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ORIGINAL PAPER

Stability study of free radicals in 10 MeV electron beam irradiated quartz as an EPR dosimetry method

Farhood Ziaie,
Ahmad Mohammadzadeh,
Mohammad Modarresi

Abstract. In this work the stability of free radicals induced by a 10 MeV electron beam in quartz samples was studied. The investigations have been done for different doses and different dose rates. For this reason, quartz samples in powder form were irradiated at different dose rates, i.e. 100, 260 and 630 kGy/min for absorbed dose range of ~ 5–80 kGy using the 10 MeV electron beam radiation. The dose values were validated using a polystyrene calorimeter system as a reference standard dosimetry system. The EPR optimum system parameters were defined and EPR responses at room temperature in air were measured and compared. The EPR signal, which is due to the free radicals induced by electron beam, was also investigated at different time intervals to check its stability. The main objective of this work was to study the feasibility of using quartz as a dosimeter. Thus, the variation of EPR signal intensities with the absorbed dose values were evaluated and plotted in a graph. The results show that the variation of dose rate, do not affect considerably the EPR response.

Key words: quartz dosimeter • EPR response • dose rate • electron beam • EPR signal stability

F. Ziaie✉
Agricultural, Medical and Industrial Research School (AMIRS),
Nuclear Science and Technology Research Institute,
P. O. Box 31485-498, Karaj, Iran,
Tel.: +98 261 442 4073, Fax: +98 261 446 4058,
E-mail: fziaie@nrcam.org

A. Mohammadzadeh
Nuclear Science Research School,
Nuclear Science and Technology Research Institute,
Tehran, Iran

M. Modarresi
Department of Physics,
Islamic Azad University of Shahre-Rey Branch,
Tehran, Iran

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Introduction

Many properties of a material, such as physical shape, crystalline or amorphous form, and particle size, may affect the quality of EPR response. The effects of mechanical treatment to a material such as grinding, crushing, and particle size on EPR signal were reported earlier [1, 9]. The EPR technique is a well known and well established method for dose measurement in several fields like radiation processing and retrospective dosimetry [8–13]. Many different materials such as alanine [5, 7, 12, 14], tooth enamel, and bone [2, 6, 12, 13, 15] have been extensively studied for their applications in radiation dosimetry using the EPR technique. It is also reported that the quartz materials have shown a good response to gamma rays in the dose ranges used for radiation processing and retrospective dosimetry, using the EPR techniques [8, 9]. These reasons plus the non-destructive nature of the radiation-induced defects when using the EPR technique, suggest that quartz could be a possible candidate for dose measurements in radiation processing and retrospective dosimetry. The main purpose of this study was to investigate the stability of the EPR response of quartz irradiated at different doses and dose rates with a 10 MeV electron beam.

Experimental procedures

Sample preparation

EPR sample holder tubes, made by the WILMAD Glass Company (USA) were used in this investigation. The CFQ tubes was powdered manually using a mortar and a pestle, followed by a milling machine, followed by separation of grain sizes of 30–50 mesh.

Sample irradiation

The powder form samples of about 35 mg, packed in plastic packages, placed in the polystyrene phantom were irradiated along with a polystyrene calorimeter to measure precisely the absorbed dose. The irradiation in the dose range of ~ 5–80 kGy was performed under different dose rates of 100, 260 and 630 kGy/min using the 10 MeV electron beam from a Rhodotron TT200 type electron accelerator.

EPR measurements

The samples were put into quartz thin-wall EPR tubes (4 mm in diameter) and measured with a Bruker EMS--104 spectrometer operating in X-band. The EPR spectrometer parameters used for this study were: 0.715 mT modulation amplitude, 100 kHz modulation frequency, 12.4 mT scan width, 1024-point field resolution, 20.5 ms time constant, 10.5 s sweep time, 20 dB receiver gain, and 4 number of scans. The microwave power was 19.9 mW. The EPR responses were measured as peak--to-peak height for the most intense EPR lines (first derivative of the absorption spectra as shown in Fig. 1) per sample mass. Reproducibility of the EPR signal intensity was done by repeating the EPR measurements, with the same instrument settings, four times for each sample and the mean values are reported with their standard deviation of the mean (SD). In our work the peak-to-peak radiation-induced signal height was calculated using a signal maximum at $g = 2.0261$, a signal minimum at $g = 2.0057$ and a position on base-line at $g = 2.0159 \pm 0.0001$. These characteristics indicate that the radical centers responsible for the signals are of the types of the well known E′-centers, the peroxy radicals (PORs) with the non-bridging oxygen hole centers (NBOHCs), which have been reported by many researchers [3, 4].

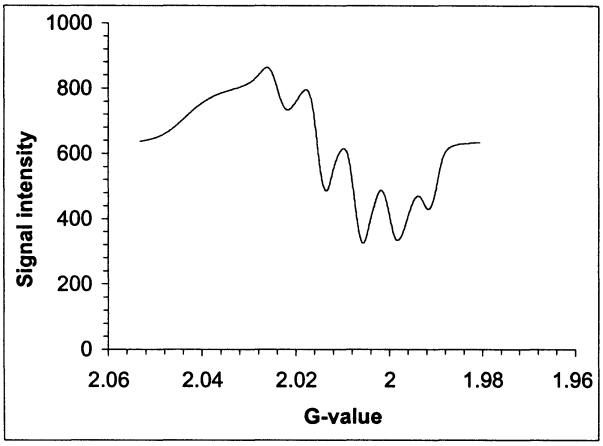

Fig. 1. The EPR signal of the quartz sample irradiated at ~ 5 kGy (signal intensity is in arbitrary units).

Results and discussion

Figure 2 presents the variation of EPR signal intensities as a function of dose for different time intervals and their associated fading for quartz samples irradiated with the 10 MeV electron beam at a dose rate of 630 kGy/min. The recombination of the free radicals induced by the electron irradiation could be seen in the EPR response reduction. According to the published literature [3, 4], there is a thermally-induced decay of E′-centers, and recovery to defect free glass. The generally accepted mechanism is that the E′-centers are converted mostly to PORs upon annealing at about room temperature due to diffusion of interstitial oxygen atoms and molecules already present in the quartz structure. The dose rate effects on the quartz samples are investigated and the results were compared via different doses in Fig. 3. The results do not show any significative difference between the curves associated with the different dose rates.

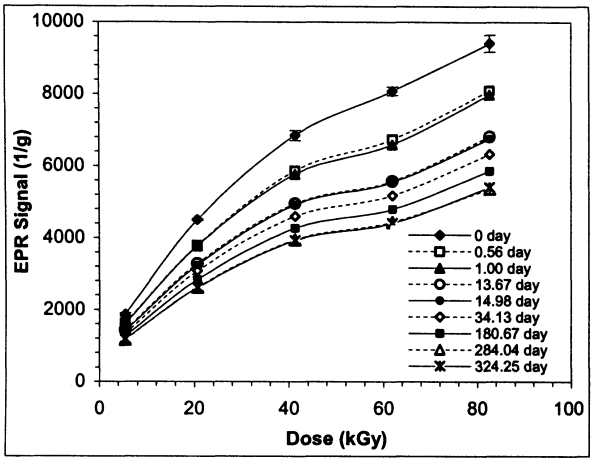

Fig. 2. Variation of the EPR signal intensity as a function of dose at different time intervals after irradiation. The dose rate was 630 kGy/min for all the samples.

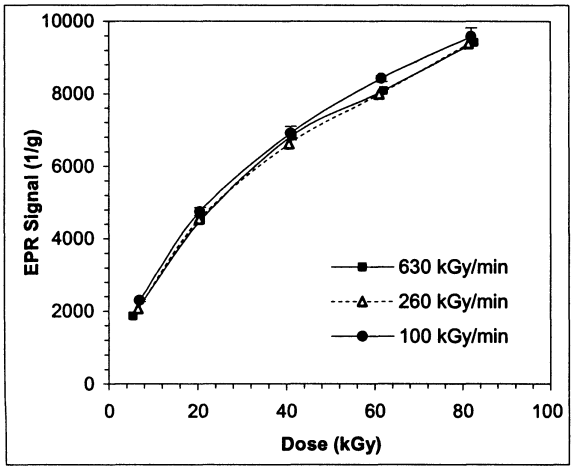

Fig. 3. Variation of EPR signal intensity for different dose rates of the e-beam irradiated samples (the EPR measurements were performed just after irradiation).

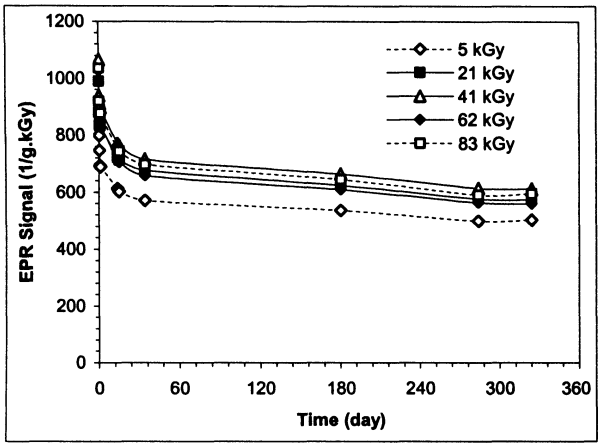

Fig. 4. Variation of EPR response (normalized to the square root of absorbed dose in kGy) of the samples with time after irradiation.

The main idea of this work was to study the use of quartz as an EPR dosimetry system. Therefore, changes in the EPR response with the time could involve an error in the estimated dose value. Thus, the variation of EPR response with time was investigated and compared for different radiation doses. To normalize the results to the irradiated dose value, the EPR response were divided by the square root of the irradiated dose value for each as reported in Fig. 4.

It can be seen that the curves associated with different doses were almost coincident by an acceptable uncertainty except the one with the lowest dose of 5 kGy. The reason for this misbehavior is due to the used reference dosimeter, polystyrene calorimeter, which shows no good precision in the lowest measurable dose and, therefore, the measured values for the 5 kGy irradiated samples are not reliable. Moreover, the average EPR response at each time interval presented in Fig. 4 are reported in Fig. 5. A logarithmic curve was fitted to the mentioned average values with a very good correlation factor (R^2). The fitted curve is presented as:

(1) $$S_{m,D} = A \ln(t) + B$$

(2) $$S_{m,D} = s/(m\sqrt{D})$$

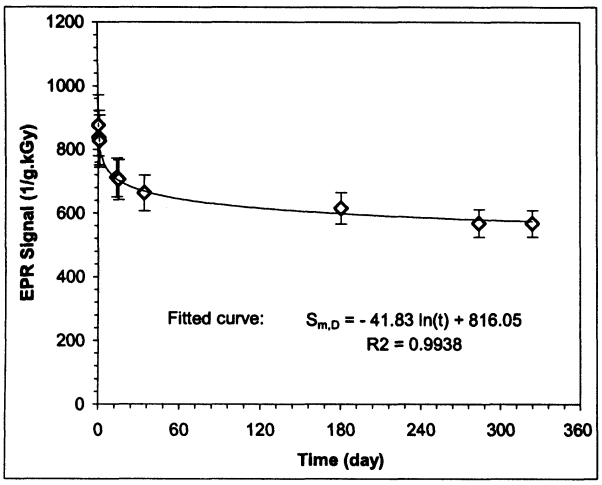

Fig. 5. Variation of average normalized EPR response of the samples with time after irradiation.

where $S_{m,D}$ is the EPR response (S) which is normalized to the sample mass (m) and square root of the radiation dose (D), t is the time after irradiation, and A and B are the constant values derived from the fitted curve in Fig. 5, which are –41.83 and 816.05, respectively. Thus, by knowing the dose at a fixed time after irradiation Eq. (3) can be used to calculate the real radiation dose value just after irradiation and *vice versa*.

(3) $$D = \left(\frac{S}{m(A\ln(t) + B} \right)^2$$

Conclusion

The EPR signal intensity of the irradiated quartz samples decreases with time. These changes can be fitted by a logarithmic expression; this helps with the calculation of the real dose by applying the correction factor that accounts for the fading of the EPR signal for any other post-irradiation time period. The EPR signal intensity variation from the electron irradiated quartz samples plotted vs. the radiation dose can be used as a calibration curve for doses below 80 kGy. The EPR responses of quartz samples are independent of dose rates.

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