

# Radiolysis products and sensory properties of electron-beam irradiated high barrier food packaging films containing a buried layer of recycled low density polyethylene

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### **Food Additives and Contaminants**



# Radiolysis products and sensory properties of electronbeam irradiated high barrier food packaging films containing a buried layer of recycled low density polyethylene

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- Radiolysis products and sensory properties of electron-beam
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- 3 layer of recycled low density polyethylene

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### Abstract

The aim of the present work was to study the effect of electron-beam irradiation on the production of radiolysis products and sensory changes in experimental high barrier packaging films composed of polyamide (PA), ethylene–vinyl alcohol (EVOH) and low density polyethylene (LDPE). Films contained a middle buried layer of recycled low density polyethylene (LDPE), while films containing 100% virgin LDPE as the middle buried layer were taken as controls. Irradiation doses ranged between 0 and 60 kGy. Generally, a large number of radiolysis products were produced during e-beam irradiation even at the lower absorbed doses of 5 and 10 kGy (approved doses for food "cold pasteurization"). The quantity of radiolysis products increased with irradiation dose. There were no significant differences in radiolysis products identified between samples containing a recycled layer of LDPE and those containing virgin LDPE (all

absorbed doses), indicating the "functional barrier" properties of external virgin polymer layers. Sensory properties (mainly taste) of potable water were affected after contact with irradiated as low as 5 kGy packaging films. This effect increased with increasing irradiation dose.

**Keywords:** electron beam irradiation; multilayer packaging films; recycled LDPE; radiolysis products; sensory evaluation

# Introduction

After more than five decades of research, food irradiation is considered as a safe and effective method of food processing and preservation (Riganakos et al., 1999; George et al., 2007). Both gamma (mainly from  $^{60}$ Co sources) and electron beam (from electron accelerators) irradiation is being commercially used for both cold pasteurization and sterilization purposes (Stoffers et al., 2004). Electron-beam (e-beam) irradiation has many advantages as compared to  $\gamma$ -irradiation, such as high dose rate, low heat, short process time and easy operation procedure, good control, continuous process (Riganakos et al., 1999; Suarez et al., 2000; Fintzou et al., 2006; Fintzou et al., 2007a).

Plastic packaging materials, in the form of single- or multilayer films are part of the irradiation processing of foods, since foodstuffs are usually packaged prior to irradiation to prevent microbial recontamination (Ezquerro et al., 2003; Chytiri et al., 2005; Chytiri et al., 2006; George et al., 2007).

The major changes produced in polymers by ionizing radiation are a) scission and/or crosslinking of the polymer chains, b) formation of gases and low molecular-

weight volatile radiolysis products and c) formation of unsaturated bonds, free radicals and oxidative degradation products (Goulas et al, 2004a). The specific results depend on the type of polymer, the specific polymer additives used and the irradiation conditions, i.e. radiation-induced crosslinking dominates under vacuum or an inert atmosphere. Chain scission on the other hand dominates during irradiation in the presence of oxygen or air (Morehouse & Komolprasent, 2004). Above phenomea may lead to changes in mechanical properties (tensile strength, brittleness) (Fintzou et al., 2007b), physical properties (discoloration, gas permeability, migration) (Goulas et al., 2004b; Zygoura et al., 2007) as well as off-odor development in polymers (Chytiri et al., 2008) or even toxicological concerns through the production of specific radiolysis products (Riganakos et al., 1999; Fintzou et al., 2007a).

Nowadays, municipal waste has become a major problem worldwide. The percentage of plastic scrap in the waste stream increases as the production of polymeric materials grows rapidly (Clough, 2001; Zenkiewicz and Dzwonkowski, 2007). The European Union (EU), through council directive 94/62/EC (EC, 1994), sets as a first priority the reduction of packaging waste combined with reuse and recycling of packaging materials.

One of the major problems associated with recycling is that melt processing may induce thermo-mechanical degradation which leads to a subsequent deterioration of mechanical properties, an increase in migration values from the packaging material into the packaging foodstuff and organoleptic changes of the contained product (Badeka et al., 2003; Chytiri et al., 2005; Chytiri et al., 2006; Scaffaro et al., 2007). Thus recycled polymers are not used in direct contact with food, but only as a buried layer of a multilayer structure in which the outer layers are composed of virgin polymer. Such virgin polymer layers are used as a "functional barrier" (Welle et al.,

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2002; Badeka	et al.,	2003;	Stoffers	et al.,	2004;	Chytiri	et al.,	2005;	Chytiri	et	al.,
2006).											

Based on the above the objective of the present study is to investigate the effect of electron beam irradiation (doses 5-60 kGy) on the formation of radiolysis products from five-layer materials based on EVOH layer (high barrier material) containing recycled LDPE as a middle layer from an analytical and sensory point of view.

### **Materials and Methods**

### Materials

Five-layer coextruded experimental films PA/ EVOH / tie / LDPE / LDPE, where the middle buried layer LDPE was recycled material, were produced in the ELVIOMET SA plant (Komotini, Greece), on a five extruder pilot scale coextrusion line (Alfa Marathon Manufacturing Co., Mississauga, Canada). The recycled layer contained either 50% virgin plus 50% recycled LDPE or 100% recycled LDPE. Control samples contained 100% virgin LDPE in the respective layer. The three coextruded structures are given in Figure 1. The three film types (V-Virgin LDPE, R1-50% recycled plus 50% recycled LDPE, R2-100% recycled LDPE) were 80 μm in thickness. LDPE was used for its high barrier to water vapour, its excellent sealability and its low cost. PA was used for its excellent mechanical properties and EVOH was used for its high barrier to oxygen and odorous compounds.

# Reagents

Methyl stearate was purchased from Fluka (Buch, Switzerland). Iso-octane, GC grade, was purchased from Merck (Darmstadt, Germany).

# Simulant/packaging material contact conditions

In order to achieve realistic food/plastic irradiation conditions, plastic films were irradiated while in contact with the food simulant iso-octane, as follows: rectangular strips of each film, dimensions 12 cm x 12 cm, were used to produce pouches via thermosealing (Boss model N 48 vacuum sealer, Bad Homburg, Germany). Pouches (inner surface area, 2 x 100 cm²) were filled with 100 ml of iso-octane (alternative fatty food simulant according to EU Directive 97/48/EC (EC, 1997), thermosealed and subsequently irradiated. The total polymer/iso-octane contact time was 48 hrs at room temperature. These conditions are identical to those of 48 hrs/20°C as defined by EU for fat stimulants (EC, 1997). Control samples were the same as above but were not irradiated. Then the pouches were cut open and the simulant was transferred to glass serum vials (capacity: 20 ml). The vials were sealed with an aluminum-faced silicone septum and an aluminum crimp cap.

### Irradiation conditions-dosimetry

The irradiation of all samples (films plus food simulant) was carried out in the presence of air at room temperature in the Institute of Process Engineering of the Federal Research Center for Nutrition and Food (Karlsruhe, Germany) using an electron accelerator LINAC- CIRCE III (Linac Technologies C.A., Orsay, France, 10 MeV, 4.3 KW) so as to achieve 5, 10, 30 and 60 kGy absorbed doses, respectively. Irradiation doses were measured using Alanin/ ETR dosimeters and the average dose rate was ~1 kGy/min.

# Determination of radiolysis products by GC/MS

A Hewlett-Packard, model 6890, gas chromatograph was used, connected to a Hewlett-Packard model 5973 mass spectrometric detector (Agilent Technologies, Wilmington, DE, USA).

The gas chromatographic conditions were as follows: column: HP – 5 MS (5% Phenyl Methyl Siloxane), 30.0 m x 250  $\mu$ m x 0.25  $\mu$ m, carrier gas flow rate (helium): 0.7 ml min<sup>-1</sup>. Column temperature program: 60°C for 10 min, increased by 3°C min<sup>-1</sup> to 120°C and then increased by 10°C min<sup>-1</sup> to 280°C and held isothermaly for 6 min (total analysis time 52 min). Injector temperature: 250°C; interface temperature: 290°C injection mode: split 1:5.

The mass spectrometer was operated under the following conditions: Scan range: 30-550, MS source temperature: 230°C, MS ion source: 150°C, Electron impact (El) ionization at 70 eV.

# Identification and semi-quantification of radiolysis products

Identification of radiolysis products was achieved by comparing the mass spectra of the recorded chromatographic peaks, with the Wiley 275 MS data base while semi-quantification was achieved by comparison of the MS detector response to methyl stearate used as an internal standard to that of the recorded peaks. Methyl stearate concentration in iso-octane used was 10 µg ml<sup>-1</sup>.

Sensory evaluation

Irradiated pouches as those described previously, made of the experimental films, were filled with 100 ml of potable water (aqueous food simulant according to EU directive 97/48/EC) and thermosealed as described previously. Pouches were then stored at 40°C for 10 days (storage conditions for aqueous food simulants according to EU directive 97/48/EC) (EC, 1997). Multilayer materials were evaluated with respect to their sensory characteristics by a panel of seven experienced judges which were asked to score odor, taste and colour of potable water after film/simulant contact. Non-irradiated samples served as "controls". Scoring was based on a 4 point scale, where: 0-no difference, 1-slight, 2-moderate, 3-strong, 4-very strong with respect to

the control sample. The score of 1 (recorded by at least of 50% of the judges) was considered to be the limit of acceptability. Potable water bottled in glass served as the "master control" sample.

### **Results and Discussion**

### Radiolysis products

The results of semi-quantitative analysis of radiolysis products from all three samples are given in Table 1. The higher concentration compounds followed by numbers in parenthesis (Table 1) are also shown in the chromatograms in Figure 2 A, B for film R2 irradiated at 60 kGy. GC chromatograms of the products from non-irradiated and irradiated at the doses 5, 10, 30 and 60 kGy films are given in Figure 3. The "control samples" (non-irradiated polymer/iso-octane) did not show any compounds, the result of migration from the polymer film into iso-octane during contact (Fig. 3 V, R1, R2, curve 1).

As shown in Table 1, the main classes of radiolysis compounds identified included saturated hydrocarbons, ketones, alcohols, alkenes, esters and carboxylic acids. Most of these compounds were identified even at the lowest irradiation dose (5 kGy). More specifically, 23 saturated hydrocarbons with 9 to 16 carbon atoms, one alkene with 7 carbon atoms, 10 alcohols with 4 to 8 carbon atoms, one ketone with 8 carbon atoms, one dicarboxilic acid with 10 carbon atoms were identified. Some of these compounds or isomers were also identified in our previous work (Chytiri et al., 2005; Chytiri et al., 2008), where we have studied five-layer  $\gamma$ -irradiated coextruded films based on LDPE containing a middle buried layer of recycled LDPE and external layers of virgin LDPE and PA. Azuma et al. (1983) identified the products from LDPE irradiated with e-beam irradiation and found that the main volatiles were

aliphatic hydrocarbons, aldehydes, ketones and carboxylic acids. More specifically, according to the same authors, aldehydes and ketones are considered to be oxidation products resulting from the reaction with oxygen in air during irradiation, while the formation hydrocarbons during irradiation is caused by the breakdown of short branches of polyethylene. Buchalla et al. (1999) identified hydrocarbons, aldehydes, ketones and carboxylic acids as radiolysis products from PE by thermal desorption-GC/MS, while some amides (mainly pentanamide) were identified from PA. Byun et al. (2007) detected several alcohols as volatile radiolysis compounds due to irradiation degradation of  $\gamma$ -irradiated EVOH films at doses up to 30 kGy. Fintzou et al. (2007) studied the effect of e-beam irradiation in polypropylene syringes at 30, 60 and 120 kGy. A number of radiolysis products, such as hydrocarbons, aldehydes, ketones and carboxylic acids were produced after contact with CH<sub>2</sub>Cl<sub>2</sub> at 60°C for 24 h which increased in concentration with increasing irradiation dose. Similar observations were recorded by the same authors in their previous work (Fintzou et al., 2006a) where they studied the formation of radiolysis products of polypropylene syringes after yirradiation at the same doses.

Data in Table 1 shows that the number and concentration of radiolysis products increased progressively with increasing absorbed dose for all three packaging films (V, R1, R2). More specifically, at 5 kGy the major compounds (>5 μg ml<sup>-1</sup>) indentified in material V were: 2-buten-1-ol (19.6 μg ml<sup>-1</sup>) 4,4-dimethyl-pentene or isomer. At a dose of 10 kGy the major compounds (>10 μg ml<sup>-1</sup>) produced were: 2,2,4-trimethyl-3-pentanone (13.5 μg ml<sup>-1</sup>) and 2-ethoxy-2-methyl-propane (10.7 μg ml<sup>-1</sup>). At a dose of 30 kGy the major compounds (>20 μg ml<sup>-1</sup>) produced were: 2,2,4-trimethyl-3-pentanone (22.8 μg ml<sup>-1</sup>), 2-ethoxy-2-methyl-propane (22.4 μg ml<sup>-1</sup>), 2-buten-1-ol (29.1 μg ml<sup>-1</sup>), an unknown compound (23.4 μg ml<sup>-1</sup>), 2,2,4,4-tetramethyl-

octane or isomers (20.5 µg ml<sup>-1</sup>and 25.6 µg ml<sup>-1</sup>respectively), 3-methyl-nonane (22.6 μg ml<sup>-1</sup>) and 2,2,4,4,6,8,8-heptamethyl-nonane or isomers (45.6 μg ml<sup>-1</sup>). Finally, the major compound produced at dose of 60 kGy (>40 µg ml<sup>-1</sup>) was 2,2,4,4,6,8,8heptamethyl-nonane (56.7 µg ml<sup>-1</sup>).

It must be noted that 1,2-benzene-dicarboxylic acid, detected at 45.31 min, may be a degradation product of Irgafos 168 (tris-(2,4-di-tert-butylphenol)-phosphite) used as a UV stabilizer additive in LDPE. The presence of benzene derivatives as radiolysis products of packaging materials constitutes a potential problem given that benzene and some of its derivatives are toxic and/or carcinogens as has been reported by several authors (Marque et al., 1998; Buchalla et al., 1999; Riganakos et al., 1999; Buchalla et al., 2000; Chytiri et al., 2005).

An important observation to be made is that no new compounds were detected as a result of the presence of recycled LDPE in the multilayer structure, while there were no differences in radiolysis products observed between sample V (containing virgin LDPE as the buried layer) and R1, R2 (containing recycled LDPE as the buried layer) at all absorbed doses (Table 1). This can be attributed to the good quality of the recycled LDPE used originating from a pre-consumer scrap, something that was also observed in our previous work (Chytiri et al., 2005; Chytiri et al., 2008), as well as to the good barrier properties of the external virgin polymer layers used in all experimental structures.

Of the compounds identified 2,4-dimethyl-pentane and 2,5-dimethyl-hexane have been found to irritate skin and may cause long-term adverse effects in the aquatic environment (very toxic to aquatic organisms) (http://www.lookchem.com). 3octanol and 1,2-benzene-dicarboxylic acid have been found to irritate eyes, respiratory system and skin. 2-ethoxy-2-methyl-propane has been found to irritate

eyes and skin (http://www.lookchem.com). Finally, 2,2,4,6,6-pentamethyl heptane may cause lung damage through ingestion (http://www.lookchem.com).

Comparison of present results to those reported in our previous work (Chytiri et al., 2005; Chytiri et al., 2008) shows that e-beam irradiation resulted to the production of a lower number of radiolysis products than  $\gamma$ -irradiation. The higher dose rate of e-beam irradiation compared to  $\gamma$ -irradiation may explain this behavior (Deschenes et al., 1995). More specifically, the lower dose rate of  $\gamma$ -irradiation corresponds to a longer irradiation time to achieve the same dose effect as compared to e-beam irradiation. This inturn results in greater oxidation or degradation of the polymer structure and production of higher number of radiolysis compounds that migrate into the stimulant (Azuma et al., 1983).

Based on present results and those reported in our previous work (Chytiri et al., 2005; Chytiri et al., 2008), it is very important to select the proper irradiation conditions in order to minimize the formation of radiolysis products. A suggested methodology is to carry out irradiation at very low oxygen concentration i.e. under vacuum, or in the presence of nitrogen in combination with temperatures lower than ambient temperature (Goldman et al., 1996; Jahan et al., 2001; Woo and Sandford, 2002). When irradiation is carried out in vacuum most of free radicals formed, decay in the absence of oxygen without any observable transformation to final radiolysis products (Azuma et al., 1984; Jahan et al., 2001; Fintzou et al., 2006; Fintzou et al., 2007a).

# Sensory evaluation

The results of sensory evaluation of potable water after contact with the experimental packaging materials for 10 days at 40°C are summarized in Table 2.

Generally, there was no color transfer to potable water observed after contact with the three packaging materials (irradiated and control samples; results not shown).

However, taste and odor of water were affected in contact with both irradiated

and non-irradiated films. The Taste Transfer to Water (TTW) and Odor Transfer to Water (OTW) in contact with all non-irradiated materials were equal to or lower than the acceptability limit of 1, respectively. According to Bravo et al., (1992) and Ezquerro et al. (2003), volatile organic compounds (aldehydes, ketones, alcohols, hydrocarbons and carboxylic acids) formed by thermooxidative degradation of polyethylene during the extrusion process used to produce multilayer packaging materials, are involved in the unpleasant wax-like or metallic off-odor development. A similar procedure was used for the production of the present coextruded multilayer structures and may be responsible for the slight to moderate taste transfer to water

As shown in Table 2, the TTW increased progressively as irradiation dose increased. At a dose of 60 kGy the taste score was high reaching the values of 3.2, 3 and 3.1 for the materials V, R1 and R2, respectively. These results are in direct correlation with the number and concentration of radiolysis products, which increased progressively with absorbed dose (Table 1). Many authors have reported that volatile products such as aldehydes, ketones and carboxylic acids have a significant contribution to the off-odor/taste of irradiated polymers (Azuma et al., 1983; Azuma et al., 1984; Deschenes et al., 1996; Buchalla et al., 2000).

after contact with non-irradiated packaging materials.

On the other hand e-beam irradiation did not result in substantial differences in the OTW scores with increasing dose. Odor scores corresponding to irradiation doses between 0-30 kGy were lower than or equal to the acceptability limit of 1. Only at a dose of 60 kGy and only for the material R2 odor scores exceeded this limit.

Interestingly, equivalent differences were recorded in taste scores (which proved to be the most sensitive sensory attribute) of potable water after contact with material V as compared to materials containing a recycled middle layer R1 and R2. Odor transfer showed a different pattern. That is, odor scores of table water in contact with material V were significantly lower (p<0.05) than those of materials R1 and R2. In general the recycled polymers are more sensitive to oxidation than virgin polymers resulting in a more evident off-odor development (Pospisil et al., 1995). Such a discrepancy between off-taste and off-odor needs further investigation. According to Devlieghere et al. (1998) a large number of migrating compounds caused an off-odor in untreated and caustic-washed recycled HDPE bottles which could easily be recognized. Stoffers et al. (2004) observed a typical off-odor in water after  $\gamma$ -irradiation at 29 and 54 kGy of LDPE and PA, while Franz and Welle (2004) reported that volatile compounds formed during irradiation of PS and PP affected the odor of water after for 1 day at 23°C.

Present results are in general agreement with those of our previous work (Chytiri et al., 2005; Chytiri et al., 2008). To the best of our knowledge this is the first report in the literature with respect to the effect of e-beam irradiation on sensory properties of multilayer packaging materials containing a middle buried layer of recycled polymer.

# **Conclusions**

Based on above results the following conclusions can be drawn:

1. A large number of radiolysis products are produced during e-beam irradiation treatment of multilayer coextruded high barrier food packaging materials containing a buried layer of recycled low density polyethylene, even at the

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- lower absorbed doses of 5 and 10 kGy (approved doses corresponding to "cold pasteurization" of foods).
- 2. The number and concentration of these compounds increased progressively with increasing dose. The toxicity and the safety of these compounds is mostly unknown and mutagenicity tests are needed given that their potential migration from the packaging material into the contained foodstuff may pose health related problems.
- 3. No differences in radiolysis compounds were observed between films containing a buried recycled LDPE layer and those containing virgin layer. This may be attributed to the good quality of the recycled LDPE used, as well as to the good barrier properties of the external virgin polymer layers.
- 4. No substantial differences were observed in taste scores between potable water in contact with virgin materials and that in contact with materials containing a recycled middle layer. Taste scores were substantially affected at doses as low as 5 kGy and increased progressively as irradiation dose increased. Such scores are in correlation with the number and concentration of radiolysis products, which increased progressively with absorbed dose and can be attributed to the post-irradiation oxidation of recycled material.
- 5. Based on present results it is suggested that recycled LDPE originating from pre-consumer scrap may be used as an internal buried layer comprising up to 50% bw of a high barrier multilayer structure without significant differences in radiolysis compounds produced during e-beam irradiation treatment as compared to virgin materials.

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457 Figure legend	ls
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Figure 1. Structure of five-layer coextruded barrier films V, R1 and R2 (percent values in the right of the figures indicate the by weight contribution of each layer to the multilayer structure).

- Figure 2. GC spectra (A) (detail 3-14 min) and (B) (detail 14-52 min) of irradiated at
- 464 60 kGy multilayer packaging film R2. Peak assignment as in Table 1.

- 466 Figure 3. GC spectra of materials V, R1 and R2 at irradiation doses of 1) 0 kGy, 2) 5
- 467 kGy, 3) 10 kGy, 4) 30 kGy and 5) 60 kGy.

Material V

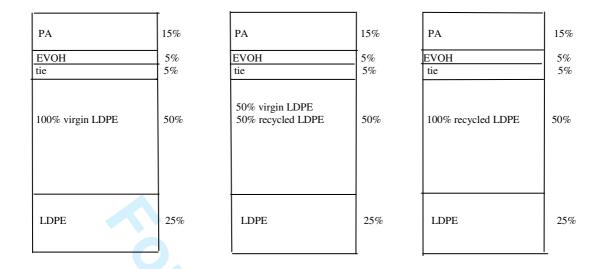
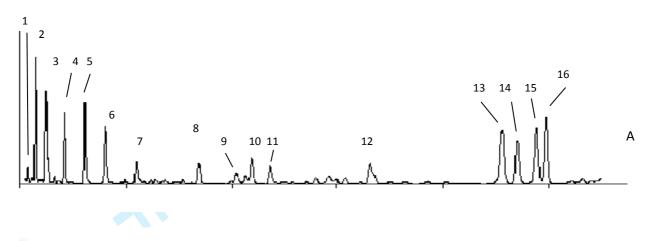
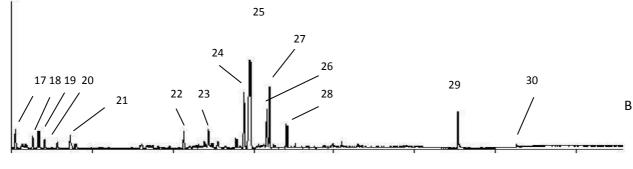
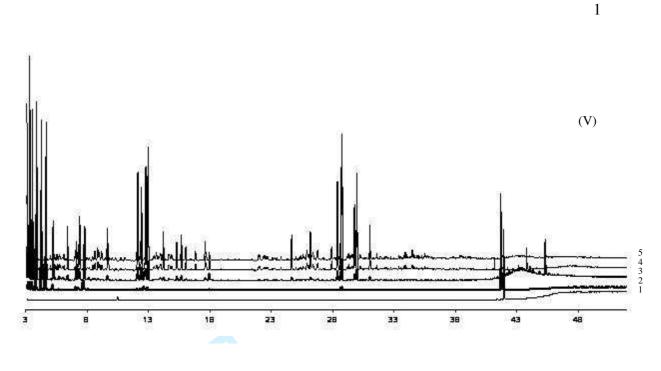
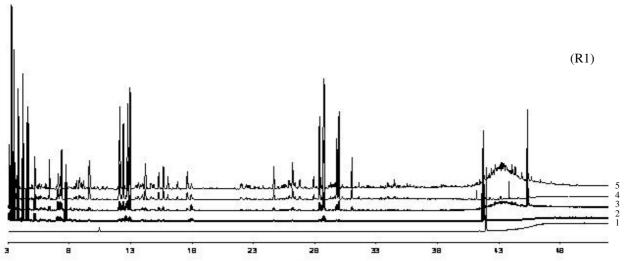


Figure 1









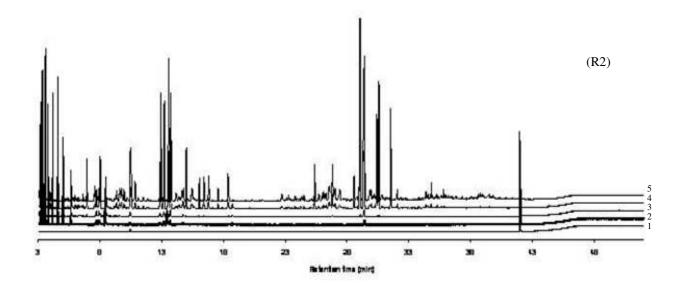


Figure 3

Table 1. Radiolysis products identified in e-beam irradiated multilayer films.

		Results of semi-quantitative analysis (µg ml <sup>-1</sup> )											
			Ma	terial V			M	[aterial ]	R1		N	Aaterial R	22
	Absorbed dose (kGy)	5	10	30	60	5	10	30	60	5	10	30	60
RT <sup>1</sup> (min)	Identified compounds												
3.12	2,2,5- trimethyl-hexane (1) <sup>2</sup>	0.9 ±0.1	2.5 ±0.3	4.8 ±1.1	2.9 ±0.3	1.0 ±0.2	2.3 ±0.1	2.7 ±0.8	2.2 ±0.6	0.5 ±0.1	2.5 ±0.4	2.8 ±0.5	2.1 ±0.1
3.35	3,3-dimethyl-heptane (2)	1.0 ±0.0	3.7 ±0.5	16.8 ±1.2	20.8 ±1.9	0.9 ±0.1	3.5 ±0.4	14.2 ±2.2	21.9 ±3.1	1.9 ±0.2	5.0 ±0.9	12.4 ±1.9	21.8 ±2.5
3.55	2,2,3,4- tetramethyl-pentane or is. <sup>3</sup> (3)	4.2 ±1.0	2.5 ±0.1	13.9 ±3.1	21.6 ±1.0	3.1 ±0.8	4.2 ±0.2	6.5 ±1.8	22.1 ±2.1	4.4 ±0.1	9.6 ±2.1	10.3 ±2.5	22.2 ±2.2
3.60	2,2,5,5- tetramethyl- hexane or is.	0.5 ±0.1	4.3 ±1.0	6.4 ±1.2	1.2 ±0.2	0.3 ±0.0	0.3 ±0.1	1.2 ±0.2	1.3 ±0.1	n.d. <sup>4</sup>	0.4 ±0.0	4.4 ±0.5	1.3 ±0.2
3.86	2,2,4- trimethyl -3- pentanone ( <b>4</b> )	0.7 ±0.0	13.5 ±2.1	22.8 ±2.1	16.5 ±1.0	9.5 ±0.8	12.2 ±3.0	22.0 ±0.9	15.1 ±0.5	11.2 ±1.5	21.1 ±1.9	16.3 ±2.8	15.9 ±0.3
4.24	2- ethoxy-2-methyl-propane (5)	10.3 ±1.9	10.7 ±1.8	22.4 ±1.9	20.2 ±0.2	7.0 ±1.2	9.9 ±1.0	20.4 ±1.8	19.1 ±1.5	8.7 ±1.1	16.4 ±0.6	16.2 ±3.6	20.2 ±2.6
4.63	2-Buten-1-ol ( <b>6</b> )	19.6 ±3.8	18.2 ±1.1	29.1 ±4.0	19.9 ±3.9	13.2 ±3.1	16.4 ±2.3	27.3 ±1.5	17.9 ±2.5	16.1 ±2.9	28.7 ±4.2	20.8 ±4.5	18.9 ±2.6
5.21	2,2,4-trimethyl-3-pentanol or is. (7)	3.7 ±0.9	3.8 ±0.1	8.4 ±1.5	7.9 ±0.1	2.4 ±0.1	3.3 ±0.8	7.0 ±1.1	7.6 ±0.3	3.2 ±0.5	6.1 ±1.1	6.1 ±0.6	7.6 ±1.0
5.75	2,5-dimethyl-2-hexanol	0.7 ±0.1	0.8 ±0.2	1.8 ±0.1	1.7 ±0.2	0.4 ±0.1	0.4 ±0.1	1.7 ±0.0	1.7 ±0.3	1.1 ±0.0	1.2 ±0.1	0.8 ±0.1	1.8 ±0.1
5.97	5-methyl-3-hexanol or is.	n.d.	0.9 ±0.1	1.9 ±0.1	1.4 ±0.2	n.d.	0.8 ±0.0	1.5 ±0.1	0.2 ±0.0	0.8 ±0.1	1.5 ±0.2	1.3 ±0.1	1.4 ±0.1
6.42	2,4-dimethyl- pentane or is. (8)	0.7 ±0.1	1.1 ±0.1	5.5 ±1.1	7.0 ±0.1	n.d.	1.0 ±0.1	4.4 ±0.5	7.3 ±0.2	0.5 ±0.1	1.4 ±0.2	4.0 ±0.8	7.5 ±0.1

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7.10	2,2,4-trimethyl-1-pentanol or is. (9)	2.3 ±0.6	2.6 ±0.8	5.4 ±0.1	4.9 ±0.1	1.7 ±0.3	2.3 ±0.3	4.7 ±0.1	4.6 ±0.6	2.1 ±0.1	4.0 ±0.3	4.0 ±0.1	4.9 ±0.1
7.27	2,4,4- trimethyl-1-pentanol or is.	2.3 ±0.3	2.7 ±0.6	5.4 ±1.3	4.6 ±1.2	1.7 ±0.3	2.3 ±0.3	4.5 ±0.8	4.1 ±0.5	2.2 ±0.2	4.1 ±1.1	3.9 ±0.5	4.3 ±0.6
7.41	2,2- dimethyl -hexanol or is. (10)	1.4 ±0.2	1.9 ±0.3	8.4 ±1.5	11.1 ±1.1	1.0 ±0.1	1.8 ±0.1	7.4 ±0.7	11.3 ±0.9	1.0 ±0.1	2.6 ±0.3	6.4 ±1.2	11.7 ±1.3
7.73	4,4-dimethyl-pentene or is. (11)	8.4 ±1.3	8.2 ±1.4	13.1 ±2.2	8.7 ±0.1	6.2 ±1.1	7.3 ±0.2	11.4 ±1.4	7.2 ±0.5	7.4 ±0.6	12.6 ±2.8	9.1 ±1.7	8.8 ±0.1
8.58	3- octanol	n.d.	0.5 ±0.0	2.6 ±0.3	3.6 ±0.1	n.d.	0.5 ±0.1	2.2 ±0.2	3.2 ±0.6	0.2 ±0.1	0.7 ±0.1	1.8 ±0.3	3.7 ±0.3
8.86	2,2,3,3,5,6,6-heptamethyl-heptane or is.	n.d.	0.7 ±0.1	3.8 ±0.8	5.2 ±1.3	0.2 ±0.1	0.6 ±0.0	2.8 ±0.2	5.1 ±1.1	0.3 ±0.1	0.9 ±0.2	2.7 ±0.2	5.4 ±1.4
9.62	2,2,4,6,6-pentamethyl-heptane or is. ( <b>12</b> )	0.1 ±0.1	1.5 ±0.1	10.4 ±0.1	13.9 ±0.1	0.4 ±0.1	1.3 ±0.1	7.8 ±0.1	13.6 ±0.1	0.8 ±0.1	1.9 ±0.1	7.8 ±0.1	14.7 ±0.1
12.11	Unknown (13)	1.5 ±0.3	4.0 ±0.7	23.4 ±3.0	30.1 ±4.0	1.1 ±0.1	3.5 ±0.4	17.8 ±2.3	31.7 ±2.7	1.5 ±0.1	4.7 ±1.4	17.7 ±3.2	31.8 ±2.1
12.40	2,2,4,4-tetramethyl-octane or is. (14)	1.7 ±0.4	3.4 ±0.5	20.5 ±3.7	22.6 ±3.1	1.2 ±0.1	3.0 ±0.4	15.7 ±2.6	23.9 ±0.9	1.6 ±0.2	4.1 ±0.1	13.5 ±3.2	23.7 ±1.1
12.75	3-methyl-nonane (15)	1.0 ±0.2	3.4 ±0.4	22.6 ±3.8	29.6 ±0.3	0.7 ±0.1	3.1 ±0.2	17.9 ±2.1	31.2 ±0.7	0.8 ±0.1	3.4 ±0.5	17.2 ±2.0	31.0 ±1.1
12.90	2,2,4,4-tetramethyl-octane or is. (16)	2.3 ±0.3	5.0 ±0.9	25.6 ±4.2	32.6 ±3.1	1.7 ±0.3	4.4 ±0.3	20.9 ±2.3	34.4 ±2.8	2.1 ±0.1	5.8 ±0.8	19.4 ±2.8	34.3 ±2.1
13.65	2,6-dimethyl-undecane	n.d.	0.4 ±0.1	2.6 ±0.3	3.4 ±0.3	n.d.	0.3 ±0.0	2.3 ±0.1	3.5 ±0.2	n.d. ±0.1	0.3 ±0.1	2.2 ±0.2	3.5 ±0.2
13.96	Unknown	1.0 ±0.3	1.3 ±0.1	2.7 ±0.1	2.4 ±0.1	0.8 ±0.1	1.2 ±0.2	3.4 ±0.6	2.3 ±0.3	0.9 ±0.0	1.9 ±0.2	2.6 ±0.2	3.6 ±0.5
14.22	2,5-dimethyl-hexane or is. (17)	n.d.	0.8 ±0.2	6.1 ±1.3	8.5 ±1.6	n.d.	0.7 ±0.0	5.0 ±1.3	9.0 ±1.0	0.2 ±0.1	0.8 ±0.1	4.7 ±1.9	8.8 ±2.0

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14.65	Unknown	n.d.	0.7 ±0.1	3.1 ±0.4	3.7 ±0.9	n.d.	0.2 ±0.0	2.5 ±0.3	3.6 ±1.0	0.1 ±0.1	0.6 ±0.1	2.2 ±0.3	1.8 ±0.4
15.30	2,2,4-trimethyl-decane or is. (18)	0.5 ±0.1	1.0 ±0.0	4.3 ±0.3	5.5 ±1.0	0.3 ±0.0	0.8 ±0.1	3.4 ±0.6	5.2 ±1.1	0.5 ±0.1	1.3 ±0.1	3.2 ±0.5	5.3 ±1.5
15.66	2,5,9-trimethyl-decane or is. (19)	0.6 ±0.1	1.1 ±0.1	5.8 ±1.3	7.6 ±1.1	0.3 ±0.0	1.0 ±0.0	4.8 ±0.3	7.6 ±0.4	0.5 ±0.1	1.3 ±0.1	4.4 ±0.9	7.8 ±0.9
16.04	2.2.6-trimethyl-decane or is. (20)	n.d.	0.4 ±0.0	3.0 ±0.3	3.8 ±0.2	n.d.	0.3 ±0.1	2.6 ±0.6	4.0 ±0.1	n.d.	0.3 ±0.1	2.1 ±0.9	4.0 ±0.6
17.62	2,5-dimethyl-decatetrane or is. (21)	n.d.	0.7 ±0.2	4.1 ±0.8	5.7 ±0.5	n.d.	0.5 ±0.1	3.0 ±0.5	6.0 ±0.1	0.1 ±0.1	0.6 ±0.1	3.3 ±0.4	5.9 ±1.0
17.95	3-octanol or is.	1.3 ±0.3	1.7 ±0.1	3.8 ±0.8	2.3 ±0.1	1.1 ±0.1	1.8 ±0.1	2.6 ±0.3	2.1 ±0.5	1.6 ±0.2	2.5 ±0.1	2.2 ±0.5	2.0 ±0.2
24.67	Unknown (22)	0.3 ±0.1	0.8 ±0.1	5.1 ±1.1	7.0 ±0.8	0.2 ±0.0	0.7 ±0.0	4.0 ±0.2	7.3 ±0.1	0.3 ±0.0	0.9 ±0.1	3.9 ±0.5	7.4 ±0.5
25.91	2,4,6-trimethyl-octane or is.	n.d.	0.4 ±0.0	2.6 ±0.4	3.8 ±0.1	n.d.	0.1 ±0.0	2.2 ±0.2	3.8 ±0.2	n.d.	0.4 ±0.1	2.1 ±0.4	4.0 ±0.2
26.18	2,2-dimethyl-1-propanol or is. (23)	0.4 ±0.1	1.2 ±0.1	7.0 ±0.1	9.9 ±0.1	0.3 ±0.1	1.0 ±0.1	5.8 ±0.1	10.9 ±0.1	0.2 ±0.1	1.3 ±0.1	5.4 ±0.1	10.4 ±0.1
26.78	Unknown	n.d.	0.5 ±0.1	2.8 ±0.1	4.5 ±0.1	n.d.	0.4 ±0.1	2.0 ±0.1	3.8 ±0.1	0.2 ±0.1	0.7 ±0.1	2.3 ±0.1	3.9 ±0.1
27.91	2,2,4,4,6,8,8-heptamethyl-nonane or is.	n.d.	0.3 ±0.1	2.5 ±0.1	3.8 ±0.1	n.d.	0.3 ±0.1	2.2 ±0.1	4.5 ±0.1	n.d.	0.2 ±0.1	2.1 ±0.1	3.9 ±0.1
28.38	2,2,3,3,6,8,8-heptamethyl-nonane or is.( <b>24</b> )	0.8 ±0.1	2.7 ±0.1	19.9 ±0.1	24.9 ±0.1	0.6 ±0.1	2.6 ±0.1	16.5 ±0.1	25.7 ±0.1	0.7 ±0.1	2.7 ±0.1	14.1 ±0.1	26.8 ±0.1
28.75	2,2,4,4,6,8,8-heptamethyl-nonane or is.( <b>25</b> )	3.7 ±0.1	8.1 ±0.1	45.6 ±0.1	56.7 ±0.1	2.7 ±0.1	7.6 ±0.1	39.2 ±0.1	57.8 ±0.1	3.3 ±0.1	9.4 ±0.1	33.3 ±0.1	60.9 ±0.1
29.79	2,2,4,4-tetramethyl-octane <b>(26)</b>	0.3 ±0.1	1.7 ±0.1	12.9 ±0.1	16.4 ±0.1	0.2 ±0.1	1.7 ±0.1	10.8 ±0.1	16.8 ±0.1	0.3 ±0.1	1.6 ±0.1	9.6 ±0.1	17.6 ±0.1

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29.97	Unknown (27)	0.6 ±0.1	2.6 ±0.1	19.0 ±0.1	23.0 ±0.1	0.4 ±0.1	2.5 ±0.1	16.3 ±0.1	23.6 ±0.1	0.5 ±0.1	2.5 ±0.1	13.7 ±0.1	24.9 ±0.1
31.03	2,2,4,4,5,5,7,7-octamethyl-octane ( <b>28</b> )	n.d.	0.8 ±0.1	6.3 ±0.1	8.3 ±0.1	n.d.	0.7 ±0.1	5.0 ±0.1	2.0 ±0.1	0.1 ±0.1	0.7 ±0.1	4.6 ±0.1	8.6 ±0.1
41.96	Octadecanoic acid methyl ester (IS <sup>5</sup> ) (29)	10.0	10.0	10.0	10.0	10.0	10.0	10.0	10.0	10.0	10.0	10.0	10.0
45.31	1,2-Benzene-dicarboxylic acid (30)	0.1 ±0.1	7.4 ±0.1	5.9 ±0.1	3.1 ±0.1	0.1 ±0.1	9.9 ±0.1	9.0 ±0.1	15.4 ±0.1	0.5 ±0.1	4.7 ±0.1	13.8 ±0.1	0.1 ±0.1

<sup>&</sup>lt;sup>1</sup>RT: retention time

<sup>&</sup>lt;sup>2</sup>Numbers in parenthesis correspond to peaks in chromatograms in Figure 2 A, B

<sup>&</sup>lt;sup>3</sup>is.: isomer(s)

<sup>&</sup>lt;sup>4</sup>n.d.: not detectable <sup>5</sup>IS: Internal standard



Table 2. Effect of e-beam irradiation on sensory properties<sup>a,b</sup> of table water in contact<sup>c</sup> with multilayer film<sup>d</sup> containing a buried recycled LDPE layer.

Absorbed Dose (kGy)	0	5	10	30	60
Taste Transfer to Water					
V	1.0±0.1	2.1±0.3	2.6±0.3	3.1±0.3	3.2±0.2
R1	0.9±0.1	1.8±0.1	2.1±0.3	2.8±0.1	3.0±0.1
R2	1.2±0.2	1.7±0.3	2.2±0.2	2.8±0.2	3.1±0.0
Odor Transfer to Water					
V	0.0±0.0	0.2±0.2	0.4±0.2	0.8±0.0	0.6±0.1
R1	0.2±0.0	0.5±0.1	0.6±0.1	0.9±0.1	1.1±0.2
R2	0.6±0.1	0.7±0.1	0.9±0.1	1.1±0.1	1.6±0.1

<sup>&</sup>lt;sup>a</sup>Scoring scale 0-4 with: 0 no difference from reference sample-table water packaged in glass; 1 slight difference; 2 moderate difference; 3 strong difference; 4 very strong difference

<sup>&</sup>lt;sup>b</sup> Values are means of seven scores ±SD

<sup>&</sup>lt;sup>c</sup> Film/water contact for 10 days at 40°C

<sup>&</sup>lt;sup>d</sup> Ratio of film (area) to water (weight): 200 cm<sup>2</sup> (100 g)<sup>-1</sup> (ml)