

Preparation and characterization of polymer blends based on waste from automotive coverings^{*})

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Abstract: This paper presents the results of the mechanical recycling of post-production automotive coverings. Shredded carpet waste was used to obtain new systems based on thermoplastic polymer materials, in this case linear low density polyethylene (LLDPE). As automotive carpet waste, we used a material composed mainly from polyamide 6, chalk, polypropylene. Optical microscopy and scanning electron microscopy characterized the structure of both the automotive lining and finished materials. The dependence of the mechanical properties and thermo-oxidative stability on the mutual content of the components was investigated.

Keywords: automotive coverings, carpet/covering wastes, material recycling, mechanical properties, thermal stability.

Otrzymywanie i właściwości mieszanin polimerowych na bazie odpadów z wykładzin samochodowych

Streszczenie: Przedstawiono wyniki prac własnych dotyczących mechanicznego recyklingu poprodukcyjnych wykładzin samochodowych. Rozdrobnione odpady wykładzinowe użyto do otrzymania układów na bazie tworzywa termoplastycznego, w tym wypadku liniowego polietylenu małej gęstości (LLDPE). W charakterze odpadów wykładzin samochodowych zastosowano materiał, w którego składzie występują głównie poliamid 6, kreda i polipropylen. Metodami mikroskopii optycznej oraz skaningowej mikroskopii elektronowej (SEM) scharakteryzowano strukturę zarówno wykładziny samochodowej, jak i nowo wytworzonych materiałów. Zbadano zależność właściwości mechanicznych oraz stabilności termooksydacyjnej od udziału poszczególnych składników.

Słowa kluczowe: wykładziny samochodowe, odpady dywanowe/wykładzinowe, recykling materiałowy, właściwości mechaniczne, stabilność termiczna.

Carpet and covering waste – in addition to various types of synthetic fibers, among others: polyamides 6 and 66 (PA6 and PA66), poly(ethylene terephthalate) (PET) [1], polypropylene (PP), polyacrylonitrile (PAN), and natural materials (wool, cotton, jute) – contain large quantities of minerals, synthetic rubbers, bituminous compounds,

etc. [2]. Due to the complex structure and permanent connection of the textile with the binding materials, post-production and post-consumer carpet wastes are unsuitable for direct, secondary textile processing and to a large extent are sent to landfills. Post-production waste (“clean ones”) account for only approx. 2% of production. While, the amount of generated post-consumer wastes (“dirty ones” or “complex ones”) is difficult to determine. The surface weight of the floor covering, depending on its type, is 1.2–5.5 kg/m², and the service life varies between 3–10 years [2]. One can, therefore, estimate, that in recent years about 60 000 tons per year of used carpets and coverings have been created in Poland [3].

The legal status regarding waste management is regulated by the Act of 14 December 2012 describing the scope of obligations to obtain appropriate decisions in the area of waste generation and the generation of waste in connection with the operation of the installation. Until now, entities providing services in the field of construction,

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demolition, renovation of facilities, cleaning of tanks or equipment, as well as cleaning, maintenance, and repairs, and the treatment of asbestos-containing waste in transportable devices were required to obtain a decision approving a waste management program. In turn, in September 2018, an amendment to the Act of Waste was adopted in which further general changes are planned and what is important – the implementation of the detailed provisions describing the guidelines to the general requirements set out in the Act on Waste. The most important changes include: (i) new regulations regarding the visual control system of waste storage locations, (ii) new rules on fire-fighting requirements for waste storage sites, and (iii) an obligation to submit an application for the adaptation of existing waste management permits. Undoubtedly, 2019 will be demanding in terms of changes in the field of waste management. This, however, allows us to believe that Poland is slowly moving towards a more responsible approach to secondary raw materials.

Just as for plastic materials, the main recycling methods used for carpets and covering waste are as follows: mechanical recycling, raw materials recycling and energy recycling [4, 5]. Mechanical or material recycling involves the regranulation or defiberification of post-consumer carpet waste and its re-use for the production of various products. Depending on the subsequent application, material recycling is carried out with a separation into components and segregation, or without such separation [2, 6, 7]. In turn, raw materials or chemical recycling consists of obtaining valuable, useful chemical compounds from waste plastics that can be used as fuel or raw materials in the chemical industry [8–10]. Energy recycling, *i.e.* burning with energy recovery, meets with great societal reluctance due to the waste gases associated with atmospheric pollution and an increased greenhouse effect [11]. Shredded carpet waste can be used in systems based on thermoplastic polymers for the construction of anti-vibration mats or thermal insulation [12], substituting 40–100% of mineral fillers, *e.g.* chalk in the bottom layers of carpet or undercoat of PVC covering [13, 14], or as a reinforcement in lightweight cementitious composites [15]. Moreover, Mirafteb and Lickfold [16] reported that carpet fibrous waste can be applied in clayey or substandard soils with the intention of enhancing soil cohesion and reinforcement. The findings suggest that the inclusion of as much as 10% fiber can be tolerated and would enhance internal cohesion, shear, and comprehensive strength as well as load-bearing capacity of this kind of soil. Nevertheless, the above-described recycling methods mainly concern post-production waste, whereas post-consumer wastes are mainly used in energy recycling.

In the Institute of Materials Science and Engineering within the research tasks of the Students' Research Group "POLIMAT", together with the patron of the group, Nicrometal S.A., we have developed a method of shredding the waste of automotive coverings and subsequently introducing the waste in a molten state to lin-

ear low density polyethylene (LLDPE). The mechanical properties and thermooxidative stability of the obtained composites were investigated and the structure was characterized by means of optical and scanning electron microscopy. The results of these analyses are the subject of this publication.

EXPERIMENTAL PART

Materials and preparation of the blends

A polymer matrix linear low density polyethylene (LLDPE) (LL 1004YB, Exxon Mobil) was used for the preparation of polymer blends. As the filler, we used post-production automotive covering waste under the trade name of 4701 Trooper Ebony (TrEb), composed of PA6 (24%), calcium carbonate (28%), PP (8%), PET (7%), *etc.* Compositions of LLDPE/TrEb containing 0–90 wt % of automotive coverings were prepared by melt blending using a counter-rotating twin-screw extruder (LSM30, Leistritz Laborextruder, Nuremberg, Germany) with a 34 mm screw diameter and a length/diameter ratio of 23, equipped with two gravimetric feeders. The extruded material was cooled in a water bath and granulated. In the compounding process, the following temperatures were determined: feed zone: 20 °C, zone 1: 100 °C, zone 2: 100 °C, zone 3: 170 °C, zone 4: 170 °C, zone 5: 170 °C, zone 6: 170 °C, zone 7: 180 °C, zone 8 (nozzle): 180 °C. For all materials, a 40 rpm of screw speed was used.

Dumbbell shape samples for tensile tests were prepared by injection molding using a Boy 15 (Dr. BOY GmbH&Co., Germany) injection molding machine with the following parameters: injection pressure 80–120 MPa, temperature 170 °C, mold temperature 30 °C, holding down pressure of 15 MPa for 15 s and cooling time of 30 s.

Methods of testing

The distribution of the fibers included in the automotive carpet, neat LLDPE and fibers themselves were analyzed by scanning electron microscopy (SEM, Hitachi SU-70, Naka, Japan). The samples for SEM analysis were cryofractured in liquid nitrogen and subsequently coated (2–5 nm) in a vacuum with a thin gold film before the tests. In addition, light microscopy observations were performed using a Nikon MM-40 microscope, operating in the transmission light mode.

The tensile properties of the LLDPE/TrEb blends were tested using a universal tensile test machine (Autograph AG-X plus, Shimadzu) equipped with an optical extensometer at room temperature according to EN ISO 527. The stress–strain curves were obtained at a strain rate of 5 mm/min. For each material, six measurements were performed, and then the results were averaged.

The thermooxidative stability of the investigated materials was evaluated by thermogravimetric analysis (TGA 92-16, Setaram). Samples were heated in the temperature

range from 20 to 700 °C at a rate of 10 °C/min in dry, synthetic air ($N_2 : O_2 = 80 : 20$ vol %).

RESULTS AND DISCUSSION

Micro and macroscopic structure of the prepared materials

The morphology of immiscible polymer blends and polymer compositions depends on the type of components, their ratios and melt viscosities, as well as processing conditions [17]. In most heterogeneous systems, we observe a morphology where one phase is distributed into another. The SEM micrographs of the fracture surfa-

ces of neat LLDPE, LLDPE/TrEb 70/30, LLDPE/TrEb 50/50 and LLDPE/TrEb 30/70 systems are shown in Figs. 1a–1d, respectively. Additionally, in Fig. 1e and 1f we observe the post-production automotive covering (at two different magnifications), provided by Nicrometal S.A, which clearly indicates the fibrous nature of the material. The SEM micrograph of the neat LLDPE shows that the surface of the polymer is rather uneven with evidence of some protuberances on the submicron scale (Fig. 1a), which may indicate the semi-crystalline nature of the polymer matrix. In the micrographs of LLDPE/TrEb systems (Figs. 1b–1d) we clearly observe fibers sticking from the fractured surface of LLDPE and their thickness was estimated to be around 30–40 μm . Moreover, the fibers seem to be “anchored” with one end in the matrix, thus

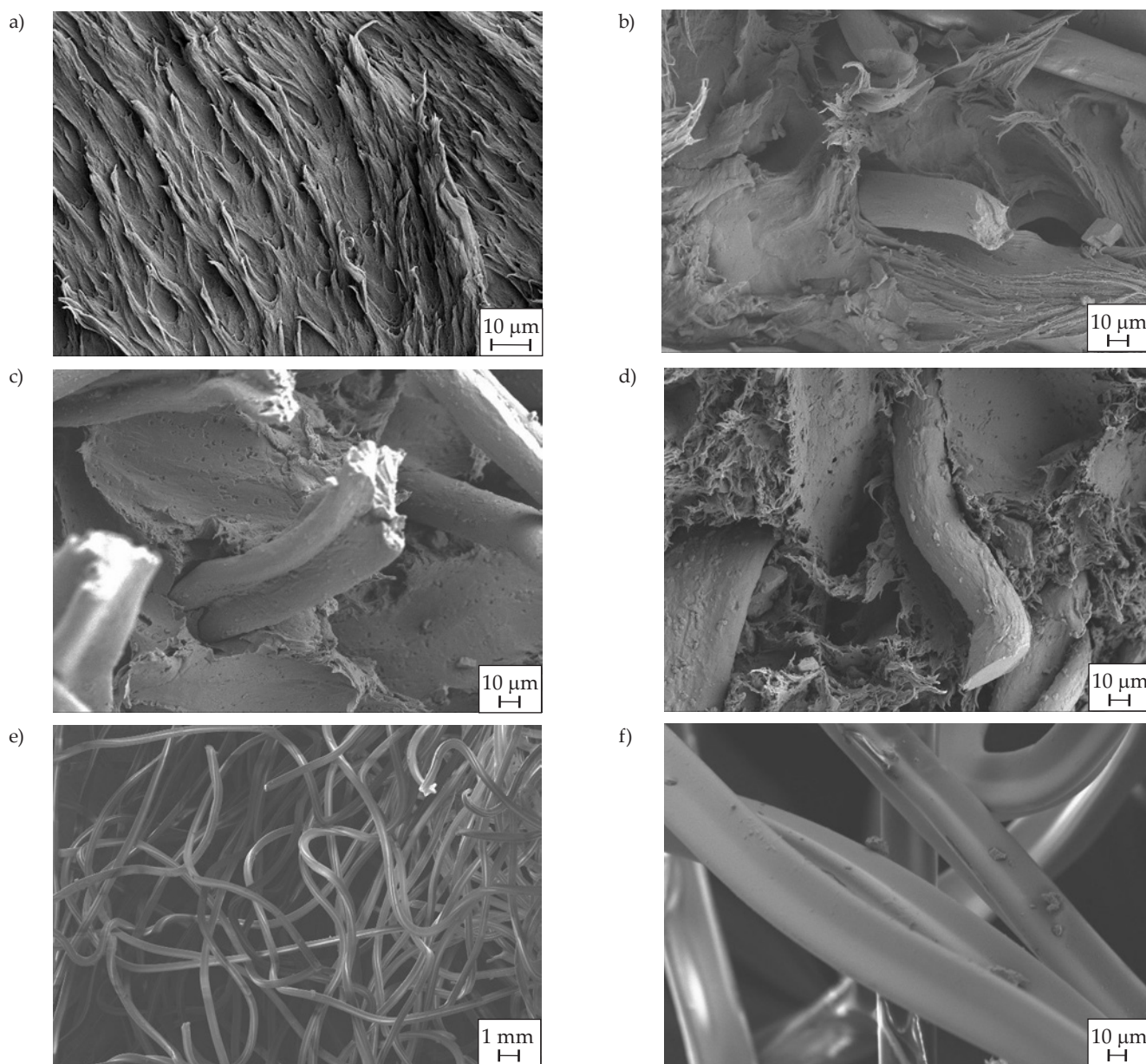


Fig. 1. SEM micrographs of: a) LLDPE, b) LLDPE/TrEb 70/30, c) LLDPE/TrEb 50/50, d) LLDPE/TrEb 30/70, e, f) TrEb (at different magnifications)

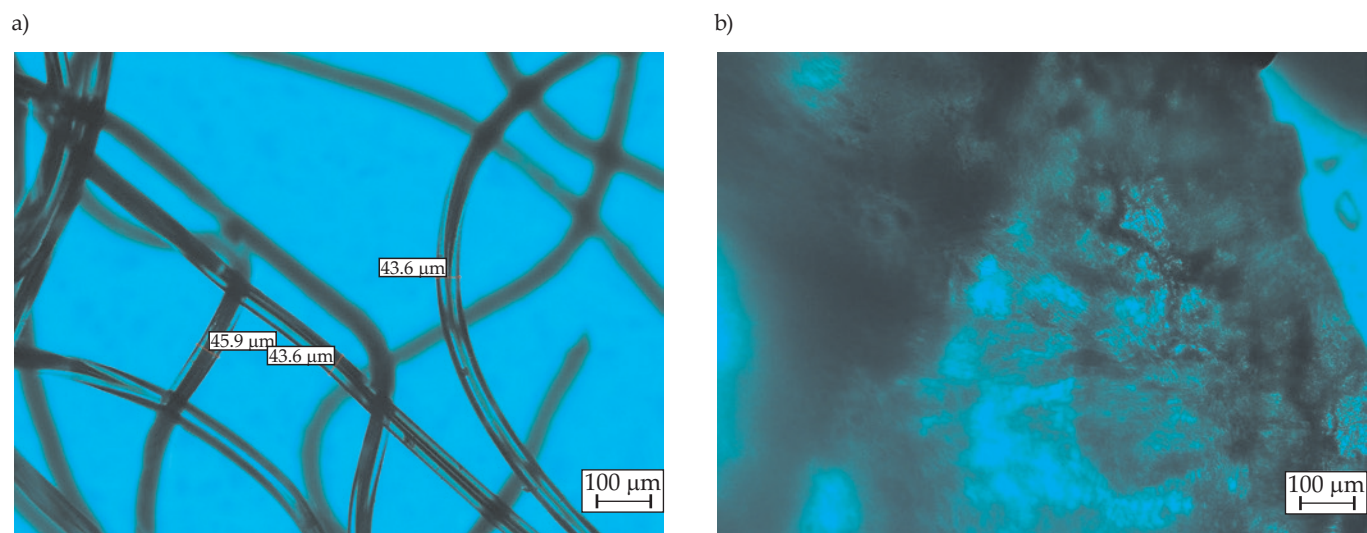


Fig. 2. Optical microscopy images of: a) automotive covering TrEb (100 \times), b) LLDPE (100 \times)

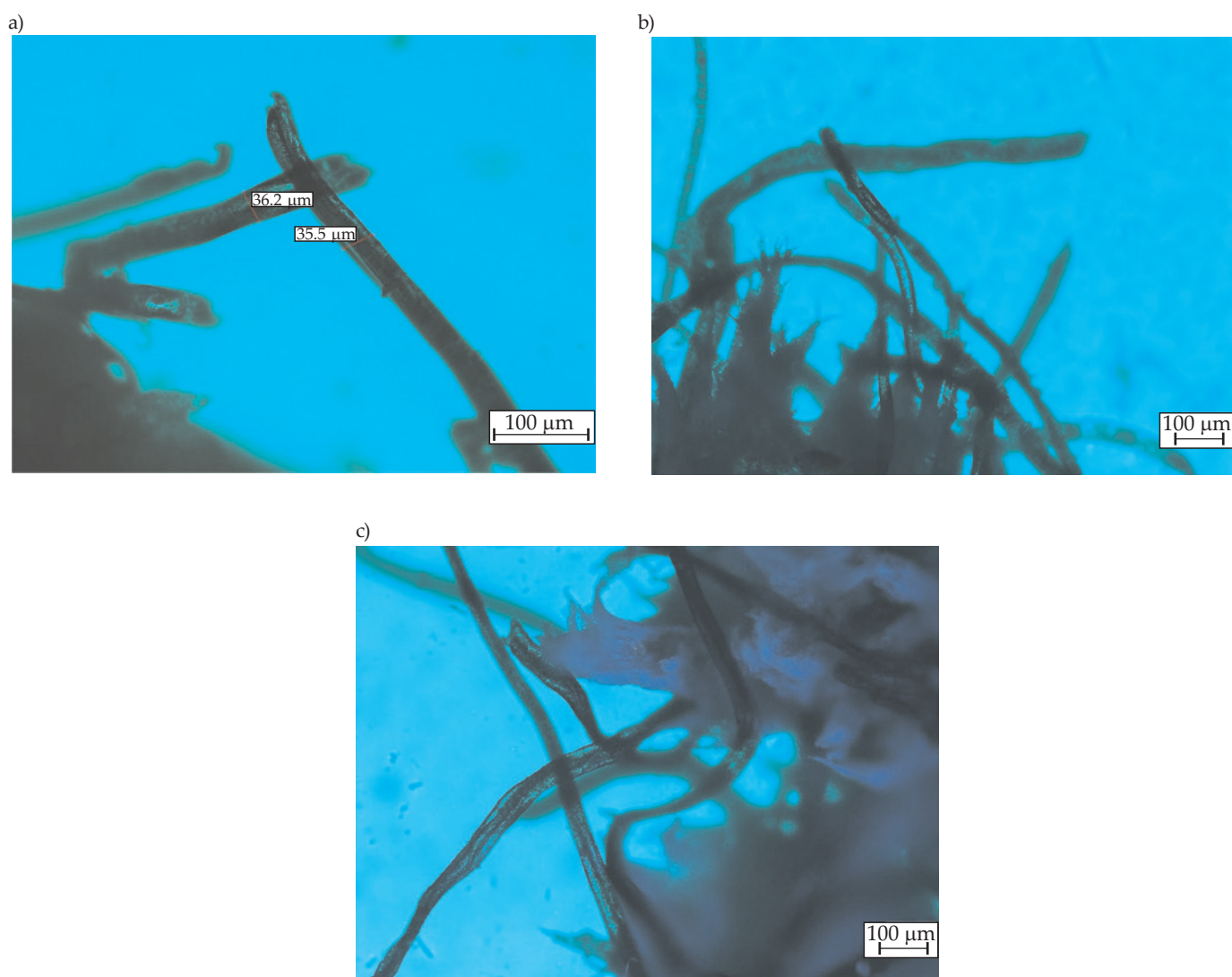


Fig. 3. Optical microscopy images of the LLDPE/TrEb systems: a) 70/30 (200 \times), b) 50/50 (100 \times), c) 30/70 (100 \times)

one can expect the improvement in functional properties of the final materials, like mechanical or thermal.

Optical microscopy was applied in order to observe the automotive covering Trooper Ebony (Fig. 2a), neat LLDPE (Fig. 2b) and LLDPE/TrEb systems (Fig. 3). The observations on the macro scale were in agreement with the observations made by SEM microscopy. A clearly defined fiber structure, with fibers exhibiting a thickness of about 40 μm , was observed in Fig. 2a. While, in the case of neat LLDPE, a smooth surface with partial transparency was visible. In turn, in all systems based on LLDPE and the automotive covering (Trooper Ebony), we rather observed the fibers than the matrix itself, regardless of the amount of post-production automotive covering in the system.

Tensile properties

From an application perspective, the analysis of the mechanical properties of LLDPE/TrEb systems is of great importance. Therefore, LLDPE/TrEb blends were subjected to uniaxial tensile tests. Representative stress-strain curves of the investigated materials are plotted in Fig. 4. Moreover, the dependence of the tensile strength (TS) and the relative elongation at break (ϵ_b) vs. composition are depicted in Fig. 5. The presented results clearly show that, along with an increase of TrEb content in the polymer blend, the values of tensile strength and relative elongation at break regularly decreased. The reason for such a deterioration in the tensile properties results mainly from the immiscibility of blend components. In particular, this was the case for polar polyamide (PA6) and non-polar polyolefins [18, 19] being either present in recycled material (PP from the carpet backing) or applied as a second component. Moreover the character of the mechanical response in LLDPE/TrEb blends strongly depends on the composition, morphology developed during compounding and purity of the stock (since recycled materials were used). Careful analysis of stress-strain deformation curves provides additional information on the nature of fabricated materials and allows us to distinguish three different types of systems within the LLDPE/TrEb series. We can characterize those types as the following: 1) ductile polymers with three stages of sample deformation, including yielding, neck forming and strain hardening (neat LLDPE and LLDPE/TrEb 90/10 sample); 2) materials fracturing without necking or strain hardening effect (compounds loaded with 20–50% of TrEb waste), and 3) systems in which deformation is characterized by a single maximum occurring between 3% to 13% of elongation followed by a gradual stress drop down to ~ 0 MPa (materials with TrEb as a predominant phase, ≥ 60 wt % TrEb).

From the collected results, it is apparent that even the addition of 10 wt % of automotive covering waste decreases the tensile properties to a great extent. Although the yield point can be distinguished at the stress-strain curve

of LLDPE/TrEb 90/10, one can immediately see that both the tensile strength and elongation at break decreased significantly, by $\sim 43\%$ (TS) and $\sim 30\%$ (ϵ_b) compared to neat LLDPE. Along with an increase in TrEb content, a decrease in the values of TS and ϵ_b were observed. In the second group, the tensile strength decreased from 10.6 to 8.1 MPa, whilst elongation at break decreased from 105.0 to 36.3% for LLDPE/TrEb 80/20 and LLDPE/TrEb 50/50, respectively. Nevertheless, the obtained results are in good agreement with the study of Kowalska *et al.* [2] dealing with comparable PE-LD based composites with carpet wastes of polyacrylonitrile and polyamide 66.

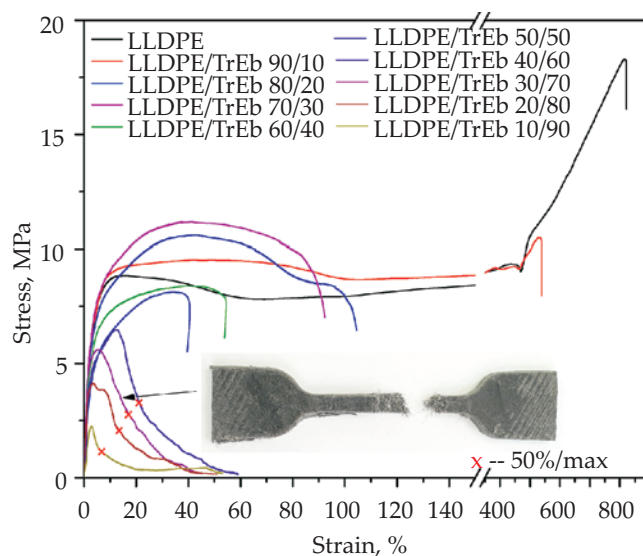


Fig. 4. Representative stress-strain curves recorded during a static tensile test

The last group of systems, containing ≥ 60 wt % of TrEb exhibited very limited mechanical properties, with tensile strengths lower than 6.6 MPa and elongation at break less than 21.4% (for the purpose of comparison, in the present work ϵ_b was determined as the elongation at which the stress dropped to 50% of the maximum value, as marked with a red x symbol in Fig. 4). This effect strongly depended on the blend morphology. Indeed, in the latter group, the blend major phase was formed by TrEb carpet waste, which is a heterologous and rather discontinuous phase, as evidenced by the microscopic examinations presented earlier. Apparently, the fibers and fillers originating from the TrEb covering were not sufficiently moistened by the LLDPE phase to develop a material with a stable, continuous morphology able to resist high mechanical loads. A visual inspection of the sample after the tensile test also reaffirmed the poor adhesion between the components. As depicted in the insert in Fig. 4, the fracture is irregular and a number of single fibers sticking from the matrix can be seen.

Such a downward trend in mechanical characteristics was already expected since our investigation covers a broad composition range thus changes in sample morphology are likely to occur (including phase-inversion).

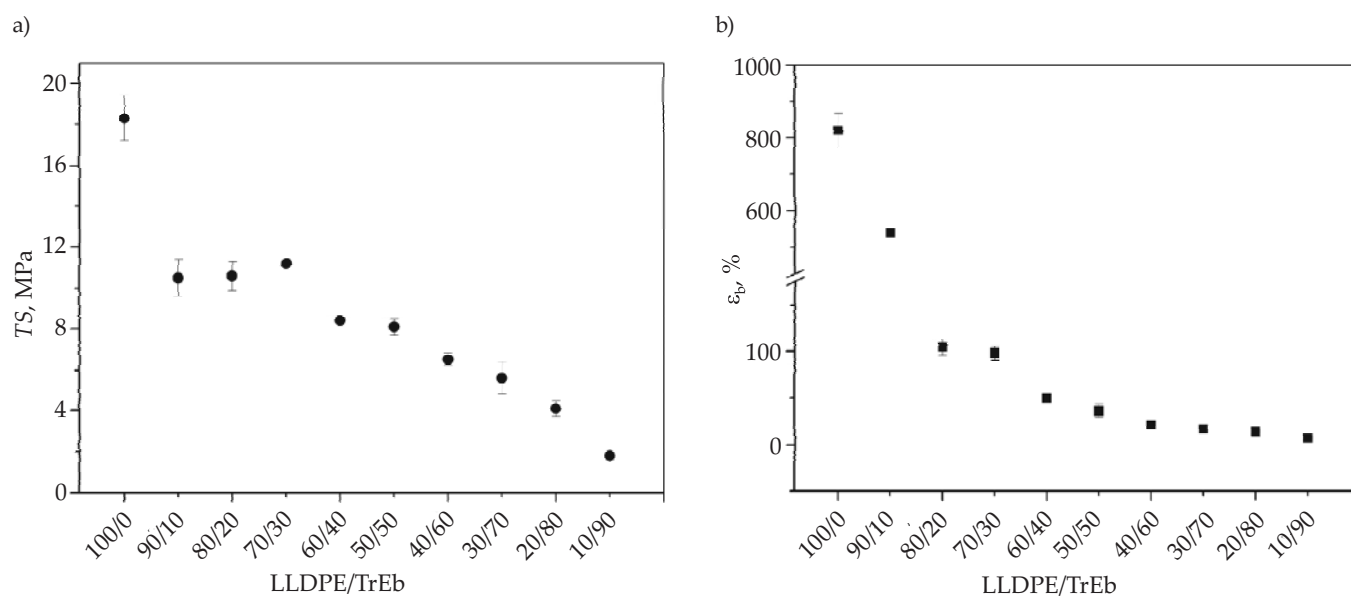


Fig. 5. Dependence of: a) tensile strength (TS), b) relative elongation at break (ϵ_{br}) as a function of the composition of LLDPE/TrEb blends

Moreover, the recycled TrEb was in the form of a dry mix of randomly oriented short fibers and organic fillers, whose poor moistening leads to discontinuities affecting the mechanical properties. To conclude, sufficient mechanical performance of LLDPE/TrEb is possible only at a TrEb recyclate content less than 60%. Moreover, at this point, it is worth highlighting that adding compatibilizer as a third component can further promote interfacial adhesion between polyamide and polyolefins [19, 20] to obtain a product with finer morphology and improved properties. Studies on compatibilized LLDPE/TrEb systems will be the subject of our forthcoming work.

Thermooxidative stability

In Fig. 6a and 6b we present the influence of the addition of the shredded automotive covering on the thermooxidative stability of the LLDPE based compositions.

The thermostability of the polymers and composites depends on many factors, including the presence of low molecular weight substances, branching or crosslinking [21]. Polyethylene, like other high molecular weight organic compounds, exhibits limited resistance to high temperatures, resulting mainly from a specific energy of CC (346 kJ/mol) and CH (411 kJ/mol) chemical bonds [21, 22], especially in the presence of oxygen. This effect is noted by analyzing the obtained results (Table 1) where it was observed that the thermal degradation in an oxidizing atmosphere of the obtained systems begins in the temperature range of 339–355 °C (5% of mass loss) and it is similar for all systems regardless of the content of TrEb, as in the case of a 10% of mass loss.

A significant change in the decomposition temperatures was noted in the case of a 50% mass loss, especially for systems containing 10 to 70% of automotive covering, TrEb, – materials were less stable and exhibited lower mass loss

Table 1. Thermal properties of the materials: temperatures of 5%, 10%, 50% and 90% of mass loss and the temperature corresponding to the maximum of mass loss (T_{DTG1} , T_{DTG2} , T_{DTG3}) in an oxidizing atmosphere

Sample	$T_{5\%}$ °C	$T_{10\%}$ °C	$T_{50\%}$ °C	$T_{90\%}$ °C	T_{DTG1} °C	T_{DTG2} °C	T_{DTG3} °C
LLDPE	348	371	450	467	374	457	496
LLDPE/TrEb 90/10	354	375	432	467	451	501	–
LLDPE/TrEb 80/20	350	373	432	500	448	494	–
LLDPE/TrEb 70/30	339	363	427	512	418	470	–
LLDPE/TrEb 60/40	350	374	435	605	422	–	–
LLDPE/TrEb 50/50	353	378	445	687	441	–	–
LLDPE/TrEb 40/60	355	377	434	–	422	464	–
LLDPE/TrEb 30/70	339	377	438	–	418	460	–
LLDPE/TrEb 20/80	358	388	447	–	414	453	–
LLDPE/TrEb 10/90	349	382	464	–	420	480	–
TrEb	349	380	472	–	426	469	503

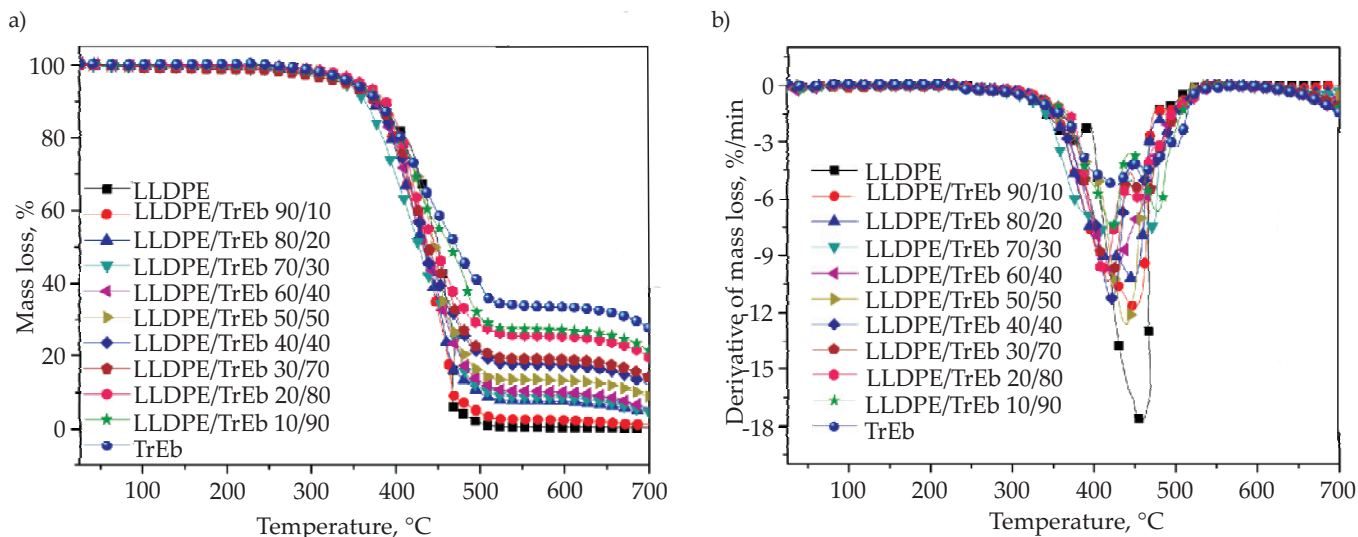


Fig. 6. a) TG, b) DTG curves as a function of the temperature for LLDPE/TrEb blends under an air atmosphere at the heating rate of 10 °C/min

temperatures than neat LLDPE. The obtained results suggest that, in the case of materials for which the mechanical properties have been reduced the most (systems contain 60–100% of TrEb), they do not show a mass loss of more than 80%. This would indicate that the residue consists of mineral fillers that are part of the material binding to the covering fleece. The same mineral component increases the temperature of 90% of mass loss in the systems containing 10–50% TrEb (increase up to 687 °C, *i.e.* by 220 °C above the LLDPE decomposition temperature).

In Fig. 6b, a multistage thermal decomposition process for the tested materials can be observed. Only two samples (LLDPE/TrEb 60/40 and LLDPE/TrEb 50/50) undergo a one-stage decomposition process and the rest decomposes in two or three stages. The temperatures corresponding to the maximum mass losses for each stage are summarized in Table 1. The lowest temperature for the first decomposition stage is 374 °C and relates to LLDPE, in which thermal degradation is a free radical process, although it may also be accompanied by non-radical molecular reactions, leading to statistical chain cracking or detachment of hydrogen atoms and formation of macroradicals. The radicals formed in the first stage of the process attack other macromolecules, which may cause subsequent chain reactions, such as reactions of transferring a hydrogen atom to another molecule or fragmentation of chains. Oxygen molecules, found in the atmosphere of the research environment, react with radicals, resulting in transition products (hydroperoxide and alkoxy radicals). In subsequent stages, degradation into products containing oxidized groups of various types (carbonyl, hydroxide) occurs, which confirms the three-stage process of mass loss (Table 1). As in the case of LLDPE, a three-stage mass loss process is indicated by TrEb. The most intensive depolymerization and thermooxidative destruction for TrEb occur at 426 °C (first stage of decomposition). In subsequent stages, the TrEb sample shows

slightly higher temperatures than neat LLDPE. In other systems, a two-stage mass loss was observed in the temperature range of 414–451 °C (first stage) and 453–501 °C (second stage). The appearance of only two maxima in DTG curves for systems containing the TrEb indicates the heterophase structure of the materials at the macrophase level (as confirmed by SEM and optical microscopy) – the phase deriving from automotive covering (fiber/fleece) degrades much later than the LLDPE-derived phase. On the basis of the conducted research, it can be concluded that the modification of LLDPE with the addition of the shredded automotive coverings has an influence on the improvement of thermooxidative stability of the obtained systems.

CONCLUSIONS

The present study confirms the successful incorporation of the shredded post-production automotive coverings (Trooper Ebony) into the LLDPE polymer matrix via the melt mixing process. It is clearly visible that the addition of post-production automotive coverings affects the morphology, mechanical properties, and thermooxidative stability. At this stage of the research it can be clearly stated that the introduction of a larger amount of TrEb (>50%) is pointless – these materials are characterized by a large decrease in the mechanical properties, with a slight effect on the thermooxidative stability. On the other hand, it seems reasonable to introduce 20–50% of post-production automotive coverings into LLDPE, or other polyolefins, in order to recycle them and obtain new materials for use in the technical industry, for instance geotextiles, anti-erosion mats, gardening geotextiles, dimpled membranes, damp proofing, *etc.* The obtained results suggest carrying out additional trials relating to the introduction of compatibilizing agents between the phases (automotive covering and polymer matrix), which

could effectively contribute to the improvement of moistening of the fibers constituting the covering and thus improve the interfacial interactions, which in turn will affect the utilitarian properties of the materials.

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