

**SYNTHESIS, CHARACTERIZATION AND  
CATALYTIC ACTIVITY OF SUPPORTED SUGAR  
CATALYSTS**

**NURLIYANA NASUHA BINTI SAFIE**



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UNIVERSITI MALAYSIA SABAH

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**THIS IS SUBMITTED IN FULFILLMENT OF THE  
REQUIREMENTS FOR THE DEGREE OF  
MASTER OF ENGINEERING**

**FACULTY OF ENGINEERING  
UNIVERSITI MALAYSIA SABAH  
2017**

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IJAZAH: **MASTER OF ENGINEERING (CHEMICAL ENGINEERING)**

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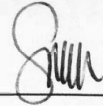
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# DECLARATION

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## ACKNOWLEDGEMENT

First of all, I want to express my highest gratitude to The Almighty Allah s.w.t that without rezq He had given to me, I would not be able to complete this thesis. Second, to my supervisor Dr Jidon Adrian Janaun, for all the guidance, tolerance when my works were not as expected and whenever I missed the weekly meetings. Thank you for all the constructive comments on my works which I believe, you might waste a lot of precious time reading it. During my journey too, I never shortage of money or any other necessities because I was financially supported by Dr.Jidon and Dr. Nancy. Thank you to both of you for being concern, supportive and understanding supervisors. Next, I dedicated my appreciation to my parents, Mr Safie and Mrs Fodhilah, as well as my brothers and sisters that supported me morally and financially throughout my studies. To my husband Mohd Izuan Bin Mahmuda for being there all the time and to all your supports too, you are my strength and the reason I hold on. To my research mates, Poyo, Ola, Valey, Veron, and Winnie, you guys have made my journey so much fun and resourceful I could not forget our memories and moments together. To my beloved friends, Monjia Belleza Mojulat Cosmas, who helped me a lot, day and night, I pray for smoothness of your studies and happiness. Thank you everyone, I could not have done this without all of you, thank you.



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## ABSTRACT

The process of synthesizing sugar catalyst is started by incomplete carbonization of D-glucose. It is followed by sulphonation to incorporate the sulphonic group ( $-\text{SO}_3\text{H}$ ). However, despite of its excellence catalytic performance, the current form of sugar catalyst is in powder form with non-porous structure maybe lack of applicability in commercial scale biodiesel production due to the potential of large pressure drop in a packed bed reactor. Hence, it is anticipated that the industrial applicability of sugar catalyst can be enhanced if its particle size can be increased. This study was conducted to investigate the feasibility of attaching sugar catalyst on different surfaces by depositing the char on external surfaces of aluminum (SCDCAI), glass (SCDCSi) and clay (SCDCCI) via dip coating method (*ex-situ*) followed by sulphonation. The sulfonation of char embedded naturally in different types of supports namely, rice husk, rice straw, and rice husk were also studied (*in-situ*). Rice husk and rice straw were pyrolyzed in a controlled condition, whereas rice husk ash, a combustion waste, was acquired from a local paddy mill. Synthesized supported sugar catalysts via *ex-situ* were sulphonated using fuming sulphuric acid via vapor-phase sulphonation and supported catalysts via *in-situ* were sulphonated via liquid phase sulphonation. The ratio of char deposited on SCDCAI, SCDCSi and SCDCCI were  $0.900 \text{ g}_c/\text{g}_s$ ,  $0.040 \text{ g}_c/\text{g}_s$ , and  $0.014 \text{ g}_c/\text{g}_s$ , respectively. The total acidity for SCDCAI, SCDCSi, and SCDCCI were  $0.9 \text{ mmol/g}$ ,  $0.2 \text{ mmol/g}$ , and  $0.4 \text{ mmol/g}$ , respectively. SCDCAI and SCDCGI had surface area of  $3.38 \text{ m}^2/\text{g}$  and  $1.2 \text{ m}^2/\text{g}$ , respectively compared to powder sugar catalyst which has less than  $<1 \text{ m}^2/\text{g}$ . The catalysts were stable up to  $673.15 \text{ K}$ . Meanwhile, the total acidity of *in-situ* sulfonation of rice straw (RSC), rice husk (RHC) and rice husk ash (RHAC) were  $3.60 \text{ mmol/g}$ ,  $3.53 \text{ mmol/g}$  and  $2.28 \text{ mmol/g}$ , respectively. FTIR data indicated the presence of silica bonded to carbon in the samples. The supported catalysts via *in-situ* undergone two stages of weight loss from  $T = 303.15 \text{ K}$  to  $673.15 \text{ K}$ . The catalytic activity of SCDCAI was tested in a packed bed reactor and conversion of oleic acid was below than 1%. The catalytic activity of RSC, RHC and RHAC on esterification of oleic acid with methanol were studied in a batch reactor at  $333.15 \text{ K}$ ,  $343.15 \text{ K}$ , and  $353.15 \text{ K}$  under reflux, molar ratio (oleic acid: methanol) of 1:10, 3 wt.% catalyst. The conversion of oleic acid using RHC and RHAC were linearly proportional to temperature. RSC had the highest oleic acid conversion compared to RHC and RHAC.  $E_a$  for esterification reaction catalyzed by RSC, RHC and RHAC were  $25.952 \text{ KJ.mol}^{-1}$ ,  $38.760 \text{ KJ.mol}^{-1}$  and  $10.842 \text{ KJ.mol}^{-1}$  respectively. The kinetic model was studied using the integral method. Based on the regression of best fit line, RSC, RHC and RHAC followed second order reaction. In conclusion, sugar catalyst can barely be deposited externally on aluminum, clay and glass via *ex-situ* process with weak adhesion strength. Meanwhile, the sulfonated carbon-silica catalyst prepared via *in-situ* had high total acidity that likely due to the presence of carbon-silica bond. It had shown high catalytic activity on esterification of oleic acid with methanol.

## ABSTRAK

### **SINTESIS, PENCIRIAN DAN AKTIVITI PEMANGKINAN SUGAR CATALYST YANG DISOKONG**

Proses penghasilan sugar catalyst dimulakan dengan proses pirolisis. Diikuti dengan sulfonasi bagi penggabungan dengan kumpulan asid sulfonik ( $\text{SO}_3\text{H}$ ). Bentuk sugar catalyst dalam serbuk adalah tidak porous dan kurang sesuai untuk dikormesialkan kerana potensi untuk menghasilkan penurunan tekanan di dalam *packed bed reactor*. Hal ini disebabkan oleh *packed bed* yang dimampatkan dengan pemangkin yang tidak porous dan luas permukaan yang rendah memerlukan lebih tenaga untuk mengatasi penurunan tekanan tersebut. Penurunan tekanan boleh mempengaruhi penghasilan biodiesel untuk tindakbalas dalam bentuk gas. Penyesuaian penggunaan sugar catalyst di dalam skala pengindustrian boleh dilakukan dengan pembesaran saiz partikel sugar catalyst. Kajian ini telah dijalankan untuk mengkaji kebolehan melekatkan sugar catalyst pada permukaan sokongan seperti aluminium (SCDCAI), gelas (SCDCGI) dan tanah liat (SCDCCI) secara berasingan melalui kaedah *dip-coating (ex-situ)* atau campuran sokongan secara semulajadi (*in-situ*) diikuti dengan proses sulfonasi untuk menghasilkan pemangkin yang disokong bersifat asid. Sekam padi, jerami padi dan abu sekam padi digunakan sebagai sampel untuk penggabungan pemangkin sugar catalyst dengan viethan melalui *in-situ proses*. Nisbah karbon yang terdapat pada SCDCAI, SCDCGI dan SCDCCI adalah 0.9 g<sub>c</sub>/g<sub>s</sub>, 0.040 g<sub>c</sub>/g<sub>s</sub>, dan 0.014 g<sub>c</sub>/g<sub>s</sub>. Jumlah keasidan SCDCAI, SCDCGI dan SCDCCI adalah 0.9 mmol/g, 0.2 mmol/g, dan 0.4 mmol/g. SCDCAI and SCDCGI mempunyai luas permukaan 3.38 m<sup>2</sup>/g and 1.2 m<sup>2</sup>/g berbanding dengan sugar catalyst bentuk serbuk yang mempunyai luas permukaan kurang dari 1 m<sup>2</sup>/g. Kajian selanjutnya yang dijalankan menunjukkan SCDCAI, SCDCGI dan SCDCCI viethan terhakis akibat daripada impak mekanikal semasa digunakan untuk ujian pengasilan viethanolvi. Jumlah keasidan sulfonasi *in-situ* jerami padi (RSC), sekam padi (RHC) dan abu sekam padi (RHAC) adalah 3.60 mmol/g, 3.53 mmol/g dan 2.28 mmol/g. Data dari FTIR juga menunjukkan kewujudan hubungan viethan-karbon dalam RSC, RHC dan RHAC. Pemangkin yang disokong melalui *in-situ* melalui dua peringkat kehilangan berat dari suhu 303.15 K hingga 673.15 K. Aktiviti pemangkin disokong y dihasilkan melalui *ex-situ* atas aluminium (SCDCAI) telah diuji dalam reaktor *packed bed* dan menghasilkan konversi asid oleik kurang dari 1%. Manakala RSC, RHC dan RHAC telah diuji dalam reaktor *batch* pada tiga suhu berbeza iaitu 333.15 K, 343.15 K, and 353.15 K di bawah refluks, nisbah ratio asid oleik : methanol (1:10) dan 3.wt % berat pemangkin. Tindakbalas esterifikasi menggunakan RSC adalah yang tertinggi berbanding RHC dan RHAC. Model ethanol ditentukan menggunakan *integral method*. Activation energy,  $E_a$  untuk reaksi esterifikasi menggunakan RSC, RHC dan RHAC adalah 25.952 Kj.mol<sup>-1</sup>, 38.760 Kj.mol<sup>-1</sup> and 10.842 Kj.mol<sup>-1</sup>. Reaksi esterifikasi menggunakan RSC, RHC dan RHAC mengikut *order* 2. Secara keseluruhannya, sugar catalyst yang dihasilkan melalui proses *ex-situ* menunjukkan lekatan yang lemah pada permukaan sokongan aluminium, gelas dan tanah liat. Manakala pemangkin karbon-silika yang dihasilkan melalui *in-situ* mempunyai jumlah keasidan tinggi yang berkemungkinan disebabkan kewujudan karbon-silika dan telah menunjukkan fungsi pemangkinan yang lebih tinggi dalam tindak balas pengeksterifikasi asid oleik dan ethanol.

# LIST OF CONTENT

	Page
<b>TITLE</b>	i
<b>DECLARATION</b>	ii
<b>CERTIFICATION</b>	iii
<b>ACKNOWLEDGEMENT</b>	iv
<b>ABSTRACT</b>	v
<b><i>ABSTRAK</i></b>	vi
<b>LIST OF CONTENTS</b>	vii
<b>LIST OF FIGURES</b>	xiv
<b>LIST OF TABLES</b>	xvii
<b>LIST OF SYMBOLS</b>	xix
<b>LIST OF ABBREVIATIONS</b>	xx
<b>LIST OF APPENDICES</b>	xxii
<b>CHAPTER 1 : INTRODUCTION</b>	1
1.1 Research Background	1
1.2 Research Problem	2
1.3 Research Topic	2



1.4	Novelty	3
1.5	Research Objective	3
1.6	Hypothesis	3
1.7	Research Scope	3
CHAPTER 2 : LITERATURE REVIEW		5
2.1	Overview of Biodiesel Production	5
2.2	Types of catalysts	7
2.2.1	Homogeneous Catalysts	7
2.2.2	Heterogeneous Catalysts	10
2.2.3	Solid Acid Catalysts	10
2.2.4	Sulphonated Carbon Based Catalysts	11
2.2.5	Mechanism of Solid Acid Catalyst	13
2.3	Limitation on Powder Size of Sugar Catalyst and Recent Advancement	14
2.4	Method for Synthesizing <i>Ex-situ</i> Supported Sugar Catalysts	15
2.4.1	Melting Method	17
2.4.2	Polymerization Method	17
2.4.3	Chemical Vapor Deposition (CVD) Method	17

2.4.4	Suspension Method	18
2.4.5	Dip-coating Method	18
2.5	Method of Synthesizing <i>In-situ</i> Supported Sugar Catalysts	19
2.5.1	Rice Plants as Precursor for <i>in-situ</i> Supported Carbon Based Catalysts	19
2.6	Potential Supports	22
2.7	Support Pre-treatment	24
2.8	Test to Determine Strength of Coating	25
2.9	Interaction between Catalyst and Support	25
2.10	Synthesis of Biodiesel in Batch Reactor and Packed Bed Reactor	28
2.10.1	Effect of Molar Ratio of Reactants	29
2.10.2	Effect of Temperature	30
2.10.3	Effect of Amount of Catalyst	30
2.10.4	Effect of Feed Flow Rate in Packed Bed Reactor	31
2.11	Methods to Re-Use Catalyst in Biodiesel Production	31
2.12	Summary	32
CHAPTER 3 : METHODOLOGY		34
3.1	Introduction	34

3.2	Preparation of <i>ex-situ</i> Supported Sugar Catalysts	35
3.3	Preparation of <i>in-situ</i> Supported Sugar Catalysts	35
3.4	Functionalization of Char	36
3.4.1	Functionalization of <i>ex-situ</i> Supported Char and <i>in-situ</i> Supported Chars	37
3.5	Characterization of Supported Sugar Catalysts	39
3.5.1	Total Acidity	39
3.5.2	Porosity	41
3.5.3	Functional Groups	42
3.5.4	Surface Morphological Structure and Elements Composition	42
3.5.5	Structure of Carbon	44
3.5.6	Thermal Stability	44
3.6	Esterification of Oleic Acid with Methanol using Supported Sugar Catalysts	46
3.6.1	Esterification of Oleic Acid with Methanol Using <i>ex-situ</i> Supported Carbon Catalysts in Packed Bed Reactor	46
3.6.2	Esterification of Oleic Acid with Methanol using <i>in-situ</i> Supported Sugar Catalysts on Silica in Batch Reactor	47

3.6.3	Product Analysis	48
3.6.4	Operating Manual of Gas Chromatography (GC) Model 7890B	48
3.6.5	Reusability of Catalysts	49
3.6.6	Reproducibility of Method	49
CHAPTER 4 : RESULTS AND DISCUSSION		51
4.1	Preparation and Characterization of <i>ex-situ</i> Supported Sugar Catalysts by Dip Coating Method	51
4.1.1	Porosity and Total Carbon Char	53
4.1.2	Total Acidity	55
4.1.3	Functional Groups	56
4.1.4	Surface Morphological Structure and Element Composition	58
4.1.5	Structure of Carbon	60
4.1.6	Thermal Stability	61
4.2	Preparation and Characterization of <i>in-situ</i> Supported Sugar Catalysts	64
4.2.1	Total Acidity	66
4.2.2	Porosity, Elemental Content and Total Acidity	67

4.2.3	Functional Groups	68
4.2.4	Surface Morphological Structure and Element Composition	69
4.2.5	Structure of Carbon	72
4.2.6	Thermal Stability	74
4.3	Catalytic Activity of <i>in-situ</i> and <i>ex-situ</i> Supported Sugar Catalysts on Esterification of Oleic Acid with Methanol in Batch Reactor	75
4.3.1	Catalytic Activity of <i>ex-situ</i> Supported Sugar Catalyst on Esterification of Oleic Acid with Methanol in Packed Bed Reactor	76
4.3.2	Catalytic Activity of <i>in-situ</i> Supported Sugar Catalysts: Effect of Temperature on Conversion of Oleic Acid using RHAC, RHC, and RSC	77
4.3.3	Catalytic Activity of <i>in-situ</i> Supported Sugar Catalysts: Effect of Total Acidity, Structure of Carbon and Porosity on Conversion of Oleic Acid at 333.15 K, 343.15 K, and 353.15 K	80
4.4	Kinetic Analysis	82
4.5	Activation Energy, $E_a$	85

4.6	Reusability	86
CHAPTER 5 : CONCLUSIONS AND RECOMMENDATION		89
5.1	Conclusions	89
5.2	Further recommendation	90
REFERENCES		91
APPENDICES		101



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## LIST OF FIGURES

	Page
Figure 2.1 : Transesterification of triglycerides with alcohol, 'R' is used to designate a generic (Unspecified) alkyl group	5
Figure 2.2 : Transesterification reactions of glycerides with methanol	6
Figure 2.3 : Formation of the active species in transesterification reactions using a base catalyst (NaOH)	8
Figure 2.4 : (a) Base catalyst reactions with FFAs to produce soap and water (b) Water promotes the formation of FFAs. These then can deactivate the catalyst and produce soap as in (a)	8
Figure 2.5 : Reaction mechanism for the transesterification of triglycerides: (1) protonation of the carbonyl group by the acid catalyst; (2) nucleophilic attack of the alcohol, forming a tetrahedral intermediate; (3) proton migration and breakdown of the intermediate. The sequence is repeated twice.	9
Figure 2.6 : Preparation of sulphonated carbon material (A) Pyrolysis, (B) carbonization, and (C) sulfonation	12
Figure 2.7 : Solid acid catalyzed simultaneous esterification and transesterification reaction.	14

Figure 2.8 :	Schematic illustration of Mechanical Adhesion	26
Figure 2.9 :	Schematic illustration of Electrostatic Adhesion	27
Figure 3.1 :	Schematic diagram of synthesis route of supported sugar catalyst on aluminum, glass and clay	35
Figure 3.2 :	Experimental setup for multiple vapor phase sulfonation	38
Figure 3.3 :	Thermo Electron Corporation (Pascal 440 Series) Automated Mercury Porosimetry	41
Figure 3.4 :	A FT-IR Spectrometer (Perkin Elmer)	42
Figure 3.5 :	Surface Electron Microscope S-3400N Hitachi for SEM-EDX Analysis	43
Figure 3.6 :	XRD Philips Xpert Pro	44
Figure 3.7 :	TGA 6 Thermo-gravimetric Analyzer (Perkin Elmer)	45
Figure 3.8 :	Schematic Diagram of Apparatus Setup of Packed Bed Reactor	47
Figure 4.1 :	The transformation from support to supported sugar catalyst on (a) aluminium, (b) glass, and (c) clay	53
Figure 4.2 :	FT-IR bands for a) SCDCAI, b) SCDCGI, and c) SCDCCI	57
Figure 4.3 :	SEM Images (a) SCDCAI (b) SCDCGI and (c) SCDCCI support at magnification 100x (left) and 1000x (right)	59



Figure 4.4 :	XRD pattern of SCDCAI, SCDCGI and SCDCCI	61
Figure 4.5 :	Thermo-gravimetric analysis of SCDCAI, SCDCGI, SCDCCI	62
Figure 4.6 :	Thermo-gravimetric analysis of SCDCCI	63
Figure 4.7 :	Derivative thermal analysis of SCDCAI, SCDCGI and SCDCCI	63
Figure 4.8 :	Derivative thermal analysis of SCDCCI	64
Figure 4.9 :	FTIR bands for RHC, RSC, and RHAC	69
Figure 4.10 :	SEM images of RHAC at magnification of 5.50 k	71
Figure 4.11 :	SEM image of RHC at magnification of 8.50 k	71
Figure 4.12 :	SEM images of RSC at magnification of 1k	72
Figure 4.13 :	XRD pattern of RHAC, RHC and RSC	73
Figure 4.14 :	Thermo-gravimetric analysis (TGA) of RHAC, RHC, and RSC	74
Figure 4.15 :	Derivative thermal analysis of RHAC, RHC, and RSC	75
Figure 4.16 :	Calibration curve of methyl oleate compound	76
Figure 4.17 :	Conversion of oleic acid, X(%) against time (h) for RHAC	79
Figure 4.18 :	Conversion of oleic acid, X(%) against time (h) for RHC	79
Figure 4.19 :	Conversion of oleic acid, X(%) against time (h) for RSC	80
Figure 4.20 :	ln k against 1/T for RHAC, RHC and RSC	85

## LIST OF TABLES

	Page
Table 2.1 : Methods of coating	16
Table 2.2 : Composition SiO <sub>2</sub> and lignin in rice straw	20
Table 2.3 : Physico-chemical characterization of RHC from a local rice mill compared to pyrolyzed RHC	21
Table 2.4: Summary of biochar properties from different pyrolysis temperatures	22
Table 2.5 : Comparison of types of catalyst support	23
Table 4.1 : Surface area and total porosity (%) of support and supported sugar catalyst	53
Table 4.2 : Ratio of char supported on aluminium, glass and clay	54
Table 4.3 : Total acidity of SCDCAI, SCDCGI and SCDCCI	56
Table 4.4 : EDX analysis of <i>ex-situ</i> supported sugar catalysts	59
Table 4.5 : Total char produced after pyrolysis	65
Table 4.6 : Total acidity of RHAC, RHC and RSC	66
Table 4.7 : Characteristics of RHAC, RHC and RSC	67
Table 4.8 : EDX analysis of RHAC, RHC and RSC	71

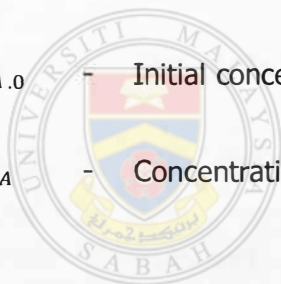
Table 4.9 :	Analysis of Rice Husk Ash	72
Table 4.10 :	Porosity and conversion at 12th hour for RHAC, RHC, and RSC	82
Table 4.11 :	Kinetic Parameters of esterification reaction catalyzed by RHAC	84
Table 4.12 :	Kinetic Parameters of esterification reaction catalyzed by RHC	84
Table 4.13 :	Kinetic Parameters of esterification reaction catalyzed by RSC	84
Table 4.14 :	Activation energy of RHAC, RHC, and RSC	86



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## LIST OF SYMBOLS

- $K$  - Kelvin
- $k$  - Rate constant
- $\alpha$  - Order of reaction
- $A$  - Preexponential factor of frequency factor
- $E$  - Activation energy, J/mol or cal/mol
- $R$  - Gas constant = 8.314 J/mol. K = 1.987 cal/mol.K
- $C_{OA.0}$  - Initial concentration of oleic acid at  $t = 0$
- $C_{oA}$  - Concentration of oleic acid at time,  $t$



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## LIST OF ABBREVIATIONS

FFA	-	Free Fatty Acids
TGs	-	Triglycerides
$C_{oA}$	-	Concentration of Oleic Acid ( $\text{mol.dm}^{-3}$ )
COOH	-	Carboxyl groups
OH	-	Hydroxyl groups
SO <sub>3</sub> H	-	Sulphonic groups
$C_{19}H_{36}O_2$	-	Methyl oleate
$C_{18}H_{34}O_2$	-	Oleic acid
CH <sub>3</sub> OH	-	Methanol
BDL	-	Below Detection Limit
d.a.f	-	dry ash free
g <sub>c</sub> /g <sub>s</sub>	-	gram of char/gram of support
SCDCAI	-	supported sugar catalyst by dip coating method on aluminum
SCDCGI	-	supported sugar catalyst by dip coating method on glass
SCDCCI	-	supported sugar catalyst by dip coating method on clay
RHAC	-	Rice husk ash catalyst

RHC - Rice husk catalyst

RSC - Rice straw catalyst

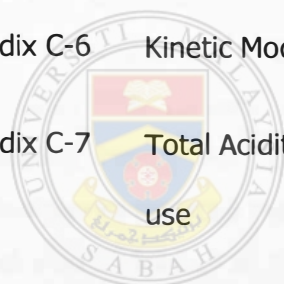


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# LIST OF APPENDICES

		Page
Appendix A	Sample of Calculation	101
Appendix A-1	Sample calculation of Weight Ratio of Catalyst Supported on Aluminum	101
Appendix A-2	Sample Calculation for Standard Solution Preparation	102
Appendix A-3	Sample Calculation Loading of Fuming Sulphuric Acid	103
Appendix A-4	Sample Calculation of Total Acidity	105
Appendix A-5	Sample Calculation of Elemental Composition	106
Appendix A-6	Sample Calculation of Packed Bed Reactor Design	107
Appendix A-7	Sample Calculation of Conversion of Oleic Acid in Esterification of Oleic Acid with Methanol using supported sugar catalyst on aluminum (SCDCAI)	109
Appendix A-8	Sample Calculation to Calculate the Actual Concentration of Methyl Oleate using RSC at 333.15 K.	110
Appendix A-9	Sample Calculation of Kinetic Model	113
Appendix A-10	Sample Calculation of Catalyst Weight for Reusability	120
Appendix B	Calibration	122
Appendix B-1	Calibration for Temperature of Heating Mantle	122

Appendix B-2	Calibration Temperature of Vertical Furnace	124
Appendix B-3	Methyl Oleate Calibration Curve	125
Appendix C	Experimental Data	126
Appendix C-1	Maximum Coating of D-glucose on Aluminum	126
Appendix C-2	Total Acidity of Supported Sugar Catalyst ( <i>ex-situ</i> )	127
Appendix C-3	EDX Data for RSC, RSC, and RHAC	129
Appendix C-4	Kinetic Models Tables and Graphs for RHAC	131
Appendix C-5	Kinetic Models Tables and Graphs for RHC	138
Appendix C-6	Kinetic Models Tables and Graphs for RSC	145
Appendix C-7	Total Acidity of RHAC, RHC and RSC for Two Times of Re-use	153
Appendix C-8	Reproducibility	155
Appendix D	Viva Voce Questions and Answers	156



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