

SYNTHESIS OF POLYSULFIDE
ADSORBENT FROM WASTE COOKING
PALM OIL FOR THE REMOVAL OF IRON (III)

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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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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 institution.

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ABSTRAK

Sulfur unsur dan minyak sawit masak sisa (WCO) masing-masing merupakan hasil sampingan industri yang banyak daripada industri petrokimia dan pemprosesan makanan. WCO telah berjaya digunakan sebagai penghubung silang untuk menyediakan polimer kandungan sulfur tinggi melalui pemvulkanan songsang untuk penyingkiran ion ferik (Fe^{3+}) dalam air sisa. Dalam kerja semasa, WCO telah dicirikan menggunakan GC-MS, FTIR, dan TGA untuk menilai kebolehlaksanaan untuk digunakan sebagai penyambung silang dalam pemvulkanan songsang. Polisulfida yang dihasilkan telah dianalisis berdasarkan parameter tindak balas yang berbeza termasuk sulfur kepada nisbah silang silang WCO, suhu, masa, dan penggunaan NaCl oleh FTIR, TGA, XRD, SEM-EDX, dan BET. Proses penjerapan dikaji dengan mempelbagaikan pH, kepekatan awal Fe^{3+} , dan dos dengan kajian isoterma dan kinetik yang sesuai. Ikatan berfungsi yang diperlukan dan ketidaktepuan WCO telah disahkan oleh FTIR dan GC-MS, dan kestabilan pada suhu yang lebih tinggi telah disahkan menggunakan TGA. Polisulfida telah disintesis di bawah kacau (500 rpm) WCO dengan unsur sulfur pada tiga suhu berbeza ($195\text{ }^{\circ}\text{C}$, $190\text{ }^{\circ}\text{C}$, dan $185\text{ }^{\circ}\text{C}$) dengan tiga nisbah silang silang (70, 60, dan 50 wt% sulfur). Dua set masa tindak balas yang berbeza; 45 min dan 60 min telah digunakan untuk sintesis polisulfida. Sifat fizikokimia polisulfida yang dihasilkan telah ditentukan dan kestabilan terma dianalisis. Spektrum FTIR termasuk pecahan C=C dan pembentukan ikatan C-S mengesahkan perubahan kumpulan berfungsi antara WCO dan polimer yang dihasilkan. Kesan trigliserida tepu dan tak tepu WCO jelas kelihatan dalam mikrograf SEM. Polisulfida dengan nisbah suapan sulfur 70 wt % menunjukkan luas permukaan yang lebih baik. TGA dan DTG menunjukkan bahawa sifat terma yang lebih baik dan polisulfida yang stabil boleh diperolehi daripada jumlah kandungan sulfur yang lebih tinggi. Keadaan optimum lain untuk pemvulkanan songsang juga direkodkan, seperti 60 minit masa tindak balas dan $195\text{ }^{\circ}\text{C}$. Permukaan diubah suai menggunakan NaCl sebagai porogen untuk menambah luas permukaan. Keputusan BET mengesahkan peningkatan luas permukaan polisulfida berliang yang disediakan menggunakan NaCl. Prestasi penyingkiran dinilai dengan mengkaji kesan parameter termasuk keliangan, pH larutan, kepekatan awal Fe^{3+} , dan jumlah dos polisulfida. Penyingkiran tertinggi dicatatkan untuk polisulfida berliang. pH berasid (pH=3) adalah baik untuk penyingkiran Fe^{3+} . Dos optimum ialah 20 mg/100 mL. Isoterma penyingkiran telah dikaji dan dipasang paling baik untuk isoterma Freundlich. Kajian kinetik proses penyingkiran telah sesuai dengan baik dalam model kinetik pseudo kedua linear. Disimpulkan bahawa polisulfida tervulkan songsang boleh digunakan dengan jayanya untuk mengurangkan Fe^{3+} dalam air sisa dan mengurangkan ancaman alam sekitar bagi WCO yang berlebihan.

ABSTRACT

Elemental sulfur and waste cooking palm oil (WCO) are abundant industrial by-products from the petrochemical and food processing industries, respectively. WCO has been successfully used as a crosslinker to prepare a high-sulfur-content polymer through inverse vulcanization for the removal of ferric ions (Fe^{3+}) in wastewater. In the current work, WCO has been characterized using GC-MS, FTIR, and TGA to assess the feasibility to be used as a crosslinker in inverse vulcanization. The produced polysulfides were analyzed based on the different reaction parameters including sulfur to WCO crosslinking ratio, temperature, time, and the application of NaCl by FTIR, TGA, XRD, SEM-EDX, and BET. The adsorption process was studied by varying pH, initial Fe^{3+} concentration, and dosage with suitable isothermal and kinetic studies. The required functional bonds and unsaturation of WCO were confirmed by FTIR and GC-MS, and the stability at higher temperatures was confirmed using TGA. Polysulfides were synthesized under stirring (500 rpm) of WCO with elemental sulfur at three different temperatures (195°C, 190°C, and 185°C) with three crosslinking ratios (70, 60, and 50 wt% sulfur). Two different sets of reaction time; 45 min and 60 min have been used for polysulfide synthesis. The physicochemical properties of the produced polysulfides were determined and the thermal stability was analyzed. The FTIR spectra including the breakdown of C=C and formation of C-S bond confirmed the change of functional groups between WCO and produced polymer. The effect of saturated and unsaturated triglycerides of WCO is clearly visible in SEM micrographs. The polysulfide with a 70 wt % sulfur feed ratio showed better surface area. TGA and DTG showed that better thermal properties and stable polysulfides can be obtained from higher amount of sulfur content. Other optimal conditions for inverse vulcanization were recorded as well, such as 60 min of reaction time and 195°C. The surface was modified using NaCl as porogen to increase surface area. The BET results confirmed the increase of the surface area of the porous polysulfides prepared using NaCl. Removal performance was evaluated by studying the effects of parameters including porosity, pH of the solution, initial Fe^{3+} concentration, and amount of polysulfide dosages. The highest removal was recorded for porous polysulfides. Acidic pH (pH=3) was favorable for Fe^{3+} removal. The optimum dosage was 20 mg/100 mL. The removal isotherm has been studied and fitted best for Freundlich isotherm. The kinetic study of the removal process has been fit well in linear pseudo second order kinetic model. It is concluded that inverse vulcanized polysulfides can be successfully used to reduce Fe^{3+} in wastewater and mitigate the environmental threats of excess WCO.

TABLE OF CONTENT

DECLARATION	
TITLE PAGE	
DEDICATION	i
ACKNOWLEDGEMENTS	ii
ABSTRAK	iii
ABSTRACT	iv
TABLE OF CONTENT	v
LIST OF TABLES	ix
LIST OF FIGURES	x
LIST OF SYMBOLS	xii
LIST OF ABBREVIATIONS	xiii
CHAPTER 1 INTRODUCTION	1
1.1 Research background	1
1.2 Problem statement	2
1.3 Research objectives	3
1.4 Research scopes	4
1.5 Thesis outline	5
CHAPTER 2 LITERATURE REVIEW	6
2.1 Waste Cooking Palm Oil (WCO)	6
2.1.1 Physicochemical Composition	7
2.1.2 Uses of WCO	9

2.2	Petroleum Refinery Wastewater	10
2.2.1	Properties of Petroleum Refinery Wastewater	11
2.2.2	Removal methods of heavy metals in wastewater	11
2.2.3	Utilization of various adsorbents in heavy metal wastewater removal	13
2.3	Polysulfides	17
2.3.1	Properties of polysulfides	18
2.3.2	Polysulfide synthesis	19
2.4	Inverse vulcanization	20
2.4.1	Use of cooking oils in inverse vulcanization	22
2.4.2	Applications of inverse vulcanized polysulfides from cooking oil	25
2.5	Inverse vulcanized polysulfide adsorbents in heavy metal removal	26
2.6	Iron (Fe) and its chemical properties	31
2.6.1	Iron toxicity mechanism	32
2.7	Adsorption study	32
2.7.1	Parameters affecting adsorption using inverse vulcanized polysulfides	33
2.7.2	Adsorption Isotherm	38
2.7.3	Adsorption Kinetics	39
2.8	Implication of literature review	39
CHAPTER 3 METHODOLOGY		41
3.1	Introduction	41
3.2	Research framework	41
3.3	Materials	42
3.4	Characterization of oil samples	43
3.4.1	Gas chromatography–mass spectrometry (GC-MS)	44

3.4.2	Fourier transform infrared spectroscopy (FTIR)	44
3.4.3	Thermogravimetric analysis (TGA)	45
3.5	Preparation of polysulfide adsorbent	45
3.5.1	Non-porous polysulfide preparation	45
3.5.2	Porous polysulfide preparation	46
3.6	Characterization of polysulfide adsorbent	47
3.6.1	Scanning electron microscopy with X-ray Microanalysis (SEM-EDX)	47
3.6.2	Brunauer-Emmett-Teller surface area (BET)	48
3.6.3	X-Ray diffraction (XRD)	48
3.7	Preparation of Fe ³⁺ solution	49
3.8	Adsorption study	49
3.8.1	Application of polysulfide adsorbent	49
3.8.2	Atomic absorption spectrometry (AAS)	50
3.9	Isothermal and kinetic modelling	50
CHAPTER 4 RESULTS AND DISCUSSION		52
4.1	Introduction	52
4.2	Characterization of FCO and WCO	52
4.3	Effect of synthesis parameters	57
4.3.1	Effect of sulfur to WCO ratio	57
4.3.2	Effect of temperature	62
4.3.3	Effect of reaction time	65
4.3.4	Effect of NaCl as porogen	66
4.4	Application of synthesized polysulfides in the adsorption of Fe ³⁺	68
4.4.1	Equilibrium time determination and effect of porosity	68

4.4.2	Effect of pH	70
4.4.3	Effect of initial Fe ³⁺ concentration	72
4.4.4	Effect of dosage	73
4.5	Post adsorption analysis	75
4.5.1	Analysis of polysulfide after Fe ³⁺ adsorption	75
4.5.2	Adsorption isotherm	77
4.5.3	Kinetic study	79
CHAPTER 5 CONCLUSION		82
5.1	Conclusion	82
5.2	Future recommendation	83
REFERENCES		85
APPENDIX A AAS Calibration Curve		106
APPENDIX B Adsorption data for freundlich isotherm		107
APPENDIX C List of publication		108

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