UNIVERSITI PUTRA MALAYSIA

PREPARATION AND CHARACTERIZATION OF HYDROGELS FROM CARBOXYMETHYL CELLULOSE AND 1- VINYL-2-PYRROLIDONE USING IRRADIATION TECHNIQUES FOR SLOW RELEASE APPLICATION

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MASTER OF SCIENCE
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By

NORHANIFAH BINTI MOHD YAZID

Thesis Submitted to School of Graduate Studied, Universiti Putra Malaysia, in Fulfilments of the Requirements for the Degree of Master of Science.

February 2011

Abstract of thesis presented to the Senate of University Putra Malaysia in fulfilment of the requirement for the degree of the Master of Science

PREPARATION AND CHARACTERIZATION OF HYDROGELS FROM CARBOXYMETHYL CELLULOSE AND 1- VINYL-2-PYRROLIDONE USING IRRADIATION TECHNIQUES FOR SLOW RELEASE APPLICATION

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February 2011

Chairman : Associate Professor Mansor bin Ahmad, PhD

Faculty : Science

Hydrogels from carboxymethyl cellulose (CMC) and 1-vinyl-2-pyyrolidone (VP) were prepared via electron beam and ultraviolet radiations. Optimization of the preparation parameters was carried out for both methods where the optimum stirring time and percentage of crosslinking agents were at 3 hour of stirring and 5 % of BIS respectively. The hydrogels were prepared by irradiating CMC/VP samples with electron beam at 5, 10, 15, 20 and 25 kGy irradiation doses. For ultraviolet radiation, the optimum irradiation time and percentage of photoinitiator were at 8 hour of exposure and 1 % of photoinitiator. The effect of electron beam dose and concentrations of CMC on gel fraction, swelling behaviour, thermal properties and surface morphology were studied. When the irradiation dose and the concentration of CMC were increased, the gel fraction increased. Increasing the irradiation dose or the concentration of CMC resulted in the decrease of degree of swelling. The highest degree of swelling was obtained in alkaline medium followed by distilled water, salt

and acidic media. In the temperature-swelling studies, the highest degree of swelling was recorded at room temperature (25 ^{o}C). The FTIR analysis showed there was a intermolecular interaction between C=O and O-H which could be due to the intermolecular hydrogen bonding of carboxylic group and non-substituted hydroxyl groups in the CMC. Thermogravimetric analysis and differential thermogravimetric of hydrogels showed that thermal stability of hydrogels increased with increasing irradiation dose, but decreased when the concentration of CMC was increased. Differential scanning spectroscopy thermograms showed that melting temperature of hydrogels was affected by irradiation dose and concentration of CMC. The surface morphology study showed the pore size of the hydrogels was dependent on irradiation dose and concentration of CMC which affected the crosslinking density of the hydrogels. For the controlled release study, the highest released obtained from hydrogels irradiated at 10 kGy. For the ultraviolet radiation study, the gel fraction obtained was less than electron beam radiation and the degree swelling of hydrogels decreased with increasing gel fraction. The thermal properties showed that the melting temperatures of the hydrogels decreased compared to pure CMC and VP. The surface morphology study showed the pores size obtained after ultraviolet radiation was heterogeneous. Overall analysis revealed that electron beam radiation was the better technique compared to the ultraviolet radiation for preparing CMC/VP hydrogels.

Abstrak tesis yang dikemukan kepada Senat Universiti Putra Malaysia sebagai memenuhi keperluan untuk ijazah Master Sains

PENYEDIAAN DAN PENCIRIAN HIDROGEL DARIPADA KARBOKSILMETIL SELULOSA DAN 1-VINIL-2-PYRROLIDONE MENGGUNAKAN TEKNIK-TEKNIK RADIASI UNTUK APLIKASI PENGALIRAN PERLAHAN

Oleh

NORHANIFAH BINTI MOHD YAZID

Februari 2011

Pengerusi : Profesor Madya Mansor bin Ahmad, PhD

Fakulti : Sains

Hidrogel daripada karboksimetil selulosa (CMC) dan 1-vinil-2-pyrrolidone (VP) telah disediakan melalui sinaran alur elektron dan ultraviolet. Pengoptimuman parameter penyediaan dilakukan untuk kedua-dua kaedah di mana masa pengadunan dan peratusan agen rangkai silang yang optimum adalah 3 jam pengadunan dan 5 % dari BIS. Hidrogel telah disediakan dengan penyinaran alur elektron kepada sample CMC / VP dengan sinaran dos 5, 10, 15 20 dan 25 kGy. Untuk sinaran ultraviolet, masa sinaran dan peratusan fotoinisiator yang optimum adalah 8 jam masa sinaran dan 1 % dari fotoinisiator. Pengaruh dos alur elektron dan kepekatan CMC pada kandungan gel, perilaku pengembangan, sifat terma dan morfologi permukaan dikaji. Ketika dos sinaran dan kepekatan CMC meningkat, kandungan gel meningkat. Peningkatan dos sinaran atau kepekatan CMC mengakibatkan penurunan darjah pengembungan. Pengembungan tahap tertinggi diperolehi melalui medium alkali diikuti dengan medium air suling, garam dan asid. Dalam kajian suhu bagi tahap

pengembungan, tahap tertinggi mencatatkan pengembungan pada suhu bilik (25 ^{o}C). Keputusan analisis FTIR menunjukkan ada interaksi intramolekul antara C = O dan OH yang disebabkan oleh ikatan hidrogen intramolekul kumpulan karboksilik dan kumpulan pengantian bukan hidroksil dalam CMC. Analisi termogravimetri dan perbezaan termogravimetri dari hidrogel menunjukkan bahawa kestabilan terma dari hidrogel meningkat dengan dos sinaran meningkat, tetapi menurun ketika ada kepekatan CMC meningkat. Termogram spektroskopi pengimbasan perbezaan menunjukkan bahawa suhu lebur hidrogel dipengaruhi oleh dos sinaran dan kepekatan CMC. Kajian morfologi permukaan menunjukkan saiz pori hidrogel bergantung pada dos sinaran dan kepekatan CMC yang mempengaruhi kepadatan rangkai silang hidrogel. Untuk kajian bagi perlepasan terkawal, perlepasan tertinggi diperolehi daripada hidrogel yang disinari pada 10 kGy. Untuk kajian sinaran ultraviolet, kandungan gel yang diperolehi kurang dari sinaran alur elektron dan darjah pengembungan hidrogel menurun dengan peningkatan kandungan gel. Sifat terma menunjukkan bahawa takat lebur hidrogel menurun berbanding dengan CMC dan VP yang asli. Kajian morfologi permukaan menunjukkan saiz pori-pori yang diperolehi selepas sinaran ultraviolet adalah heterogen. Keseluruhan analisis mendedahkan bahawa sinaran alur elektron adalah teknik yang lebih baik berbanding dengan sinaran ultraviolet untuk penyediaan CMC / VP hidrogel.

ACKNOWLEDGEMENTS

First of all, thanks and Alhamdullillah to Allah for giving me faith and strength to complete my project.

I would like to express my deepest gratitude to Assoc. Prof. Dr. Mansor B. Hj. Ahmad for his supervisions and guidance throughout this study. He is rightfully top the list who has carried me through this study. Thank also to Dr. Kamaruddin Hashim and Dr. Norazowa Bt. Ibrahim. Without their guidances, advices and undying efforts, surely my project would not success.

I also would like to take this opportunity to express uncountable thanks to my parents; Mohd Yazid B. Noh and Fauziah Bt. Ismail, my siblings for their supports and encouragements with spare me the strength to finish this project.

Special thanks must go to all Chemistry Department's staffs especially Mrs Zaidina and Mrs Norazlina, also to Agency Nuclear Malaysia's staffs ; Miss Sabariah, Miss Nurain and Miss Nor Akmal who are always there with their technical expertise.

My friends and all polymer group members, thanks for your support, faith and always with me up and down. Thanks also Mr Muhammad Firdaus B. Haji Dasuki and I really appreciated the encouragement and understanding given by him during doing this study.

Last but not the least, thanks to Ministry of Science, Technology and Innovation (MOSTI) and Universiti Putra Malaysia (UPM) for facilities financial support during my study.

I certify that an Examination Committee met on date on viva to conduct the final examination of Norhanifah Bt. Mohd Yazid on her Master of Degree thesis entitle " Preparation and Characterization of Hydrogel from Carboxymethyl Cellulose and 1-Vinyl-2-Pyrrolodone using Irradiation Techniques for Slow Release Application " in accordance with Universiti Pertanian Malaysia (Higher Degree) Act 1980 and Universiti Pertanian Malaysia (Higher Degree) Regulations 1981. The Committee recommends that the student be awarded the relevant degree.

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DECLARATION

I declare that the thesis is my original work except for quotations and citations which have been duly acknowledged. I also declare that it has not been previously, and is not concurrently, submitted for any other degree at Universiti Putra Malaysia or any other institution.

NORHANIFAH BINTI MOHD YAZID

Date: 9 February 2011

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