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APPENDICES

Appendix A Thermodynamic Study

For primary reaction Ethanol + Benzene = EB + H_2O

	Ethanol	Benzene	EB	H_2O
(J/mol) Enthalpy (ΔH^o_{298})	-234810	82930	29790	-241800
(J/mol) Gibb free (ΔG^o_{298})	-168280	129660	130580	-228600

So, ΔH^o_{298} = -60,130 J/mol

ΔG^o_{298} = -59,400 J/mol

For secondary reaction Ethanol + EB = p-DEB + H_2O

	Ethanol	EB	p-DEB	H_2O
(J/mol) Enthalpy (ΔH^o_{298})	-234810	29790	-22260	-241800
(J/mol) Gibb free (ΔG^o_{298})	-168280	130580	137860	-228600

So, ΔH^o_{298} = -59040 J/mol

ΔG^o_{298} = -53040 J/mol

Evaluating the equilibrium conversion (€) of benzene and ethanol at 400 °C, 1 bar, and molar feed ratio of benzene to ethanol is 1/1.

$$K_a = \frac{K_y K_\varphi K_p}{K_{f^o}}$$

$$K_a = \ln\left(-\frac{\Delta G^o}{RT}\right)$$

For

$$\frac{\Delta G^o}{RT} = \frac{\Delta G^o{}_0 - \Delta H^o{}_0}{RT_0} + \frac{\Delta H^o{}_0}{RT} + \frac{1}{T}\int_{To}^{T} \frac{\Delta Cp^o}{R} dT - \int_{T0}^{T} \frac{Cp^o}{R}\frac{dT}{T} \quad (1)$$

At

$$\int_{To}^{T} \frac{\Delta Cp^o}{R} dT = (\Delta A)T_0(\tau - 1) + \left(\frac{\Delta B}{2}\right) T_0{}^2(\tau^2 - 1) + \left(\frac{\Delta C}{3}\right) T_0{}^3(\tau^3 - 1) + \left(\frac{\Delta D}{T_0}\right)\left(\frac{\tau - 1}{\tau}\right) \quad ; \tau = \frac{T}{T_0} \tag{2}$$

At

$$\int_{T0}^{T} \frac{Cp^o}{R}\frac{dT}{T} = \Delta A \ln\tau + \left[\Delta B T_o + \left(\Delta C T_o{}^2 + \frac{\Delta D}{\tau^2 T_o{}^2}\right)\left(\frac{\tau+1}{2}\right)\right](\tau - 1) \tag{3}$$

Then

$$K_{a,T} = \ln\left(-\frac{\Delta G^o}{RT}\right)$$

So, $K_{a1,400} = 48{,}130.17$

$K_{a2,400} = 5{,}015.264$

For K_φ

$$K_{\varphi,1} = \frac{\varphi_{EB} \times \varphi_{H2O}}{\varphi_{benzene} \times \varphi_{ethanol}}$$

$$K_{\varphi,2} = \frac{\varphi_{p-DEB} \times \varphi_{H2O}}{\varphi_{EB} \times \varphi_{ethanol}}$$

@ pressure 1 bar $K_\varphi = 1$

For $K_{f^o} = \pi(1bar)^{\nu_i} = 1$

For $K_p = \pi(P)^{\nu_i} = 1$

For K_y

	mole in	mole react	mole out	yi
Benzene	1	-€1	1-€1	(1-€1)/2
Ethanol	1	-€1-€2	1-€1-€2	(1-€1-€2)/2
EB(c8H10	0	€1-€2	€1-€2	(€1-€2)/2
H20	0	€1+€2	€1+€2	(€1+€2)/2
p-DEB	0	€2	€2	€2/2
		∑	2	

From; $K_{y,1} = \frac{y_{EB} \times y_{H2O}}{y_{benzene} \times y_{ethanol}}$

So; $K_{y,1} = \frac{(€1-€2)\times(€1+€2)}{(1-€1)\times(1-€1-€2)}$

(2)

From; $K_{y,2} = \frac{y_{pDEB} \times y_{H2O}}{y_{EB} \times y_{ethanol}}$

So; $K_{y,1} = \frac{€2\times(€1+€2)}{(€1-€2)\times(1-€1-€2)}$

(3)

From; $K_{a,1} = K_{y,1}$ and $K_{a,2} = K_{y,2}$

So, $48{,}130.17 = \frac{(€1-€2)\times(€1+€2)}{(1-€1)\times(1-€1-€2)}$

$5{,}015.264 = \frac{€2\times(€1+€2)}{(€1-€2)\times(1-€1-€2)}$

So, €1 =0.80742

€2 =0.1925

	yi
Benzene	0.09629
Ethanol	4E-05
EB	0.30746
H_2O	0.49996
p-DEB	0.09625
Σ	1

Then, calculated with vary any of temperature.

T(°C)	benzene	ethanol	EB	H_2O	pDEB	€1	€2
150	0.0935	8.57E-08	0.31300	0.50000	0.09350	0.81300	0.18700
200	0.0938	5.11E-07	0.31200	0.50000	0.09381	0.81237	0.18763
300	0.0952	1.5E-05	0.31000	0.49999	0.09521	0.80955	0.19042
400	0.0963	4E-05	0.30700	0.49996	0.09625	0.80742	0.19250
500	0.0970	0.000125	0.30600	0.49988	0.09691	0.80593	0.19382
600	0.0979	0.000345	0.30500	0.49966	0.09756	0.80420	0.19511

T(°C)	T(K)	Selectivity EB	Selectivity(%) EB
150	423	0.5274	52.7380
200	473	0.5260	52.6041
300	573	0.5201	52.0107
400	673	0.5157	51.5691
500	773	0.5128	51.2840
600	873	0.5099	50.9946

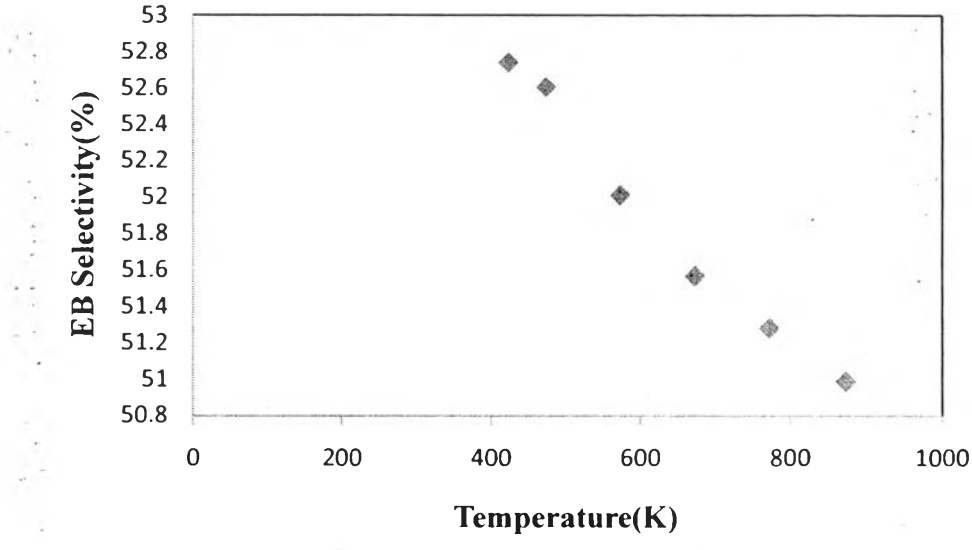


Figure A1 EB Selectivity versus Temperature.

T(K)	Ka1	Ka2	1/T	lnK1	lnK2
423	21537398	1927229.463	0.0023641	16.8853	14.47159
473	3721077	347814.4354	0.0021142	15.12952	12.75942
573	286226.9	28539.47133	0.0017452	12.56454	10.25904
673	48130.17	5015.264335	0.0014859	10.78166	8.520241
773	12955.19	1393.254864	0.0012937	9.469252	7.239398
873	4726.5	520.4856058	0.0011455	8.46094	6.254762

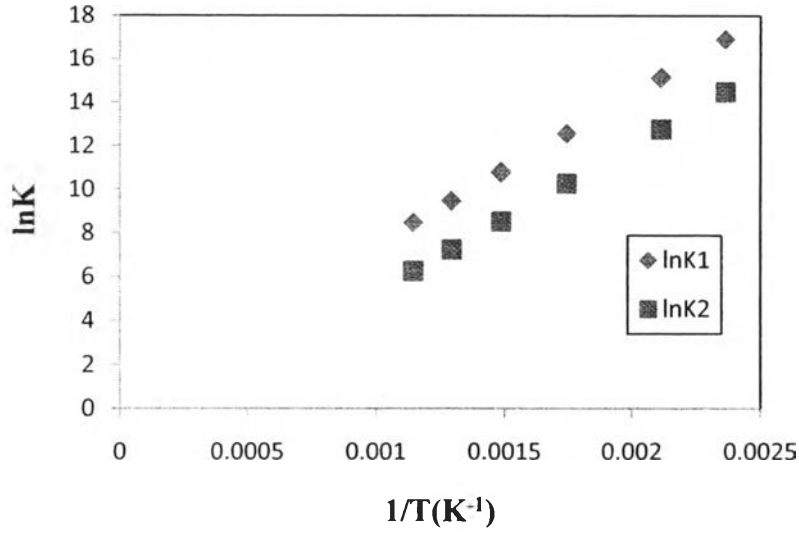


Figure A2 lnK versus 1/T.

Appendix B Experimental Data of Liquid Feed Calibration of GC 5890

1. Benzene

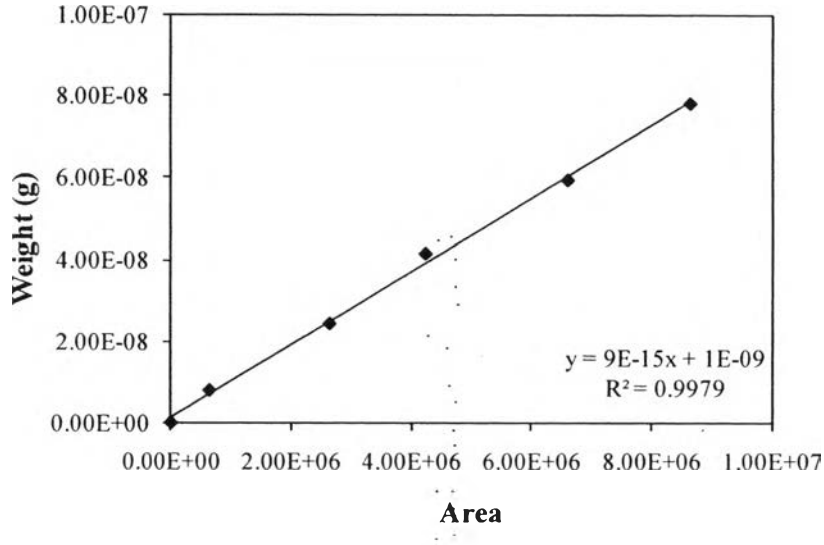


Figure B1 Calibration curve of benzene.

2. Ethanol

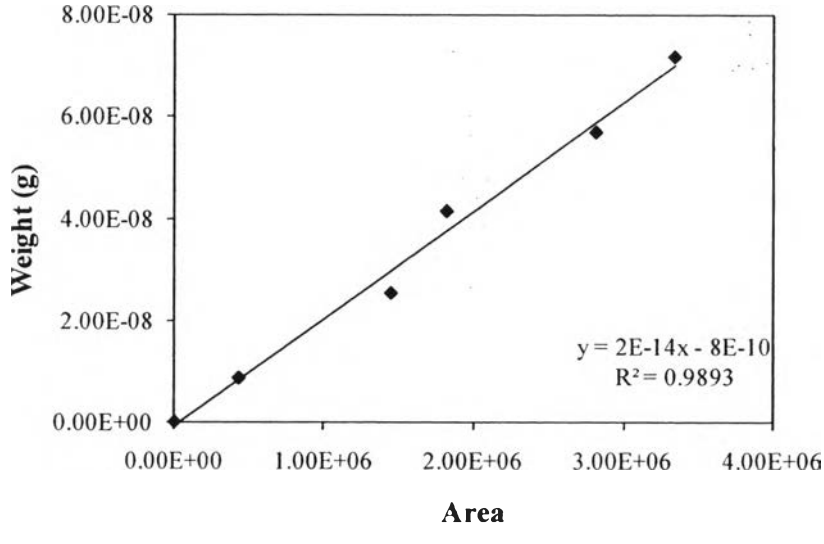


Figure B2 Calibration curve of ethanol.

Appendix C Experimental Data of Gas Flow Calibration of Sierra C100L Mass Flow Controller

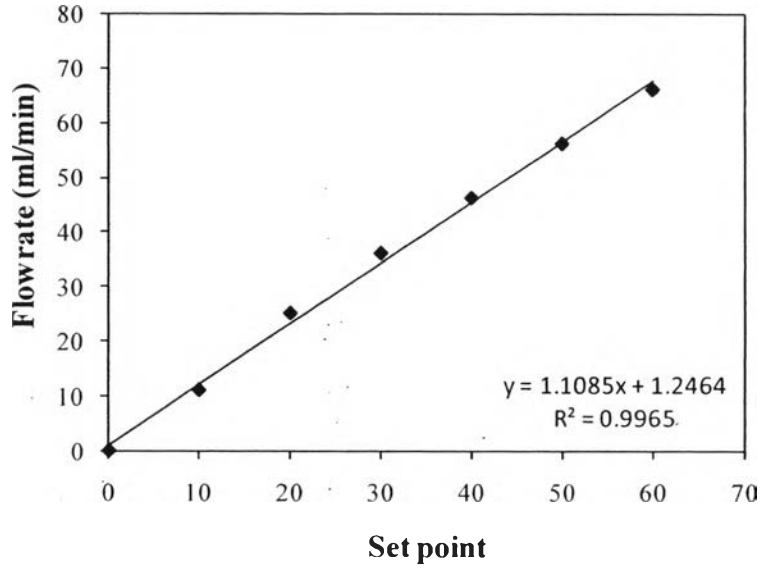


Figure C1 Calibration curve of nitrogen.

Appendix D Experimental Data of Liquid Feed Flow Calibration of Gilson 307 Pump

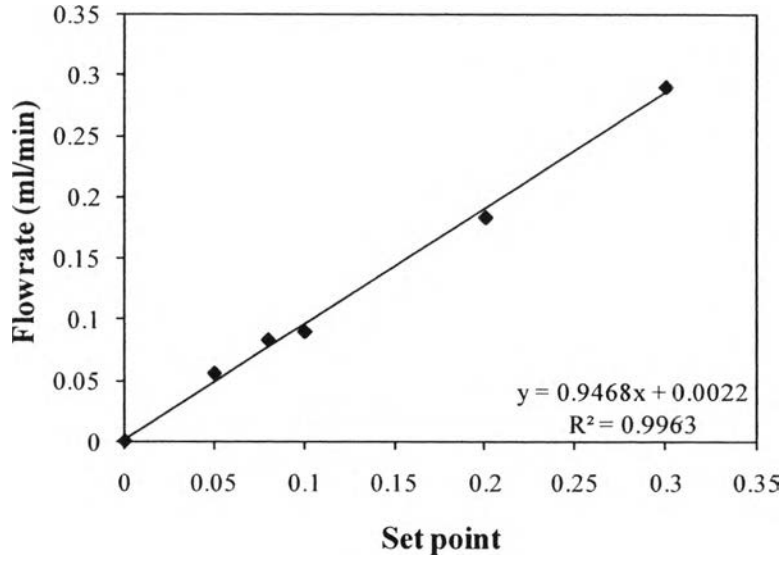


Figure D1 Calibration curve of liquid feed.

Appendix E Calculation of Si/Al Ratio and Theoretical Acidity

From the chemical composition determined by XRF method, the Si/Al ratio is calculated as follows:

The general formula of ZSM-5 is $Na_nAl_nSi_{96-n}O_{192}$

In the case of HZ5B2,

Si = 98.842 wt% Al = 1.063 wt%

Si = 3.519325 mol Al = 0.039397 mol

Si/ Al = 89.32907

From $Al_nSi_{96-n}O_{192}$,

Si/ Al = 89.32907 = (96-n)/n

90.32907 n = 96

n = 1.062781

So, Si = 94.93722

Al = 1.062781

From the chemical composition determined by XRF method, the theoretical acidity of zeolite is calculated as follows:

The general formula of HZSM-5 is $H_nAl_nSi_{96-n}O_{192}$

In the case of HZSM-5 (B1) with,

Si = 94.93722

Al = 1.062781

From the above, the general formula of HZSM-5 is $H_{1\ 062781}Al_{1\ 062781}Si_{94.93722}O_{192}$.

The weight of unit cell of HZSM-5 (U) is

U = 1.062781 (1) + 1.062781 (26.98) + 94.93722 (28.09) + 192(16.00)

U = 5768.098 g

The theoretical acidity ($[H^+]$) of HZSM-5 (B1) is

$[H^+]$ = 1.062781 /5768.098

$[H^+]$ = 0.181 mmol/g

However, from the chemical composition determined by XRF method was noticed some remained of Na.

In the case of HZSM-5 (B1),

Na = 0.095 wt%

Na = 0.111 mol%

So, H = 1.062781 -0.111 = 0.951781

From the above, the general formula of HZSM-5 is $H_{0.951781}Al_{1.062781}Si_{94.93722}O_{192}$.

The weight of unit cell of HZSM-5 (U) is

U = 0.951781 (1) + 1.062781 (26.98) + 94.93722 (28.09) + 192(16.00) + 0.05043(23)

U = 5767.987 g

The actual acidity ([H+]) of HZSM-5 (B1) is

[H+] = 0.951781 /5767.987

[H+] = 0.165 mmol/g

Appendix F The Other Catalyst Preparation

In this work, the other method for synthesizing ZSM-5 zeolites was adopted from the work of Chareonpanich and coworkers (Chareonpanich *et al.*, 2003) The detailed procedure is described below.

The agents used for synthetic reaction were sodium silicate (Na_2SiO_3), aluminium nitrate ($Al(NO_3)_3 \cdot 9H_2O$), tetrapropylammonium bromide (TPABr), sodium hydroxide pellets (NaOH,), hydrochloric (HCl), and de-ionized water. Firstly, TPABr solution was stirred and adjusts pH at 10.5 by 1 molar HCl and NaOH. After that, the TPABr solution was droped by Na_2SiO_3 and $Al(NO_3)_3 \cdot 9H_2O$. Then, the solution was stir for half an hour until it forms gel. After 0.5 h they were put in stainless steel autoclave, and heated in an oven at various conditions. After the hydrothermal synthesis, the autoclave was cooled to room temperature. Then, they were washed powder for reduce pH from 10.5 to 7 by water. Then, the as-synthesis zeolte ZSM-5 was dried at 120 °C overnight. Finally, calcination is applied to remove the precursor at 550 °C for 4 h.

Results & Disscusion

F.1 Effect of Aging Time

The ZSM-5 zeolites obtained at aging time 24 h and 48 h. Their surface areas are shown in Table 6.1. It was noticed that the surface area of ZSM-5 zeolite synthesized with aging time 48 h is higher than the one synthesized with aging time 24 h. Therefore, the longer aging time the higher surface area of ZSM-5 could be obtained. The SEM photographs of the ZSM-5 catalysts synthesized with aging time of 24 h, 48 h and 72 h at temperature 150 °C are shown in Fig. 6.1. With aging time 24 h, the unidentified amorphous solids of irregular shape and crystalline were found. It was represented that the ZSM-5 zeolite could not be produced under short aging time, which is at 24 h, however, the crystalline form of ZSM-5 zeolites increased as an aging time increased, especially, at 72 h.

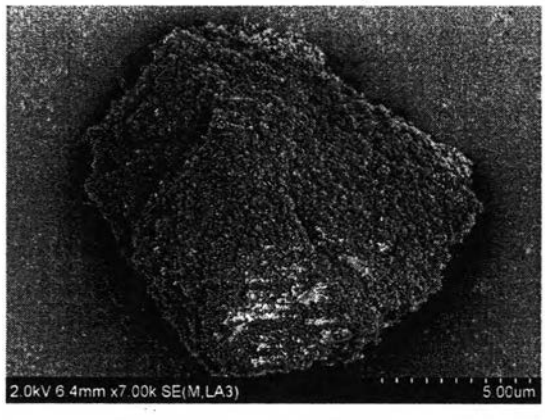
Table F.1 Surface characteristics of the investigated ZSM-5 catalysts

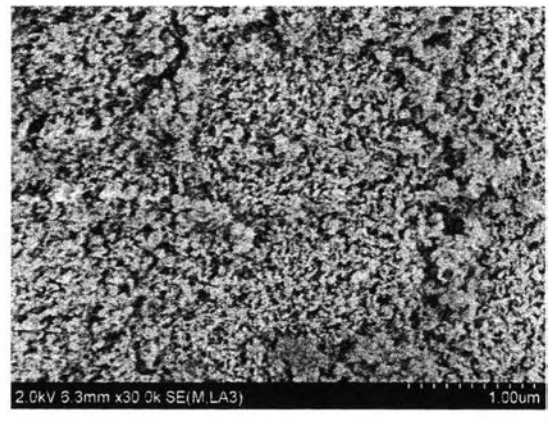
Catalyst	BET surface area (m^2/g)	Total pore volume[b] (cm^3/g)	Micropore volume[c] (cm^3/g)	Median pore width[c] (Å)
ZSM-5 (24h)[a]	129.62	0.506	0.0526	8.958
ZSM-5 (48h)[a]	156.68	0.788	0.0643	5.959

[a] Holding temperature 150 °C

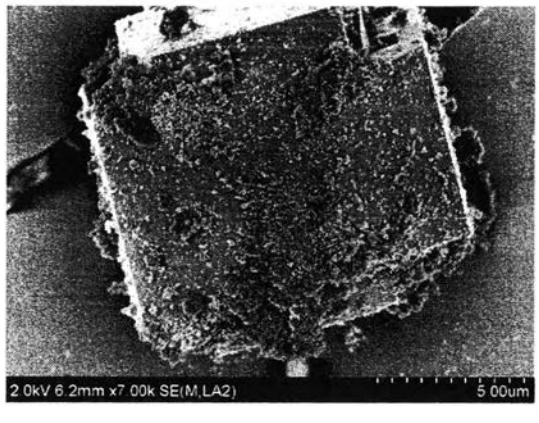
[b] Determined at $P/P_0 = 0.99$

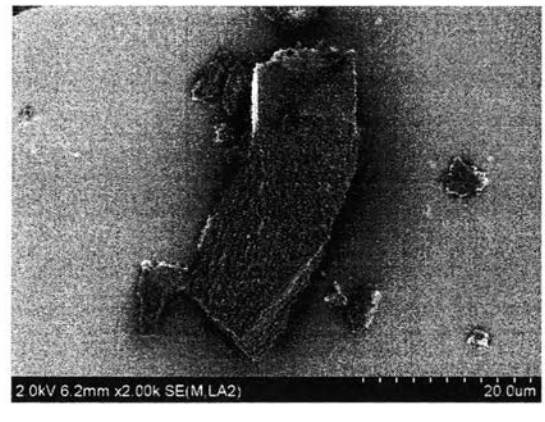
[c] Determined by SF method



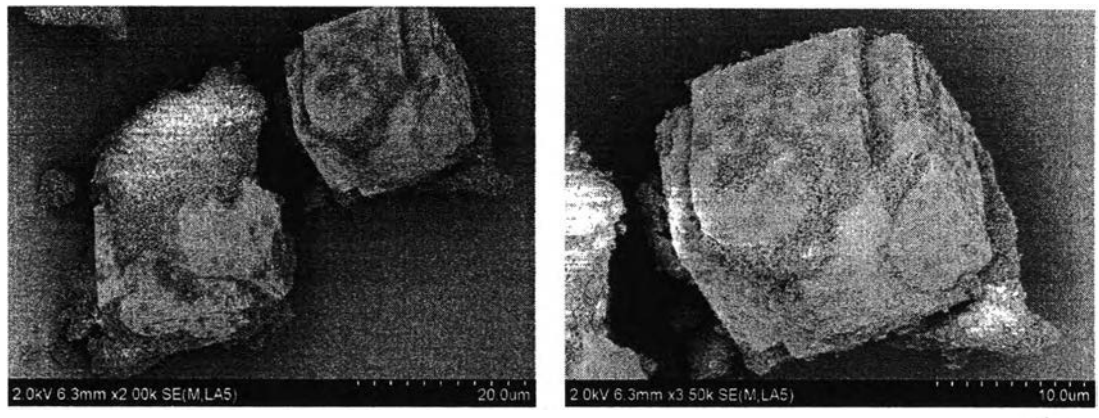


(a)





(b)

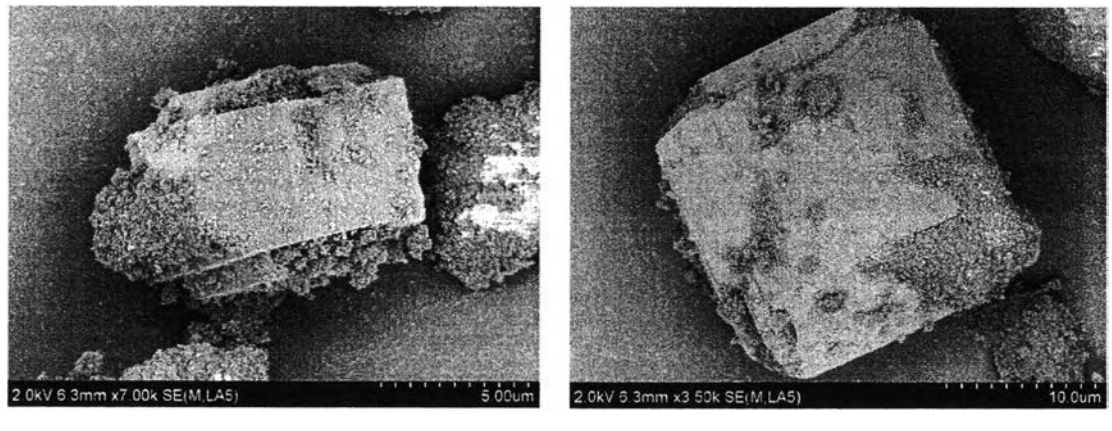


(c)

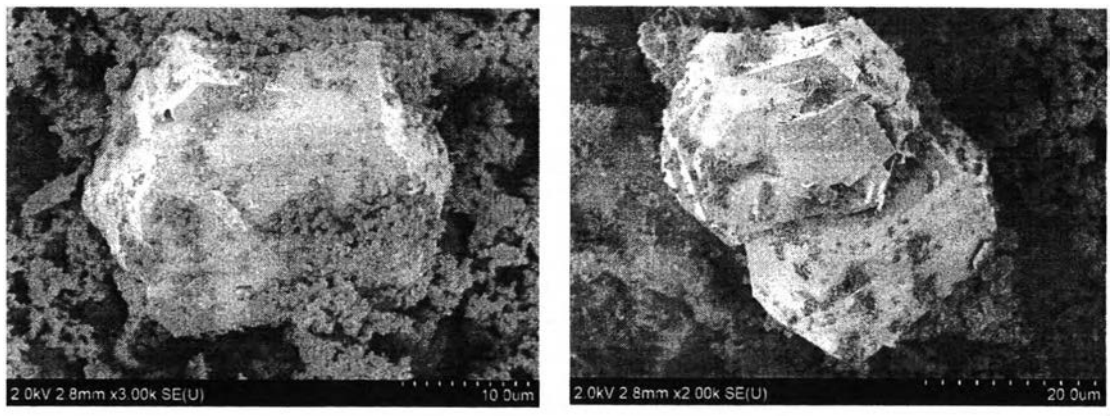
Figure F.1 SEM photographs of the ZSM-5 zeolites obtained from aging time at (a) 24 h, (b) 48 h and (c) 72 h ; SiO2/Al2O3 molar ratio of 195, temperature 150 °C

F.2 Effect of Temperature

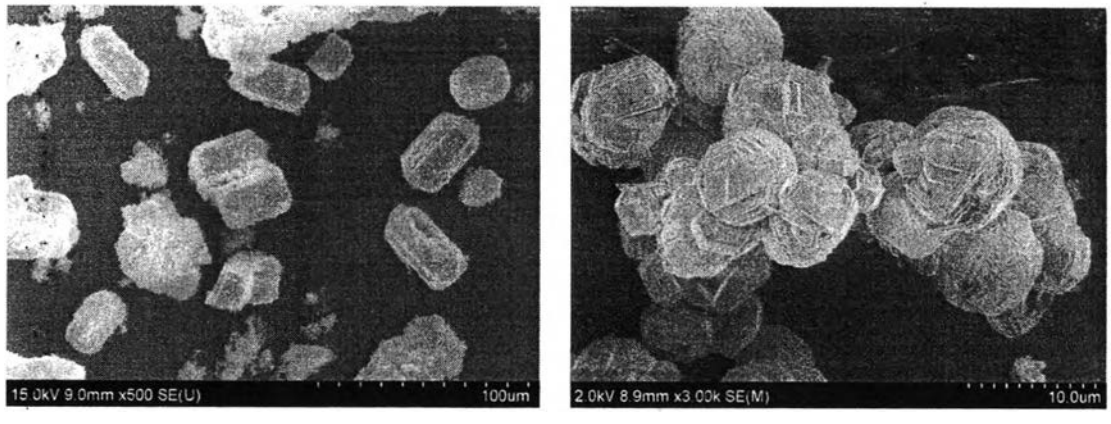
In this study, ZSM-5 zeolites were synthesized at temperature 150 °C, 180 °C and 240 °C. SEM photographs of the synthesized ZSM-5 are shown in Fig. 6.2. At temperature of 150 °C with aging time 72 h, the unidentified amorphous solids of irregular shape and crystalline were found. An increasing temperature tends to reduce the amount of unidentified amorphous solids and provides higher crystalline structure.



(a)



(b)



(c)

Figure F.2 SEM photographs of the ZSM-5 zeolites obtained from temperature at (a) 150 °C, (b) 180 °C with aging time 72 h and (c) 240 °C with aging time 18 h; SiO2/Al2O3 molar ratio of 195.

Appendix G Experimental Data of Catalytic Activity Test for Alkylation of Benzene with Ethanol over Synthesized HZSM-5 Catalyst

Table G1 Catalytic Activity Testing over HZSM-5 with Different Synthesis at Temperature 500°C, B/E = 4, WHSV = 20 h^{-1}

Component	catalyst	Conversion (%)					
		15min	60min	130 min	200 min	270 min	340 min
Benzene	HZ5A2	1.37	24.31	21.78	21.05	21.31	21.64
	HZ5A3	1.29	20.36	15.60	17.23	15.60	15.76
	HZ5B1	1.68	11.23	11.43	11.14	11.88	14.83
	HZ5B2	1.22	11.24	11.81	11.18	11.81	13.39
	HZ5B3	1.68	15.62	13.39	13.82	17.21	13.04
Ethanol	HZ5A2	82.34	98.32	97.32	98.72	99.55	99.87
	HZ5A3	81.34	98.23	97.12	97.82	99.38	99.98
	HZ5B1	83.23	98.14	98.43	99.51	99.72	99.82
	HZ5B2	81.34	98.56	98.61	97.99	99.54	99.97
	HZ5B3	80.42	97.94	98.54	98.10	99.12	99.98

Table G2 Catalytic Activity Testing on Different Temperature for HZ5B3, B/E = 4, WHSV = 20 h^{-1}

Component	Temperature (°C)	Conversion (%)					
		15min	60min	130 min	200 min	270 min	340 min
Benzene	300	2.67	11.23	12.38	12.05	11.24	11.48
	500	1.68	15.62	13.39	13.82	14.02	13.04
	600	1.22	5.34	4.78	4.14	4.25	3.87
Ethanol	300	82.14	98.12	99.56	99.78	99.90	99.54
	500	81.31	98.45	99.12	99.56	99.12	99.99
	600	85.22	98.14	99.67	99.51	99.51	99.84

Table G3 Catalytic Activity Testing on Different Feed Ratio for HZ5A2, WHSV = 20 h^{-1}, T = 500 °C

Component	B/E (mol/mol)	Conversion (%)					
		15min	60min	130 min	200 min	270 min	340 min
Benzene	1	2.87	33.1	35.05	37.78	39.61	36.37
	2	1.61	20.10	19.84	19.87	20.05	21.79
	4	1.37	24.31	21.78	21.05	21.31	21.64
Ethanol	1	85.12	99.32	97.23	98.33	98.12	99.91
	2	80.12	99.10	97.69	98.23	97.98	99.86
	4	80.78	99.42	97.91	98.42	99.10	99.87

Table G4 Product Distribution of Liquid Sample over HZSM-5 with Different Synthesis at Temperature 500°C, B/E = 4, WHSV = 20 h^{-1}, and TOS 340 minutes

Component	Product distribution (wt%)				
	HZ5A2	HZ5A3	HZ5B1	HZ5B2	HZ5B3
ethylene	0.02	0.02	0.01	0.01	0.01
methanol	0.01	0.02	0.01	0.01	0.01
toluene	0.20	0.13	0.30	0.23	0.77
EB	94.13	94.08	93.38	92.71	90.71
m-xylene	0.15	0.24	0.34	0.37	0.66
xylene	0.71	1.56	1.64	0.85	2.13
o-xylene	0.01	0.01	0.00	0.05	0.06
cumene	0.02	0.01	0.03	0.02	0.04
propyl-benzene	0.04	0.04	0.10	0.13	0.17
p-ethyl toluene	0.03	0.01	0.03	0.04	0.08
o-ethyl toluene	0.04	0.03	0.05	0.03	0.09
1,2,3-trimethylbenzene	0.05	0.03	0.10	0.03	0.06
(2-methylpropyl)-benzene	0.01	0.01	0.05	0.01	0.01
(1-methylpropyl)-benzene	0.18	0.10	0.30	0.12	0.31
indane	0.15	0.18	0.28	0.25	0.63
1-propenyl benzene	0.03	0.04	0.12	0.05	0.29
1,3-diethylbenzene	0.66	0.70	0.15	0.92	0.89
1,4-diethylbenzene	3.16	2.51	2.48	3.83	2.30
1,2-diethylbenzene	0.02	0.01	0.01	0.02	0.02
2-butenylbenzene	0.00	0.05	0.01	0.00	0.08
1-butenylbenzene	0.00	0.08	0.11	0.04	0.12
1-ethyl-3-(1-methylethyl)-benzene	0.03	0.00	0.01	0.09	0.00

Table G4 Product Distribution of Liquid Sample over HZSM-5 with Different Synthesis at Temperature 500°C, B/E = 4, WHSV = 20 h^{-1}, and TOS 340 minutes (Continued)

Component	Product distribution (wt%)				
	HZ5A2	HZ5A3	HZ5B1	HZ5B2	HZ5B3
1-ethyl-4-(1-methylethyl)-benzene	0.09	0.00	0.01	0.01	0.00
1-methyl-4-(1-methylpropyl)-Benzene	0.02	0.00	0.03	0.01	0.01
1-butynyl-benzene	0.05	0.00	0.01	0.11	0.00
1-methyl-1H-Indene	0.01	0.01	0.10	0.01	0.05
1,2-dihydro-Naphthalene	0.01	0.06	0.00	0.04	0.12
1,2,3,4-tetrahydronaphthalene	0.01	0.01	0.00	0.00	0.02
naphthalene	0.00	0.05	0.10	0.01	0.25
(1-ethyl-1-propenyl)-Benzene	0.00	0.01	0.03	0.01	0.01
2-methyl-Naphthalene	0.15	0.01	0.22	0.02	0.10
Total	100.00	100.00	100.00	100.00	100.00

Table G5 Product Distribution of Liquid Sample over HZ5B3 at Different Temperature, B/E = 4, WHSV = 20 h^{-1}, and TOS 340 minutes

Component	Product distribution (wt%)		
	300 (°C)	500 (°C)	600 (°C)
ethylene	0.01	0.01	0.05
methanol	0.01	0.01	0.01
toluene	0.12	0.77	8.44
EB	69.85	90.71	63.92
m-xylene	0.06	0.66	0.56
xylene	0.29	2.13	17.27
o-xylene	0.00	0.06	0.00
cumene	1.84	0.04	0.07
propyl-benzene	0.59	0.17	0.25
p-ethyl toluene	1.05	0.08	0.10
o-ethyl toluene	0.04	0.09	0.10
1,2,3-trimethylbenzene	0.00	0.06	0.46
(2-methylpropyl)-benzene	0.03	0.01	0.04
(1-methylpropyl)-benzene	0.58	0.31	0.15
indane	0.07	0.63	1.25
1-propenyl benzene	0.00	0.29	0.00
1,3-diethylbenzene	1.68	0.89	0.98
1,4-diethylbenzene	22.90	2.30	3.97
1,2-diethylbenzene	0.41	0.02	0.03
2-butenylbenzene	0.00	0.08	0.03
1-butenylbenzene	0.00	0.12	0.03
1-ethyl-3-(1-methylethyl)-benzene	0.04	0.00	0.14

Table G5 Product Distribution of Liquid Sample over HZ5B3 at Different Temperature, B/E = 4, WHSV = 20 h^{-1}, and TOS 340 minutes (Continued)

Component	Product distribution (wt%)		
	300 (°C)	500 (°C)	600 (°C)
1-ethyl-4-(1-methylethyl)-benzene	0.09	0.00	0.00
1-methyl-4-(1-methylpropyl)-Benzene	0.00	0.01	0.00
1-butynyl-benzene	0.00	0.00	0.00
1-methyl-1H-Indene	0.13	0.05	0.12
1,2-dihydro-Naphthalene	0.11	0.12	0.59
1,2,3,4-tetrahydronaphthalene	-	0.02	0.05
naphthalene	0.04	0.25	1.00
(1-ethyl-1-propenyl)-Benzene	0.03	0.01	0.03
2-methyl-Naphthalene	0.05	0.10	0.37
Total	100.00	100.00	100.00

Table G6 Product Distribution of Liquid Sample over HZ5B3 at Different Feed Ratio of B/E, Temperature 500 °C, WHSV = 20 h^{-1}, and TOS 340 minutes

Component	Product distribution (wt%)		
	B/E=1	B/E=2	B/E=4
ethylene	0.01	0.01	0.02
methanol	0.01	0.01	0.01
toluene	0.10	0.18	0.20
EB	90.37	94.00	94.13
m-xylene	0.13	0.15	0.15
xylene	0.30	0.54	0.71
o-xylene	0.02	0.02	0.01
cumene	0.05	0.01	0.02
propyl-benzene	0.03	0.04	0.04
p-ethyl toluene	0.02	0.03	0.03
o-ethyl toluene	0.03	0.02	0.04
1,2,3-trimethylbenzene	0.02	0.03	0.05
(2-methylpropyl)-benzene	0.01	0.01	0.01
(1-methylpropyl)-benzene	0.09	0.09	0.18
indane	0.08	0.08	0.15
1-propenyl benzene	0.01	0.02	0.03
1,3-diethylbenzene	1.85	1.06	0.66
1.4-diethylbenzene	6.68	3.51	3.16
1,2-diethylbenzene	0.03	0.04	0.02
2-butenylbenzene	0.00	0.00	0.00
1-butenylbenzene	0.03	0.03	0.00
1-ethyl-3-(1-methylethyl)-benzene	0.08	0.07	0.03

Table G6 Product Distribution of Liquid Sample over HZ5B3 at Different Feed Ratio of B/E, Temperature 500 °C, WHSV = 20 h^{-1}, and TOS 340 minutes (Continued)

Component	Product distribution (wt%)		
	B/E=1	B/E=2	B/E=4
1-ethyl-4-(1-methylethyl)-benzene	0.00	0.00	0.09
1-methyl-4-(1-methylpropyl)-Benzene	0.00	0.00	0.02
1-butynyl-benzene	0.00	0.00	0.05
1-methyl-1H-Indene	0.01	0.01	0.01
1,2-dihydro-Naphthalene	0.03	0.02	0.01
1,2,3,4-tetrahydronaphthalene	0.00	0.00	0.01
naphthalene	0.01	0.02	0.00
(1-ethyl-1-propenyl)-Benzene	0.01	0.00	0.00
2-methyl-Naphthalene	0.00	0.01	0.15
Total	100.00	100.00	100.00

Appendix E Calculation of the minimum ratio the bed length to the particle size

$$\frac{L_b}{d_p} > \frac{8n}{Pe_p} \ln\left(\frac{1}{1-x}\right)$$

L_b = length of bed

d_p = diameter of particle

Pe = Peclet number

n = order of reaction

x = conversion of reaction

Taking Pe_p = 0.5 for the low Reynolds region of interest for laboratory-scale operation.

Taking d_p = 0.05 cm for the particle sieve at mesh 20-40

Assume n = 1

If x = 0.4

$$\frac{L_b}{0.05} = \frac{8}{0.5} \ln\left(\frac{1}{1-0.4}\right)$$

L_b= 0.04 cm

If x = 0.5

$$\frac{L_b}{0.05} = \frac{8}{0.5} \ln\left(\frac{1}{1-0.5}\right)$$

L_b= 0.55 cm

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