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Original article

# FTIR study of gamma and electron irradiated high-density polyethylene for high dose measurements



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#### ABSTRACT

A reliable and well-characterized dosimetry system which is traceable to the international measurement system, is the key element to quality assurance in radiation processing with cobalt-60 gamma rays, Xrays, and electron beam. This is specifically the case for health-regulated processes, such as the radiation sterilization of single use medical devices and food irradiation for preservation and disinfestation. Polyethylene is considered to possess a lot of interesting dosimetric characteristics. In this work, a detailed study has been performed to determine the dosimetric characteristics of a commercialized highdensity polyethylene (HDPE) film using Fourier transformed infrared spectrometry (FTIR). Correlations have been established between the absorbed dose and radiation induced infrared absorption in polyethylene having a maximum at 965  $\text{cm}^{-1}$  (transvinylene band) and 1716  $\text{cm}^{-1}$  (ketone-carbonyl band). We have found that polyethylene dose-response is linear with dose for both bands up to1000 kGy. For transvinylene band, the dose-response is more sensitive if irradiations are made in helium. While, for ketone-carbonyl band, the dose-response is more sensitive when irradiations are carried out in air. The dose-rate effect has been found to be negligible when polyethylene samples are irradiated with electron beam high dose rates. The irradiated polyethylene is relatively stable for several weeks after irradiation. © 2021 Korean Nuclear Society, Published by Elsevier Korea LLC. This is an open access article under the CC BY-NC-ND license (http://creativecommons.org/licenses/by-nc-nd/4.0/).

#### 1. Introduction

Radiation processing technology consists of subjecting materials to the ionizing radiation in order to modify and/or to improve their physical, chemical, and biological properties [1].

The main sources of ionizing radiation for radiation processing purposes include  $\gamma$ -rays from radioactive isotopes such as Co-60 (<sup>60</sup>Co), electron beams from electron accelerators, and X-rays converted from electron beams [2]. Some of the major industrial applications of radiation processing are the sterilization of single use medical devices, foodstuff preservation and disinfestation, material modification and environmental applications. Because of worldwide growth in radiation processing industry, the role of quality control is a basic requirement. The absorbed dose is a key

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parameter for the quality assurance of all these applications. Radiation dosimetry plays important roles in the quality control of the process, and it is necessary to guarantee suitable treatments of the products. Particularly, as public health and safety depend on dose measurement accuracy, such as food irradiation and medical products sterilization, accuracy requirements must be strict.

Various polymer dosimetry systems are commercially available and are being successfully used for absorbed dose measurements in gamma and electron beam radiation processing industry, such as Harwell polymethylmethacrylate (PMMA) dosimeters [3–5], Cellulose triacetate strips and radiochromic dyed films (FWT-60, B3, Gafchromic) [6–9].

However, some of the dosimetric systems present certain limitable disadvantages with respect to their use [10-12]. A dosimetric system must satisfy some criteria. It should be easy to use and to readout, reproducible, limited variation in response to influence quantities, and low cost. The dosimeter cost should be negligible compared to the radiation processing final price [13].

Polyethylene is a polymer formed from ethylene  $(C_2H_4)$ . The

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generic chemical formula for polyethylene is  $-(C_2H_4)n-$ , where n is the degree of polymerization. Because of its wide uses in radiation processing industry, the effect of ionizing radiation on polyethylene has been the subject of extensive studies [14–16]. The crosslinking is the preponderate effect upon irradiation in vacuum or in inert atmosphere, while chain scission and oxidative degradation occur in the presence of oxygen [17,18]. Besides crosslinking and degradation, the main changes induced by ionizing radiation in polyethylene, are the unsaturation formation [19–21] and, if oxygen is present, the formation of carbonyl and other oxidation products [22,23]. Here, we are mainly concerned with changes in main chain (transvinylene unsaturation) as well as in ketone carbonyl content.

In this study, polyethylene was chosen for its many attractive characteristics: polyethylene is considered among the simplest polymers and relatively low cost when compared with other polymers. When polyethylene is a component of the irradiated product, it serves itself as a quantitative monitor of process success such as in the curing and/or crosslinking of polyethylene [24]. In such cases, dosimeters are not necessary. Clear and transparent PE films are commercially available in large batches and can be used for routine high dose dosimetry after a systematic dosimetric characterization.

The most of commercial dosimetry systems are not designed for use above 100 kGy. Polyethylene is a material sensitive to ionizing radiation and can be used as a dosimeter in a wide dose range up to 1000 kGy in nuclear reactors. Indeed, polymers are present at different levels in the nuclear industry. They are used as constituent insulators for the sheaths of electric cables in the reactor buildings of nuclear power plants. To ensure the proper functioning of these cables in the event of an accident or during normal operation, knowledge of the absorbed irradiation dose is essential for the installation safety.

There are only a few literature studies on polyethylene dosimeter for radiation processing. These studies were carried out using several types of polyethylene and different analytical techniques [25–27].

This study has been undertaken to extend and enrich the valuable data available in the literature with respect of the possible use of high-density polyethylene (HDPE) as radiation sensitive material for high dose measurements in a wide dose range by irradiation with gamma rays and electron beam. The main dosimetric properties of polyethylene have been tested: (1) Effect of the absorbed dose and the irradiation atmosphere on the Fourier transformed infrared spectrometry (FTIR) spectra; (2) Effect of dose rate; (3) Determination of dose response curves; (4) Influence of the irradiation atmosphere on the post-irradiation behaviour.

#### 2. Materials and methods

The film used in this study is a High-density polyethylene (HDPE) type A4009 (melt flow rate at 190 °C and 2.16 kg = 0.8 dg min<sup>-1</sup>, density at 23 °C = 0.96 g cm<sup>-3</sup>, average molecular weight 75,000 g mol<sup>-1</sup>, crystallinity between 70 and 80 %) manufactured by Solvay, Belgium. Its nominal thickness is 28  $\mu$ m. Polyethylene samples have been cut (30 mm × 20 mm) from the same sheets for FTIR analysis.

Electrons irradiations have been performed using a 2.5 MeV Van de Graaff accelerator (LSI, Ecole Polytechnique, Palaiseau, France). Polyethylene films have been irradiated at room temperature in air and under helium atmosphere and with different dose rates of 100, 500 and 1000 kGy/h and with an intensity of up to 250  $\mu$ A. The irradiation cell is connected to the mixing ramp used for the injection of helium for the irradiations.

Three replicate polyethylene samples were irradiated for each point of measurement. After irradiation, the films have been stored under nitrogen at room temperature.

Gamma irradiations have been performed at the Tunisian semiindustrial cobalt-60 gamma irradiation facility [28]. Polyethylene films have been irradiated at room temperature of about 22 °C in air and under nitrogen atmosphere at the dose rate of 5.5 kGy/h. For each point of measurement, three replicate films were irradiated in polyethylene sachets sealed by heat and in nitrogen filled ampoules. After irradiation, the films have been stored in the dark at room temperature from 20 °C to 25 °C and in relative humidity between 40 % and 60 %. The traceability has been established with alanine/EPR dosimetry system in terms of absorbed dose to water traceable to Aerial Secondary Standard Dosimetry Laboratory (SSDL), Strasbourg-France. In order to ensure electron equilibrium conditions, polyethylene films have been put into polystyrene phantom used for in-plant gamma irradiation calibration [29].

Fourier Transform Infrared spectra of polyethylene have been recorded, before and after irradiation, between 500 and 4000 cm<sup>-1</sup> at a resolution of 2 cm<sup>-1</sup> and with 32 accumulated scans using NICOLET Magna IR-750 spectrometer. To observe only the irradiation effect, the non-irradiated spectra were subtracted from the irradiated ones.

#### 3. Results and discussion

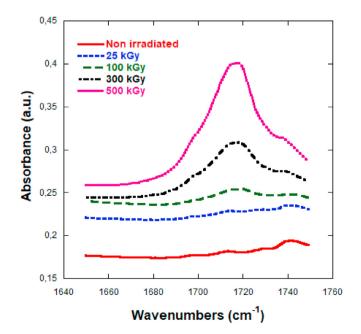
#### 3.1. FTIR absorption spectra of irradiated polyethylene in air

Figs. 1 and 2 show respectively the absorption spectra of the ketone carbonyl band at.

1716 cm<sup>-1</sup> (-CH<sub>2</sub> = 0) and the transvinylene band at 965 cm<sup>-1</sup> (-CH = CH-) of non-irradiated and irradiated polyethylene samples with 2.5 MeV electron beam accelerator in air at doses 25, 100, 300 and 500 kGy. It is noted that their intensities increase gradually with the increase of the dose.

#### 3.2. Effect of the irradiation atmosphere on the FTIR spectra

The changes induced by electron beam irradiation on the polyethylene spectra have been observed on samples irradiated simultaneously in air and in helium with dose of 500 kGy.



**Fig. 1.** FTIR normalized spectra of the ketone carbonyl absorption band at 1716 cm<sup>-1</sup> of electron beam irradiated polyethylene in air at different absorbed doses.

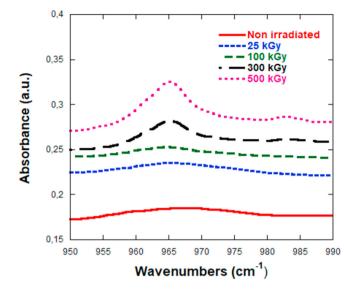
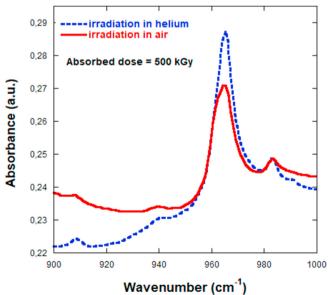


Fig. 2. FTIR normalized spectra of the transvinylene band at 965  $\rm cm^{-1}$  of electron beam irradiated polyethylene in air at different absorbed doses.

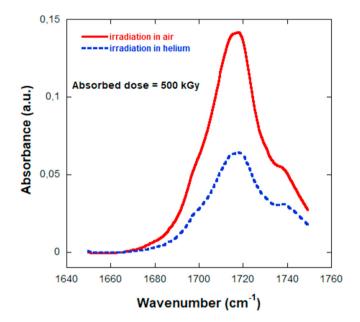
Figs. 3 and 4 respectively show the effect of the irradiation atmosphere on the two bands at 965 cm<sup>-1</sup> and at 1716 cm<sup>-1</sup>. Fig. 3 indicates that the peak at 965 cm<sup>-1</sup> is more intense with irradiation in helium than in air. While Fig. 4 shows that the peak intensity at 1716 cm<sup>-1</sup> is significantly greater for the irradiated sample in air.

These observations are conformed to those of McLaughlin et al. [27] (HDPE) and Wenxiu et al. [25] Low density polyethylene (LDPE). Indeed, McLaughlin has suggested that irradiation formation of unsaturated transvinylene might be more a molecular than a free radical process. Wenxiu et al. have observed that the absorption band of the ketone carbonyl group still appears at 1716 cm<sup>-1</sup> when irradiated in nitrogen, but its intensity is lower than that obtained with the same dose delivered air. This confirms the suggestion of Tabb et al. [30] (HDPE), namely the increase of the ketone function is not due to the free oxygen, but to the oxygen initially dissolved in the polyethylene. When polyethylene is exposed to



beam irradiated polyethylene in air and in helium.

Fig. 3. FTIR normalized spectra of the transvinylene band at 965 cm<sup>-1</sup> of electron



**Fig. 4.** FTIR normalized spectra of the ketone carbonyl absorption band at 1716 cm<sup>-1</sup> of electron beam irradiated polyethylene in air and in helium.

high-energy electron beam in an inert atmosphere such as helium or nitrogen, the oxygen initially dissolved in the polyethylene is totally consumed and is not renewed. If the sample is irradiated in air, the oxygen diffuses into the sample to renew the spent oxygen.

On the other hand, Wenxiu could not detect this band when the polyethylene sample has been stored in vacuum for 12 h before irradiation since there is no oxygen-containing group produced during irradiation.

#### 3.3. Dose response curves

In order to find out the useful electrons dose range for dosimetry use of PE dosimeter, the response curves (absorbance versus dose) have been established in the dose range 25–1000 kGy. All measurements have taken 24 h after irradiation. Figs. 5 and 6 show the response curves corresponding to the increase of absorbance with dose, respectively, for the two absorption bands at 1716 and 965 cm<sup>-1</sup>. Both curves show linear growth up to the maximum doses of current experiments. The FTIR response has not yet reached saturation at these dose levels.

As shown and explained in Section 3.2, Fig. 3 shows that transvinylene band (965 cm<sup>-1</sup>) is more sensitive to radiation in helium atmosphere than in air. Conversely, the response of the ketone carbonyl band (1716 cm<sup>-1</sup>) is more intense when irradiation occurs in air (Fig. 4).

These results agree with McLaughlin et al. [27] and Wenxiu et al. [25].

To minimize the contribution of irradiation atmosphere on the polyethylene response, calibration curves have to be used for different irradiation atmosphere.

#### 3.4. Dose rate effect

Polyethylene films are irradiated in air for 100 kGy with electron beam accelerator (2.5 MeV) at relatively high dose rates 100, 500 and 1000 kGy/h and with gamma rays from  $a^{60}$ Co source (1.25 MeV) at dose rate of 5.5 kGy/h. The FTIR absorbance values at 965 cm<sup>-1</sup> and 1716 cm<sup>-1</sup> bands for each dose rate are shown in Table 1.

From this table, it can be noticed that transvinylene and ketone

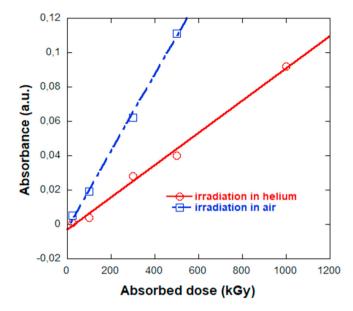


Fig. 5. Dose-response curve for electron irradiated polyethylene, absorbance at 1716  $\rm cm^{-1}.$ 

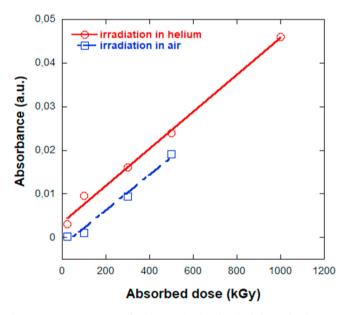


Fig. 6. Dose-response curve for electron irradiated polyethylene, absorbance at 965  $\rm cm^{-1}.$ 

carbonyl absorption bands intensities do not depend strongly on dose rate when irradiated by electron beam high dose rates. This result has been found to be like that described by McLaughlin et al. [27]. Except for the highest dose rate, 1000 kGy/h, where the intensity of the absorbance decreases significantly for both bands.

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#### gamma irradiation in air (300 kGy)

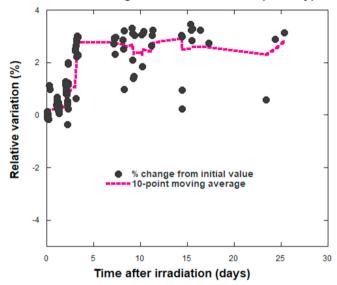


Fig. 7. The percent change in the response ratio for polyethylene film irradiated in air with gamma rays, absorbance at  $965 \text{ cm}^{-1}$ .

This result can be explained by the fact that, at the highest dose rate the number of electrons produced becomes very large in a short period of time, which gives more chance for their fast recombination rather than for the formation of transvinylene and ketone carbonyl groups resulting in less absorbance.

A very significant decrease in absorbance for both absorption bands is observed when the polyethylene films are irradiated with gamma rays compared to the electron-beam irradiated samples. This effect could be avoided by using different calibration curve for gamma rays and electron beam irradiations.

#### 3.5. Post-irradiation effect

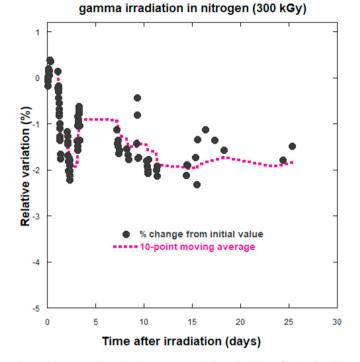
In order to analyse the post-irradiation behaviour as a function of time of transvinylene and ketone carbonyl bands, two polyethylene samples have been irradiated with gamma rays at 300 kGy in air and in nitrogen. The FTIR absorbance changes have been followed up to 25 days. After each measurement, polyethylene samples have been stored in the dark at room temperature from 20 °C to 25 °C and in relative humidity between 40 % and 60 %.

## 3.5.1. Post-irradiation stability of the transvinylene group at 965 $\rm cm^{-1}$

Figs. 7 and 8 show the percent change in response ratio as a function of storage time of transvinylene signal at 965 cm<sup>-1</sup> for polyethylene samples irradiated respectively in air and in nitrogen. The line in the figures is a 10-point moving average. This has been chosen as a simple means of identifying any drift in the ratio within the random scatter of data points. This method makes it possible to

Table 1	
Dose rate effect on the FTIR absorbance bands at 965 cm <sup>-1</sup>	and 1716 $\rm cm^{-1}$ .

Type de rayonnement	Dose rate (kGy/h)	Absorbance (a. u.)	
		965 $cm^{-1}$	1716 cm <sup>-1</sup>
Electrons (2.2 MeV)	100	0.024	0.0080
Electrons (2.2 MeV)	500	0.022	0.0078
Electrons (2.2 MeV)	1000	0.016	0.0050
γ ( <sup>60</sup> Co: 1.25 MeV)	5.5	0.005	0.0013



**Fig. 8.** The percent change in the response ratio for polyethylene film irradiated in nitrogen with gamma rays, absorbance at 965  $\rm cm^{-1}$ .

visualize the longer-term trend of the transvinylene variation, by suppressing the transient fluctuations. The sample irradiated in air experienced an increase of the overall response by approximately 4 % that ranged from -0.4 % to 3.4 % of the initial value. The sample irradiated in nitrogen experienced a decrease of overall response by about 3 % that ranged from 0.4 to -2.3 % of the initial value. These results show that the post-irradiation trend of the transvinylene signal at 965 cm<sup>-1</sup> depend on whether the irradiation is carried out in air or in nitrogen. However, the transvinylene signal is relatively stable for both irradiation conditions. These results are in good agreement with similar observations performed by other

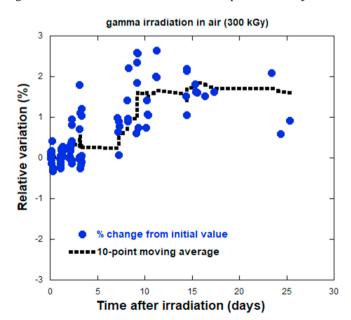


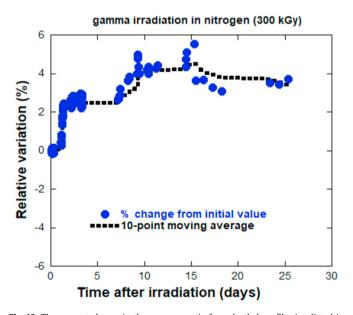
Fig. 9. The percent change in the response ratio for polyethylene film irradiated in air with gamma rays, absorbance at  $1716 \text{ cm}^{-1}$ .

researchers. Carpentieri et al. [22] has reported that transvinylene signal does not increase significantly with storage time after the end of irradiation in air in different polyethylene.

Costa et al. [31] has proved that the crystallinity rise occurs mostly in the early 150 h of ageing in air could be responsible for the apparent transvinvlene decrease detected at the same time of ageing in orthopaedic UHMWPE irradiated in vacuum. On the other hand, McLaughlin et al. [27] have shown that HDPE films irradiated in air with electron beam have been relatively stable for several weeks after irradiation. However, those irradiated in vacuum or in nitrogen have showed a significant post-irradiation decrease in their absorbance by about 10 % over 10 days. McLaughlin et al. have explained this post-irradiation behaviour by the slow oxygen diffusion and the attachment at unsaturations in the polymer backbone at transvinylene sites. Earlier, by studying different types of HDPE and LDPE polyethylene, Dole et al. [32] have proposed that transvinylene post-irradiation decay does result in cross-linking formation. Pearson [33] has proposed a mechanism for radiation crosslinking involving the transvinylene group.

### 3.5.2. Post-irradiation stability of the ketone carbonyl group at 1716 $\rm cm^{-1}$

Figs. 9 and 10 show the percent change in response ratio of ketone carbonyl signal at 1716  $\text{cm}^{-1}$  as a function of storage time for polyethylene samples irradiated respectively in air and in nitrogen. The polyethylene film irradiated in air experienced an overall increase in response of about 3% that ranged from de -0.3 %–2.6 % of the initial value. The film irradiated in nitrogen also experienced an overall increase in response by approximately 6% that ranged from -0.2% to 5.5% of the initial value. These results show that the post-irradiation stability of the ketone carbonyl signal at 1716 cm<sup>-1</sup> depend on whether the irradiation is carried out in air or in nitrogen. Indeed, the ketone carbonyl signal is relatively more stable when irradiated in air. It is obvious that, during irradiation, ketone concentration has grown with the irradiation dose much faster when irradiated in air than in nitrogen. The kinetics of ketone formation are dependent on the concentration of radical species and of oxygen. At the end of irradiation and when the two polyethylene films are exposed to the air, the ketone formation reaction keeps occurring in both samples.



**Fig. 10.** The percent change in the response ratio for polyethylene film irradiated in nitrogen with gamma rays, absorbance at  $1716 \text{ cm}^{-1}$ .

#### 4. Conclusions

In the current study, HDPE was investigated in order to evaluate the possibility to use it as radiation sensitive material for high dose measurements by means of FTIR spectrometer at 965 cm<sup>-1</sup> and 1716 cm<sup>-1</sup>. Correlations have been established between the absorbed dose and radiation induced infrared absorption having a maximum at 965 cm<sup>-1</sup> and 1716 cm<sup>-1</sup>.

We have found that radiation induced transvinylene and ketone-carbonyl formation response of polyethylene are linear with dose in the dose range from 25 to 1000 kGy. The FTIR response has not yet reached saturation at these dose levels. The influence of the irradiation environment on the dose response has showed that, the dose response is more sensitive for transvinylene band at 965 cm<sup>-1</sup> if irradiations are made in helium, whereas the dose response is more sensitive for ketone-carbonyl at 1716 cm<sup>-1</sup> when irradiations are carried out in air.

The dose-rate effect has been found to be negligible for transvinylene and ketone carbonyl absorption bands when polyethylene samples are irradiated by electron beam high dose rates. However, a very significant decrease in absorbance for both absorption bands has been observed when the polyethylene films are irradiated with gamma rays.

The post-irradiation response of the absorption bands at 965 cm<sup>-1</sup> and 1716 cm<sup>-1</sup> has been followed during several weeks after irradiation and found to be relatively stable.

In conclusion, the present investigation has shown good performances of HDPE polyethylene as a radiation sensitive material and that may be used as a routine dosimeter for wide dose range by FTIR spectrophotometry of transvinylene and ketone carbonyl bands, but the effects of the type of radiation and the irradiation atmosphere are not negligible and can give rise to significant errors during its use as routine dosimeter. These effects must be reduced to a minimum; this may be achieved by using in-plant calibration method under conditions similar to those of routine use. Ensuring that the environment conditions are carefully controlled during the use of this dosimeter. In-plant-calibration must be done for each new batch of polyethylene.

#### **Declaration of competing interest**

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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