	0.3	43.43	43.73

The results show that sterilization has a different effect on the surface topography PET track membrane. The TM surface before sterilization is smooth ( $R_a=0.23 \mu\text{m}$ ) but there is local ups and downs with peaks high of 0.5  $\mu\text{m}$ . Obtained peaks are the result of mechanical action on the material during the preparation of the starting membrane film.

The steam sterilization at 120 ° C for 20 minutes leads to appearance of domed peaks of up to 0.6  $\mu\text{m}$  height on the TM surface. The surface roughness increase to 0.39  $\mu\text{m}$  ( $R_a$  parameter) due to TM texture changes and surface peak counters increases (tab. 1, fig. 1a). The wetting angle increases to 8.8° which is a negative effect for the ophthalmology's implants. The full surface energy increases twice.

The figure 1 illustrates the results of texture changes and number of peaks ( $\gamma$  at the Fig.1b) in the horizontal section, which is formed on the sample center.

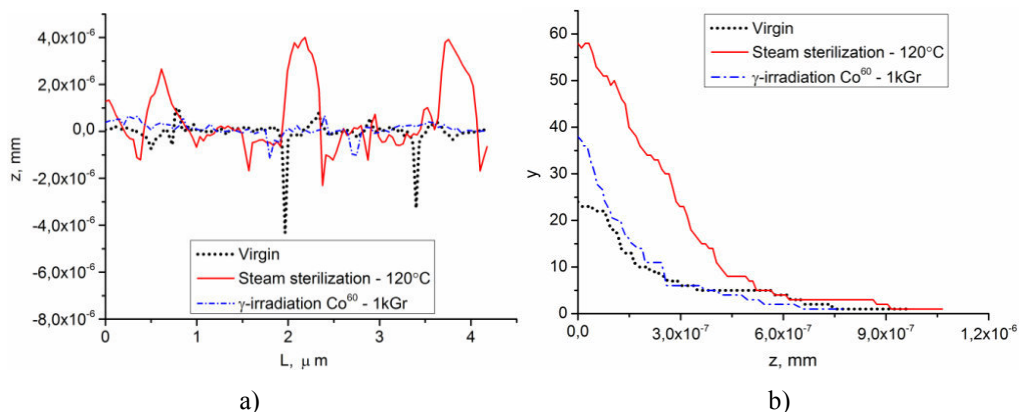


Fig. 1. Surface texture (a) and peak count (b) of the PET TM before and after sterilization

Figure 2 demonstrates the contact angle dependence on storage time after sterilization.

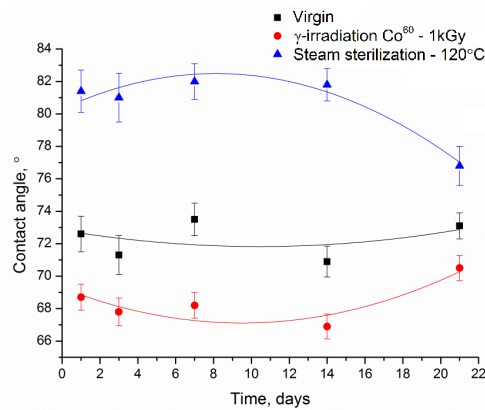


Fig. 2. Contact angle of the PET TM before and after sterilization.

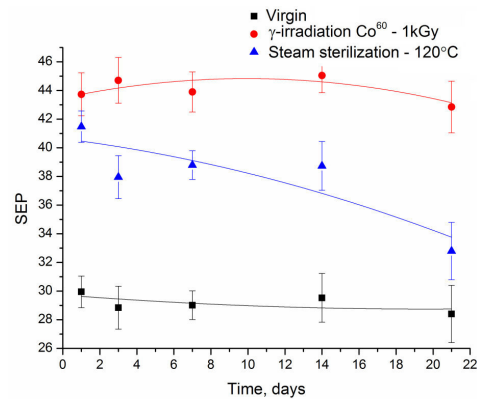


Fig. 3. Full surface energy of the PET TM before and after sterilization procedure

Data at the figure 2 show that contact angle changes during 21 days after steam and  $\gamma$ -irradiation  $\text{Co}^{60}$  sterilization insignificantly. Nevertheless the trend of alteration of wettability during the storage at the dark is different for  $\gamma$  and steam sterilization (see Fig.2). The surface becomes more hydrophilic in the case of steam sterilization whereas surface wettability varies slightly for the case of  $\gamma$ -sterilization. Minor changes of contact angle after sterilization indicates the long-term stability of the sterilized membrane properties.

The figure 3 illustrates the dependence of full surface energy on the storage time during 21 days after steam sterilization and  $\gamma$ -irradiation  $\text{Co}^{60}$  sterilization. The surface energy of PET TM increases after  $\gamma$ -irradiation sterilization and remain unchanged during 21 days. The surface energy of PET TM decreases after steam sterilization.

Thus, the positive effects of  $\gamma$ -irradiation is increasing of hydrophilic surface properties and unchanged during 21 days. Both sterilization methods lead to a change of surface topography. The degree of influence on the relief membrane surface is less after  $\gamma$ -irradiation sterilization than steam sterilization. So, the sterilization by  $\gamma$ -irradiation is preferred over steam sterilization in an autoclave.

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