

Infectious Behavior in Photo-oxidation of Polymers

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Abstract When a polymer is used together with others, its aging process will be affected by the adjacent polymers. This infectious behavior between polymers makes the aging process more complex than that of an individual material. In this study, infectious behavior in photo-oxidation of polymers was investigated. Polypropylenes (PPs), an unstabilized PP and a commercial PP, were chosen as the infection sources. Six typical polymers, high density polyethylene (HDPE), low density polyethylene (LDPE), polystyrene (PS), polycarbonate (PC), poly(ethylene terephthalate) (PET), and polyamide 6 (PA6), were used as the targets. The degree of oxidation of the targets was evaluated by attenuated total reflection-Fourier transform infrared spectroscopy (ATR-FTIR). An accelerating effect of two infection sources on the photo-oxidation of the target polymers was observed. Potential infectious agents from the infection sources were detected by pyrolysis-gas chromatography-mass spectrometry (Py-GC-MS) and gas chromatography (GC). The acceleration effect of two main infectious agents, *i.e.* acetone and acetic acid, on the photo-oxidation of the commercial PP was verified. The infectious effect of the infection source on the target polymer was considered to be a comprehensive result of the effects of a variety of infectious agents.

Keywords Photo-oxidation; Infection source; Infectious agent; Polypropylene

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INTRODUCTION

In practical applications, different polymers are usually used together rather than individually.^[1–4] When a polymer is used together with others, its aging behavior may be influenced by the adjacent materials. This infectious behavior between polymers, similar to the spreading of a disease, makes the aging process more complex and attracts the attention of researchers. Infection phenomena between polymers during thermo-oxidation have been reported.^[5–8] Celina *et al.*^[9] discovered the spreading of thermo-oxidation between polypropylene (PP) particles for the first time. Then, Ahlblad *et al.*^[10,11] found a similar phenomenon between ethylene-propylene-diene monomer (EPDM) particles. Thereafter, Celina *et al.*^[12] developed a dual stage chemiluminescence (CL) detection system to probe specially for the infectious behavior in thermo-oxidation. PP was found to be able to infect hydroxyl terminated polybutadiene (HTPB) located 25 mm away by carrying infectious agents through oxygen flow. Some potential infectious agents such as benzoyl peroxide, formaldehyde, and acetic acid were placed together with PP and proved to accelerate the thermo-oxidation of PP.^[13] Recently, our group^[14] investigated the cross-infection between unstabilized PP and seven typical polymers. Infectious behavior was demonstrated to be a common phenomenon in thermo-oxidation of various types of polymers and different polymers were observed to exhibit different susceptibility.

Photo-oxidation is another crucial factor that affects the

service life of polymeric materials, especially for outdoor applications.^[15–17] It is possible for different polymers to “meet” with each other in relatively confined space under solar irradiation, for example in automobiles, aircraft, and buildings. Does infectious behavior occur in photo-oxidation as it does in thermo-oxidation? This question has not been given prominence. Our group^[18] reported the acceleration effect of PMMA, PA6, and styrene-butyl acrylate copolymer (PSB) on the photo-oxidation of PP. Pre-oxidized PMMA had higher infectivity than the virgin material. However, compared to the various applications with polymers used together, research on the infectious behavior among different polymers during photo-oxidation is still lacking and, furthermore, the mechanism of the infectious behavior has not yet been clarified.

In this study, the infectious effects of two PPs as the infection sources on photo-oxidation of six target polymers were investigated. An acceleration effect was observed. Infectious behavior was thus demonstrated to be a common phenomenon in photo-oxidation. The infectivity of the two PPs was different, related to the concentration of potential infectious agents that could be generated in their aging process. The acceleration effects of two main infectious agents, *i.e.* acetone and acetic acid, on the photo-oxidation of the commercial PP were verified. The infectious effect of the infection source on the target polymer was considered to be a comprehensive result of the effects of a variety of infectious agents.

EXPERIMENTAL

Materials

Six target polymers, high density polyethylene (HDPE,

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DMDA8920, PetroChina Dushanzi Petrochemical Company), low density polyethylene (LDPE, LD200-BW, PetroChina Yanshan Branch Company), polystyrene (PS, 1001006354, Sigma-Aldrich), polycarbonate (PC, L-1225, Daikin Industries Ltd.), poly(ethylene terephthalate) (PET, SA-1206, Daikin Industries Ltd.), and polyamide 6 (PA6, 1013B, Ube Industries Ltd.) were used. The polymer pellets were hot-pressed into films of about 0.4 mm in thickness. The hot-pressing temperatures were 190 °C for HDPE and LDPE, 250 °C for PS, 270 °C for PC, and 280 °C for PET and PA6. Unstabilized PP powder (PetroChina Panjin Petrochemical Company) and commercial PP pellets (F401, Sinopec) were hot-pressed at 190 °C into films of about 0.3 mm in thickness. The two PPs were used as infection sources. All the films were cut into strips of 40 mm × 15 mm for the subsequent photo-oxidation and characterization. Acetone (AR, Beijing Tongguang Fine Chemical Co., Ltd.) and acetic acid (AR, Beijing Chemical Works) were used as received.

Photo-oxidation

Photo-oxidation of samples was carried out in sealed quartz tubes in a Xenon test chamber (Q-Sun Xe-3, Q-Lab Corporation). The ultraviolet (UV)-visible light with a wavelength range of 295–800 nm and intensity of 0.35 W·m⁻² at 340 nm irradiated the samples vertically. The black panel temperature was 60 °C and the total aging time was 288 h. Target films were photo-oxidized individually (Fig. 1a) or together with a PP film (Fig. 1b). In the latter case, the distance between them was about 10 mm. When a PP film was aged with acetone or acetic acid, the total aging time was 168 h. 20 µL of liquid acetone or acetic acid was injected into the tube before aging and did not contact the PP film directly (Fig. 1c).

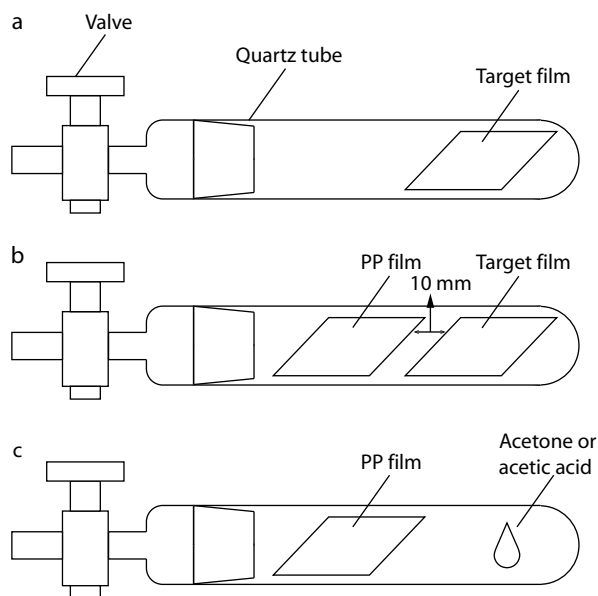


Fig. 1 Location of samples in the quartz tube: (a) A target film aging individually; (b) A target film aging with a PP film; (c) A PP film aging with acetone or acetic acid.

Attenuated Total Reflection-Fourier Transform Infrared Spectroscopy (ATR-FTIR)

ATR-FTIR spectra were obtained using a Thermo-Nicolet 6700 FTIR spectrometer by signal averaging 32 scans at a resolution

of 4 cm⁻¹. An accessory with a diamond crystal was used and the wavenumber range was 600–4000 cm⁻¹. Three different positions on a film were tested and averaged. For PP, carbonyl and hydroxyl indices were calculated using the band areas of carbonyl and hydroxyl groups divided by the area of the band at 2722 cm⁻¹ as the reference.^[19] For target polymers, the relative intensities of C=O groups were calculated using the C=O area divided by the areas of reference bands at 1471 cm⁻¹ for HDPE^[20] and LDPE,^[21] at 1601 cm⁻¹ for PS,^[22] and at 1462 cm⁻¹ for PA6;^[23] the relative intensities of C—O groups were calculated using the C—O area divided by the areas of reference bands at 1503 cm⁻¹ for PC^[24] and at 1505 cm⁻¹ for PET.^[25]

Pyrolysis-Gas Chromatography-Mass Spectrometry (Py-GC-MS)

An EGA/PY-3030D multi-shot pyrolyzer (Frontier Laboratories, Ltd.) was installed on a gas chromatography-mass spectrometer (GCMS-QP2010 SE, Shimadzu). Flash evaporation was carried out to detect volatile oxidation products in samples by heating 10.0 mg of film at 300 °C for 30 s. The volatile components were carried by helium (He) carrier gas through an Ultra Alloy-5 column (5% diphenyl and 95% dimethylpolysiloxane, 30.0 m, i.d. 0.25 mm × 0.05 µm stationary phase thickness), and the mixture was separated. The oven temperature was held at 50 °C for 1 min, then raised at a rate of 8 °C·min⁻¹ to 300 °C, and held for 30 min. Each component was identified by the MS detector with an electron ionization (EI) source in a mass range of *m/z* 33–600. The MS spectra were compared to standards in the NIST 11 database composed of ~2.4 × 10⁵ standard spectra. Two parallel measurements were carried out for each sample.

Gas Chromatography (GC)

GC analysis was conducted in a 7890B GC System (Agilent Technologies) to detect the gaseous degradation products of samples in the quartz tube. 200 µL of gas was extracted from the quartz tube by an injector and injected into the GC system. The gaseous components were carried by argon (Ar) carrier gas through an G3591-81023 column (0.5 m in length, o.d. 3.175 mm, i.d. 2 mm), and the mixture was separated. The oven temperature was held at 50 °C for 4 min, then raised at a rate of 20 °C·min⁻¹ to 250 °C, and held for 20 min. A flame ionization detector (FID) was used to detect carbon monoxide (CO) and hydrocarbons. A thermal conductivity detector (TCD) was used to detect hydrogen (H₂) and carbon dioxide (CO₂).

RESULTS AND DISCUSSION

Infectious Effect of Unstabilized PP

Changes in functional groups of the target polymers during photo-oxidative aging were detected by ATR-FTIR. Figs. 2(a)–2(d) show the ATR-FTIR spectra of HDPE, LDPE, PS, and PA6 before and after aging, with and without unstabilized PP. The carbonyl bands of these four polymers were increased after aging, indicating the oxidation of C—H groups in the macromolecular chains. With the presence of the unstabilized PP, the carbonyl bands were increased further. Figs. 2(e) and 2(f) show the ATR-FTIR spectra of PC and PET. Changes in the spectra were not obvious. Since it was demonstrated that C—O bonds in PC and PET were fractured during photo-oxidative aging,^[26] the relative intensities of C—O groups were used to show the changes.

The relative intensities of functional groups in the target po-

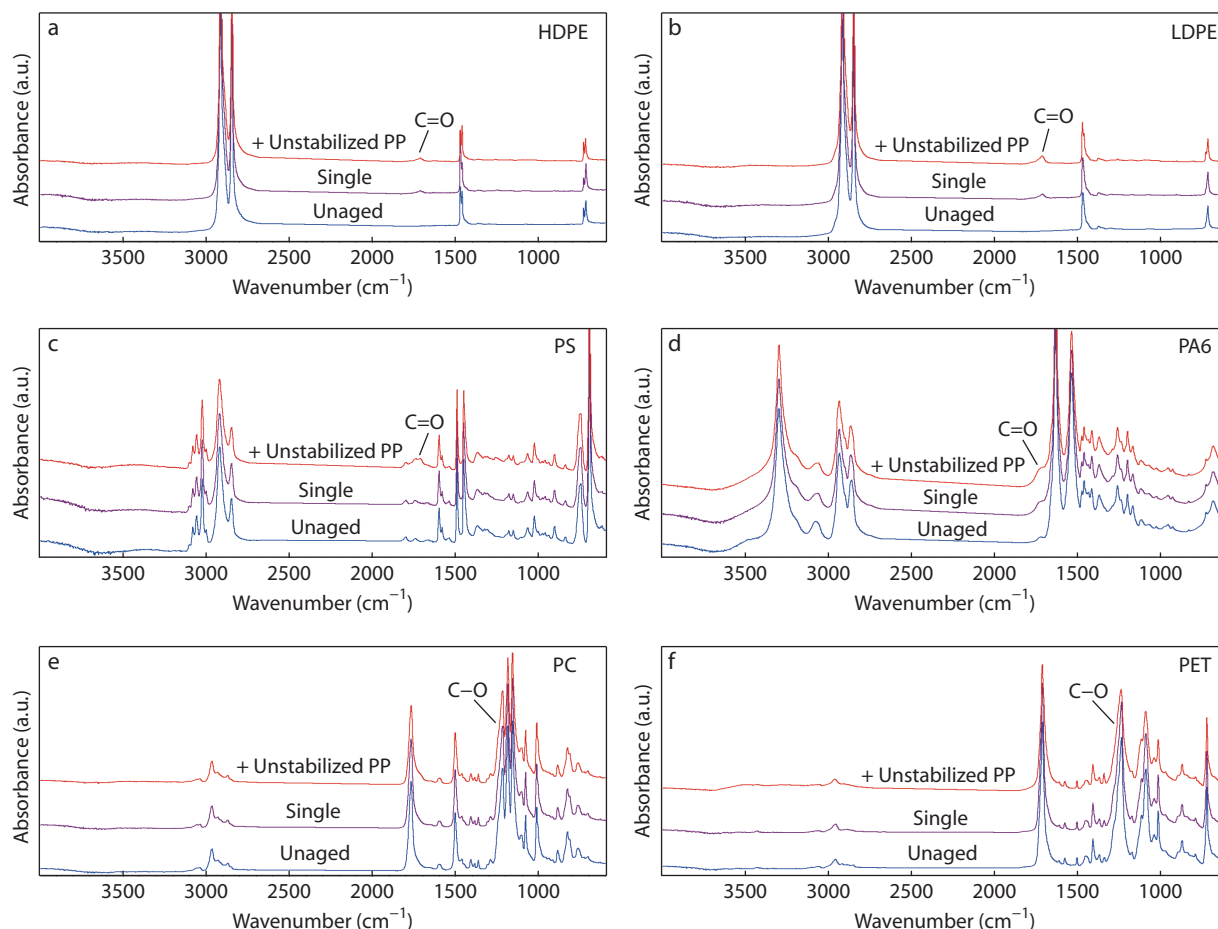


Fig. 2 ATR-FTIR spectra of (a) HDPE, (b) LDPE, (c) PS, (d) PA6, (e) PC, and (f) PET before and after aging, with and without unstabilized PP. (The spectra are normalized by respective reference bands, at 1471 cm^{-1} for HDPE and LDPE, at 1601 cm^{-1} for PS, at 1462 cm^{-1} for PA6, at 1503 cm^{-1} for PC, and at 1505 cm^{-1} for PET.)

lymers before and after aging, with and without unstabilized PP, calculated according to Fig. 2, are summarized in Fig. 3.

HDPE, LDPE, PS, and PA6 (Figs. 3a–3d) were easily photo-oxidized to generate carbonyl-containing products in the macromolecular chains. The intensities of C=O groups increased obviously after aging individually. When aged with the unstabilized PP, the increase was more remarkable. The accelerating effect of the unstabilized PP on HDPE, LDPE, PS, and PA6 could be observed clearly.

The intensities of C—O groups in PC and PET (Figs. 3e and 3f) remained almost unchanged after aging individually, attributed to their high stability during photo-oxidative aging. PC was more stable, showing only a tiny decrease even after aging with the unstabilized PP. The intensity of C—O groups in PET was decreased after aging with the unstabilized PP, indicating fracture of the macromolecules under the influence of the unstabilized PP.

Infectious Effect of Commercial PP

Commercial PP was used as another infection source. The relative intensities of functional groups in the target polymers before and after aging, with and without commercial PP, are collected in Fig. 4. After aging with the commercial PP, the intensities of C=O groups in HDPE, LDPE, PS, and PA6 were higher

than when they were aged individually, and the intensity of C—O groups in PET was lower. PC was extremely stable and not affected by the commercial PP, in which the intensity of C—O groups remained almost unchanged in both conditions.

Similar to the unstabilized PP, the commercial PP accelerated photo-oxidation of the target polymers. Nevertheless, the acceleration effects of these two infection sources were not identical. The acceleration effect was evaluated by the acceleration ratio, which was defined as the ratio of the absolute values of the difference between the intensities of functional groups with and without an infection source to the intensity in the individual condition, as shown in the following equation:

$$\text{Acceleration ratio (\%)} = \frac{|I_T - I_S|}{I_S} \times 100 \quad (1)$$

where I_T is the relative intensity of functional groups with an infection source and I_S is the relative intensity of functional groups in the individual condition. The calculation results are plotted in Fig. 5. The acceleration ratio was higher with the unstabilized PP than with the commercial PP for all the target polymers. Especially for PS, the acceleration ratio with the unstabilized PP was 4.6 times that with the commercial PP. Higher acceleration ratio with the unstabilized PP suggested the higher

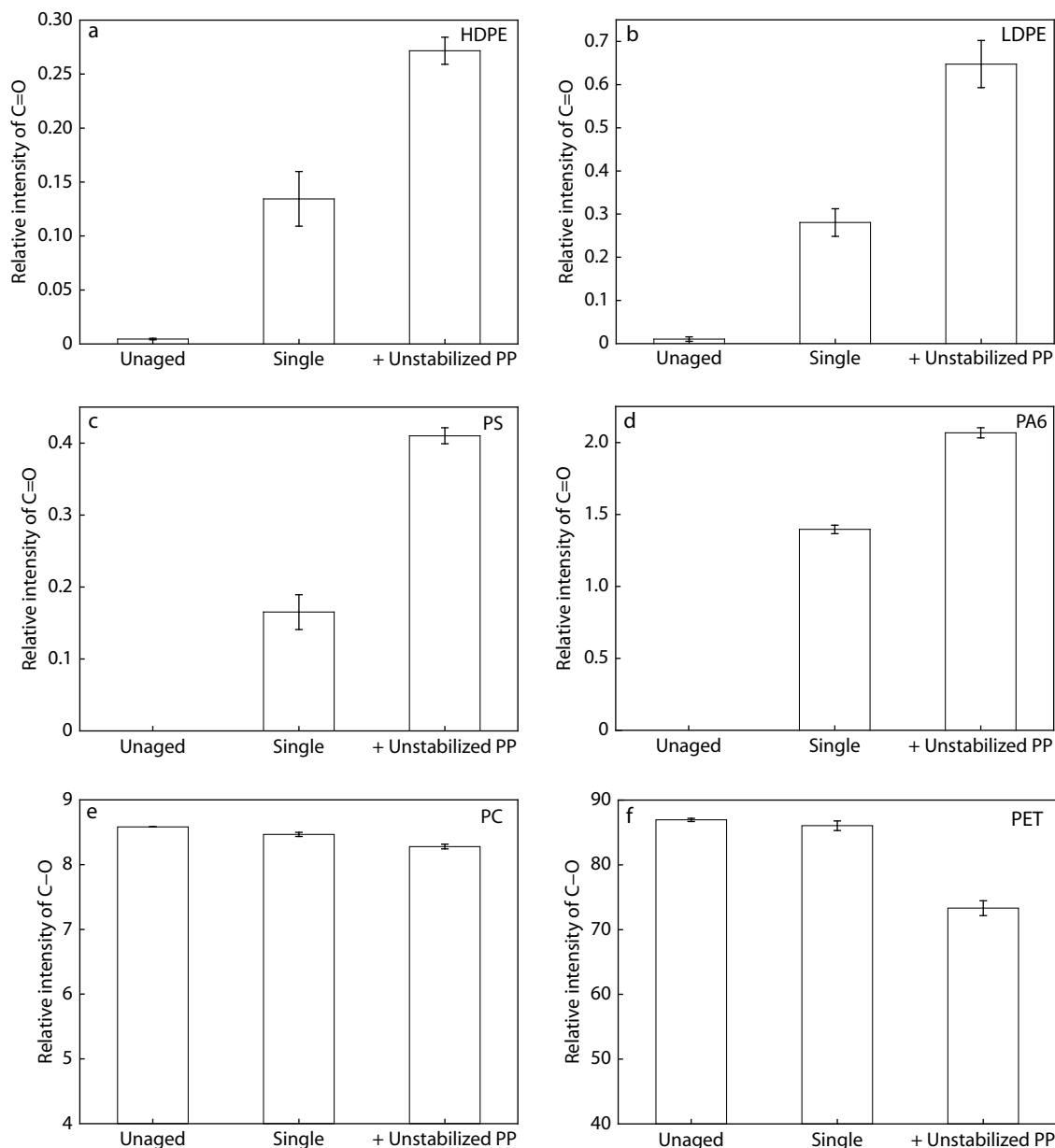


Fig. 3 Relative intensity of C=O in (a) HDPE, (b) LDPE, (c) PS, and (d) PA6, and relative intensity of C—O in (e) PC and (f) PET before and after aging, with and without unstabilized PP.

infectivity of the unstabilized PP. Compared to the unstabilized PP, the commercial PP was a relatively weak infection source.

Also, aging conditions might affect the infectivity of the same infection source. According to our previous study,^[14] the unstabilized PP accelerated the thermo-oxidation of HDPE, LDPE, PS, and PA6, but its influence on PET was negligible. Here, in the photo-oxidative aging process, the acceleration effect of both PPs on PET could be observed clearly. The intensity of C—O groups was decreased by 9.2% with the commercial PP and by 14.8% with the unstabilized PP.

Susceptibilities of different target polymers could also be compared in Fig. 5. With the unstabilized PP, the susceptibilities of HDPE, LDPE, and PS were much higher than those of PA6 and PET. With the commercial PP, the susceptibility of

LDPE was higher than HDPE, PS, PA6, and PET. PC was not sensitive to the two infection sources.

Potential Infectious Agents

In the photo-oxidative aging process, the infection source and the target polymer did not contact each other. Therefore, the infectious agents were assumed to exist in the atmosphere around the target polymer. PP, as the infection source, generated a large amount of small molecule products when photo-oxidized, either volatile or gaseous molecules.^[27,28] They might act as infectious agents.

A variety of volatile degradation products were detected and identified in the flash evaporation-gas chromatograms of the two PPs after photo-oxidative aging individually, including alkenes, ketones, carboxylic acids, and esters (Fig. 6).

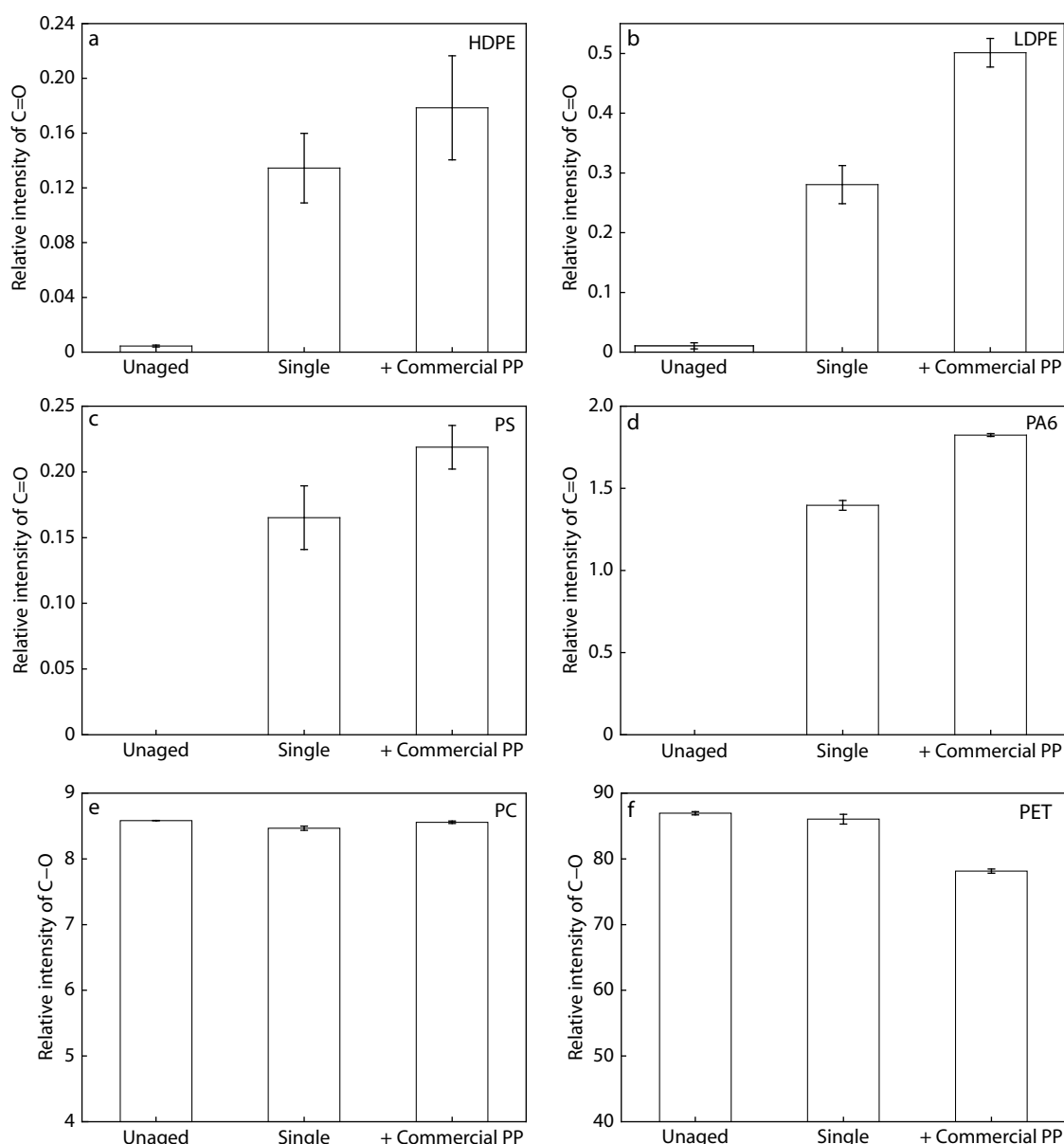


Fig. 4 Relative intensity of C=O in (a) HDPE, (b) LDPE, (c) PS, and (d) PA6, and relative intensity of C—O in (e) PC and (f) PET before and after aging, with and without commercial PP.

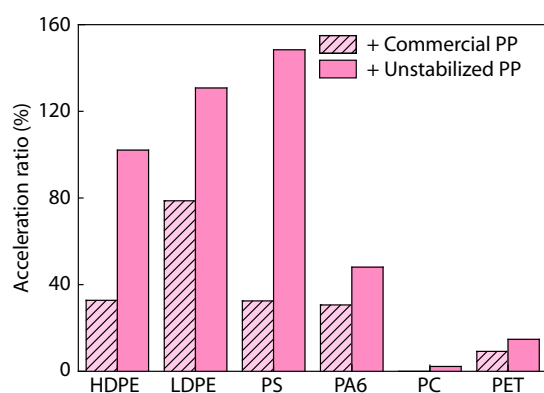


Fig. 5 Acceleration effect of unstabilized and commercial PP on the photo-oxidation of target polymers.

Acetone and acetic acid were the two main volatile degradation products. The intensities of the volatile degradation products in the commercial PP were lower than those in the unstabilized PP due to the presence of antioxidants and the resulting lower degree of oxidation. During photo-oxidative aging, the degradation products of the infection source volatilized and migrated to the target polymers. As shown in Fig. 7, acetone and acetic acid were found in almost all the target polymers after aging with two PPs, and their intensities were higher with the unstabilized PP than with the commercial PP.

The gaseous degradation products of unstabilized and commercial PP during photo-oxidative aging were detected by GC. Six typical products were identified as shown in Fig. 8: H_2 , CO_2 , CO , methane (CH_4), ethylene (C_2H_4), and ethane (C_2H_6). The intensities of gaseous degradation products were higher

in the unstabilized PP than in the commercial PP.

With a large amount of volatile and gaseous degradation

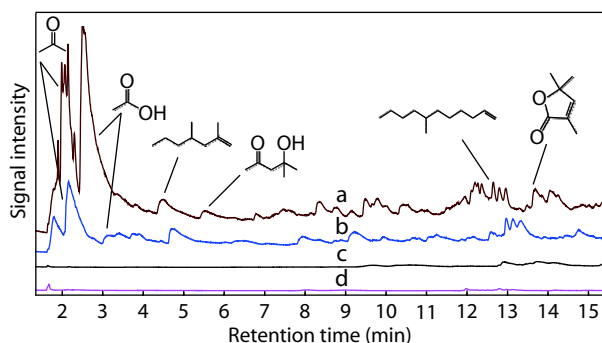


Fig. 6 Flash evaporation-gas chromatograms of (a) unstabilized PP and (b) commercial PP after photo-oxidative aging individually; Flash evaporation-gas chromatograms of (c) unstabilized PP and (d) commercial PP before aging as the reference. (The chromatograms are normalized by the weight of the samples.)

products, these two infection sources provided an atmosphere rich in photochemically active small molecules that might be involved in the photo-oxidation of the target polymers. The unstabilized PP produced higher concentrations of volatile and gaseous species, corresponding to higher infectivity, as shown in Fig. 5.

Effects of Acetone and Acetic Acid on PP

Acetone and acetic acid are two main volatile degradation products of PP. Since it has been demonstrated that acetic acid accelerated the thermo-oxidation of PP,^[13] how could they influence the photo-oxidation of polymers?

In the study of infectious behavior of polymers, PP was the preferred choice as either the infection source or the target polymer,^[12–14,18] because it is easily oxidized and has both high infectivity and susceptibility. Here, the commercial PP was chosen as the target polymer to verify the effects of two main potential infectious agents, *i.e.* acetone and acetic acid. 20 μL of liquid acetone or acetic acid was injected into the tube before aging. They could volatilize and be absorbed by

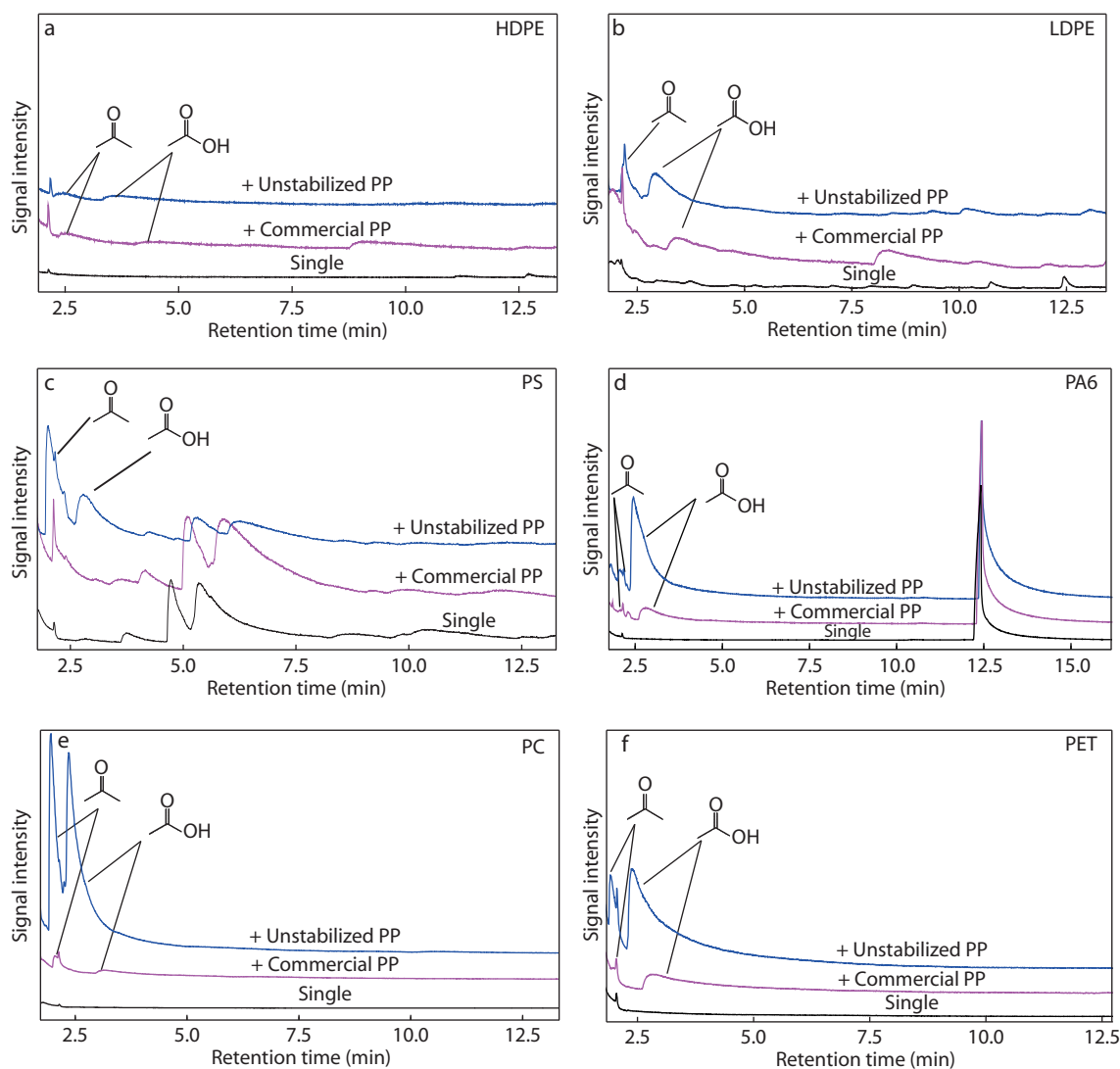


Fig. 7 Flash evaporation-gas chromatograms of target polymers after photo-oxidative aging with and without two infection sources: (a) HDPE, (b) LDPE, (c) PS, (d) PA6, (e) PC, and (f) PET. (The chromatograms are normalized by the weight of the samples.)

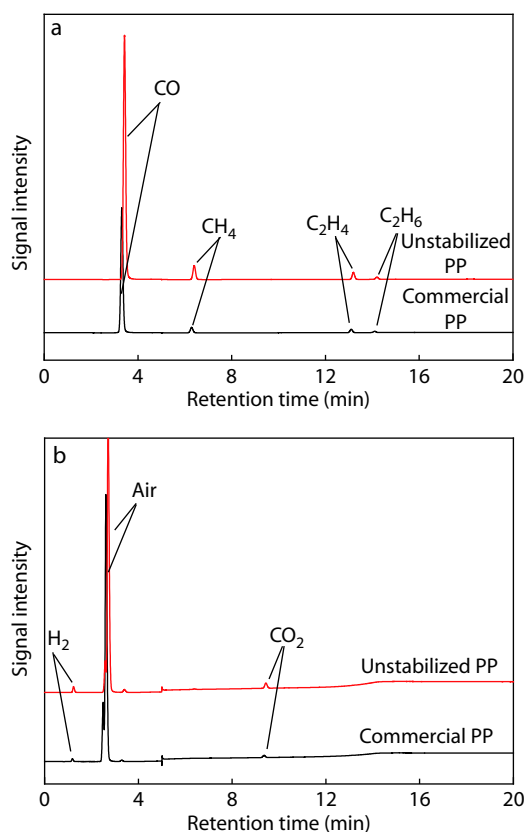


Fig. 8 Gas chromatograms of gaseous degradation products of two infection sources after photo-oxidative aging individually: (a) Signal from FID detector; (b) Signal from TCD detector. (The chromatograms are normalized by the volume of the samples.)

the commercial PP, simulating the spreading of infectious agents from an infection source to a target polymer.

Fig. 9 shows the ATR-FTIR spectra of commercial PP before and after aging individually and with acetone or acetic acid. Both the carbonyl and hydroxyl bands of the commercial PP were increased after aging, indicating the oxidation of C—H groups and the generation of carbonyl- and hydroxyl-containing oxidation products in the macromolecular chains. Carbonyl and hydroxyl indices are shown in Fig. 10. Both acetone and acetic acid led to higher carbonyl and hydroxyl indices of the commercial PP after photo-oxidative aging. The influence of acetone was greater. With acetone, the carbonyl index was increased by 147%, and the hydroxyl index by 34%, compared with the value in the individual condition.

A carbonyl band existed in acetone and acetic acid, and a hydroxyl band existed in acetic acid, so absorbed acetone or acetic acid might also contribute to the increase of the carbonyl and hydroxyl indices of the commercial PP. In order to distinguish the infectious effect and the absorption contribution, gaseous degradation products were also used to evaluate the effects of acetone and acetic acid on the photo-oxidation of the commercial PP (Fig. 11). The intensities of all the gaseous degradation products were higher with acetone or acetic acid than those in the individual condition. Although photo-degradation products of acetone included H₂, CO, and CH₄, and photo-degradation products of acetic acid included

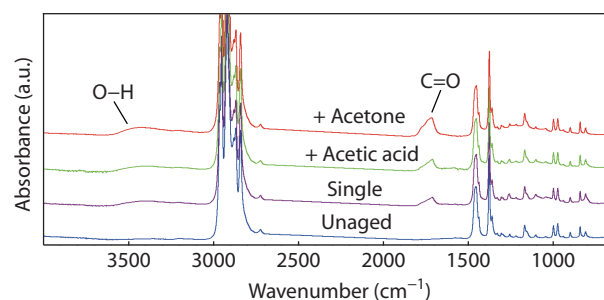


Fig. 9 ATR-FTIR spectra of commercial PP before and after aging individually and with acetone or acetic acid. (The spectra are normalized by the reference peak at 2722 cm⁻¹.)

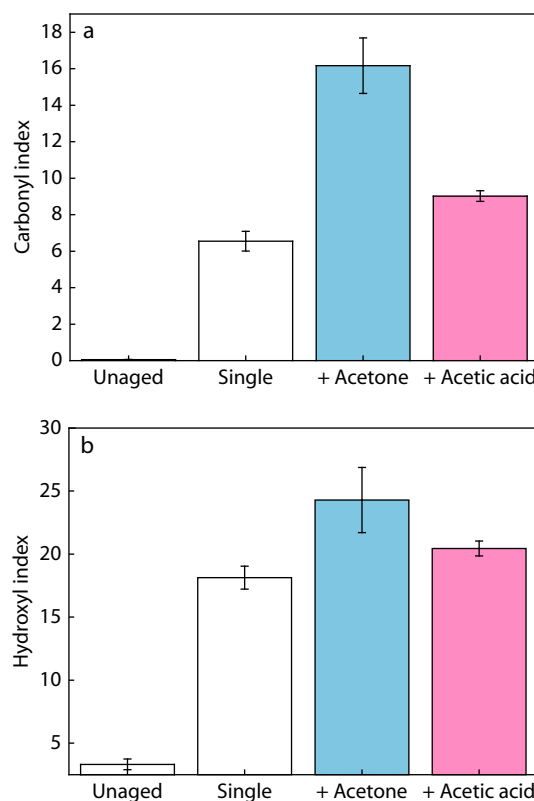


Fig. 10 (a) Carbonyl and (b) hydroxyl indices of commercial PP before and after aging individually and with acetone or acetic acid.

CO₂ and CH₄, there was other strong evidence of infectious effects of acetone and acetic acid. Acetone led to much higher intensities of CO₂, C₂H₄, and C₂H₆, while acetic acid led to much higher intensities of H₂, CO, C₂H₄, and C₂H₆. The significant increase of these gaseous degradation products demonstrated the acceleration effect of acetone and acetic acid on photo-oxidation of the commercial PP. C₂H₄ and C₂H₆ were highlighted because they were not generated from either acetone or acetic acid. Therefore, the higher intensities of C₂H₄ and C₂H₆ were exclusively attributed to the accelerated degradation of the commercial PP.

According to the above results, acetone and acetic acid were indeed demonstrated to be efficient infectious agents. Acetone exhibited extremely high efficiency, probably due to its high photochemical activity to generate radicals easily un-

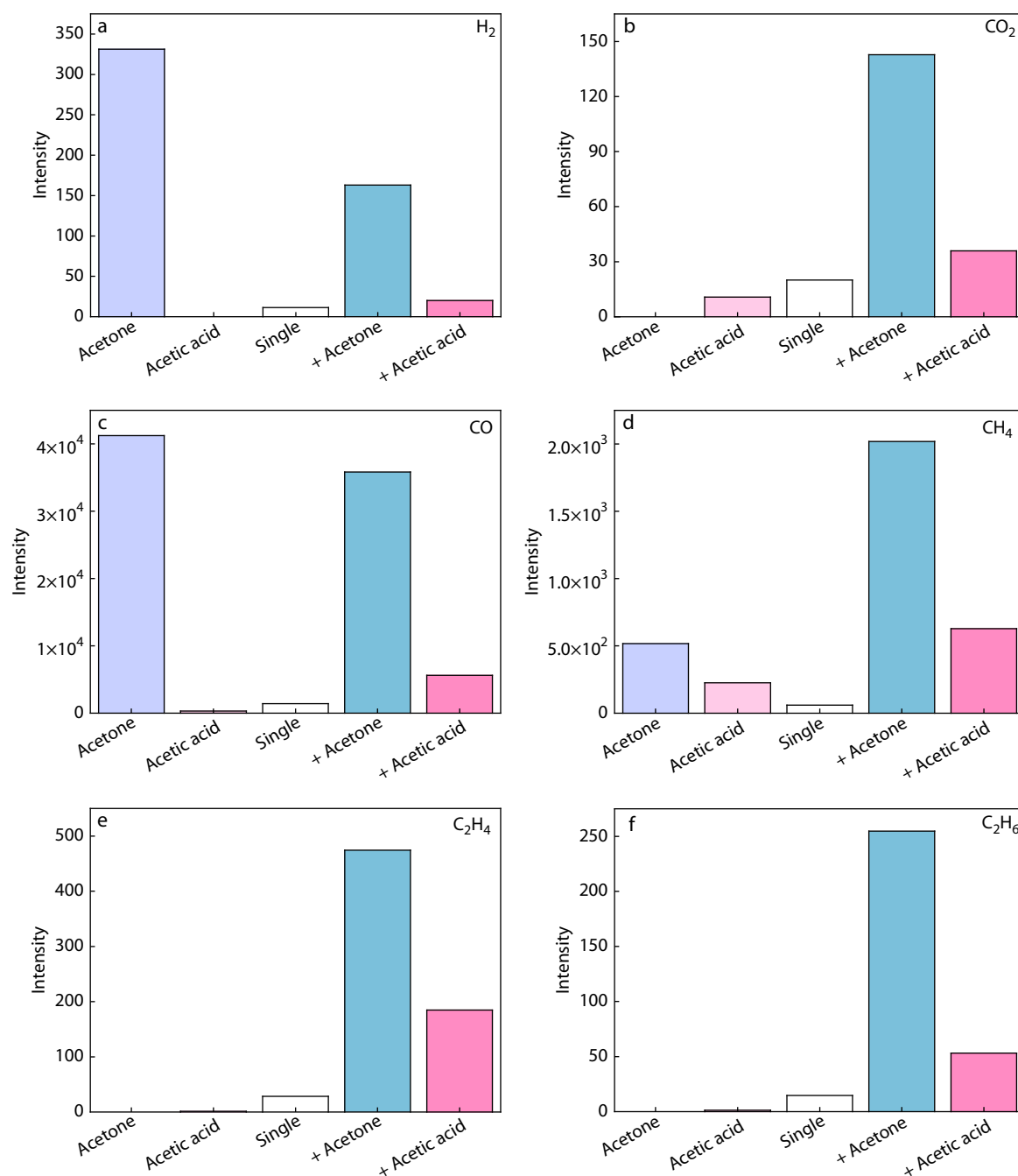


Fig. 11 Intensity of gaseous degradation products of commercial PP after aging individually and with acetone or acetic acid: (a) H_2 , (b) CO_2 , (c) CO , (d) CH_4 , (e) C_2H_4 , and (f) C_2H_6 . (Gaseous degradation products of acetone and acetic acid under the same aging condition were employed as the reference.)

der UV irradiation.^[29] In addition to acetone and acetic acid, other degradation products from the infection source might also act as infectious agents. Their effects deserve further investigation.

CONCLUSIONS

Infectious behavior in photo-oxidation of polymers was investigated. Unstabilized PP and commercial PP were chosen as two

types of infection sources. Their acceleration effects on the photo-oxidation of six target polymers were demonstrated. The two infection sources exhibited different infectivities. The influence of the unstabilized PP was greater than that of the commercial PP, which was attributed to the higher concentration of potential infectious agents generated by the unstabilized PP with higher degree of oxidation. The potential infectious agents included volatile and gaseous degradation products from the infection source, which could be absorbed by the target poly-

mers and involved in the photo-oxidative aging process of those polymers. Two main infectious agents, *i.e.* acetone and acetic acid, were aged together with the commercial PP to verify their effects. Both acetone and acetic acid accelerated the photo-oxidation of the commercial PP. The acceleration effect of acetone was greater, probably due to its high photochemical activity to easily generate radicals under UV irradiation. The infectious effect of the infection source on the target polymer was a comprehensive result of the effects of a variety of infectious agents. In order to clarify the underlying mechanism of infectious behavior during aging of polymers, the effects of various infectious agents on different polymers need further investigation.

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