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Characterization of the chemical and structural modifications induced by X rays on the HEMA based polymer gel

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Abstract: The use of polymer gels in the radiation dosimetry field is rapidly increasing due to the possibility of 3 dimensional (3D) dosimetry. The aim of this study is to produce a new polymer gel with high dose sensitivity. This involved the production of polymer gel compositions containing different percentages of 2-hydroxyethyl methacrylate (HEMA) monomer and Di(ethylene glycol) dimethacrylate (DEGDMA) and 1-Vinyl-2-pyrrolidinone (VP) crosslinkers and these gels were irradiated with radiation dose between 0.5 Gy to 11 Gy, using 6 MV X-ray energy of the medical linear accelerator. The degree of polymerization was assessed by using magnetic resonance imaging (MRI) based on the R_2-dose response. Then, Fourier Transform Infrared Spectroscopy (FTIR) analysis and Scanning Electron Microscope (SEM) images of the gels were taken. Polymer gels consisting of DEGDMA as crosslinker and Tetrakis (Hydroxymethyl) phosphonium chloride (THPC) as antioxidant were found to have a potential for use in radiation therapy dosimeter. The concentration of HEMA showing the most effective dose response was identified as 12 wt%. It was found that HEMA polymer gels containing DEGDMA crosslinker provide a better dose response than HEMA and HEMA-VP normoxic polymer gels.

Keywords: Dosimetry concepts and apparatus; Radiotherapy concepts; MRI (whole body, cardiovascular, breast, others), MR-angiography (MRA); Radiation damage evaluation methods

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Contents

1 Introduction

Radiation dosimetry is of paramount importance in modern radiotherapy because it ensures the accurate delivery of radiation therapy, enhances patient safety, enables treatment adaptation, and supports ongoing research and development efforts in the field. It plays a central role in optimizing the balance between effectively treating cancer and minimizing the potential side effects on healthy tissues [1].

In radiation dosimetry, the energy accumulated in the environment by exposure of the material to ionizing radiation can be quantitatively determined. Numerous materials have been investigated for use as a dosimeter. Conventional radiation dosimeter includes 1- and 2-dimensional (D) dosimeters, such as ion chamber, semiconductor dosimeter, thermoluminescent (TLD) dosimeter and film dosimeters. However, these dosimeters have only limited potential to determine the absorption and scattering of radiation dose in 3D dose distribution, and also pose numerous problems in measuring isodose curves and deep dose distributions in regions with high dose gradients [2, 3]. Ion chambers and semiconductor dosimeters provide in sufficient spatial resolution for the planning of many treatments. Additionally, TLD dosimeters, even small in size, are cumbersome and time-consuming in the production of 2D or 3D dose distributions [3].

Polymeric materials are a new class of materials suitable for use in the construction of dosimeters due to their ease of synthesis, low cost, flexibility and adaptability for various applications. Polymer gel dosimeters are radiation sensitive and are made of chemical materials that polymerize as a function of absorbed dose [4, 5]. Unlike traditional radiation dosimeters, these are 3D dosimeters that can record and integrate complex 3D dose distributions and offer volumetric verification capability [6].

Alexander et al. (1954), investigating the effects of ionizing radiation on polymethylmethacrylate, first suggested that polymer systems could be used in radiation dosimetry [7]. Subsequently different formulations of polymer gel dosimeters were investigated [8, 9]. Polymer gel dosimeters needed to be manufactured in an oxygen-free environment (e.g., in a glove box flushed with inert gases such as nitrogen or argon) because they were susceptible to atmospheric oxygen, which inhibits polymerization

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processes [10, 11]. To overcome this limitation, a new type of polymer gel dosimeter, normoxic gel dosimeters, was proposed by Fong et al. (2001) [12]. To date, only two antioxidants (i.e. Ascorbic acid and THPC) have been reported as effective in fabricating normoxic polymer gel dosimeters [12]. Ascorbic acid has been reported to destructively reduce the dose response of gel dosimeters, even in small concentrations [13], and thus, THPC is the most applicable antioxidant for gel dosimetry.

After exposure to irradiation, the physical and chemical properties of polymers change due to induced cross-links, chain cleavage, and formation of new chemical bonds [14]–[20]. Radiation causes structural changes that can affect different properties of polymer gel dosimeters, such as proton nuclear magnetic resonance relaxation times, mass density, opacity, and elasticity [21]. These features can be evaluated by magnetic resonance imaging (MRI) [22], X-ray computed tomography (CT) [23], optical scanning [24], and ultrasonography [25].

The transverse relaxation time (T_2) of water protons obtained from MRI is inversely proportional to the absorbed dose, and is therefore used for dose evaluation [26]. Despite its disadvantages, such as long image acquisition time and temperature sensitivity of the transverse relaxation time (T_2), MRI is generally preferred as the most accurate method for evaluating a gel dosimeter [2]. The cross-linked polymers formed in the gel's irradiated regions increase nuclear magnetic resonance (NMR) relaxation rates of neighboring water protons due to reduced mobility of the surrounding water molecules ($R_1 = 1/T_1$ and $R_2 = 1/T_2$). It is assumed that polymer gels have a linear response within a certain dose range. R_2-dose response (calibration) curves are obtained using homogeneously irradiated test tubes (batch) with prescribed doses (D_i);

$$R_{2i} = \alpha D_i + R_{20} \tag{1.1}$$

where α ($s^1\,Gy^{-1}$) represents the gel response (dose sensitivity) and R_{20} the relaxation rate of the unirradiated gel, R_{2i} is the mean R_2 value in a region of interest (ROI) of uniform dose in the test tube (batch) with index i and D_i is the (mean) dose measured in that region.

HEMA monomer can irritate eyes and skin, but is still much safer than acrylamide [27]. The aim of this study is to prepare a new safer normoxic polymer gel dosimeter containing the less toxic HEMA monomer and THPC antioxidant, and to investigate different factors, such as dose variation, magnetic properties, particle size and molecular structure in order to determine the most suitable gel for use as a 3D dosimeter.

2 Materials and methods

2.1 Preparation of polymer gel dosimeters

The chemicals used in this experiment are gelatin bovine skin, Type B (225 g Bloom, Type B, Hayfene, TR), HEMA (Sigma-Aldrich, U.S.A.), VP (Sigma-Aldrich, U.S.A.), DEGDMA (Sigma-Aldrich, U.S.A.), THPC (Sigma-Aldrich, U.S.A.), and distilled water. Polymer gels containing different percentages of HEMA monomer were prepared in the laboratory under normal atmospheric conditions. The compositions of HEMA-polymer gels with DEGDMA and VP are summarized in table 1. The materials used in the experiment were measured using measuring cups and magnetic stirrer (DLab MS-H280-Pro). The distilled water further heated to 50°C in a magnetic stirrer, gelatin was added and mixed for 20 minutes until a homogeneous mixture was obtained. The temperature was reduced to 35°C and monomers were added and mixed for 5 min. Finally, 2.5 mL THPC was added and mixed

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Table 1. The different chemical compounds of the HEMA-polymer gel with different percentages.

Component	Concentration, % (w/w)								
	A	**B**	**C**	**D**	**E**	**F**	**G**	**H**	**I**
Distilled Water	83	83	83	81	81	81	79	79	79
Gelatin	7	7	7	7	7	7	7	7	7
2-Hydroxyethyl Methacrylate	10	5	5	12	6	6	14	7	7
1-Vinyl-2-Pyrrolidinone	—	5	—	—	6	—	—	7	—
Di(Ethylene glycol) Dimethacrylate	—	—	5	—	—	6	—	—	7

for 5 more minutes. The prepared mixture was poured into polypropylene Eppendorf tubes. Then, the solution was placed in a refrigerator at low temperature, 4°C, to solidify.

2.2 Irradiation of polymer gels

The gel mixtures were kept in the refrigerator for 1 day after production and then irradiated with a medical linear accelerator (TrueBeam STx, Varian, U.S.A.). During irradiation, gel mixtures of different batches were prepared to receive the same dose, and were placed in a line on 10 cm solid phantom and 1 cm bolus. A 1.5 cm thick bolus was placed on the tubes and the collimation area was set to 20×20 cm at SSD = 100 cm. For irradiation, X-rays with 6 Megavolt (MV) energy and 600 MU/min dose rate were used, and irradiation was performed at a gantry angle of 0 degrees. In each batch, 1 tube was left without irradiation as background (BG), the other tubes were irradiated up to 11 Gy at 0.5 Gy intervals.

2.3 Characterization analysis systems

2.3.1 MRI measurements

The irradiated gels were kept in the refrigerator for 24 hours and then scanned with Philips Achieva 1.5 T MRI device. Imaging was performed in a head coil. The images were obtained in two 5 mm slices. T_2 measurements were carried out by a spin echo pulse sequence with multi echoes (16 equidistant echo times). Repetition time (TR) was set at 5000 ms and echo delays (TE) were increased from 20 ms to 320 ms. After T_2 relaxation measurements, it was possible to see the signal difference produced in the gel by changing radiation dose of a different gel samples in each row. T_2 relaxation times were obtained from the acquired T_2 map image (figure 1). R_2 ($= 1/T_2$) relaxation rate was calculated, and R_2-dose response curves were plotted in the OriginLab program. The slope of each relation was taken as proton relaxivity [28].

2.3.2 SEM measurements

The surface morphology of the gels was performed by Quanta 250 S FEG model SEM (Scattering Electron Microscopy) microscope. Irradiated and scanned polymer gels were dried in the oven at 40°C overnight for SEM analysis. Before the analysis, HEMA-gels were covered with gold for 2 min to increase conductivity of membrane surfaces. SEM images were used to calculate the average particle size and size distribution for different gel samples.

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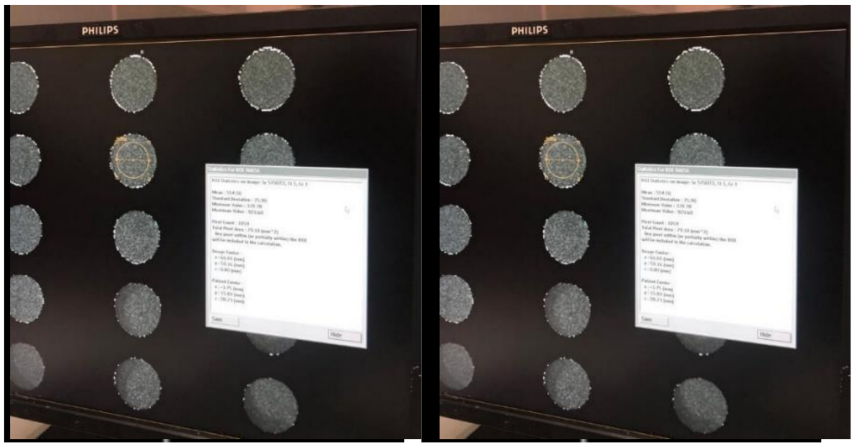

Figure 1. Acquisition of T_2-weighted MR images of the gels.

2.3.3 FTIR measurements

The chemical characterization of HEMA-gels were performed by attenuated total reflection- Fourier transform infrared spectrometer (ATR-FTIR, PerkinElmer Spectrum 100) in the transmittance mode in the wavelength range of 4000–400 cm^{-1}. For this analysis, irradiated and scanned polymer gels were dried in the oven at 40°C overnight.

3 Result and discussion

When gels with HEMA are irradiated, it causes the radical polymerization and crosslinking of its monomers. Typically, for deoxygenated dilute aqueous solutions of vinyl monomers, these reactions are initiated by the water radiolysis products: hydroxyl radicals $OH^{\bullet}$, hydrogen atoms $H^{\bullet}$ and hydrated electrons e^{-}_{aq} [29]. To eliminate the inhibition of polymerization by oxygen, THPC was used to bound free oxygen in gels volume. Large polymeric aggregates in gels change NMR relaxation properties of the water protons in their vicinity, causing difference in the measured T_2 relaxation time values in various parts of the inhomogeneously irradiated gel composition. This phenomenon allowed us to derive a basic relation between the relaxation rate ($R_2 = 1/T_2$) and the absorbed dose for gels composition.

3.1 Dose response of polymer gels

The gel mixtures produced were irradiated up to 11 Gy, but after 4 Gy the linearity began to deteriorate. Therefore, evaluation was made by plotting R_2-dose response curves in the range of 0–4 Gy (figure 2).

Firstly, gel mixtures containing 10, 12 and 14% HEMA monomer were tested to determine the optimum concentration. Our results show a good correlation between the relaxation rates measured in the samples prepared at room temperature (22–24 C°) and the increasing dose values. As seen from table 2, the best dose response was observed at 12% HEMA (r^2: 0.922), however, this was found insufficient in terms of linearity, and to remedy this, it was decided to add two different cross-linkers (DEGDMA and VP) to HEMA in equal proportions. It is seen that α, dose sensitivity ($Gy^{-1}\ s^{-1}$) and r^2 values of polymer gel samples with DEGDMA have higher values than polymer gels containing only HEMA and formed with VP as a cross-linker after irradiation in the range of 0–4 Gy.

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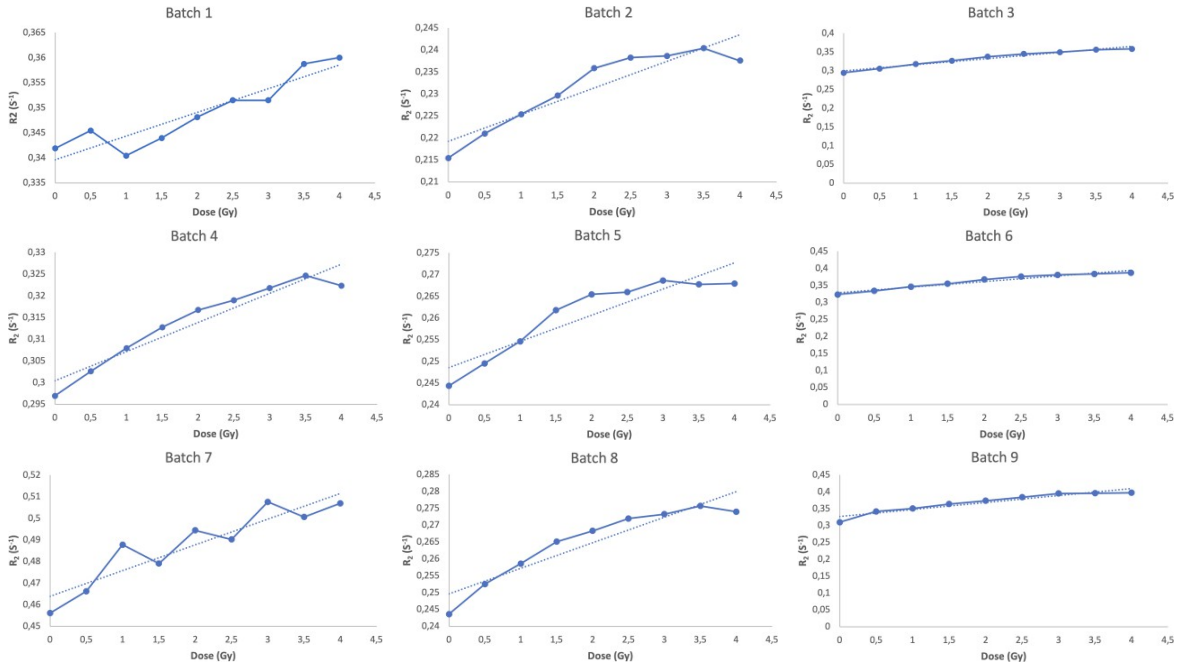

Figure 2. R_2-dose response curves of gels containing 10%, 12%, and 14% monomers. (a) 10 HEMA, (b) 5 HEMA+5 VP, (c) 5 HEMA+5 DEGDMA, (d) 12 HEMA, (e) 6 HEMA+6 VP, (f) 6 HEMA+6 DEGDMA, (g) 14 HEMA, (h) 7 HEMA+7VP, (i) 7 HEMA+7 DEGDMA.

Table 2. Dose sensitivity, offset and r^2 values of gels in different combinations. α = dose sensitivity (slope); R_{20} = offset (intercept); r^2 = regression; α/R_{20} = slope-to-intercept ratio.

Batch No.	**Monomer Percentage (%)**	**α [$Gy^{-1}\,s^{-1}$]**	**R_{20} [s^{-1}]**	**α/R_{20}**	**r^2**
1	10 HEMA	0.005 ±0.001	0.340 ±0.002	0.015	0.860
2	5 HEMA+5 VP	0.006 ±0.001	0.219 ±0.002	0.027	0.860
3	5 HEMA+5 DEGDMA	0.016 ±0.001	0.300 ±0.002	0.053	0.968
4	12 HEMA	0.007 ±0.000	0.300 ±0.001	0.023	0.922
5	6 HEMA+6 VP	0.006 ±0.001	0.249 ±0.002	0.024	0.848
6	6 HEMA+6 DEGDMA	0.017 ±0.000	0.328 ±0.003	0.052	0.958
7	14 HEMA	0.012 ±0.001	0.464 ±0.005	0.026	0.845
8	7 HEMA+7 VP	0.008 ±0.000	0.250 ±0.002	0.032	0.882
9	7 HEMA+7 DEGDMA	0.021 ±0.001	0.326 ±0.003	0.064	0.913

Also, R_{20} offset relaxation rate (s^{-1}) values of polymer gels with HEMA and HEMA-DEGDEMA remained almost the same.

The r^2 values demonstrate that the gels that gave the best dose response contained 5% HEMA-5% DEGDMA and 6% HEMA-6% DEGDMA (r^2: 0.968 and 0.958). Similarly, Maryanski et al. reported that the dose sensitivity of BANG polymer dosimeter significantly depends on the composition ratio between monomer and crosslinker [30]. Rabaeh et al. also reported that dose sensitivity increased in correspondence to the total co-monomers concentration [31]. The highest dose sensitivity was obtained as 0.021 $Gy^{-1}\,s^{-1}$ for 7% HEMA-7% DEGDMA gels. Baldock et al. also calculated the dose

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sensitivity of PAG gels as 0.0285 Gy^{-1} s^{-1} [10]. Additionally, Lepage et al. found the dose sensitivity of the gel dosimeter with 3% HEMA to be 0.046 Gy^{-1} s^{-1} [32].

Fong et al. showed that an important determinant of the sensitivity in detection of small dose changes is the slope-intercept ratio of the dose-response curve. These ranged from 0.08 to 0.17, which is comparable to previously described hypoxic gels [12]. Therefore, simply increasing slope is not sufficient to ensure appropriate dose resolution, the slope-to-intercept relation must also be maximized, which means minimizing the intercept value. The slope-to-intercept ratio values given in table 2 show that the composition with the highest value is 7% HEMA-7% DEGDMA (0.064). Similarly, Kozicki et al., found that the slope-to-intercept ratio value of VIPAR polymer gel dosimeters containing N-vinylpyrrolidone monomer was 0.026 [33].

3.2 FTIR analysis of gels

The FTIR spectra of gels containing 12% HEMA, 6% HEMA-6% VP and 6% HEMA-6% DEGDMA are shown in figure 3. When the curve is examined, FTIR spectra showed characteristic bands at 3219 cm^{-1} attributed to -OH stretching; about 2950 cm^{-1} related to C−H bond stretching of alkyl group; 1721 cm^{-1} relating to C=O bond stretch of the ester carboxyl groups; and 1642 cm^{-1} relating to C=C bond stretching vibrations [33]–[38].

An examination of the FTIR spectrum of 12% HEMA shows that the observation of the band at 1642 cm^{-1} indicates the presence of unreacted HEMA monomer. However, the peak intensity of the C=C groups decreased when DEGDMA crosslinker was added to the HEMA monomer. This change in peak intensity may indicate increased polymerization in the mixture of HEMA and DEGDEMA, and conversion of vinyl groups of HEMA and DEGDMA in cross-linked samples. The FTIR spectrum of HEMA-VP reveals an increase in intensity at about 1640 cm^{-1}. The C=O band of the VP monomer overlaps with the C=C band of HEMA, suggesting an increase in the intensity of the 1642 cm^{-1} band [39]–[41].

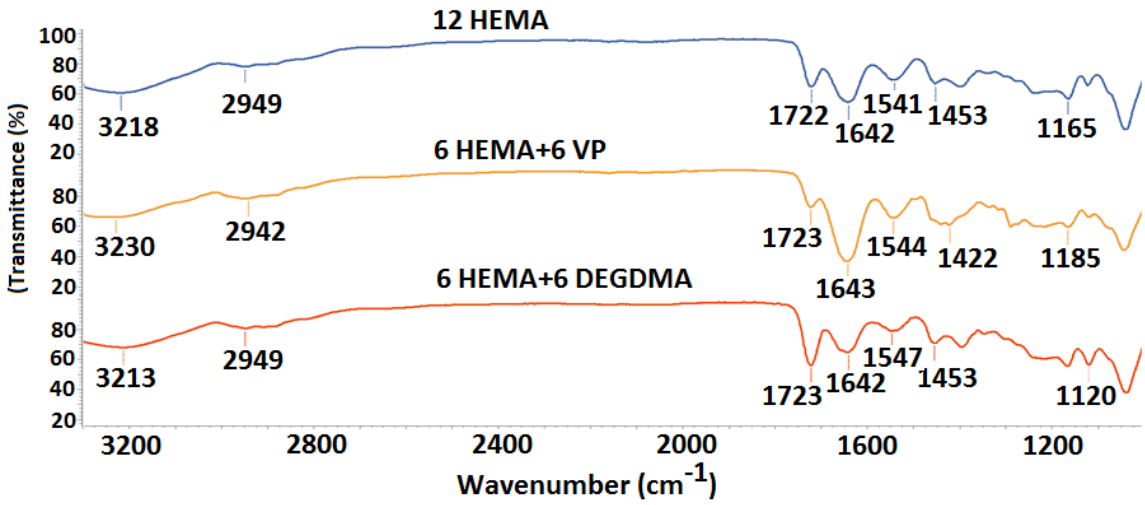

Figure 3. FTIR spectra of samples.

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3.3 SEM analysis of gels

SEM analyzes could not be performed because, during drying, HEMA-VP gels melted to a soft consistency. Figure 4 shows SEM micrographs at different magnifications of 12% HEMA gels that were not irradiated (background) and dosed at 4 Gy. An examination of the SEM images of all samples reveals no significant difference between the particle sizes of the gels receiving 0 (background) and 4 Gy doses. The spaces between the particles and the particle configurations show differences, i.e., distribution was not homogeneous.

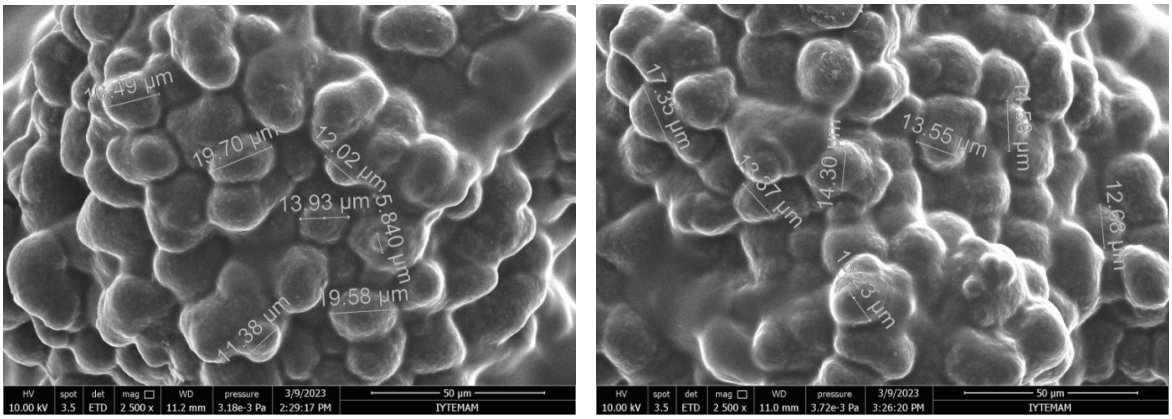

Figure 4. SEM images (2500x; scaled of 50 µm) of 12% HEMA gels with background (a) and irradiated with a dose of 4 Gy (b).

Figure 5 shows SEM images of gels irradiated with a dose of 4 Gy, containing 10% HEMA, 5% HEMA-5% DEGDMA, 12% HEMA and 6% HEMA-6% DEGDMA. The SEM images of the mixtures show that the particle sizes in the gels containing 10% and 12% HEMA are in the order of µm, and for those containing 5% HEMA-5% DEGDMA and 6% HEMA-6% DEGDMA, in the order of nm, that is, the average particle size decreases when the crosslinker is added. The relaxation rate increased with decreased particle size.

Dipolar interaction between the superparamagnetic cores and surrounding solvent protons result in increased longitudinal and transverse relaxation rates, especially for small size domain particles [42]. The comparison of r^2 values given in table 2 with the SEM images show that the addition of DEGDMA crosslinker decreases the particle sizes, and accordingly, increases the relaxation rate.

4 Conclusion

The normoxic HEMA gel dosimeters prepared were found to have potential as radiation dosimeters. The dose response can be varied by altering the precise composition of the gels. The sensitivity and stability of gel dosimetry increased when an equal amount of DEGDMA cross-linker was added to the HEMA monomer. Further research is necessary to understand other variables' effects on the gel response, such as dose rate, radiation quality, and composition, and in the light of these effects, reconsider the R_2-dose relationship. However, these preliminary results highlight the possibility of developing polymer gels that respond well in normal atmosphere, and that are suitable for use in radiation dosimetry.

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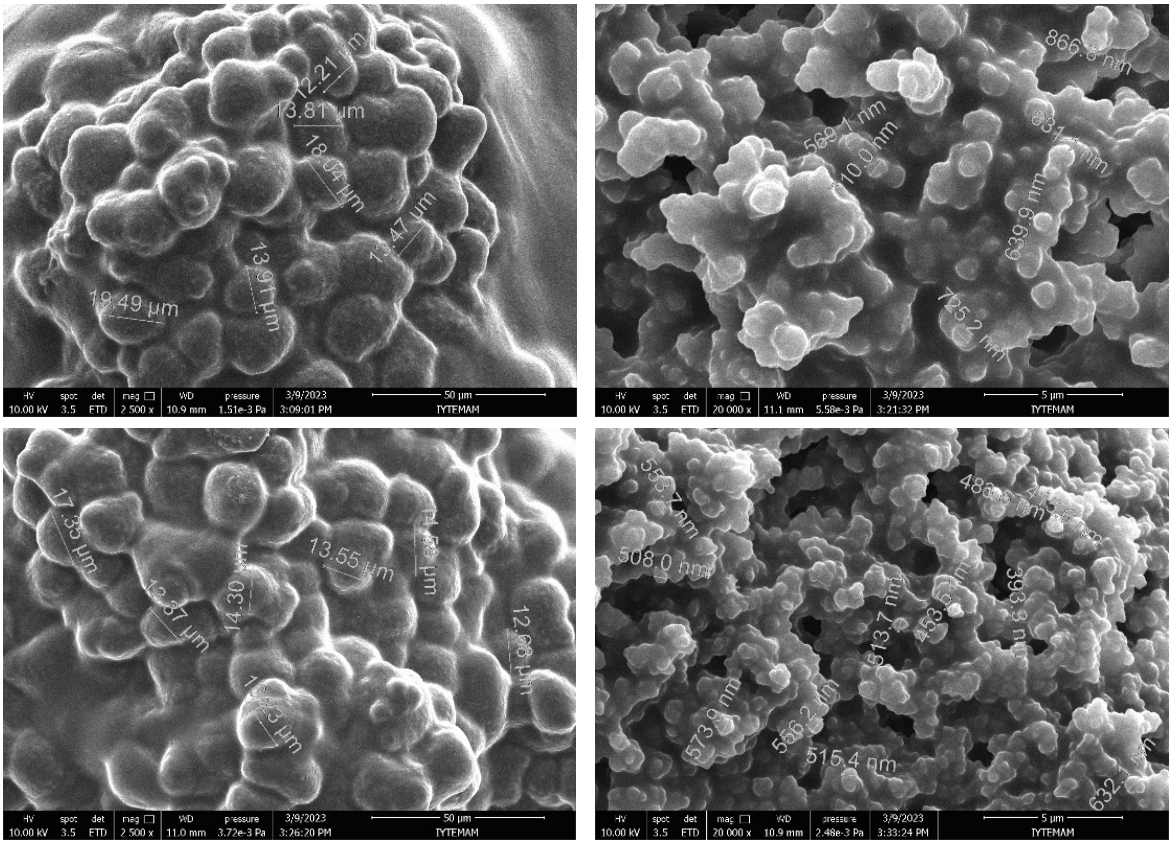

Figure 5. SEM micrographs of 10% HEMA (a), 5% HEMA-5% DEGDMA (b), 12% HEMA(c), 6% HEMA-6% DEGDMA gels (d) dosed with 4 Gy.

Acknowledgments

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