

The Effects of Crosslinker and Monomer to the Polymerization of Polymethacrylamide Gel Dosimeters by Direct and Indirect Methods

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Abstract: Radiation-induced polymerization in Polymethacrylamide Gels (PMAAGs) potentially used for 3D dose verification in radiotherapy has been studied using both Raman spectroscopy and Nuclear Magnetic Resonance (NMR) method. The dosimeters are composed of aqueous Methacrylamide (MAA) monomer and N, N' methylene-bis-acrylamide (BIS) crosslinker at various concentrations from 2 to 6% (w/w) and gelatin at 6% (w/w). The dosimeters were irradiated to doses up to 30 Gy using ⁶⁰Co teletherapy γ -ray source at a constant dose rate. The formation of the polymer increases with increasing dose and was followed directly by Raman spectroscopic measurement for CH₃ stretching mode assigned to polymethacrylamide and indirectly by the transverse spin-echo pulse NMR R₂ relaxation rate of water protons within the polymer gel network. The half dose $D_{1/2}$ values of both direct and indirect methods were used to evaluate the effects of initial concentrations of monomer and crosslinker to the dose required to produce 50% of the polymer in PMAAGs. The PMAAGs containing more crosslinker than monomer show larger $D_{1/2}$ values, indicating that the crosslinker has a larger effect on the increase in dose required to produce 50% of the polymer. The $D_{1/2}$ value of the direct method is consistently higher than that of the indirect method, indicating that the indirect method is more sensitive to the dose response, but fundamentally does not measure the amount of polymethacrylamide formed. There is a correlation between $D_{1/2}$ value and concentrations of monomer and crosslinker. The correlation factor, k_C of the crosslinker is always greater than k_M of monomer, for both the direct and indirect methods, suggesting the crosslinker reacts more efficiently than monomer to produce 50% of the polymer of polymethacrylamide.

Key words: Crosslinker, monomer, polymerization, direct method, polymethacrylamide gels

INTRODUCTION

Polymer gel dosimeter used in conjunction with magnetic resonance imaging (MRI) is the most popular dosimeter imaging modality as a potential tool for mapping complex dose distributions^[1-5]. The original polymer gel is based on the high molecular weight compounds consisting of acrylamide (AA, monomer) and N, N' methylene-bis-acrylamide (BIS, crosslinker)^[3] dissolved in a gelatin/agarose hydrogel. Upon irradiation, water molecules dissociate into OH and H radicals that break the double C=C bonds of monomers (AA and BIS). The resulting monomer radicals, in turn, interact with other monomers and produce a chain reaction to form 3D polymer aggregates that are spatially retained in a gelatin matrix. The amount of polymer formed is related to absorbed dose received by the polymer gel. These polymer aggregates are usually evaluated indirectly using nuclear magnetic resonance (NMR) technique, the

principle of MRI, which measures the proton relaxation times of the surrounding water molecules. The proton relaxation rate increases with dose and the dose distribution of polymer gels may be constructed from the relaxation rate images obtained from MRI scans.

The primary objective of a study of polymer gel dosimeters is to manufacture more efficient and stable 3D dosimeters that have the highest R₂-dose sensitivity which gives the lowest dose resolution so that two doses of slightly different values can be mapped and visualized correctly with the lowest uncertainty^[1,6]. Recently, there has been an interest in the study of the basic physical and chemical properties of polymer gel dosimetry, which could provide invaluable information on various factors affecting the overall dosimeter performance^[1,2,7-12]. The sensitivity of a polymer gel dosimeter is dependent to some extent, on physical parameters such as radiant energy, temperature during MRI evaluation, the time between irradiation and NMR evaluation and magnetic strength^[7]. Murphy *et al.*^[2] has

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observed the effect of pH during synthesis on the dose response of a modifier polymer gel dosimeter. It is well known fact that dose response of gel dosimeters is dependent on the temperature during MRI measurement^[13,14]. The temperature rise during polymerization may have considerable influence on the polymerization processes. Only recently, there has been reported the significant change in local temperature during irradiation of polymer gel dosimeters^[15].

Two gelling agents have so far been used in the manufacture of polymer gels i.e. agarose^[3] and gelatin^[4]. The emphasis in the current literature has been on the dose resolution optimization of polymer gel dosimeters using different monomers^[4,5,8,12,16,17]. The use of acrylamide monomer is common in the polymer gel dosimeter studies. However, details of the use of methacrylamide as monomer in polymer gel dosimeter have not been reported. The choice of this monomer, which has $-CH_3$ group in its structure instead of $-H$ in acrylamide structure, leads to an increase in molecular mass of the polymer gel dosimeters. It would be very interesting to understand the effect of higher molecular mass polymer gel to the polymerization process and to the proton relaxation rate in the polymethacrylamide gel dosimeters, potentially used in MRI 3D dose verification for radiotherapy treatment planning.

The slope or R_2 -dose sensitivity at low doses derived from a plot of R_2 versus dose generally accepts as a parameter able to quantify and compare the performance of different polymer gel dosimeters. The assumption of linearity at low doses is commonly applied, although a divergence from linearity has been observed^[18,19]. The polymer gel performance as a dosimeter depends on the type and the concentration of comonomers in different formulations of gelatin-based or agarose-based^[6]. It has also been shown that polymer gels with different concentrations of gelatin produced different dose sensitivity^[1,6,8].

There has been a study using FT-Raman spectroscopy and NMR T_2 that the dose response of polymer gel dosimeters increases in monoexponential fashion as a function of the monomer concentration and the gelatin concentration^[9]. Polymer formation and monomer consumptions have been observed in the Raman spectra^[10-12]. The results show that the crosslinker is consumed at a greater rate than the monomer consumption in polyacrylamide gel dosimeters. The formation of polymers has been directly correlated with the consumption of monomer^[10]. These studies are, by no means complete and more works are needed, such as to understand the fundamental relations between initial concentrations of monomer and crosslinker on the formation of polymer induced by ionizing radiation.

In this communication the correlation factors km and KC were obtained between $D_{1/2}$ value and the initial concentrations of monomer and crosslinker to the dose

required to produce 50% of the polymer in polymethacrylamide gel (PMAAG) dosimeters.

MATERIALS AND METHODS

Synthesis of PMAAG dosimeters: The polymethacrylamide gel dosimeters were synthesized in a nitrogen glove-box according to Deene *et al.*^[19]. The initial concentrations of the dosimeters were varied from 2 to 6% (w/w) for both methacrylamide (MAA) monomer and *N, N'*-methylene-bis-acrylamide (BIS) crosslinker, 6% (w/w) gelatine and completed with deionised water. Both monomers (MAA and BIS) were obtained from the SIGMA chemical Co (St. Louis, Mo, USA) and aware of electrophoresis grade (99%). The comonomers and gelatine were dissolved separately in two reaction flasks with equal amount of the total water volume. In the first reaction flask, the comonomers in half of the amount of deionised water were heated to a constant temperature at 55°C for 2 h. In the second reaction flask, the gelatine and another half of the amount of deionised water were also heated to a constant temperature at 55°C for 2 h to dissolve the gelatin.

Subsequently, both solutions were allowed to cool down to 30°C for about 1 h to avoid spontaneous heat-induced polymerization before mixing. A peristaltic pump was used to mix the comonomers with the gelatin via Tygothane flexible tubing and stirred at 1000 RPM to form a polymethacrylamide gel (PMAAG). The gel PMAAG was pumped into screw-top "P6" glass vials using the second peristaltic pump. The manufacture and collection of the gel dosimeters were conducted in an oxygen free environment inside a glove box, which was flushed with nitrogen at the flow rate of 60 ml min⁻¹ in order to expel oxygen that inhibits polymerization prior to gamma irradiation. The oxygen concentration was determined at less than 0.1 mg L⁻¹ throughout. The final gel dosimeters were sealed and kept in a refrigerator overnight at 20°C before irradiation.

Irradiation: All irradiations were performed using an Eldorado 6&8 Co-60 teletherapy gamma unit (Atomic Energy of Canada Limited) with the maximum dose rate at 0.58 Gy/min calibrated using a Fricke dosimeter. Each vial filled with PMAAG was placed in a polystyrene holder in a water-phantom acrylic tank. The sample was irradiated at 15 cm depth, 60 cm surface to source distance (SSD) set-up and 60 x 60 cm² field size. Five vials of PMAAG were irradiated with the same dose between 1 and 30 Gy. The phantom temperature during irradiation was constant at 22°C. The samples were transferred back to the refrigerator and stored for about 18-24 h before Raman spectra and NMR measurements. It was estimated that to complete polymerization required at least 12 h post-irradiation for polyacrylamide gels^[2].

Determination of inelastic scattering Raman shift:

Raman spectra of PMAAG were acquired on a 25mW Raman spectrometer (RSI 2001 B, Raman system, INC) equipped with a solid-state Nd:YAG green laser emitting at 532 nm and a thermoelectrically cooled CCD array of 2048 elements (125 μm x 200 μm per element). This spectrometer was chosen for its high signal to noise. The Raman signal intensity is inversely proportional to λ⁴, where, λ is the wavelength of the visible laser. Low power laser is preferred in this study to avoid excessive sample heating. The ambient temperature during the measurements was 25°C. The Laser excitation and signal collection was performed using a probe head inserted inside the sample compartment as shown in Fig. 1. The fundamental limitation of using a visible laser to perform Raman spectroscopy is the interference from fluorescence. Grams/32, version 6 software was used to analyze the spectra and perform corrections for baseline, smoothing and Fourier transform on the dispersive spectra. The baseline correction utilized the multiple point level method in which the baseline is levelled at a value that is the average of the baseline points. A constant correction factor of the degree of smoothing parameter was used throughout the data collection. A constant correction factor of 80% of the degree of smoothing parameter was used throughout the data collection. The Fourier smoothing was accomplished by the peak data, applying a triangular filter function at the specified cutoff point of 40% and then reverse Fourier transforming the data.

Determination of T₂ relaxation time: A Carr Purcell Meiboom Gill (CPMG) sequence or spin-echo pulse method (90°-τ-180°) was used to measure the proton spin-spin relaxation time, T₂ in the polymer matrix. T₂ values were determined using an NMR instrument PC 120 IBM (Bruker, Germany) at low frequency of 20 MHz and the magnetic strength of 0.47 T. This spectrometer was specifically designed for proton relaxation measurement. The measurement was conducted at ambient temperature of 25°C.

RESULTS AND DISCUSSION

The half-dose D_{1/2} of direct method: The amount of polymer formation in PMAAG is proportional to the Raman intensity. The dose response of the polymer gel dosimeters is therefore represented by the relative Raman intensity as a function of dose^[10]. Figure 2 shows the relative Raman intensity that corresponds to the peak area of Raman shift of CH₃ bending mode of polymethacrylamide (2880 cm⁻¹) at different doses. Polymer formation is monoexponential in the dose range between 0 and 30 Gy. The formation of polymers may be represented as the change of the Raman intensity Δy = y - y₀ as the fit equation dose and fit equation (1):

$$\Delta y = y - y_0 = A(1 - e^{-D/D_0}) \tag{1}$$

where, D₀ is the dose sensitivity parameter, y₀ is the Raman intensity at zero dose, y is the Raman intensity of dose D and A is a constant. Figure 3 illustrates the relative Raman intensity as a function of dose for MAA varied from 2 to 6% and at 2 and 6% BIS. At low doses, the formation of the polymer increases with the increase of the concentration of MAA and is higher for higher BIS concentration. However, very little additional polymer is being formed at higher doses, in which the amount of polymer has insignificantly changed with dose. Figure 4 illustrates the relative Raman intensity as a function of dose for BIS varied from 2 to 6% and at 2 and 6% MAA.

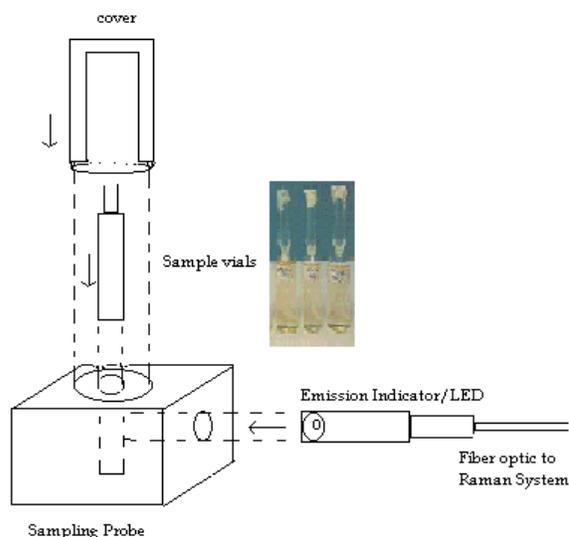


Fig. 1: Schematic diagram of the Raman probe

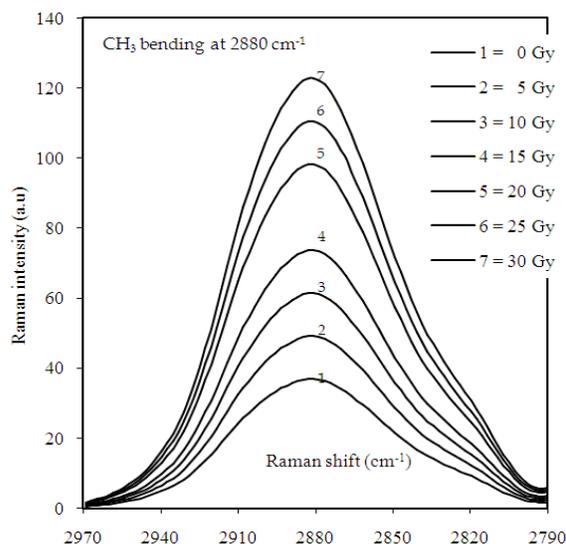
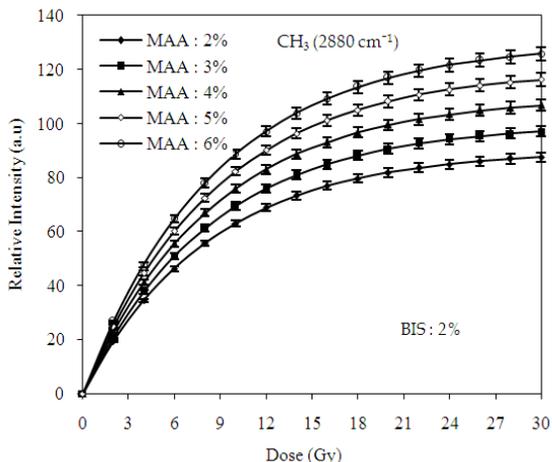
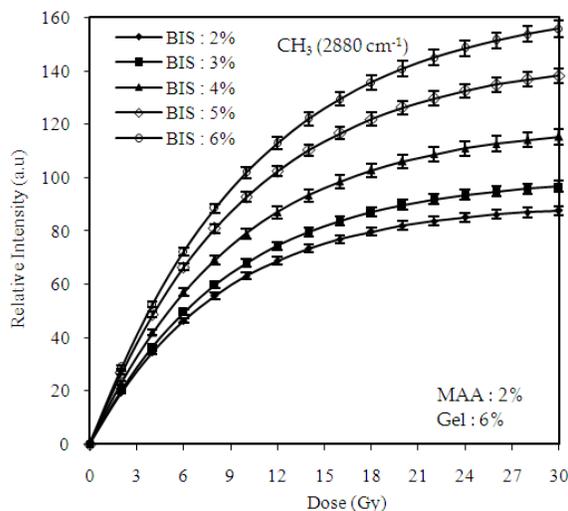


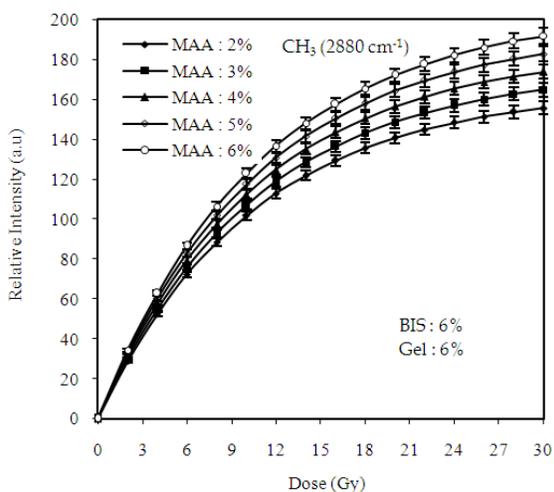
Fig. 2: The area under the peaks at Raman shift of 2880 cm⁻¹ CH₃ bending band used to analyze the polymer formation as a function of dose



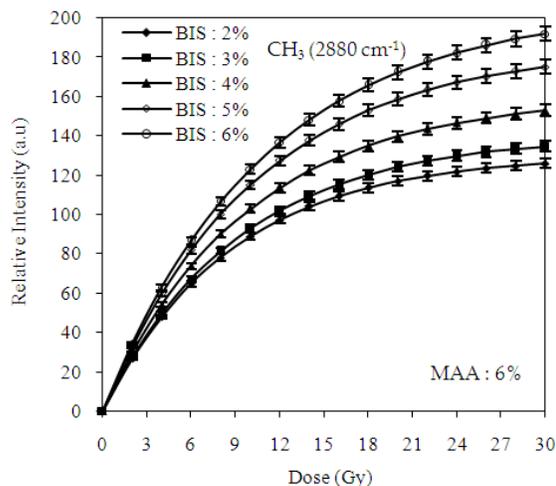
(a)



(a)



(b)



(b)

Fig. 3: Change in the relative Raman intensity Δy as a function of dose D at various monomer concentrations for (a) 2% crosslinker and (b) 6% crosslinker

Fig. 4: Change in the relative Raman intensity Δy as a function of dose D at various crosslinker concentrations for (a) 2% monomer and (b) 6% monomer

Here, the polymer formation increases with the increase of BIS concentration and is higher for higher MAA concentration. More polymers are being formed at increasing dose and it is higher for higher BIS concentration. This suggests that BIS were consumed more in the formation of polymethacrylamide. At higher doses, very little additional polymer is being formed and the amount of polymer is insignificantly increased. The amount of polymer becomes constant. The results are qualitatively consistent with the previous studies but for polyacrylamide gels that BIS is consumed at a greater rate than acrylamide^[10,12].

The latter describes the absorbed dose at which the polymerization has reached 50% in value. The half dose $D_{1/2}$ values are expected to increase with the increase of gelatin concentration as reported elsewhere^[6,10]. Figure 5 illustrates the correlation between $D_{1/2}$ and concentrations of monomer and crosslinker. It is noted that $D_{1/2}$ increases slowly with MAA concentration as shown by the less steeper slopes of $D_{1/2}$ vs. MAA concentration relationship in Fig. 5(a). At 2% BIS, $D_{1/2}$ value increases from 5.76 Gy for 2% MAA to 6.05 Gy for 6% MAA. At 6% BIS, $D_{1/2}$ value increases from 7.23 Gy for 2% MAA to 7.58 Gy for 6% MAA. This suggests that $D_{1/2}$ value increases less strongly with MAA concentration.

The reciprocal of the slope of linear plot $\ln\left(1 - \frac{\Delta y}{A}\right)$ versus dose D was used to determine the dose sensitivity parameter D_0 and the half dose $D_{1/2} = D_0 \ln 2$.

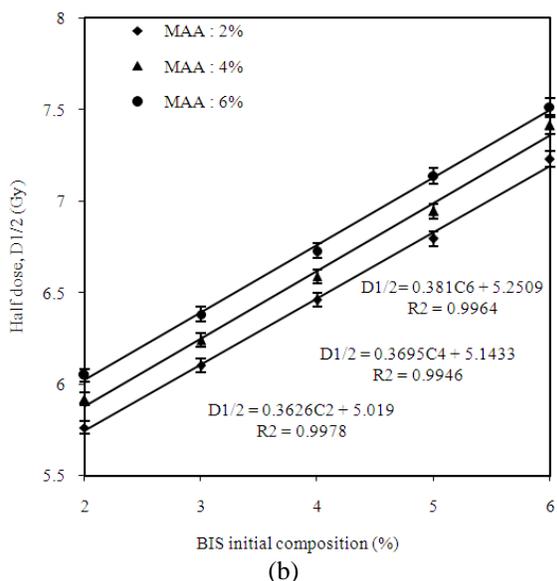
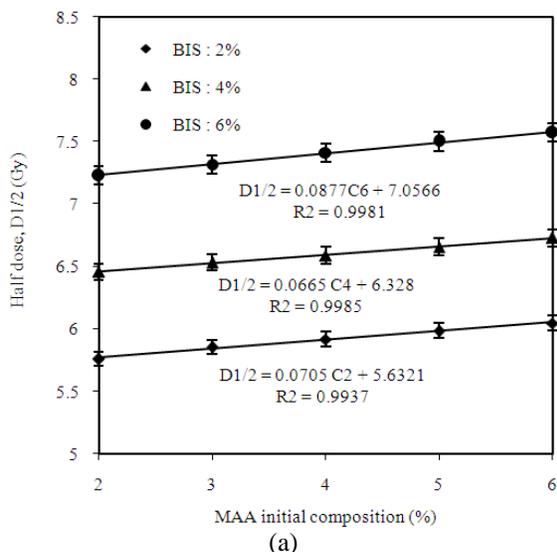


Fig. 5: Correlation between $D_{1/2}$ (direct) and (a) initial concentration of monomer at 2, 4, 6% crosslinker, represented by parameter k_M (b) initial concentration of crosslinker at 2, 4, 6% monomer, represented by parameter k_C

Figure 5 (b) illustrates the correlations between $D_{1/2}$ and BIS concentration from 2 to 6 % for MAA concentrations at 2, 4 and 6%. It is noted that $D_{1/2}$ increases strongly with the BIS concentration as shown by the steeper slopes of $D_{1/2}$ vs. BIS concentration relationship. At 2% MAA, $D_{1/2}$ value increases from 5.76 to 7.23 Gy for BIS from 2 to 6%. At 6% MAA, $D_{1/2}$ value increases from 6.05 in 7.58 Gy for MAA from 2 to 6%. These results are consistent with the previous study for polyacrylamide gel, where $D_{1/2}$ value increases in an approximately linear fashion as a function of the acrylamide concentration and is higher for higher BIS concentrations^[10].

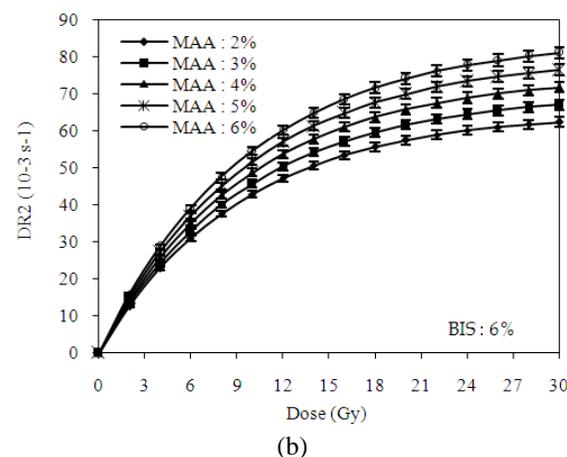
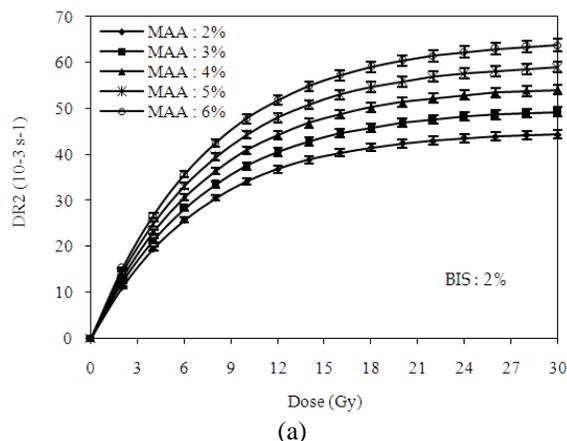


Fig. 6: Change in the transverse relaxation rate ΔR_2 as a function of dose D at various monomer concentrations for (a) 2% crosslinker and (b) 6% crosslinker

The effects of monomer and crosslinker in to produce 50% of the polymer may be represented by the correlation factors, k_M and k_C defined as the gradient of $D_{1/2}$ vs. MAA initial concentration and $D_{1/2}$ vs. BIS initial concentration respectively. Note that k_C is larger than k_M , which suggests that the concentration of BIS has a larger effect to produce 50% of the polymer. The reason is that BIS forms clusters upon polymerization of polymethacrylamide. This process terminates efficiently so that a higher dose is required in order to obtain 50% of the total amount of polymer when more BIS is present in the gels. Thus, a higher concentration of BIS is more efficient to produce 50% of the polymer than a higher concentration of MAA.

The half-dose $D_{1/2}$ of indirect method: The relationships between proton relaxation rate ΔR_2 and dose for all concentrations used were fitted to the monoexponential equation (1). The proton relaxation characteristics of $D_{1/2}$ vs. comonomer concentrations are of similar fashion to those obtained from the Raman scattering method.

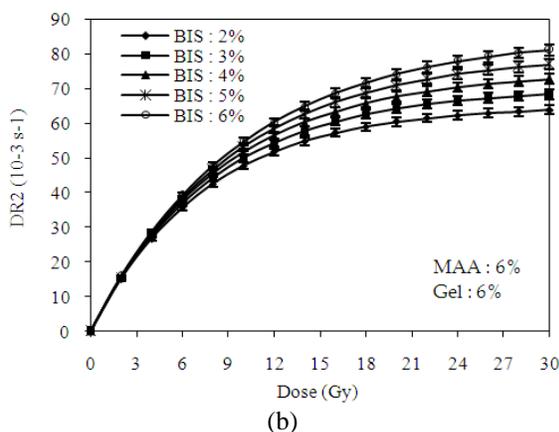
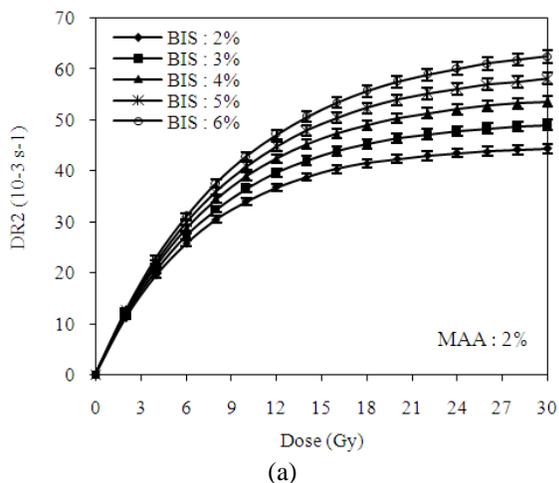
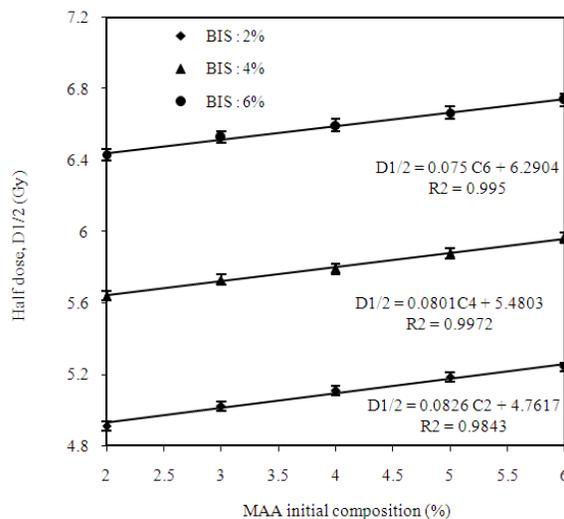
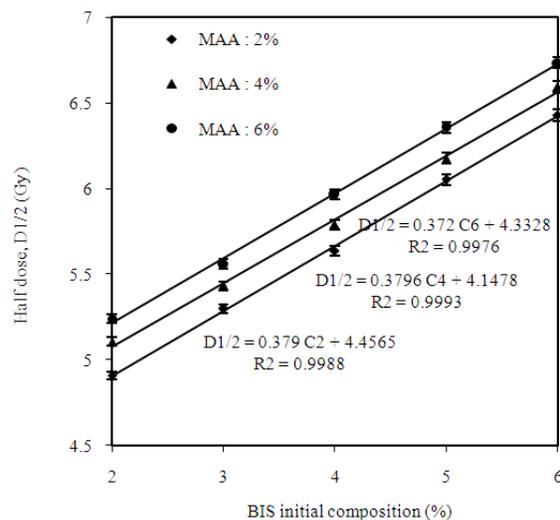


Fig. 7: Change in the transverse relaxation rate ΔR_2 as a function of dose D at various crosslinker concentrations for (a) 2% monomer and (b) 6% monomer

Figure 6 (a) and 6 (b) illustrate the change of transverse relaxation rate (ΔR_2) as a function of dose for MAA concentrations from 2 to 6% at 2 and 6% BIS respectively. The change of relaxation rate increases with the increase of MAA concentration and is higher for a higher BIS concentration. Figure 7 (a) and (b) illustrate the change of relaxation rate (ΔR_2) as a function of dose for different BIS concentrations from 2 to 6% at 2 and 6% MAA respectively. The relaxation rate of protons increases with the increase of BIS concentration. The relaxation rate is assumed to correspond to the formation of polymer. The formation of the polymer increases with the increase of the concentration of MAA and is higher for a higher BIS concentration. This indicates that the commoners are consumed more efficiently at the lower concentrations. However, at higher doses very little additional polymer is being formed. The similar trend was also observed in the direct method. The values of $D_{1/2}$ (direct) are higher than $D_{1/2}$ (indirect).



(a)



(b)

Fig. 8: Correlation between $D_{1/2}$ (indirect) and (a) initial concentration of monomer at 2, 4, 6% crosslinker, represented by parameter k_M (b) initial concentration of crosslinker at 2, 4, 6% monomer, represented by parameter k_C

The indirect method does not measure the amount of polymer formed. However, using an appropriate model, spectroscopic and NMR measurement is reconciled in representing polymerization of the polymer gels.

Figure 8 (a) shows the correlation between $D_{1/2}$ and MAA concentration at different BIS concentrations. It is noted that $D_{1/2}$ increases slowly with MAA concentration as shown by the correlation factor k_M , the slope of $D_{1/2}$ vs. MAA concentration relationship. At 2% BIS, $D_{1/2}$ value increases from 4.91 Gy for 2% MAA to 5.24 Gy for 6% MAA. At 6% BIS, $D_{1/2}$ value increases from 6.33 Gy for 2% MAA to 6.74 Gy for 6% MAA.

Table 1: Correlation factor k_M and k_C values at different BIS and MAA concentrations

BIS	k_M (direct)	k_M (indirect)	ratio	MAA	k_C (direct)	k_C (indirect)	ratio
2%	0.0625	0.0359	1.741	2%	0.3626	0.3506	1.034
3%	0.0697	0.0357	1.952	3%	0.3628	0.3568	1.017
4%	0.0665	0.0358	1.858	4%	0.3645	0.3542	1.029
5%	0.0860	0.0377	2.281	5%	0.3669	0.3556	1.032
6%	0.0877	0.0379	2.314	6%	0.3690	0.3590	1.028

This suggests that $D_{1/2}$ value increases strongly with BIS concentration. Figure 8 (b) illustrates the correlations between $D_{1/2}$ and BIS concentration from 2 to 6 % for MAA concentrations at 2, 4 and 6%. At 2% MAA, $D_{1/2}$ value increases from 4.91 Gy for 2% BIS to 6.33 Gy for 6% BIS. At 6% MAA, $D_{1/2}$ value increases from 5.24 Gy for 2% BIS to 6.74 Gy for 6% BIS. It is noted that $D_{1/2}$ increases strongly with the BIS concentration as shown by the increase of the correlation factor k_C , the slope of $D_{1/2}$ vs. BIS concentration relationship. Note that k_C is larger than k_M because BIS has a larger effect on the increase in dose to produce 50% of the polymer due to the formation of clusters upon polymerization.

Comparison of k_M and k_C values between direct and indirect method:

The k_M and k_C values of the direct and indirect method are shown in Table 1. Generally, the correlation factors k_M and k_C values are higher for higher MAA and BIS concentration. It also shows that k_M (direct) is greater than k_M (indirect) by a factor from 1.7 to 2.3 times for BIS concentrations from 2 to 6%. k_C values are almost constant for all MAA concentrations. This suggests that the correlation factor is strongly influenced by the concentration of crosslinker. The k_C (indirect) values are greater than k_C (direct) values indicating the NMR method is more radiosensitive than the Raman method, but does not measure the actual formation of polymer in PMAAG dosimeters. Also shown is k_C (direct) is slightly higher than k_C (indirect) by about 3% for each MAA concentration. However, using an appropriate model, spectroscopic and NMR measurements are reconciled in representing polymerization of the polymethacrylamide gel dosimeter. Thus, for the PMAAG system to be used as polymer gel dosimeter, other than radiate energy, the strength of the magnetic field, transportation time from the irradiation of the measurement and gelatin concentration^[7,9], the concentration of crosslinker is the primary important for optimizing the formation of polymer in PMAAG dosimeters.

CONCLUSION

This study has shown the fundamental characteristics of polymethacrylamide gels irradiated with γ -rays up to 30 kGy. The PMAAG composed of MAA monomer and BIS crosslinker at varying concentrations from 2 to 6% and at 6% gelatin. The polymer gels exhibit increasing polymerization with increasing dose from which the change of Raman

intensity or proton relaxation $\Delta y = A(1 - e^{-D/D_0})$ was conveniently used to determine the half dose $D_{1/2}$ at different concentrations of comonomers. The PMAAGs containing more BIS than MAA show larger $D_{1/2}$ values, indicating that the BIS produced a larger effect on the increase in dose required to produce 50% of the polymer. There is a correlation between $D_{1/2}$ values and concentrations of BIS and MAA. This has been shown by the correlation factors k_C and k_M , where k_C is greater than k_M , indicating BIS reacts more efficiently than MAA to produce 50% of the polymer formation. The k_C (indirect) value is greater than k_C (direct) value suggesting the NMR method is more radiosensitive than the Raman method, but does not measure the actual formation of polymer in PMAAG dosimeters. However, using an appropriate model such as the ratio of correlation factor between direct and indirect methods, both spectroscopic and NMR measurements are reconciled in representing polymerization of the polymethacrylamide gel dosimeter.

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