SYNTHESIS AND OPTIMIZATION OF POLY(HEMA-*CO*-EGDMA-*CO*-VBC) FOR PREPARATION OF HYPERCROSSLINKED AMINATED POLYMER USING DoE METHOD

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SUPERVISOR'S DECLARATION

I hereby declare that I have checked this thesis and in my opinion, this thesis is adequate in terms of scope and quality for the award of the degree of Master of Science in Chemical Engineering.

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STUDENT'S DECLARATION

I hereby declare that the work in this 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 degree at Universiti Malaysia Pahang or any other institutions.

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Thesis submitted in fulfillment of the requirements
for the award of the degree of
Master of Science

Faculty of Chemical Engineering & Natural Resources

UNIVERSITI MALAYSIA PAHANG

JULY 2017

ACKNOWLEDGEMENTS

I would like to express my special appreciation and thanks to my supervisor, Dr. Norhayati Bt Abdullah. You have been a brilliant mentor for me. I would like to thank you for your never ending support during my tenure as research student under your guidance, for giving insightful comments and suggestions of which without it, my research path would be a difficult one . Your advice on my research has been valuable.

A special thanks to my family. Words cannot express how grateful I am to my mother, father, brother and sister for the love and support throughout these years. Your prayer for me was what sustained me thus far. I am also indebted to the Ministry of Higher Education and Universiti Malaysia Pahang for funding my study.

I would also like to thank all of my friends who supported me in writing, and motivate me to strive towards my goal. I am sincerely grateful to the staffs of Faculty of Chemical Engineering and Natural Resources Faculty who helped me in many ways and made my stay in UMP pleasant and unforgettable.

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LIST OF SYMBOLS

W_s	Weight of swollen beads (g)
W	Weight of dry beads (g)
RUN	Experimental run
A	Effect of EGDMA content
B	Effect of VBC content
C	Effect of monomer concentration
R^2	Coefficient of determination
P-value	Calculated probability

LIST OF ABBREVIATIONS

AIBN	Azobisisobutyronitrile
ANOVA	Analysis of Variance
BET	Brunauer-Emmet-Teller
BPO	Benzoyl Peroxide
DMBA	Dimethylbutylamine
DoE	Design of Experiment
DVB	Divinylbenzene
EDMA	Ethylene Dimethacrylate
FESEM	Field Emission Scanning Electron Microscopy
FTIR	Fourier Transform Infrared Spectroscopy
HEMA	2-hydroxyethylmethacrylate
HHP	Hydrophilic Hypercrosslinked Polymer
HP	Hydrophilic Precursor
HPLC	High Performance Liquid Chromatography
HQAR	Hydrophilic Quarternary Ammonium Resin
PMMA	Poly(methyl methacrylate)
PSD	Particle Size Distribution
PtBMA	Poly(t-butyl methacrylate)
PVP	Polyvinylpyrrolidone
RSM	Response Surface Methodology
SEM	Scanning Electron Microscope
SPE	Solid-Phase Extraction
SSA	Specific Surface Area
TGA	Thermogravimetric Analysis
XRF	Xray Flourescent

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ABSTRAK

Hidrofilik kuarternari resin ammonia (HQAR) telah berjaya dihasilkan yang berasal daripada persilangan tertinggi polimer Poly(HEMA-*co*-EGDMA-*co*-VBC). Objektif projek ini adalah untuk menghasilkan HQAR melalui tiga peringkat, sintesis pelopor (pempolimeran suspensi), tindak balas persilangan tertinggi dan tindak balas aminasi. Untuk pempolimeran suspensi, beberapa pemboleh ubah telah divariasikan seperti nisbah pelarut, putaran pengacau, jumlah pemula, jumlah penstabil, jumlah penyilang, jumlah pembantu monomer, dan kepekatan monomer. Untuk mendapatkan partikel yang berkualiti tinggi, jumlah penyilang sepatutnya digunakan pada jumlah tertinggi. Kesan penyilang, jumlah pembantu monomer dan kepekatan monomer telah dipilih untuk penapisan (Design of Experiment) DoE bagi menentukan interaksi antara ketiga-tiga faktor tersebut. Menerusi metode DoE, kesan penyilang didapati sebagai pemboleh ubah terpenting. Manakala, kesan penyilang dan jumlah pembantu monomer dipilih untuk pengoptimuman DoE bagi mengoptimum zarah untuk reaksi persilangan itertinggi. Dalam reaksi persilangan tertinggi, kesan penyilang, jumlah pembantu monomer dan nisbah pemangkin terhadap luas permukaan spesifik (LPS) zarah dikaji. Bagi bahagian terakhir dalam reaksi persilangan tertinggi melibatkan pertukaran pelopor optimum kepada persilangan tertinggi polimer dengan peningkatan nilai LPS. Dalam peringkat terakhir projek ini, optimum persilangan tertinggi polimer difungsikan dengan Dimethyl butylamine (DMBA) untuk mengasilkan HQAR dengan ion positif berjaya dilekatkan pada tulang belakang polimer. Secara keseluruhannya, pencirian yang terlibat adalah Scanning Electron Microscope (SEM), Fourier Transform Infrared Spectrospcopy (FTIR), X-ray Flourescence (XRF), CHNS analysis, Brunauer-Emmet-Teller (BET) dan Thermal Gravimetric Analysis (TGA).

ABSTRACT

Hydrophilic quarternary ammonium resin (HQAR) was successfully derived from hypercrosslinked Poly(HEMA-*co*-EGDMA-*co*-VBC) polymer. The objective of this project is to develop HQAR *via* three stages, precursor synthesis (suspension polymerization), hypercrosslinking reaction, and functionalization reaction (amination). For first stage which suspension polymerization, several parameters were varied which were solvent mixture, solvent/porogen ratio, stirrer rotation, amount of initiator, amount of stabilizer, amount of crosslinker, amount of co-monomer and monomer concentration. To obtain highest quality particles, amount of crosslinker should being used at highest amount. Effect of crosslinker, co-monomer amount and monomer concentration were chosen for screening part using Design of Experiment (DoE) to study the interaction between these three factors. DoE analysis showed that the most significant factor was amount of crosslinker. Meanwhile, effects of crosslinker and co-monomer amount were chosen for DoE optimization part to optimize the particles for hypercrosslinking reaction. In second stage that involved hypercrosslinking reaction, the effect of crosslinker, co-monomer amount and catalyst ratio on particles specific surface area (SSA) were studied. Co-monomer and crosslinker amount to be significant parameters in order to obtain high SSA particles. Optimized precursors were chosen for optimization of hypercrosslinked polymer. In the last stage of this study, the optimized hypercrosslinked polymer was functionalized with Dimethylbutylamine (DMBA) to produce HQAR with positively charge group successfully attached to the backbone. The characterization involved in all stages were Scanning Electron Microscope (SEM), Fourier Transform Infrared Spectrospcopy (FTIR), X-ray Flourescence (XRF), CHNS analysis, Brunauer-Emmet-Teller (BET) and Thermal Gravimetric Analysis (TGA).

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