



**UNIVERSITI PUTRA MALAYSIA**

**RADIATION-INDUCED POLYMERIZATION OF ACRYLAMIDE AND  
METHACRYLAMIDE-BASED POLYMER GEL DOSIMETERS**

**ARIS DOYAN**

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METHACRYLAMIDE-BASED POLYMER GEL DOSIMETERS**

**By**

**ARIS DOYAN**

**Thesis Submitted to the School of Graduate Studies, Universiti Putra  
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the Degree of Doctor of Philosophy**

**March 2005**



*In the Name of Allah, the Beneficent, the Merciful*

*Read in the name of your Lord who created, created man from clots of blood.*

*Read ! your Lord is the most Bounteous who has taught the use of the pen,  
has taught man what he did not know.*

**Al-Qur'an, 96:1-5**



## DEDICATION

This dissertation dedicates to my late father Happu Lintong, my mother Martha Nurgayah, my parent in law; H. Hambali and Hj. Ahilmi, my wife Susilawati PhD, my sons Muhammad Ikhsan, Ikhlasul Amal, my late daughter Kurnia Ramadhani, my brother, my sister and to my all family.



Abstract of the thesis presented to the Senate of Universiti Putra Malaysia in fulfillment of requirement for the degree of Doctor of Philosophy

**RADIATION-INDUCED POLYMERIZATION OF ACRYLAMIDE AND METHACRYLAMIDE-BASED POLYMER GEL DOSIMETERS**

By

**ARIS DOYAN**

**March 2005**

**Chairman: Associate Professor Elias Saion, PhD**

**Faculty: Science**

Radiation-induced polymerization of acrylamide and methacrylamide-based polymer gel dosimeters potentially used for the verification of complex dose distributions in 3D associated with conformal radiotherapy has been studied using nuclear magnetic resonance (NMR), Raman spectroscopy and impedance analyzer methods at room temperature. Three polymer gels of polyacrylamide (PAAmG), polymethacrylamide (PMAAmG) and poly(acrylamide-co-methacrylamide) (PAAm-co-MAAmG) were synthesized in oxygen free condition from acrylamide (AAm) and methacrylamide (MAAm) as monomers and N, N'-methylene-bis-acrylamide (BIS) as Cross-linker at various concentration from 2% to 6% and 6% gelatin. The comonomers (AAm, MAAm and BIS) were dissolved in an aqueous gelatin



of deionized water in appropriate proportion by weight. In the manufacture of PAAm-co-MAAmG, the BIS was fixed at 2% and allowing the monomers varied from 2% to 6%. The dosimeters, each placed in a closed vial, were irradiated at constant dose rate with single doses ranging from 1 to 20 Gy for PAAmG, from 1 to 30 Gy for PMAAmG and from 1 to 40 Gy for PAAm-co-MAAmG using  $^{60}\text{Co}$  teletherapy  $\gamma$ -ray source.

The dose sensitivity of polymerization was determined indirectly using inversion recovery pulse sequence method (IR) and spin-echo based on the Carr-Purcell sequence of NMR method, which measures the spin-lattice relaxation rate  $R_1$  and the spin-spin relaxation rate  $R_2$  of proton in the polymer matrix. Analysis of the change of the relaxation rate  $\Delta R$  as a function of dose  $D$  has revealed that the dose response of polymerization is monoexponential function of the form  $\Delta R = A (1 - e^{-D/D_0})$ . The dose sensitivity  $D_0$  obtained for  $\Delta R_1$  is greater than that obtained for  $\Delta R_2$ , indicating the polymerization that determined from the spin-spin relaxation rate  $R_2$  method is more radiosensitive than that of the spin-lattice relaxation rate  $R_1$  method. The dose sensitivity  $D_0$  is proportional to the concentrations of monomer and cross-linker by factor  $k_A$  and  $k_B$  respectively. The value of  $k_B$  is greater than  $k_A$ , indicating that the dose resolution of polymerization is influenced by the cross-linker concentration, irrespective of the concentrations of monomer.

The dose sensitivity of polymerization has been determined directly using photon inelastic scattering of Raman spectroscopic technique by following the change of the Raman shift intensity at CH<sub>2</sub>, CH<sub>3</sub> and C=O stretching peaks for polymer formations and at C=C stretching peak for monomer and cross-linker consumptions. Analysis of the change of Raman shift intensity as a function of dose  $D$  has revealed that the dose response of polymerization is also monoexponential function of the form  $\Delta I = A (1 - e^{-D/D_0})$  and  $\Delta I = -A (1 - e^{-D/D_0})$  for the polymer formation and the consumption of monomer and cross-linker respectively. Analysis of the dose sensitivity  $D_0$  has confirmed that the dose sensitivity of polymerization by Raman method is in proportion with that of the NMR method. However,  $k_B$  value of NMR method is always greater than that of Raman method, indicating that the dose resolution of polymerization obtained from NMR method had overestimated the actual polymerization.

The dielectric study of PAAmG, PMAAmG and PAAM-co-MAAmG has showed that the dielectric properties relationship with dose is a quasi-dc response in series with the conductance  $G$ . The dielectric constant  $\epsilon'(\omega)$  and dielectric loss  $\epsilon''(\omega)$  of polymer gels increases with dose and concentration of BIS. The increase of  $\epsilon'(\omega)$  value with dose is due to an increase polymer formations with increasing dose and BIS consumption. The dielectric loss  $\epsilon''(\omega)$  also increases with dose and concentration of BIS as more polymers are formed and free ions are created with increasing dose and BIS consumption.



Furthermore, the conductivity study of PAAmG, PMAAmG and PAAm-co-MAAmG revealed that an increase of ac and dc components of conductivity with increasing dose. The power law type of ac conductivity increases with dose and BIS concentration and the frequency exponent  $s$  obtained decreases with increasing dose in the range of (0.798 - 0.776), (0.792 - 0.756) and (0.785 - 0.746) for PAAmG, PMAAmG and PAAm-co-MAAmG respectively. This has been attributed to hopping of ions trapped in the localized sites of the polymer gel matrix. The flat response of dc conductivity increases with dose. The dose sensitivity  $D_0$  obtained from the Arrhenius relationship, increases with increasing BIS concentration in the range of (12.72 - 13.35)Gy, (18.21 - 20.12)Gy and (22.47 - 27.70)Gy for PAAmG, PMAAmG and PAAm-co-MAAmG respectively, attributed to the increase of free ionic carriers in the polymer gels with increasing dose.





Abstrak tesis yang dikemukakan kepada Senat Universiti Putra Malaysia  
bagi memenuhi keperluan untuk ijazah Doktor Falsafah

**PEMPOLIMARAN DOSIMETER POLIMER GEL BERASASKAN  
AKRILAMIDA DAN METAKRILAMIDA  
OLEH ARUHAN SINARAN**

Oleh

**ARIS DOYAN**

**March 2005**

**Pengerusi: Profesor Madya Elias Saion, PhD**

**Fakulti : Sains**

Pempolimeran oleh aruhan sinaran terhadap dosimeter polimer gel berasaskan akrilamida dan meta-akrilamida yang berpotensi digunakan dalam menentukannya taburan dos kompleks dalam 3D yang dikaitkan dengan pengesahan radioterapi telah diselidiki dengan menggunakan kaedah resonans magnet nuklear (NMR), spektroskopi Raman dan analisis impedans pada suhu bilik. Tiga jenis polimer gel bebas oksigen telah disintesis iaitu terdiri daripada poliakrilamida (PAAmG), polimeta-akrilamida (PMAAmG) dan poliakrilamida-co-meta-akrilamida (PAAm-co-MAAmG) daripada akrilamida dan meta-akrilamida sebagai monomer dan N, N'-metalin-bis- akrilamida (BIS) sebagai pautsilang pada komposisi berubah daripada 2% hingga 6% dan 6% gelatin. Komonomer (AAm, MAAm dan BIS) dilarutkan dalam gelatin berair nyahion pada kadar tertentu



mengikuti berat. Dalam penyediaan PAAm-co-MAAmG, komposisi BIS ditetapkan pada 2% dan komposisi monomer berubah daripada 2% hingga 6%. Semua dosimeter yang setiap satu diisikan dalam vial tertutup, disinarkan dengan sinar  $\gamma$  pada kadar dos malar tetapi berbeza dos tunggal dalam julat 1 - 20 Gy untuk PAAmG, 1 - 30 Gy untuk PMAAmG dan 1 - 40 Gy untuk PAAm-co-MAAmG dengan menggunakan sumber  $^{60}\text{Co}$  daripada jenis teleterapi.

Dos sensitif pada pempolimeran telah ditentukan secara tak terus dengan menggunakan kaedah NMR berasaskan metoda pulsa inversi rekoveri dan spin-gema menurut turutan Carr-Purcell yang dapat mengukur kadar santaian spin-kekisi  $R_1$  dan kadar santaian spin-spin  $R_2$  bagi proton dalam matrik polimer. Analisis perubahan terhadap kadar santaian  $\Delta R$  sebagai fungsi dos  $D$  menunjukkan bahawa tindak balas dos pada pempolimeran adalah menurut fungsi monoeksponen dalam bentuk  $\Delta R = A (1 - e^{-D/D_0})$ . Dos sensitif  $D_0$  didapati untuk  $\Delta R_1$  lebih besar daripada untuk  $\Delta R_2$ , menunjukkan bahawa pempolimeran diukur dengan kaedah kadar santaian spin-spin  $R_2$  lebih sensitif terhadap sinaran daripada kaedah kadar santaian spin-kekisi  $R_1$ . Dos sensitif  $D_0$  didapati juga berubah terus dengan komposisi monomer dan petautsilang masing-masing dengan faktor  $k_A$  and  $k_B$ . Nilai  $k_B$  lebih besar nilai  $k_A$ , menunjukkan bahawa resolusi dos pada pempolimeran ditentukan oleh komposisi petautsilang tanpa bergantung kepada komposisi monomer.

Dos sensitif pada pempolimeran juga telah ditentukan juga dengan kaedah terus menggunakan penyerakan foton tak kenyal menurut teknik keamatan anjakan Raman pada puncak-puncak regangan  $\text{CH}_2$ ,  $\text{CH}_3$  and  $\text{C=O}$  bagi pembentukan dan pada puncak regangan  $\text{C=C}$  bagi penggunaan monomer and petautsilang. Analisis perubahan keamatan anjakan Raman sebagai fungsi dos  $D$  menunjukkan bahawa tindak balas dos pada pempolimeran juga mempunyai fungsi monoeksponen dalam bentuk  $\Delta I = A (1 - e^{-D/D_0})$  dan  $\Delta I = -A (1 - e^{-D/D_0})$  masing-masing untuk pembentukan polimer dan penggunaan monomer dan petautsilang. Analisis dos sensitif  $D_0$  telah mengesahkan dos sensitif pada pempolimeran dengan kaedah Raman adalah setara dengan kaedah NMR. Bagaimanapun, nilai  $k_B$  kaedah NMR lebih besar daripada nilai kaedah Raman, menunjukkan resolusi dose pada pempolimeran kaedah NMR melebihi jangkaan daripada pempolimeran sebenar.

Pengajian dielektrik PAAmG, PMAAmG dan PAAm-co-MAAmG telah menunjukkan bahawa hubungan sifat dielektrik dengan dos adalah sambutan quasi-dc secara siri dengan konduktans  $G$ . Nilai pemalar dielektrik  $\epsilon'(\omega)$  dan lesapan dielektrik  $\epsilon''(\omega)$  bertambah dengan dos dan komposisi BIS. Penambahan nilai  $\epsilon'(\omega)$  dengan dos adalah kerana lebih banyak polimer terbentuk semasa penambahan dos dan BIS. Lesapan dielektrik  $\epsilon''(\omega)$  juga



bertambah dengan dos dan BIS kerana lebih banyak polimer dan ion bebas terbentuk semasa penambahan dos dan BIS.

Lanjutan daripada pengajian kekonduksian PAAmG, PMAAmG dan PAAm-co-MAAmG menunjukkan bahawa komponen kekonduksian arus ulangalik (a.u) dan arus terus (a.t) bertambah dengan dos. Komponen kekonduksian a.u mematuhi sambutan hukum kuasa yang bertambah dengan dos dan nilai eksponen frekuensi yang diperolehi berkurangan dengan pertambahan dos dalam julat (0.798 - 0.776), (0.792 - 0.756) and (0.785 - 0.746) bagi masing-masing PAAmG, PMAAmG dan PAAm-co-MAAmG. Ini disebabkan oleh ion hop tertahan pada kedudukan setempat dalam matrik polimer. Sambutan kekonduksian a.t yang mendatar bertambah dengan dos. Dos sensitif  $D_0$  yang diperolehi daripada perkaitan jenis Arrhenius didapati bertambah dengan bertambah komposisi BIS dalam julat (12.72 - 13.35)Gy, (18.21 - 20.12)Gy and (22.47 - 27.70)Gy masing-masing bagi PAAmG, PMAAmG dan PAAm-co-MAAmG disebabkan oleh penambahan pembawa cas bebas dalam polimer gel dengan penambahan dos.



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All praises due to Allah, Lord of the universe. Only by His grace and mercy this thesis can be completed.




I certify that an Examination Committee met on 16<sup>th</sup> March 2005 to conduct the final examination of Aris Doyan on his Doctor of Philosophy thesis entitled "Radiation-Induced Polymerization of Acrylamide and Methacrylamide-Based Polymer Gel Dosimeters" in accordance with Universiti Pertanian Malaysia (Higher Quantity) Act 1980 and Universiti Pertanian Malaysia (Higher Quantity) Regulations 1981. The Committee recommends that the candidate be awarded the relevant degree. Members of the Examination Committee are as follows:

**Azmi Zakaria, PhD**  
Associate Professor  
Faculty of Science  
Universiti Putra Malaysia  
(Chairman)

**Wan Mahmood Mat Yunus, PhD**  
Professor  
Faculty of Science  
Universiti Putra Malaysia  
(Internal Examiner)

**Zainul Abidin Hasan, PhD**  
Associate Professor  
Faculty of Science  
Universiti Putra Malaysia  
(Internal Examiner)

**Abd Aziz Tajuddin, PhD**  
Professor  
School of Physics  
Universiti Sains Malaysia  
(External Examiner)

  
\_\_\_\_\_  
**GULAM RUSUL RAHMAT ALI, PhD**  
Professor / Deputy Dean  
School of Graduate Studies  
Universiti Putra Malaysia

Date: **19 MAY 2005**

This thesis submitted to the Senate of Universiti Putra Malaysia and has been accepted as fulfillment of the requirement for the degree of Doctor of Philosophy. The members of the Supervisory Committee are as follows:

**Elias Saion, PhD**  
Associate Professor  
Faculty of Science  
Universiti Putra Malaysia  
(Chairman)

**Abdul Halim Shaari, PhD**  
Professor  
Faculty of Science  
Universiti Putra Malaysia  
(Member)

**Wan Daud Wan Yusoff, PhD**  
Associate Professor  
Faculty of Science  
Universiti Putra Malaysia  
(Member)

**Zaki Abd Rahman, PhD**  
Associate Professor  
Faculty of Science  
Universiti Putra Malaysia  
(Member)

**Kamaruzzaman Hj. Dahlan, PhD**  
MINTS Bangi  
(Member)



---

**AINI IDERIS, PhD**  
Professor/Dean  
School of Graduate Studies  
Universiti Putra Malaysia

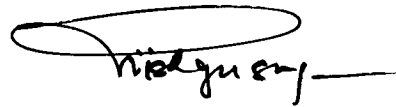
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## DECLARATION

I hereby declare that the thesis is based on my original work except for quotations and citations, which have been duly acknowledged. I also declare that it has not been previously or concurrently submitted for any other quantity at UPM or other institutions.

A handwritten signature in black ink, appearing to read 'Aris Doyan', is written above a horizontal line.

ARIS DOYAN

Date: 2 May 2005

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