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Short communication

Strain-reporting pyrene-grafted polyethylene

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ABSTRACT

High Density and Very Low Density PolyEthylene-*graft*-Maleic Anhydride (HDPE-MAH and VLDPE-MAH respectively) functionalized with 1-aminopyrene (AP) are prepared via reactive extrusion. Covalent attachment of AP is confirmed by lack of residual free AP after extraction with dichloromethane. Differential scanning calorimetry, tensile test, dynamical mechanical thermal analysis and rheology are employed to investigate the influence of AP incorporation on thermal, mechanical and rheological properties when grafted on both HD and VLDPE-MAHs. Fluorescent emission spectroscopy reveals pronounced changes in fluorescent behavior under stress due to the breakup of the pyrene excimers. For HDPE-MAH-AP this change is sudden with a clear drop of excimer content (I_E/I_M) of around 50% due to necking of the material stretched above 50% strain. In contrast, VLDPE-MAH-AP shows no necking and a linear decrease of I_E/I_M ratio down to 30% when elongated up to 1100% strain, while HDPE-MAH-AP broke after 200% strain.

1. Introduction

The monitoring of strain in polymeric materials is of great importance to prevent premature failure like stress fracture or fatigue [1]. Polyethylene (PE) is the most widely used commodity thermoplastics due to its good solvent resistance, excellent flexibility, low cost and ease of processing, thanks to which it finds application in automotive, medicine, aerospace and electronics [2,3]. Despite the wide use, PE suffers from premature failure like stress cracking of pipes [4–6] cables [7,8] and storage tanks [9] as well as of laboratory supplies and implants for medical applications, which could be prevented by suitable monitoring. One of the most promising tools for strain detection in polymers is fluorescent emission spectroscopy [1]. This noninvasive method allows for a real time in situ analysis and it is based on a mechanochromic polymer containing a fluorophore showing changes in fluorescence in response to a mechanical stimulus [10,11]. Changes in fluorescence during deformation can be induced by an altered aggregation or alignment of the fluorophore, typically transition from excimer to monomer due to the separation of the aggregates [11,12] or aggregation-induced emission phenomena [13,14]. Fluorophores are typically dispersed in the bulk of the appropriate polymer matrix by means of solution or melt blending. The polymer chains remain structurally unaltered and the obtained material is generally biphasic unless the used dye is fully soluble in the pristine polymer [10]. During the

lifetime of the material, the dye usually migrates from the core to the surface and will alter the fluorescence properties of the material. To circumvent that problem, efforts are being put into the preparation of mechanochromic polymers with fluorophores covalently attached to the macromolecular chains. This method allows to obtain a material with homogeneously distributed fluorophore molecules preventing its diffusion, leaching and segregation. However, the need of appropriate functional groups on both macromolecules and fluorophores limits its applications [1,10]. Even more demanding is the grafting procedure, which must be the simplest and cheapest possible, particularly for commodity polymers, like PE.

Despite the extensive research on mechanochromic polymers in recent years, PE with covalently attached fluorophores remains mainly unexplored, as PE lacks proper functionality for grafting. The group of Weder and Crenshaw published reports on mechanochromic blends of PE and cyano-oligo(p-phenylene vinylene) [15,16]. The mechanical deformation of those materials led to a pronounced change in photoluminescent characteristics and when the strain reached 500%, the monomer to excimer emission ratios I_M/I_E were increased by a factor of up to 10. Kunzelman et al. reported on self-assessing polymer blends based on poly(ethylene terephthalate glycol) and linear low density polyethylene (LLDPE) with a cyano-substituted oligo(p-phenylene vinylene) obtained by melt-processing and subsequent quenching below T_g [17]. Ruggeri et al. reported on two perylene tetracarboxylic acid

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bis-imides containing linear or branched alkyl chains dispersed in LLDPE at low loadings by melt processing [18]. To the best of our knowledge, strain detection in PE with covalently attached fluorophores is unprecedented. Brown et al. investigated selectivity and efficiency of pyrene attachment to PE films by bombardment with MeV-range protons; however, the potential of this material as strain detector was not explored [19].

Herein, we describe a strain-reporting polymer based on polyethylene-*graft*-maleic anhydride (PE-MAH) with covalently attached pyrene as a fluorophore, produced directly via reactive extrusion. Both HDPE-MAH and VLDPE-MAH were functionalized and their changes in fluorescence as a response to stress were investigated. The thermal, rheological and mechanical properties of both pyrene functionalized polymers were also tested in comparison with those of the pristine samples.

2. Experimental section

2.1. Materials

Chloroform (Sigma–Aldrich), Dichloromethane (DCM, Sigma–Aldrich), 1,2-dichlorobenzene (oDCB, Sigma–Aldrich), deuterated chloroform (CDCl_3 , Sigma–Aldrich), deuterated tetrachloroethene ($\text{TCE-}d_2$, Sigma–Aldrich), 1-aminopyrene (AP, 97%, Sigma–Aldrich) high density polyethylene-*graft*-maleic anhydride (HDPE-MAH) was kindly provided by SABIC, very low density polyethylene-*graft*-maleic anhydride (VLDPE-MAH) was purchased from YPAREX. All materials were used as received unless otherwise stated.

2.2. Typical procedure for pyrene reactive extrusion grafting onto polyethylene-*graft*-maleic anhydride (PE-MAH)

VLDPE-MAH (9.9 g) and AP (0.1 g, 0.46 mmol) were mixed in a metal cup and subsequently fed into the 15 mL corotating twin-crew micro extruder. The reaction mixture was processed for 10 min at 160 °C and a screw speed of 100 RPM after which the discharge valve was opened. The amount of grafted AP was determined from the weight ratio of the polymer and AP fed into the extruder. Extraction experiments proved that all AP has reacted (see below) and ^1H NMR showed the resonances of grafted pyrene at AP concentration of 1% (Figs. S1 and S2).

2.3. Typical procedure for PE-MAH-AP extraction with DCM

A piece of PE-MAH-AP polymer (around 200 mg) was placed in a vial and 5 mL of DCM was added. The vial was closed and left at RT for 24 h. Subsequently DCM was evaporated, the vial was washed with 0.5 mL of CDCl_3 and ^1H NMR of the solution was recorded to detect if any free AP was present (Figs. S3 and S4).

2.4. Measurements

^1H NMR analysis was carried out either at room temperature or at 120 °C using deuterated chloroform (CDCl_3) or deuterated tetrachloroethene ($\text{TCE-}d_2$) as solvents and recorded in 5 mm tubes on a Varian Mercury spectrometer operating at frequencies of 400 MHz. Chemical shifts are reported in ppm versus tetramethylsilane and were determined by reference to the residual solvent signal.

The molecular weight and polydispersity were studied by Size exclusion chromatography (SEC) measurements performed at 150 °C on a Polymer Char GPC-IR[®] built around an Agilent GC oven model 7890, equipped with an autosampler and the Integrated Detector IR4. oDCB was used as an eluent at a flow rate of 1 mL min⁻¹. The SEC data were processed using Calculations Software GPC One[®]. The molecular weights were calculated with respect to polyethylene standards.

Melting temperatures (T_m) and enthalpies of the transition (ΔH_m)

were measured by differential scanning calorimetry (DSC) using a DSC Q100 from TA Instruments. The measurements were carried out at a heating and cooling rate of 10 °C min⁻¹ from -20 °C to 150 °C. The transitions were deduced from the second heating curves (Figs. S5 and S6).

Fluorescence emission spectra were measured in the solid state (film thickness around 100 μm) on a Horiba Jobin Yvon Fluoromax-3 spectrofluorometer equipped with a xenon arc lamp as a light source using excitation wavelength of 320 nm.

Tensile tests were performed with a Zwick Z100 tensile tester equipped with a 100 N load cell. The tests were performed on injection molded tensile bars. The samples were pre-stressed to 0.3 MPa, then loaded with a constant cross-head speed of 50 mm min⁻¹ (Figs. S7 and S8).

Rheology was measured using TA Instruments DHR-2 equipped with a parallel plate geometry. Discs with diameter of 25 mm and thickness of 1 mm were injection molded at 160 °C. Frequency sweeps were measured from 100 to 0.1 rad s⁻¹ (strain amplitude of 0.4%) at a temperature of 160 °C (Figs. S9 and S10).

Dynamical mechanical thermal analysis (DMTA) was measured using TA Instruments Q800 in tensile mode. The specimens were compressed molded at 160 °C. Samples were measured from -100 to 200 °C with a heating speed of 3 °C min⁻¹ and a fixed oscillation (amplitude 10 micron, frequency 1 Hz, Figs. S11 and S12).

3. Results and discussion

3.1. AP reactive extrusion grafting onto PE-MAHs

1-Aminopyrene (AP) was used as fluorophore of choice thanks to pyrene's clear and well-separated monomer and excimer emissions in the solid state, long singlet monomer excited state fluorescence life time, high luminescence quantum yield as well as chemical and thermal robustness [20,21]. For the synthesis of pyrene functionalized PE, two types of polyethylene grafted with maleic anhydride were selected: highly crystalline, stiff HDPE-MAH and more elastic VLDPE-MAH with low crystallinity (Table 1). To graft AP onto PE-MAHs a reaction between amino group of AP and maleic anhydride forming a robust succinimide was used (Scheme 1) [22–25]. The reactions were performed at 160 °C in a corotating twin-screw micro extruder allowing to eliminate the use of solvents.

Increasing amount of AP was introduced ranging from 0.01 to 1% leaving always the excess of MAH to achieve complete grafting which was confirmed by extraction with DCM, as no free AP could be detected by ^1H NMR in the extracted solution (Figs. S3 and S4). The covalent attachment of AP to polyethylene backbone prevents leeching, ensuring a long-term stability [26–28]. Incorporation of AP within the studied range did not influence significantly thermal properties of PE-MAHs as indicated by DSC measurements (Figs. S5 and S6). AP content, melting temperatures, enthalpies of the transition and glass transition temperatures of the samples are listed in Table 1. The introduction of AP at 0.01 wt% decreases slightly the crystallinity of HDPE (around 10%) but remains roughly the same when more AP is introduced up to 1 wt%.

Tensile test, DMTA and rheology were employed to investigate the impact of AP on mechanical performance and flow behavior of PE-MAH-APs. Tensile and DMTA curves before and after introduction of 1% of AP were almost identical as well as rheology frequency sweeps, proving that mechanical properties and flow behavior remained unaltered (Figs. S7–S12).

3.2. PE-MAH-AP fluorescent characterization

To investigate the amount of excimer present in PE-MAHs containing different amount of AP, fluorescence emission spectra were recorded (Fig. 1). Samples containing low amount of pyrene (0.01–0.1 wt%) exhibit a low excimer formation ($I_E/I_M < 0.1$) and

Table 1

MAH and AP content, melting temperatures and enthalpies of the transition as well as molecular weights and the molecular weight distributions (PDI) of PE-MAHs and corresponding PE-MAH-APs.^a

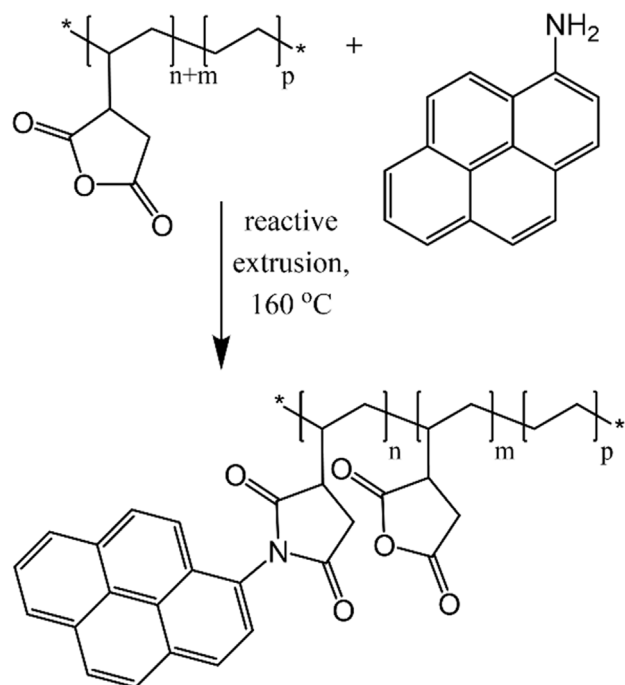
| Polymer | MAH ^b [mol%] | AP [wt%] | T_m ^c [°C] | ΔH_m ^c [°C] | M_n ^d [g mol ⁻¹] | PDI ^d |
|------------------|-------------------------|----------|-------------------------|--------------------------------|---|------------------|
| HDPE-MAH | 0.7 | – | 125.7 | 154.2 | 6000 | 3.7 |
| 1 (HDPE-MAH-AP) | – | 0.01 | 127.7 | 121.9 | – | – |
| 2 (HDPE-MAH-AP) | – | 0.1 | 128.2 | 120.8 | – | – |
| 3 (HDPE-MAH-AP) | – | 1 | 127.9 | 118.0 | – | – |
| VLDPE-MAH | 1.2 | – | 76.6 | 10.8 | 4500 | 5.3 |
| 4 (VLDPE-MAH-AP) | – | 0.01 | 73.4 | 11.8 | – | – |
| 5 (VLDPE-MAH-AP) | – | 0.1 | 73.6 | 10.7 | – | – |
| 6 (VLDPE-MAH-AP) | – | 1 | 73.6 | 9.7 | – | – |

^a Conditions: reactive extrusion grafting was carried in a 15 mL twin crew micro extruder for 10 min at 160 °C and a screw speed of 100 RPM.

^b MAH content was calculated from the ¹H NMR (120 °C, TCE-d₂).

^c Melting temperatures (T_m) and enthalpies of the transition (ΔH_m) were determined by DSC from the second heating scan.

^d Molecular weight and polydispersity were determined by SEC in oDCB at 150 °C with respect to polyethylene standards.



only when the pyrene content was increased to 1 wt% a pronounced broad peak around 465 nm was observed, confirming the excimer formation in the PE matrix. Interestingly, HDPE-MAH-AP materials contain more excimer than VLDPE-MAH-AP ones. Since maleic anhydride is excluded from the PE crystalline phase, AP grafted onto the maleic anhydride has to be located in the amorphous phase as well [29]. VLDPE contains higher amount of amorphous phase than HDPE therefore, AP is more concentrated in the amorphous region of HDPE-MAH-AP with respect to VLDPE-MAH-AP, leading to increased excimer formation.

Samples grafted with 1 wt% of AP, containing the highest amount of excimer, were selected to investigate the fluorescent behavior under stress. Specimens for testing were prepared by compression molding and mounted in a metal frame between clamps and subjected to stretching with 50 mm increments. After each stretching step the frame containing the sample was placed in the spectrofluorimeter and the emission spectrum was recorded. Stress induced plastic deformation broke the excimers which manifested itself in the decrease of the I_E/I_M ratio. Due to the different tensile properties of HDPE-MAH and VLDPE-MAH, a different fluorescent behavior under stress was observed. Polymer 3 (HDPE-MAH-AP 1%) showed necking after 50% strain and a

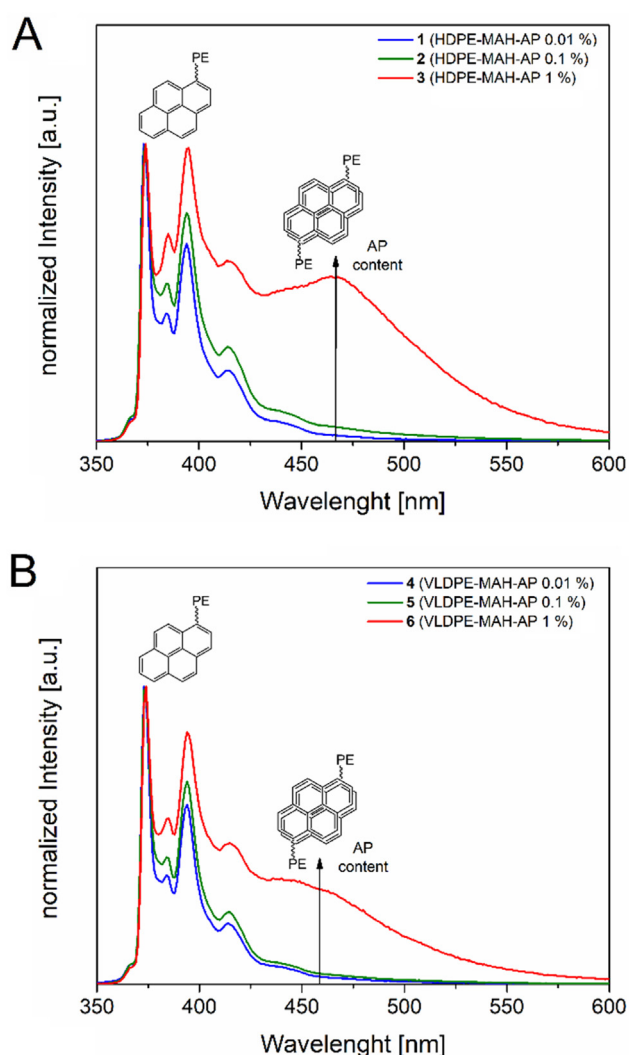


Fig. 1. Normalized fluorescence spectra of A: HDPE samples grafted with AP (1, 2 and 3) and B: VLDPE samples grafted with AP (4, 5 and 6).

clear drop of around 50% of I_E/I_M ratio was observed when the emission spectrum was collected from the neck area (Fig. 2A). Samples of polymer 3 failed when the strain exceeded 200%. In contrast, polymer 6 (VLDPE-MAH-AP 1%) showed no necking and a linear decrease of I_E/I_M ratio with elongation down to around 30% for 1100% strain was observed, which is the maximum elongation the device can allow (Fig. 2B). Interestingly, there is a linear relationship between the

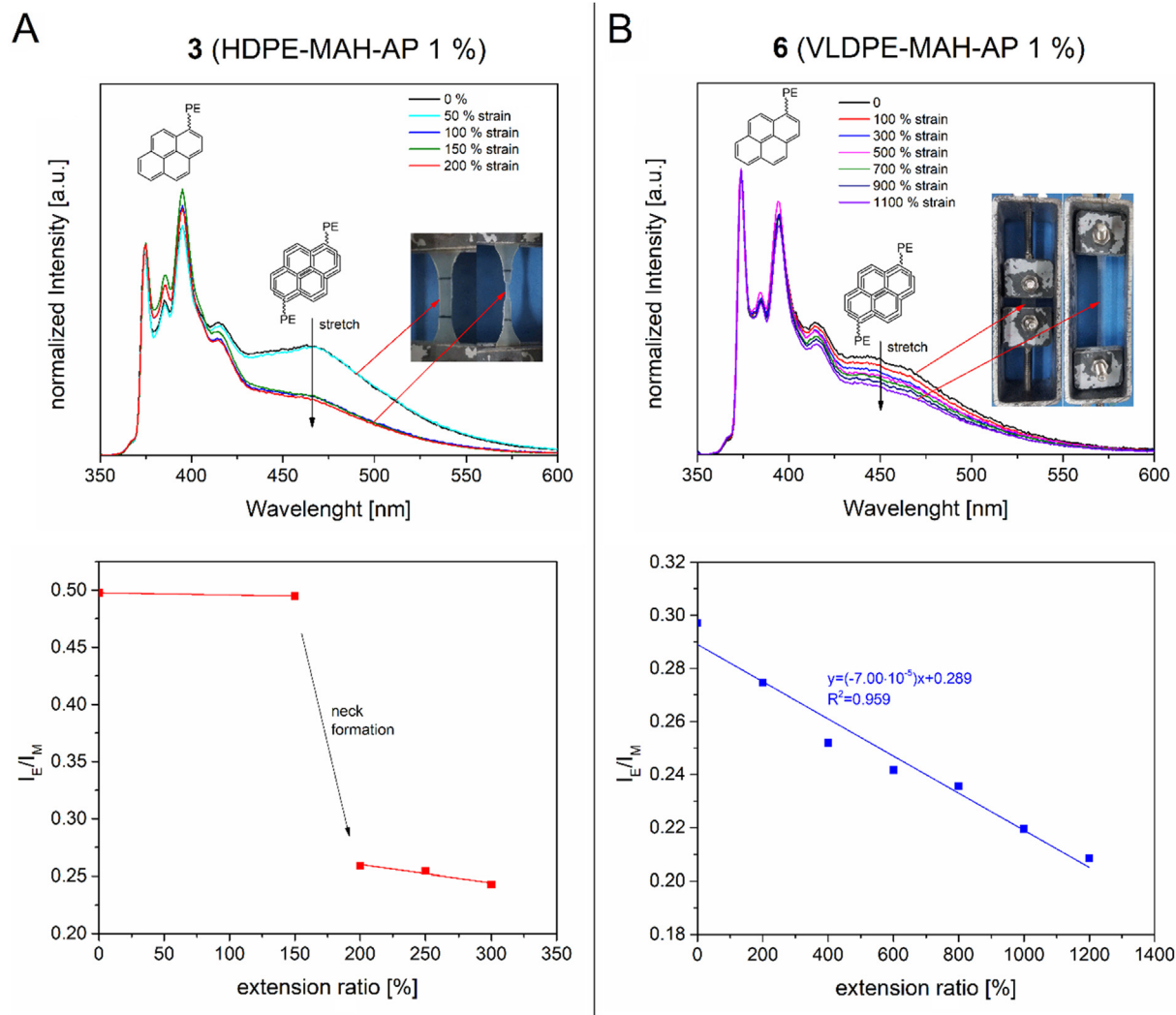


Fig. 2. Fluorescent behavior under stress of A: 3 (HDPE-MAH-AP 1%) and B: 6 (VLDPE-MAH-AP 1%).

extension ratio and excimer content, which can be used as a calibration line for the evaluation of strain in the material.

4. Conclusions

This work reports a simple and practical way to transform commercially available PE-MAH into a strain-reporting polymer. Reactive extrusion was successfully employed as an efficient and practical method to covalently functionalize PE-MAHs with increasing amounts of AP (from 0.01 to 1 wt%). The resulting strain-reporting PE retains similar mechanical, thermal and rheological properties to that of the starting PE-MAH materials. Both polymers **3** (HDPE-MAH-AP 1 wt%) and **6** (VLDPE-MAH-AP 1 wt%) showed pronounced fluorescence changes under stress which could be correlated to their different tensile properties. Around 50% drop of the excimer content (I_E/I_M) in polymer **3** was observed when the elongation exceeded 50% which could be related to a neck formation. In contrast, polymer **6** showed no necking with a linear decrease of the excimer content (I_E/I_M) with elongation up to 1100%. The results demonstrate a significant potential for developing strain-reporting PE directly via reactive extrusion. This solution is applicable not only for PE but also for any thermoplastic grafted with maleic anhydride units.

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Conflict of interest

The authors declare no conflict of interest.

Appendix A. Supplementary material

Supplementary data to this article can be found online at <https://doi.org/10.1016/j.eurpolymj.2018.12.016>.

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