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APPENDICES

APPENDIX A

EXPERIMENTAL AND DATA ANALYSIS

A-1 Experimental data of esterification reaction in batch reactor

Table A-1.1 The percent of methyl ester at the molar ratio 1:6 for the reaction time 30 minutes, at the various temperature between 240-300(°C)

Tem(°C)	Sample			Average	SD
	%Methyl ester (1)	%Methyl ester (2)	%Methyl ester (3)		
240	39.67	41.37	57.21	46.0833	9.6733
250	66.15	70.98	55.57	64.2333	7.8817
260	75.2	62.27	56.9	64.7900	9.4066
270	73.67	56.62	76.51	68.9333	10.7577
280	75.52	64.88	77.21	72.5366	6.6844
290	79.25	96.5	81.55	85.7666	9.3662
300	92.49	98.61	92.83	94.6433	3.4394

Table A-1.2 The percent of methyl ester at 300 °C, molar ratio of palm fatty acid to methanol 1:1 to 1:12 at the reaction time 30 minutes

Molar ratio	Sample			Average	SD
	%Methyl ester (1)	%Methyl ester (2)	%Methyl ester (3)		
1:1	68.1300	71.8700	74.04	71.3467	2.9895
1:2	72.9000	78.2100	82.43	77.8467	4.7753
1:3	80.9900	87.9800	73.52	80.8300	7.2313
1:6	92.4900	98.6000	92.84	94.6433	3.4310
1:9	79.6900	85.6100	88.93	84.7433	4.6805
1:12	77.3700	65.3900	78.88	73.8800	7.3912

Table A-1.3 The percent of methyl ester at 250 °C, molar ratio of palm fatty acid to methanol 1:1 to 1:12 at the reaction time 30 minutes

Molar ratio	Sample		Average	SD
	%Methyl ester (1)	%Methyl ester (2)		
1:1	49.04	49.75	49.3950	0.5020
1:2	58.92	63.31	61.1150	3.1042
1:3	65.4	68.68	67.0400	2.3193
1:6	77.82	77.44	77.6300	0.2687
1:9	55.3	67.44	61.3700	8.5843
1:12	51.25	65.66	58.4550	10.1894

A-2 The data of the effect of water on yield of methyl esterification product

Table A-2.1 The data of effect of water on yield of methyl esterification at various water (5-30%) the reaction time 30 minutes, 300 °C

%Water	%Methyl ester (1)	% Methyl ester (2)	% Methyl ester (3)	Average	SD
5	77.66	72.39	72.9	74.3166	2.2288
10	72.87	69.83	67.15	69.9500	1.9466
15	72.51	68.32	64.6	68.4766	2.6888
20	68.51	69.95	64.43	67.6300	2.1333
25	66.18	66.21	61.85	66.1950	0.0150
30	48.41	57.3	56.8	52.8550	4.4450

A-3 The data of hydrolysis of methyl ester

Table A-3.1 The data of hydrolysis of methyl ester by hydrolysis at various the reaction time between 0-30 minutes, 300 °C at the water content to methyl ester 1 %

Times	%Methyl ester (1)	%Methyl ester (2)	Average	SD
0	100	100	100	0.0000
5	99.09	93.87	96.48	3.6911
10	91.54	89.48	90.51	1.4566
15	86.38	84.16	85.27	1.5698
20	86.12	82.12	84.12	2.8284
25	85.99	79.32	82.655	4.7164
30	74.79	82.26	78.525	5.2821

Table A-3.2 The data of hydrolysis of methyl ester by hydrolysis at various the reaction times between 0-30 minutes, 300 °C at the water content to methyl ester 10 %

Times	%Methyl ester (1)	%Methyl ester (2)	Average	SD
0	100	100	100	0.0000
5	93.13	88.74	90.94	3.1042
10	89.52	85.60	87.56	2.7719
15	88.39	81.54	84.97	4.8437
20	84.45	79.37	81.91	3.5921
25	79.22	74.99	77.11	2.9911
30	75.28	75.53	75.41	0.1768

Table A-3.3 The data of hydrolysis of methyl ester hydrolysis at various the reaction times between 0-30 minutes, 300 °C at the water content to methyl ester 20 %

Times	%Methyl ester (1)	%Methyl ester (2)	Average	SD
0	100	100	100	0.0000
5	86.63	88.65	87.64	1.4284
10	80.54	79.05	79.795	1.0536
15	81.32	75.99	78.655	3.7689
20	76.01	74.52	75.265	1.0536
25	73.4	75.07	74.235	1.1809
30	72.45	75.53	73.99	2.1779

Table A-3.4 The data of hydrolysis of methyl ester by hydrolysis at various the reaction times between 0-30 minutes, 300 °C at the water content to methyl ester 30 %

Times	%Methyl ester (1)	%Methyl ester (2)	Average	SD
0	100	100	100	0.0000
5	80.46	81.12	80.79	0.4667
10	76.06	80.04	78.05	2.8143
15	78.75	77.31	78.03	1.0182
20	75.57	77.50	76.535	1.3647
25	67.17	66.81	66.99	0.2546
30	42.99	45.45	44.22	1.7395

APPENDIX B

B-1 Calculation of molecular weight of palm fatty acids

The molecular weight of palm fatty acids is calculated from the weighted average of the molecular weight of the five key fatty acids: palmitic acid, oleic acid, stearic acid, linoleic acid, and linolenic acid. The compositions of palm fatty acids are shown in Table B-1.1

Table B-1.1 Composition and molecular weight of key components in of palm fatty acids.

Palm fatty acids	% Weight Fraction	Molecular weight
Palmitic acid	42.8	256.43
Oleic acid	40.5	282.47
Stearic acid	4.5	284.5
Linoleic acid	10.1	268
Linolenic acid	2.1	278.43

The data in Table B-1.1 can be used to compute the molecular weight of palm fatty acids as shown below:

1 mole of palm fatty acids

$$M_w = \text{Sum } (M_{Fa} \times \% \text{ Weight fraction fatty acids}) \dots\dots\dots \mathbf{B-1.1}$$

Where

M_w = Molecular weight of fatty acids

M_{Fa} = Molecular weight of each fatty acids

Such as (Data in the table B-1.1) apply in equation 1

$$= (0.428 \times 256.43) + (0.405 \times 282.47) + (0.045 \times 284.5) + (0.101 \times 268) + (0.021 \times 278.43)$$

$$= 269.85 (\sim 270)$$

B-2 Calculate the percent yield of fatty acids to methyl esters

The percent yield methyl ester is defined as

$$\begin{aligned} \% \text{Yield of methyl ester} = \% \text{ Methyl ester} &= \frac{W_{ME}}{W_{Fa} \times (x_i)} \times 100 \dots\dots\dots \text{B-2.1} \\ &= \frac{W_{MP} + W_{MS} + W_{MO} + W_{ML}}{(W_{Fa}) \times (x_i)} \times 100 \end{aligned}$$

Where

- W_{ME} = weight of methyl ester (g)
- W_{Fa} = weight of fatty acid (g)
- W_{MP} = weight of methyl palmitate (g)
- W_{MS} = weight of methyl stearate (g)
- W_{MO} = weight of methyl oleate (g)
- W_{ML} = weight of methyl linoleate (g)
- W_P = weight of palmitic acid (g)
- W_S = weight of stearic acid (g)
- W_O = weight of oleic acid (g)
- W_L = weight of linoleic acid (g)
- x_i = weight fraction of fatty acid

Calculation weight of each methyl ester

$$W_{ME} = \left(\frac{C \times V_{TD}}{V_S} \right) \times V_P \dots\dots\dots \text{B-2.2}$$

Where

- W_{ME} = weight of methyl ester (g)
- C = concentration of each methyl ester from calibration curve (g/ml)
- V_{TD} = total volume dilute (ml)
- V_S = volume product dilute (ml)
- V_P = total volume of product (ml)

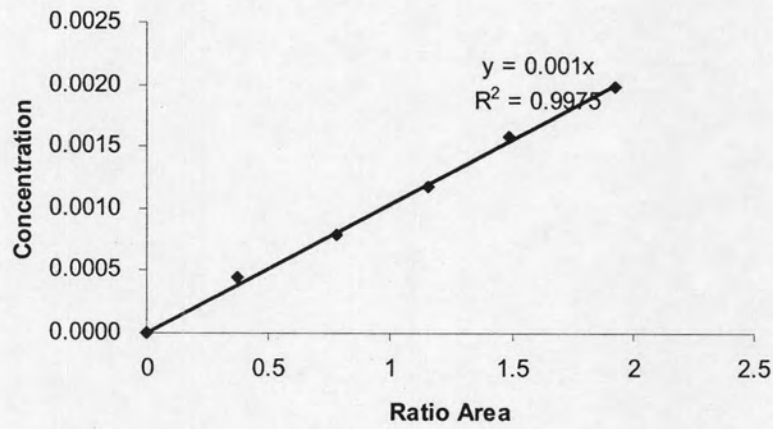


Figure B-2.2 Standard calibration curve for methyl plamitate (corrected with use of ecosane as an internal standard).

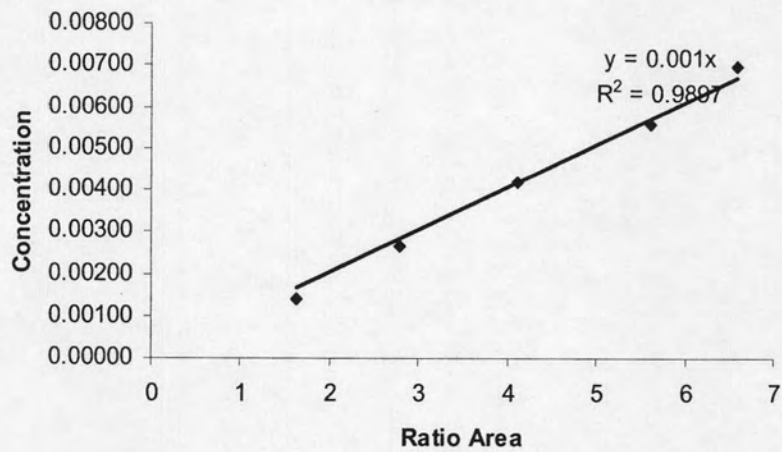


Figure B-2.3 Standard calibration curve for methyl oleate (corrected with use of ecosane as an internal standard).

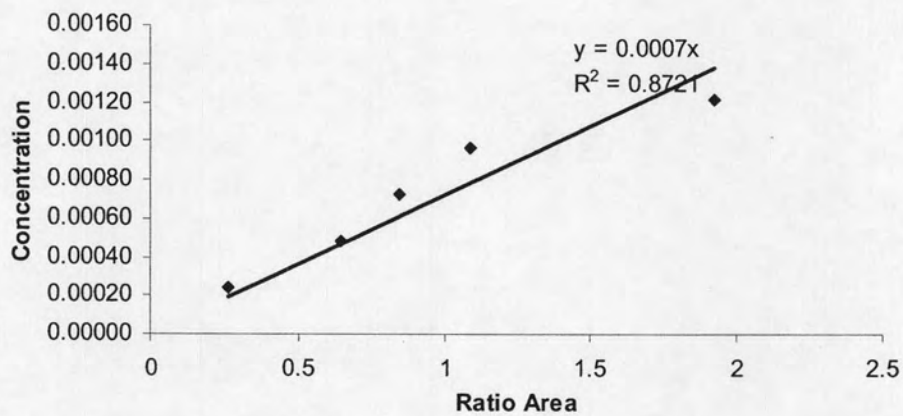


Figure B-2.4 Standard calibration curve for methyl stearate (corrected with use of ecosane as an internal standard).

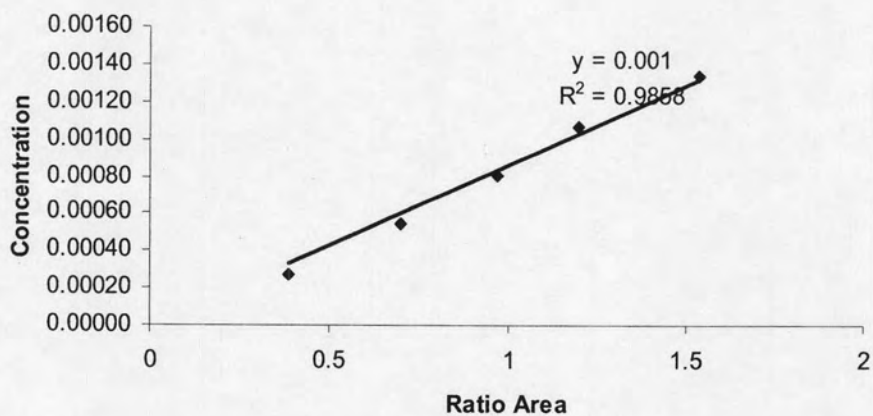


Figure B-2.5 Standard calibration curve for methyl linoleate (corrected with use of ecosane as an internal standard).

Example percent yield of methyl ester based on the data from the chromatogram in Figure B-2.1 and the standard calibration curves for different methyl esters figure B-2.2-B-2.5). From the data of peak GC, it can convert to concentration of each methyl ester and then apply in equation B-2.2

Concentration of each of fatty acid (g/ml) = ratio peak area x calibration constant

Example Concentration of palmitate = ratio peak area of area of palmitate x 0.001

$$= \frac{74716}{8788} \times 0.001 = 6.21 \times 10^{-3} \text{ g/ml}$$

$$\text{Thus, wt. of methyl palmitate} = \frac{6.21 \times 10^{-3} \times 1 \text{ ml} \times 6 \text{ ml}}{0.1 \text{ ml}} = 0.3729 \text{ g}$$

Wt of methyl oleate and methyl linoleate

Because the chromatographic peaks of methyl oleate and methyl linoleate are not separated but the calibration factors are exactly the same, the amount of the two can be calculated using the total peak area.

$$\begin{aligned} \text{Wt. of methyl oleate and methyl linoleate} &= \frac{6.43 \times 10^{-3} \text{ g/ml} \times 1 \text{ ml} \times 6 \text{ ml}}{0.1 \text{ ml}} \\ &= 0.3861 \text{ g} \end{aligned}$$

$$\text{Wt. of methyl stearate} = \frac{6.26 \times 10^{-4} \text{ g/ml} \times 1 \text{ ml} \times 6 \text{ ml}}{0.1 \text{ ml}} = 0.0376 \text{ g}$$

$$\text{Sum of wt fatty acid} = 0.7966 \text{ g}$$

Then apply in equation B-2.1

$$\% \text{ Yield of Methyl Ester} = \frac{0.7966 \times 100}{0.92 \times 0.979} = 88.44 \%$$

B-3 Calculate mole fraction

Example Mole fraction of palm fatty acids and methanol such as 1:3

Palm fatty acids = 270 g Density = 0.92 g/ml

Methanol = 96.12 g Density = 0.79 g/ml

If we used 1 ml of palm fatty acids = 0.0034 mol.

Calculate methanol $3 \times 0.0034 = 0.0102$ mol, will use methanol = 0.3264 g

\therefore volume methanol = $0.3264/0.79 = 0.4132$ ml

APPENDIX C

Table C-1.1 Critical Property Increment Lydersen's Structural Contribution

Symbols	ΔT	ΔP	ΔV
Nonring increments			
-CH ₃	0.020	0.227	55
-CH ₂	0.020	0.227	55
-CH ₂	0.012	0.210	51
-C-	0.00	0.210	41
=CH ₂	0.018	0.198	45
-CH	0.018	0.198	45
=C-	0.0	0.198	36
-C-	0.0	0.198	36
=CH	0.005	0.153	(36)
=C-	0.005	0.153	(36)
Ring increments			
-CH ₂ -	0.013	0.184	44.5
-CH	0.012	0.192	46
-C-	(-0.007)	(0.154)	(31)
=CH	0.011	0.154	37
-C-	0.011	0.154	36
-C=	0.011	0.154	36
Halogen increments			
-F	0.018	0.221	18
-Cl	0.017	0.320	49
-Br	0.010	(0.50)	(70)
-I	0.012	(0.83)	(95)
Oxygen increments			
-OH (alcohols)	0.082	0.06	(18)
-OH (phenols)	0.031	(-0.02)	(3)
-O- (nonring)	0.021	0.16	20
-O- (ring)	(0.014)	(0.12)	(8)
-C=O (nonring)	0.040	0.29	60

Symbols	ΔT	ΔP	ΔV
Oxygen increments (cont)			
$\begin{array}{c} \\ -C=O \text{ (ring)} \end{array}$	(0.033)	(0.2)	(50)
$\begin{array}{c} \\ HC=O \text{ (aldehyde)} \end{array}$	0.048	0.33	73
$-COOH \text{ (acid)}$	0.085	(0.4)	80
$-COO- \text{ (ester)}$	0.047	0.47	80
$=O \text{ (except for combinations above)}$	(0.02)	(0.12)	(11)
Nitrogen increments			
$-NH_2$	0.031	0.095	28
$\begin{array}{c} \\ -NH \text{ (nonring)} \end{array}$	0.031	0.135	(37)
$\begin{array}{c} \\ -NH \text{ (ring)} \end{array}$	(0.024)	(0.09)	(27)
$\begin{array}{c} \\ -N- \text{ (nonring)} \end{array}$	0.014	0.17	(42)
$\begin{array}{c} \\ -N- \text{ (ring)} \end{array}$	(0.007)	(0.13)	(32)
$-CN$	(0.060)	(0.36)	(80)
$-NO_2$	(0.055)	(0.42)	(76)
Sulfur increments			
$-SH$	0.015	0.27	55
$-S- \text{ (nonring)}$	0.015	0.27	55
$-S- \text{ (ring)}$	(0.008)	(0.24)	(45)
$=S$	(0.003)	(0.24)	(47)
Miscellaneous			
$\begin{array}{c} \\ -Si- \end{array}$	0.03	(0.54)	
$\begin{array}{c} \\ -B- \end{array}$	(0.03)		

The critical point of each fatty acids used the Lydersen's Structural Contribution and calculate the critical property in the equation C-1.1-C-1.4

$$\bullet \text{ Critical Temperature (Tc)} = \frac{\text{boiling point FFA}}{0.567 + (N\Delta T) - (N\Delta T)^2} \dots\dots\dots \text{C-1.1}$$

$$\bullet \text{ Critical Pressure (Pc)} = \frac{\text{molecular weight of FFA}}{[0.34 + (N\Delta P)]^2} \dots\dots\dots \text{C-1.2}$$

$$\bullet \text{ Critical Volume (Vc)} = 40 + (N\Delta V) \dots\dots\dots \text{C-1.3}$$

$$\bullet \text{ Zc} = \frac{Pc \times Vc}{82.06 \times Tc} \dots\dots\dots \text{C-1.4}$$

Palmitic Acids

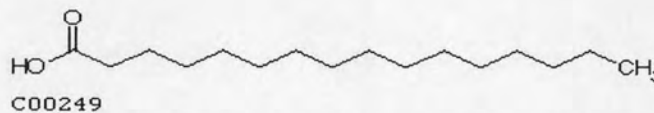


Table C-1.2 The property of critical palmitic acid with Lydersen's Structural

Mw	bp (oC)	bp (K)		N	ΔT	$N\Delta T$	ΔP	$N\Delta P$	ΔV	$N\Delta V$
256.42	271.5	544.65	COOH	1	0.085	0.085	0.4	0.4	80	80
			CH3	1	0.02	0.02	0.227	0.227	55	55
			CH2	14	0.02	0.28	0.227	3.178	55	770
			SUM			0.385		3.805		905
			total			0.385		3.805		905

The data in the table C-1.2 apply in the equation C-1.1-C-1.4

Tc (K)	Pc (atm)	Vc (cm ³ /(g.mol))	Zc
677.62	14.92	945	0.25

$$R = 82.06 \text{ cm}^3\text{atm/g.mol K}$$



Stearic acid

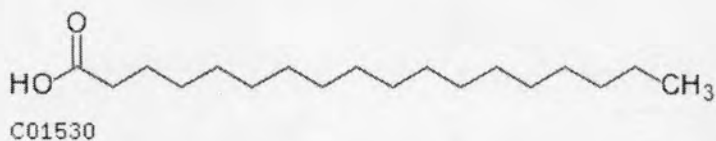


Table C-1.3 The property of critical stearic acid with Lydersen's Structural Contribution

Mw	bp (oC)	bp (K)		N	ΔT	$N\Delta T$	ΔP	$N\Delta P$	ΔV	$N\Delta V$
284.47	291	564.15	COOH	1	0.085	0.085	0.4	0.4	80	80
			CH3	1	0.02	0.02	0.227	0.227	55	55
			CH2	16	0.02	0.32	0.227	3.632	55	880
			SUM			0.425		4.259		1015
			total			0.425		4.259		1015

The data in the table C-1.3 apply in the equation C-1.1-C-1.4

Tc (K)	Pc (atm)	Vc (cm ³ /(g.mol))	Zc
695.30	13.45	1055	0.25

$$R = 82.06 \text{ cm}^3\text{atm/g.mol K}$$

Oleic acid

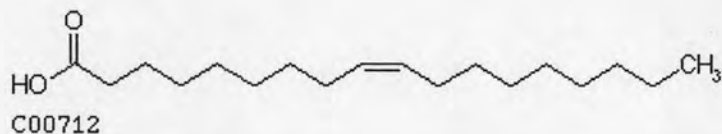
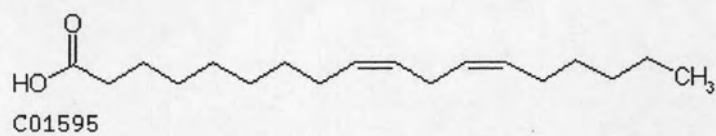


Table C-1.4 The property of critical oleic acid with Lydersen's Structural Contribution

Mw	bp (oC)	bp (K)		N	ΔT	$N\Delta T$	ΔP	$N\Delta P$	ΔV	$N\Delta V$
282.2559	285	558.15	COOH	1	0.085	0.085	0.4	0.4	80	80
			CH3	1	0.02	0.02	0.227	0.227	55	55
			CH2	14	0.02	0.28	0.227	3.178	55	770
			HC=	2	0.018	0.036	0.198	0.396	45	90
			SUM			0.421		4.201		995
			total			0.421		4.201		995

Tc (K)	Pc (atm)	Vc (cm ³ /(g.mol))	Zc
688.43	13.69	1035	0.25

$$R = 82.06 \text{ cm}^3\text{atm/g.mol K}$$

Linoleic acid**Table C-1.5** The property of critical linoleic acid with Lydersen's Structural Contribution

Mw	bp (oC)	bp (K)		N	ΔT	N ΔT	ΔP	N ΔP	ΔV	N ΔV
280.4455	229.445	502.595	COOH	1	0.085	0.085	0.4	0.4	80	80
			CH3	1	0.02	0.02	0.227	0.227	55	55
			CH2	12	0.02	0.24	0.227	2.724	55	660
			HC=	4	0.018	0.072	0.198	0.792	45	180
			SUM			0.417		4.143		975
			total			0.417		4.143		975

Tc (K)	Pc (atm)	Vc (cm ³ /(g.mol))	Zc
620.40	13.95	1015	0.28

$$R = 82.06 \text{ cm}^3\text{atm/g.mol K}$$

Table C-1.6 The critical property of total fatty acid by average with fraction fatty acids

Fatty acids	T _c	%FFA	MW	Mol	Mol%	% Fraction	T _c Fraction	Pc	Pc Fraction	Vc	Vc Fraction	Zc	Zc Fraction
Palmitic	607.32	42.8	256.42	0.1669	46.0786	0.4608	279.8443	14.92	6.8749	945	435.44	0.22	0.1014
Stearic	695.30	4.5	284.47	0.0158	4.3670	0.0437	30.3637	13.45	0.5874	1055	46.07	0.22	0.0096
oleic	688.43	40.5	282.25	0.1435	39.6121	0.3961	272.7017	13.69	5.4229	990	392.16	0.23	0.0911
linoleic	620.40	10.1	280.44	0.0360	9.9423	0.0994	61.6822	13.95	1.3870	925	91.97	0.3	0.0298
Total	2611.45	97.9		0.3622		1	644.5919		14.2721		965.64		0.2319

The critical point of mixture between palm fatty acids with methanol used Lorentz-Berthelot-type rules for binary mixture method, the data in the table C-1.6 apply in the equation the critical point of mixture binary system

Critical point of mixture (binary system)

The critical points of mixture were calculated by Lorentz-Berthelot-type mixing rules as following equations:

$$T_{cm} V_{cm} = \sum_i \sum_j x_i x_j T_{ij} V_{ij} = x_i^2 T_{ci} V_{ci} + 2x_i x_j T_{cij} V_{cij} + x_j^2 T_{cj} V_{cj}$$

$$V_{cm} = \sum_i \sum_j x_i x_j V_{cij} = x_i^2 V_{ci} + 2x_i x_j V_{cij} + x_j^2 V_{cj}$$

$$Z_{cm} = \sum_i \sum_j x_i x_j Z_{cij} = x_i^2 Z_{ci} + 2x_i x_j Z_{cij} + x_j^2 Z_{cj}$$

$$P_{cm} = \frac{Z_{cm} R T_{cm}}{V_{cm}}$$

The terms of T_{cij} , V_{cij} and Z_{cij} were calculated by the combining rule as the following equations:

$$T_{cij} = \sqrt{T_{ci} T_{cj}}$$

$$P_{cij} = \frac{1}{V_{cij}} \sqrt{P_{ci} P_{cj} V_{ci} V_{cj}}$$

$$Z_{cij} = 0.5(Z_{ci} + Z_{cj})$$

$$V_{cij}^{1/3} = \frac{1}{2}(V_{ci}^{1/3} + V_{cj}^{1/3})$$

Where

i and j = subscripts for component i and j

x = mole fraction

T_{cm} = critical temperature of mixture

V_{cm} = critical volume of mixture

P_{cm} = critical pressure of mixture

Z_{cm} = critical compressibility factor of mixture

Example the critical point of mixture at the molar ratio 1:6

	mole	mole fraction
MeOH	6	0.86
PFA	1	0.14
total	7	1

	Tc (K)	Pc (atm)	Vc (L/mol)	Zc
MeOH	512.6	79.9	0.118	0.224
PFA	644.59	14.27	0.96	0.23

$$\begin{aligned}
 T_{cij} &= 574.819 \text{ K} & 301.819 \text{ C} \\
 V_{cij}^{1/3} &= 0.73849 & V_{cij} = 0.402742 \\
 P_{cij} &= 28.2186 \\
 Z_{cij} &= 0.227 \\
 \\
 V_{cm} &= 0.20 \\
 T_{cm} V_{cm} &= 113.763 & T_{cm} = 555.17 \text{ K} \\
 & & 282.17 \text{ C} \\
 Z_{cm} &= 0.22 \\
 P_{cm} &= 48.89
 \end{aligned}$$

VITA

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