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Radiation sensitivity and EPR dosimetric potential of gallic acid and its esters

Hasan Tuner^{a,*}, M. Oktay Bal^a, Mustafa Polat^b^a Department of Physics, Faculty of Art and Science, Balikesir University, 10145 Cagis, Balikesir, Turkey^b Department of Physics Engineering, Faculty of Engineering, Hacettepe University, Beytepe Ankara, Turkey

HIGHLIGHTS

- Radiation sensitivity of gallic acid and its esters were studied in intermediate and low radiation dose range using EPR.
- While the irradiated samples of GA were presented complex EPR spectra the esters presented singlet ESR spectra.
- Samples were compared to alanine in terms of the dosimetric point of view.
- The radiation sensitivities of the investigated materials were very low at intermediate doses.
- Lauryl ester of gallic acid was found to present a good sensitivity below 10 Gy.

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ABSTRACT

In the present work the radiation sensitivities of Gallic Acid anhydrous and monohydrate, Octyl, Lauryl, and Ethyl Gallate (GA, GAm, OG, LG, and EG) were investigated in the intermediate (0.5–20 kGy) and low radiation (< 10 Gy) dose range using Electron Paramagnetic Resonance (EPR) spectroscopy. While OG, LG, and EG are presented a singlet EPR spectra, their radiation sensitivity found to be very different in the intermediate dose range. At low radiation dose range (< 10 Gy) only LG is found to be present a signal that easily distinguished from the noise signals. The intermediate and low dose range radiation sensitivities are compared using well known EPR dosimeter alanine. The radiation yields (G) of the interested material were found to be 1.34×10^{-2} , 1.48×10^{-2} , 4.14×10^{-2} , and 6.03×10^{-2} , 9.44×10^{-2} for EG, GA, GAm, OG, and LG, respectively at the intermediate dose range. It is found that the simple EPR spectra and the noticeable EPR signal of LG make it a promising dosimetric material to be used below 10 Gy of radiation dose.

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1. Introduction

Ionizing radiations generally produce some intermediates that have unpaired electron. The Electron Paramagnetic Resonance (EPR) spectroscopy is very sensitive technique in detection materials that have unpaired electron. This sensitivity makes EPR spectroscopy one of the most used methods to detect the exposed radiation dose after the irradiation or radiation accident (Bradshaw et al., 1962; Ikeya, 1993). A high radical yield, linear EPR signal intensity increase with dose, narrow linewidth and stable signal at room temperature are the criteria that the candidate dosimetric materials are expected to be met (Ikeya et al., 2000; Lund et al., 2002). Alanine (AL) was proposed to be used as a dosimetric material in intermediate and high radiation dose

ranges (ASTM, 1999; Bradshaw et al., 1962; Kojima and Tanaka, 1989; Regulla and Deffner, 1982). Although, some researcher reported that AL could be used in the determination of radiation doses below 10 Gy by using some extra techniques (Anton, 2006; Bafa and Kinoshita, 2014; Castro et al., 2006; Sharpe et al., 1996; Sharpe, 2003; Haskell et al., 1998) many researchers exploring to find new materials sensitive to low radiation doses (Alzimami et al., 2014). In this regard, smart phone screen glass, sugar, some tartrate salts, compounds of formic acid and dithionate salts have been evaluated in the literature (Fattibene et al., 2014; Trompier et al., 2011; Yordanov et al., 2002; Mikou et al., 2009; Olsson et al., 2000; Yordanov and Gancheva, 2004; Bartolotta et al., 2001; Tuner and Korkmaz, 2009; Korkmaz et al., 2012; Bal and Tuner, 2014a; Vestad et al., 2003; Gustafsson et al., 2004; Danilczuk et al., 2008; Baran et al., 2006).

While radiation and oxidation effect of GA in aqueous solution, and powder form of propyl gallate have been reported in the

* Corresponding author. Fax: +90 266 612 12 15.

E-mail address: htuner@balikesir.edu.tr (H. Tuner).

literature using different spectroscopic methods (Eslami et al., 2010; Melo et al., 2009, 2011; Bal and Tuner, 2014b) the dosimetric features of GA and its esters have not been reported. Therefore the aim of the present work is to investigate the radiation sensitivity and dosimetric features of different compounds of gallic acid in the dose ranges of 0.5–20.0 kGy, and 0.74–10.00 Gy using EPR spectroscopy.

2. Material and methods

Gallic acid (3,4,5-trihydroxybenzoic acid) is one of the most important polyphenolic compounds (Eslami et al., 2010; Tachibana et al., 2004; Friedman and Jurgens, 2000). There are two forms of gallic acid; the anhydrous (GA) and monohydrate (GAm) forms. Gallic acid octyl ester (OG), lauryl (LG) or dodecyl ester, and ethyl ester (EG) were widely used as additive in food and cosmetic industry, and have the E numbers of E311, E312 and E313, respectively. It is reported that gallates have an antioxidant, anticancer, antiviral, antifungal properties in the literature (Aruoma et al., 1993; Inoue et al., 1995; Gunckel et al., 1998; Yen et al., 2002; Gomes et al., 2003; Ow and Stupans, 2003; Li et al., 2005; Lu et al., 2006, 2010; Priscilla and Prince, 2009; Nabavi et al., 2012; Kubo et al., 2001; Xin et al., 2014). The molecular structures of the interested materials are given in Fig. 1. The gallate samples were supplied from Aldrich and stored at normal laboratory conditions. No further purification was performed. All irradiation were performed at ambient temperature in air using samples in well closed polycarbonate vials. The ^{60}Co source (PX- γ -30 Isslodovatelj) with a dose rate of 0.65 kGy/h, and ^{137}Cs source (Mark I-22M) with a dose rate of 8.83 Gy/min were used to irradiate the samples to the intermediate and low dose ranges, respectively. The dose rate at the sample sites was measured by a Fricke dosimeter with an uncertainty of ± 1 Gy/min. A-type uncertainty calculations were performed related to the signal intensities, and they were determined from 5 independent spectra. The EPR measurements were carried out using Bruker EMX-131 X-band EPR spectrometer equipped with a high sensitive cylindrical cavity.

The radiation sensitivity of the investigated samples were determined by calculation of the radiation yield value (G), which is described as number of radicals produced by the absorption of 100 eV of radiation energy. The area of the absorption curves of the EPR spectra is proportional to the number of the radicals which have unpaired electrons. Thus calculation of the spectrum area is important to determine the G -value. In this purpose the area of the absorption curve was calculated by double integration of the experimental first derivative EPR spectra using the Bruker WINEPR program. This method was described by Barr et al. (1998).

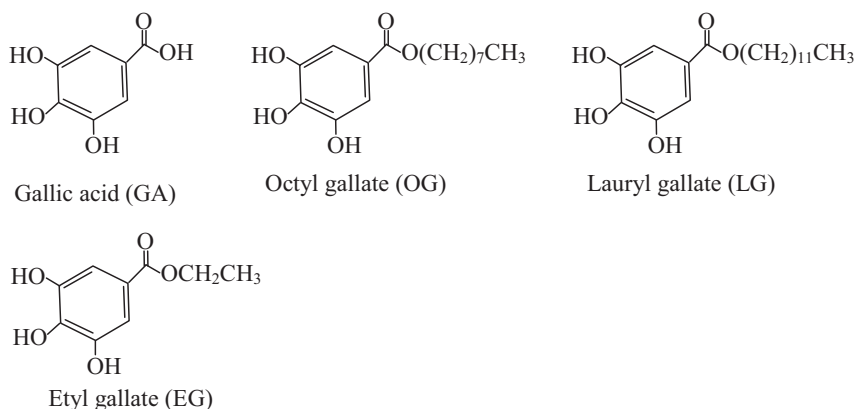


Fig. 1. Molecular structures of gallic acid and its esters.

The calculated areas were normalized to the mass and radiation dose, and the average of the normalized areas was used to determine the G -value of the interested materials. The formula of $G_{(X)} = (A_X/A_{STD}) \times G_{STD}$ was used to determine the relative G -value. Here A_X and A_{STD} are the average of the normalized areas of the investigated and the standard material, respectively, and G_{STD} is the radiation yield value of the standard material. In the present work alanine (AL) was used as the standard material. The AL G -value proposed by Ikeya (1993) was used in the present work ($G_{AL}=1$). Ikeya also accepted that the number of the radiation-induced radical number per kg is $\sim 6.3 \times 10^{16} \text{ Gy}^{-1}$ for material having the G -value equal to 1.

3. Experimental results and discussion

While the unirradiated samples were not presented any EPR signal, irradiated samples were presented different EPR spectra. Namely, irradiated GA and GAm were presented one intense and many weak EPR signals, and irradiated OG, LG and EG were observed to present a singlet EPR signal (Fig. 2). Although, the irradiated OG, LG and EG were observed to have almost the same linewidths (~ 0.64 mT) the g value of OG was measured to be different than other's ($g_{OG} = 2.0024$, $g_{LG} = 2.0046$, $g_{EG} = 2.0045$, and $g_{GA-GAm} = 2.0046$). The EPR spectra are given together in Fig. 2 to make comparison. The EPR signal intensities of GA and EG were multiplied by 10 to make them visible and comparable with other gallates EPR spectra (Fig. 2).

3.1. Intermediate dose range findings

Samples irradiated at the dose of 0.5, 1.0, 3.5, 5.0, 11.0, 15.0 and 20.0 kGy were used to determine the dosimetric features of the interested materials. The EPR spectra are recorded at the same environment and spectrometer conditions, and normalized to the mass of the samples. The operation conditions were as follows: central field, 349.5 mT; microwave power, 0.5 mW; microwave frequency, ~ 9.86 GHz; scan range, 10 mT; modulation amplitude, 0.2 mT; receiver gain, 2.0×10^4 ; modulation frequency, 100 kHz; sweep time, 83.89 s. The dose–response curves of gallates are given in Fig. 3. As it is seen from the figure each compounds were started to saturate above 5.0 kGy with different rates. Namely, the EPR signal intensities were increased almost linearly with increasing the radiation dose, then increasing the radiation dose did not cause a significant increase in the signal intensities, and the intensities were started to saturate (Fig. 3). An exponential function has the form of $I_{(D)} = I_{\max}(1 - e^{-aD})$ is used to determine the

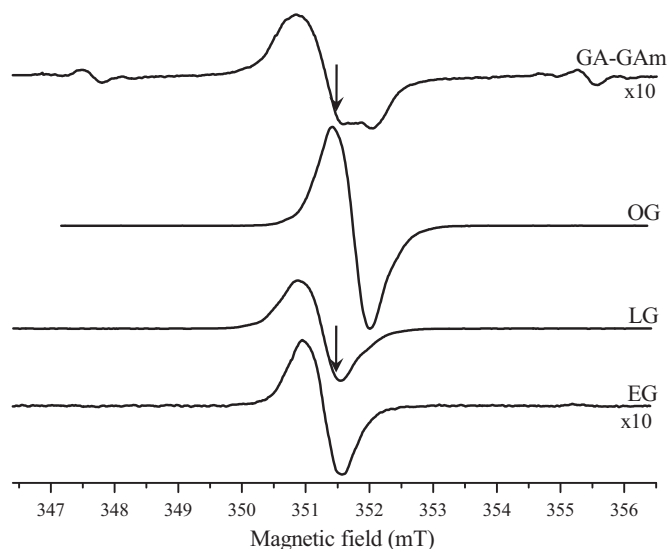


Fig. 2. EPR spectra of gallic acid and its esters irradiated at 11 kGy. Arrows indicate the position of the DPPH g value ($g=2.0036$).

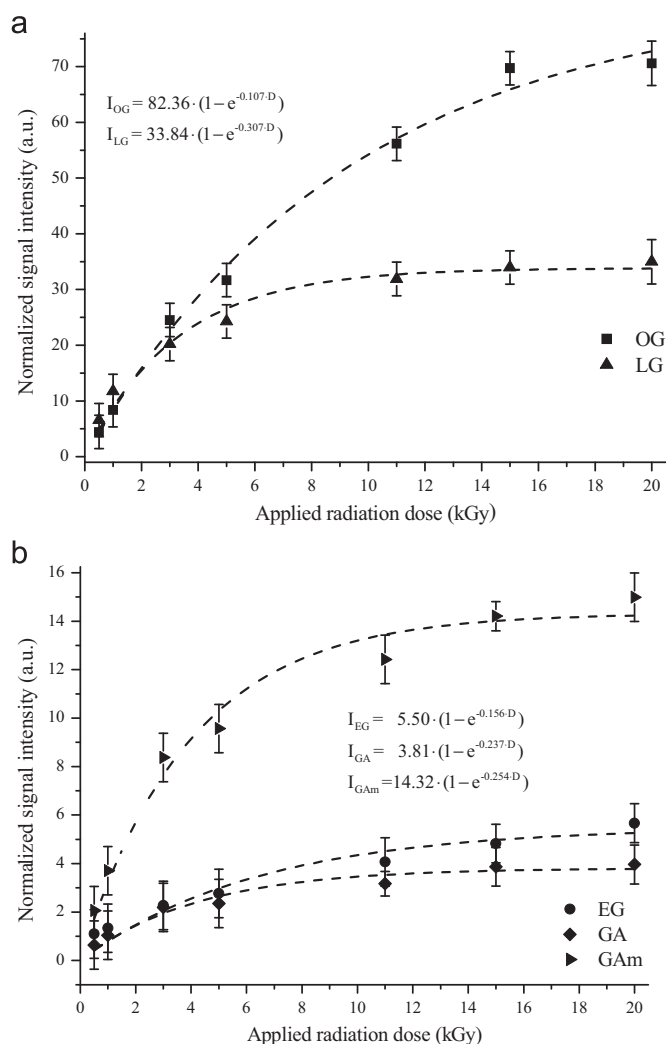


Fig. 3. Dose–response curves at the intermediate dose range. (a) OG (■), and LG (▲); (b) GA (◆), GAm (▴), and EG (●).

experimental dose–response data (Fig. 3). Here I_{max} represents the maximum intensity, and a is a parameter to find.

The areas of the investigated materials were calculated at the linear parts of the dose–response curves. Thus the areas were calculated for the first three doses of gallates and all doses of AL. The normalized spectrum areas of the interested materials and AL are given in Fig. 4. While the response of OG at high doses (> 10 kGy) is better than other gallates (Fig. 3a), the normalized spectrum area findings were showed that the radiation yield was lower than LG in the linear part of the dose–response curve (Fig. 4). It is also found that the radiation sensitivities of the interested materials are very small compared to the AL sensitivity (Fig. 4). The relative G-values of LG, OG, GAm, GA, and EG were calculated to be 9.44×10^{-2} , 6.03×10^{-2} , 4.14×10^{-2} , 1.48×10^{-2} , and 1.34×10^{-2} , respectively.

The decays of the EPR signal intensities of the interested materials were also investigated during two months of storage time under laboratory condition open to air. A sample irradiated to 11 kGy was used, and its spectra were recorded at a regular time intervals. While the signal of EG was completely disappeared almost after three days of storage about 60% of the signal intensities of other gallates were found to be decayed after two months of storage.

3.2. Low radiation dose findings

The radiation sensitivities of the interested materials were also investigated below 10 Gy. To achieve this goal samples irradiated at the dose of 0.74, 1.50, 2.20, 5.00, and 10.00 Gy were used. The EPR spectra of irradiated GA, OG and EG were not distinguished from noise below 10 Gy, only spectra of LG were observed. EPR spectra of LG and AL irradiated with a dose of 1.50 Gy are given together in Fig. 5 to make a comparison. From line intensities measurement points of view (dose–response curve) both compounds were found to have almost the same behavior (Fig. 6). Namely, the slopes of the dose–response curves were measured to be 3.98 and 4.17 for LG and AL, respectively. On the other hand, from radiation yield point of view the picture was slightly different, thus the relative G-value of LG was calculated to be about 0.10 at low radiation dose range.

The modulation amplitude effect on EPR signal intensity of LG was also investigated. The intensity is increasing linearly with modulation amplitude until 0.5 mT. Subsequently it is increasing non-linearly until achieving a maximum at about 1.2 mT, than start to decrease due to the linewidth broadening (inset of Fig. 6).

4. Conclusion

Irradiation was caused different effects on gallic acid and its esters. While the gallic acid esters were presented singlet EPR spectra the EPR spectra of GA and GAm were observed to be complex (Fig. 2). The dose–response curves of interested materials were found to be in accordance with a function has the form of $I_{(D)} = I_{max}(1 - e^{-aD})$, which were started to saturate about 5.0 kGy of radiation dose. This finding makes the interested materials are not suitable dosimetric materials at the intermediate radiation dose ranges. Additionally, the relative G-values of the interested materials were calculated to be very low compared to the AL's. Thus, the relative G-values of LG, OG, GAm, GA, and EG were found to be 9.44×10^{-2} , 6.03×10^{-2} , 4.14×10^{-2} , 1.48×10^{-2} , and 1.34×10^{-2} , respectively. However, the relative G-value of OG was been high enough at the intermediate radiation doses, the EPR spectra of it below 10 Gy were not distinguished from the noise signals even at different spectrometric conditions (high modulation amplitude, microwave power and receiver gain). Only

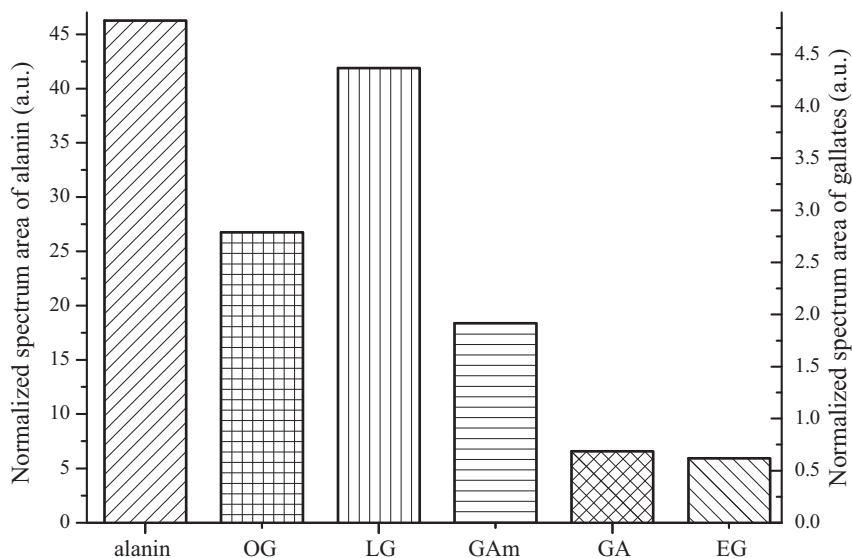


Fig. 4. The average of the normalized spectrum areas of the radiation doses between 0.5 and 5.0 kGy.

the spectra of LG were observed at radiation doses below 10 Gy (Fig. 5). While the radiation induced radical structures of the investigated gallates were accepted to be similar the radiation sensitivities were found to be different. The similar behavior was found in the literature for tartrates, compounds of formic acid and dithionate salts (Olsson et al., 2000; Yordanov and Gancheva, 2004; Bartolotta et al., 2001; Tuner and Korkmaz, 2009; Korkmaz et al., 2012; Bal and Tuner, 2014a; Vestad et al., 2003; Gustafsson et al., 2004; Danilczuk et al., 2008; Baran et al., 2006). While the intermediate dose-response (sensitivity) of AL was high enough, Bal and Tuner (2014a) reported that the radiation sensitivities of the tartrates and some other well known radiation sensitive materials at low doses were comparable with the AL sensitivity. The

relative G -value of LG was calculated to be higher than its value at the intermediate radiation dose range. This unexpected increase in the relative G -value of LG was concluded to be due to the using different radiation source.

It is also concluded that while the radiation sensitivity of GA and its esters were low at intermediate radiation dose range the radiation sensitivity of LG in the low dose range (0.74–10 Gy) was comparable with the sensitivity of AL, from the signal intensity measurement point of view. Its simple EPR spectra, tissue equivalent molecular structure, and linear dose–response curve at the low radiation dose range were indicated that LG has the potential to be used as low dose dosimeter. However, the fast decay at room temperature seems to be its negative features.

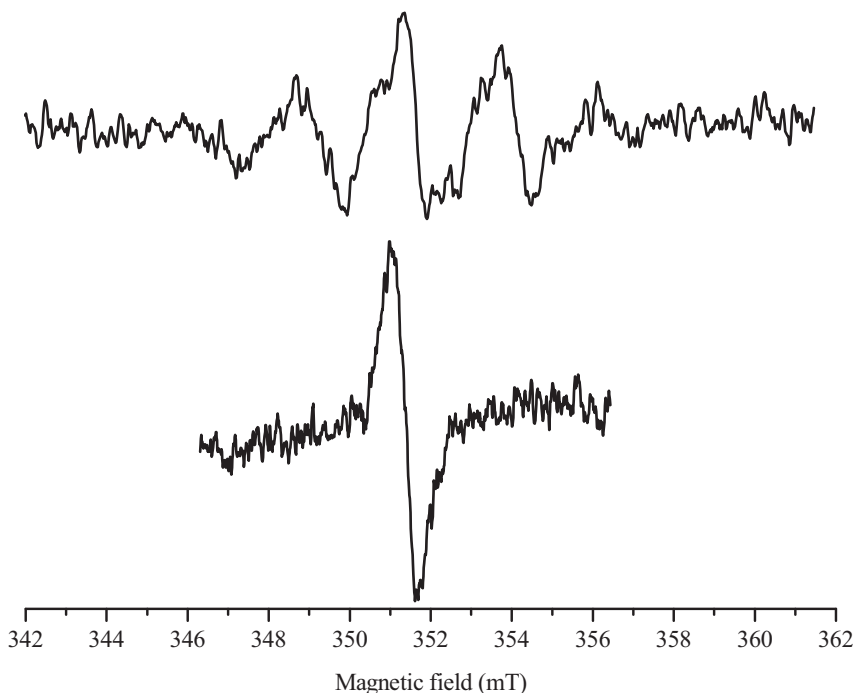


Fig. 5. Normalized EPR spectra of LG and AL samples irradiated to a dose of 1.5 Gy.

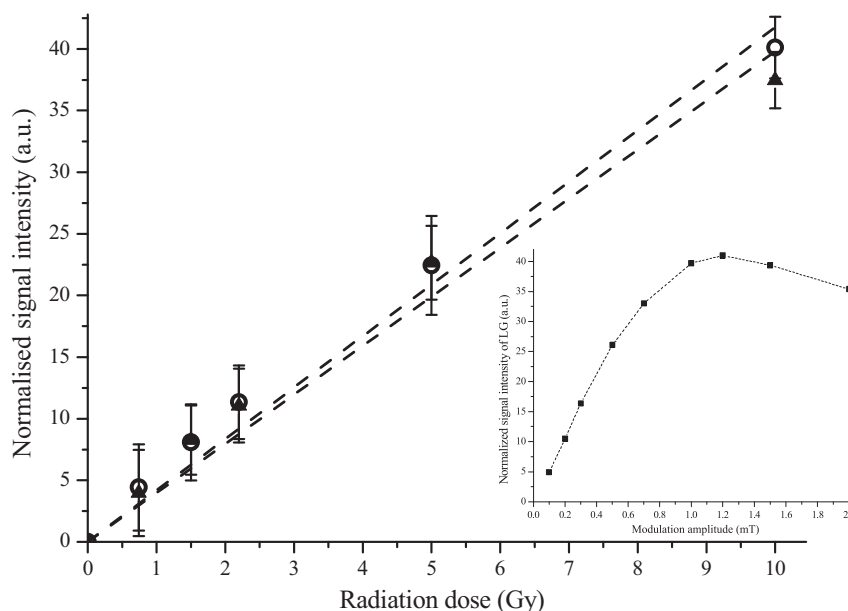


Fig. 6. Dose response curves of LG (▲) and AL (○) at the low dose range (inset: modulation amplitude dependency of LG).

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