Research Article

Difference in polypropylene fragmentation mechanism between marine and terrestrial regions

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Abstract

Two kinds of marine polypropylene (M1-PP and M2-PP) and one land PP (L-PP) samples were collected from two beaches and land in Japan, respectively, to study the fragmentation mechanisms. Delamination was observed on both M1-PP and M2-PP surfaces. Moreover, there was no delamination but an abrasion patch structure on the surface of L-PP. The delamination was studied using an advanced oxidation process-degraded PP as the marine PP model. The number and shape of cracks varied with an increase in degradation time. The fluctuations in the values and ratios of the carbonyl index as well as the weight change ratio were due to repeated oxidation and delamination. We found that the delamination behavior depends on the oxidation state. Poly(oxyethylene)$_8$ octylphenyl ether (POE8) surfactant treatment caused the delamination to speed up, which is a typical characteristic of polyolefin environmental stress cracking (ESC). These results reveal that delamination is based on ESC.

Article Highlights

- Two kinds of marine and one land polypropylene (PP) samples were collected from two beaches and land, respectively, to study the fragmentation mechanisms.
- Delamination was observed on both of marine PP surfaces. Moreover, there was no delamination but an abrasion patch structure on the land PP surface.
- We found that the delamination was based on environmental stress cracking mechanism by employing a marine PP model.

1 Introduction

Several plastic materials have yielded for a long time, resulting in the accumulation of a high amount of litter in the marine environment [1–15]. Plastic litter has become one of the major problems in the marine environment. In particular, microplastics (MP) regenerated by larger plastic debris have raised a growing environmental concern ([15–19]. Polypropylene (PP), polyethylene (PE), and expanded polystyrene (EPS) have low

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densities, and they are hugely produced worldwide. The MPs float on the sea surfaces. Besides, they are not biodegradable; hence, they semipermanently remain in the marine environment. The MP production place has not been clarified yet. For instance, one group has been generated in the terrestrial region (on land) and others in the sea. In some cases, MP partially leaves the sea to the atmosphere [20]. They occur in various places and circulate. Halle et al. reported the MP formation mechanism in the sea [9], however, the driving force for the fragmentation is not yet clarified. It has been reported that several kinds of MP are formed in water by light exposure in the visible and/or UV regions [21–23]. In our previous study, a PP film photodegradation test was performed in water with a specific photocatalyst under visible light irradiation, and MP particles were obtained by planar exfoliation [24]. We infer that MP formation is certainly associated with autoxidation and water. However, the detailed formation mechanism has not been clarified yet. It is unclear MPs are formed in the sea. The mechanism does not explain MP formation on dried terrestrial regions. The difference between the MP formation mechanism in marine and terrestrial regions is unknown. There is a need to clarify the difference in the MP formation mechanisms by studying the surfaces of weathered plastic debris in the sea and on land. The obtained hypothesis should be verified using an imitation model of the actual phenomenon.

In this study, two samples of marine PP and one land PP were collected to study the MP formation mechanism. The obtained mechanism was verified using an advanced oxidation process (AOP)-degraded PP as the marine PP model. The fragmentation mechanism of marine plastic litter has been presented for the first time in the world.

2 Experiment

2.1 Materials

PP (product name: J-700GP) was supplied by Prime Polymer Co., Ltd., Japan. The melt flow rate and density were 8 g/10min at 230 °C and 0.9 g/cm^3. Potassium hydroxide (KOH) and potassium persulfate ($K_2S_2O_8$) and poly(oxyethylene)$_8$ octylphenyl ether (POE8) were purchased from Wako Pure Chemical Industries., Japan.

2.2 PP sample collection

Two kinds of marine PP litter pieces (M1-PP and M2-PP) were collected from Iguchihama beach at Tsushima island and Head land beach at Chigasaki city in Japan, respectively. One kind of terrestrial PP litter pieces (L-PP) was done from a dried island of Arakawa River at Tokyo in Japan. The detailed locations were shown in Fig. 1. A pyrolysis gas chromatography/mass (py-GC/MS: SHIMADZU GCMS-QP2010 PLUS, Japan) analysis was used to discriminate the PP litter pieces from other plastics. In Figure S1, the py-GC/MS chart of M1-PP was shown as representative results. All of them exhibited typical homo isotactic PP spectra. These pieces were pretreated using KOH 1N aqueous solution.

2.3 Advance oxidation process (AOP) degradation

The PP was molded into a thin film (50×50×0.065 mm) by compression molding at 180 °C under 10 MPa for 10 min, and then the film was cut to 10×10×0.065 mm. In the case of heat pretreatment, the film was performed using an oven at 130 °C for 144 h in air.

Fig. 1 Sampling locations of M1-PP , M2-PP and L-PP samples. Letters and numbers represent sampling sites, respectively. Local names and other information are given in Text
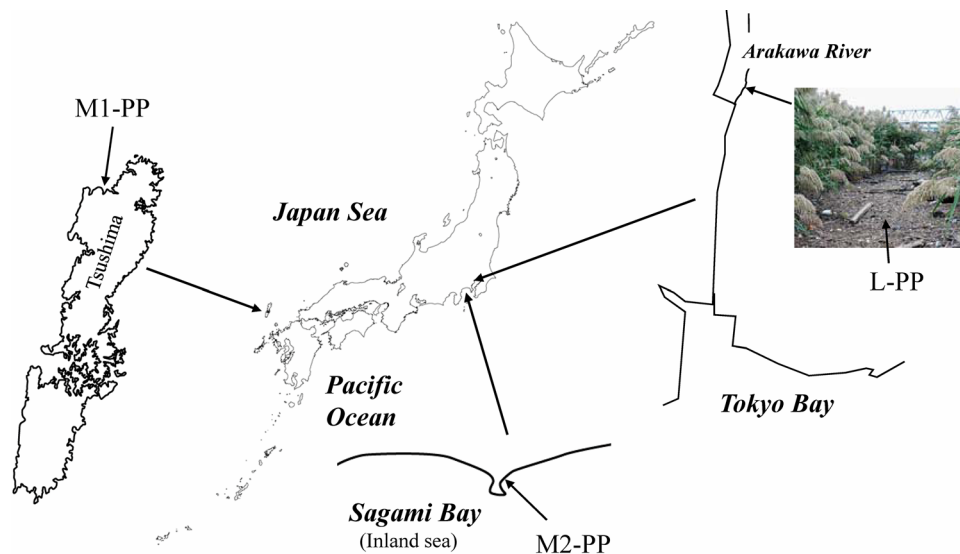
They were employed as "with heat pretreatment and without heat pretreatment" to a water oxidative degradation test, respectively. The AOP degradation procedure was according to some reports [25, 26]. Each three pieces of the films were put into a 100 ml glass vessel equipped with a 20 ml aqueous solution containing 0.54 g $K_2S_2O_8$ at ca. 65 °C for 12 h under stirring with a stirrer tip speed of ca. 100 rpm, and the equal amount of $K_2S_2O_8$ aqueous solution was replaced every 12 h due to the consumption of the oxidant. The pieces were filtrated and were washed every 2 days to prevent the accumulation of K_2SO_4 in the solution and were again introduced into the fresh $K_2S_2O_8$ aqueous solution. The films were continuously treated according to the above procedures for different time periods. The oxidative degradation using $K_2S_2O_8$ was named as "AOP".

2.4 Poly(oxyethylene)$_8$ octylphenyl ether (POE8) surfactant treatment

The POE8 surfactant treatment of the 9 days-AOP degraded sample with the heat pretreatment was performed using a 100 ml glass vessel equipped with a 50 ml aqueous solution containing 5 wt%-POE8 at r.t. for 2 days.

2.5 Weight measurement of PP and pre-PP films treated by water oxidative degradation using $K_2S_2O_8$

The 3 pieces (ca. 15 mg) of the film at every one day were carefully picked up from the glass vessel with tweezers and were rinsed with methanol. After drying with vacuum oven at 60 °C for 7 h, the weight was measured.

2.6 Characterization

The morphology of PP sample surface was observed through SEM (JEOL SM-7500FAM, Japan), polarized optical microscope (POM: Nikon ECLIPSE 50/POL, Japan) and 3D digital microscope (Keyence VHX-7000, Japan). Then the 3D topological profile was subsequently evaluated on the Keyence VHX-7000.

The Fourier transform infrared (FT-IR) spectra were measured using an FT-IR spectrometer (Jasco IRT-5200, Japan) with an Attenuated Total Reflectance (ATR) diamond accessory at a resolution of 4 cm^{-1} over the full mid-IR range (400–4000 cm^{-1}).

3 Results and discussion

Figure 2 shows the photograph, polarized optical microscope (POM) and microphotographs of M1-PP, M2-PP, and L-PP. M1-PP and M2-PP were collected from different beaches, and L-PP was collected from a place (see Fig. 1). The surface of M2-PP was too rough to be observed with POM; it was observed using a microscope. Tile-like cracks were observed in the entire surface of M1-PP. Meanwhile, a rectangular crack pattern was observed on the L-PP surface. Crack growth causes fragmentation. The difference in the crack patterns suggests that the fragmentation mechanisms in the two samples are different. Several small flakes called "delamination" [27, 28] were observed on the M2-PP surface. Besides, the M2-PP surface showed a complicated structure composed of horizontal planes and protuberances, such as mirrors and hills. Figures S2 and S3 show the photographs of M2-PP. The 3D topological profiles show a step structure on the surfaces, which is attributed to multiple factors. Oxidative degradation is a dominant factor that induces fragmentation in PP [29]. However, a simple degradation model based on autoxidation cannot determine the formation mechanism of such a step structure. Figure 3 shows the SEM micrographs of M1-PP and L1-PP. Mirror planes and hills were distinctly observed in the SEM photograph of the M1-PP surface. The sampling locations are very far apart (over 1000 km), and they face on the open-sea with high wave intensity and on inland-sea with weak wave intensity (Fig. 1), respectively. Various factors, such as voyage length, exposure history, and wave intensity to wave action, were different among the samples. The mirror plane area, hill magnitude, and delamination amount were dependent on the sampling location. For instance, M2-PP showed considerably rough surfaces compared with M1-PP. Moreover, step structures were observed on M1-PP and M2-PP, regardless of the sampling location. Weinstein et al. observed the delamination behavior in PP and PE degradation in a salt marsh [27, 28]. The surface morphology of PP beach debris was also studied by Cooper et al., and they reported deep and defined fractures, indicating the occurrence of mechanical-related degradation [30]. The morphology of M1-PP(a) is similar to that reported by Cooper et al. We infer that the step structure is inherent in MP obtained in the sea and sea-like places. Waves in the sea continuously apply stresses on the debris, leading to environmental stress cracking (ESC). Delamination likely occurs in the sea due to ESC [31]. The morphology of the L-PP surface is considerably different from that of M1-PP, as shown in Fig. 3. It is composed of linear fractures and patches, and defined step structures were not observed. It implies that L-PP was exposed to common weathering without ESC. The L-PP fragmentation is attributed to weathering and surface abrasion by wind and other factors. These results suggest that there the fragmentation mechanism differs in marine and terrestrial regions.

The delamination mechanism is based on ESC. This hypothesis needs to be thoroughly tested using an
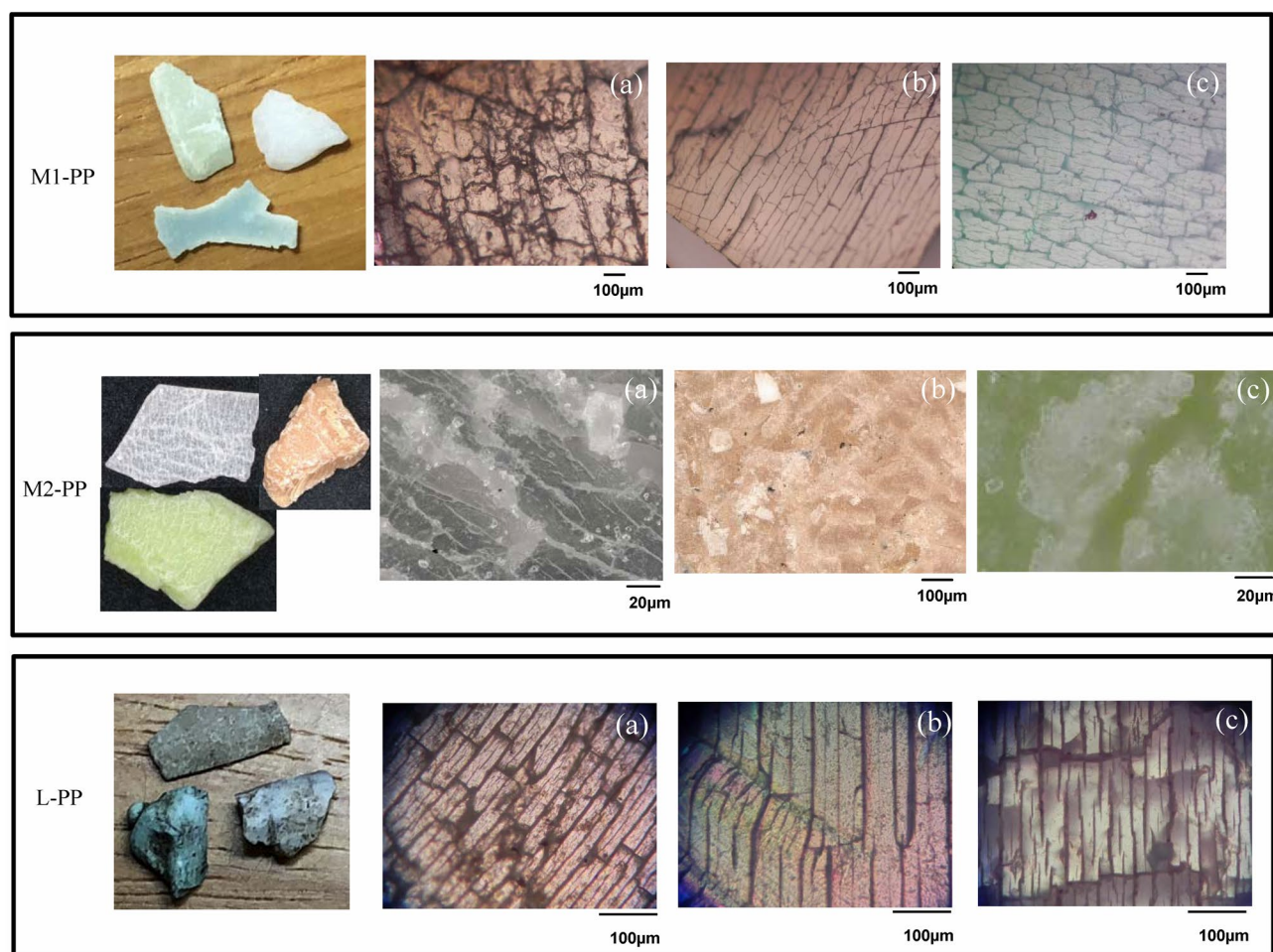
Fig. 2 Photographs, POM and microphotographs of M1-PP, M2-PP and L-PP. M1-PP and L-PP: POM. M2-PP: Microscope

imitation model of the actual phenomenon. PP degradation is initiated by autoxidation, as shown in Fig. 4 [32, 33], leading to the onset of ESC. The initiation rate of autoxidation is considerably slow under sunlight irradiation. Therefore, $K_2S_2O_8$ was employed as the initiator [25, 26]. Highly reactive sulfate radicals are generated by cleaving the peroxide bond in $K_2S_2O_8$ and attacking PP chains. Autoxidation effectively advances, leading to the growth of cracks at the surface of the PP sample. The crack formation is related to the chemi-crystallization process, of which the driving force is autoxidation [34, 35]. Linear cracks named "linear fractures" are frequently observed on the surfaces of PP and PE debris [30, 36] and they were also observed on our PP debris samples. The cracks grow along flow-lines, i.e., the polymer chain orientation [34, 35]. The orientation generates a residual stress point to initiate cracks. An oriented surface part supplies linearly marked cracks, such as linear fracture, as shown in Figure S4(a). Moreover, the orientation degree depends on the depth direction of the sample. The surface has a higher orientation degree than

the inner part. Therefore, the orientation becomes weaker as autoxidation advances to the inner part. The number and shape of cracks change with an increase in degradation time when ESC occurs on AOP-degraded PP as an imitation model of marine MP degradation. Longer AOP-degraded PP showed a mixture of tile- and rectangle-like crack patterns, as shown in Figure S4(b). The tile-like crack pattern represents crack growth around PP spherulites. In our previous work, we confirmed that the PP spherulite structure contributes to the tile-like crack pattern [24]. The occurrence of such a crack pattern is inherent in PP when the orientation does not exist in the matrix. Only the tile-like crack pattern was observed due to the release from the orientation by the heat pretreatment, as shown in Figure S4(c). Linear fracture is formed on the surface by orientation. However, it gradually changes to the tile-like crack pattern. The orientation degree becomes lower down the internal part due to the increase in delamination. FT-IR spectra of the AOP-degraded PP samples reveal that autoxidation, as well as typical weathering of PP, occurred

Fig. 3 SEM micrographs and plausible mechanisms to marine and land PP debris
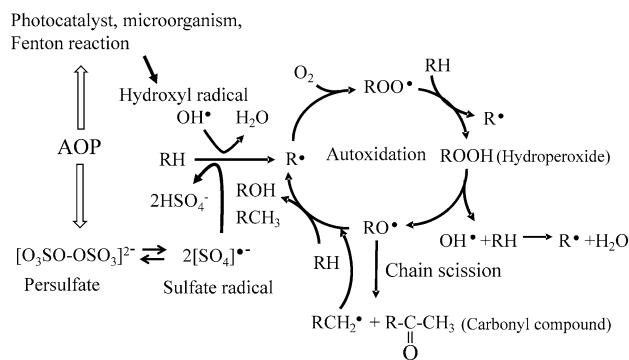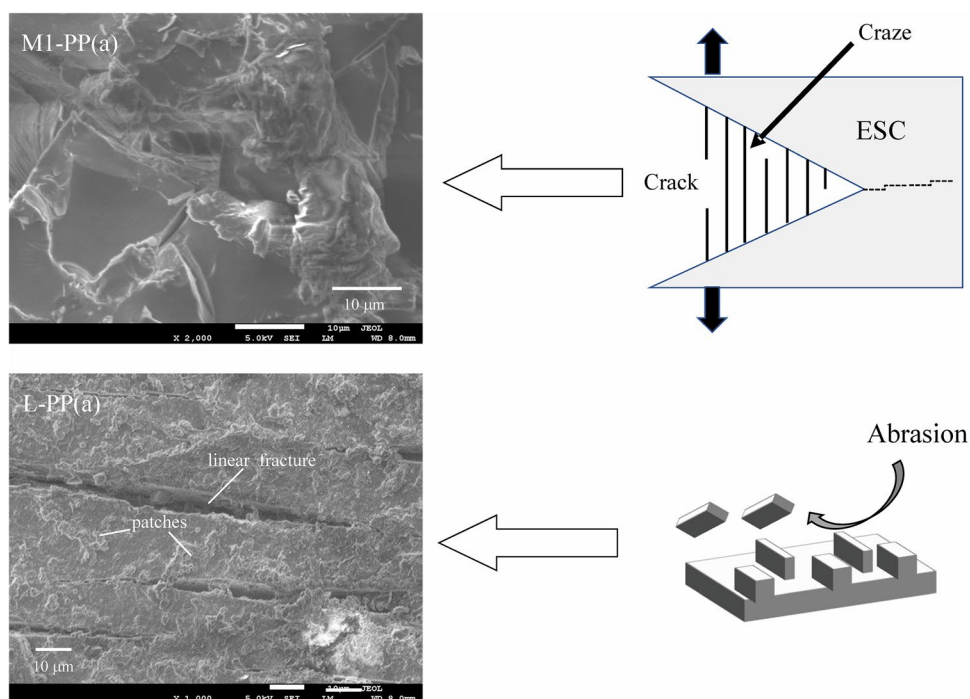

Fig. 4 Autoxidation mechanism initiated by advanced oxidation process (AOP)

(Figure S5). Figure 5 shows the value and change rate of carbonyl index (CI), ratio, and the rate of weight change vs AOP degradation time for PP films with and without heat pretreatment. CI was calculated using the carbonyl group (ca. 1720 cm^{-1})/methylene group (ca. 1452 cm^{-1}) band intensity ratio [37–39]. The positive and negative values, as well as the ratios, are attributed to the repeated oxidation and delamination [24]. In particular, the values and ratios of the samples with no heat pretreatment rise up and down unlike those for heat-pretreated samples. The difference is a result of the initial oxidation degree. The initial CI values were 0.20 and 0.14 with and without heat pretreatment. The heat treatment brings about oxidation and leads to initial delamination speedup. In addition, heat-pretreated samples oxidize to the inside and change to a rather degraded medium than pristine PP. The delamination behavior substantially depends on the oxidation state. Interestingly, the CI and rates of weight change of the sample without heat pretreatment show a simple oscillation to the degradation day. The less degraded PP, i.e., nearly homogeneous matrix, showed repetition motion, of which delamination is simply repeated. Moreover, the pretreated sample composed of heterogeneously degraded matrix exhibited irregular delamination behavior. The dependence on PP matrix conditions can be interpreted by a delamination mechanism based on ESC. Figure 6 shows the ESC mechanism of PP delamination. The onset of crack is initiated by autoxidation, and a series of delamination proceeds as follows: (I) water ingress, (II) craze birth and crack growth, and (III) delamination. These processes are repeated. The weight is increased by autoxidation during the (I) and (II) steps because delamination is not accompanied, and it decreases at (III). The simple oscillation of CI and rates of weight change are observed by the cycle of (I), (II), and (III) steps, as shown in Fig. 6. The delamination of marine PP samples is initiated as well. Notably, delamination occurs by ESC. Also, the ESC of polyolefin, such as polyethylene, shows surfactant-induced fracture in water [40]. Thus, delamination is likely facilitated in a water solution of surfactant. To assess the effect of the surfactant, the delamination mechanism is clarified. Figure 7 shows the optical SEM micrograph and 3D image of nine-day AOP-degraded PP with heat pretreatment. As shown in Fig. 7(a), there were no delamination parts on the surface before the POE8 surfactant treatment. Although the trace was
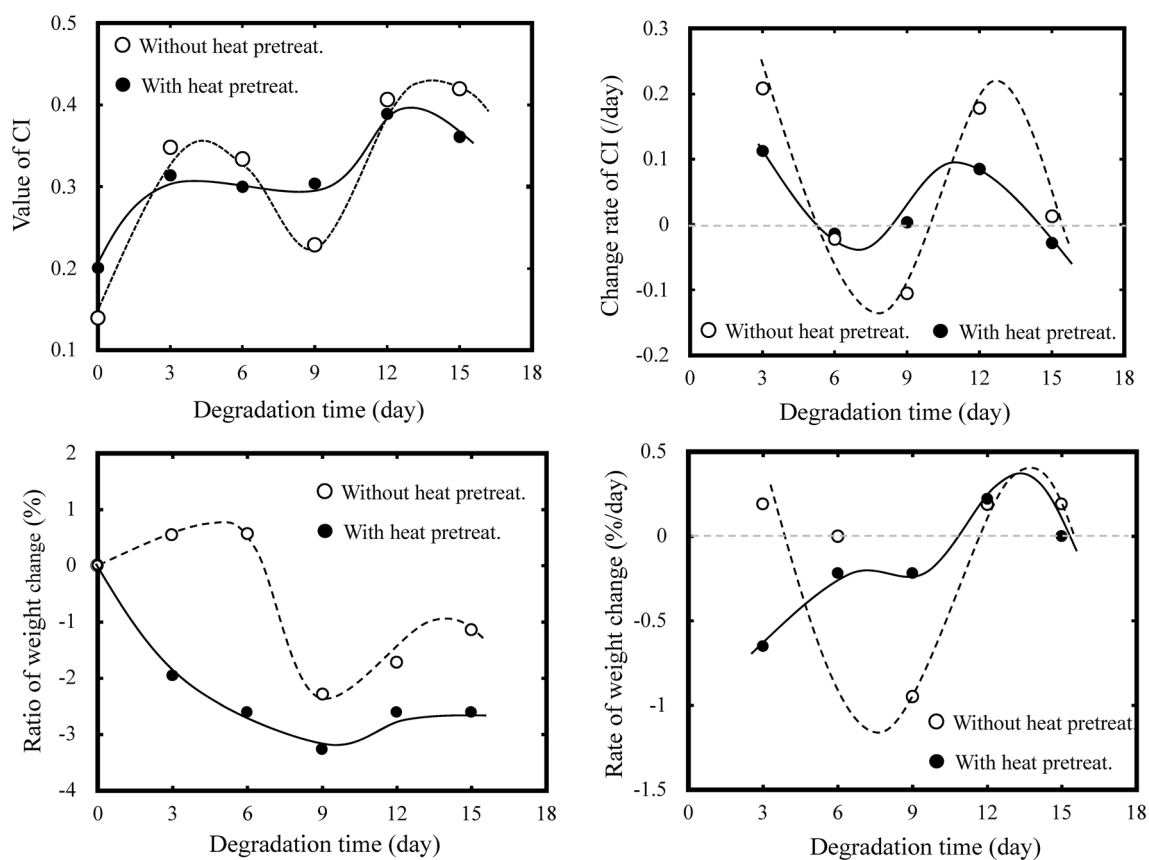
Fig. 5 Value and change rate of carbonyl index (CI), ratio and rate of weight change vs AOP degradation time of PP film with and without heat pretreatment

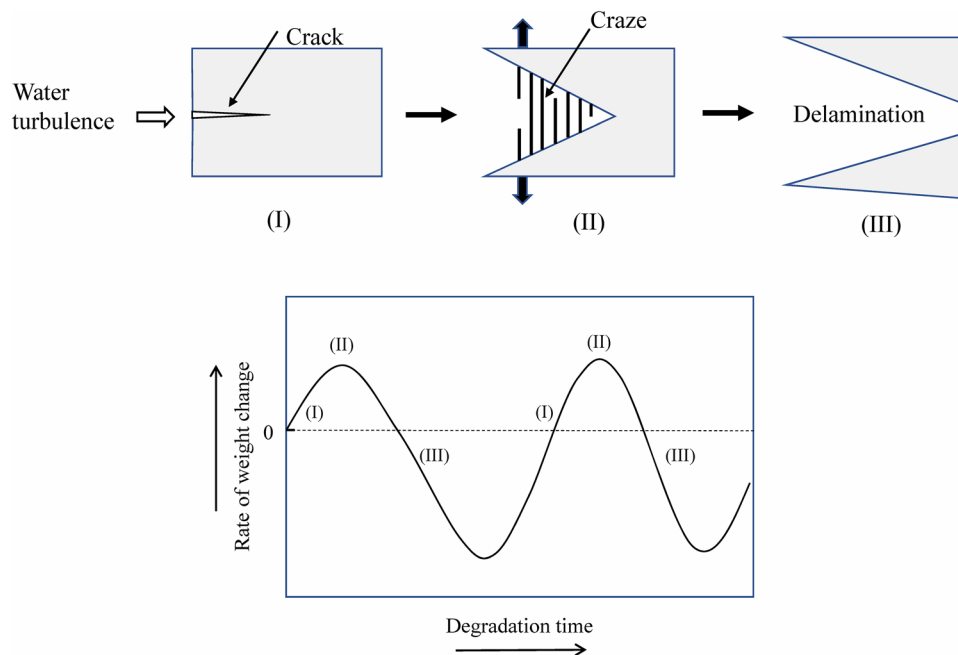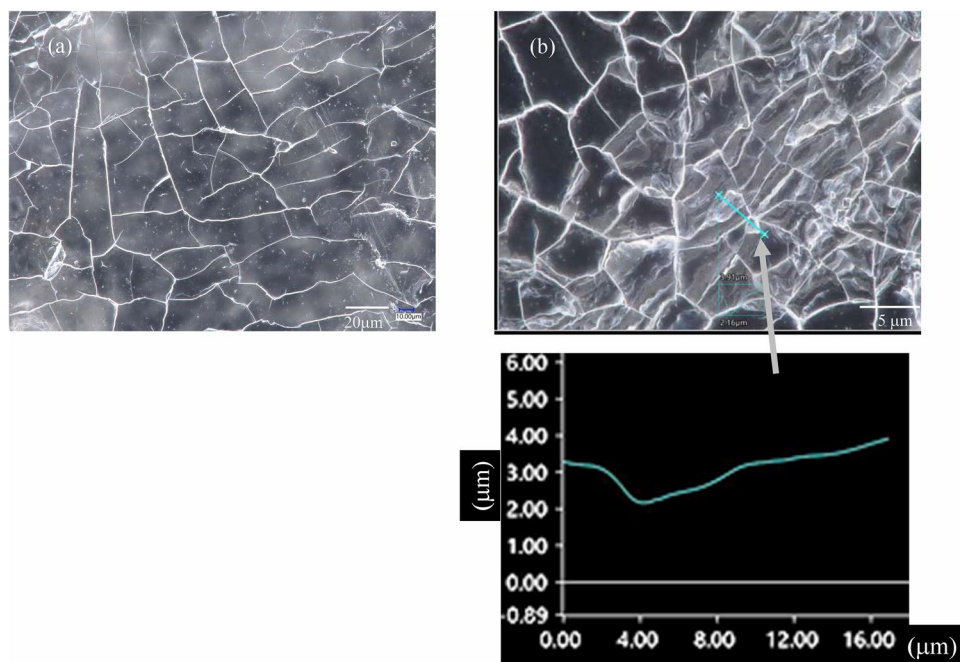**Fig. 6** ESC mechanism for PP delamination

Fig. 7 Optical SEM micrograph and 3D image of AOP degraded PP with heat pretreatment: AOP degradation time = 9 days. **a** Before surfactant treatment. **b** After surfactant treatment

shallow (ca. 1 μm) and uneven, the surfactant treatment provided many delamination traces, which resulted in the speedup. The result shows that delamination is based on the ESC mechanism. In addition, the cross-section was considerably rough and not a mirror plane. The force of craze fibril breakdown was considerably weaker since the water turbulence intensity is lower than the sea waves. The roughness of the cross-section depends on the water turbulence intensity, e.g., wave strength. Other M1-PP samples showed rough cross-sections similar to that of the surfactant-treated sample (Figure S6). In addition, as shown in Figure S7, there are many delamination traces on the large area. The trace shapes reveal that tile-like peeling off occurs. These results suggest that the shape of the delaminated part is affected by factors, such as wave intensity and drifting history. The fragmentation behavior of PP degradation is generally not due to ESC but surface embrittlement. It is a well-known fact [41]. However, all of the degradation test have been performed under dry condition without water. The water plays an important role in the degradation process. Julienne et al. suggested that water worked as plasticizer in polyolefin degradation process [22, 23]. In fact, we succeeded in recovering peeling off parts from AOP-degraded PP surface as shown in Figure S8. The recovery part composes of many sheet-like slices. If the fragmentation occurs by surface embrittlement, the powdery recovery is obtained. The sheet-like slices support that ESC of PP can occur in the water presence. The delamination behavior is attributed to the ESC mechanism, however, more detailed studies are required to clarify the delamination shape and production rate.

4 Conclusions

Two marine PP (M1-PP and M2-PP) and one land PP (L-PP) samples were collected from two beaches and the land, respectively, to study the fragmentation mechanisms. The delamination behavior was observed on the surfaces of both marine PP samples. Meanwhile, L-PP showed no delamination but abrasion patches on the surface. The delamination mechanism was studied using AOP-degraded PP as the marine PP model. The number and shape of cracks changed with the degradation time, showing the occurrence of ESC. The positive and negative CI values, as well as the ratios of the weight change, are attributed to repeated oxidation and delamination. In particular, the values and ratios of the sample without heat pretreatment fluctuated up and down, unlike those of the heat-pretreated samples. This is because of the different initial degrees of oxidation. The delamination behavior was dependent on the oxidation state, suggesting that the mechanism is based on ESC. Moreover, the POE8 surfactant treatment caused delamination speedup, which is a typical characteristic of polyolefin ESC. These results reveal that delamination is based on the ESC mechanism.

Declarations

Conflict of interest The authors declare that they have no conflict of interest.

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