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

ARIS DOYAN

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By

ARIS DOYAN

Thesis Submitted to the School of Graduate Studies, Universiti Putra Malaysia, in Fulfillment of the Requirements for the Degree of Doctor of Philosophy

March 2005

UPM

In the Name of Allah, the Beneficient, 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

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of deionized water in appropriate proposition 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}Co teletherapy γ-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 ΔR as a function of dose D has revealed that the dose response of polymerization is monoexponential function of the form $\Delta R = A\ (1 - e^{-D/Do})$. The dose sensitivity D_0 obtained for ΔR_1 is greater than that obtained for ΔR_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_2, CH_3 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 $\Delta I = A\ (1 - e^{-D/Do})$ and $\Delta I = -A\ (1 - e^{-D/Do})$ 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 $\varepsilon'(\omega)$ and dielectric loss $\varepsilon''(\omega)$ of polymer gels increases with dose and concentration of BIS. The increase of $\varepsilon'(\omega)$ value with dose is due to an increase polymer formations with increasing dose and BIS consumption. The dielectric loss $\varepsilon''(\omega)$ 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**

Pempolimaran oleh aruhan sinaran terhadap dosimeter polimer gel berasaskan akrilamida dan meta-akrilamida yang berpotensi digunakan dalam menentusahkan taburan dos komplek 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 disentisis 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 petautsilang pada komposisi berubah daripada 2% hingga 6% dan 6% gelatin. Komonomer (AAm, MAAm dan BIS) dilarutkan dalam gelatin berair nyahion pada kadar tertentu

mengikut 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 vail tertutup, disinarkan dengan sinar γ 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}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 ΔR sebagai fungsi dos D menunjukkan bahawa tindak balas dos pada pempolimeran adalah menurut fungsi monoeksponen dalam bentuk $\Delta R = A\ (1 - e^{-D/Do})$. Dos sensitif D_0 didapati untuk ΔR_1 lebih besar daripada untuk ΔR_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 CH_2, CH_3 and C=O bagi pembentukan dan pada puncak regangan 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 $\Delta I = A\ (1 - e^{-D/Do})$ dan $\Delta I = -A\ (1 - e^{-D/Do})$ 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 $\varepsilon'(\omega)$ dan lesapan dielektrik $\varepsilon''(\omega)$ bertambah dengan dos dan komposisi BIS. Penambahan nilai $\varepsilon'(\omega)$ dengan dos adalah kerana lebih banyak polimer terbentuk semasa penambahan dos dan BIS. Lesapan dielektrik $\varepsilon''(\omega)$ 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 frekuens s 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 16th 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:

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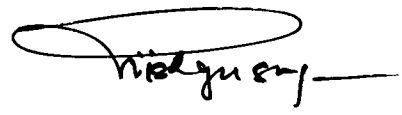
DECLARATION

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.



ARIS DOYAN

Date: 2 May 2005

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