SYNTHESIS AND CHARACTERIZATION OF ZEOLITE FROM PALM OIL FUEL ASH (POFA) BY ULTRASONIC ASSISTED HYDROTHERMAL APPROACH

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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 Engineering (Chemical).

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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 institutions.

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TABLE OF CONTENT

DECLARATION

TITLE PAGE

ACKNOWLEDGEMENTS **ii**

ABSTRAK **iii**

ABSTRACT **iv**

TABLE OF CONTENT **v**

LIST OF TABLES **ix**

LIST OF FIGURES **xi**

LIST OF SYMBOLS **xiv**

LIST OF ABBREVIATIONS **xv**

CHAPTER 1 INTRODUCTION **1**

1.1 Research Background 1

1.2 Problem Statement 3

1.3 Objectives of the Research 4

1.4 Scope of the Study 4

1.5 Significance of the Study 5

1.6 Overview of the Thesis 6

CHAPTER 2 LITERATURE REVIEW **7**

2.1 Introduction 7

2.2 Sources and Utilization of POFA 7

2.3 Physical Properties of POFA 9

2.4 Chemical Features of POFA 10

2.5 Zeolites: Historical background 11

2.6 Properties of Zeolites 13

2.6.1 Ratio of Silica and Alumina in Zeolite 15

2.7 Natural Zeolites 17

2.8 Synthetic Zeolites 17

2.9 Zeolite from Fly Ash 18

2.10 Zeolite from Agro-wastes 19

2.11 Synthesis of Zeolites 19

2.11.1 Effects of Parameters on Zeolitization 20

2.11.2 Mechanism of Zeolite Formation 22

2.11.3 Hydrothermal Synthesis (Conventional) 25

2.11.4 Fusion with Hydrothermal Technique 26

2.11.5 Microwave Treatment Method 28

2.11.6 Ultrasound Application in Zeolite Synthesis 29

2.12 Ultrasound Applications in Other Sectors 31

2.13 Zeolite Applications 32

2.14 Design of Experiment 37

2.14.1 Fractional Factorial Designs 38

2.14.2 Central Composite Design (CCD) 38

2.15 Summary 41

CHAPTER 3 RESEARCH METHODOLOGY 42

3.1 Overview 42

3.2 Research Methodology Process Flow Diagram 42

3.1 Chemicals and Material 44

3.2 Preparation of Palm Oil Fuel Ash (POFA) 44
3.3 Characterization of POFA 44
3.4 Calcination of Raw POFA 45
3.5 Sieving Process 45
3.6 Mixing Process 45
3.7 Hydrothermal Process with Ultrasound Effect 49
3.8 Aging Process 50
3.9 Washing and Drying 50
3.10 Characterization of POFA and Zeolite 51
3.10.1 X-Ray Fluorescence (XRF) 51
3.10.2 X-Ray Diffraction (XRD) 51
3.10.3 Scanning Electron Microscopy (SEM) 52
3.10.4 Fourier Transform Infrared Spectroscopy (FTIR) 52
3.10.5 BET Surface Area 52
3.11 Design of Experiment-Factorial Design 52
3.12 Design of Experiment-Central Composite Design 54

CHAPTER 4 RESULTS AND DISCUSSION 56

4.1 Introduction 56
4.2 Characteristics of Raw Palm Oil Fuel Ash (POFA) 56
4.2.1 Physical Characteristics 56
4.2.2 Chemical Characteristics 57
4.3 Effect of Different KOH/POFA Ratios on Zeolite Formation 63
4.4 Effect of Ultrasound Water Bath Temperature 69
4.5 Effect of Ultrasound Exposure Time on Zeolite Formation 74
4.6 Effect of Ultrasound Power on Zeolite Formation 81

4.7 Effect of KOH Concentration on Zeolite Formation 86

4.8 Design of Experiment: Factorial Analysis 91

4.9 Design of Experiment: Optimization Using Central Composite Design (CCD) 102

CHAPTER 5 CONCLUSION 113

5.1 Conclusion 113

5.2 Future Works 114

REFERENCES 116

APPENDIX A X-RAY FLUORESCENCE ANALYSIS FOR SILICA AND ALUMINA 126

APPENDIX B LIST OF PUBLICATIONS AND AWARDS 130

LIST OF TABLES

Table 2.1	Chronological history of zeolite	11
Table 2.2	Ratio of Si/Al ratio for zeolite	16
Table 2.3	Silica and alumina ratio of zeolite	16
Table 2.4	Pore size of zeolites	18
Table 2.5	Types of zeolite with their applications	34
Table 3.1	List of chemicals and material	44
Table 3.2	List of equipment for zeolite synthesis research	44
Table 3.3	Parameters for zeolite synthesis using KOH/POFA ratios from 1:1 to 5:1	47
Table 3.4	Parameters for zeolite synthesis using KOH/POFA Ratios 1:1 within 40 °C to 80 °C	47
Table 3.5	Parameters for zeolite synthesis using KOH/POFA ratios 2:1 within 40 °C to 80 °C	47
Table 3.6	Parameters for zeolite synthesis using KOH/POFA ratios 1:1 within 30 minutes to 150 minutes ultrasound exposure time	47
Table 3.7	Parameters for zeolite synthesis using KOH/POFA ratios 2:1 within 30 minutes to 150 minutes	48
Table 3.8	Parameters for zeolite synthesis using KOH/POFA Ratios 1:1 within 400 W to 800 W	48
Table 3.9	Parameters for zeolite synthesis using KOH/POFA ratios 2:1 within 400 W to 800 W	48
Table 3.10	Parameters for zeolite synthesis using KOH/POFA ratios 1:1 within 1 M to 5 M	48
Table 3.11	Parameters for zeolite synthesis using KOH/POFA ratios 2:1 within 1 M to 5 M	49
Table 3.12	Range of parameter for fractional factorial design	53
Table 3.13	Fractional factorial design list of experiments by standard order	53
Table 3.14	Central composite design experimental layout by standard order	54
Table 4.1	Comparison of POFA surface area between past and present studies	57

Table 4.2	Chemical compositions of POFA in past and present studies	58
Table 4.3	Silica and alumina composition in researches	59
Table 4.4	Phases of raw POFA	61
Table 4.5	FTIR range of raw POFA	63
Table 4.6	Phases of zeolite at KOH/POFA ratio from 1:1 to 5:1	65
Table 4.7	Characteristic peaks for types of zeolite	67
Table 4.8	Phases of zeolite within temperature range of 40-80 ^{o}C at KOH/POFA ratio of 1:1	69
Table 4.9	Phases of zeolite at 40-80 ^{o}C at KOH/POFA ratio of 2:1	72
Table 4.10	Phases of zeolite at KOH/POFA ratio 1:1 within 30-150 minutes of exposure time	75
Table 4.11	Phases of zeolites at KOH/POFA ratio 2:1 within 30-150 minutes	78
Table 4.12	Phases of zeolite at KOH/POFA ratio 1:1 for 400-800 W	82
Table 4.13	Phases of zeolites at KOH/POFA ratio 2:1 within 400-800 W	84
Table 4.14	Phases of zeolite at KOH/POFA ratio 1:1 within 1-5 M concentration level	87
Table 4.15	Phases of zeolite at KOH/POFA ratio 2:1 for 1-5 M range of concentration	90
Table 4.16	ANOVA for half factorial design	94
Table 4.17	Summary statistics model	94
Table 4.18	Model parameters and regression analysis	95
Table 4.19	The best condition from half factorial design	102
Table 4.20	ANOVA results for changes in silica per alumina ratio	103
Table 4.21	Percent error from actual experiment	106
Table 4.22	Phases of zeolite at optimum condition	106
Table 4.23	Characteristic peaks for optimum condition zeolite	111
Table 4.24	Textural properties of POFA and zeolites	112
Table 4.25	Pore diameter classification by IUPAC	112

LIST OF FIGURES

Figure 2.1	Palm oil residues	8
Figure 2.2	Palm oil fuel ash	9
Figure 2.3	SEM image of raw POFA at magnification 500	10
Figure 2.4	SEM images of POFA at magnification (a) 200x (b) 1000x	10
Figure 2.5	Ideal structure of zeolite framework	14
Figure 2.6	Mechanism of fly ash zeolite by batch hydrothermal	23
Figure 2.7	Radicals for aluminosilicate formation	24
Figure 2.8	Mechanism of zeolite formation by sonochemical method	24
Figure 3.1	Research methodology process flow diagram	43
Figure 3.2	Summary of methodology steps	43
Figure 3.3	Screen of calcined POFA using Retch sieve shaker	46
Figure 3.4	Materials for mixing process	46
Figure 3.5	Fusion using hydrothermal and ultrasonic treatment	49
Figure 3.6	Aging with stirring by orbital shaker	50
Figure 3.7	Grinding process of dried products using mortar and pestle	51
Figure 4.1	SEM microstructures of raw POFA before treatment at 1000x magnification	57
Figure 4.2	Diffractogram of raw POFA	60
Figure 4.3	XRD patterns of POFA with SiO2 peaks	61
Figure 4.4	XRD patterns of fly ash	62
Figure 4.5	FTIR transmission spectrum of POFA	62
Figure 4.6	XRD patterns of using KOH/POFA ratios from 1:1 to 5:1	65
Figure 4.7	Variations SiO_2/Al_2O_3 ratio of different KOH/POFA ratio	66
Figure 4.8	FTIR spectra of different KOH/POFA ratios from 1:1 to 5:1	68
Figure 4.9	XRD patterns using 40-80 ^oC at KOH/POFA ratio 1:1	70
Figure 4.10	SiO2/Al2O3 ratio at KOH/POFA ratio of 1:1 at different temperatures	71
Figure 4.11	XRD patterns using 40-80 ^oC at KOH/POFA ratio 2:1	73
Figure 4.12	SiO_2/Al_2O_3 ratio at KOH/POFA ratio 2:1 with different temperature	74

Figure 4.13	Figure 4.13 XRD pattern at KOH/POFA ratio 1:1 within 30-150 minutes of exposure time	76
Figure 4.14	SiO_2/Al_2O_3 ratio at KOH/POFA ratio 1:1 with different exposure times	77
Figure 4.15	XRD pattern at KOH/POFA ratio 2:1 within 30-150 minutes of exposure time	79
Figure 4.16	SiO_2/Al_2O_3 ratio at KOH/POFA ratio 2:1 with different ultrasonic exposure time	80
Figure 4.17	XRD pattern using 400-800 Watt ultrasound power at KOH/POFA Ratio 1:1	81
Figure 4.18	SiO_2/Al_2O_3 ratio at KOH/POFA ratio 1:1 with different ultrasound power	83
Figure 4.19	XRD pattern using 400-800 Watt ultrasound power at KOH/POFA ratio 2:1	84
Figure 4.20	SiO_2/Al_2O_3 ratio at KOH/POFA ratio 2:1 with different ultrasound power	85
Figure 4.21	XRD patterns using KOH concentration level range of 1-5 M at KOH/POFA ratio 1:1	86
Figure 4.22	SiO_2/Al_2O_3 ratio at KOH/POFA ratio 1:1 with different KOH concentration	88
Figure 4.23	XRD pattern using 1-5 M KOH concentration at KOH/POFA ratio 2:1	89
Figure 4.24	SiO_2/Al_2O_3 ratio at KOH/POFA ratio 2:1 with different KOH concentration levels	91
Figure 4.25	Percent contribution calculated from Design Expert	92
Figure 4.26	Pareto chart	93
Figure 4.27	Normal probability plot	96
Figure 4.28	Interaction between KOH/POFA ratio and ultrasonic irradiation power	97
Figure 4.29	Interaction between KOH/POFA ratio and KOH concentration	98
Figure 4.30	Interaction between ultrasonic water bath temperature and ultrasonic irradiation power	99

Figure 4.31	Effect of ultrasonic exposure time on silica per alumina ratio	100
Figure 4.32	Normal probability plot of residual	101
Figure 4.33	Comparison between experimental and predicted value using eq. 4.1	101
Figure 4.34	Three-dimensional response surface (Ultrasonic exposure time and KOH/POFA ratio relationship)	104
Figure 4.35	Three-dimensional response surface (KOH concentration and KOH/POFA ratio relationship)	105
Figure 4.36	Three-dimensional response surface (KOH concentration and ultrasonic exposure time relationship)	105
Figure 4.37	XRD image of optimum condition	107
Figure 4.38	XRD image by using ultrasound irradiation	107
Figure 4.39	XRD image of reference zeolite P	108
Figure 4.40	XRD image of zeolite A	108
Figure 4.41	XRD image of zeolite X	109
Figure 4.42	SEM microstructures of POFA-based zeolite at optimum condition at 1000x magnification	110

LIST OF SYMBOLS

°C	Degree Celcius
Hz	Hertz
L	Litre
M	Molarity
W	Watt

LIST OF ABBREVIATIONS

ASTM	American Society for Testing and Materials
BET	Brunauer-Emmet-Teller Surface Area
CCD	Central Composite Design
CEC	Cation Exchange Capacity
EFB	Empty Fruit Bunches
FA	Coal Fly Ash
FD	Factorial Design
FTIR	Fourier Transformed Infrared Spectroscopy
IUPAC	International Union of Pure and Applied Chemistry
POFA	Palm Oil Fuel Ash
RHA	Rice husk ash
RSM	Response Surface Methodology
SCBA	Sugarcane Bagasse Ash
SEM	Scanning Electron Microscopy
SME	Small Medium Enterprise
XRD	X-Ray Diffraction
XRF	X-Ray Fluorescence

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ABSTRAK

Abu bahan api minyak sawit atau *palm oil fuel ash* (POFA) ialah sejenis bahan buangan daripada kilang kelapa sawit. Ia mempunyai komponen yang hampir serupa dengan komponen abu terbang dan bahan buangan pertanian seperti abu sekam padi dan abu hampas tebu iaitu silika dan alumina. Kehadiran dua sebatian tersebut mendorong kepada sintesis satu bahan yang sangat berguna, yakni, zeolit. Penyelidikan ini bertumpu pada penyiasatan terhadap sintesis zeolit daripada POFA menggunakan kaedah hidrotermal dan kaedah ultrasonik. Kajian ini telah dijalankan dalam pelbagai keadaan menggunakan beberapa nisbah KOH: POFA (1:1- 5:1), suhu rendaman air ultrasonik (40-80 °C), tempoh pendedahan ultrasonik (30-150 minutes), tahap kuasa ultrasonik (400-800 W) dan tahap kepekatan KOH (1-5 M). Secara umumnya, teknik hidrotermal dan teknik pelakuran telah digunakan secara meluas dalam penghasilan zeolit. Namun, kedua-duanya memerlukan penggunaan tenaga yang tinggi lalu menjadikan kedua-duanya kurang menjimatkan dari sudut ekonomi dan mengambil masa sehingga beberapa hari untuk menghasilkan zeolit. Kesan lima jenis parameter terhadap zeolit yang terhasil telah dikaji bagi mendapatkan keadaan yang terbaik untuk menghasilkan kaedah sintesis yang ringkas, pantas, tidak mencemarkan alam sekitar dan mesra alam semasa dan selepas eksperimen dijalankan. Ciri-ciri POFA mentah dan zeolit yang terhasil ditentukan oleh XRF, XRD, luas permukaan BET, FTIR dan SEM. Seterusnya, kajian untuk menentukan keadaan optimum zeolit telah dilakukan yang menggabungkan reka bentuk faktoran separa dan reka bentuk komposit pertengahan. Hasil kajian menunjukan bahawa keadaan optimum untuk penghasilan zeolit daripada POFA telah ditemui pada nisbah KOH: POFA (2:1), suhu ultrasonik rendaman air 80 °C, tempoh pendedahan ultrasonik 150 minit, kuasa ultrasonik 600 W dan kepekatan KOH 1 M. Zeolit yang terhasil secara optimum mempunyai nisbah silika per alumina yang memenuhi julat standard bagi nisbah silika per alumina untuk zeolit (2-10). Berdasarkan eksperimen yang menggunakan keadaan optimum, keputusan XRD menunjukkan fasa zeolit yang terhasil sepadan dengan zeolit A, P dan X. Spektra FTIR menunjukkan kehadiran kumpulan fungsi yang kebiasaannya ada pada zeolit. Nisbah silika per alumina untuk keadaan optimum menunjukkan sifat penjerap yang hidrofobia. Seperti yang ditunjukkan oleh SEM, permukaan zeolit adalah sebanding dengan permukaan zeolit A, P dan X. Kajian-kajian sebelum ini membuktikan bahawa zeolit A, P dan X telah biasa digunakan sebagai penjerap. Oleh itu, dapatlah dikatakan bahawa kajian ini telah berjaya menemui keadaan sintesis optimum bagi menghasilkan zeolit dengan nisbah silika per alumina yang tinggi dengan sifat penjerap. Untuk meningkatkan kualiti zeolit, pemisahan zeolit mentah serta pembinaan kaedah dan parameter proses lain perlulah diberi perhatian.

ABSTRACT

Palm oil fuel ash (POFA), is a type of waste from palm oil mill. Its components are almost similar as those of fly ash and agro-wastes such as rice husk ash, and sugarcane bagasse ash, which is, silica and alumina. The presence of both compounds has led to the synthesis of zeolite, which is a highly useful material. Abundance of POFA in palm oil mill, lack of its application and lead to serious environmental problem became concern to minimize POFA. This research concentrated on the investigation of the synthesis of zeolites from POFA by using ultrasonic assisted hydrothermal method. The present study was conducted in various conditions using several KOH: POFA ratios (1:1- 5:1), ultrasound water bath temperatures (40-80 °C), ultrasound exposure times (30-150 minutes), ultrasound power levels (400-800 W) and KOH concentration levels (1-5 M). Commonly, hydrothermal and fusion techniques have been widely used in the production of zeolite. However, these techniques would require high energy consumption, making them less economical and time-consuming because zeolite production may take several days. The effect of the five parameters were investigated on the synthesized zeolites in order to achieve best conditions for pollution-free and environmenal friendly synthesis during and after the experimental process. Characterization of raw POFA and synthesized zeolites were determined by X-Ray Fluorescence (XRF), X-Ray Diffraction (XRD), BET surface area, Fourier Transform Infrared Spectroscopy (FTIR) and Scanning Electron Microscopy (SEM). This was followed by the optimisation study of zeolites in combination with half factorial design and central composite design. Results showed that the optimum conditions for zeolite synthesis from POFA were KOH: POFA ratio of 2:1, ultrasonic water bath temperature at 80 °C, ultrasonic exposure time of 150 minutes and KOH concentration level at 1 M which eventually produced zeolites with high silica per alumina ratio (10.60) that fulfilled zeolite standard range of silica per alumina (2-10). Based on the optimum conditions, XRD results indicated the phases of zeolite synthesized matched those of zeolites A, P and X. FTIR spectra detected the presence of functional groups that were common for zeolites. Silica per alumina ratio for the optimum conditions which is 10.60, indicated the hydrophobic adsorbent property of the synthesized zeolite. As shown by SEM, the surface was comparable to that of zeolites A, P and X. It has been evident from past studies that zeolite A, P and X have been commonly used as adsorbent. Thus, it can be said that the present study has successfully achieved the optimum synthesis condition that would be able to produce high silica per alumina zeolite with adsorbent property. To improve the quality of zeolites, separation of crude zeolites as well as development of other methods and process parameters should be given consideration.

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