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# Influence of Gamma Irradiation on the Surface Energy Characteristics and Wetting of Polytetrafluoroethylene

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Abstract—The effect of  $\gamma$ -radiation dose on the surface energy characteristics of commercial polytetrafluoroethylene (PTFE) of five different grades has been studied. In the course of PTFE irradiation in air, radiation oxidation leads to the appearance of functional groups and an increase in the acid—base component of the free surface energy and surface polarity; in this case, the dispersive component of the free surface energy decreases with dose because of the radiation-induced loosening, amorphization, and degradation of the polymer. The effect of  $\gamma$ -irradiation of PTFE on its wetting with aqueous solutions of the anionic surfactant sodium dodecyl sulfate has been studied for the first time. It has been found that irradiation increases the wetting and the work of adhesion of the surfactant solution due to the appearance of polar groups in the surface layer.

**Keywords:** polytetrafluoroethylene,  $\gamma$ -irradiation, free surface energy and its components, surfactant, sodium dodecyl sulfate, wetting

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The structure peculiarities of polytetrafluoroethylene (PTFE), which are responsible for the high strength of bonds between fluorine and carbon atoms in its macromolecules, determine a combination of valuable properties that distinguish this material from all other polymers: PTFE is chemically inert to any acids and their mixtures, alkalis, and solvents, and it has a wide range of operating temperatures and high electrical insulating and mechanical strengths. Due to its properties, PTFE is widely used in the chemical industry, electrical engineering, electronics, everyday life, and medicine [1, 2].

Like other perfluorinated polyolefins, PTFE has an extremely low free surface energy (FSE); therefore, it has a low coefficient of friction and antiadhesive properties. In studies related to the wetting of polymer surfaces by various liquid media, PTFE is a standard reference substance with nonpolar surface [3, 4]. The poor wettability and low surface adhesion of PTFE severely limit its application in various fields.

At the same time, the wettability of polymer surfaces is one of the most important characteristics for determining the applicability of a material in a specific area. It is a fundamental property of solid surfaces, which plays an important role in the pharmaceutical, cosmetic, paint and varnish, and construction industries, in adhesive technologies, in coating applications, in the processing of polymeric materials, and in the production of nanocomposites. In recent decades, wetting measurement has been used as a method for assessing the structure and properties of the surface layers of polymer materials [5, 6].

The surface modification of PTFE by radiation grafting polymerization of various monomers onto the polymer surface is frequently used to improve the contact properties of the PTFE surface and impart adhesion properties to it [7]. Electron beams [8], ion beams [9], various types of radiation ( $\gamma$ -rays [10, 11] and laser radiation [12]), and sequential radiation-laser irradiation, which stimulates the occurrence of chemical reactions in the surface layer of PTFE, can also be used to modify the surface of PTFE.

The wetting of polymer surfaces can be controlled using the solutions of surfactants. Based on the classical concepts of colloid chemistry, the wetting ability depends on both the adsorption interaction of a surfactant with the liquid—solid interface and a decrease in the surface tension at the liquid—gas interface. A relationship between the surface energy characteristics of polymers and their wetting with surfactant solutions has been established [13].

The main factor that limits (and excludes in most cases) the use of PTFE both in its pure form and as a constituent of compositions in special equipment

operating under radiation exposure is its extremely low radiation resistance [14]. The effect of radiation on PTFE leads to a significant decrease in its mechanical strength at relatively low absorbed doses from the point of view of radiation chemistry. We believe that the FSE and its components can be a criterion and indicator of changes in the structure of the surface layer and in the bulk of the polymer if we take into account the penetrating nature of  $\gamma$ -radiation. The acid-base component of the FSE, which is closely related to the presence of functional groups in the boundary layer, is especially informative; the functional groups are active centers that contribute to the occurrence of surface phenomena: wetting, adhesion, and adsorption. The FSE and its components are highly sensitive parameters, the measurement of which can be used to study effects on the surface layers of polymers and to obtain information on interphase processes in thermal and radiation oxidation, the deposition of nanolayers of amphiphilic compounds, the formation of a surface from a melt and solution, and the introduction of various modifying and functionalizing additives.

Thus, the action of  $\gamma$ -radiation causes changes in the structure and physicomechanical characteristics of polymeric materials due to the processes of structuring or destruction. These processes are reflected in changes in the surface energy characteristics: FSE, its dispersion and acid—base components, surface polarity, and acidity parameter.

The aim of this work was to evaluate the surface energy characteristics and surface polarity of PTFE samples of various brands depending on the dose of  $\gamma$ irradiation in atmospheric oxygen and a vacuum and to study the effect of the  $\gamma$ -radiation dose on the wetting of PTFE with aqueous solutions of an anionic surfactant, sodium dodecyl sulfate.

#### **EXPERIMENTAL**

Various brands of commercial polymer were used to study the effect of  $\gamma$ -irradiation on the surface properties of PTFE: PTFE in the form of a film 0.936 mm thick (Teflon® virgin electrical grade film of Teflon® (PTFE-1), a film made of Teflon® PTFE (PTFE-2), sheets made of Teflon® PTFE with a thickness of 1.56 mm and a width of 30 × 30 cm (PTFE-3), and a mechanical grade PTFE sheet (PTFE-4). They were purchased from McMaster-Carr Supply Company (Atlanta, Georgia). A Ftoroplast F-4 PTFE sheet (PTFE-5) from the Polymer Plant of the Kirovo-Chepetsk Integrated Chemical Works was also used in this study.

Sodium dodecyl sulfate from AppliChem (Spain) was used as a surfactant.

The contact angle was determined by the sessile drop method in a cell with a hydraulic seal using a KM-8 cathetometer equipped with a micrometric attachment. Liquid drops were applied using a microsyringe. For the good reproducibility of experiments, at least five drops of the same size were applied so that their diameter did not exceed 2-3 mm. All of the measurements were carried out at  $20 \pm 1^{\circ}$ C. Polymer samples onto which liquid droplets were applied were thoroughly degreased with acetone. The relative measurement error was 1.7%.

The FSE ( $\gamma_s$ ) of a polymer material and its acid– base ( $\gamma_s^{ab}$ ) and dispersive ( $\gamma_s^d$ ) components were determined from the contact angles of test liquids on the material surface. The surface tension of liquids ( $\gamma_l$ ) used in this work and its acid–base ( $\gamma_l^{ab}$ ) and dispersive ( $\gamma_l^d$ ) components were given in our previous works. The measured values were processed based on the Owens–Wendt equations [15]

$$0.5\gamma_{1}(1+\cos\theta) = (\gamma_{l}^{d})^{0.5} (\gamma_{s}^{d})^{0.5} + (\gamma_{l}^{ab})^{0.5} (\gamma_{s}^{ab})^{0.5}.$$

The dependence can be represented as a straight line in the Fowkes coordinates:

$$x = \left(\gamma_l^{ab} / \gamma_l^d\right)^{0.5}, \quad y = 0.5\gamma_1 (1 + \cos \theta) / \left(\gamma_l^d\right)^{0.5}.$$

The *y*-intercept is  $(\gamma_s^d)^{0.5}$ , and the slope, which corresponds to the acid–base FSE component, is  $(\gamma_s^{ab})^{0.5}$ . According to Fowkes [16], the free surface are different to depend on the surface of the

be represented as a sum of components due to different forces, and it is sufficient to take into account only two of them: the dispersive and acid—base components

$$\gamma_s = \gamma_s^{ab} + \gamma_s^d.$$

The wetting of solids by liquids is greatly influenced by surface roughness. The roughness of the polymer surfaces was determined using a profilometer. The profilometer operated based on the principle of feeling the irregularities of the measured surface with an inductive sensor probe—a diamond needle—in the course of tracing (moving the probe along the measured surface at a constant speed) and converting the height of the probe into a digital signal with the subsequent processing. Roughness parameters were calculated according to GOST [State Standard] 2789-73 with a base length of 0.8 mm and an integration path length of 12.5 mm.

The surface tension of surfactant solutions, which is required for calculating the work of adhesion, was determined with a K6 KRUSS tensiometer by the Du Nouy ring detachment method in a cell thermostatically controlled at 25°C. The relative measurement error was no greater than 0.3%.

The test materials were irradiated with  $^{60}$ Co  $\gamma$ -rays at 23°C using a Gammatok-100 radiation research facility with a dose rate of 4 Gy/s at the Institute of Problems of Chemical Physics, Russian Academy of Sciences. For irradiation in a vacuum, polymer sam-

ples were first evacuated to a residual pressure of 0.13 Pa; then, they were irradiated with  $\gamma$ -rays in molybdenum glass ampoules.

# **RESULTS AND DISCUSSION**

Table 1 summarizes the results of the calculations of FSE and its components by the Fowkes graphical relations from data on the wetting of the initial and irradiated surfaces of the PTFE test samples with test liquids. Table 1 also shows the polarity of the surfaces, which was determined as the ratio  $x^p = \gamma_s^{ab} / \gamma_s$  according to Vijayendran [17].

As an example, Fig. 1 shows plots in the Fowkes coordinates used for calculating the total FSE of PTFE-1.

The analysis of the surfaces of the initial PTFE samples showed that they had low values of FSE and the acid—base component; this fact is expected and consistent with published data [18]. It is more convenient to carry out a comparative analysis of the effect of radiation on a set of surface characteristics considering their dependences on radiation dose, which are given in Table 1. In this case, the individual columns of the table show the parameters of surface characteristics obtained by irradiation of the polymer in the presence of atmospheric oxygen and in a vacuum.

An analysis of the data presented in Table 1 showed that the history of PTFE samples (various preparation processes) can have a significant effect on the given dependences. Irradiation conditions (in atmospheric oxygen or in a vacuum) also play a significant role in changing the surface properties. The irradiation of PTFE in the presence of atmospheric oxygen led to an increase in the surface polarity mainly due to an increase in the acid—base component of the FSE in almost all of the samples. Thus, the acid—base component of the FSE of PTFE-1 increased by a factor of 7 upon irradiation with a dose of 60 kGy.

The largest difference in the acid-base components of the FSE was achieved in the sample of PTFE-3: it increased by a factor of 9.7 upon irradiation with a dose of 120 kGy. For the sheet fluoroplastic PTFE-5, at relatively small values of  $\gamma_s^{ab}$ , the surface polarity increased by a factor of 18 upon irradiation with a dose of 400 kGy. The sample of PTFE-4 exhibited a moderate increase in the surface polarity, whereas the polarity of sample 2 did almost not increase and even somewhat decreased after irradiation; this can be due to a possible decrease in crystallinity. However, the surface polarity can decrease at high radiation doses, as it was observed in the samples of PTFE-2, PTFE-3, and PTFE-4. This can be due to the destruction and amorphization of the PTFE surface. The possibility of amorphization upon the radiolysis of a PTFE matrix has been repeatedly noted in the



**Fig. 1.** Fowkes plots for calculating the total FSE of PTFE-1 samples with ten test liquids: (1) unirradiated, (2) irradiated with  $\gamma$ -rays in oxygen, and (3) irradiated with  $\gamma$ -rays in a vacuum. Radiation dose, 60 kGy.

case of polymer irradiation with accelerated MeV particles [13, 19] and  $^{60}$ Co  $\gamma$ -rays.

A decrease in the dispersion component of the FSE also indicated the surface amorphization of  $\gamma$ -irradiated PTFE (Table 1); obviously, the surface structure of the  $\gamma$ -irradiated polymer became partially or completely amorphous. The oxidative postradiation functionalization of the PTFE surface occurs as a result of the reaction of stabilized radicals formed on the irradiated PTFE surface with oxygen. Ultimately, this can lead to the formation of macromolecules with hydroxyl, carbonyl, and carboxyl bonds.

Indeed, according to published data [9], the chemical composition of the polymer surface changed noticeably after the  $\gamma$ -irradiation of commercial Ftoroplast F-4 PTFE powder. The concentration of fluorine decreased in radiolyzed PTFE, and the concentrations of carbon and oxygen increased as a result of the destruction of the polymer backbone with the formation of low-molecular-weight compounds and due to the oxidation reactions of free radicals stabilized on the irradiated polymer surface.

According to the experimental data, the sample surface of PTFE-2 had the highest resistance to  $\gamma$ -radiolysis. The FSE parameters changed in the course of the  $\gamma$ -irradiation of PTFE-2, but these changes were not so great in terms of quantity. This fact indicates that the modification and functionalization of some grades of PTFE can be performed without significant degradation. We will study these processes in the future because the practical use of various constructions requires improving the wettability and contact properties of PTFE surfaces and imparting adhesion properties to them.

A decrease in the values of the acid-base component of the FSE was observed upon the irradiation of

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	$^{1}x^{\mathrm{P}} \times 10^{2}$		$^{2}\gamma_{s}^{ab}$ , mN/m		$^{3}\gamma_{s}^{d}$ , mN/m		$^{4}\gamma_{s}$ , mN/m		
D, kGy	5	6	5	6	5	6	5	6	
PTFE-1									
0	3.07	3.07	0.61	0.61	19.28	19.28	19.89	19.89	
0.5	1.83	4.7	0.37	1.15	19.9	20.36	20.27	21.51	
2	2.82	6.82	0.67	1.69	23.05	23.08	23.72	24.77	
4	6.62	6.89	1.82	1.78	25.69	24.45	27.51	26.23	
6	15.31	7.32	4.26	1.96	23.56	25.92	27.82	27.88	
8	10.99	8.7	2.66	2.3	21.55	24.14	24.21	26.44	
PTFE-2									
0	3.00	3.00	0.62	0.62	20.07	20.07	20.69	20.69	
0.5	1.45	0.49	0.30	0.11	20.44	22.42	20.74	22.53	
1	1.18	2.30	0.28	0.53	23.39	22.36	23.67	22.89	
2	1.18	2.68	0.30	0.60	23.19	22.26	23.49	22.86	
4	1.90	2.08	0.41	0.47	21.21	22.11	21.62	22.58	
6	0.83	4.08	0.17	1.01	20.29	23.74	20.46	24.75	
PTFE-3									
0	3.31	3.31	0.87	0.87	25.39	25.39	26.26	26.26	
1	4.23	3.97	1.09	0.76	24.66	18.37	25.75	19.13	
2	2.08	5.3	0.54	1.04	25.47	18.67	26.01	19.71	
4	3.56	2.24	0.97	0.42	23.56	18.37	24.53	18.79	
6	6.78	0.54	1.72	0.11	23.64	20.39	25.36	20.50	
8	9.96	1.39	2.67	0.28	24.13	19.82	26.80	20.10	
PTFE-4									
0	3.97	3.97	0.82	0.82	19.82	19.82	20.64	20.64	
0.5	5.39	2.07	1.29	0.38	22.6	17.98	23.89	18.36	
1	7.11	2.07	1.8	0.36	23.5	17.04	25.30	17.40	
2	8.06	2.58	2.22	0.4	25.32	15.08	27.54	15.48	
4	8.73	2.31	2.47	0.4	25.82	16.93	28.29	17.33	
6	7.02	2.58	1.86	0.5	24.63	18.86	26.49	19.36	
8	6.38	0.84	1.68	0.17	24.63	20.09	26.31	20.26	
PTFE-5									
0	0. 49		0.09		18.2		18.29		
1	0.94		0.19		19.97		20.16		
4	1.99		0.37		18.22		18.59		
8	9.83		1.62		14.86		16.48		
20	8.87		1.52		15.62		17.14		
40	10.15		1.84		16.28		18.12		

Table 1. Surface energy characteristics of PTFE samples irradiated with  $\gamma$ -rays

<sup>1</sup> surface polarity; <sup>2</sup> acid–base component of FSE; <sup>3</sup> dispersive component of FSE; <sup>4</sup> total FSE; <sup>5</sup> polymer irradiation in air; <sup>6</sup> polymer irradiation in vacuum.

PTFE in a vacuum. This is evident from the tabular data for PTFE-1, PTFE-3, and PTFE-4. The dispersive component of sample 1 changed insignificantly, and this component of sample 3 decreased, as compared to that upon irradiation in air. The value of  $\gamma_s^{ab}$ 

for the PTFE-1 sample increased with radiation dose; however, the values in the range of 60–80 kGy were lower than those in the case of irradiation in atmospheric oxygen, and the curve exhibited no maximum. However, the acid–base component of the FSE for

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Fig. 2. Dose dependences of (a) the surface polarity, (b) the acid–base component of FSE, (c) the dispersive component of FSE, and (d) the total surface energy of PTFE-5  $\gamma$ -irradiated in atmospheric oxygen.

PTFE-3 and PTFE-4 remained almost unchanged upon irradiation in a vacuum.

The increase of  $\gamma_s^{ab}$  with absorbed dose for PTFE-2 is unusual, but the values themselves were insignificant: the increase was only 0.4 mN/m, and it was 1.7 MN/m for PTFE-1. A comparison of the data obtained after polymer irradiation in a vacuum and in the presence of air showed that, in general, the radiation-induced oxidation of the surface significantly decreased in a vacuum. The surface polarity was pre-

dominantly caused by changes in  $\gamma_s^{ab}$ .

The FSE dispersive component in a vacuum for the PTFE-3 and PTFE-4 samples was smaller than that under the conditions of irradiation in air. For PTFE-1 and PTFE-2, it changed insignificantly. All these data indicate the specifics of the preparation and structure of PTFE films; additional studies are needed in order to determine the mechanism of radiolysis. It is likely that the results were influenced by different sample densities.

The data presented in Fig. 2 for a Ftoroplast F-4 sheet (PTFE-5) in a wide range of irradiation doses in

air show predictable changes in surface energy characteristics: radiation-induced oxidation led to the appearance of functional groups and an increase in the acid—base component of the FSE and the polarity of the surface; in this case, the dispersion component of the FSE decreased with the dose, apparently, due to the loosening, amorphization, and degradation of the polymer.

The oxidation reactions of free radicals generated by irradiation can be the main chemical processes leading to changes in the surface polarity of radiolyzed PTFE. In the case of PTFE irradiation at 77 K, signals from several paramagnetic centers overlapped in the EPR spectrum. The components of signals from the central radicals  $\sim F_2 CFC \cdot CF_2 \sim$  [20] and allyl  $\sim$ F<sub>2</sub>CFC<sup>•</sup>CF=CFCF<sub>2</sub> $\sim$  macroradicals can be detected in the spectrum, and the spectra of positive and negative charges captured by various traps can be recorded [21]. There are contradictory published data on the presence of a signal due to terminal  $\sim F_2 C F_2 C^{-1}$ macroradicals in the EPR spectrum of irradiated PTFE. According to Milinchuk et al. [20], terminal macroradicals were formed by C-C bond scission



**Fig. 3.** Wetting isotherms of PTFE-5 surfaces with aqueous solutions of sodium dodecyl sulfate: (1) unirradiated PTFE-5, (2) PTFE-5  $\gamma$ -irradiated to 40 kGy, and (3) PTFE-5  $\gamma$ -irradiated to 400 kGy.

during the radiolysis of PTFE at both 77 and 300 K, and their concentration in the dose range of 10– 100 Mrad in a vacuum was approximately 10 times lower than the concentration of central macroradicals [22]. On the other hand, according to Astakhov et al. [23], terminal macroradicals were not detected in the EPR spectrum of PTFE irradiated at 77 K.

Studies carried out with linear perfluoroalkanes (*n*-PFAs) and PTFE in order to clarify the effects of the chain length and physical state on the mechanism of radiolysis of *n*-PFAs showed [14] that terminal radicals formed by C-C bond breaking were not stabilized in *n*-PFA and PTFE matrices in the radiolysis at both 77 and 300 K. In this case, only the  $\sim F_2 C F_2 C$ . radicals, which are formed upon the detachment of F atoms from terminal  $F_3C$  groups, were detected in the EPR spectra of irradiated *n*-PFAs and PTFE. As a result, the proportion of trapped terminal radicals in a high-molecular-weight *n*-PFA, such as PTFE with a molecular weight of  $\sim 10^6$ , was almost a thousand times smaller than that of central radicals. Therefore, the spectrum of the  $\sim F_2 C F_2 C'$  radicals was almost absent from the EPR spectrum of irradiated PTFE.

Thus, central macroradicals formed as a result of the abstraction of fluorine atoms are the main radical product of the  $\gamma$ -radiolysis of PTFE in a vacuum. Note that the mechanism of PTFE radiolysis changes significantly in the course of radiolysis at high temperatures (near or above the melting point of PTFE crystallites) [24]) or in the presence of oxygen [25, 26]. The formation of terminal ( $-CF_2-CFOO \cdot$ ) and central ( $-CF_2-CFOO \cdot -CF_2-$ ) peroxide radicals is the dominant process in the radiation modification of PTFE in the presence of molecular oxygen [20]. Perfluoroalkyl and peroxide radicals in the PTFE chain have very long lifetimes at room temperature. Their presence is reflected in changes in the surface energy characteristics of the polymer.

The effect of the dose of  $\gamma$ -irradiation of PTFE-5 on its wetting with aqueous solutions of an anionic surfactant (sodium dodecyl sulfate) was studied.

Table 1 summarizes the surface energy characteristics, such as FSE, its components, and surface polarity, of PTFE-5 depending on the radiation dose. As can be seen, PTFE-5 had low values of  $\gamma_s^{ab}$ , which indicated the almost complete absence of polar groups from the surface layer. The study of the wetting of such polymers is useful both for practical purposes and for expanding our understanding of the combined effect of surfactants, the nature of the polymer, and the intensity of irradiation on the character of wetting.

Figure 3 shows the wetting isotherms of PTFE-5 surfaces with aqueous solutions of SDS. These isotherms indicate that the surface of PTFE-5 is hydrophobic:  $\cos \theta < 0$ . Irradiation with <sup>60</sup>Co  $\gamma$ -rays at a dose of to 400 kGy did not lead to hydrophilicity, although it slightly increased the surface polarity: on irradiated surfaces,  $\cos \theta$  was also below zero. An inversion of wetting was observed at certain surfactant concentrations upon applying a surfactant solution to the surface of PTFE-5; this fact indicates the hydrophilization of the surface as a result of the orientation of surfactant molecules in a saturated adsorption monolayer by hydrocarbon moieties toward the polymer surface. As the surfactant concentration was increased,  $\cos \theta$ increased to a certain value. A further increase in the surfactant concentration on the surface of PTFE-5 did not change  $\cos \theta$ . A comparative analysis of curves 1-3 showed that irradiation increased wetting; it is likely that this was due to the appearance of polar groups in the surface layer. An increase in the radiation dose by a factor of 10 (40 and 400 kGy) increased  $\cos \theta$  by a factor of 2 at a 0.25% concentration of surfactant solution.

Based on the results of evaluating the wetting and data on the surface tension of surfactant solutions, we calculated the work of adhesion of sodium dodecyl sulfate solutions to the surface of PTFE-5 according to the Young–Dupré equation

$$W_{ad} = \gamma_l (1 + \cos \theta).$$

Table 2 summarizes the results of calculating the work of adhesion. The data in Table 2 show that, in general, the adhesion of the surfactant to the surface of irradiated PTFE-5 samples was somewhat higher than that to the initial one. Thus,  $W_{ad}$  increased by an average factor of 1.2. It should be noted that sheet F4 fluoroplastic exhibited a sufficiently high radiation resistance, and this was confirmed by an analysis of the interaction of the surfactant with the surfaces of the initial and irradiated samples.

	$W_{ad}$ , mN/m						
<i>C</i> , %	Radiation dose, kGy						
	0	40	400				
0.125	61.32	61.80	64.80				
0.25	59.05	63.92	67.26				
0.50	51.24	59.24	60.96				
1.00	51.24	55.55	58.19				

Table 2. Work of adhesion of surfactant solutions to the surfaces of unirradiated and  $\gamma$ -irradiated PTFE-5

#### CONCLUSIONS

• An analysis of FSE and its components was carried out for the surfaces of five brands of commercial PTFE irradiated with <sup>60</sup>Co  $\gamma$ -rays in the dose range from 5 to 80 kGy (to 400 kGy for PTFE-5) in a vacuum or in the presence of oxygen.

• The irradiation of PTFE in the presence of atmospheric oxygen leads to an increase in the surface polarity mainly due to an increase in the acid—base component of the FSE. The results can be associated with oxidative processes in the surface layer.

• The irradiation of PTFE in the presence of atmospheric oxygen leads to an increase in the surface polarity mainly due to an increase in the acid—base component of the FSE. The results can be associated with oxidative processes in the surface layer.

• The irradiation of PTFE in a vacuum does not lead to a significant increase in the FSE, its acid—base component, and the surface polarity.

• At high radiation doses, the polarity of the PTFE surface and the dispersion component of the FSE decrease; this can be due to the destruction and amorphization of the polymer.

• The wetting isotherms of PTFE surfaces with aqueous solutions of a surfactant (sodium dodecyl sulfate) show that irradiation leads to an increase in wetting.

• The work of adhesion of the solutions of sodium dodecyl sulfate to the surface of PTFE increases with radiation dose.

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