Surface free energy of composite materials with high calcium carbonate filler content

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DOI: dx.doi.org/10.14314/polimery.2017.434

Abstract: The adhesive properties of ternary composites based on middle density polyethylene (PE-MD) and isotactic polypropylene (*i*-PP) with high content (48–64 wt %) of inorganic fillers were studied. As fillers two kinds of calcium carbonate were used. Two fillers differed in their fineness degree and one of them had also stearic acid modified surface. It was stated that the kind and amount of filler affect mean value and scatter of surface free energy (γ_S) as well as its polar and dispersive components. More stable adhesive properties were observed for composites with stearic acid modified filler.

Keywords: polymer composites, polyethylene, polypropylene, calcium carbonate, surface free energy, adhesive properties.

Energia powierzchniowa kompozytów o wysokim stopniu napełnienia węglanem wapnia

Streszczenie: Zbadano właściwości adhezyjne trójskładnikowego kompozytu na bazie polietylenu średniej gęstości (PE-MD) i izotaktycznego polipropylenu (*i*-PP) z wysoką zawartością (48–64 % mas.) nieorganicznych napełniaczy. Jako napełniaczy użyto dwu rodzajów węglanu wapnia, które różniły się stopniem rozdrobnienia, a ponadto ten o drobniejszych cząstkach miał powierzchnię modyfikowaną kwasem stearynowym. Stwierdzono, że rodzaj i ilość napełniacza wpływają na rozrzut i wartość średnią swobodnej energii powierzchniowej (γ_S) oraz jej komponentów: polarnego i dyspersyjnego. Bardziej stabilne właściwości adhezyjne zaobserwowano w grupie kompozytów z napełniaczem modyfikowanym kwasem stearynowym.

Słowa kluczowe: kompozyty polimerowe, polietylen, polipropylen, węglan wapnia, energia powierzchniowa, właściwości adhezyjne.

The examination of adhesive properties of material surfaces is critical for determining their suitability or behavior of materials during processes in which adhesion occurs.

Adhesive properties are among the vital parameters that determine the suitability of surface layer preparation for bonding or making adhesive joints (*e.g.*, in adhesive bonding, sealing, painting, coating) [1–2]. Good adhesive properties (good adhesion) have a positive effect on the mentioned adhesion processes. These properties can be described using a variety of physical quantities. One of them is surface free energy (*SFE*) [1, 3–8].

An analysis of the available literature demonstrates that one of the most widely applied methods to determine *SFE* is the Owens-Wendt method (also known as the Kaelble-Owens-Wendt method) [1, 6, 7]. This method is widely employed to determine *SFE* of polymers [3, 6, 7]. It consists in determining dispersive and polar components of *SFE* based on Bertholt's hypothesis, which says that interactions between particles present in the surface layer of two different bodies are equal to the geometric mean of interactions between the particles of these bodies. Polymer surfaces are often difficult to wet and bond, due to the low surface energy, incompatibility, chemical inertness, or the presence of contaminants and weak boundary layers [8]. The knowledge of polymer surface properties is very important to prepare the adhesive joining or other technology. The knowledge of the adhesive properties helps to work out the method of surface treatment. They influence all the operations of joining, printing, or coating processes.

Scientific research on new materials and their applications is more and more often focused on composite materials with polymer matrix and various organic and inorganic fillers of fibrous or powdered structure and diverse size [9–15]. The main aim of producing polymer composite materials is to enhance the properties of a polymer matrix applied, including rheological, mechanical, thermal, and electric properties [16–22]. Calcium carbonate

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($CaCO_3$) is a popular and readily available inorganic filler [19, 23–26] which exhibits good dispersion in polymer matrix [23]. Owing to its very good properties (chemically passive, nontoxic, highly pure, odorless, and tasteless [27–29]) calcium carbonate is also used as cost-effective means of increasing both thermal strength of polymers and notched impact strength, as well as improving product rigidity and its surface quality [30–32].

The studies published so far mainly focus on investigating the effect of modified and non-modified $CaCO_3$ on mechanical properties of polyolefins [27–29, 33–35] with low filler content, *i.e.*, maximum 50 wt %. The results demonstrated that the mechanical properties of composite products change significantly. It was also observed that introducing calcium carbonate to polymer matrix leads to an increase in resistances of flow of the plasticized composite material, in other words – to increased viscosity of this composite material.

The majority of research conducted on polymer composites filled with $CaCO_3$ concerns binary composite systems: polymer-filler (modified or non-modified $CaCO_3$) [13, 25, 27–29, 36, 37]. However, ternary composite materials (polymer-polymer-filler, with high $CaCO_3$ content) are also worth examining due to their applications. The matrix of this composite material is made up of a mixture of polyolefins (polyethylene and polypropylene), with an adequate weight ratio of the polymers used. The content of calcium carbonate filler can be up to 65 wt % of the composite material. If the filler is adequately modified, such composites can be processed using conventional techniques, including injection molding and extrusion molding [38, 39].

The aim of this study was to determine adhesive properties of ternary polyolefin composites with a high $CaCO_3$ content.

EXPERIMENTAL PART

Materials

The following materials were applied in our investigations:

– middle density polyethylene (PE-MD, trade name HF 513), produced by Total Petrochemicals, used as matrix in the investigated composites;

– isotactic polypropylene (*i*-PP, trade name Malen HP 456J), produced by Basell Orlen Polyolefins Sp. z o.o.;

– calcium carbonate high chemical purity and ultrafine particles (1 μm), with stearic acid modified surface ($CaCO_3$, trade name Omyalite 95 T), produced by Omya;

– calcium carbonate with particle size 2.5 μm, chemical purity ($CaCO_3$, trade name Omyacarb 2-VA), produced by Omya.

Sample preparation

The homogenization of composite materials and reference mixtures was performed by the rolling process using a Buzulk rolling mill. The operating temperature of the work rolls was maintained at 230 ± 5 °C. Obtained sheets were first subjected to shearing and then to grinding using a Shini SG-1417 low-speed granulator. The granulate prepared thereby was processed by the injection molding method. Test samples in form of dumbbells (type 1A) compliant with EN ISO 527-2:2012 standard were manufactured by injection molding using a Battenfeld PLUS-35 hydraulic injection molding machine with a screw diameter D = 25 mm and screw L/D ratio of 15.

The examined materials involved ternary composite systems (UM and M series) and binary polymer reference mixtures (R series). Compositions of individual constituents making up the investigated materials are listed in Table 1.

Table 1. Compositions and denotations of produced mixtures and polymer composites

Denotation	Investigated composites, wt %			
	PE-MD	*i*-PP	Omyacarb 2-VA	Omyalite 95 T
R1	77	23	–	–
R2	68	32	–	–
R3	56	44	–	–
UM1	40	12	48	–
UM2	30	14	56	–
UM3	20	16	64	–
M1	40	12	–	48
M2	30	14	–	56
M3	20	16	–	64

Methods of testing

Surface free energy

Values of surface free energy of solid materials were determined using the Owens-Wendt method. This method assumes that surface free energy of a solid (γ_S) is the sum of: polar (γ_S^p) and dispersive (γ_S^d) components [7]:

$$\gamma_S = \gamma_S^d + \gamma_S^p \tag{1}$$

The polar component can be defined as the sum of components from interparticle interactions: polar, hydrogen bond type, inductive and acid-base ones, excluding dispersive interactions. Dispersive interactions can be defined as a dispersive component of surface free energy.

In order to determine the component of surface free energy, it is necessary to measure the wetting angle of surfaces of the examined materials using: polar and nonpolar liquids. Distilled water is most often used as the polar liquid, while diiodomethane is used as the nonpolar liquid [40, 41].

Values of the surface free energy γ_S and its components determined with the measuring liquids are listed in Table 2 [40].

T a b l e 2. Values of the surface free energy (γ_S) and its components determined with the measuring liquids applied (Owens-Wendt method)

Surface free energy and its components	Measuring liquids	
	Distilled water	Diiodomethane
Surface free energy mJ/m^2	72.8	50.8
Dispersive component *SFE*, mJ/m^2	21.8	48.5
Polar component *SFE* mJ/m^2	51.0	2.3

To determine values of γ_S, the wetting angle of the examined materials was measured using the measuring liquids given in Table 2. Next, the polar and dispersive components of γ_S as well as the total value of γ_S were determined using relevant dependences given in the studies [40–43]. The value of γ_S of the examined composites was determined based on eq. (1). The components γ_S^d and γ_S^p of the examined materials may be calculated from equations [42]:

$$(\gamma_S^d)^{0.5} = \frac{\gamma_d(\cos\Theta_d + 1) - \sqrt{\dfrac{\gamma_d^p}{\gamma_w^p}}\,\gamma_w(\cos\Theta_w + 1)}{2\left(\sqrt{\gamma_d^d} - \sqrt{\gamma_d^p\dfrac{\gamma_w^d}{\gamma_w^p}}\right)} \tag{2}$$

$$(\gamma_S^p)^{0.5} = \frac{\gamma_w(\cos\Theta_w + 1) - 2\sqrt{\gamma_S^d\gamma_w^d}}{2\sqrt{\gamma_w^p}} \tag{3}$$

where: γ_d – surface free energy of diiodomethane, γ_d^d – dispersive component of diiodomethane surface free energy, γ_d^p – polar component of diiodomethane surface free energy, γ_w – surface free energy of water, γ_w^d – dispersive component of water surface free energy, γ_w^p – polar component of water surface free energy, Θ_d – contact angle of diiodomethane, Θ_w – contact angle of water.

The measurements of the wetting angle were performed at a temperature of 22 ± 2 °C and air humidity of 28 ± 2 %. The volume of drops of the measuring liquids ranged from 0.6 to 1.4 mm^3. The wetting angle was measured immediately following the application of a drop of the measuring liquid on the sample surface (after a few seconds). The measurements were made using a PGX goniometer manufactured by Fibro System (Sweden) and a program for computer image analysis. The analysis was performed on 5 samples for each composite material type; each test sample was subjected to 25–30 measurements (4–6 drops of each measuring liquid). γ_S was determined by arithmetic means of the wetting angle measured using distilled water and diiodomethane.

Based on the hypothesis and the appropriate statistical tests in literature [44] the statistical analysis of γ_S value was performed. The mean values of γ_S were calculated after rejecting the extreme value. For comparison, the average values of the characteristic in two populations of general significance test were used, which is taken into account an error of the first kind (significance level is $\alpha = 0.5$). The essence of these tests is based on the rejection of the hypothesis, if it is true. Value of γ_S was determined by arithmetic means of the wetting angle measured using distilled water and diiodomethane. The selection of an adequate analysis method depends on the truth of preliminary assumptions. The main criterion for selection of a suitable statistical model rests upon the assumption of equality of two population variances. The truth of this assumption was tested using Fisher's test. The Student's t-test is used when there is no basis for rejecting the hypothesis about equality of variances on the assumption of equality of standard deviations. In contrast, Cochran-Cox test is applied when the hypothesis about equality of variances has to be rejected.

Scanning electron microscopy

Scanning electron microscopy (SEM) observations were performed with a Zeiss EVO40 instrument (Germany) with a contrast of secondary electrons (SE) at the acceleration voltage 17 kV, to observe the possible agglomerations and dispersion level of the powder filler. The scanning electron microscopy observations were performed on the cryogenic fracture surface.

RESULTS AND DISCUSSION

The obtained values of γ_S and its components for PE-MD/*i*-PP reference composites (series R) are presented in Fig. 1. Analyzing the results of the adhesive properties of the reference composites (series R), it was observed that scatter of γ_S values measured for the same samples was quite high. For R1, the difference between the lowest and the highest obtained value was 15 %, for R2 – 24 %, for R3 – 34 % and this may prove that the surface layer has highly variable properties. Since the mean γ_S values obtained for R1, R2 and R3 mixtures are similar. It can be concluded that the content of *i*-PP does not affect the γ_S, whereas noticeable increase of γ_S^p with increase of *i*-PP content was found.

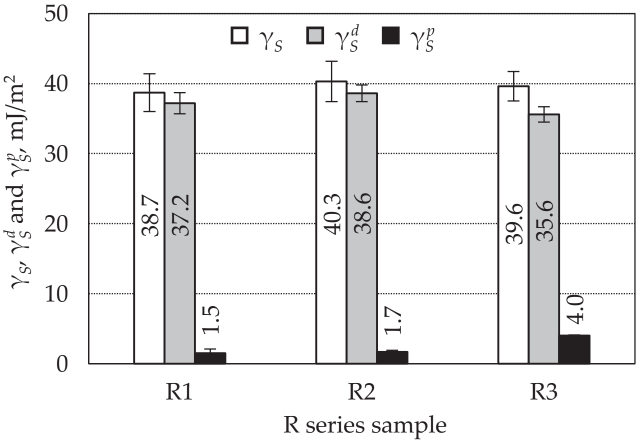

Fig. 1. Surface free energy and its components for surfaces of the R series of composites

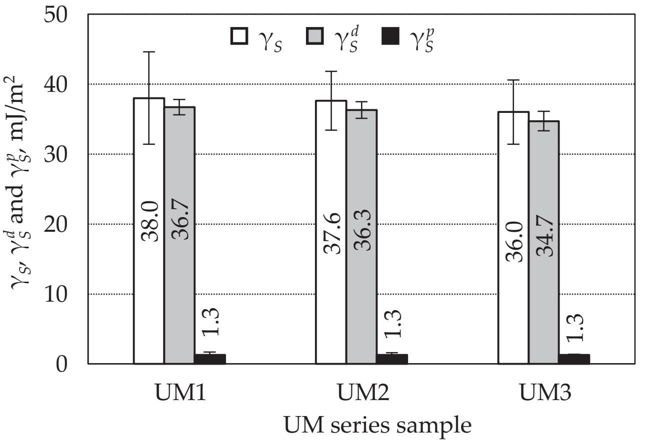

Fig. 2. Surface free energy and its components for surfaces of the UM series of composites

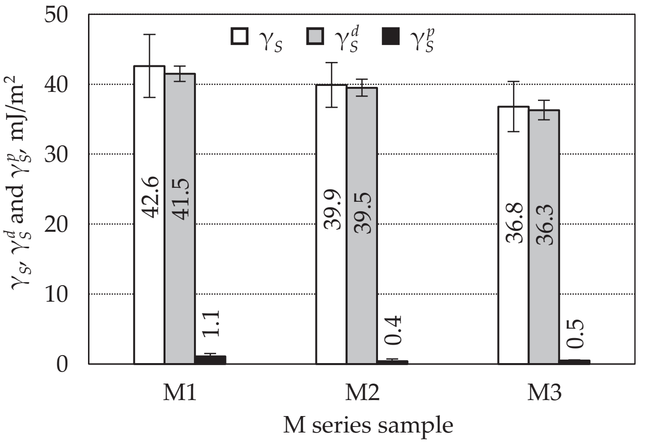

Fig. 3. Surface free energy and its components for surfaces of the M series of composites

The results of γ_S and its components for the ternary composite materials (series UM and M) are given in Figs. 2 and 3.

For UM series of composites (Fig. 2) no significant differences in the obtained γ_S values were observed. Value of γ_S for UM2 and UM3 samples decreases only by 1 and 5 %, respectively, compared to the UM1 composite with the lowest content of Omyacarb 2-VA. In addition, γ_S^p component had the same value for all composite types. A low scatter of results was observed for the wetting angle measured using both distilled water and diiodomethane. Hence, γ_S results do not differ much, either. The scatter of γ_S results for consecutive samples of this series was as follows: for UM1 6.6 mJ/m^2 (constituting 16 % of the maximal γ_S value), for UM2 4.2 mJ/m^2 (11 %) and for UM3 4.6 mJ/m^2 (12 %). It can be concluded that Omyacarb 2-VA as the inorganic filler has no effect on adhesive properties of the examined group of composite materials.

Examining series M of composites the effect of Omyalite 95 T content was investigated (Fig. 3). Values of γ_S and its components decrease with an increase of the filler content in the composite material. In comparison to M1 sample (48 wt % of filler) values of γ_S for M2 sample (56 wt % of filler) were lower by approx. 6 %, while for the M3 composites (64 wt % of filler) by approx. 13 %. A visible effect of the applied amount of Omyalite 95 T was also observed with regard to rheological parameters of the composite materials and their processability, which is described in detail in the studies [1, 39].

The analysis of the wetting angle results for the examined composite materials was performed. For some composites, low scatter of results was observed, while for others considerable differences in obtained values of the wetting angle are noted. The considerable scatter of results is observed only in some cases for distilled water. It was observed that lower scatters of results were obtained for the composites with Omyacarb 2-VA filler characterized by lower fineness degree (UM series).

Based on the comparison of the results for the composites with examined filler content (48, 56 or 64 wt %) it can be observed that higher values of γ_S and γ_S^d were obtained when Omyalite 95 T filler was used. It was observed that the application of this filler with a higher fineness degree (M series) leads in most cases to a higher value of γ_S, *i.e.*, higher adhesive properties. It should however be stressed here that Omyalite 95 T is modified chalk.

Comparing the results obtained (Figs. 2, 3), it was observed that fillers with the most favorable effect with regard to γ_S value obtained in most cases are Omyalite 95 T stearic acid modified $CaCO_3$. It can be presumed that the composite materials with these fillers will exhibit better adhesive properties compared to composite materials

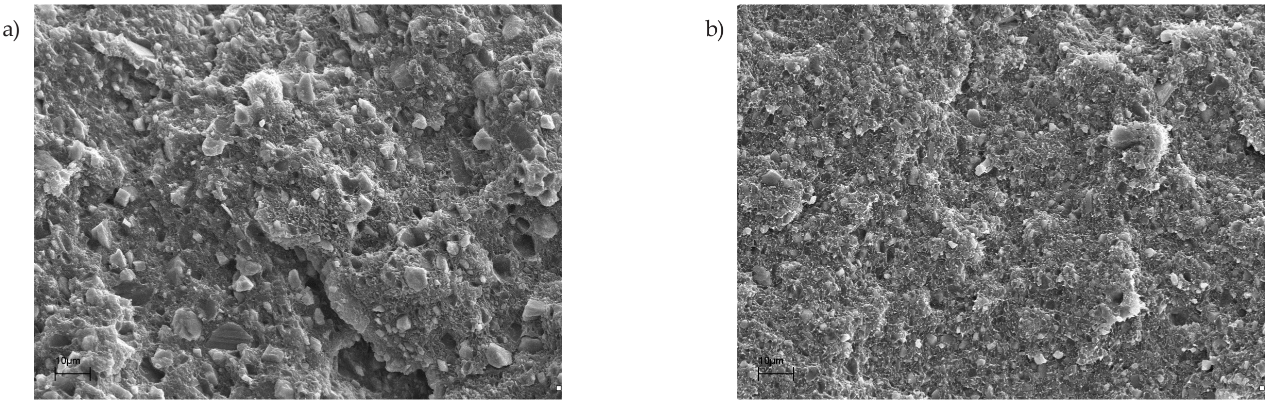

Fig. 4. SEM micrographs of samples: a) UM1, b) M1

Table 3. Statistical analysis results of γ_S values for R series of composites

Type of composite	Value of statistic F_{obl}	Value of statistic F_{α}	Result	Value of statistic T_{obl}	Value of statistic F_{α}	Conclusion
R1-R2	1.780	2.039	$\sigma_1^2 = \sigma_2^2$	3.427	2.009	$m_1 \neq m_2$
R2-R3	1.224	2.039	$\sigma_2^2 = \sigma_3^2$	1.753	2.009	$m_2 = m_3$
R1-R3	1.951	2.039	$\sigma_1^2 = \sigma_3^2$	4.201	2.009	$m_1 \neq m_3$

Table 4. Statistical analysis results of γ_S values for UM series of composites

Type of composite	Value of statistic F_{obl}	Value of statistic F_{α}	Result	Value of statistic T_{obl}	Value of statistic F_{α}	Conclusion
UM1-UM2	1.342	2.039	$\sigma_1^2 = \sigma_2^2$	1.955	2.009	$m_1 = m_2$
UM2-UM3	0.975	2.039	$\sigma_2^2 = \sigma_3^2$	2.107	2.009	$m_2 \neq m_3$
UM1-UM3	1.287	2.039	$\sigma_1^2 = \sigma_3^2$	2.488	2.009	$m_1 \neq m_3$

Table 5. Statistical analysis results of γ_S values for M series of composites

Type of composite	Value of statistic F_{obl}	Value of statistic F_{α}	Result	Value of statistic T_{obl}	Value of statistic F_{α}	Conclusion
M1-M2	1.107	2.039	$\sigma_1^2 = \sigma_2^2$	3.993	2.009	$m_1 \neq m_2$
M2-M3	0.672	2.039	$\sigma_2^2 = \sigma_3^2$	2.987	2.009	$m_2 \neq m_3$
M1-M3	0.871	2.039	$\sigma_1^2 = \sigma_3^2$	4.375	2.009	$m_1 \neq m_3$

with non-modified $CaCO_3$. The differences in γ_S values of the composites with modified and in those with non-modified $CaCO_3$ can be caused by a more homogeneous distribution of the modified filler in the polymer matrix. According to the data given in the literature, stearic acid modification of $CaCO_3$ surface leads to an increase in its dispersion in the nonpolar component (polymer matrix) as well as it prevents agglomeration of its particles [43]. This is confirmed by the SEM micrographs presented in Fig. 4, showing fractures of the examined composite UM1 and M1.

As shown in Fig. 4b, a more homogeneous distribution of the modified $CaCO_3$ filler was achieved. Better homogenization of modified $CaCO_3$ can directly affect surface properties of composites being manufactured. To confirm this assumption, it is however necessary that additional research on surface properties of the composites be conducted, including atomic force microscopy (AFM) method and friction factor determination.

Due to slight differences in γ_S values of the tested polymers, the statistical analysis of results was performed and the results of this analysis are shown in Tables 3–5 (where: σ – variance, m – mean).

The statistical analysis results of γ_S for R, UM and M series of composites demonstrate that the hypothesis about equality of variances of two populations is met in all cases. On the basis of the results of statistical analysis it can be concluded that γ_S value only for variants: R2-R3 and UM1-UM2 shows no statistically significant differences on an assumed significance level of $\alpha = 0.05$ (the hypothesis about equality of means was found to be true for two cases). In contrast, the surface free energy value for others variants is statistically different from the other.

CONCLUSIONS

Based on the results obtained, it is found that a type of composite material affects the value of γ_S and its components. It was also observed that the type of filler applied is one of the factors affecting the scatter of results for γ_S values, which leads to a certain variation in adhesive properties. In all investigated cases, γ_S^p had a slight share, ranging between 0.5 and 4 %. By comparing the results obtained for the UM series and M series of composites, it was observed that fillers with higher degrees of fineness (M series) gave greater variability in results. However, the filler in the M series of composites has been stearic acid modified and it is more likely that this is causing the difference in variability. If the UM series of composites, containing unmodified filler, is compared low variability in measurements can be seen.

This observation seems quite significant and can be used when subjecting the examined composite materials to adhesion-based processes. This will help predict some consequences of such adhesive properties of the surfaces subjected to processes such as painting, coating, or adhesive bonding. The results demonstrate a positive effect of applying stearic acid modification to the fillers (Omyalite 95 T) on γ_S. This can result from a better distribution of the modified filler in the polymer matrix compared to non-modified filler.

It was observed that lower scatters of results were obtained for the composite materials with the lowest fineness degree filler (Omyacarb 2-VA), *i.e.*, for the UM series composites. Therefore, it can be concluded that the application of the filler with a lower fineness degree will allow obtaining a surface with more stable adhesive properties.

This work was supported by the Ministry of Science and Higher Education (Poland) – Grant N N209 106837.

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Received 6 VII 2016.

Instytut Inżynierii Materiałów Polimerowych i Barwników,
Oddział Elastomerów i Technologii Gumy w Piastowie
oraz
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zapraszają do udziału

w **17. Międzynarodowej Konferencji**

ELASTOMERY'2017 „Z gumą przez życie"

Warszawa, 21–23 listopada 2017 r.

Przewodniczący Komitetu Naukowego: prof. dr hab. inż. Dariusz BIELIŃSKI
Przewodniczący Komitetu Organizacyjnego: mgr Urszula PAWŁOWSKA

Tematyka konferencji:
Nowe elastomery i składniki mieszanek
Fizykochemia i modyfikacja elastomerów
Wzmacnianie i sieciowanie elastomerów
Elastomery termoplastyczne i specjalne
Procesy technologiczne i urządzenia do przetwórstwa
Metody badań i kontroli – materiały, procesy i wyroby
Symulacje procesów, zjawisk i zachowań elastomerów i gumy
Guma, kompozyty i nanokompozyty elastomerowe – synteza i aplikacje
Starzenie, eksploatacja i czas życia wyrobów gumowych
Recykling odpadów gumowych i ochrona środowiska

Program konferencji obejmuje: referaty plenarne, referaty, komunikaty oraz sesje plakatową.

Język obrad: polski, angielski (z tłumaczeniem symultanicznym).

Terminy: 31 maja 2017 r. – przesłanie formularza zgłoszeniowego; 30 czerwca 2017 r. – streszczenie, artykuł do publikacji w Elastomerach; 31 sierpnia 2017 r. – opłata konferencyjna; 20 października 2017 r. – pełne teksty wystąpień.

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