Energy characteristics of poly(olefin ketone) surfaces at different interfaces

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The energy characteristics of surfaces of new polymers belonging to the class of poly(olefin ketone)s at the interfaces with air and polar (water) and nonpolar (octane) liquids were determined by the contact angle method. The objects of the study were binary copolymer (propylene—carbon monoxide, PCO) and ternary copolymers (propylene—carbon monoxide—ethylene—carbon monoxide, PECO; but-1-ene—carbon monoxide—ethylene—carbon monoxide, BECO). It was shown that the ternary copolymers can be recommended for further medicobiological tests as a base for the development of hemocompatible materials. The equilibrium values of the interfacial energy of poly(olefin ketone)s at the interfaces with water and octane make it possible to predict their adhesion properties for designing composite materials.

Key words: poly(olefin ketone)s, wetting, surface energy, adhesion, hemocompatibility, polymer composite materials.

Poly(olefin ketone)s (POK) are new strictly alternating copolymers of carbon monoxide with olefins. They possess a complex of unique properties and, therefore, attract researchers´ attention. These properties are due to the presence of the polar comonomer in the POK chain and a possibility to introduce various functional substituents at the carbonyl group.[1,2] In addition, POK possess high strength, thermal stability, and relatively low prime cost.[3]

Structural morphological organization of POK surfaces is being under intensive investigation.[4,5] Information about the energy characteristics of surfaces of these polymers is very restricted.[6] In particular, data on the surface energy of POK are lacking, whereas this is the parameter that determines the occurrence of many processes on contact of polymer with the environment (adsorption, chemical reactions, strength decrease, and adhesion of living cells). Prolong contact with various media changes the surface properties of polymers modifying the energy characteristics of the surface.[7] Therefore, for the prediction of the behavior of the polymer upon a prolong contact with various media it is insufficient to only determine the specific free surface energy of the polymer at the interface with air. Under these conditions, the mechanical stability of the polymer material will be determined by the equilibrium value of the interfacial energy at the polymer—medium interface.

For the practical use of polymers, for instance, in medicine, biotechnology, or in designing composite materials, control over changes in the energy characteristics of the surface is needed. For this purpose, the contact angle method is a very convenient and informative express procedure. This allows one to determine the specific free surface energy, *viz.*, interfacial energy of polymer in contact with the polar and nonpolar phases, on the basis of measurements of contact advancing and receding angles and selective contact angle.[8]

For the determination of the specific free energy of solids by the contact angle method, several approaches and procedures are used, which are based on the molecular wetting theory.[8,9] The studies in the field of determination of the equilibrium interfacial energy of polymers contacting for a long time with liquid media of various polarity resulted in the development of a procedure that makes use of values of contact angles to estimate the state of the surface layer of polymer equilibrated with the polar and nonpolar phases.[7,10]

The purpose of this work is to determine the specific free surface energy of poly(olefin ketone)s and the interfacial energy at the interface of polymers with polar and nonpolar media and to predict a possibility of practical use of these polymers on the basis of the data obtained.

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Experimental

The following copolymers synthesized at the Institute of Problems of Chemical Physics of the Russian Academy of Sciences (Chernogolovka, Moscow Region) were used: binary copolymer propylene—carbon monoxide (PCO, **1**), ternary copolymers propylene—carbon monoxide—ethylene—carbon monoxide (PECO, **2**), and but-1-ene—carbon monoxide—ethylene—carbon monoxide (BECO, **3**) (Table 1).

^{1}H NMR spectroscopy was used to compare the content of the ethylene—carbon monoxide comonomers in the ternary copolymers. The ^{1}H NMR spectra were recorded on a Bruker-Avance instrument with the working frequency 400 MHz in $CDCl_3$. The ^{1}H NMR spectra of the PECO and BECO samples contained characteristic broadened signals of protons of the methyl and ethyl groups at δ 0.8—1.2 and signals of protons of the main chain at δ 2.4—3.0. A comparison of the integral intensities of the proton signals in these spectral regions showed that in the BECO sample the content of the ethylene—carbon monoxide comonomers is approximately 5 times higher than in the PECO sample. Copolymers of the purity not less than 98% were used, the content of the third comonomer being 5—7%.

Polymer films were obtained by depositing a 3% solution of the corresponding polymer in chloroform on the surface of aluminum plates 1.0×1.5×0.1 cm in size followed by drying for 24 h at room temperature. The film thickness was 200 nm.

Contact angles were measured with an MG horizontal microscope with a goniometric attachment. Under the advancing conditions (θ_a) droplets were deposited on the solid surface (Fig. 1, *a*), while under receding conditions (θ_r) an air bubble was brought to the surface of the sample immersed in water (see Fig. 1, *b*). Finally, selective wetting conditions (θ_{wo}) assume bringing an octane droplet to the surface of the sample immersed in water (see Fig. 1, *c*) and depositing a water droplet on the sample placed in octane (see Fig. 1, *d*). The volumes of droplets (bubbles) was 10 μL. The accuracy of angle measurement was ±1°. The contact angles of six—nine droplets was measured for each sample. The root-mean-square deviation for measurements of contact angles was ±(1—2)°. All measurements were carried out at 20 °C in a closed chamber 2—3 min after deposition of droplets (bubbles) on the sample surface.

The advancing and receding contact angle values were used to characterize the polymer films. The samples were used for which deviations of the contact angles of water from those listed in Tables 2 did not exceed ±1°. The film surfaces were examined by scanning electron microscopy on a JSM-6490 LV instrument (Jeol). The microelectronic images show that the surface of the PCO films is more rough than those of the ternary copolymers (Fig. 2). This corresponds to the difference in the ordinal hysteresis value of the contact angles $\Delta\theta = \theta_a - \theta_r$ (see Table 2).

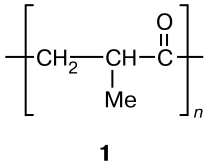
Table 1. Molecular weights (M), melting points (T_m), glassy transition temperatures (T_g), and crystallinity (α) of poly(olefin ketone)s **1—3**

Copolymer	M*	T_g	T_m	α (%)
		/°C		
PCO (**1**)	48000	19.8	127.3	—
PECO (**2**)	255000	10.0	63.9	1.2
			85.1	1.2
			145.4	8.15
BECO (**3**)	38000[11]	−7.9	151.7	4.10
		44.8		

* The weight-average molecular weights are presented for PCO and PECO, and the number-average molecular weight is given for BECO.

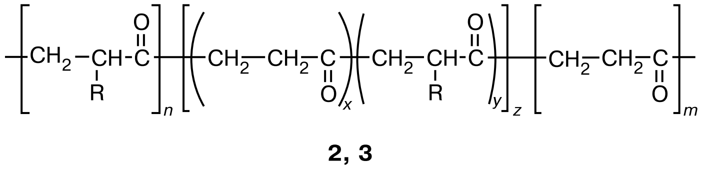
Several theoretical approaches are known for the calculation of the energy characteristics of solids using experimental values of the contact angles of test liquids with the known surface tension (γ_L) and values of the polar ($\gamma^p{}_L$) and dispersion ($\gamma^d{}_L$) components of surface tension.[8,12] The most part of them are based on the molecular wetting theory using the concepts about intermolecular interactions of a solid and a test liquid at the interface. It is assumed that the adsorption of vapors of the wetting liquid is negligibly low on the low-energy solid surfaces among which are the majority of polymers.[8] At present the problem of description of the contribution of the polar component of the specific free surface energy is not finally solved, and there is no single point of view concerning the approach that should be used in calculations.[8] It should be mentioned that the results of determination of the specific free surface energy depend on the choice of test liquids; in addition, some researchers use literature data rather than the experimental values of the $\gamma^p{}_L$ and $\gamma^d{}_L$ parameters obtained in their own studies. Although there is no universal theoretical model describing all possible cases of interaction of the liquid with the solid surface and many questions about the interpretation of data obtained by the contact angle method remain unanswered, the measurement of contact angles is most widely used for the determination of the energy characteristics of solid surfaces.

In this work, the specific free surface energy of polymers (γ_S) was determined by the two-liquid method.[13] According to this method, dispersion and polar interactions of the solid and test liquid near the interface obey the geometric-average rule, and the $\gamma^p{}_S$ value includes all types of polar intermolecular forces. The two-liquid method makes it possible to obtain more reliable data, because the sensitivity of the results to the choice of test liquids is minimum for this approximation. Bidistilled water and ethylene glycol (purity at least 99.8% with the water content not



$$-\!\left[CH_2-\underset{Me}{CH}-\overset{O}{\overset{\|}{C}}\right]_n\!-$$

1



$$-\!\left[CH_2-\underset{R}{CH}-\overset{O}{\overset{\|}{C}}\right]_n\!\left[\left(CH_2-CH_2-\underset{O}{\underset{\|}{C}}\right)_x\left(CH_2-\underset{R}{CH}-\overset{O}{\overset{\|}{C}}\right)_y\right]_z\!\left[CH_2-CH_2-\underset{O}{\underset{\|}{C}}\right]_m\!-$$

2, 3

R = Me (**2**), Et (**3**)

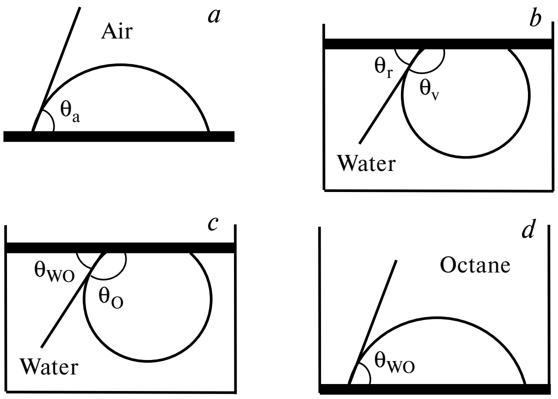


Fig. 1. Scheme of measurements of the advancing (*a*) and receding (*b*) contact angles and the selective contact angle (*c*, *d*). The contact angles are designated according to the known procedure.[8]

more than 0.005% (Sigma Aldrich), which was stored above molecular sieves 3 Å (Sigma Aldrich)) were used as test liquids.

The choice of water and ethylene glycol as test liquids for the determination of the γ_S parameter is due to the fact that they are prone to oxidation to a lesser extent than other test liquids. In addition, rather high surface tension of these liquids and a low viscosity make it possible to obtain reliably measured values of equilibrium contact angles. The interfacial energy (γ_{SL}) was determined using water and octane (purity ≤99%, Merck) according to a procedure described earlier.[10] The surface tension of water, ethylene glycol, and octane was determined by the Wilhelmy method (equilibration of the plate of sponge platinum) with the accuracy $\Delta\gamma_L = \pm 0.5$ mJ m^{-2}. The polar and dispersion components of γ_L of water and ethylene glycol were determined by the advancing contact angles of these liquids on Teflon-4, accepting $\gamma_S = 18$ mJ m^{-2} (see Refs 8, 9, and 14). The accuracy of determination was $\Delta\gamma_S = \pm(0.5-0.7)$ mJ m^{-2}. The results obtained (Table 3) are well consistent with the literature data.[15]

The surface energy of polymers (γ_S) and its polar ($\gamma^p{}_S$) and dispersion ($\gamma^d{}_S$) components were calculated using the system of two equations[13]

$$\begin{cases}(1 + \cos\theta_{L_1})\gamma_{L_1} = 2(\gamma^d{}_{L_1}\gamma^d{}_S)^{0.5} + 2(\gamma^p{}_{L_1}\gamma^p{}_S)^{0.5}, & (1)\\[1em] (1 + \cos\theta_{L_2})\gamma_{L_2} = 2(\gamma^d{}_{L_2}\gamma^d{}_S)^{0.5} + 2(\gamma^p{}_{L_2}\gamma^p{}_S)^{0.5}, & (2)\end{cases}$$

where θ_{L_1} and θ_{L_2} are the advancing contact angles of water and ethylene glycol, respectively; $\gamma^p{}_{L_1}$, $\gamma^p{}_{L_2}$, $\gamma^d{}_{L_1}$, and $\gamma^d{}_{L_2}$ are the polar and dispersion components of the surface tension of water and ethylene glycol; $\gamma_S = \gamma^d{}_S + \gamma^p{}_S$.

Table 2. Contact angles of the test liquids (θ/deg) on the surface of poly(olefin ketone)s **1**—**3** and in the polymer—liquid model systems

Polymer	Water			EG*	Polymer—water		Polymer—octane
	θ_a	θ_r	$\Delta\theta$	θ_a	θ_o	θ_v	θ_{wo}
PCO (**1**)	80	43	37	60	110	141	131
PECO (**2**)	63	43	20	50	122	142	99
BECO (**3**)	60	43	23	50	130	150	126

* EG is ethylene glycol.

The molecular wetting theory allows one to calculate γ_{SW} by the equation

$$\gamma_{SW} = \gamma_S + \gamma_W - 2(\gamma^d{}_S \cdot \gamma^d{}_W)^{0.5} - 2(\gamma^p{}_S \cdot \gamma^p{}_W)^{0.5}, \quad (3)$$

where γ_S, $\gamma^d{}_W$, and $\gamma^p{}_W$ are the surface tension of water and its dispersion and polar components, respectively.[9] The γ_{SW} parameter was calculated using the values γ_W, $\gamma^d{}_W$, and $\gamma^p{}_W$ (see Table 3) and γ_S, $\gamma^d{}_S$, and $\gamma^p{}_S$ (Table 4) calculated from the experimental values of the contact angles of the test liquids by Eqs (1) and (2). However, upon a long contact of the polymer with the liquid medium the γ_{SW} values calculated by Eq. (3) can be far from the equilibrium values. This is related to a possible reorientation of functional groups of the surface layer and to the inclusion of water into the surface layer of the polymer upon the prolong contact with water.[10]

The interfacial energy of the polymers equilibrated with water at the interface with water ($\gamma_{SW(W)}$) and polymers equilibrated with octane at the interface with water ($\gamma_{S(O)W}$) were determined by the known procedure.[7,10] The polymer films were stored for 24 h in water, and then the contact angles of octane droplets (θ_O) and air bubbles (θ_V) brought to the surface of the samples immersed in water (see Fig. 1, *b*, *c*) were measured. According to Refs 7 and 10, the $\gamma_{S(W)W}$ and $\gamma_{S(O)W}$ parameters were calculated on the basis of these measurements. The $\gamma_{S(W)W}$ value was calculated by the equation

$$\gamma_{S(W)W} = [(\gamma^p{}_{SW})^{0.5} - (\gamma^p{}_W)^{0.5}]^2 + {} + [(\gamma^d{}_{SW})^{0.5} - (\gamma^d{}_W)^{0.5}]^2, \quad (4)$$

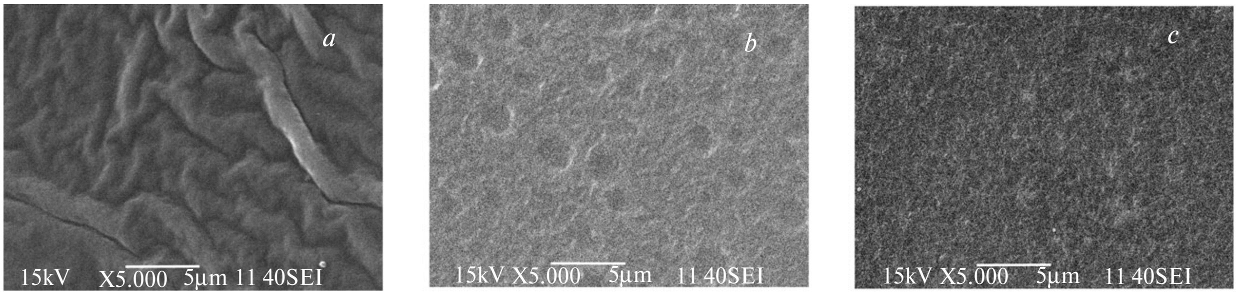


Fig. 2. Microelectronic images of the surfaces of the PCO (*a*), PECO (*b*), and BECO (*c*) films.

Table 3. Surface tension (γ_L) and the dispersion ($\gamma^d{}_L$) and polar ($\gamma^p{}_L$) components of the surface tension for the test liquids

Liquid	$\gamma^p{}_L$	$\gamma^d{}_L$	γ_L
		/mJ m^{-2}	
Water	50.8	21.8	72.6
Ethylene glycol	19.0	29.3	48.3
Octane	—	21.8	21.8

where $\gamma^p{}_{SW}$ and $\gamma^d{}_{SW}$ are the polar and dispersion components of the specific free surface energy of the polymers equilibrated with water. The $\gamma^p{}_{SW}$ and $\gamma^d{}_{SW}$ values were determined using the following equations:

$$\gamma^p{}_{SW} = (\gamma_W - \gamma_O - \gamma_{OW}\cos\theta_O)^2/4\gamma^p{}_W, \tag{5}$$

where γ_{OW} = 50.8 mJ m^{-2} is the interfacial tension at the octane—water interface[10]; γ_W and γ_O are the surface tension of water and octane, respectively, and

$$\gamma^d{}_{SW} = (\gamma_{OW}\cos\theta_O - \gamma_W\cos\theta_V + \gamma_O)^2/4\gamma_O. \tag{6}$$

The calculation of $\gamma_{S(O)W}$ was performed by the equation

$$\gamma_{S(O)W} = \gamma^p{}_{SW} + [(\gamma^d{}_{SO})^{0.5} - (\gamma^d{}_W)^{0.5}]^2. \tag{7}$$

In the calculations, the assumption that the dispersion components of the surface energy of a solid are independent of the nonpolar medium nature, *i.e.*, they are the same for all hydrocarbons and for air $\gamma^d{}_{OW} \approx \gamma^d{}_S$, was used (see Ref 7 and 10).

To determine the interfacial energy of the copolymers equilibrated with octane at the interface with octane γ_{SO}, the samples were stored for 24 h, and the contact angles of water droplets (θ_{WO}) deposited on the surface of the samples in octane (see Fig. 1, *d*) were measured. The γ_{SO} values were calculated by the equation

$$\gamma_{SO} = \gamma^d{}_{SO} + \gamma^p{}_{SO} + \gamma_O - 2(\gamma^d{}_{SO}\gamma_O)^{0.5}, \tag{8}$$

where the polar component of the interfacial energy of the octane-saturated polymer at the interface with octane $\gamma^p{}_{SO}$ = $= (\gamma_{WO}\cos\theta_{WO} + \gamma_W - \gamma_O)^2/4\gamma^p{}_W$ (see Ref. 10).

Results and Discussion

Surfaces of all polymers are wetted with water (θ < 90°). In this case, the surfaces of the ternary copolymers are more hydrophilic due to the introduction of fragments containing the blocks (ethylene—carbon monoxide)$_n$ into the polymer chain structure. An increase in the hydrophilicity of the surface results in an increase in the polar component of the specific surface and a higher γ_S value (see Table 4). An elongation of the side chain in the ternary copolymers is accompanied by an increase in the $\gamma^p{}_S$ parameter and a decrease in the $\gamma^d{}_S$ parameter. This result and the ^{1}H NMR data indicate that the content of the comonomers ethylene—carbon monoxide in the polymer chain structure determines the γ_S value of ternary POK.

For PCO the γ_{SW} value calculated by Eq. (3) is higher than that for the ternary copolymers; for PECO and BECO the γ_{SW} parameter values are close (see Table 4). This result is related to the higher hydrophilicity of the ternary copolymers. For all copolymers studied $\gamma_{SW} > \gamma_{S(W)W}$, which can be due to the reorientation of the surface carbonyl groups when the polymers are equilibrated with water and the difference in polarities at the interface "polymer equilibrated with water—water" decreases. Another possible reason can be the inclusion of water into the surface layer of the polymer. A comparison of the values of the contact angles θ_r and ($180 - \theta_v$) presented in Table 2 indicates a decrease in the interfacial energy of the polymer—water interface upon equilibration.

The $\gamma_{S(W)W}$ parameter value makes it possible to predict hemocompatibility of polymer materials.[7,16] A complex of studies is usually used for the evaluation of hemocompatibility of polymers, such as studies of toxicity of a polymer, reactions of a living system to the introduction of a foreign surface, influence of the biological medium on the mechanical stability of the material, and changes in the structure of the material surface upon the interaction with components of blood and upon oxidation.[16] Therefore, the development of criteria for hemocompatibility on the basis of simpler and faster physicochemical tests is a topical problem. The behavior of the polymer in a biological medium is determined by the equilibrium value of the interfacial energy at the polymer—water interface ($\gamma_{S(W)W}$). This value should be close to the interfacial energy of the cell—blood plasma interface, which is equal to 1—3 mJ m^{-2} (Ruckenstein´s criterion).[7,10] No adsorption of fibrinogen leading to thrombosis and no adhesion of

Table 4. Specific free surface energy of poly(olefin ketone)s **1**—**3** at different interfaces and its polar (γ^p) and dispersion (γ^d) components

γ/mJ m^{-2}	PCO (**1**)	PECO (**2**)	BECO (**3**)
		Polymer—air	
γ_S	27.2	38.8	42.3
$\gamma^d{}_S$	15.7	8.4	6.2
$\gamma^p{}_S$	11.5	30.4	36.1
		Polymer—water	
γ_{SW}	14.5	5.7	6.0
$\gamma_{S(W)W}$	8.9	3.6	2.4
$\gamma^d{}_{SW}$	42.4	31.1	31.0
$\gamma^p{}_{SW}$	22.8	29.7	34.2
		Polymer—octane	
$\gamma_{S(O)W}$	23.3	32.8	39.0
γ_{SO}	2.0	12.1	6.9
$\gamma^p{}_{SO}$	1.5	9.1	2.2

pathogenic bacteria that provoke inflammatory processes occur on surfaces with such an energy characteristic. The determination of the $\gamma_{S(W)W}$ parameter and a comparison of the data obtained with the criterion value represent an important stage when optimizing the choice of the polymer for further medical biological studies as a hemocompatible material.

The results listed in Table 4 show that for the ternary polymers the $\gamma_{S(W)W}$ parameter value corresponds to Ruckenstein´s criterion.[7,10] The increase in the contact angles θ_O in the model system "polymer—water" in the order PCO < PECO < BECO (see Table 2) indicates the enhancement of the role of polar interactions of the polymer with the aqueous medium upon the introduction of the ethylene—carbon monoxide comonomers into the structure of the polymer chain of POK in order to provide hemocompatibility.

The determination of the $\gamma_{S(W)W}$, $\gamma_{S(O)W}$, and γ_{SO} parameters for the model systems polymer—polar phase and polymer—nonpolar phase allows one to predict the adhesion properties of polymers on contact with phases of different nature. The work of adhesion of the polymer to the liquid modeling the polar or nonpolar phase with the surface tension (γ_L), $W_a = \gamma_S + \gamma_L - \gamma_{SL}$, depends on the specific free surface energy of the polymer at the interface with air (γ_S) and the equilibrium value of the interfacial energy (γ_{SL}). The γ_{SL} value is determined by intermolecular interactions polymer—model liquid and is minimum for good adhesion. Thus, the values of the $\gamma_{S(W)W}$, γ_{SO}, and $\gamma_{S(O)W}$ parameters can be used for the calculation of the work of adhesion and prediction of the adhesion properties of polymers when two polar, two nonpolar, and polar and nonpolar phases, respectively, are combined.

The interrelation of the adhesion properties of the ternary copolymers with the energy characteristics of their surface is illustrated by the data in Tables 5 and 6. The data[11] on the tensile strength of the monolayer glass fiber plastics, in which PECO and BECO were used as a dressing for the combination of the glass fiber with polymer matrices of different nature, are given in Table 5. The work of adhesion to the polar phase (W_{a_1}) for the ternary copolymers is higher than that for PCO, and BECO is a better adhesive to the polar phase than PECO (see Table 6). This result shows that the enhancement of POK adhesion to the polar phase is due to an increase in the content of the ethylene—carbon monoxide comonomers in the polymer chain structure. Thus, BECO is a better dressing than PECO when polar phases are combined. This assumption agrees with the data in Table 5: the glass fiber plastic based on the polar polyamide matrix, in which the glass fiber is dressed by BECO, has a higher strength. The PCO and PECO copolymers are characterized by a larger work of adhesion when the polar and nonpolar phases are combined (W_{a_2}) (see Table 6). The decrease in the W_{a_2} parameter on going from PECO to BECO corresponds to a decrease in the rupture strength of the glass fiber plastic based on the nonpolar polyethylene matrix (see Table 5). The values of the work of adhesion to the nonpolar phase (W_{a_3}) for PCO and PECO are close (see Table 6). The elongation of the side chain in the ternary copolymer increases the work of adhesion W_{a_3} on going from PECO to BECO. This result demonstrates that of all copolymers studied BECO provides the most efficient adhesion when two nonpolar phases are combined.

Table 5. Rupture strength (*P*) of the monolayer glass fiber plastics based in polyethylene and polyamide matrices[11]

Object	*P*/MPa	
	Polyethylene	Polyamide
Initial matrix	26±2	56±4
Microplastic without sublayer	60±8	123±6
PECO (**2**)	124±14	109±10
BECO (**3**)	113±10	156±14

Table 6. Work of adhesion (W_a) of polymers **1**—**3** to the phases of various polarity*

Polymer	W_{a_1}	W_{a_2}	W_{a_3}
	/mJ m^{-2}		
PCO (**1**)	90.9	51.3	47.0
PECO (**2**)	107.8	51.9	48.5
BECO (**3**)	112.4	40.5	57.2

* $W_{a_1} = \gamma_S + \gamma_W - \gamma_{S(W)W}$; $W_{a_2} = \gamma_{SO} + \gamma_W - \gamma_{S(O)W}$; $W_{a_3} = \gamma_S + \gamma_O - \gamma_{SO}$.

Thus, the energy characteristics of the poly(olefin ketone) surfaces at different interfaces show that these polymers can be promising for the construction of hemocompatible materials and as adhesives in the development of composite materials. The contact angle method has doubtless advantages for the prediction of possibilities of the practical use of polymers.

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