PREPARATION AND CHARACTERIZATION OF POLY(MMA-*CO*-EGDMA-*CO*-VBC) *VIA* PHOTOINITIATED DISPERSION POLYMERIZATION ROUTE TO SULFONATED HYPERCROSSLINKED RESIN

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SUPERVISOR'S DECLARATION

We hereby declare that we have checked this thesis and in our opinion, this thesis is adequate in terms of scope and quality for the award of the degree of Master of Science.

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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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TABLE OF CONTENT

LIST OF TABLES

LIST OF FIGURES

LIST OF SYMBOLS

A	Amount of EGDMA
B	Amount of VBC
C	Amount of PI
D	Reaction times
R^2	Correlation coefficient
p-value	Calculated probability
F-value	F statistic
α-value	Confidence level
RUN	Experimental run

LIST OF ABBREVIATIONS

AA	Acrylic Acid
ANOVA	Analysis of Variance
BET	Brunauer-Emmet-Teller
CCD	Central Composite Design
DCE	1,2-Dichloroethane
DoE	Design of Experiment
DVB	Divinylbenzene
EGDMA	Ethylene-Glycol-Dimethacrylate
$FeCl_3$	Iron Chloride
FTIR	Fourier Transform Infrared Spectroscopy
HCPs	Hypercrosslinked Polymers
HNO_3	Acid Nitric
HPLC	High Performance Liquid Chromatography
IEC	Ion Exchange Capacity
MAA	Methacrylic Acid
MMA	Methyl Methacrylate
PI	Photoinitiator
PMEV	Poly(MMA-*co*-EGDMA-*co*-VBC)
PMMA	Poly(Methyl-Methacrylate)
PS	Polystyrene
PSD	Particle Size Distribution
PSEV	Poly(Styrene-*co*-EGDMA-*co*-VBC)
PVP	Polyvinylpyrrolidone
RSM	Response Surface Method
SEM	Scanning Electron Microscopy
SPE	Solid-Phase Extraction
SSA	Specific Surface Area
TGA	Thermogravimetric Analysis
VBC	4-vinylbenzyl chloride
XRF	X-ray Flourescent

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ABSTRAK

Resin polimer berpemaut silang hiper sulfonat telah dihasilkan daripada poli(MMA-*co*-EGDMA-*co*-VBC). Dalam kajian ini, resin polimer berpemaut silang hiper sulfonat dihasilkan melalui dua peringkat; pempolimeran penyebaran fotopemula dilaksanakan untuk mensintesis pelopor dan pempolimeran secara lanjutan yang melibatkan tindak balas paut silang hiper dan tindak balas fungsian untuk menghasilkan resin sulfonat. Pada peringkat pertama (pempolimeran penyebaran fotopemula) dilaksanakan untuk mengatasi masalah berkaitan dengan kaedah terdahulu dalam pempolimeran penyebaran dalam aspek gangguan reagen fungsi semasa peringkat nukleasi. Beberapa parameter dikaji pada peringkat pertama kajian semasa kajian awal dijalankan iaitu jenis monomer, jenis fotopemula atau *photoinitiator* (PI), masa tindak balas, jumlah pemaut silang, jumlah ko-monomer, jumlah penstabil, serta nisbah pelarut kepada porogen. Dalam ujian saringan yang menggunakan reka bentuk faktoran penuh 2^4 telah digunakan untuk menentukan parameter penting yang mungkin mempengaruhi hasil pelopor polimer yang diperoleh dari segi jumlah pemaut silang, jumlah ko-monomer, jumlah PI dan masa tindak balas. Analisis DoE menunjukkan bahawa masa tindak balas ialah parameter yang paling penting. Selanjutnya, jumlah pemaut silang, jumlah ko-monomer dan jumlah PI dipilih sebagai parameter dalam kajian pengoptimuman untuk mendapatkan hasil yang banyak dan pelopor polimer yang berkualiti. Pada peringkat kedua (pempolimeran secara lanjutan), pelopor yang teroptimum menjalani dua pendekatan iaitu tindak balas paut silang hiper dilaksanakan terlebih dahulu sebelum tindak balas fungsian dan pendekatan kedua tindak balas paut silang hiper dilaksanakan selepas tindak balas fungsian. Kedua-dua pendekatan dilaksanakan untuk mengatasi luas permukaan spesifik yang rendah pada resin sulfonat. Pencirian yang terlibat adalah mikroskop electron pengimbas atau *scanning electron microscope* (SEM), pendarflour sinar-X atau *X-ray fluorescence* (XRF), analisis CHNS, analisis Brunauer-Emmett-Teller (BET), taburan saiz zarah atau *particle size distribution* (PSD) dan analisis gravimetric terma atau *thermal gravimetric analysis* (TGA) untuk kedua-dua peringkat manakala nilai kapasiti pertukaran ion (IEC), luas permukaan spesifik, morfologi dan kestabilan terma terlibat dalam pempolimeran secara lanjutan. Kesimpulannya, kajian ini memberi garis panduan yang penting dan kefahaman asas tentang resin polimer berpemaut silang hiper sulfonat yang dihasilkan daripada poli(MMA-*co*-EGDMA-*co*-VBC). Dapatan kajian menunjukkan bahawa pendekatan pertama dalam pempolimeran secara lanjutan menghasilkan luas permukaan spesifik dan nilai IEC yang lebih tinggi berbanding pendekatan kedua.

ABSTRACT

A sulfonated hypercrosslinked polymer resin was derived from poly(MMA-*co*-EGDMA-*co*-VBC) polymer. In the present study, the sulfonated hypercrosslinked polymer resin was synthesized *via* two stages; photoinitiated dispersion polymerization employed to synthesis polymer precursor and post-polymerization which involved hypercrosslinking reaction and functionalization reaction to obtained sulfonated resin. In the first stage (photoinitiated dispersion polymerization) was conducted to counter the problems regarding the method of conventional dispersion polymerization in terms of the disturbance of functional reagents during nucleation stage. In this stage, which was the preliminary stage of the study, several parameters had been investigated: types of monomer, types of photoinitiators (PI), reaction times, and the amount of crosslinker, co-monomer, stabilizer as well as solvent/porogen ratio. In a screening test using a full factorial design of experiment (DoE), a 2^4 full factorial design was used to determine the significant parameters that could affect the yield of polymer precursor obtained in terms of the amounts of crosslinker, co-monomer, PI and also reaction times. The DoE analysis showed that the most significant factor was the reaction times. For further optimization study, central composite design (CCD) approach were design using the factor the amounts of crosslinker, co-monomer and PI were chosen as parameters in order to obtain high yield and good quality of polymer precursor. In the second stage (post-polymerization), the optimized precursor underwent two approaches which is hypercrosslinking reaction was conducted first before functionalization reaction and second approach hypercrosslinking reaction conducted after functionalization. Both approaches were conducted to overcome the low specific surface area (SSA) of sulfonated resin. The characterizations involved were the scanning electron microscopy (SEM), Fourier transform infrared spectroscopy (FTIR), X-ray fluorescence (XRF), CHNS analysis, Brunauer-Emmet-Teller (BET) analysis, particle size distribution (PSD) and thermal gravimetric analysis (TGA) in both stages meanwhile ion exchange capacity (IEC) value, specific surface area, morphology and thermal stability involved in the post-polymerization. In conclusion, the present study has provided significant guidelines and basic understanding of the synthesized sulfonated hypercrosslinked polymer resin derived from poly(MMA-*co*-EGDMA-*co*-VBC) polymer. It was found that first approach in post-polymerization resulting higher SSA and IEC value compared to second approach.

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