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## Ionizing Radiation Effects on Polymer Biodegradation

M. Negrin<sup>\*1</sup>, E. Macerata<sup>1</sup>, G. Consolati<sup>2,3</sup>, L. Di Landro<sup>2</sup>, M. Mariani<sup>1</sup>.

<sup>1</sup> *Department of Energy, Politecnico di Milano, Piazza L. da Vinci, 32, 20133 Milano, Italy.*

<sup>2</sup> *Department of Aerospace Science and Technology, Politecnico di Milano, Piazza L. da Vinci, 32, 20133 Milano, Italy.*

<sup>3</sup> *INFN, Sezione di Milano, Via Celoria 16, 20133 Milano, Italy.*

*\*Corresponding author: maddalena.negrin@polimi.it*

## Abstract

Plastic waste has become a major worldwide environmental threat. In this view, six different polymer films, characterized by a different biodegradation rate, were exposed to gamma radiation and their surface properties along with the effect on their biodegradation rate in compost has been investigated. In particular two polyesters, poly(butylene succinate) (PBS) and poly(propylene cyclohexane dycarboxylate) (PPCE), have been selected as biodegradable and slow degrading polymers, while, as recalcitrant polymers, commercial films of high density polyethylene (HDPE), low density polyethylene (LDPE) and polyethylene terephthalate (PET). Polymers have been irradiated in air and water at absorbed doses up to hundreds of kGy. Surface properties have been analyzed by means of water contact angle (WCA) measurements and the composting tests have been performed up to 180 days.

WCA measurements revealed positive changes that could affect the polymer biodegradability. For polyesters, the wettability is highly increased when the samples are irradiated in water. PE wettability is increased for all the absorbed doses, while PET is not affected. In general, the degradability in compost of the considered systems is increased: PBS tends to degrade faster when irradiated in water while PPCE in both irradiation conditions. HDPE and LDPE did not evidence relevant variations in the surface morphology after 180 days in compost but a difference in the thermal properties suggests a beginning of the degradation process. PET degradability appeared to be not affected after 180 days in compost, even after irradiation up to 1 MGy. The research suggests that the radiation-induced degradation could be an effective pre-treatment to enhance the biodegradation rate of some polymeric systems.

*Keywords:* polymer, gamma radiation, biodegradation, polyester, polyethylene, hydrophilicity.

## 1 Introduction

The great properties of conventional synthetic plastics have allowed a wide range of applications. Packaging constitutes the larger market segment and, due to its specific function, it rapidly becomes waste that accumulates in the environment (1, 2). Plastic waste has been creating risks not only for the environment, but also for human and animal health (3, 4). Most

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3 of the conventional petrochemical-based plastics are not biodegradable and the production of  
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5 biodegradable and bio-based plastics is still limited by the high production costs and the  
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7 poorer properties. Nowadays efforts have been devoted to enhancing conventional plastic  
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9 degradability (5) and to tailoring biodegradable polymer properties (6, 7).

12 Finding a cost-effective and environmental-friendly solution for the treatment of  
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14 plastic waste is extremely important. A sustainable technique to improve conventional plastic  
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16 waste degradation still does not exist and a huge amount of waste goes into environment. It is  
17  
18 well known that ionizing radiation can modify polymers, affording many practical  
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20 applications, among which inducing their degradation (8). In particular, gamma radiation can  
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22 facilitate the material degradation by inducing oxidative fragmentations of polymer backbone  
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24 (9). In this view, experimental activities have been addressed to investigate if a radiation  
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26 treatment of bio-based plastics could represent an effective pre-treatment to improve their  
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28 biodegradation.  
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33 Different polymers used as packaging have been considered, with a different  
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35 biodegradation rate. Among the class of biodegradable biopolymers, poly(butylene succinate)  
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37 (PBS) has interesting mechanical and physical properties along with a good processability. It  
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39 is commercially available since 1993 and it is completely biodegraded in 180 days under  
40  
41 controlled composting conditions. Its behavior under gamma irradiation has been investigated  
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43 in different conditions (10-12). Irradiation in air has been shown to slow down its degradation  
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45 in compost but low absorbed doses or different irradiation environments can enhance its  
46  
47 degradability. Among the class of slow-degrading biopolymers, poly(propylene cyclohexane  
48  
49 dicarboxylate) (PPCE), thanks to the presence of the cycloaliphatic unit, is characterized by  
50  
51 good mechanical properties, good resistance to weather, heat, light and moisture, still  
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53 maintaining biodegradability. Its high crystallinity degree significantly lowers its degradation  
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55 rate: tests have shown that, after 140 days in compost, its residual weight is about 97% (13).  
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3 Polyethylene (PE) is one of the most used plastic as packaging (14), belonging to the  
4 polyolefins family. Due to its high stability, associated with a slow rate of degradation in  
5 natural conditions, PE strongly contributes to the increase of plastic pollution (15). Its  
6 radiation stability has been widely investigated (16-18): it is known to be fairly resistant  
7 towards radiation but the overall effects depend on both irradiation and sample conditions.  
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12 Previous works showed that gamma radiation can affect the properties related to  
13 biodegradability (12, 19). In this view, the present research aims at evaluating how gamma  
14 radiation in two different irradiation environments (air and water) could affect the  
15 hydrophilicity and thus the rate of disintegration in compost of PBS, PPCE, PE and PET  
16 films.  
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## 28 **2 Experimental**

### 29 **2.1 Materials**

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32 Different systems characterized by a different radiation sensibility and a different time to  
33 complete disintegration in compost have been selected: months, years and decades (Figure 1).  
34 Poly(butylene succinate) (PBS), synthesized according to the two-stage procedure (20)  
35 reported in literature, has been selected as biodegradable system. Poly(propylene cyclohexane  
36 dicarboxylate) (PPCE) (13) has been studied as slow-degrading system. Polyesters in form of  
37 powder have been moulded as 200  $\mu\text{m}$  films. The recalcitrant system selected is polyethylene  
38 (PE). Samples of high-density polyethylene (HDPE) and low-density polyethylene (LDPE)  
39 have been acquired by Sigma-Aldrich<sup>®</sup> as 10 and 15  $\mu\text{m}$  films respectively. Some  
40 experiments have been carried out also on commercial packaging samples: HDPE films of 20  
41  $\mu\text{m}$  (HDPE1). Moreover, 13  $\mu\text{m}$  polyethylene terephthalate (PET) films (Sigma-Aldrich<sup>®</sup>),  
42 have been considered as radiation-resistant and recalcitrant system.  
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3 Irradiation has been carried out by a Co-60 source with a dose rate of 2.5 kGy/h up to  
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5 hundreds of kGy. Samples have been irradiated in two different environments, air and  
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7 plunged in water.  
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12 [Insert Figure 1 near here]  
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## 16 **2.2 Methods**

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18 Surface properties have been analyzed by static water contact angle measurements (WCA).  
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20 Measurements were performed using an optical contact angle apparatus (OCA 15 Plus – Data  
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22 Physics Instruments GmbH, Filderstadt, Germany) equipped with a video measuring system.  
23  
24 Software SCA 20 (Data Physics Instruments GmbH) was used for data acquisition. Prior to  
25  
26 the analysis, the films surface was properly washed with 70% ethanol aqueous solutions and  
27  
28 then dried overnight in a sealed desiccator. Measurements were performed at ambient  
29  
30 conditions by recording the side profiles of deionized water drops for image analysis. For  
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32 each polymeric sample, at least ten measurements were performed. Image analysis was  
33  
34 carried out with a Drop Shape Analysis software. The data reported correspond to the average  
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36 values. In the case of the commercial HDPE sample from the shopping bag, in order to  
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38 simplify the measurement, the sample have been fixed on a glass surface by using carbon  
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40 tape.  
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46 Disintegration experiments have been performed by a procedure based on the ISO-  
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48 20200 standard method. Prior to disintegration tests, each sample was put under vacuum to  
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50 constant weight and then weighted to obtain the initial mass. Then, each specimen was placed  
51  
52 inside a vessel with a proper amount of mature compost and then incubated at 58°C for a  
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54 specific incubation time, maintaining the humidity at the 90% of the water holding capacity of  
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56 the system. The minimum incubation period was set at 45 days and then extended with  
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3 increasing the stability of the system towards biodegradation: 90 days for the slow-degrading  
4 system and 180 days for the recalcitrant polymers. After the incubation period, samples were  
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6 sieved at 2 mm and then washed following a procedure to remove the microbial cells adhered  
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8 on the surface. The samples were then dried under vacuum to constant mass. The rate of  
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10 disintegration was then quantified by evaluating the samples weight loss. Moreover, changes  
11  
12 in the visual appearance and surface morphology have been analyzed. SEM images of  
13  
14 polymer films were acquired using a field emission scanning electron microscope (FE-SEM)  
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16 working in high vacuum Zeiss Supra 40 equipped with the GEMINI column. Images were  
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18 acquired under vacuum by gluing films on aluminum stabs with carbon tape.  
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24 The thermal behavior has been investigated by differential scanning calorimetry  
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26 (DSC). A Mettler Toledo differential scanning calorimeter (DSC 822e) calibrated with high  
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28 purity standards (indium and zinc) was used to evaluate the thermal properties. Samples (c.a.  
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30 1 to 5 mg) were encapsulated in aluminum pans and heated to about 20 °C above melting  
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32 temperature at a rate of 20 °C min<sup>-1</sup> (first scan) under nitrogen flux. For each sample, two  
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34 heating and cooling cycles were performed. The melting temperature ( $T_m$ ) was determined as  
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36 the peak value of the endothermic phenomenon. The melting enthalpy per unit mass ( $\Delta H_m$ )  
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38 was calculated from the area of the DSC endothermic peak. The first scan has been  
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40 considered to take into account the modifications induced by irradiation on the crystallinity.  
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### 46 **3 Results and Discussion**

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48 WCA measurements on polymer films evidenced variations of the hydrophilicity induced by  
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50 irradiation for all the considered systems and the results are shown in Figure 2. As evidenced  
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52 in a previous work, the surface properties are strictly connected to an increased rate of  
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54 biodegradability (12, 19). The biodegradable system PBS, irradiated in air, does not show an  
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56 improvement of hydrophilicity: with increasing the absorbed dose, the water contact angle  
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3 increases of about  $10^\circ$ . Differently, when the sample is irradiated in water, the sample at 100  
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5 kGy shows a decrease of about  $10^\circ$  compared to the pristine sample. When the sample is  
6  
7 immersed in water, the most of the damage induced by radiation goes to the water, leading to  
8  
9 the production of very reactive radiolytic species among which hydroxyl radicals ( $\text{HO}^\cdot$ ) and  
10  
11 reducing aqueous electrons ( $\text{e}_{\text{aq}}^-$ ). Since the experiments have been performed in aerated  
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13 conditions, the aqueous electrons could be scavenged by the oxygen dissolved in the solution  
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15 leading to the production of the superoxide radical ion ( $\text{O}_2^-$ ). The subsequent reaction with  
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17 hydrogen ions ( $\text{H}^+$ ) produced hydroperoxy radicals ( $\text{HO}_2^\cdot$ ) that, along with superoxide  
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19 radicals, lead ultimately to the formation of hydrogen peroxide that acts as oxidizing agent.  
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21 The damage in the bulk of the polymer is lower, since the degradation induced by oxidation  
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23 acts only thanks to the permeated oxygen. On the polymer surface, however, the contact with  
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25 the oxidizing agents can enhance the degradation of the polymer surface and thus favor the  
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27 attack by microorganisms.  
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33 PPCE, the slow-degrading system, does not display any improvement of wettability  
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35 except for the highest absorbed doses, 200 kGy and 500 kGy. In the case of the sample  
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37 irradiated in water, the samples appear more hydrophilic than the ones irradiated in air at  
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39 equal absorbed dose.  
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42 In the case of polyethylene, radiation induced oxidation leads to the creation of new  
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44 functional groups, that clearly enhances hydrophilicity in the irradiated samples. Irradiated  
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46 HDPE shows an increased wettability, especially when treated in water. The commercial  
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48 HDPE bag (HDPE1) shows a similar behavior, but the treatment in air appears to be less  
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50 effective. LDPE samples, characterized by an higher thickness than HDPE, show an increase  
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52 of wettability with the absorbed dose more limited than HDPE but still more evident in water.  
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56 PET is widely known to be resistant toward ionizing radiation thanks to the presence  
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58 of the aromatic ring that dissipates the absorbed energy without collisions. In this case the  
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3 hydrophilicity does not show any variation, even for the sample irradiated at 1 MGy,  
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5 confirming its high radioresistance.  
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10 [Insert Figure 2 near here]  
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13 The modifications induced on the wettability are different among the three systems,  
14 the enhanced wettability observed in several samples, along with the degradation induced by  
15 oxidation, can favor the attack by microorganisms. As shown in Figure 3, coherently, some  
16 effects have been observed in the composting tests. In each composting experiment a Mater-  
17 Bi® sample was included and it was found to be completely destroyed after 45 days in  
18 compost, thus demonstrating the effectiveness of the test.  
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29 [Insert Figure 3 near here]  
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33 PBS specimens after 52 days in compost show a residual weight that is coherent with  
34 the wettability measurements: samples irradiated in water degrade faster, while the ones  
35 irradiated in air appear to be less degraded than the pristine sample. This can be explained by  
36 the crosslink induced by radiation that, decreasing the hydrophilicity of the sample, reduce the  
37 attack by microorganisms. Interestingly, samples irradiated at 25 kGy evidence an  
38 enhancement of the degradation rate in both the irradiation environments: this suggests that a  
39 low absorbed dose is enough to alter the biodegradation rate of the biodegradable system.  
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49 PPCE samples after 70 days in compost show limited weight losses. While the pristine  
50 sample is not degraded, the irradiated samples show an increased degradation with increasing  
51 the absorbed dose. This improvement can be clearly seen even at 25 kGy, highlighted by the  
52 SEM images acquired (Figure 4). The surface of the irradiated samples shows the presence of  
53 holes and depleted zones, more evident for the sample irradiated in water.  
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[Insert Figure 4 here]

Within an incubation period of 90 days, PE samples do not show any remarkable improvement in the biodegradation process and, differently to polyesters, samples irradiated in water are even less affected. HDPE1 samples show an higher degradation when irradiated in air at 300 kGy and 500 kGy (Figure 3), however, the decrease of the residual weight is strongly affected by the sample fragility induced by ionizing radiation. As an example, the residual weight of the sample irradiated at 300 kGy, completely fragmented (Figure 5), was about 65% but SEM images acquired after the test did not evidence any relevant sign of attack by microorganisms (Figure 6). The same observations could be extended to the HDPE1 (Figure 6) and HDPE samples (Figure 7) treated at 500 kGy.

[Insert Figure 5 near here]

[Insert Figure 6 near here]

[Insert Figure 7 near here]

The incubation period of HDPE, LDPE and PET films has been extended to 180 days but the weight losses are still limited (Figure 3). The residual weight of the HDPE samples irradiated at 200 kGy in air was about 50% but, also in this case, the sample fragility caused by ionizing radiation is strongly enhanced by incubation in compost. Similar results have been obtained for LDPE samples: up to the incubation time considered no evident signs of degradation can be observed by SEM measurements. As reported in literature (21-23), the beginning of the biodegradation process could be monitored by differential scanning

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3 calorimetry (DSC). DSC analyses on irradiated samples after the composting tests showed a  
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5 variation of the melting enthalpy (Table 1), thus suggesting that the polymer modifications  
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7 induced by the radiation treatment, even if not relevant, were enough to initiate the  
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9 degradation process. PE degradability in compost in general appears to be affected by  
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11 irradiation at high absorbed doses but further experiments are foreseen to check if an extended  
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13 incubation period could evidence higher modifications lowering the absorbed dose.  
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19 [Insert Table 1 near here]  
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24 In the case of PET, the polymer biodegradability is not affected in the 180 days  
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26 incubation period; the sample irradiated in air at the highest absorbed dose of 500 kGy was  
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28 completely recovered after the composting test and no evidence of degradation was observed.  
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### 34 **Conclusions**

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37 The present work studied the effect of ionizing radiation on the wettability and on the  
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39 biodegradability of polymers with different degradability: PBS, PPCE, PE and PET. The  
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41 treatment proved to be effective for systems more sensitive to ionizing radiation. The  
42  
43 radiation-induced oxidation affects the surface properties and consequently also the  
44  
45 degradation rate in compost. PBS and PPCE biodegradability is mostly enhanced for the  
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47 samples irradiated in water, already at the low absorbed dose of 25 kGy. Radiation treatment  
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49 strongly influences PE wettability, nevertheless after an incubation period of 180 days the  
50  
51 degradability appeared to be slightly affected and DSC analyses evidenced a beginning of the  
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53 degradation process only at the highest absorbed doses. As expected, no evidence of  
54  
55 degradation process only at the highest absorbed doses. As expected, no evidence of  
56  
57 degradation was observed for PET.  
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The research performed confirmed that the radiation-induced degradation could be considered as an effective pre-treatment to enhance the degradation in compost of polymeric systems sensitive towards ionizing radiations.

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## Tables

Table 1 - DSC thermal data (1<sup>st</sup> scan) for LDPE films as a function of the absorbed dose.

Sample		Incubation Period	
		0 gg	180 gg
Pristine	$T_m$ [°C]	106	105
	$\Delta H$ [J/g]	53.1	127.4
200 kGy air	$T_m$ [°C]	105	106
	$\Delta H$ [J/g]	73.3	133.1
200 kGy water	$T_m$ [°C]	104	107
	$\Delta H$ [J/g]	77.5	131.0

## Figures

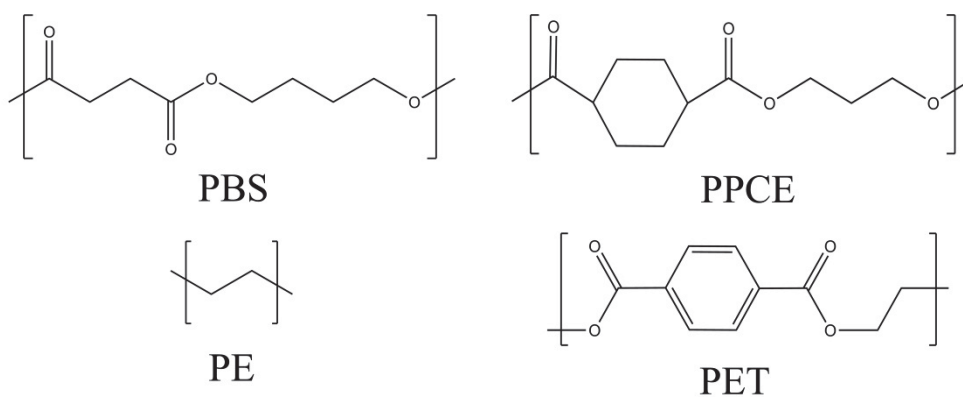


Figure 1 – Chemical Structure of the Repeating Units of Poly(butylene succinate) (PBS), Poly(propylene cyclohexane dicarboxylate) (PPCE), Polyethylene (PE) and Polyethylene terephthalate (PET) polymers.

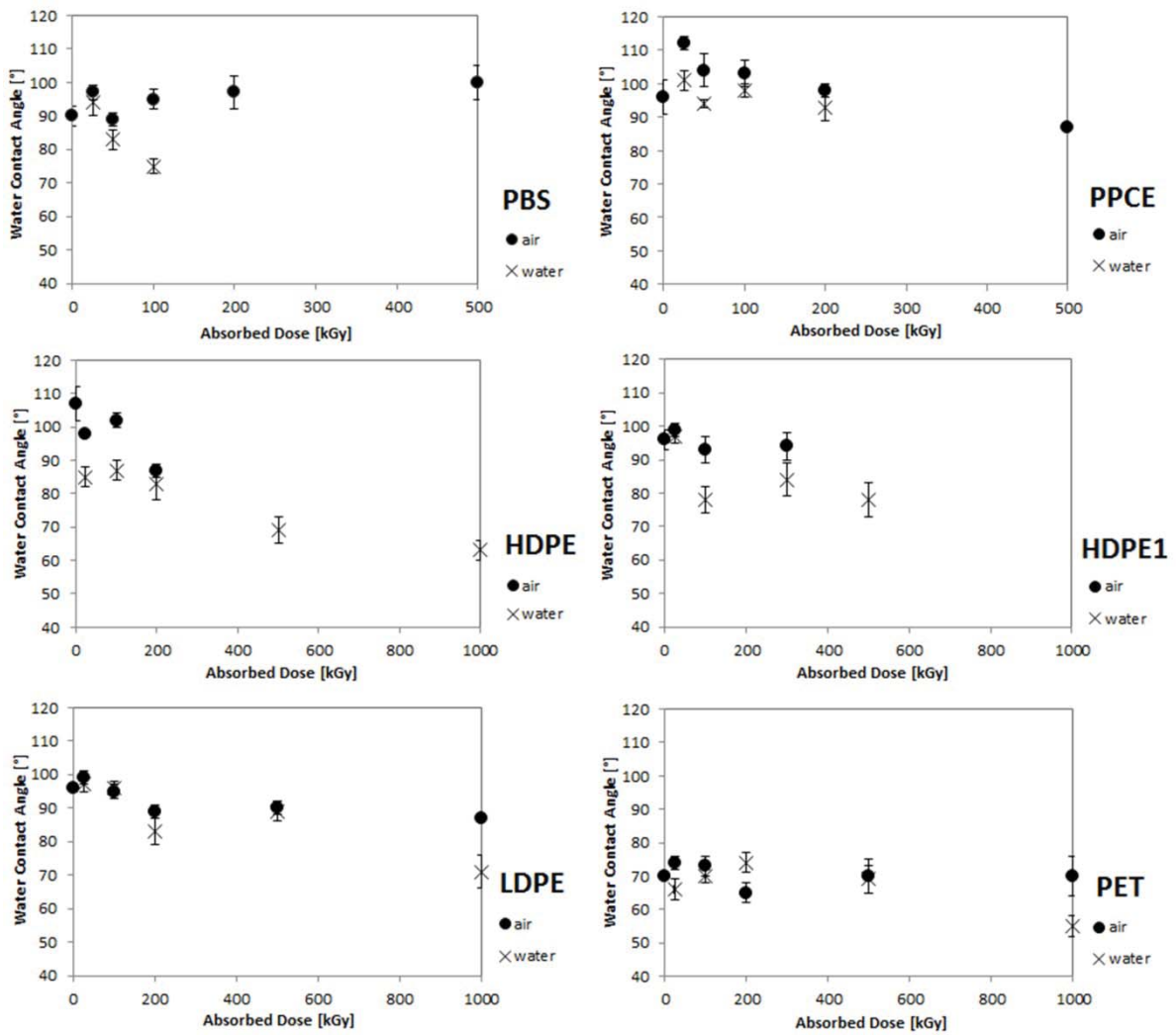


Figure 2 – Water Contact Angle measurements vs absorbed dose for polymer films irradiated in air and water: PBS (12), PPCE (19), HDPE, HDPE1, LDPE, PET.



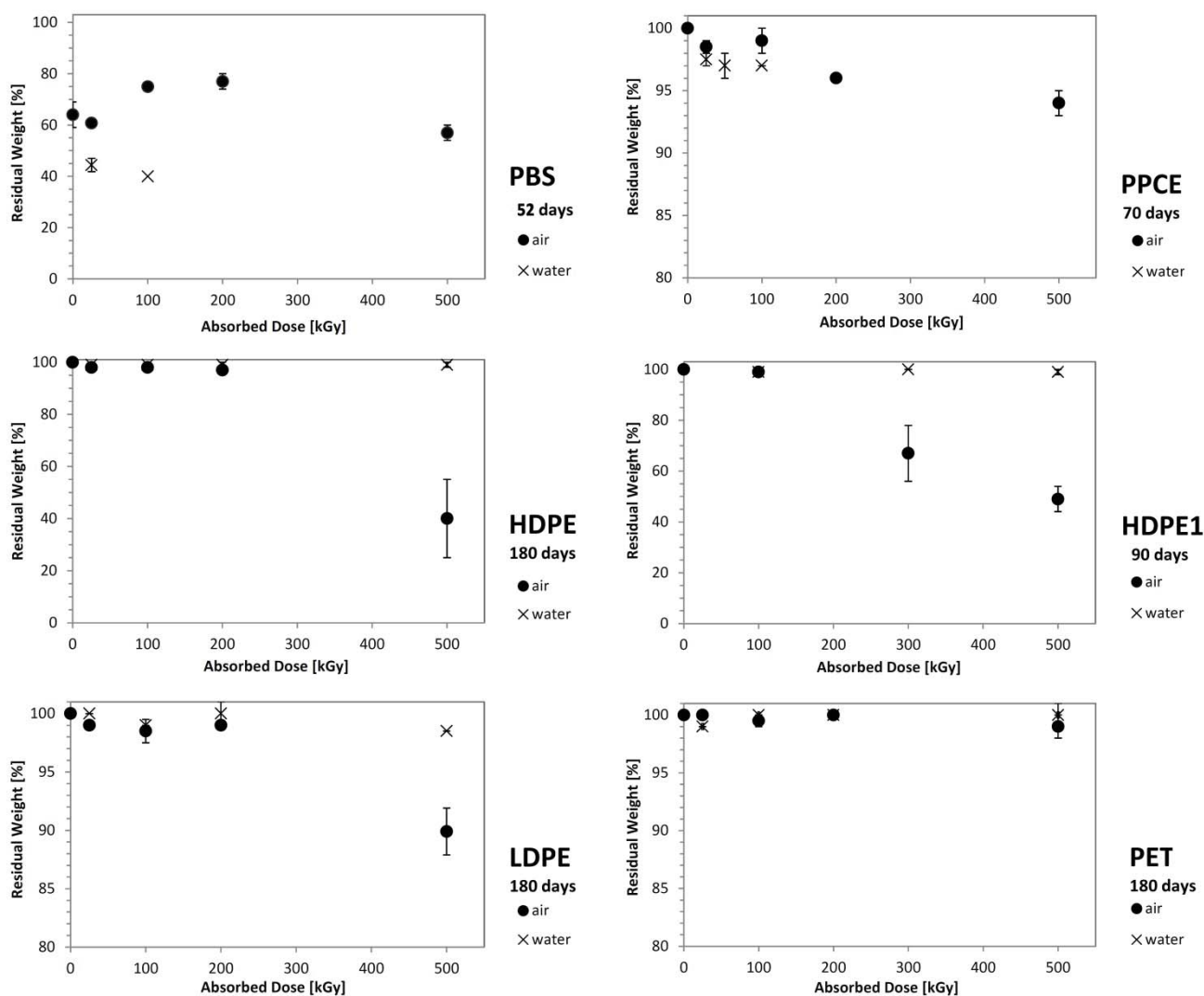


Figure 3 – Residual weight [%] as a function of the absorbed dose for the polymer samples irradiated in air and water after different incubation periods: 52 days for PBS (12); 70 days for PPCE (19); 90 days for HDPE1; and 180 days for HDPE, LDPE and PET.

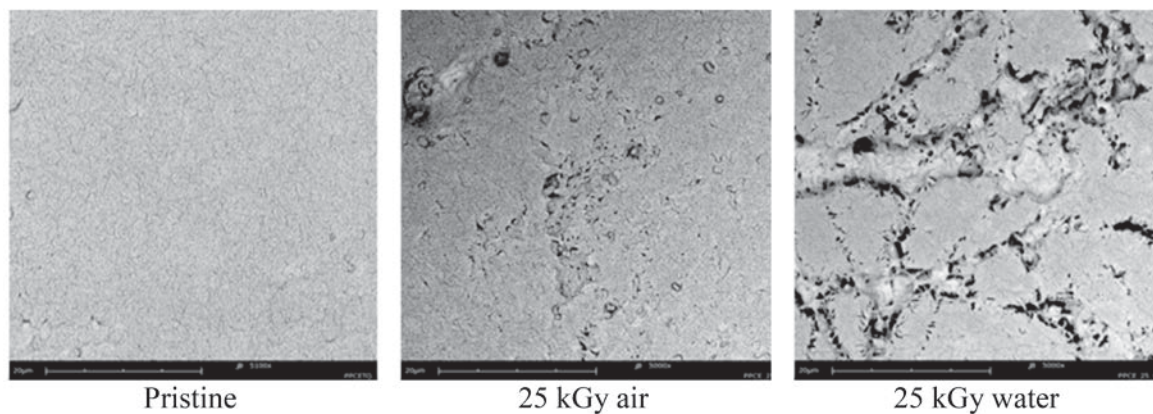


Figure 4 – SEM images of PPCE samples, pristine and irradiated at 25 kGy in air and water, after 70 days in compost (Magnification 5000X) (19).

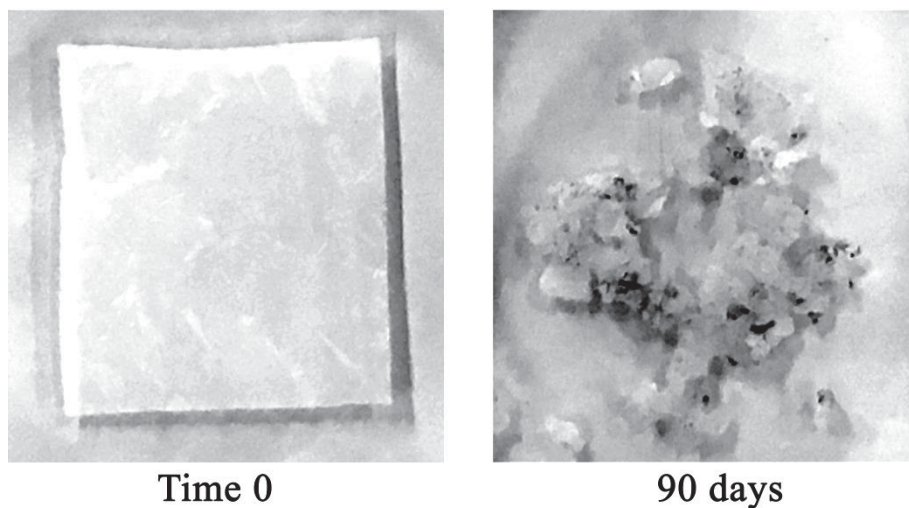
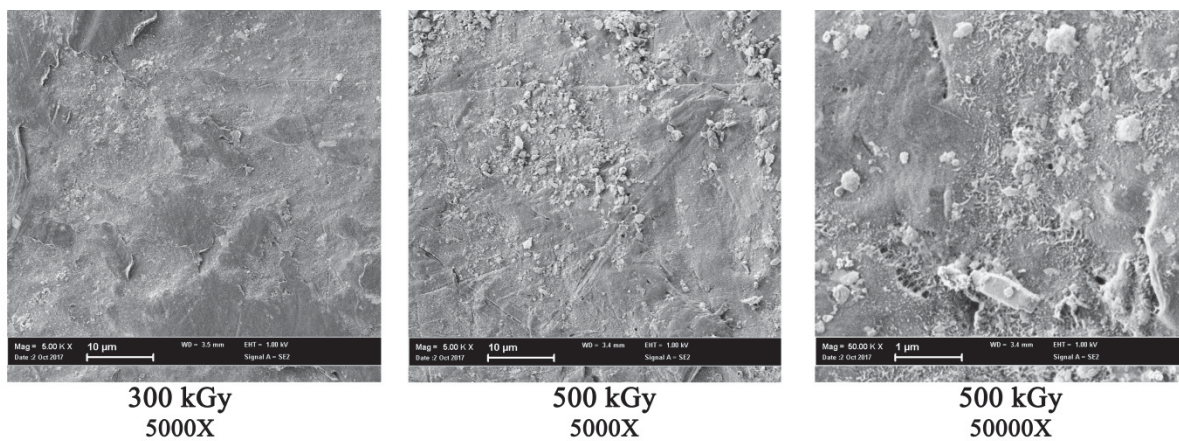
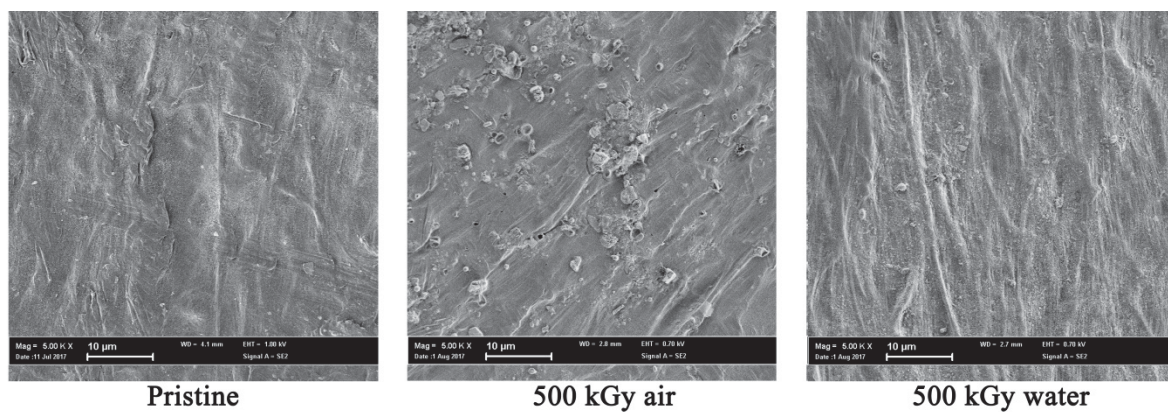


Figure 5 – Visual appearance of HDPE1 sample irradiated at 300 kGy before (Time 0) and after 90 days in compost.



20 Figure 6 – SEM images of HDPE1 samples irradiated at 300 kGy and 500 kGy in air after 90  
21 days in compost (Magnification 5000X and 50000X).  
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21 Figure 7 – SEM images of HDPE samples pristine and irradiated at 500 kGy in air and water  
22 after 90 days in compost (Magnification 5000X).  
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## Figures Caption

Figure 1 – Chemical Structure of the Repeating Units of Poly(butylene succinate) (PBS), Poly(propylene cyclohexane dicarboxylate) (PPCE), Polyethylene (PE) and Polyethylene terephthalate (PET) polymers.

Figure 2 – Water Contact Angle measurements vs absorbed dose for polymer films irradiated in air and water: PBS (12), PPCE (19), HDPE, HDPE1, LDPE, PET.

Figure 3 – Residual weight [%] as a function of the absorbed dose for the polymer samples irradiated in air and water after different incubation periods: 52 days for PBS (12); 70 days for PPCE (19); 90 days for HDPE1; and 180 days for HDPE, LDPE and PET.

Figure 4 – SEM images of PPCE samples, pristine and irradiated at 25 kGy in air and water, after 70 days in compost (Magnification 5000X) (19).

Figure 5 – Visual appearance of HDPE1 samples irradiated at 300 kGy before (Time 0) and after 90 days in compost.

Figure 6 – SEM images of HDPE1 samples irradiated at 300 kGy and 500 kGy in air after 90 days in compost (Magnification 5000X and 50000X).

Figure 7 – SEM images of HDPE samples pristine and irradiated at 500 kGy in air and water after 90 days in compost (Magnification 5000X).