

Influence of Natural and Accelerated Weathering on Performances of Photoselective Greenhouse Films

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ABSTRACT: The research aims to investigate the effects of natural and accelerated weathering on polyethylene-based films. At this regard, monolayer films of low density/linear low density polyethylene blends, containing commercially available organic pigments and an UV absorber of the benzophenone type, have been considered. The samples were weathered on field (natural weathering) or using two different artificial procedures: UV lamp and QUV chamber. Conditioned film samples were, then, analyzed by performing several physical tests taking as-received films as a reference. Rheological measurements showed an increase in viscosity of weathered sample melts as a consequence of photodegradation phenomena, inducing the formation of double bonds and crosslinks. This latter result was also confirmed by gel content measurements. UV-visible spectroscopic tests indicated that in both cases of natural and artificial weathering an increase of the transmittance of films occurred. Tensile tests indicated the increase of films stiffness, especially in case of samples conditioned using the UV lamp, and a large decrease of the strain at break, both in machine and in transverse directions, especially for film weathered using the QUV chamber. © 2012 Wiley Periodicals, Inc. *J. Appl. Polym. Sci.* 000: 000–000, 2012

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INTRODUCTION

In the last decades, the increased diffusion of protect cultivations has significantly contributed to increase the amount of plastics used for agriculture. In particular, it is well established that the right selection of the cover material can enhance crop productivity, advance harvest time, and save energy.

The most important factor affecting horticultural production is the natural light as it provides energy for photosynthesis, the process by which plants produce carbohydrates and oxygen from carbon dioxide and water. At this regard, the part of the global radiation spectra between 400 and 700 nm, necessary for the occurrence of this vital process, is called photosynthetically active radiation (PAR).¹ To make the most of this portion of the solar spectrum, a considerable effort has been focused on the development of transparent greenhouse covering material with improved optical properties.^{2–4}

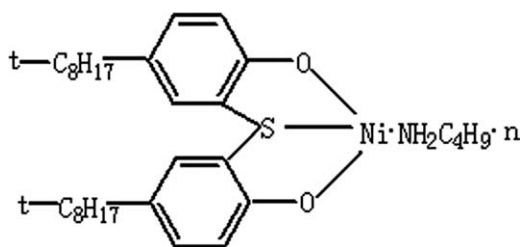
Moreover, considering that the exposure to intensive sun irradiation, combined with many factors such as humidity, air pollutants, chemicals, and so on is usually responsible for rapid deg-

radation effects on plastic coverings, a specific research interest has been paid to understand the complex mechanisms involved^{5–8} and, consequently, to improve their durability in outdoor applications.⁹

In this framework, the main objective of the researchers is to increase PAR transmission of films and simultaneously improve the light spectrum meeting the needs of plants or protected cultivations. At this regard, even if glass still appears to be one of the most interesting materials for coverings in Northern Europe, plastic films, containing fluorescent pigments seem to have potential and offer valuable perspectives.^{10,11}

For this purpose, many products have been developed by now both at laboratory and industrial levels and, undoubtedly, thin low-density polyethylene (LDPE) films are the most widely used for greenhouse coverings. The widespread diffusion of these latter is essentially due to the relatively good mechanical and optical properties of this material as well as its competitive market price.

However, it has well known that thin LDPE films used as greenhouse covering material are subject to numerous environmental



Scheme 1. Chemical structure of yellow pigment.

factors responsible for chemical and/or physical degradation processes. As extensively witnessed by the literature,^{12,13} the degradation of LDPE films involves complex photodegradation processes catalyzed by UV radiations, chemical degradation through reactions with air pollutants and agrochemicals, and finally mechanical degradation phenomena by bond rupture under the influence of mechanical stress.¹⁴

As an example, tests have confirmed that agrochemicals decrease the aging resistance of covering polyethylene films,^{15,16} especially under the concomitant action of oxygen and UV rays.

To improve the durability of plastic covering films, specific additives are typically included during their production, usually performed by blown extrusion technique. These additives include UV absorber and hindered amine light stabilizer to protect the film against UV degradation,^{17–19} surface-active additives to prevent coalescence of condensed small water droplets into larger ones able to drip on plant causing their disease,^{20–22} chalk as light diffuser²³ and others.

To increase knowledge about the complex mechanisms of degradation that inevitably occur during the life cycle of greenhouse films, many researchers have tried to reproduce the natural weathering (NW) by the development of appropriate procedures for accelerated weathering.

Accelerated tests are usually performed by exposing the materials to environments more aggressive than those generally encountered in practice, that is, very high temperatures, prolonged immersion periods and higher loads. In standardized procedures, proposed to achieve accelerated weathering, one or more weather-like conditions are intensified to levels greater than those occurring naturally. As a consequence, accelerated tests are reliable only if a precise correlation exists between the data obtained from accelerated weathering and the performances of the materials exposed for long time to environment. At the present time, the results of the accelerated weathering, even if carried out in accordance with the current standards (ISO, ASTM), are mainly regarded as qualitative indicators and used as a reference limit value.

In this work, accelerated weathering tests were performed on different polyethylene greenhouse film samples, assuming that samples characterized by lower resistance to accelerated weathering will show lower resistance to NW. The physical properties (transparency, viscosity, and mechanical properties) of the films artificially weathered were compared with those measured on as-received samples of the same films. Finally, some films, simi-

lar to those analyzed in the study and exposed for 2 years on fields, were characterized to detect if the accelerated weathering procedure used can be representative to the true conditions encountered by the greenhouse films on field.

MATERIALS AND METHODS

Film Samples and Weathering Procedures

Three different UV-stabilized polyethylene greenhouse films, supplied by Plastik S.p.A., were analyzed: pink, yellow (mark Orolene), and white (unpigmented) films. The thickness of the films was $170 \pm 3 \mu\text{m}$.

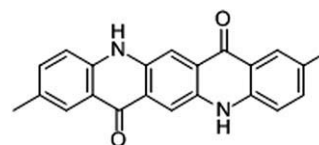
Colored film sample contain 5% by weight of a commercial pigment provided, in both cases, by Clariant as master-batch having, in turn, a content of active pigment equal to 15% by weight.

In more details, the yellow pigment is a nickel salt having the chemical structure reported in Scheme 1. Fourier Transform Infrared Spectroscopy (FTIR) analysis revealed that the pigment is characterized by absorbance peaks at 3317, 3214, 3134, 3049, 2953, 2901, 2872, 1595, 1475, 1394, 1363, 1313, 1277, 1238, 1168, 1146, 1097, 1058, 977, 922.5, 875, 855, and 824 cm^{-1} .

The pink pigment, commercialized under the trade name Hostaperm Pink E02, belongs to the family of quinacridones, whose base chemical structure is reported in Scheme 2. In this case, the commercial product is a master batch containing 15% by weight of the pigment.

Referring to the FTIR spectrum of quinacridone, it is characterized by absorbance peaks at 3267, 3228, 3165, 2980, 2915, 1635, 1604, 1578, 1554, 1502, 1478, 1413, 1339, 1297, 1260, 1234, 1201, 1143, 1120, 915, 873, 808, 792, 771, 710, 701, 612, 556, 537, and 418 cm^{-1} .

The pigmented greenhouse films naturally exposed for 2 years in an area of Apulia region (Italy) were compared with similar films weathered in an environmental chamber “UV weathering” QUV (Q-Lab), in agreement with the UNI EN ISO 4892.²⁴ The UV weathering chamber is equipped with a UV-B type lamp, with a power of 0.76 W/cm^2 . Each accelerated weathering cycle provided 8 h of irradiation at 60°C followed by 4 h of condensation at 50°C , in agreement with the standard, parts 1, 2, and 3. The total time of exposure was set up to 2100 h, equivalent to 2 years of natural exposure, as reported by the standard. Therefore, the total time of the tests, including exposure and condensation, was set at 3300 h. Samples were also extracted from the QUV chamber after 1100 h of test (corresponding to 733 h of exposure) to be characterized at an intermediate time. For comparison purposes, the unpigmented (white) films were



Scheme 2. Chemical structure of pink pigment.

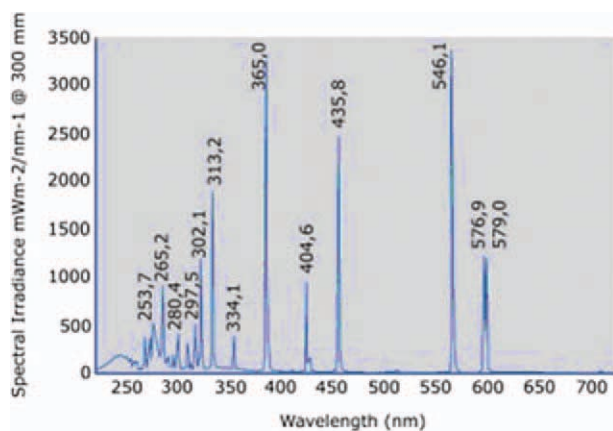


Figure 1. Emission spectrum of UV mercury lamp used. [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

weathered in the UV weathering chamber under the same conditions.

In addition, for the yellow film, another weathering procedure was considered by the film producer: it was exposed to a mercury UV lamp with an emission spectrum reported in Figure 1. The conditioning of film was performed at 40°C for 48, 52, 54, and 56 h.

Characterization Techniques

The degradation of the different films, occurred as a consequence of natural or accelerated weathering, was analyzed by performing several physical tests on the weathered and, for comparison purposes, on as-received films.

Rheological measurements were performed on the weathered and as-received films as a means to quantify the degradation occurred in the materials. To this aim, a strain controlled rheometer (Ares, Rheometric Scientific) was used, using a parallel plate geometry (radius of the plate 12.5 mm) and a gap of 0.2 mm, in steady state mode. The tests were conducted at 180°C, a temperature well above the melting point of the polyethylene, in a shear rate range between 0.01 and 10 s⁻¹. Each result reported is obtained as the average of at least three experiments.

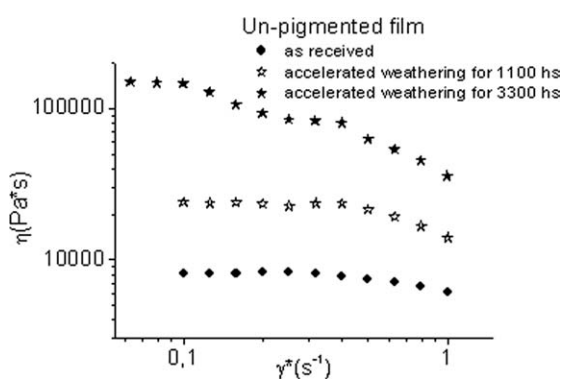


Figure 2. Flow curves of unpigmented films. Comparison among not-exposed and QUV weathered samples for 1100 and 3300 h, respectively.

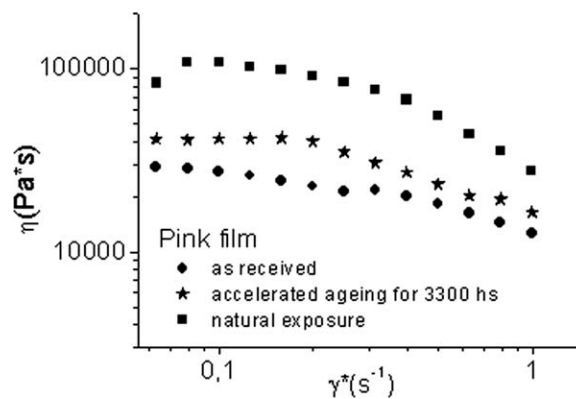


Figure 3. Flow curves of pink films. Comparison among not-exposed, QUV weathered samples for 3300 h and natural weathered samples.

The gel content of yellow and pink films was performed by extraction in hot xylene at 120°C for 48 h. The tests were performed on as-received and artificially weathered films. FTIR spectra on the same films were also collected between 2000 and 1000 cm⁻¹, using a FTIR (mod. 6300, Jasco).

Ultraviolet–visible spectroscopy was performed on the films in the range of wavelength 200–1200 nm, using a Varian Cary 500 UV-Vis Near InfraRed (NIR) spectrophotometer. The instrument measures the transmittance of the films during the test, that is, the amount of radiation transmitted by each film as a function of the wavelength of the radiation itself.

Finally, tensile properties were determined using an Instron mechanical testing machine (model 3360), according to American Standard Test Method (ASTM) test method D638,²⁵ on the weathered and as-received yellow film. Specimens were cut in the machine direction (MD) and transverse direction (TD) with respect to the direction of extrusion and were tested at two strain rates: for modulus measurement purposes, the speed was 1 mm/min up to a deformation of 10%; then, the speed increased to 150 mm/min until break. For each specimen, the strain gauge length was limited by markers to highlight any slipping that may occur at high tensile stresses, whereas to account for slight prestraining of the film sample during the tightening of grips, a manual reset of the tensile load has been always done before starting tests.

Tensile properties (elastic modulus, maximum strength, and elongation at break) were calculated, averaging the results of at least five tests per kind of film.

RESULTS AND DISCUSSION

Referring first to the rheological analysis performed on as received (reference) and QUV weathered film samples, a fairly Newtonian behavior at lower values of shear rates (<0.1–0.2 s⁻¹) was always observed. Conversely, at higher shear rates, a pseudoplastic behavior has been detected.

It is generally observed that accelerated weathering treatments produced an increase in viscosity of melts, irrespective of the presence of pigments.^{26,27} This increase in viscosity has been explained by the occurrence of structural changes as a consequence of degradation. Degradative phenomenon, in fact, can

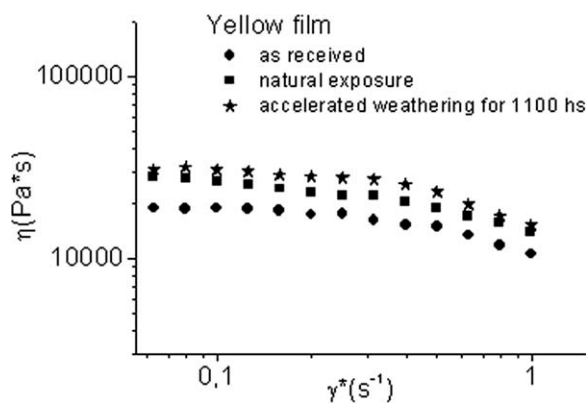


Figure 4. Flow curves of yellow films. Comparison among not-exposed, QUV weathered samples for 1100 h and natural weathered samples.

produce the formation of double bonds and some crosslinking in the polymer, which in turn, can lead to an increase in viscosity. This was confirmed by the rheological measurement performed on the unpigmented film: the longer the accelerated weathering time used, the higher the increase in viscosity, as shown in Figure 2.

Referring to pink films, similar results were found when the films were exposed to accelerate weathering in the UV weathering chamber, as shown in Figure 3. The increase in viscosity as a consequence of accelerate weathering was in this case of a lower entity. It must be underlined, however, that the pink film collected after the weathering performed in the UV weathering chamber was mostly damaged, with diffused cracks and fractures. This suggests that a higher degree of degradation occurred in the pink film, with a predominance of chain scission on the polymeric structure and, in turn, a decrease of the molecular weight and the viscosity of the polymer. When the same film is externally exposed on field, conversely, a certain increase in melt viscosity is observed, indicating in such exposure regime the preponderance of the formation of double bonds and crosslinking. The sample of pink films externally exposed, moreover, were found substantially undamaged. The comparison of the results found revealed that the accelerated weathering performed on the pink films was unable to reproduce what truly happened in real field conditions, the latter being much less harsh than the former.

The rheological results found on the yellow films, QUV weathered or as received, are shown in Figure 4. The increase in viscosity found as consequence of both the weathering regimes, that is, accelerated or natural, suggests that the degradation took place in both cases with the predominant formation of double bonds and crosslinking in the polymeric structure.

The effects on the viscosity of an accelerated weathering treatment, performed on the yellow film for a time even lower than that indicated in the standard to reproduce a 2 years of natural exposure, were slightly more severe than those produced by a true outdoor exposure for 2 years. Degradative phenomena in this case produced mostly the formation of double bonds and crosslinking in the polymer, with a consequent increase in vis-

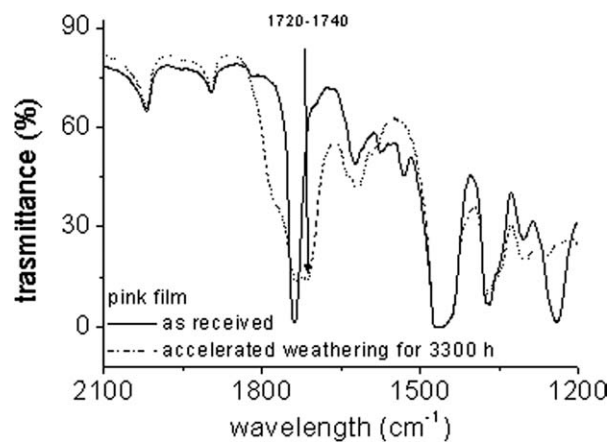


Figure 5. FTIR spectra of pink film before and after artificial weathering procedure, respectively.

cosity. However, all the yellow film samples exposed to accelerated or NW were found undamaged.

Gel content measurements were performed on yellow and pink films to confirm the hypothesis stated for rheological results, that is, crosslinking formation as a result of degradative phenomena. Gel content values equal to 0.024% and 0.02% were found on the artificially weathered yellow and pink films, respectively. The as-received films, conversely, did not show any residual after the extraction procedure in xylene, proving the absence of any crosslink. The formation of crosslinking, in fact, is typical of the degradation of polyethylene films.

Further, FTIR spectra of the artificially weathered films were produced. Results are reported in Figure 5 for the pink film and Figure 6 for the yellow one. For both pigmented films, the appearance of a medium intensity peak at 1720–1740 cm^{-1} is indicative of the formation of carbonyl groups, which normally appear during thermo-oxidative degradation of polyethylene.²⁸ In conclusion, the results obtained from FTIR analysis and gel content indicate that both crosslinking and chain-scission occur during artificial weathering. Crosslinking is normally associated with a deficiency of oxygen at the reaction site, which leads to the formation of relatively stable peroxidic crosslinks (possibly

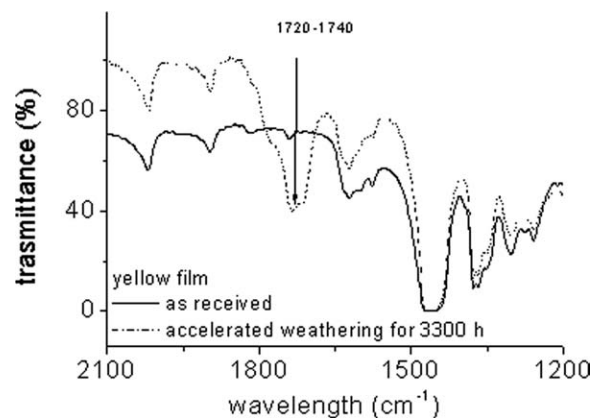


Figure 6. FTIR spectra of yellow film before and after artificial weathering procedure, respectively.

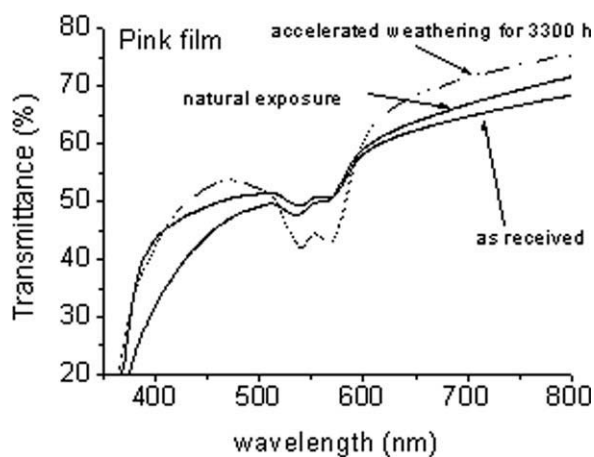


Figure 7. Influence of weathering on transmittance UV spectra of pink films.

even carbon–carbon crosslinks). Conversely, in the presence of highly redox-active transition metal ions, as those present in the yellow pigment, the metal ion rapidly converts hydroperoxides to alkoxy radicals.²⁹ Consequently, the absorbance peak of the pink film at $1720\text{--}1740\text{ cm}^{-1}$ is much higher than that of the yellow film, which in turn shows a higher gel content, due to the formation of alkoxy radicals and subsequent crosslinking.

In Figures 7 and 8, the comparison of as-received and QUV weathered films in terms of transmittance, limited to the visible wave length range (i.e., $380\text{--}760\text{ nm}$), is reported for pink and yellow samples, respectively.

Referring to the pink film, the NW brought about an appreciable increase of the transparency properties of the films only at the higher wavelengths. Conversely, no appreciable variation of the intensity of the absorbance peak can be detected with respect to the as-received film. The artificially weathered film also showed a dramatic increase of the transparency at the higher wavelengths. In contrast to the natural weathered film, however, the artificially weathered film showed a significant increase of the absorbance peak. A double peak is observed in

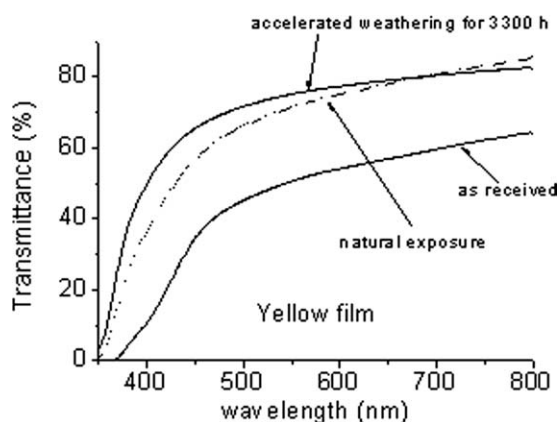


Figure 8. Influence of weathering on transmittance UV spectra of yellow films.

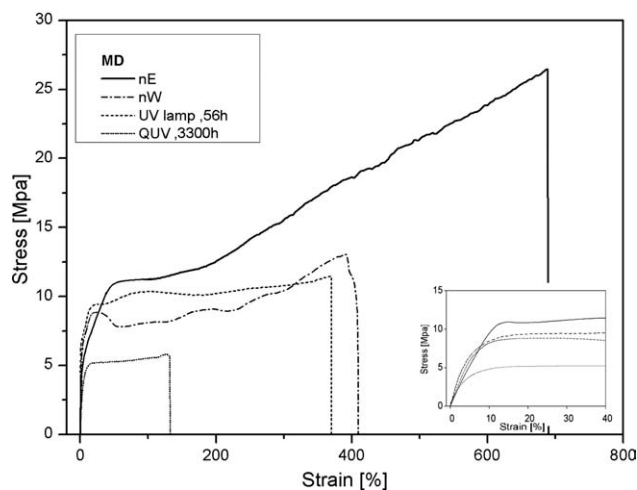


Figure 9. Tensile stress–strain curves. Comparison among NE, NW, exposed for 56 h to the UV Lamp ($UV_{lamp, 56h}$) and to the QUV chamber for 3300 h (QUV_{3300h}) films, respectively.

the spectrum of pink film irrespective to the weathering procedure performed, due to the presence of two different kinds of quinacridones, characterized by absorbance peak comprised in the range of $500\text{--}600\text{ nm}$.²⁹

In the case of the yellow film, no absorbance peak is observed, in the examined range, as the peak is hidden by the absorbance decrease occurring around 400 nm . The plot of Figure 8 clearly shows that, as previously observed for the pink film, weathering of the film involves an increase of the transmittance. This is true in both cases of natural and artificial weathering in the whole wavelength range. The films exposed to the mercury UV lamp for 56 h, not reported in Figure 8, showed a transmittance spectrum very similar to that of the artificially QUV weathered film.

Regarding tensile properties measured on yellow film samples, weathering phenomena induced relevant variations of the stress–strain curves, as shown in Figure 9.

The picture compares the tensile behavior in the MD of film samples subjected to NW, exposed for 56 h to the UV Lamp and conditioned using a QUV chamber for 3300 h, respectively, taking the not exposed (NE) sample as the reference one. The stiffening effect of weathering is highlighted in the inset reported within the same Figure 9.

Quantitatively, the influence of the different conditioning procedures on elastic modulus, yield strength, and elongation at break of accelerated and natural weathered samples, with respect to NE ones, is shown in Figures 10–12, respectively. In these pictures, artificially weathered samples, coded as $UV_{lamp,t}$ (i.e., weathered using a mercury UV lamp), QUV_t (i.e., weathered in QUV chamber), and NW (naturally weathered), respectively, where the subscript t indicates the exposition time, have been compared with NE film samples both in MD and TD.

Clearly, natural and UV lamp weathered films show an increase of the elastic modulus (see Figure 10) with respect to NE ones as an indication of the occurrence of modifications, that is,

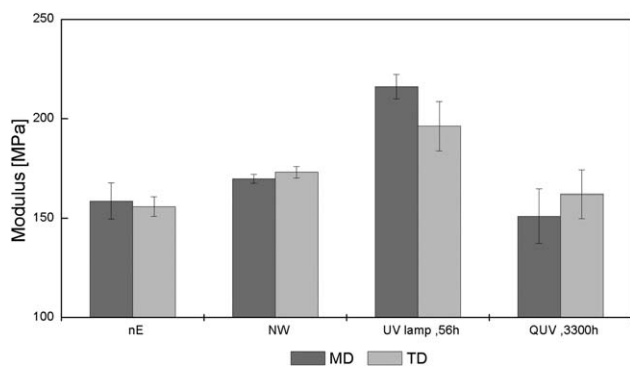


Figure 10. Weathering effects on tensile modulus of yellow films.

formation of double bonds and crosslinking, already appreciated from gel content measurements. In any case, small discrepancy revealed between MD and TD are not worthy to be highlighted being within the experimental errors, as indicated by the error bars reported in Figures 10–12.

Regarding the tensile strength at yield of film samples reported in Figure 11, a slight decrease of this parameter, found only in samples conditioned in QUV chamber with respect to as-received (nE) ones, can be ascribed to oxidative degradation phenomena essentially responsible of higher ordering effects in the amorphous region.^{30,31} On the contrary, for samples naturally weathered (NW) or exposed to the mercury UV lamp for 56 h, averaged values of the same mechanical parameter are comparable within the limit of experimental error with ones characterizing the NE film sample. Moreover, in these last two cases, it seems that the conditioning of the films has unexpectedly allowed a balance of the mechanical properties of these samples at least with regard to the strength at yield.

Finally (Figure 12), weathering procedures, as expected by the previous considerations, always induce a relevant decrease of the elongation at break, scarifying their further use for the reference applications.

As a general comment, the films subjected to NW showed lower modifications of the mechanical properties with respect to the artificially weathered films. In fact, the films subjected to artificial weathering in the QUV chamber or using a mercury UV

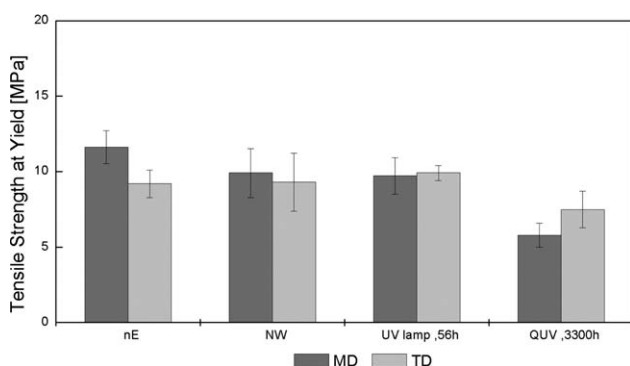


Figure 11. Weathering effects on the maximum tensile strength of yellow films.

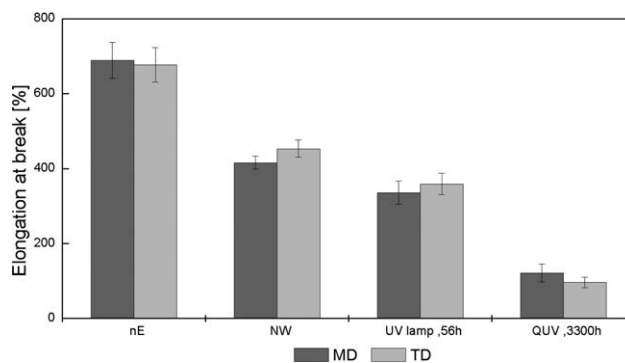


Figure 12. Weathering effects on elongation at break of yellow films.

lamp showed a dramatic decrease of both elongation at break and tensile strength. In agreement with the previous observations made on results obtained from rheological and transmittance tests, this suggests that artificial weathering generally provides an overestimation of the degradation effects occurring in real outdoor applications.

Finally, considering that the elongation at break is the most sensitive parameter to the structural and morphological variations of the polymers, Dilara and Briassoulis¹⁴ established that the useful lifetime of a greenhouse film can be represented by the time at which the value of this tensile parameter is halved with respect to the initial one. Thus, although conditioning procedures always involve nearly balanced degradation effects of tensile properties, from the results obtained it is possible to deduce that the durability of considered films on Apulia territory is more than 2 years.

CONCLUDING REMARKS

The weathering behavior of monolayer films of low-density/linear low-density polyethylene blends, containing commercially available organic pigments and an UV absorber of the benzophenone type, was analyzed by subjecting the films to both artificial weathering procedures and natural exposure.

The increased viscosity of weathered films, although variable with the type of included pigment, and the concomitant reduction of both tensile strength and elongation at break of yellow films clearly testify effects related to the occurrence of photodegradation phenomena, formation of double bonds and crosslinking.

UV-visible spectroscopy measurements enhanced changes of optical properties of weathered film samples with respect to NE ones.

Finally, besides the direction in which the sample was stretched, tensile measurements of all investigated films showed a clear increase of the stiffness at expense of the elongation at break for weathered films. These findings were accompanied by a slight increase in yield strength observed especially in case of the films weathered in a QUV chamber and ascribed to photo-oxidative degradation phenomena already invoked in the literature.

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