Effect of low-temperature plasma treatment on rubber properties

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Abstract: The effect of the distance (0.5 and 5 mm) and exposure time (10 and 30 s) of the atmospheric plasma on the structure and surface properties (contact angle, adhesion) of unvulcanized rubber was examined. FT-IR and SEM were used to assess the structure. After plasma treatment, a higher intensity of the OH and COO- bands was observed, while the intensity of the bands from methylene groups decreased. SEM showed that plasma significantly reduces the size and number of agglomerates, resulting in a more uniform structure. The plasma distance has a significant effect on the surface properties of the rubber, with a better effect achieved at a smaller distance (0.5 mm). In this case, the rubber character changed from hydrophobic to hydrophilic, which is related to the introduction of oxygen species. A greater plasma distance (5 mm), regardless of the exposure time, resulted in a lower adhesion force. The exposure time had no significant effect on the tested rubber properties.

Keywords: atmospheric plasma, DCSBD, rubber blend, FT-IR, SEM, adhesion.

Wpływ niskotemperaturowej obróbki plazmowej na właściwości gumy

Streszczenie: Zbadano wpływ odległości (0,5 i 5 mm) oraz czasu ekspozycji (10 i 30 s) plazmy atmosferycznej na strukturę i właściwości powierzchniowe (kąt zwilżania, adhezja) niewulkanizowanej gumy. Do oceny struktury stosowano FT-IR i SEM. Po obróbce plazmą zaobserwowano większą intensywność pasm pochodzących od grup OH i COO-, natomiast intensywność pasm pochodzących od grup metylenowych zmniejszyła się. Metodą SEM wykazano, że plazma znacząco zmniejsza wielkość i liczbę aglomeratów, czego efektem jest bardziej jednorodna struktura. Odległość plazmy ma istotny wpływ na właściwości powierzchniowe gumy, przy czym lepszy efekt uzyskano przy mniejszej odległości (0,5 mm). W tym przypadku charakter gumy zmienił się z hydrofobowego na hydrofilowy, co jest związane z wprowadzeniem ugrupowań tlenowych. Większa odległość próbki od plazmy (5 mm), niezależnie od czasu ekspozycji, skutkowała mniejszą siłą adhezji. Czas ekspozycji nie miał istotnego wpływu na badane właściwości gumy.

Słowa kluczowe: plazma atmosferyczna, DCSBD, guma, FT-IR, SEM, adhezja.

Plasma source operating in diffuse coplanar surface barrier discharge (DCSBD) represents a suitable and environmentally friendly technique but remains an active way of materials treatment. Plasma based on the DCSBD discharge is one of the non-isothermal atmospheric pressure plasma [1] sources often used for treatment of different types of materials such as glass [2, 3], films [4], biomaterials [5–8] and fibers, but also finds an objective application in treatment of polymeric material surface [9], while enables the treatment at large surface areas, according to the electrode dimensions within high speed of process and still maintains producing homogenous in-line source of plasma [10–14]. In the last few years, the plasma working at the atmospheric pressure is more preferred than low pressure plasma, due to its easier industrialization and faster plasma-chemical processes [3, 15].

The principle of diffuse coplanar surface barrier discharge plasma is explained in many recent papers [16, 17]. Already, the discharge has been intensely investigated and characterized by several authors [18–22]. The plasma source produces active particles (radicals, ions, electrons,

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photons, atoms) as a form of energy generated in a submillimeter layer above the dielectric ceramic electrode [6, 23–25].

In compliance with the higher power of the plasma reactor, the DCSBD discharge becomes more diffuse and therefore more homogenous, still it is formed by several micro discharges [8, 23, 24, 26, 27]. In connection to the power of discharge itself, the highest advantage of DCSBD is its high-volume density of electric power [23, 24, 26, 27].

In accordance with the system of diffuse coplanar surface barrier discharge, it is drafted by multiple micro discharges moving along the flat dielectric forming a thin layer of plasma above the plate which refers to be macroscopically homogenous at full power [6, 19, 23, 28–30].

In terms of using plasma as a surface modification technique for polymer materials, it can be stated that the polymeric surface can be activated by specific functional groups linked to the polymeric surface to create novel operational superficies for industrial applications. In general, plasma activation takes place in the interaction of free charged electrons colliding neutral gas molecules while energy transfers with a solid surface leading into desired modifications of surface properties [31–34].

In addition, the important role in activation of polymeric surface by plasma takes place the process gas and the composition of a material´s surface. As described in literature, the reactive environment of oxygen plasma forms wide amount of components such as: oxygen anions O^-, cations O^+, ozone O_3, O^{2+} and O^{2-} capable of releasing energy which leads to the breaking of C-C and C-H covalent bonds [35, 36] and to form various functional groups, for example OH, CHO, COOH etc. to the plasma subjected surface [35, 37]. According to the literature, the use of oxygen plasma ensures the enhancement of materials adhesion [31–33]. On the other side, the usage of fluorinated plasma improves the chemical inertness [34] and noble gases affect surface initial crosslinking [38].

Hence, the objective of the current research is to study the effect of the distance (0.5 and 5 mm) and exposure time (10 and 30 s) of the atmospheric plasma on the structure and surface properties (contact angle, adhesion) of unvulcanized rubber. The structure was assessed using FTIR and SEM analyses.

EXPERIMENTAL PART

Materials

In this work, the rubber based on complex polymer matrix made of styrene butadiene rubber (SBR 1723, SBR 1500) and natural rubber (NR SMR 10) in loading 100 phr was treated. The rubber was filled (0–200 phr) with the carbon black (N339, MAKROchem, Lublin, Poland). As an activator (0–40 phr) was used the zinc oxide (ZnO, SlovZinc, a.s., Košeca, Slovakia) with density of 5.5–5.7 g/cm^3 and stearic acid (Aldrich Chemical Company Inc. Burlington, MA, USA) with density of 941 kg/m^3), sulfur 0.25–40 phr (Istrochem a.s., Bratislava, Slovakia) was added as a curing agent and other ingredients 0–10 phr (accelerators, inhibitors) were blended. The rubber was examined in the shape of foil with the thickness of 1.2 mm. The conditions of plasma treatment used in this work with rubber labelling are listed in Table 1.

Table 1. Labelling of rubber samples according to applied plasma conditions

Rubber	Exposure time, s	Plasma distance, mm	Power, W
T0 (as received)	–	–	–
T101	10	5.0	375
T301	30	5.0	375
T102	10	0.5	375
T302	30	0.5	375

Methods

Plasma treatment

Rubber samples were treated by laboratory device for in-line plasma treatment based on diffuse coplanar surface barrier discharge – plasma reactor KPR 200 mm (Research Institute for Man-Made Fibers, Svit, Slovak Republic) working in ambient air at atmosphere with plasma bulk density of 80 W/cm^3 and operating at power of 375 W. The macroscopically homogeneous plasma is generated at alumina (96%) dielectric electrode. The samples were exposed to DCSBD plasma using an exposure time of 10 s and 30 s and a distance from the plasma of 0.5 mm and 5 mm, respectively.

Fourier transform infrared spectrometry

The infrared spectra of plasma treated samples were obtained using a FT-IR spectrometer Nicolet iS50 Thermo Scientific (Waltham, MA, USA) via ATR technique with the diamond crystal to study chemical structure with identification of chemical bonds. The spectra were measured in the range of 4000–600 cm^{-1} with resolution of 4 cm^{-1}.

Scanning electron microscopy

Scanning electron microscope Tescan VEGA 3 (Brno, Czech Republic) in the secondary electron mode was used to analyze morphological features on treated rubber surface. Plasma treated samples with dimensions of 10 × 10 mm were placed in the beaker with an isopropyl alcohol inside of ultrasonic cleaner to reduce the fatness, humidity, and free particles on the surface of rubber samples. The samples were coated using SC7620 Mini Sputter Coater by Quorum Technologies (East Sussex, United

Kingdom) to achieve an optimal signal from the analyzed areas to ensure high-quality visualization of the samples surface. The electric voltage applied to the tungsten cathode was 20 kV to obtain electron emission.

Adhesion

The Mecmesin Multitest 1-d apparatus (West Sussex, United Kingdom) was applied to determine adhesion. Herein, the force needed to peel off two rubber surfaces (50 × 60 mm) was measured before and after plasma treatment.

Contact angle (CA)

The wetting properties before and after plasma treatment were determined by contact angle measurements using testing liquids: distilled water and diiodomethane via sessile drop method. The rubber test pieces were placed in the beam captured by a camera system Quick Photo 52 Industrial 2.2. The contact angles were evaluated using Matlab software.

RESULTS AND DISCUSSION

FT-IR analysis

ATR-FT-IR spectra of plasma treated and untreated rubbers are illustrated in Figs. 1–6.

As discussed earlier, the ATR-FTIR spectra of plasma treated rubbers should have changed a presence of functional groups with a content of oxygen species, responsible for the surface modifications [37, 39, 40]. For the infrared spectra of the untreated rubber (T0) and plasma treated rubber samples (T101, T301, T102, T302), different effects of plasma treatment can be observed:

- formatting of new chemical bonds (transitions),
- extinction of chemical bonds.

In the all-infrared spectra can be seen a broad absorbance peak of stretching vibration of OH [41] and NH [42] in the region of 3400–3100 cm^{-1}. The peak at the wavenumber of 3316 cm^{-1} can be assigned to stretching vibrations of υ(OH) and it can be seen an increase of relative intensity of peaks depending on plasma treatment position and time, for T301, T302 and T102. Typical peaks of the rubber polymer matrix were observed in the area between 3000 cm^{-1} to the 2840 cm^{-1} related to the symmetric and asymmetric stretching vibrations of CH, -CH$_2$ and -CH$_3$ [43–52]. Stretching vibrations of υ(CH) from the styrene butadiene rubber were observed near the wavenumber of 3000 cm^{-1}, overlapping stretching vibrations of υ(C=C-H) of natural rubber. The peaks between 2840 cm^{-1} and 2950 cm^{-1} corresponds to the natural rubber and styrene butadiene rubber symmetric and asymmetric stretching vibrations of υ(CH$_2$) and υ(CH$_3$). As can be seen in Fig. 1 and Fig. 4, the peaks relative intensity changed after the plasma treatment following from lowest to highest: T301<T101<T0 and T302<T102<T0. Due to these observations, we can suggest that during the plasma treatment occurs a reduction of methylene groups as in [39, 51] and/or migration (removal) of waxes originated in rubber composition [47, 49, 50]. At the 1732 cm^{-1} for T301, and 1726 cm^{-1} for T101 were identified stretching vibrations υ(C=O) [39, 47], a significant shift was observed for T302 (1706 cm^{-1}) and T102 (1707 cm^{-1}). The main changes were seen in the wavenumber area between 1600 cm^{-1} to 1300 cm^{-1}. Oxygen related groups (C=O, COO$^-$) with varying intensity were assigned [39, 49, 53] but in many cases were overlapped by stretching vibrations of aromatic ring. According to that, a significant change of relative intensity was seen around 1590 cm^{-1} which could be attributed to the stretching vibration of carboxylic anion υ_{as}(COO$^-$) [47, 48]. Herein, the relative intensity of plasma treated surfaces changed following from lowest to highest: T0<T101<T301 as can be seen in Fig. 2 and T0<T302<T102 in Fig. 5. Another significant change in relative intensity was seen at the wavenumber of 1513 cm^{-1} which could be assigned to the nitrogen related moieties (NO$_2$) [51]. The relative intensity changed following from lowest to highest: T0<T101<T301 as can be seen in Fig. 2 and T0<T302<T102 in Fig. 5. The same trend was seen in 1490 cm^{-1} which could be assigned to the stretching vibrations of benzene ring [41, 53, 54]. According to the [54] the increase of absorbance peak´s relative intensity could be caused due to the bombardment of electric charged particles (electrons, ions) that originated from ionized air during the process of plasma treatment in the area between 1600–1400 cm^{-1}. Thus, the breakage of C-H bonds occurred, and polymer radical formed here. Eventually, the forming C=C double bond occurred as can be seen in this study in Fig. 2 and 5. The stretching vibration υ_s(COO$^-$) can be seen in 1380 cm^{-1} [48]. Another large contrast in relative intensity was seen at the wavenumber in 1300 cm^{-1}. This peak can be related to the vibrations of oxygen functional groups – bending vibration of δ(OH) [55]. Herein, the relative intensity was changed following from lowest to highest: T0, T101<T301 (Fig. 3) and T0<T302<T102 (Fig. 6). An exposure time of 10 s and a plasma distance of 5 mm did not cause significant changes compared to the untreated rubber. However, comparing the distance of 0.5 mm and 5 mm, the smaller the distance, the greater the changes in the examined area visible after 10 s of exposure.

SEM analysis

Fig. 7a shows SEM image of T0 with a developed surface with unevenly distributed large agglomerates of irregular shapes. Plasma significantly reduces the number and size of agglomerates, which are most often hidden under the surface. The most homogeneous structure was obtained for T101 and T301 (Fig. 7b and 7c). The presence of dark spots in T301 can be explained by the removal of agglomerates because of sample fracture. T102 (Fig. 7d)

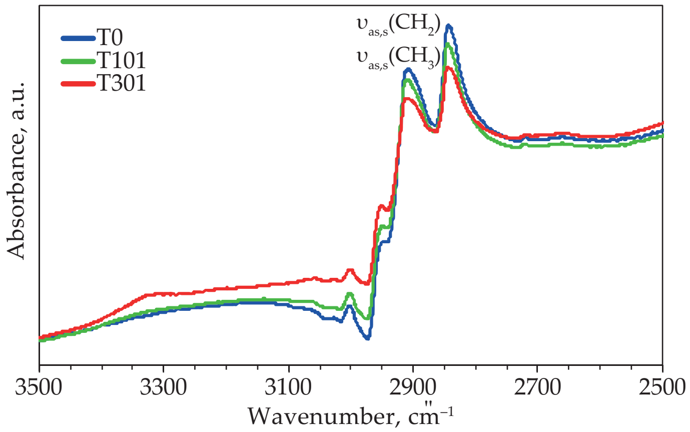

Fig. 1. IR spectra of T0 rubber and T101, T301 between 3500–2500 cm^{-1}

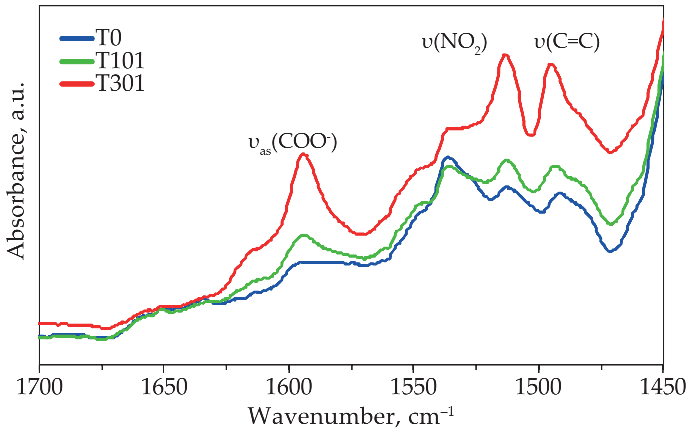

Fig. 2. IR spectra of T0 rubber and T101, T103 between 1700–1450 cm^{-1}

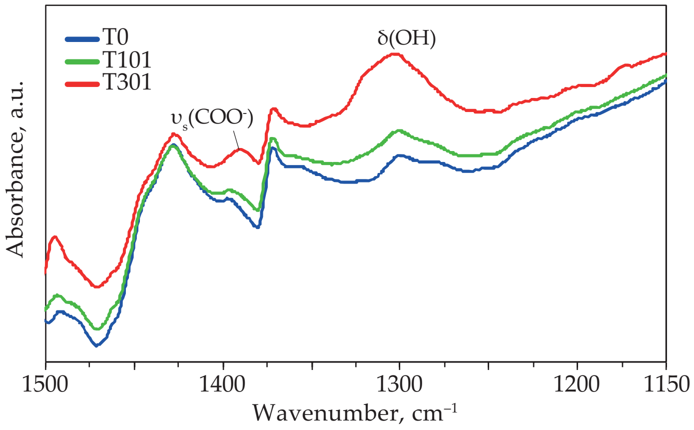

Fig. 3. IR spectra of T0 and T101, T103 between 1500–1150 cm^{-1}

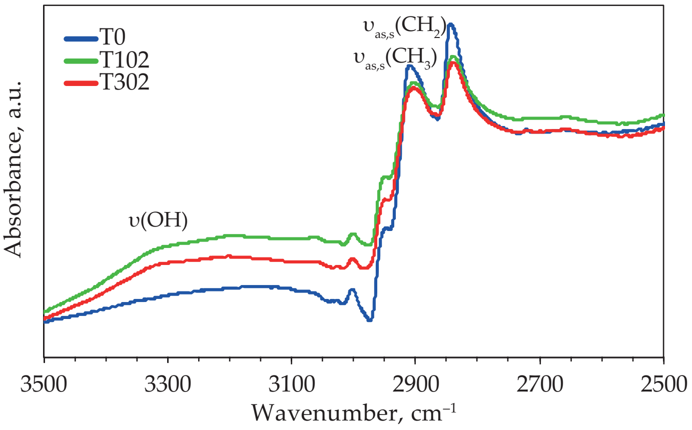

Fig. 4. IR spectra of T0 and T102, T302 between 3500–2500 cm^{-1}

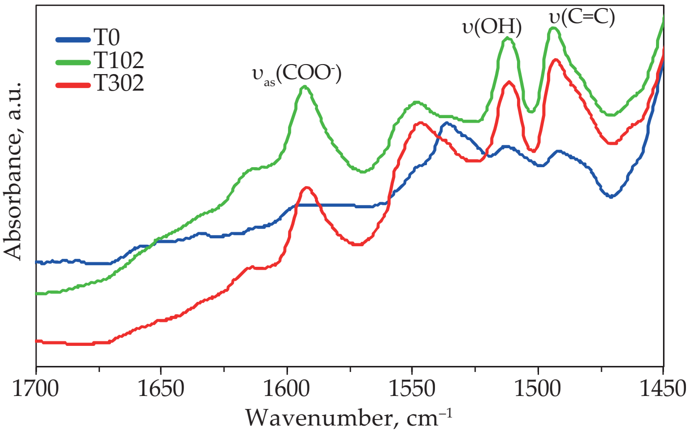

Fig. 5. IR spectra of T0 and T102, T302 between 1700–1450 cm^{-1}

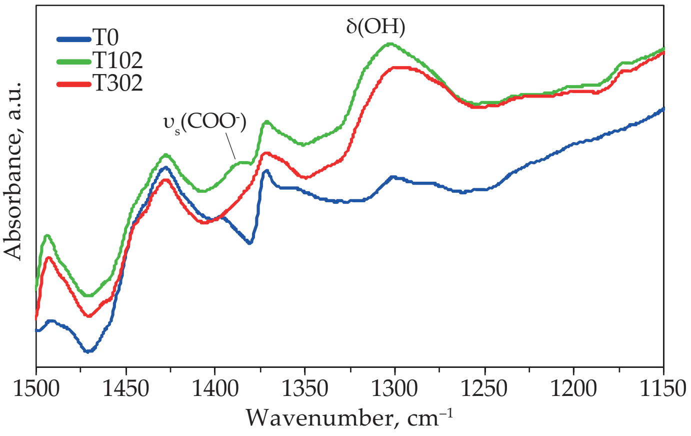

Fig. 6. IR spectra of T0 and T102, T302 between 1500–1150 cm^{-1}

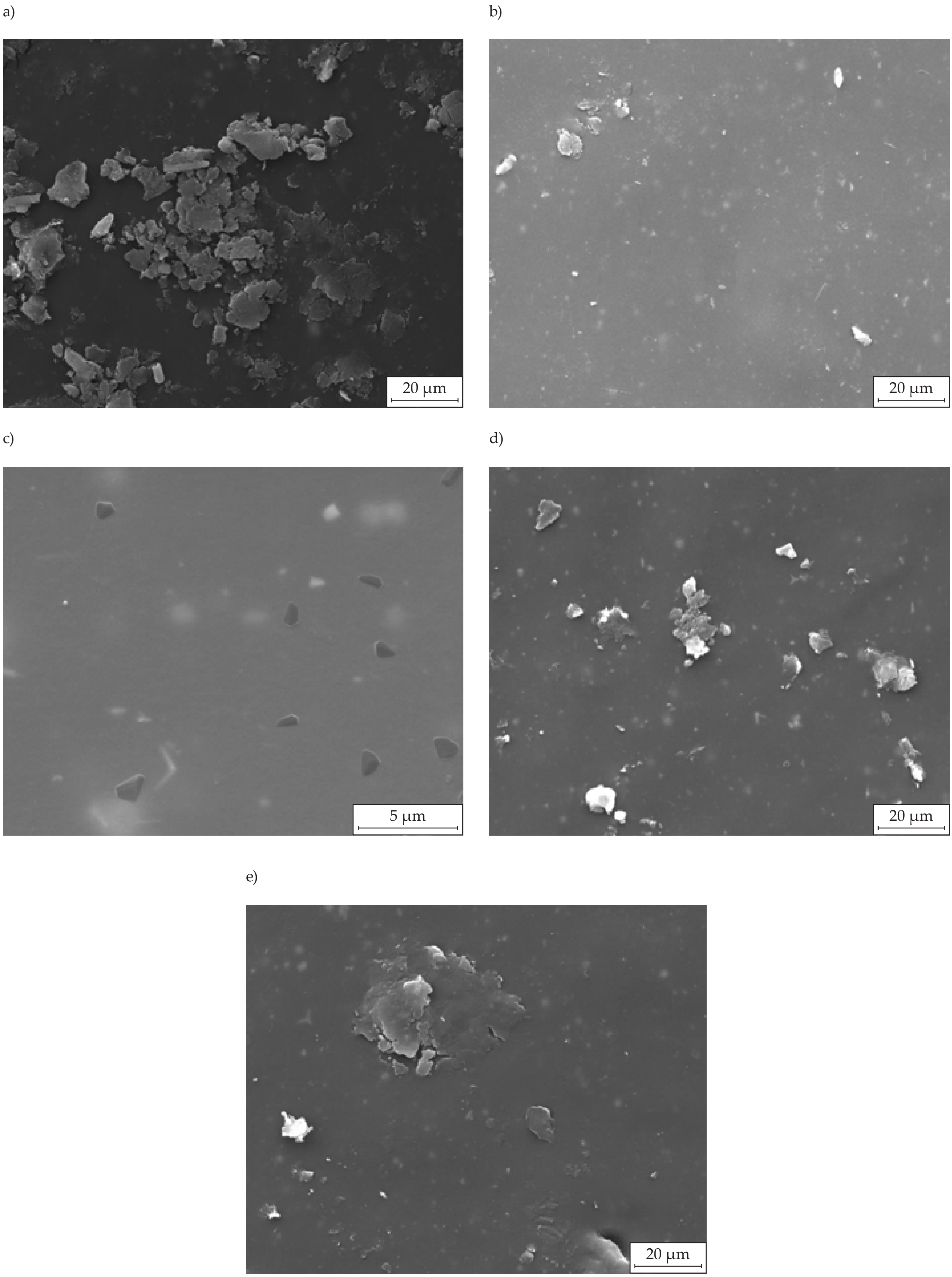
a)
b)
c)
d)
e)

Fig. 7. SEM images of a) T0, b) T101, c) T301, d) T102, e) T302

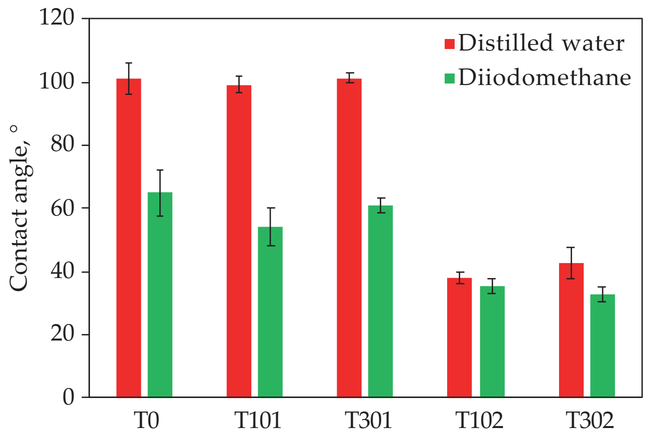

Fig. 8. Contact angle before and after plasma treatment

and T302 (Fig. 7e) show more clearly visible agglomerates compared to T101 and T301. In the case of T102, a larger number of smaller agglomerates were found than in T302. Moreover, in the case of T302, the presence of surface decohesion caused by 30 seconds of plasma treatment at a distance of 0.5 mm was also observed.

Contact angle measurement

The contact angle T0 indicates poor wettability of both tested liquids (Fig. 8). For distilled water, the contact angle was 101.01° and for diiodomethane 64.89°. After plasma treatment from 5.0 mm distance, the water contact angle of T301 (101.27°) and T101 (99.16°) was comparable to T0. However, the diiodomethane contact angle decreased after plasma treatment for T101 to 53.98° but was comparable to T301. The smaller the plasma distance, the more hydrophilic the rubber is [56, 57]. Comparing exposure times, both T102 and T302 had similar water contact angles of 37.89° for T102 and 42.56° for T302. The smallest contact angle was observed for T302 for diiodomethane (32.61°). As can be seen from Fig. 9, the smaller the plasma distance, the greater the effect of plasma treatment and, as a result, the greater the surface modification. However, the exposure time does not significantly affect the surface character of the rubber samples.

Determination of adhesion force

As can be seen from Fig. 10, samples treated from higher distance above the plasma electrode reflect significantly lower adhesion forces. In comparison, T0 achieved the adhesion force of 6.88 N, T101 of 3.85 N and T301 of 4.01 N. However, the rubber samples treated from 0.5 mm distance shows higher adhesion forces, e.g., T102 of 7.25 N and T302 of 7.06 N. The adhesion force increased as the distance of the plasma from the sample decreased [32], but the effect of the plasma treatment time was minimal.

CONCLUSIONS

In this research, the effect of the distance (0.5 and 5 mm) and exposure time (10 and 30 s) of the atmospheric plasma on the structure and surface properties (contact angle, adhesion) of unvulcanized rubber was demonstrated. Plasma significantly modified the structure and surface properties of the rubber by introducing hydroxyl and carboxyl groups. SEM showed that the plasma treated samples had a smoother surface compared to the untreated rubber.

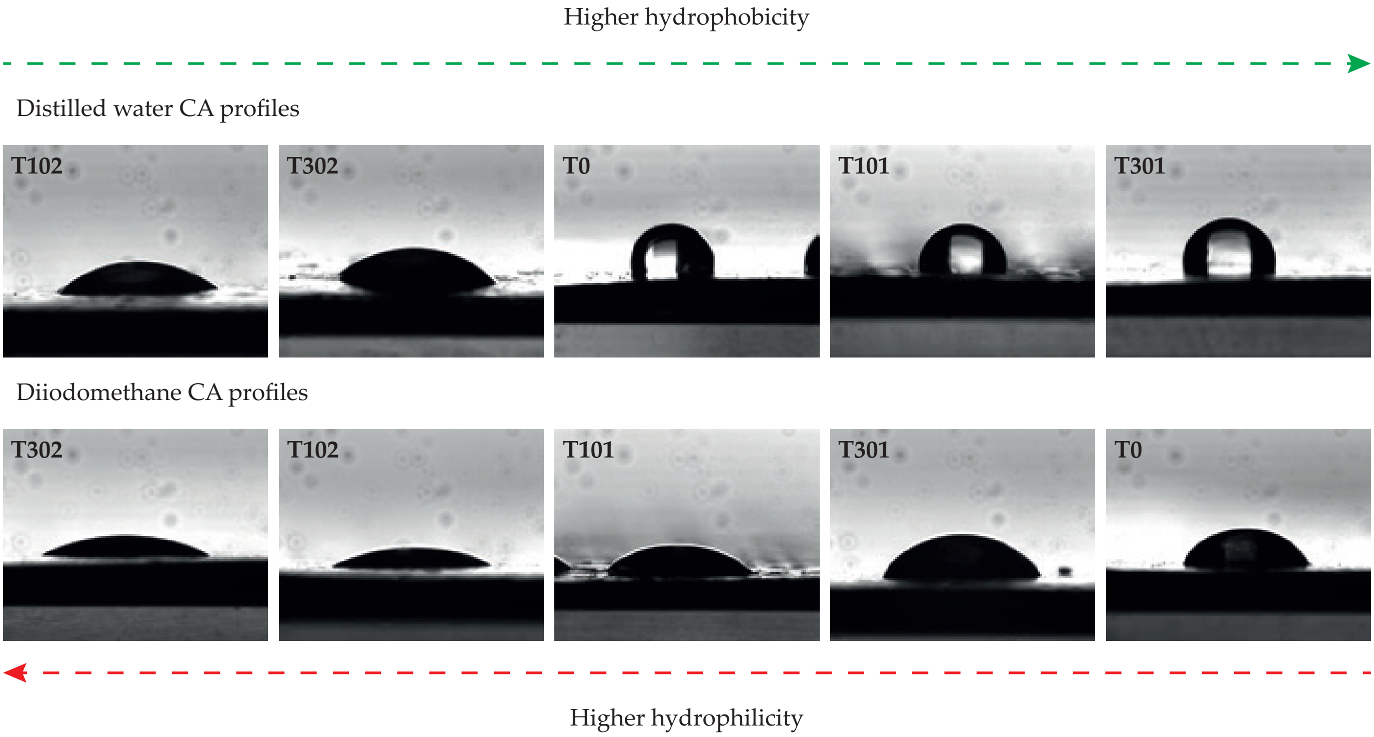

Fig. 9. Dependence of contact angle on plasma exposure time and sample positioning

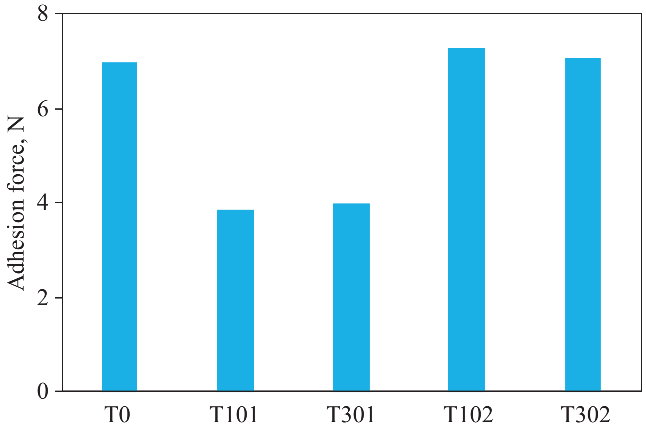

Fig. 10. Adhesion force of examined samples

Plasma treatment at 0.5 mm distance changed its character from hydrophobic to hydrophilic, which is related to the introduction of oxygen species. The 5 mm plasma distance at both exposure times resulted in a reduction in the adhesion force. Moreover, our research shows that the plasma distance has a significant effect on the obtained surface properties, with a better effect when using 0.5 mm.

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Authors contribution

S.D. – writing – original draft, writing review and editing, methodology, investigation, references; M.P – writing – original draft, writing review and editing, supervision, methodology, investigation, visualization; D.O. – validation, supervision, writing review and editing; R.J. – investigation, visualization, validation; Z.M. – methodology, investigation, references; A.D. – methodology, investigation, writing – original draft; J.P. – methodology, investigation, visualization; J.Š. – references, methodology, investigation, conceptualization; S.B. – references, methodology, investigation; J.V. – visualization, validation, investigation; T.W.K. – investigation, visualization, validation.

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Conflict of interest

The authors declare no conflict of interest.

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