

Research Article

Investigating the Degradability of HDPE, LDPE, PE-BIO, and PE-OXO Films under UV-B Radiation

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The changes in structural properties of high density polyethylene films (HDPE), low density polyethylene films (LDPE), biodegradable polyethylene (PE-BIO), and oxodegradable polyethylene (PE-OXO) films exposed to UV-B radiation were studied. The carbonyl (I_{CO}) and vinyl (I_V) index, the crystalline phase fraction, and the dichroic ratio were used to evaluate the photooxidation of these polymers. The results obtained show that LDPE and HDPE undergo a major degree of oxidation and an increase in the crystalline phase fraction comparing to PE-BIO and PE-OXO. If the LDPE and HDPE are pretreated by an accurate radiation UV-B dosage before its different commercial uses or in its final disposition, they can become an option for biodegradable material without the necessity of adding organic agents or photosensitizers.

1. Introduction

The widespread use of polyethylene PE as packaging films is due to its mechanical properties, enabling the production of mechanically strong films [1]. These plastic films accumulate in the environment due to their low degradability, generating pollution and accumulating in landfills. Moreover, because the films have very small masses and are usually contaminated, recycling is economically unfeasible [2].

Polyethylene has a simple and semicrystalline molecular structure, high molecular weight but does not easily degrade in the natural environment because it does not absorb UV radiation [3, 4]; however, the presence of impurities like catalyst remnants, plasticizers, and antioxidants can induce the PE to absorb UV-B radiation [5–8]. These properties that make plastics the material of choice for many applications, particularly for single use packaging, together with an increase in usage have also created disposal problems at the end of the useful life of these products [9].

In recent years, due to the low rate of degradability of PE, the need for degradable PE has become a major topic of research, and hence organic molecules and prooxidants agents have been included in the molecular structure of

polyethylene, in order to obtain biodegradable (PE-BIO) and oxodegradable (PE-OXO) materials, respectively [9–12].

Degradation of polymers is mainly due to photooxidation and thermooxidation reactions [6, 7, 13, 14], causing the chain scission and cross-linking of polymer backbone, the formation of carbonyls (C=O) and vinyl ($\text{CH}_2=\text{CH}$) groups, and, finally, changes in the conformation and crystallinity of the polymer [15–17]. PE is a low degradability polymer because it is mainly composed of C–C and C–H bonds (σ bonds) whose bond energy is in the order of 300–600 kJ/mol.

The principle of degradation states that the amount of energy absorbed by a molecule must exceed the bond energy in order to cause degradation. So, UV-A radiation energy is not enough to break the chemical bonds of the PE and cause the degradation of the polymer [18]. UV-B radiation is particularly efficient in bringing about photodamage in synthetic polymers.

Although the UV-C has sufficient energy to break σ bonds, it is not a commonly used technique in the degradation of plastics because plastics materials lost their mechanical properties; furthermore, the UV-C radiation emitted by the sun is absorbed by the atmosphere and does not reach the earth's surface [2].

IR spectroscopy is a useful technique for determining changes in the crystallinity degree of various polymers [19]. The IR spectrum of semicrystalline PE presents a bifurcated band at 710–750 cm^{-1} , rocking vibration of the methylene group (CH_2) from the amorphous and crystalline phase, as well as a bifurcated band at 1450–1480 cm^{-1} which is due to the wagging vibration of the methylene (CH_2) group of the crystalline phase [20, 21].

The orientation and conformation of the molecular chains influence the mechanical properties of the polymers. The specificity of infrared absorption bands to particular chemical functional groups makes infrared spectroscopy especially attractive for measurement of chain orientation. For a given absorption band, the dichroic ratio is the parameter commonly used to characterize the degree of optical anisotropy in stretched polymers. Dichroic ratio measurements require two different absorbance spectra of the sample, parallel (A_{\parallel}) and perpendicular (A_{\perp}) to the polymer stretching direction. The dichroic ratio is defined by $R = A_{\parallel}/A_{\perp}$ [21–23].

The photodegradation of PE, such as the formation of oxidation products, the loss of molecular weight, and the crystallinity, has been characterized by other authors using technics such as differential scanning calorimeter (DSC), electronic microscopy (SEM), X-ray diffraction (XRD), and infrared spectroscopy (FTIR) [5, 24–27]. In this paper, the effect of UV-B radiation in unmodified, LDPE and HDPE, and modified, PE-BIO and PE-OXO, PE films was evaluated by changes in carbonyl index, vinyl index, the crystalline phase fraction, and dichroic ratio.

2. Experimental

2.1. Material. LDPE, HDPE, PE-BIO, and PE-OXO bags samples were obtained in supermarkets from Aguascalientes, Mexico. These bags were made in Mexico and are commercialized in Latin America [28]. These bags were obtained from different suppliers in order to gather the most representative ones on the market.

128 samples, 40 × 50 mm with a thickness of 0.05 mm, of low density polyethylene (LDPE), high density polyethylene (HDPE), biodegradable polyethylene (PE-BIO), and oxodegradable polyethylene (PE-OXO), were analyzed.

2.2. Photodegradation of PE. PE films were irradiated with $3.5 \times 10^{-4} \text{ W/cm}^2$ UV-B lamps (280 and 320 nm). Samples were mounted on racks at 5 cm from the lamp. Samples were analyzed by various techniques at regular time intervals (0, 5, 15, 30, 45, and 60 days).

2.3. Evaluation of Photodegradation. The photodegradation of the PE was characterized by IR spectroscopy with Attenuated Total Reflectance (ATR), using a spectrophotometer *Thermo Nicolet* model iS10 with a Germanium mirror (Ge), and a resolution of 2 cm^{-1} in the range of 650–4000 cm^{-1} .

2.3.1. Carbonyl Index. Carbonyl index (I_{CO}) was calculated using (1), the IR absorption band at 1740 cm^{-1} ($A_{(1740)}$),

and stretching vibration of the carbonyl group (C=O); the absorbance at 1835 cm^{-1} ($A_{(1835)}$) is used as reference [27]:

$$I_{\text{CO}} = \frac{(A_{(1740)} - A_{(1835)})}{(0.008 * t)}, \quad (1)$$

where t is the thickness of the sample (mm).

2.3.2. Vinyl Index. Vinyl index (I_V) was calculated using (2), ratio of absorbance of the band on 909 cm^{-1} ($A_{(909)}$), stretching vibration of the vinyl group ($\text{CH}_2=\text{CH}$), and absorbance at 2020 cm^{-1} [27]:

$$I_V = \frac{(A_{(909)})}{(A_{(2020)})}. \quad (2)$$

2.3.3. Crystalline Phase Fraction. The PE crystalline phase fraction (α) was calculated from the integrated intensities of IR absorption bands of the methylene (CH_2) at 717 cm^{-1} ($I_{\text{int}(717)}$), amorphous phase fraction, and crystalline phase fraction, 730 cm^{-1} ($I_{\text{int}(730)}$), according to (3); the integrated intensities of the IR bands were calculated using a Gaussian fitting of these bands [19, 29]:

$$\alpha_{\text{phase}(i)} = \frac{\text{Integrated area}_{\text{phase}(i)}}{\text{Integrated area}_{\text{all phases}}}. \quad (3)$$

2.3.4. Dichroic Ratio. The dichroic ratio of the PE exposed to UV-B radiation was calculated by using the IR absorbance at 1460 cm^{-1} corresponding to the wagging vibration of the methylene group (CH_2) of the PE crystalline phase [23].

3. Results and Discussion

3.1. Infrared Spectroscopy. The infrared spectra of LDPE, HDPE, PE-BIO, and PE-OXO without exposure to UV-B radiation are shown in Figure 1; these spectra show the IR bands characteristics of PE: the stretching vibration of Carbon-Hydrogen (CH) group of the main chain at 2772–3038 cm^{-1} and the wagging and rocking vibration of methylene (CH_2) at 1440–1490 cm^{-1} and 700–750 cm^{-1} , respectively [20, 30]. IR spectra of HDPE and PE-BIO show a single peak at 700–750 cm^{-1} and at 1440–1490 cm^{-1} because they are polymers of linear structure, while for LDPE and PE-OXO, these same bands are bifurcated because they are branched polymers.

The IR spectra of the PE-BIO films show an IR absorption at 1740 cm^{-1} , stretching vibration of the carbonyl group (C=O), whereas the IR spectrum of the PE-OXO films shows an IR absorption band at 909 cm^{-1} , stretching vibration of the vinyl group ($\text{CH}_2=\text{CH}$); these functional groups are chromophores groups and are considered as precursors of the photochemical reactions of PE [2].

3.1.1. Carbonyl Index. The photooxidation of the PE films exposed to UV-B radiation is developed according to

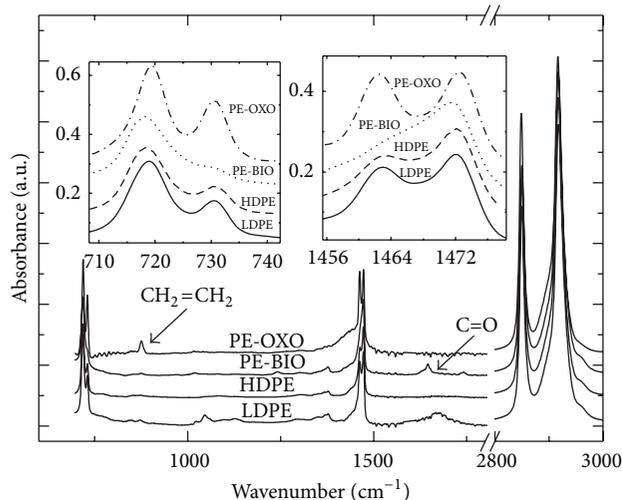


FIGURE 1: IR spectrum of the LDPE, HDPE, PE-BIO, and PE-OXO films without exposure to UV-B radiation.

the reaction mechanism Norrish Type II [31, 32], considered as a homolytic reaction consisting in four steps: initiation, propagation, chain branching, and termination [33]. The initiation step is predominantly caused by the hydroperoxides formed during polymerization reactions of PE, which decomposes by the effect of UV-B radiation, causing the formation of functional groups such as hydroxyls, anhydrides, and carbonyls. When the dose of UV-B radiation increases, reactive chemical groups like tertiary carbons react and form the vinyl groups [18, 34]. A PE with greater amount of tertiary carbons on its molecular structure presents a greater degree of photooxidation, because the degree of branching controls the amount of more labile hydrogen attached to the tertiary carbon atoms [18]. When increasing the dose of UV-B radiation on PE films, the carbonyl and vinyl groups can be studied as an indicative of polymer backbone scission [35]. The carbonyl and vinyl groups are considered the main photooxidation products of the PE [8]; therefore, carbonyl index (I_{CO}) and vinyl index (I_V) are parameters used to evaluate the effect of UV-B radiation in PE films [33, 36].

I_{CO} of LDPE and HDPE films presents a similar behavior: it increases from the fifth day of exposure to UV-B radiation, indicating that after this time the photooxidation of these polymers will start; from 5 to 30 days of exposure to UV-B radiation, I_{CO} increases due to photooxidation of stabilizers and plasticizers present in LDPE and HDPE [26]; from 30 to 60 days of exposure to UV-B radiation, the increase of I_{CO} indicates a greater polymer backbone scission. Because the LDPE is a branched material, it has a greater number of tertiary carbon, which makes it more susceptible to undergo photooxidation reactions; so, I_{CO} of LDPE is greater than the other three types of PE; see Figure 2.

The IR spectrum of the PE-BIO films without exposure to UV-B radiation presents the characteristic IR band of the carbonyl group (1640 cm^{-1}), indicating that the PE-BIO material is oxidized during the manufacturing process. The concentration of C=O decreases during the first 5 days of

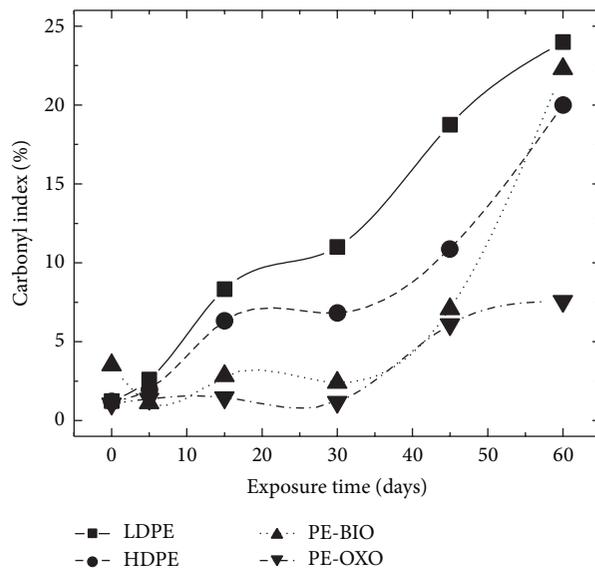


FIGURE 2: Carbonyl index (I_{CO}) of the LDPE (■), HDPE (●), PE-BIO (▲), and PE-OXO (▼) films exposed to UV-B radiation.

exposure to UV-B radiation; the band intensity decreases, because this functional group is susceptible to undergoing photooxidation reactions by UV-B radiation effects, so the carbonyl index decreases; see Figure 2 [18]. From 5 to 30 days of exposure to UV-B radiation, I_{CO} of the PE-BIO remains relatively constant due to the presence of the carbonyl group since the start of exposure to UV-B radiation slows down the photooxidation of PE-BIO. From 30 to 60 days, I_{CO} increases exponentially due to photooxidation of the stabilizers and plasticizers present in the polymer.

PE-OXO does not undergo photooxidation reactions during the first 30 days of exposure to UV-B, due to the content of prooxidant agents, transition metals, which cause inhibition of the hydroperoxides decomposition under the effect of UV-B radiation [18], inducing a slowdown in the photooxidation of the polymer, that is, the formation of the carbonyl group. After 30 days of exposure to UV-B radiation, the slowing down effect of prooxidants tends to decrease causing a slight increase in I_{CO} of this polymeric material.

3.1.2. Vinyl Index. In Figure 3, the vinyl index (I_V) of LDPE, HDPE, PE-BIO, and PE-OXO is shown. From 0 to 60 days of exposure to UV-B radiation, I_V of LDPE, HDPE, and PE-BIO tends to increase with an almost linear tendency; this is due to embrittlement process and the breaking of the bonds of the tertiary carbons of the branches of the polymer backbone, which give rise to the formation of the vinyl group [33].

I_V of PE-OXO does not increase during the whole exposure time to UV-B radiation, due to the prooxidant agents, transition metal, which when participating in a homolytic reaction does not allow embrittlement process of the material, just after photooxidation [2, 37].

3.2. Crystalline Phase Fraction. The polymers are semicrystalline solids with amorphous and crystalline zones in

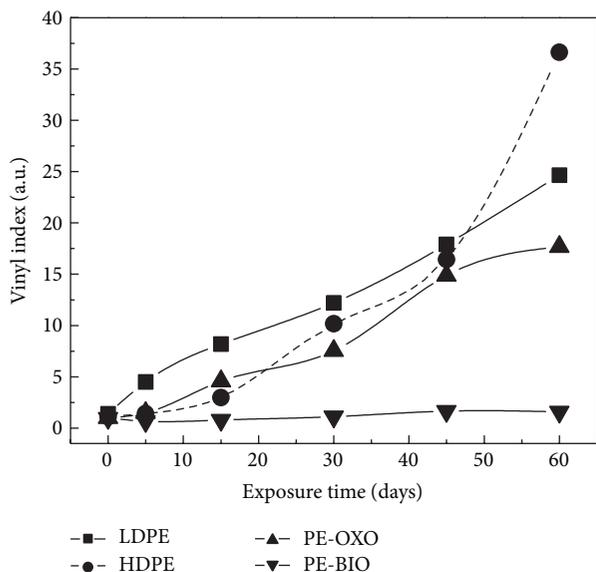


FIGURE 3: Vinyl index (I_V) of the LDPE (■), HDPE (▲), PE-BIO (▼), and PE-OXO (●) films exposed to UV-B radiation.

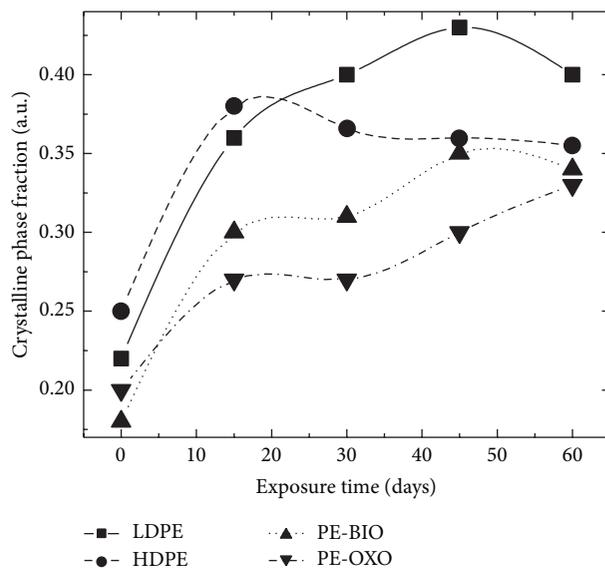


FIGURE 4: Crystalline phase fraction of LDPE (■), HDPE (▲), PE-BIO (▼), and PE-OXO (●) films exposed to UV-B radiation.

the polymer matrix. The degree of crystallinity depends on the polymerization process. The crystalline phase fraction of LDPE film increases from 0 to 45 days of exposure to UV-B radiation, due to the formation of new crystalline zones in the material as a result of breakdown branches of the polymer backbone and the bending on themselves [38]; however, after 45 days of exposure to UV-B radiation, the crystalline phase fraction decreases due to decrystallization process, that is, the formation of smaller crystals and subsequent rearrangement of the crystalline zones.

The HDPE is almost a linear polymer with a higher degree of crystallinity, so its crystalline phase fraction has a different behavior than the LDPE, PE-BIO, and PE-OXO. From 0 to 15 days of exposure to UV-B radiation, the HDPE starts its embrittlement process, forming new crystalline regions and causing the increasing of crystalline phase fraction; from 15 to 60 days of exposure to UV-B radiation, the crystalline phase fraction decreases, because the increase of the dose of UV-B radiation in the material causes the decrystallization process [39]. The crystalline phase fraction of PE-BIO and PE-OXO shows a constant increase during the experiment, slowing down only from 15 to 30 days of exposure to UV-B radiation. This behavior could be due to the presence of carbonyl groups and prooxidant agents in the polymer molecules, respectively.

3.3. Dichroic Ratio. Dichroic ratio of LDPE, HDPE, PE-BIO and PE-OXO exposed to UV-B is shown in Figure 5. The increase of the dichroic ratio for LDPE and HDPE films is due to the embrittlement process since the breakdown branches was oriented parallel to the polymer backbones; these molecular chains were oriented in the same direction of lamination of the films. LDPE dichroic ratio is greater than that of HDPE, due to the fact that it is a branched polymer and the breakdown branches from the polymer backbone are orientated parallel to the stretch direction of the films, causing

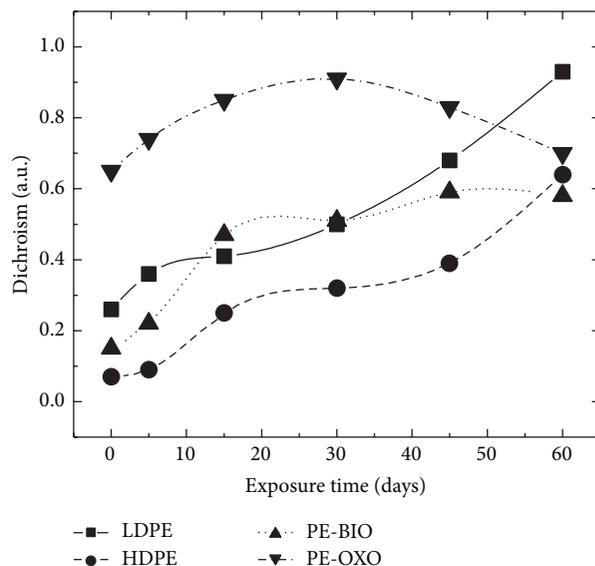


FIGURE 5: Dichroic ratio of the LDPE (■), HDPE (▲), PE-BIO (▼), and PE-OXO (●) films exposed to UV-B radiation.

a higher degree of ordering and therefore an increase in the crystalline phase fraction of the material; see Figure 4 [40].

The PE-BIO dichroic ratio has its greatest increase during the first 30 days of exposure to UV-B radiation, due to the breakdown branches of the polymer backbone and their reordering parallel to the stretch direction of the polymer; from 30 to 60 days of exposure to UV-B radiation, the dichroic ratio remains constant because there are no more branches neither breakdown nor reordering.

From 0 to 30 days of exposure to UV-B radiation, the dichroic ratio for PE-OXO is higher than LDPE, HDPE, and

PE-BIO, because the presence of prooxidants agents generally leads to the formation of functional macromolecules, reordered parallel to the stretch direction of the films [39]; dichroic ratio decreases from 30 to 60 days of exposure to UV-B radiation; this could be due to the presence of the prooxidants agents in the molecule of the material, causing the functional macromolecules to be ordered in different directions to the stretch direction of the polymer chains of PE-OXO.

4. Conclusion

In this paper, it has been demonstrated that PE of modified structure, PE-BIO and PE-OXO, which include organic and prooxidants agents in the molecule, respectively, in order to increase their biodegradability, show a lower degree of photooxidation than PE of unmodified structure, LDPE and HDPE, when exposed to the same conditions of exposure to UV-B radiation.

Until 45 days of exposure to UV-B radiation, both I_{CO} and I_V of PE-BIO and PE-OXO are lower than those of LDPE and HDPE; from 45 to 60 days of exposure, I_{CO} of PE-BIO tends to be the same as those of LDPE and HDPE. The increase in these indices implies the increased biodegradability of these polymeric materials.

The crystalline phase fraction of LDPE and HDPE increases further to 30 days of exposure to UV-B radiation, thereby affecting their physical properties such as stiffness, dissolution resistance, and dimensional stability. Additionally, during the first 30 days of exposure to UV-B radiation, the dichroic ratio increases for all types of polyethylene, thereby indicating that branches scission from polymer backbone is reordered parallel to the stretch direction of the films. So, we conclude that UV-B radiation induces the increase of crystallinity and the orientation of the chains, which in turn have a crucial influence on the physical properties of the polyethylene.

The results obtained in this work show that the LDPE and HDPE treated with UV-B radiation have a higher degree of oxidation, a major increase in the crystalline phase fraction, and a higher molecular orientation than the PE-BIO and PE-OXO, treated under the same conditions of exposure to UV-B radiation. Therefore, the outcome of this study is that LDPE and HDPE pretreated with the correct dose of UV-B radiation, before its commercial uses or after its final disposition, may be an option of biodegradable material without the need to add organic or prooxidant agents. This can be implemented as a new technology related to plastic manufacturers applied in LDPE and HDPE polymers to take advantage of the environ-ecological aspects, reduction and reinstatement of polymers as innocuous material to the environment.

The final dispositions of PE-BIO and PE-OXO films must be done according to plastic manufacturers recommendations in order for these polymers to degrade, which otherwise would not suffer any degradation and would be accumulated in landfills.

Conflict of Interests

The authors declare that there is no conflict of interests regarding the publication of this paper.

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