

Investigating the Effect of Photo-Oxidative Degradation on the Ageing Resistance of the Car Mudflaps Manufactured with Post-Production High-Density Polyethylene Wastes

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ABSTRACT

The paper presents the results of the ageing resistance of automotive mudflaps made of recycled polyethylene blends. The mudflaps were manufactured by a plastic sheet extrusion-calendaring process in Novotech Kostrzyn nad Odrą company. The changes of the mechanical properties in the tensile test and impact toughness test as well as in Shore hardness due to the accelerated aging process were characterized, taking into account the aging sensitivity coefficient (KI), and changes in the structure within the wavenumber range 4000–400 cm⁻¹, taking into account the CI coefficient. The results showed a higher melting point and crystallinity for the recycled HDPE blends during photo-oxidative degradation. Moreover, the results presented decreased tensile strength and ductility, due to macromolecular chain scission caused by oxidation. It was found that the blends of HDPE based on recycled materials are more sensitive to the aging process than virgin HDPE. Finally, it can be concluded that the sensitivity to ageing blends increases with the increase of recycled HDPE content in the HDPE matrix.

Keywords: recycling, crystallinity, mechanical behavior, ageing process, carbonyl index, mudflap, polyethylene.

INTRODUCTION

The ageing process is determined by many factors that entail a whole set of changes in the strength and usage properties of plastic products [1–4]. These changes usually occur very slowly, with difficulty to catch and the factors at work are difficult to measure. The aging process has a slightly different course for each polymer because it depends both on the construction features related to the chemical structure of the macromolecule, and on the structural features of the polymers [3, 5–7]. All the more, the problem becomes more serious in the case of structural plastic products

such as car mudflaps made of recycled polymers exposed to environmental factors. These are exposed to strong effects of water, mud, sands, and snow, hence it is important to assess the behavior of this product during operation.

Mudflaps are made of a developed by authors within a project blend of virgin polyethylene (vPE) with different content of recycled polyethylene (rPE) modified with linear low-density polyethylene (LLDPE). HDPE is one of the most frequently used polymers in the production of films and sheets, due to its tensile strength ductility, and chemical resistance [8], but polyethylene presents some limitations because of severe

conditions of low temperature and high speeds of deformation, and also for recyclates it becomes brittle [9]. Nowadays, due to environmental and reduction costs the plastics recycling industry applies new strategies to improve properties by mixing processed plastics with virgin polymers together with stabilizers, or compatibilizers, and chain extenders [10–13]. Several papers have been carried out aiming at the toughening of HDPE by the modification with linear low-density polyethylene [5, 14].

Polyethylene is characterized by low resistance to oxygen, which oxidizes in air, and UV radiation accelerates this process. In the case of products, i.e. car mud flaps operated in difficult conditions, the influence of weather conditions (radiation, heat, rain, wind) may deteriorate the mechanical properties of these products [7, 15]. The set of these factors, referred to as atmospheric aging, may follow the photo-oxidation or thermo-oxidation mechanisms.

Polyethylene products undergo a photo-oxidation process consisting of polymer degradation under the influence of UV radiation in the presence of oxygen [16, 17]. As a result, the fracture of bonds in the main chain of macromolecules or the phenomenon of depolymerization is observed. Photo-oxidation is a process that takes place mainly on the surface of the irradiated polymer, as opposed to thermo-oxidation, which can take place throughout the entire volume of the product. As a result of photo-oxidation, there may be some changes in the chemical structure of the polymer or a decrease in the average molecular weight (chain scission reactions) or its increase (cross-linking), changes in the surface morphology (crazing, blister formation, loss of surface) [8, 16, 18]. Sometimes the chain scissions may lead to chemical crystallization, a type of secondary crystallization. Therefore, photo-oxidation may change both the average molecular weight and crystallinity of polymers, thus affecting their mechanical properties [19]. For example, Torikai et al. [20] studied the photodegradation of tree types of polyethylene and found that crosslinking improved the photostability of polyethylene. A measure of the photostability of polyethylene was the relationship between the number of oxidation products produced and the decrease of mechanical strength [20]. Rodrigez and al. [19] observed the effect of UV-aging on the fracture behavior of low-density polyethylene. They showed the strengthening of polyethylene over a narrow

range of UV radiation doses may be oxidation-induced [17, 19]. Jin H. et al. [21] stated that simulated recycling did not significantly change the crystallization and melting temperature of LDPE.

There are some published papers regarding the effect of environmental factors on the change of polyethylene properties, i.e. reduction of elongation and impact strength, change of colour or transparency, etc. [6, 9, 22]. Gnatowski and al. [6] showed that UV irradiation induces irreversible changes in polyethylene pipes leading to a deterioration of polymer mechanical properties, i.e. decreased storage modulus and loss of tangent values, reduced hardness, and tensile strength, and changed gloss and colour tone after UV aging.

Adaptation of the mud flap for proper water drainage during operation is exposed to hydrolytic degradation or the formation of free radicals in the presence of radiation. This simultaneous effect of solar radiation combined with the action of water increases the ageing efficiency of the polymer products, causing the surface layer of the product to change, which increases the susceptibility to cracking of the automotive mudflap. To assess the resistance of the HDPE mudflap to weather conditions, the ageing tests must be carried out in conditions corresponding to the natural conditions of use. The mudflap was aged under accelerated conditions corresponding to the exposure for 1 year in natural conditions. Accelerated ageing is carried out in a climatic cabinet using xenon lamps with a filter as the radiation source. The ageing test device provides constant control of temperature, radiation, and humidity in the chamber, and water agitation as a rain simulation [8].

The research aimed to evaluate the resistance to the ageing process of a car mudflap made from recycled polyethylene blends. The changes in the mechanical properties in the static tensile test and impact toughness test as well as in hardness were assessed, taking into account the ageing sensitivity K_p , and changes in the structure within the wavenumber range $4000\text{--}400\text{ cm}^{-1}$, taking into account the CI coefficient.

MATERIALS AND METHODOLOGY

The high-density polyethylene (HDPE) and its blends with different percentages of recycled polyethylene were tested. The HDPE a medium molecular weight grade for blow molding (trade name SABIC B5429) with a density of 0.954 g/cm^3 ,

Table 1. Sample codes and polymer blends composition subject to the ageing process

Sample code (virgin/recycled)	Composition of vHDPE/rHDPE/LLDPE blend [wt.%]
100vPE	100/0/0
v70/r30	70/29/1
v60/r40	60/39/1
v50/r50	50/49/1
v30/r70	30/69/1

and a mass flow index of 0.3 g/10 min (190 °C and 2.16 kg) was supplied by Sabcin Poland Sp. z o.o. (Warsaw, Poland). Recycled HDPE (grinding in-house) came from shredded post-production waste of the Novotech Kostrzyn nad Odrą company, which manufactures products for the automotive industry. To optimize the composition of the blend, recycled polyethylene was added in an amount of 30, 40, 50, and 70 wt.% to the virgin material modified by 1 wt.% linear low-density polyethylene as a masterbatch. Several series of HDPE/rHDPE blends were made with the composition presented in Table 1. Recycled PE sheets were extruded-calendered from each of the blends, from which standardized samples were then cut.

ACCELERATED AGEING

To measure resistance to weather conditions, accelerated weathering ageing was measured in a XANTEST 2200 model BGD 866/A chamber

(ANTICORR Gdańsk Sp. z o.o. Gdańsk, Poland) with a Xenon lamp and a wet phase according to ISO 4892 [23]. The samples were exposed in the chamber for 360 h divided into 1 cycle of 102 min each, at 60±3 °C under UV light followed by 108 minutes under UV light and distilled water sprinkling, under 0.35 Wm⁻² at 340 nm of average irradiance. This procedure was identified as Method 1. The samples were prepared into a dumbbell-shaped specimen, according to ISO. During the test, the irradiation, humidity, and temperature in the chamber were controlled. The criterion for ending the ageing process was the dose of the emitted radiation of 3.6 GJ.

METHODS

A Jasco, model FT/IR-4600, FTIR spectrometer, ATR mode, with 64 scans and 4 cm⁻¹ resolution, was used for obtaining molecular absorption spectra in the infrared region, ranging from 4000 cm⁻¹ to 400 cm⁻¹. This research allowed us to assess the structural changes in HDPE with recycled blends that occurred as a result of the accelerated ageing process. The carbonyl index (CI) was calculated from the ratio between the absorption at 1715 cm⁻¹ (stretching vibration of the ketone C=O bond) and 1465 cm⁻¹ (methylene groups) as reference [24]. With this technique, the carbonyl index, for the aged samples, was determined, based on the equation 1 [25, 26]:

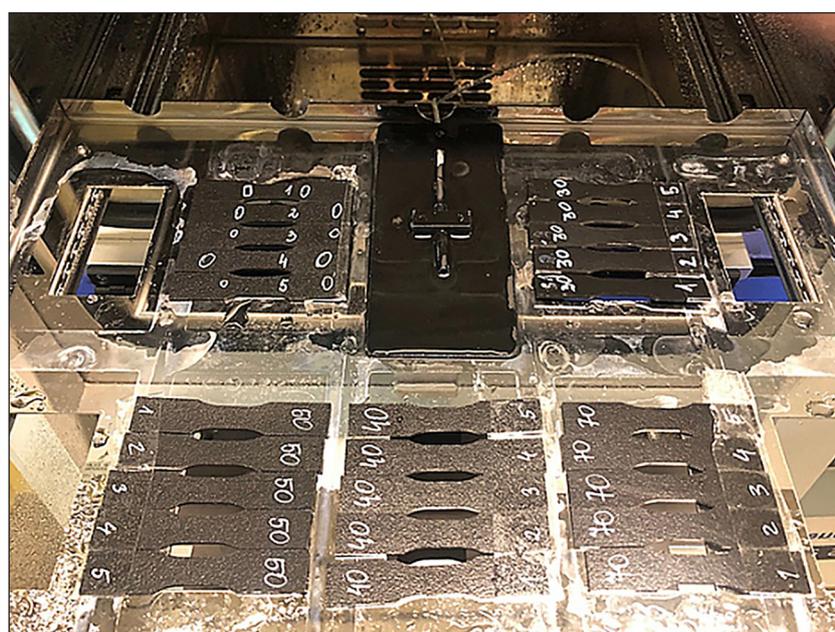


Fig. 1. The view of samples inside ageing chamber

$$CI (\%) = \frac{A_{\text{peak}}}{A_{\text{ref}}} \quad (1)$$

where: CI – a carbonyl index (%);

A_{peak} – an area of peaks attributed to carbonyl, groups at 1715 cm^{-1} , at 360 h of UV exposure;

A_{ref} – an area of reference peak for PE corresponds to the methylene group at 1465 cm^{-1} [24–26].

The melting and crystallization behavior of the virgin HDPE and recycled HDPE blends were studied using the DSC apparatus Netzsch, DSC 204 F1 Phoenix (NETZSCH GmbH, Selb, Germany) operating with a protective atmosphere (a nitrogen flow of 20 mL/min). Samples of about 8 mg were first heated at a rate of $10 \text{ }^\circ\text{C}/\text{min}$ from $-30 \text{ }^\circ\text{C}$ to $200 \text{ }^\circ\text{C}$ temperature and kept at this temperature for 10 min followed by cooling to $-30 \text{ }^\circ\text{C}$ at the rate of $10 \text{ }^\circ\text{C}/\text{min}$ to eliminate the thermal and mechanical history of the polymer. DSC studies were conducted to determine the melting temperature (T_m), crystallization temperature (T_c), melting enthalpy (ΔH_m), and degree of crystallization (X_c). The degree of crystallinity (X_c) of the high-density polyethylene (HDPE) was determined using the following equation 2:

$$X_c (\%) = \frac{\Delta H_m}{\Delta H_m^0} \cdot 100 \quad (2)$$

where: ΔH_m – the melting enthalpy of the sample in J/g;

$\Delta H_m^0 = 293 \text{ J/g}$ the melting enthalpy for a 100% crystalline polyethylene [10].

The mechanical properties of the virgin HDPE and HDPE/rHDPE blends were determined by tensile tests performed using the universal testing machine Zwick/Roell Z010 (ZwickRoell GmbH&Co. KG, Ulm, Germany) cooperating with the computer program

testXpert II. The samples were dog bond-shaped. according to the ISO 527-2:2012 standard [27]. The tensile tests were run at room temperature. The traverse speed was set to $1 \text{ mm}/\text{min}$ during the determination of the tensile modulus and $50 \text{ mm}/\text{min}$ during the remaining part of the tensile test. Tensile stress and elongation at break (EB) were evaluated from the tensile stress-strain curves. The reported data were the average of the results of 5 specimens. The Shore hardness (D scale) of samples was measured using a Shore hardness tester, according to the ISO 868:2005 standard [28].

The impact resistance of HDPE and HDPE/rHDPE blends was examined by pendulum impact testers (model Psd 50/15 HECERT, hammer 25J). The tensile impact strength test was carried out according to ISO 8256:2004 [29] at room temperature. Standardized samples with dimensions ($h \times b \times l$) were used: the thickness of $2.0 \pm 0.2 \text{ mm}$, the width of $10.0 \pm 0.2 \text{ mm}$, and the length of $80.0 \pm 2 \text{ mm}$. Five experiments were performed, and the average impact tensile strength resistance value (a_{cU}) was measured as follows in equation 3:

$$a_{cU} = \frac{E_c}{b \cdot h} \cdot 10^3 \left(\frac{\text{kJ}}{\text{m}^2} \right) \quad (3)$$

where: E_c – the absorb impact energy at sample break [J];

b – the width sample [mm];

h – the thickness sample [mm] [29].

RESULTS AND DISCUSSION

The melting and crystallinity of aged HDPE and its recycled HDPE were performed by DSC. The DSC thermograms of the second scans are analyzed and the results are summarized in Table 2 and presented in Fig. 4 as functions of recycled HDPE content.

Table 2. DSC data for the cooling and second heating cycle of virgin HDPE and polymer blends before and after accelerated ageing process

Sample composition (virgin/recycled)	Before ageing			After ageing		
	T_m [$^\circ\text{C}$]	ΔH_m [J/g]	T_c [$^\circ\text{C}$]	T_{ma} [$^\circ\text{C}$]	T_{ca} [$^\circ\text{C}$]	ΔH_{ma} [J/g]
100vPE	134.8	-246.8	113.6	134.1	114.3	-258.6
v70/r30	133.9	-186.0	114.2	133.0	115.2	-232.7
v60/r40	133.5	-222.1	113.8	132.5	115.2	-232.0
v50/r50	131.7	-210.0	115.3	131.3	115.9	-217.9
v30/r70	130.1	-192.9	114.6	131.5	115.6	-217.8

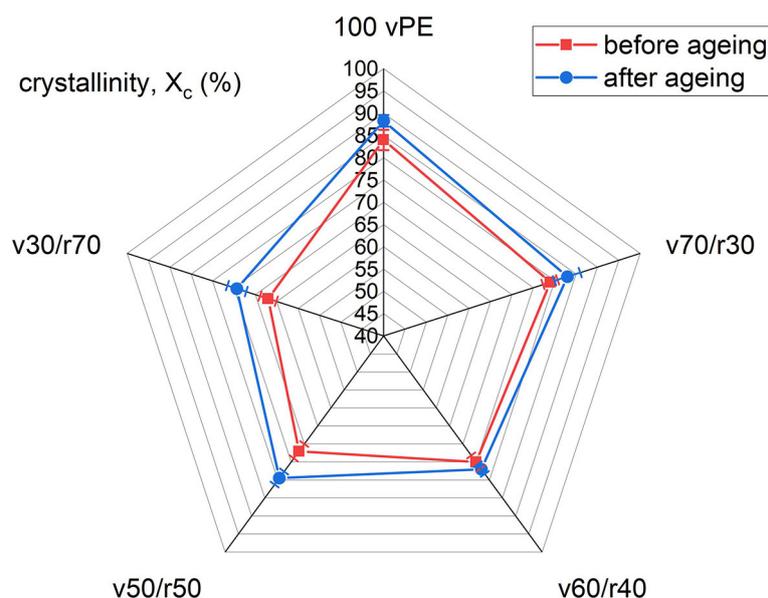


Fig. 2. The changes in the degree of crystallinity of HDPE and its recycled blends after ageing

From Table 2, it can be found that the melting point (T_m) and melting enthalpy (ΔH_m) of blends decrease with the increase of recycled content in the HDPE matrix during the ageing process. This phenomenon indicates that the macromolecular chains could organize into entities that exhibit a lower melting temperature [30]. The DSC results have shown also that photo-oxidation of the recycled blends provokes a shift of the crystallization peak to higher temperatures. However, a significant role in the changes of the mechanical properties of the thermoplastics holds their degree of crystallinity, so to better understand the structural changes that took place during photodegradation, a study of thermal properties was performed [2].

As seen in Figure 2 the highest crystallinity value (about 80%) after the aging process was noted for virgin HDPE comparison with HDPE before the aging. Next, can be observed that the degree of crystallinity for recycled HDPE/rHDPE blends increased (at about 30%) during UV -light exposure, which indicated that HDPE recrystallized due to the heat generated. Moreover, this behavior is a consequence of the recrystallization of the degraded molecules and crosslinking after UV irradiation [31]. As well as, increasing the ratio of the crystalline phase in the polyethylene results in an easier flow of the melted polymer, which is demonstrated by an increase in the mass flow index (MFI), which is consistent with literature reports. Increasing the proportion of the crystalline phase in the polymer results in an easier flow of the plasticized material, which is manifested by

an increase in the MFI index, which is consistent with literature reports [31].

The results of mechanical tests of aged HDPE and recycled blends are shown in Fig. 3. The effect of the photo-degradation process on changes in mechanical properties i.e. tensile strength (TS), elongation at break, tensile impact strength, and Shore hardness was analyzed.

Fig. 3 shows the influence of the aging process on the change of mechanical properties, including impact strength, strength, elongation, and hardness of the tested HDPE/rHDPE mixtures. From Fig. 3a-b, we can observe a decrease in strength and breaking elongation for polyethylene mixtures after the aging process, by 20% and 23%, respectively, with a simultaneous increase in stiffness, which indicates an increase in the brittleness of the material, resulting from the degradation of the macromolecule chain. On the other hand, for pure polyethylene, the tensile strength slightly increased by 10%, probably due to of cross-linking of the material. A photo-oxidation that may lead to transient strengthening has been reported by some authors, but this phenomenon is left generally unexplained [19, 32]. According to Rodriguez et al., the transient strengthening manifests in an increasing flow strength over a narrow range of UV radiation doses [19]. Fig. 3c shows a slight decrease in impact toughness (by about 10%) and an increase in hardness both for polyethylene (by about 3%) and for mixtures subjected to the aging process, with the intensity of the changes being the highest in the case of the HDPE/70 wt.%

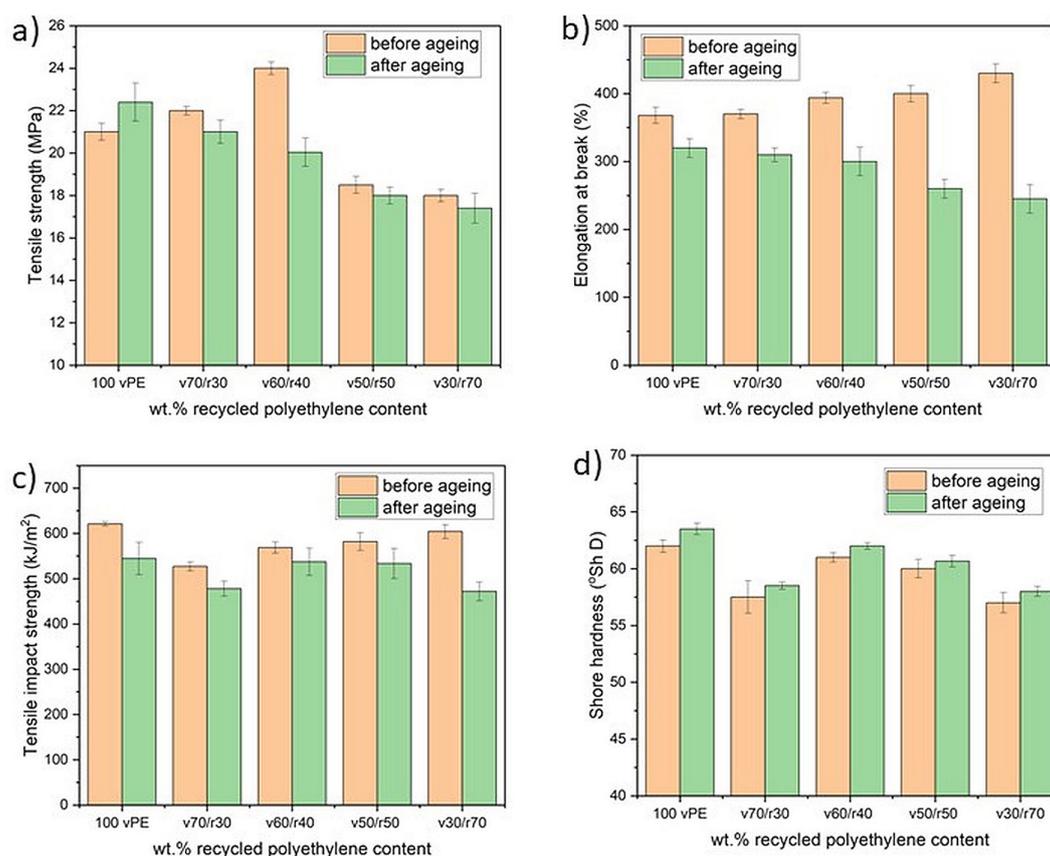


Fig. 3. The comparison of mechanical properties of virgin HDPE and HDPE/rPE blends before and after the accelerated ageing process: a) tensile strength; b) elongation at break; c) tensile impact strength; d) shore hardness

blend with the highest content of recycled HDPE, which means that the v30/r70 blend shows the highest susceptibility to aging compared to the virgin polymer 100vPE. These changes were accompanied by a significant increase in the hardness of the blends (by about 10%) (Fig. 3d), which can be explained by the photo-oxidation phenomenon on the surface of the samples.

Fig. 3c presents the results of the tensile impact strength of virgin HDPE and HDPE with recycled blends during photo-oxidation. Based on the research (Fig. 3), it was found that the virgin polyethylene (approx. 620 kJ/m²) had the highest tensile strength, and for blends (over 40 wt.% recycled polymer content), the impact tensile strength was similar to the virgin PE. The results showed that aging causes a slight reduction in impact tensile strength for both virgin HDPE and its blends with different content of recycled. The lowest impact strength was recorded for the HDPE with 70 wt.% of recycled polymer content. Based on the analysis of the sample impact test results, it was found that the majority of HDPE with 40 wt.% blends showed comparable resistance to impact loads as compared to virgin HDPE before

aging. This is possibly related to the fact that the more ductile LLDPE partially absorbs the impact energy of the hammer.

Generally, the DSC results showed that all recyclate-based HDPE blends, compared to pure HDPE after aging, show a lower tensile strength, which indicates a photo-oxidative process due to the increased degree of crystallinity, and elastic modulus, e.c. Moreover, the increase in the degree of crystallinity enhancement the stiffness of the recycled plastic and consequently reduces the impact resistance. A similar result was obtained by Santos et. al [33], which verified the influence of maleic anhydride-grafted LLDPE (LLDPE-g-MA) in ultra-high molecular weight polyethylene (UHMWPE) with and without linear low-density polyethylene (LLDPE) on aging resistance and thermo-mechanical properties.

The aging process most often causes polymer degradation, i.e. a change in its average molecular weight, and an increase in the melt flow index (MFI), which results in a decrease in impact tensile strength for the tested materials, regardless of their structure. The parameters of the aging process indicated that the main factors responsible

for the observed changes were UV radiation and the action of water, the negative effect of which is evidenced by the change in discoloration and white deposition on the surface of the samples.

In addition, to show the susceptibility of mechanical features, i.e. tensile strength, elongation at break, tensile impact strength, and shore hardness, the aging coefficient (K) was calculated after accelerated aging according to the Eq.1, and its results are presented in Fig. 4. Overall we know, that the coefficient CI informs the degree of polymer degradation [25].

The coefficient $K_1 = 100$, corresponds to the insensitivity to the aging process, so the higher the K_1 value, the more resistant the polymer is to degradation. While the $K_1 \geq 100$, i.e. the aging has an unfavorable effect on the tested feature, and its value decreases. In the case of $K_1 \leq 100$, it means that favorable structural changes take place in the polymer, as a result of which the tested property is improved. The intensity of the changes taking place, both positive and negative, is the greater the difference between the coefficient K and the value of 100 [34].

Based on the analysis of the test results and statistical calculations, it can be concluded that the results of determinations of samples subjected to the aging process are characterized by a relatively large scatter of results. The coefficient of variation, which is a measure of the scatter of values in the study population for the samples before accelerated aging, was much smaller than

after aging. Thus, it can be assumed that the aging process disrupts the virgin material equilibrium system. The coefficient K_1 of aging resistance for virgin HDPE and recycled HDPE blends was calculated from Eq. 4.

$$K_I(\%) = \frac{A_t \text{ before ageing}}{A_0 \text{ after ageing}} \times 100 \quad (4)$$

where: A_t – a value of average means of the property after aging;
 A_0 – a value of average means of the property before aging.

Based on the obtained values, the coefficient K_1 indicators for individual strength characteristics were determined and a collective diagram was drawn up (Fig. 4) of the sensitivity of the tested materials to the aging process carried out in laboratory conditions in an accelerated manner. Figure 4 illustrates the changes in mechanical properties of virgin HDPE and its blends after accelerated ageing (K index).

From Fig. 4 can be seen that the HDPE mixture with 40 wt.% recyclate content (index $K_1 = 95\%$) showed the greatest resistance to aging, compared to the virgin 100vPE polymer ($K_1 = 87.5\%$). On the other hand, with the increase in the content of recyclate in the blend, the sensitivity of the mixtures to photo-oxidation processes increased. From Figure 4 can be seen the coefficient K results from the elongation at break is the most subject to photo-degradation (K about 60%).

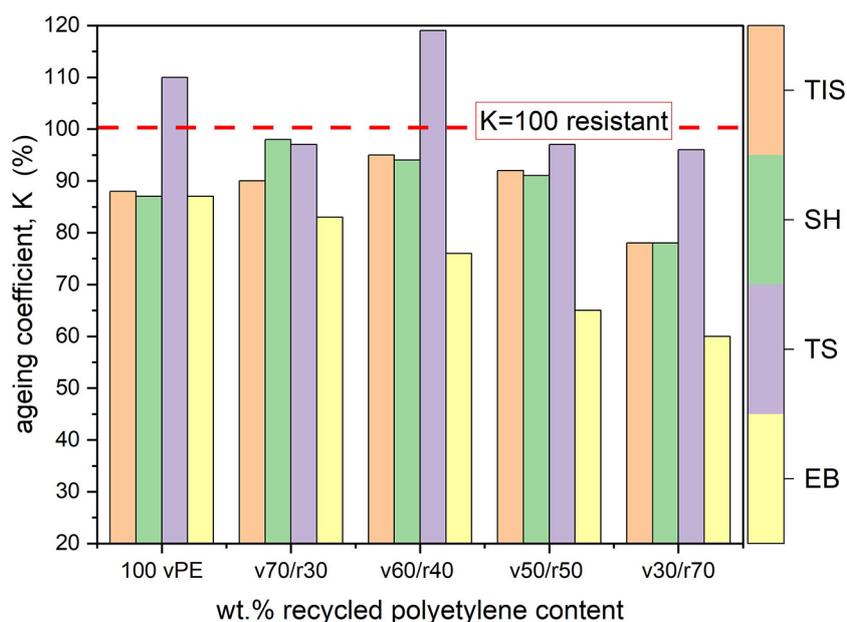


Fig. 4. The percentage change in the mechanical properties over time of virgin HDPE and its blends after accelerated aging (coefficient K in %)

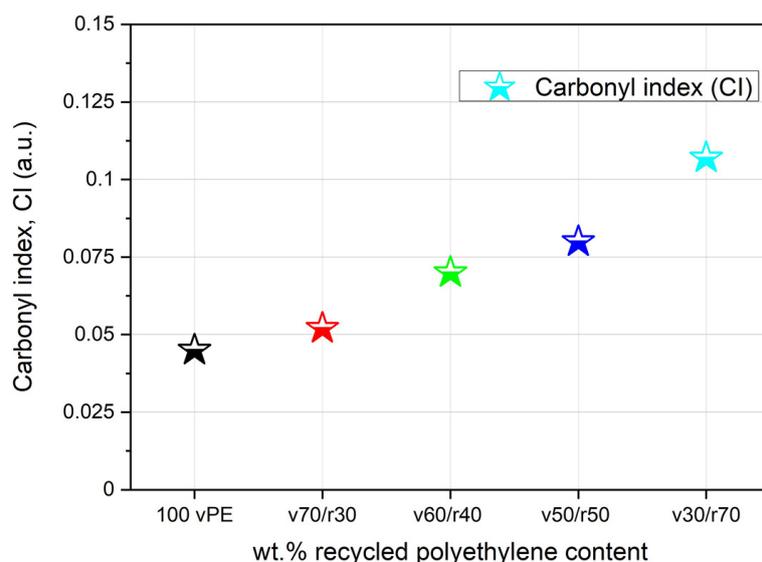


Fig. 5. The changes in carbonyl index in [a.u.] of virgin HDPE and its blends as a function of recycled HDPE content after the accelerated ageing process

FTIR spectroscopy is a numerously used analytical technique for monitoring the oxidation process of polyethylene [27]. Based on the measurement of CI, structural changes in HDPE resulting from the ageing process were determined. For oxidative degradation of high-density polyethylene (HDPE), this change occurs in the carbonyl band. This data can be looked at qualitatively in the entire carbonyl functional (C=O) groups wavenumber range (1700-1750 cm^{-1}). A quantitative measure of the carbonyl absorption is the carbonyl index CI, which was calculated according to Eq. 1, by the ratio between a peak of interest A_{peak} and the reference peak A_{ref} and results were presented in Fig. 5.

From Figure 5 can be seen that the CI values are much higher about 75% for 70 wt.% recycled HDPE compared to virgin polyethylene, which is associated with chain scission. This determination value of the CI indicated that the degradation of the HDPE/rHDPE blend with the highest recycled polyethylene (100vPE) content has proceeded much faster than virgin polymer. These results show that the recycled blends of HDPE there are photo-oxidation is more sensitive. Similar results were obtained by Zhao et al. [9], which proved that the degree of oxidation and chain scission on HDPE surfaces is high when with the increasing aging time.

CONCLUSIONS

The mechanical changes during the photo-degradation process i.e. impact resistance, tensile strength, elongation at break, hardness, aging coefficient (K_t),

and carbonyl index (CI) in high-density polyethylene with recycled polymers have been investigated.

The results demonstrated that the aging process reduces the impact toughness and tensile strength of the sheets made with recycled HDPE blends used for the production of sheets of mudflap, due to macromolecular chain scission provoked by oxidation, which was confirmed from FTIR spectroscopy. The results showed that the photo-degradation causes changes to a moderate extent, which does not exclude the use of a blend with a 40 wt.% content of recycled for the production of car mudflaps with good resistance to weather conditions, which certainly does not affect the loss of elasticity of the surface layer of the mudflap.

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