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## Experimental study of the degradation of HDPE packaging under accelerated thermal aging

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ARTICLEINFO	ABSTRACT
Article history: Received 21 May 2023 Accepted 31 August 2023 Available online 2 September 2023 Keywords: HDPE Accelerated thermal aging Kissinger model	In this work, accelerated thermal aging at different temperatures was performed on high-density polyethylene (HDPE) bottles and specimens for food packaging. The degradation of HDPE was followed macroscopically by tensile and compression tests to evaluate the decrease in mechanical properties. The data obtained from these tests are used to calculate a new static damage law and the life fraction of this polymer. A prediction of the lifetime of HDPE was determined by thermogravimetric analysis (TGA) in dynamic mode. The evolution of the crystallinity rate during accelerated thermal aging was carried out using FOURIER transform infrared spectrometry (FTIR). The results obtained show a remarkable degradation of the mechanical properties in traction and
Static damage Life fraction	compression by calculation of the static damage, and the physical and chemical properties by the analysis of the rate of crystallinity as well as the prediction by the law of Arrhenius.
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# 1. Introduction

High-density polyethylene (HDPE) is widely used in food, cosmetic, pharmaceutical, and food packaging, due to several advantages such as flexibility, recyclability, economical and unbreakable, it is more consumable in the form of bottles and vials. The consumption of HDPE packaging is expected to reach 17.5 million tons by 2028 according to a market study published by Global Market Insight with an annual growth rate of about 4.5% (Wee & Choi, 2020). High-density polyethylene is a thermoplastic polymer sensitive to temperatures above 70°C and becomes less protective (Whelton & Dietrich, 2009). HDPE works well above its glass transition temperature (Tg), melting at 130°C (Simar, 2014). This material is sometimes used as reinforcing additive and improving the performance and life of asphaltic binders (Shahryari et al. 2021). In the packaging industry, thermal and hydraulic aging are the most common causes of aging in practice, thermal aging causes a change, instability, and degradation of the HDPE material (Ayad et al., 2002), which leads to a change in brightness, color, and surface of packaging (Audouin et al., 2007). The increase in temperature causes several phenomena such as oxidation, pyrolysis, thermolysis, mechanical creep, and chemical attack (Li, et al., 2021). Thermal aging is a process that can affect the safety, quality, and stability of packaged products either in storage or transport, so it is necessary to study and understand the mechanisms of degradation of physical and chemical properties, for this reason, the literature has proposed several kinds of research to study the behavior of different polymers under thermal aging. YOUN SONG has noticed the correlation between the change of color of Polyetherimide reinforced with glass fibers with the change of mechanical properties in traction and flexion under different thermal aging (Song, et al., 2021). Another approach was extrapolated from the thermal aging experiments with UV radiation of propylene-ethylene rubber (EPM), the results were shown the difficulty of observing chemical degradation for this type of aging (Gillen & Celina, 2018), the study of Tcharkhtchi et al was focused on the effect of thermal aging on the mechanical properties of Polyurethane, it was shown that there are two characteristic periods where

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the mechanical properties change (Tcharkhtchi et al., 2014). The degradation mechanisms of low-density polyethylene (LDPE) were observed by chemical and physical technics (Suraci et al., 2020). On the other hand, Haoyu et al determined the kinetics of the pyrolysis process for HDPE waste (Shah et al., 1994), the study of Chaitanya et al based on the prediction of the life of polymer composites by the cumulative theory of damage by photo-oxidation, hydrolysis, and irradiation (Alothman et al., 2013). Another study was conducted on predicting the service life of HDPE 100 pipes under thermal aging using comparisons of the mechanical behavior of this material in the presence/absence of antioxidants, the activation energy, and the shift factor by the oxidation index method ("OIT") (Eddine et al., 2000). Studies by researchers (Celina et al., 2005; Fouad, 2010; Al-Bayaty et al., 2020; Varley et al., 2000; Choi, E et al., 2020 ; Dorleans et al., 2021) report that the degradation of HDPE under temperature leads to a change in mechanical and physical behavior at temperatures between 40°C and 120°C, after these temperatures the results of the degradation of this material may be erroneous. The degradation of polymeric materials can focus on the variation of elongation at break, tensile strength, and density (Loos & Dara, 1987; Rühl et al., 2017; Kurtz et al., 2002). Giannuzzi et al related HDPE recycling to thermal degradation and thermodynamic degradation kinetics during initiation, propagation, and termination (Giannuzzi et al., 1998).

In accelerated thermal aging, the Arrhenius law is widely applied to predict the behavior of polymers under the effect of high temperatures for short periods of time, this law allows for estimating the life of polymers by several methods, such as the coupling of differential scanning calorimetric (DSC) with thermogravimetric analysis, elongation at break, and thermogravimetric analysis in non-isothermal mode (Tries et al., 1997; Bystritskaya et al., 2013; Krishnaswamy et al., 2005; Khelidi et al., 2006; Montanari et al., 1989; Liu et al., 2016; Ciardiello et al., 2020; Gates et al., 1999; Shi et al., 2000; Jemii et al., 2020; Jakubowska et al., 2022). Elena has used this law as a technique for monitoring the properties of Rubber (Budrugeac & Segal, 1998), and extrapolation of the performance of construction materials of a nuclear power plant by Arrhenius law was developed by Ramteke et al (Ramteke et al., 2010). The accelerated thermal aging caused mechanical degradation of HDPE, thus causing damage to the mechanical properties and characteristics. The damage mechanics was discovered by Kachanov in 1958 (Lemaitre & Desmorat, 2005), he considered that the degradation of mechanical properties of fatigue, whereas the value of the damage to the imposed value of 0 for a material does not undergo any type of damage, and equal to 1 for a perfectly damaged material (Majid et al., 2018). The theory of continuum Damage Mechanics (CDM) has been established by several works (El-Bagory et al., 2015; Sanada et al., 2015; Eftekhari et al., 2016; Ayadi et al., 2018; Ennaji et al., 2019).

The objective of this paper is to study the kinetics of the degradation of the mechanical properties in tension-compression, and the chemical and physical properties of HDPE bottles intended for food packaging. The novelty of this article is:

1) To establish accelerated thermal aging on HDPE packaging to show the role of thermal stability during the manufacturing process of these packages.

2) To help researchers to follow an experimental methodology of accelerated thermal aging of polymers based on this study since in the literature there is a lack of experimental methodologies.

3) Determine the effect of accelerated thermal aging on the mechanical properties of HDPE in compression (for the first time) and in tension.

4) Discovered new static damage laws in relation to accelerated thermal aging.

In short, the novelty of this work is the real use of HDPE packaging to understand how it degrades over several years of storage or use, by simulating these conditions over short periods. In this way, the influence of thermal aging on mechanical and chemical properties can help to design sustainable packaging solutions. The TGA technique makes it possible to estimate service life and therefore facilitates the selection of the best packaging design and manufacturing method. FTIR enables us to understand molecular and microstructural changes during aging.

First the methodology of accelerated thermal aging, the methodology of tensile and compression tests was described, a calculation of the crystallinity rate during the aging time was done using FTIR, then a prediction of the material lifetime using TGA. Finally, a mechanical analysis of the life fraction by a new damage law was discovered based on the tensile strain at break, and the compressive strength for compression tests.

#### 2. Experimental Procedure

#### 2.1 Materials and instruments

In this work, we are interested in the effects of thermal aging on HDPE packaging.

- The bottles used were obtained from the HDPE extrusion process, the HDPE material with a density of 0.995 g/cm3 and melt index of 0.35g/10min, these values were obtained from the manufacturing company of these bottles.

- For accelerated thermal aging at 43°C and 80°C, a MEMMERT oven (UN 30, Gemini BV) was used.

- The tensile and compression tests were performed in the MTS machine (MTS criterion series 40).
- The FTIR tests were performed with the SHIMADZU-IRRAFINITY apparatus.
- The thermogravimetric tests were carried out in the SHIMADZU DTG-60H apparatus.

#### 2.2 Accelerated thermal aging method

To perform the aging tests, we started by cutting the bottles into parallelepipedic specimens of the same thickness of 1.10mm, length of 100mm, and width of 20mm (Fig.1).

The characteristics of this treatment are:

- Maintain the specimens and bottles at 80°C for 9, 18, 27, 36, 54, 72 and 108 days.
- Maintaining the specimens and bottles at 43°C for 9, 18, 27, 36, 54, 72, and 108 days.
- All the aged samples are placed in the oven at the same time to avoid a decrease in the holding temperature (Fig. 2).
- All aging tests were performed according to ASTM D3045-92.

The temperatures 43°C and 80°C were chosen because they represent the typical temperature ranges to which HDPE may be exposed in real applications. On the one hand, the 43°C temperature is the simulation of the use in temperate climates or long-term storage, on the other hand, the 80°C temperature is used to test the resistance of HDPE packaging to extreme conditions, such as direct long-term exposure to UV radiation, as well as storage of these packages in summer in trucks and warehouses. The aging times were chosen according to the standard, compatibility with the industrial requirements of HDPE packaging, as well as to facilitate the comparison between the two temperatures since these times were selected based on the rule of Rounding to the nearest integer. And finally, to ensure reproducible, uniform, and reasonable conditions for this accelerated thermal aging.



Fig. 1. Cutting of HDPE bottles into parallelepipedic specimens

**Fig. 2.** Test setup, (a): dog bone shape, (b): Specimens and bottles are placed in the oven for thermal aging

#### 2.3 Methodology of tensile and compression tests

The evolution of mechanical properties during aging is determined by tensile and compression tests under the following conditions:

- For each aging time and temperature, three specimens are tested in tension according to ISO1798.
- For compression tests, three bottles at each aging temperature and time are tested according to ASTM D2659.

- The characterization of HDPE in tension is done by unaged dumbbell specimens (Fig.3), these specimens were obtained using a standardized punch.

- The characterization in compression is done by unaged cylinders.
- The strain rate in tension is 10mm/min, and in compression is 250mm/min.



Fig. 3. Dimensions of the dumbbell specimens used for the characterization of HDPE extracted from the bottles.

#### 2.4 Static damage

Thermal aging of materials is a type of fatigue, where the degradation of mechanical properties of the material is due to the superposition of time and temperature factors. In this work, a model of static damage in tension and compression has been discovered.

The damage defined by Majid and Elghorba (2018) is:

$$D_{\rm s} = 1 - \frac{\sigma_{\rm eff}}{\sigma} \tag{1}$$

With  $\sigma_{eff}$  is the effective stress.

In accelerated thermal aging, for tensile tests on aged specimens, the static damage is:

$$D_{s} = \frac{\varepsilon_{rti} - \varepsilon_{rtmin}}{\varepsilon_{rtmax} - \varepsilon_{rtmin}}$$
(2)

 $\begin{cases} \epsilon_{rtmin} & \text{is the strain at break in the unaged state} \\ \epsilon_{rti} & \text{is the strain at break at aging time i, } & 9 \text{ days} \leq i \leq 108 \text{ days} \\ \epsilon_{rtmax} & \text{is the strain at break at the maximum aging time} \end{cases}$ 

Static damage of cylinders in compression is:

$$D_{s} = \frac{RCV_{ti} - RCV_{tmin}}{RCV_{tmax} - RCV_{tmin}}$$
(3)

 $\begin{array}{ll} \text{RCV}_{tmin} & \text{is the vertical compressive strength in the unaged state} \\ \text{RCV}_{ti} & \text{is the vertical compressive strength at aging time i, 9 days} \leq i \leq 108 \text{ days} \\ \text{RCV}_{tmax} & \text{is the vertical compressive strength at maximum aging time} \end{array}$ 

The life fraction in both tests (Eq. (1) and Eq. (2)) is:

$$LF = \frac{t_i - t_{min}}{t_{max} - t_{min}} \tag{4}$$

 $\begin{cases} t_{min} & \text{is the time in the unaged state} \\ t_i \text{ is the aging time i. 9 days} \leq i \leq 108 \text{ days} \\ t_{max} & \text{is the maximum aging time} \end{cases}$ 

Reliability is a parameter that describes the ability of a material to function without failure under required conditions of use such as aging, defect, etc., for a specified period of time. The relationship between damage and reliability is:

(5)

Damage + Reliability = 1

2.5 TGA and FTIR

The thermogravimetric experiments were performed at temperatures ranging from room temperature to 600°C, with an alumina crucible under air and under three heating rates, 5°C/min, 10°C/min, and 20°C/min.

Blank and aged samples at 18, 36, and 108 days under thermal aging at 43°C and 80°C were heat treated in TGA. After the thermogravimetric analysis, the results obtained in TGA are used for the calculations of the apparent activation energy and the pre-exponential factor during thermal aging.

The chemical degradation during this thermal treatment for different aging times was performed by FTIR. For each sample, the specimens are tested in IR in transmission mode in order to calculate the crystallinity rate of HDPE.

#### 3. Results and Discussion

#### 3.1 Tensile properties

The mechanical behavior of HDPE under tensile tests in the virgin state is a ductile behavior (Fig. 4), and is characterized by a high rate of stretching between 38% and 158% of deformation. The range between 0 and 48MPa characterizes linear viscoelasticity that allows us to calculate Young's modulus which equals 1370MPa and the elastic limit of value 42MPa. During accelerated thermal aging, the most important remark relates to the propagation of striction that decreases as a function of aging time for the two temperatures studied (Fig. 4), that is, the disappearance of the plastic plate from 9 days to 108 days, where the stress at break continues to increase from 15MPa in 9 days to 20MPa in 108 days, and from 12MPa for 9 days of aging to 16MPa at 108 days for the temperature 43°C (Fig. 4). The decrease in the strain at break (Fig. 5) and tensile strength (Table 1) describes the fragility of HDPE during the increase of temperature and aging time, which induces such rapid aging at 80°C, so it can be seen that the results of strain at break are more compatible to describe the degradation of the mechanical properties of HDPE in tension.



Fig. 4. Stress-strain curves for aging at 43°C and 80°C.

Table 1. Comparison of tensile strength at different aging times and temperatures.

Aging Temperature (°C)	Aging Time (days)	Tensile strength (MPa)
43	9	42,98
43	18	39,83
43	27	37,20
43	36	28,91
43	54	24,05
43	72	21,60
43	108	19,18
80	9	38,70
80	18	34,68
80	27	29,91
80	36	25,11
80	54	20,18
80	72	16,85
80	108	10,51

We can relate the strain at failure (Fig. 5) fail to an induction time, this time appearing before the drop catastrophic of the strain at failure. For the temperature 43°C the induction time is 27 days, but for 80°C it is 18 days. These results have been observed for PP and PET (Kufel & Kuciel, 2019; Arhant et al, 2022).



Fig. 5. Variation of strain at failure as a function of aging time



**Fig. 6.** Force-displacement curves were obtained by the Compression tests at 43°C and 80°C

#### 3.2 Compressive properties

Fig. 6 shows the compressive response of HDPE bottles aged at different temperatures. The results show a linear elastic behavior from 0 to 1400 N for unaged bottles, from 0 to 600 N, and from 0 to 500 N between 9 to 108 days for temperature 43 °C (Fig. 6), on the other hand at 80°C the range of the linear elastic response varied from 0 to 500 N and from 0 to 400N, from 9 to 108 days in series. Then the force slowly continues to increase with the displacement until the HDPE bottles break. It can be noticed that the degradation of the bottles is less at 43°C compared to 80°C for the vertical compressive strength (RCV) (Fig. 7), which means that the thermal stability of HDPE is very important and one of the main criteria for the use of HDPE bottle in the packaging field.



Fig. 7. Comparison of the Vertical Compressive Strength (RCV) for the two temperatures

Comparing the vertical compressive strength, a decrease in RCV from 2 kN in the unaged state to 578 N in 108 days at 43°C, and to 468N in 108 days at 80°C is noted. The results obtained are comparable with PLA and PVC (Abbés et al., 2010; Widiastuti et al., 2014).

#### 3.3 Static Damage

As mentioned in this article, we have performed two types of tests to determine the mechanical properties of HDPE, we used the relationships presented in section 1.3 (Eqts 1-4). We obtained damage-reliability curves as a function of the life fraction as shown in the following figures.



Fig. 8. Damage-reliability for tensile test under aging at 43°C



Fig. 9. Damage-reliability for tensile test under aging at 80°C



Fig. 10. Damage-Reliability for compression test under aging at43°C

Fig. 11. Damage-Reliability for compression test at 80°C

The damage-reliability superposition curves allowed us to define the critical life fraction at which the material starts to weaken, for each test and each heat treatment. The critical life fraction at 43°C is 0.16 (Fig. 8 and Fig. 10), while at 80°C the life fraction is 0.08 (Fig. 9 and Fig. 11). We can observe the presence of damage stages for both temperatures, the first stage of initiation between 0 and 0.16 for 43°C, and from 0 to 0.08 for 80°C. The second stage of propagation is between 0.25 and 0.33 at 43°C, and from 0.16 to 0.25 at 80°C. Consequently, the last propagation stage is 0.33 to the unit for 43°C, and 0.25 to the unit for 80°C. The mechanical performance at 43°C is different compared to the performance obtained at 80°C, these results confirm the results obtained by M Majid et al., 2017 and Rabiaa et al., 2023.

#### 3.4 FTIR Analysis

Fourier transform infrared spectrometry analysis allowed to obtain the evolution of the chemical properties of HDPE during treatment at 43°C and 80°C by calculating the crystallinity rate. We chose to work in the range 1800 cm<sup>-1</sup> to 2500 cm<sup>-1</sup> (Figs. 12 and 13) to show the influence of temperature on the carboxylic acids RCOOH.



The increase in temperature increases the absorbance intensity between 2350 cm<sup>-1</sup> and 2360 cm<sup>-1</sup> from 1.8% in the unaged state to 2.2% after 108 days of aging at 43°C (Fig. 12), and for the temperature of 80°C the intensity increases to 2% at 108 days of aging, we note that aging does not affect the C=C bonds at 1900 cm<sup>-1</sup>. The crystallinity rate Xc is calculated from the characteristic bands between 720 cm<sup>-1</sup> and 730 cm<sup>-1</sup> (Chamas et al., 2020) (Fig. 14).



**Fig. 14.** The areas between  $720 \text{ cm}^{-1}$  and  $730 \text{ cm}^{-1}$ 



Fig. 15. Crystallinity rates at 43°C and 80°C

$$X_{c}(\%) = 100 - \left(\frac{\frac{1 - \frac{l_{a}}{l_{b}}}{1 - \frac{1}{l_{b}}}}{1 + \frac{l_{a}}{l_{b}}} \cdot 100\right)$$

- Ia and Ib are the sub-areas of the peaks determined from the bands selected.
- 1.233 is the relationship of the band intensities of Ia and Ib for a completely crystalline HDPE.

The following figure represents the calculation results of the crystallinity rate obtained (See Fig. 15). The increase of the crystallinity rate by a rapid way at 80°C, this increase describes the accelerated method of molecular chains cession and molar mass decrease due to the material oxidation, from 9 days of aging until reaching a critical aging time at 32 days from which the crystallinity rate starts to increase by a weak way. On the other hand, at 43°C, we find a slight decrease of the crystallinity rate at 9 days of aging, then a critical value at 18 days is reached where the crystallinity rate starts to increase slowly. These phenomena explain the variation of the strain at break (Fig. 5) and the embrittlement of the HDPE material.

#### 3.5 TGA Analysis

The Arrhenius method is a time-temperature superposition method, allows to predict the activation energy, the preexponential factor and the lifetime of HDPE, thus highlights the relationship between the heating rate and the temperature.

The Arrhenius law is given by (Mourad et al., 2009):

$$K(T) = Ae^{\frac{-E_a}{RT}}$$
(7)

$$ln(K(T)) = \frac{-E_a}{RT} + ln(A)$$
<sup>(8)</sup>

where Ea is the apparent activation energy, A is the pre-exponential factor, R is the constant of perfect gases and T is the absolute temperature.

The linear curve of Ln (K(T)) versus 1/T is the Arrhenius curve, where -Ea/R is the slope.

TGA analysis provides weight loss versus temperature degradation curves at different heating rates (Fig. 16).

The derivative of the TGA curves gives the maximum or peak temperature at different heating rates.

The dependence of the maximum temperature Tp and the heating rate  $\beta$  is expressed by an isothermal Kissinger's free isoconversion law (Komesu et al., 2017):

$$Ln\left(\frac{\beta}{T_p^2}\right) = Ln\left(\frac{AR}{E_a}\right) - \frac{E_a}{RT_p} \tag{9}$$

The following curves represent the results of TGA, derived from TGA and the extrapolation by the Kissinger model.





Fig. 16. TGA analysis at different heating rates and different aging times, (a): unaged sample, (b): aged 18 days at 43°C, (c): aged 108 days at 43°C, (d): aged 18 days at 80°C, (d): aged 108 days at 80°C.

Thermal degradation of HDPE was produced in a single step for the different samples tested, where the weight losses varied with aging time, aging temperature, and heating rate, all curves shift towards the higher heating rate. All the samples aged at different aging times started to decompose at 450 °C, on the other hand, the unaged samples were degraded at 500 °C, i.e., the samples retained their weights below 450 °C, although great degradation was produced in the range of 450 °C to 600 °C, these phenomena have been by several researchers (Nassim et. al, 2018; Niang et al., 2022; Hedir et al, 2020 ; Chiaraa et al., 2020), by which the degradation of HDPE at different condition varies from 380 °C, 400 °C, and 500 °C. A significant weight loss at 108 days of aging for both temperatures used was seen at 600 °C. By comparison of the weight loss curves at 43°C and 80°C, the loss of thermal stability at 80 °C was observed in a shorter aging time compared to 43°C. The derivative of the TGA curves allows us to know the maximum temperature used in the Kissinger model, the following figures represent the plots of Ln( $\beta/Tp^2$ ) versus (1/Tp) at different aging times.



Fig. 17.Linear Kissinger curves at different aging times, (a): unaged sample, (b): aged 18 days at 43 °C, (c): aged 108 days at 43 °C, (d): aged 18 days at 80 °C, (d): aged 108 days at 80 °C.

The obtained curves were represented by linear regression, with the apparent activation energy at each aging time as the slope, and the pre-exponential factor was represented by the intercept of these curves. The following table summarizes the data extracted from these curves.

Aging Temperature (°C)	Aging Time (days)	Ea (kJ/mol)	A (min <sup>-1</sup> )	$\mathbb{R}^2$	
Not aged	Not aged	116,23	10,17 105	0,972	
43	18	111,98	9,77 10 <sup>5</sup>	0,9876	
43	36	124,728	10,98 10 <sup>5</sup>	0,9814	
43	108	136,46	12,18 10 <sup>5</sup>	0,9999	
80	18	127,38	11,23 10 <sup>5</sup>	0,9793	
80	36	101,73	11,56 10 <sup>5</sup>	0,9974	
80	108	77,83	6,39 10 <sup>5</sup>	0,9979	

**Table 2.** Kinetic parameters of HDPE during thermal aging

From Table 2, we can notice the instability of the apparent activation energy during thermal aging, although after 18 days of aging at 80°C Ea increases from 116 kJ/mole to 127 kJ/mole, we can explain this increase by the increase of crystallinity and a reorganization of polymeric chains of the HDPE material. Conversely, a slight decrease of 5 kJ/mole at 43°C was observed, this is the slowing down effect of HDPE at low temperatures, i.e. it requires less activation energy to initiate the chemical reactions. After 36 days of aging, the activation energies obtained at 43 °C are higher and increase with the aging time due to the formation of intermolecular bonds. On the other hand, at 80 °C the energies decrease due to the deterioration of HDPE. The correlation coefficient for all cases studied is greater than 0.97, showing a good prediction of the activation energy and the pre-exponential factor. The decrease of the activation energy can be related to the transfer of molecular chains and oxidation phenomena during the increase of temperature, this phenomenon is already observed by the increase of the crystallinity rate (Fig. 15). These results are in good agreement with the literature (Tippaha et al., 2016; Verho & Vaari, 2022; Lee et al., 2022).

#### 4. Conclusion

In this paper, the problem of the thermal stability of HDPE bottles for food packaging was studied by the process of accelerated thermal aging. The chemical properties during this aging process were analyzed using FTIR. The prediction of the apparent activation energy value and the pre-exponential factor was done by the Kissinger method under TGA analysis.

The mechanical properties and damage during aging were determined by tensile and compression tests, the degradation of mechanical properties during increasing temperature and aging time was observed, and confirmed by the damage laws used.

The results obtained show that HDPE resists better to thermal aging at temperatures below 80°C. Otherwise, thermal instability with deterioration of chemical, physical and mechanical compositions, and properties can occur. Thus, accelerated thermal aging of HDPE is a valued tool for predicting the life of HDPE, and also for evaluating the stability and durability of this material, which allows companies and manufacturers to develop new reliable methods for various packaging applications.

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