

# Development of Polypropylene-Based Ultraviolet-Stabilized Formulations for Harsh Environments

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*(Submitted 27 August 2001; in revised form 16 January 2002)*

This work reports the outdoor weathering performance of ultraviolet (UV)-stabilized polypropylene (PP) products (using PP resins from Saudi Basic Industries Corporation [SABIC]). Different hindered amine light stabilizers (HALS) were used to stabilize PP-film-based formulations that were exposed for 10 months in harsh outdoor weather of Dhahran, Saudi Arabia. Characterization of the exposed PP film products was done in terms of mechanical and Fourier transform infrared (FTIR) spectroscopic properties. HALS are a very effective light stabilizer for polyolefins.<sup>[14-16]</sup> They do not act by absorbing UV radiation, but by inhibiting degradation of the polymer, which has started the formation of free radicals. They function by scavenging radicals. HALS has low volatility and high extraction resistance. The effectiveness of HALS is independent of product thickness. Another advantage is that it provides a significant level of stabilization at relatively low concentration.<sup>[18]</sup> Some important types of high molecular weight HALS are Chimassorb 944 and Tinuvin 622, and a low molecular weight HALS is Tinuvin 770. These HALS, together with other UV stabilizers, are commonly used in polyolefin stabilization. The PP film formulations were divided into five categories based on the type of HALS incorporated. This was done to derive meaningful comparison of the various film formulations. The characterization data are presented for an exposure period of 10 months. The unstabilized PP films were degraded within 2.5 months of the exposure period. The performance in terms of decrease in mechanical properties and FTIR spectroscopic properties was assessed as a result of natural weathering for UV-stabilized samples. Following outdoor weathering trials, the lifetimes of certain formulations were determined. On the basis of the FTIR spectroscopic properties, it was determined that generally, the HALS-stabilized PP film formulations delayed the formation of oxidation products including esters, carbonyls, and trans-vinylenes.

**Keywords** Fourier transform infrared (FTIR) spectroscopy, hindered amine light stabilizers (HALS), mechanical properties, natural weathering, polymer degradation, polypropylene, ultraviolet (UV) stabilizers

## 1. Introduction

Plastics used for outdoor applications (e.g., agricultural films, polypropylene [PP] tapes, thick molded plastics materials, etc.) are typically subjected to ultraviolet (UV) radiation, high temperatures, temperature cycling from day to night, high humidity, and dust. Exposure of plastic materials to the weather causes various irreversible chemical reactions within the plastics that lead to the deterioration of their useful characteristics.<sup>[1-4]</sup> Either controlling or inhibiting the degradation reactions has considerable economic and technical advantages. Increased utilization of plastics in newer applications requires that ways and means be identified to prevent or reduce the damage caused by weathering parameters. This has been achieved by appropriately incorporating a single additive or a

combination of special additives such as light or UV stabilizers, antioxidants, and pigments such as titanium dioxide (TiO<sub>2</sub>) in plastic formulations. The type, combination, and concentration of these additives depend on the types of plastics, chromophores (chemical species such as hydroperoxides, ketones, catalyst residues, charge-transfer complexes, etc.), introduced during manufacturing or processing of polymers, which absorb UV light above 290 nm that may lead to photochemical reactions), or impurities present in the polymer, weather conditions, end-use applications, and the nature of outdoor utilization.<sup>[5-8]</sup>

Various stabilizer classes are available commercially for achieving a longer lifetime of plastics in an outdoor environment. UV radiation is considered to be the root cause of detrimental reactions in plastics used outdoors.<sup>[9-12]</sup> Degradation reactions are initiated when UV radiation reaches the chromophores present in the plastic. Impairment of mechanical properties of polyethylene or PP under sunlight and oxygen is due to oxidation.<sup>[13]</sup> The excitation due to chromophores can be reduced by incorporating a UV absorber as a stabilizer. The purpose of a UV absorber is to transform the UV energy into harmless heat energy. A basic disadvantage of UV absorbers is that they need a certain layer thickness (a minimum of 100-150  $\mu\text{m}$  thickness) to be effective; consequently, they provide limited protection for thin-section products, fibers, films, or tapes. The two most important types of commercial UV absorbers are ortho-hydroxybenzophenones and 2-(2-hydroxyphenyl)-benzotriazoles. Examples of benzophenone

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UV absorbers are Chimassorb 81, and benzotriazole UV absorbers are Tinuvin 326 and Tinuvin 327.

Another class of polymeric stabilizer, known as hindered amine light stabilizer (HALS), are very effective for polyolefins.<sup>[14-16]</sup> It does not act by absorbing UV radiation, but by inhibiting degradation of the polymer, which starts the formation of free radicals. It functions by scavenging radicals. It has low volatility and high extraction resistance. The effectiveness of HALS is independent of product thickness.<sup>[17]</sup> Another advantage is that it provides a significant level of stabilization at a relatively low concentration.<sup>[18]</sup> Some important commercial-grade HALS from Ciba Specialty Chemicals are Tinuvin 622, Chimassorb 944, and Tinuvin 770. These HALS, together with other UV stabilizers, are commonly used in PP stabilization.

If chromophores are excited by UV radiation and a radical is formed, then another class of UV stabilizer, called a quencher, can be used as a stabilizer, which will quench this excited state. If the formation of radicals cannot be avoided, then radical scavengers are used as stabilizers.<sup>[19,20]</sup> These energy transfer agents/quenchers act by transferring the excited-state polymer molecules back to a stable ground state.

These three classes of stabilizers and their combinations in the form of master batches are being used with polyethylenes and PPs to develop UV-stabilized formulations for agricultural films, molded products, and tapes. In addition, the hindered phenol type of commercial antioxidant, called Irganox 1010 (Ciba Specialty Chemicals, Basel, Switzerland), is added to the master batches to provide the polymer with processing stability and resistance to color formation. These antioxidants are meant to prevent the process of autocatalytic oxidation. These are free radical scavengers and interrupt the chain propagation reaction.

The weather conditions in Saudi Arabia are very severe. For example, levels of up to 150-180 kilo Langley (k) of UV radiation, 30-50 °C ambient temperature, and 50%-95% relative humidity occur in many areas. Large day-to-night temperature variations and high wind velocities are also encountered in certain geographic regions. In view of the harsh weather conditions, weathering trials on selected UV-stabilized PP-based formulations exposed in an outdoor environment become a necessity in Saudi Arabia.

PP is widely used as a commodity polymer and in technological applications. Its thermal and photochemical degradation has been extensively studied.<sup>[21-23]</sup> Most degradation studies generally involve a single initiation process—thermal, photochemical, or mechanical. Although several initiation processes are involved in most practical applications of polymers, the combined effects of two or more initiation processes have seldom been discussed in the literature.<sup>[24]</sup> The performance data on the lifetime of PP products exposed to natural harsh weather conditions, such as those in Saudi Arabia, are particularly absent in the literature.

This study discusses the development of UV-stabilized PP products (using PP resins from Saudi Basic Industries Corporation [SABIC]) that were exposed at an outdoor site. Different HALS-stabilized, PP-film-based formulations were exposed for 10 months in the harsh outdoor weather of Dhahran, Saudi Arabia. Characterization of the exposed film products was done in terms of mechanical and Fourier transform infrared (FTIR) spectroscopic properties. The outcome of natural weathering trials carried out at Dhahran, Saudi Arabia, and the

**Table 1 Polypropylene Film Formulations**

Sample ID	Antioxidant			
	Irganox 1010 (wt.%)	Chimassorb 944 (wt.%)	Tinuvin 783 (wt.%)	Tinuvin 770 (wt.%)
F-1	0	0	0	0
F-2	0.25	0.20	...	...
F-3	0.25	...	0.10	...
F-4	0.25	...	...	0.10
F-5	0.25	...	...	0.50
F-6	0.25	0.05	0.05	...

effects of weather on the stabilized PP film formulations, monitored in terms of mechanical and FTIR spectroscopic properties, are presented in this paper.

## 2. Experimental

### 2.1 PP film Sample Preparation

PP from SABIC designated as grade Ladene 520L, having a melt flow index of 10 g/10 min (at 2.16 kg and 230 °C) was used to prepare the PP films for the natural exposure trials at Dhahran, Saudi Arabia. The selected PP grade is basically a homopolymer, which can be used to produce cast and tubular water-quenched films with high melt strength, and good gloss and clarity. It contains slip and an antiblocking agent in the base resin from SABIC. The following steps were followed to prepare the PP films for natural exposure trials.

**2.1.1 Design of PP film Formulations.** An experimental program consisting of 15 different UV-stabilized PP film formulations were designed. Six PP film formulations consisting of appropriate combinations and additive levels of antioxidants and HALS were selected. Stabilization systems including the commercial HALS-type stabilizers Tinuvin 770, Chimassorb 944, and Tinuvin 783, and antioxidant Irganox 1010 were used in stabilizing the PP film products. The Tinuvin 783 was composed of 50% Tinuvin 622 and 50% Chimassorb 944. The Chimassorb 944 and Tinuvin 622 are high molecular weight (HMW) HALS, whereas Tinuvin 622 is a low molecular weight (LMW) HALS. The details of the six selected PP film formulations used in preparing PP film samples are presented in Table 1.

**2.1.2 Processing of PP Films.** The UV stabilizers, according to the formulation design shown in Table 1, were first mixed with a PP batch size of 1 kg. The polymer and additive mix was then fed into a laboratory-sized, single-screw extruder (model 100-25 HC, Brabender, Germany). The extruder screw diameter ( $D$ ) is 3.17 cm, and the length-to-diameter ratio ( $L/D$ ) is 25:1. Films with thickness in the range of 200 to 290  $\mu\text{m}$  were extruded at a film processing temperature of 215 °C.

### 2.2 Outdoor Exposure Trials at Dhahran, Saudi Arabia

UV-stabilized PP film samples were mounted first on aluminum frames and these frames were mounted on exposure racks facing South at 45°, per the requirements of American Society for Testing and Materials (ASTM) D-1435 standard.<sup>[25]</sup> The natural weathering trials at the Dhahran weather-

ing site were scheduled for a total duration of 10 months. Six PP film samples were exposed. Exposed PP film samples were withdrawn after completion of 2.5, 5, 7.5, and 10 months.

### 2.3 Characterization of Exposed PP film Samples

The four withdrawals, consisting of five PP film samples in each withdrawal, were characterized in terms of their mechanical and FTIR spectroscopic properties. The mechanical properties determined for the exposed PP films included percent elongation at break and stress at break. Mechanical properties were determined according to the procedures of ASTM D-882 method<sup>[26]</sup> using an Instron (UK) mechanical testing system. Five measurements were made to determine the mechanical properties. An average of the five values are reported. To determine the variation in the experimentally determined data, the standard deviations for percent elongation at break and stress at break were found to be within 32.3% and 2.15 MPa, respectively.

The FTIR spectroscopic properties were determined to monitor the extent of degradation. The FTIR spectroscopic properties included determination of concentrations of carbonyl, trans-vinylene, and ester groups. The model 1600 FTIR spectroscope (Perkin-Elmer, USA) was used.

It has been indicated in the literature that the growth in carbonyl is an indication of the effect of photooxidation processes taking place in the polymer matrix.<sup>[8,27]</sup> The FTIR spectra in the region of 500 to 4000  $\text{cm}^{-1}$  were obtained on the exposed film samples. The carbonyl content was determined by measuring the absorbance at around 1715  $\text{cm}^{-1}$ . The trans-vinylene and ester absorbance was measured at 973  $\text{cm}^{-1}$  and 1735  $\text{cm}^{-1}$ , respectively. Two FTIR measurements for each sample were obtained. An average of two measurements for the FTIR data is reported. However, to determine the variation in experimental measurements, the standard deviation was found to be within 0.0093.

## 3. Results and Discussion

The results and discussion of the exposed PP film samples are based on four withdrawals from the Dhahran exposure site. Characterization results of the film samples exposed for 10 months in light of mechanical properties and FTIR spectroscopic properties are discussed.

### 3.1 Mechanical Properties

Degradation trends have been monitored in terms of decrease in mechanical properties, i.e., percent elongation at break and stress at break. Changes in mechanical properties with exposure time for the exposed PP films are presented in Fig. 1 and 2.

The mechanical property failure depends not only on the extent of chemical degradation but also on spherulite sizes, (super structures formed by arrangement of crystallites during process of crystallization) and surface cracks.<sup>[28]</sup> Many researchers<sup>[29,30]</sup> have observed the impairment of mechanical properties under the influence of sun and oxygen.

The unstabilized PP films were generally completely de-

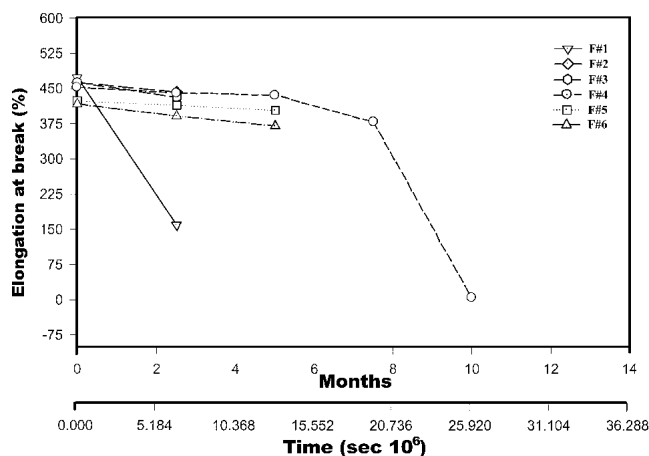


Fig. 1 Percent elongation at break of unstabilized and stabilized PP films exposed at Dhahran, Saudi Arabia

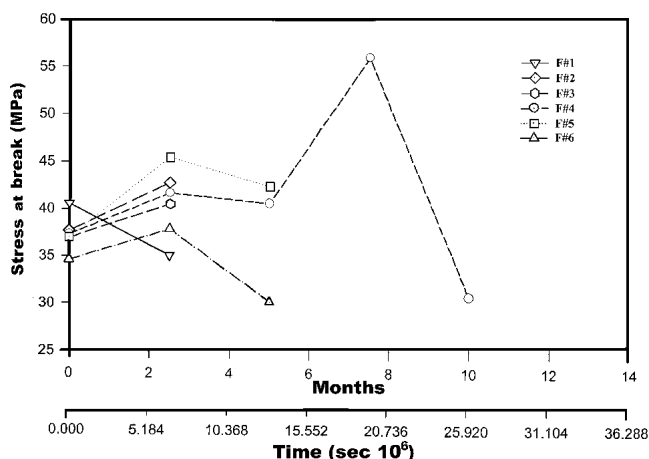
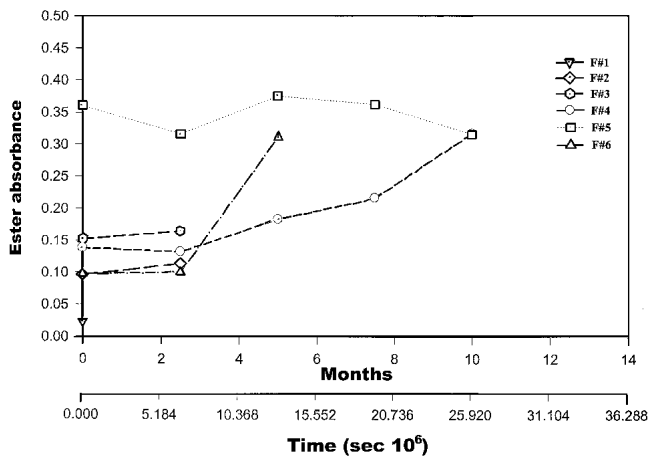


Fig. 2 Stress at break of unstabilized and stabilized PP films exposed at Dhahran, Saudi Arabia

graded during the initial 2.5 months of outdoor exposure. The PP films were classified into five categories on the basis of the type of HALS incorporated in the formulations. The first category consisted of an unstabilized film sample (F-1) and one stabilized film (F-2) using different levels of Chimassorb 944. The Chimassorb 944 content in PP film sample F-2 was 0.2 wt.%. F-2 shows degradation within 5 months of natural exposure. The second category of PP films included stabilized film with the identification F-3. This film formulation, which had Tinuvin 783 with addition levels of 0.1 wt.%, showed degradation within 5 months.

The third category of PP films had the identification F-4. The UV stabilizer Tinuvin 770 was added at addition levels of 0.1 wt.% of PP films. The F-4 film having a lower level of Tinuvin 770 retained its original mechanical properties at an acceptable level of up to 7.5 months. Films with the identification F-5 showed good retention of mechanical properties after up to 10 months of exposure.

The fourth category of PP film formulations included is F-6, which contained a combination of Chimassorb 944 and Tinuvin



**Fig. 3** Changes in ester group absorbance in unstabilized and stabilized PP films exposed at Dhahran, Saudi Arabia

783. The F-6 film has 0.05 wt.% of each of the two HALS included in the formulation. It shows a lifetime of 5 months.

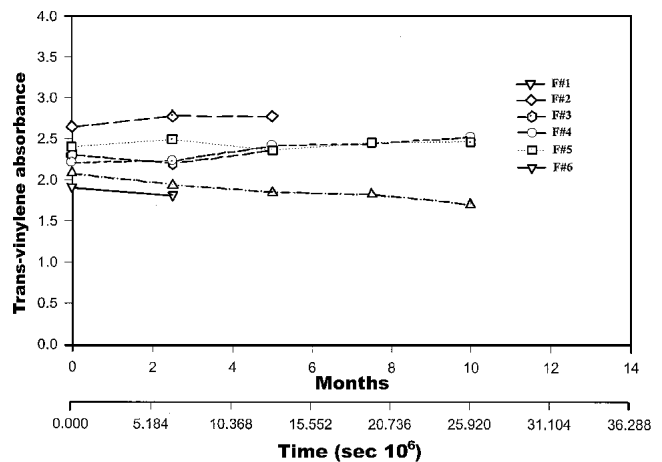
### 3.2 FTIR Spectroscopic Properties

Characterization data obtained on the exposed PP film samples using FTIR spectroscopy are presented in Fig. 3, 4, and 5. Infrared spectra were obtained in transmission through the exposed films using a model 1600 FTIR spectrometer (Perkin-Elmer). The main products of degradation (carbonyls, trans-vinylenes, and esters) in the wavelength range of 400 to 1800  $\text{cm}^{-1}$  were determined. Quantitative determinations of the above peaks were made and the absorbance levels are presented in Fig. 3, 4, and 5. The carbonyl peak was detected at a wavelength of 1715  $\text{cm}^{-1}$  and esters were detected at 1735  $\text{cm}^{-1}$ . The trans-vinylene group absorbance was detected at 973  $\text{cm}^{-1}$ .

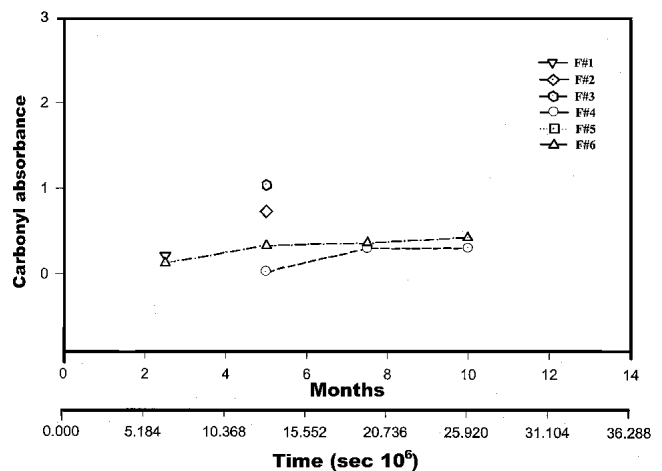
The changes in FTIR spectroscopic properties with respect to exposure time, as a result of the degradation products formed for the four categories of the exposed PP film formulations, are presented. The changes in ester group absorbance with exposure time are shown in Fig. 3. The changes in trans-vinylene absorbance with respect to exposure time are presented in Fig. 4. Carbonyl absorbance growth with respect to exposure time for all formulations is shown in Fig. 5.

The first category consisted of three film formulations (F-2) stabilized with different levels of Chimassorb 944. The FTIR spectra showed a high level of development of carbonyl groups within 5 months of exposure. The mechanical test results also confirm these results. The trans-vinylene group formation slightly increased, as shown in Fig. 4. The FTIR spectra of film F-2 showed an increase in esters during the 10 months of exposure (Fig. 3).

The second category of PP film is F-3. This film formulation had Tinuvin 783 with addition levels of 0.1 wt.%. Tinuvin 783 is a blend of 50 wt.% Chimassorb 944 and 50 wt.% Tinuvin 622. The FTIR spectra showed development of carbonyl groups within the initial 5 months of exposure (Fig. 5). The growth of esters in all three formulations was slight (Fig. 3). The ester peak determined in these formulations was due to two



**Fig. 4** Changes in trans-vinylene absorbance in unstabilized and stabilized PP films exposed at Dhahran, Saudi Arabia



**Fig. 5** Changes in carbonyl absorbance in unstabilized and stabilized PP films exposed at Dhahran, Saudi Arabia

factors. These included the presence of Tinuvin 622 (which was displayed in an FTIR spectra at 1735  $\text{cm}^{-1}$ ) and the effect of weathering. Figure 4 showed a slight development of trans-vinylene groups in these formulations.

The third category of PP films with identifications F-4 and F-5 had LMW HALS, Tinuvin 770, at levels of 0.1, 0.2, and 0.5 wt.%, respectively. The film F-4, having a lower level of Tinuvin 770, showed a significant increase in the level of carbonyl group at 7.5 months of exposure (Fig. 5). The FTIR spectra of F-5 did not show any development of carbonyl. Figure 4 shows a slight increase in trans-vinylene group in all three formulations. The effect of delaying the trans-vinylene group and other oxidation group formation has also been observed by other research.<sup>[28]</sup> The ester groups showed a significant increase in F-4 during the 10 months of exposure. F-4 displayed a slight increase in ester group formation, as shown in Fig. 3.

The fourth category of PP film formulation included F-6. The formulations contained a combination of Chimassorb 944 and Tinuvin 783. F-6 film showed a significant increase in

carbonyl within 7.5 months of exposure and resulted in complete degradation of the film. Weathering results in the trans-vinylene group changed with respect to exposure time, as presented in Fig. 4. A slight decrease of trans-vinylene functionality was observed in the formulation (F-6), as shown in Fig. 4. The ester group formation showed a significant increase (Fig. 3). The ester peaks determined in these formulations were due to the presence of Tinuvin 622 and the effect of weathering. The increase was generally due to the formation of ester groups as a result of photooxidation reactions as a result of outdoor weathering.

#### 4. Conclusions

The lifetimes for the PP film formulations exposed for 10 months were determined. The lifetimes for the six naturally exposed PP films included: 0 to 2.5 months for F-1, 2.5 to 5.0 for F-2, 2.5 to 5.0 for F-3, 7.5 to 10 months for F-4, greater than 10 months for F-5, and 5.0 to 7.5 months for F-6.

The greatest lifetime (greater than 10 months) was determined for PP film F-5 stabilized with 0.5 wt.% Tinuvin 770. An intermediate lifetime (5 to 10 months) was determined for PP film formulations F-4 and F-6. The F-4 was stabilized with 0.1 wt.% Tinuvin and F-6 was stabilized with a combination of 0.05 wt.% each of the stabilizers Chimassorb 944 and Tinuvin 783. A lower lifetime (2.5 to 5 months) was observed for PP film formulations F-2 and F-3. The F-2 was stabilized with 0.2 wt.% Chimassorb 944 and F-3 was stabilized with 0.1 wt.% Tinuvin 783. The unstabilized PP film F-1 was completely degraded within 0 to 2.5 months during outdoor weathering trials.

The HALS-stabilized PP films that were completely degraded showed a significant increase in carbonyl functional groups. Generally, the weathered samples showed an increase in ester functional groups with exposure duration. These increases in oxidation functional groups were due to photooxidative reactions that had taken place in the polymer matrix.

The HALS stabilizers together with antioxidants have significantly contributed toward UV stabilization of the base polymer and also the increase in lifetimes of PP films. These films can be used in outdoor applications (particularly in agricultural films and packaging applications) in geographical locations with higher UV radiations and higher temperatures, such as those prevailing in Saudi Arabia.

#### Acknowledgment

The author acknowledges the support of King Fahd University of Petroleum & Minerals in conducting this study.

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