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APPENDICES

Appendix A Chemical Properties

Table A1 Chemical Properties

Chemical	**Full name**	**Density (g/cm^3)**	**Mw (g/mole)**
DVB	Ddivinylbenzene	0.914	130
VBC	Vinyl benzyl chloride	1.074	152.6
Toluene	Toluene	0.8669	92
Span80	Sorbitan monooleate	0.995 ~ 1.0	428.6
CTAB	Cetyl trimethyl ammonium bromide	-	364.5
DDBSS	Dodecylbenzene sulfonic acid sodium salt	-	348.5
H_2O	Water	1	18
$CaCl_2.2H_2O$	Calcium chloride dihydrate	-	147
$K_2S_2O_8$	Potassium persulfate	2.477	270.3
50 wt% PEI	Polyethyleneimine	0.9 ~ 1.08	1200
Salicylaldehyde	Salicylic aldehyde	1.146	122.12

Appendix B PolyHIPEs Preparation

- Preparation of polyHIPE with different monomer ratio and 0 wt% PEI

Table B1 Theoretical weight and volume of each component

Oil phase (total 2 mL)						Aqueous phase (total 18 mL)		
DVB (mL)	VBC (mL)	Toluene (mL)	SPAN80 (g)	CTAB (g)	DDBSS (g)	$CaCl_2.2H_2O$ (g)	$K_2S_2O_8$ (g)	H_2O (mL)
1.00	**0.00**	**1.0**	**0.3600**	**0.0171**	**0.0229**	**0.2000**	**0.0400**	**18**
1.00	0.00	1.0	0.3599	0.0172	0.0233	0.2001	0.0401	18
1.00	0.00	1.0	0.3605	0.0170	0.0226	0.2004	0.0402	18
1.00	0.00	1.0	0.3603	0.0171	0.0231	0.2000	0.0407	18
0.90	**0.10**	**1.0**	**0.3600**	**0.0171**	**0.0229**	**0.2000**	**0.0400**	**18**
0.90	0.10	1.0	0.3602	0.0174	0.0231	0.2000	0.0404	18
0.90	0.10	1.0	0.3606	0.0175	0.0230	0.2004	0.0401	18
0.90	0.10	1.0	0.3608	0.0171	0.0228	0.2007	0.0403	18
0.80	**0.20**	**1.0**	**0.3600**	**0.0171**	**0.0229**	**0.2000**	**0.0400**	**18**
0.80	0.20	1.0	0.3604	0.0174	0.0230	0.2001	0.0401	18
0.80	0.20	1.0	0.3609	0.0172	0.0228	0.2002	0.0400	18
0.80	0.20	1.0	0.3610	0.0170	0.0234	0.2008	0.0401	18
0.70	**0.30**	**1.0**	**0.3600**	**0.0171**	**0.0229**	**0.2000**	**0.0400**	**18**
0.70	0.30	1.0	0.3598	0.0178	0.0230	0.2001	0.0409	18
0.70	0.30	1.0	0.3601	0.0171	0.0229	0.2001	0.0401	18
0.70	0.30	1.0	0.3605	0.0173	0.0233	0.2003	0.0402	18
0.60	**0.40**	**1.0**	**0.3600**	**0.0171**	**0.0229**	**0.2000**	**0.0400**	**18**
0.60	0.40	1.0	0.3602	0.0171	0.0229	0.2000	0.0402	18
0.60	0.40	1.0	0.3601	0.0172	0.0234	0.2001	0.0401	18
0.60	0.40	1.0	0.3607	0.0169	0.0231	0.2003	0.0405	18
0.50	**0.50**	**1.0**	**0.3600**	**0.0171**	**0.0229**	**0.2000**	**0.0400**	**18**
0.50	0.50	1.0	0.3603	0.0170	0.0233	0.2002	0.0402	18
0.50	0.50	1.0	0.3609	0.0172	0.0232	0.2001	0.0400	18
0.50	0.50	1.0	0.3600	0.0171	0.0233	0.2001	0.0401	18

Note: "**Bold**" number = Theoretical value

"Normal" number = Actual value

- Preparation of polyHIPE with different monomer ratio and 10 wt% PEI

Table B3 Theoretical weight and volume of each component

Oil phase (total 2 mL)						Aqueous phase (total 18 mL)				
DVB (mL)	VBC (mL)	Toluene (mL)	SPAN80 (g)	CTAB (g)	DDBSS (g)	$CaCl_2.2H_2O$ (g)	$K_2S_2O_8$ (g)	30 wt% PEI stock solution	H_2O (mL)	wt% PEI
1.0	**0.0**	**1.0**	**0.3600**	**0.0171**	**0.0229**	**0.2000**	**0.0400**	**6**	**12**	**10**
1.0	0.0	1.0	0.3607	0.0174	0.0224	0.2001	0.0402	6	12	10
1.0	0.0	1.0	0.3603	0.0171	0.0231	0.2000	0.0409	6	12	10
1.0	0.0	1.0	0.3601	0.0175	0.0230	0.2008	0.0406	6	12	10
0.9	**0.1**	**1.0**	**0.3600**	**0.0171**	**0.0229**	**0.2000**	**0.0400**	**6**	**12**	**10**
0.9	0.1	1.0	0.3598	0.0172	0.0230	0.2004	0.0401	6	12	10
0.9	0.1	1.0	0.3605	0.0174	0.0231	0.2002	0.0405	6	12	10
0.9	0.1	1.0	0.3606	0.0175	0.0230	0.2009	0.0401	6	12	10
0.8	**0.2**	**1.0**	**0.3600**	**0.0171**	**0.0229**	**0.2000**	**0.0400**	**6**	**12**	**10**
0.8	0.2	1.0	0.3601	0.0178	0.0229	0.2001	0.0405	6	12	10
0.8	0.2	1.0	0.3600	0.0173	0.0228	0.2003	0.0402	6	12	10
0.8	0.2	1.0	0.3597	0.0175	0.0233	0.2009	0.0401	6	12	10
0.7	**0.3**	**1.0**	**0.3600**	**0.0171**	**0.0229**	**0.2000**	**0.0400**	**6**	**12**	**10**
0.7	0.3	1.0	0.3604	0.0172	0.0232	0.2002	0.0402	6	12	10
0.7	0.3	1.0	0.3602	0.0171	0.0229	0.2000	0.0402	6	12	10
0.7	0.3	1.0	0.3607	0.0171	0.0234	0.2001	0.0403	6	12	10
0.6	**0.4**	**1.0**	**0.3600**	**0.0171**	**0.0229**	**0.2000**	**0.0400**	**6**	**12**	**10**
0.6	0.4	1.0	0.3602	0.0171	0.0226	0.2001	0.0400	6	12	10
0.6	0.4	1.0	0.3606	0.0173	0.0230	0.2003	0.0403	6	12	10
0.6	0.4	1.0	0.3608	0.0170	0.0233	0.2002	0.0401	6	12	10
0.5	**0.5**	**1.0**	**0.3600**	**0.0171**	**0.0229**	**0.2000**	**0.0400**	**6**	**12**	**10**
0.5	0.5	1.0	0.3607	0.0174	0.0228	0.2000	0.0402	6	12	10
0.5	0.5	1.0	0.3605	0.0172	0.0231	0.2004	0.0401	6	12	10
0.5	0.5	1.0	0.3608	0.0171	0.0230	0.2001	0.0401	6	12	10

Note: "**Bold**" number = Theoretical value

"Normal" number = Actual value

- Preparation of polyHIPE with monomer ratio 60/40 and different wt% PEI

Table B5 Theoretical weight and volume of each component

Oil phase (total 2 mL)						Aqueous phase (total 18 mL)				
DVB (mL)	VBC (mL)	Toluene (mL)	SPAN80 (g)	CTAB (g)	DDBSS (g)	$CaCl_2.2H_2O$ (g)	$K_2S_2O_8$ (g)	30 wt% PEI stock solution	H_2O (mL)	wt% PEI
0.6	**0.4**	**1.0**	**0.3600**	**0.0171**	**0.0229**	**0.2000**	**0.0400**	**0**	**18**	**0**
0.6	0.4	1.0	0.3605	0.0174	0.0231	0.2002	0.0405	0	18	0
0.6	0.4	1.0	0.3601	0.0171	0.0229	0.2001	0.0401	0	18	0
0.6	0.4	1.0	0.3603	0.0171	0.0231	0.2000	0.0409	0	18	0
0.6	**0.4**	**1.0**	**0.3600**	**0.0171**	**0.0229**	**0.2000**	**0.0400**	**3**	**15**	**5**
0.6	0.4	1.0	0.3608	0.0170	0.0233	0.2002	0.0401	3	15	5
0.6	0.4	1.0	0.3607	0.0174	0.0224	0.2001	0.0402	3	15	5
0.6	0.4	1.0	0.3602	0.0171	0.0229	0.2000	0.0402	3	15	5
0.6	**0.4**	**1.0**	**0.3600**	**0.0171**	**0.0229**	**0.2000**	**0.0400**	**6**	**12**	**10**
0.6	0.4	1.0	0.3606	0.0170	0.0228	0.2001	0.0403	6	12	10
0.6	0.4	1.0	0.3601	0.0175	0.0230	0.2008	0.0406	6	12	10
0.6	0.4	1.0	0.3604	0.0172	0.0233	0.2002	0.0402	6	12	10
0.6	**0.4**	**1.0**	**0.3600**	**0.0171**	**0.0229**	**0.2000**	**0.0400**	**9**	**9**	**15**
0.6	0.4	1.0	0.3601	0.0171	0.0234	0.2001	0.0404	9	9	15
0.6	0.4	1.0	0.3600	0.0173	0.0228	0.2003	0.0402	9	9	15
0.6	0.4	1.0	0.3605	0.0175	0.0235	0.2002	0.0401	9	9	15
0.6	**0.4**	**1.0**	**0.3600**	**0.0171**	**0.0229**	**0.2000**	**0.0400**	**12**	**6**	**20**
0.6	0.4	1.0	0.3602	0.0174	0.0230	0.2002	0.0405	12	6	20
0.6	0.4	1.0	0.3609	0.0173	0.0232	0.2001	0.0403	12	6	20
0.6	0.4	1.0	0.3604	0.0170	0.0228	0.2006	0.0400	12	6	20
0.6	**0.4**	**1.0**	**0.3600**	**0.0171**	**0.0229**	**0.2000**	**0.0400**	**15**	**3**	**25**
0.6	0.4	1.0	0.3607	0.0171	0.0235	0.2003	0.0401	15	3	25
0.6	0.4	1.0	0.3605	0.0173	0.0229	0.2000	0.0402	15	3	25
0.6	0.4	1.0	0.3602	0.0171	0.0230	0.2001	0.0406	15	3	25
0.6	**0.4**	**1.0**	**0.3600**	**0.0171**	**0.0229**	**0.2000**	**0.0400**	**18**	**0**	**30**
0.6	0.4	1.0	0.3605	0.0172	0.0230	0.2004	0.0407	18	0	30
0.6	0.4	1.0	0.3606	0.0171	0.0231	0.2001	0.0402	18	0	30
0.6	0.4	1.0	0.3601	0.0104	0.0233	0.2005	0.0403	18	0	30

Note: "**Bold**" number = Theoretical value, "Normal" number = Actual value

Appendix C Example of Calculation

- Calculate weight of 50 wt% PEI solution for preparing PEI solution for using as an water phase

  (50 wt% PEI solution = 50 g of PEI + 50 g of water)

Example: 10 wt% PEI, 18 mL aqueous solution

Define: weight of PEI must be calculated in term of weight of monomer

For the polyHIPE with DVB/VBC ratio 100/0,

- DVB volume = 1 mL
- DVB density = 0.914 g/mL

Mass = Density x Volume

Hence, weight of monomer (DVB) = 0.914 x 1 = 0.914 g

In order to load 10 wt% PEI into this polyHIPE, weight of pure PEI needed

= 0.914 x (10/100)

= 0.0914 g

The purchased PEI is 50 wt% PEI in water. In order to get 0.0914 g pure PEI, the weight of 50 wt% PEI solution is double.

Weight of 50 wt% PEI solution needed = 0.0914 x 2

= 0.1828 g

The aqueous phase was made up of 0.1828 g of 5%wt PEI solution and distilled water added to reach 18 mL volume. Different amount of PEI in the aqueous phase was calculated and shown in Table C1.

Table C1 Theoretical preparation of PEI solution

PEI solution concentration (wt%)	Volume (mL)	Calculated weight of 50%PEI solution(g)
1	18	0.0183
10	18	0.1828
1	250	0.2539
10	250	2.5390
20	250	5.0780
30	250	7.6170

- Calculation of the Amount of Amine in PEI Solution

Example: At 10 wt% PEI solution (Mw of PEI = 1200 g/mol)

Weight of 50 wt% PEI solution	=	0.1828	g
Thus, weight of pure PEI (exclude water)	=	0.1828/2	
	=	0.0914	g
Mole of PEI	=	g/Mw	
	=	0.0914/1200	
	=	7.617×10^{-5}	mol PEI

1 repeating unit of PEI (with 25% primary amine, 50% secondary amines and 25% tertiary amine (Harpe *et al.*, 2000)) has molecular weight = 172 g/mol, the repeating unit of PEI is shown in Figure C1.

So, PEI 1 molecule (with Mw =1200 g/mol):

- contains = 1200/172 = 7 repeating units
- each repeating unit contains nitrogen (N) 4 atoms
- there are nitrogen (N) or amine in 10 wt% PEI solution
  - = $7 \times 4 \times 7.617 \times 10^{-5}$ mol N
  - = 2.1327×10^{-3} mol N / 7 repeating units
- in each repeating unit, there are primary, secondary and tertiary amine
- The ratio of primary/secondary/tertiary amines are 1/2/1

Thus, mole fraction of primary amine $= \frac{1}{4} \times 2.1327 \times 10 - 3$

$= 5.327 \times 10^{-4}$ mol

mole fraction of secondary amine $= \frac{2}{4} \times 2.1327 \times 10 - 3$

$= 1.065 \times 10^{-3}$ mol

mole fraction of tertiary amine $= \frac{1}{4} \times 2.1327 \times 10 - 3$

$= 5.327 \times 10^{-4}$ mol

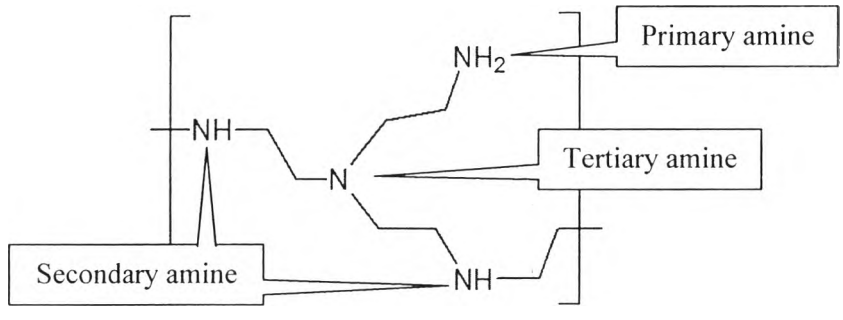

Figure C1 Repeating unit of polyethyleneimine.

Table C2 mmol of primary amine in each 18 mL PEI solution

Percent of PEI solution	Actual weight (g)	mmol of PEI in solution	mmol of overall amines in PEI solution	mmol of primary amine
1	0.0091	0.0076	0.2133	0.0533
5	0.0457	0.0381	1.0663	0.2666
10	0.0914	0.0762	2.1327	0.5332
15	0.1371	0.1143	3.1990	0.7998
20	0.1828	0.1523	4.2653	1.0663
25	0.2285	0.1904	5.3317	1.3329
30	0.2742	0.2285	6.3980	1.5995

- Salicylaldehyde Calculation (For UV-VIS method)

Preparation of 0.02 M salicylaldehyde in methanol

$$= \frac{(0.02\ mol\ salicylaldehyde)(122.12\ g)}{(1\ L\ of\ solution)(1\ mol\ salicylaldehyde)}$$

= 2.4424 g salicylaldehyde/ 1 L solution

1 L solution uses salicylaldehyde = 2.4424 g

250 mL solution uses salicylaldehyde = 2.4424 x (250/1000)

= 0.6106 g salicylaldehyde

the concentration 0.2 M was prepared and then diluted to 0.02 M using Eq. 3.1.

$$C_1V_1 = C_2V_2 \qquad \textbf{Eq. 3.1}$$

$$(0.2\ M)\ (V_1) = (0.02)\ (250\ mL)$$

$$V_1 = 25\ mL$$

The purchased salicylaldehyde has concentration 98 %w/w

If 98 g of salicylaldehyde was in 100 g of this purchased salicylaldehyde

So, there was 6.1060g salicylaldehyde in $= \frac{6.1060\ x\ 100}{98}$

= 6.2306 g salicylaldehyde

- Preparation of 1 wt% Acetic Acid in Water

The purchased acetic acid has concentration 99.8 %w/w

Dilute to 1 wt% and 250 mL by:

$C_1V_1 = C_2V_2$ **Eq. 3.1**

$(99.8\ \%)\ (V_1) = (1\ \%)\ (250\ mL)$

$V_1 = 2.505\ mL$

400 times dilution factor

First: $C_1V_1 = C_2V_2$ **Eq. 3.1**

(0.02 M salicylaldehyde) (0.5 mL) = (C_2) (10 mL)

C_2 = 0.001 M salicylaldehyde

Second: $C_1V_1 = C_2V_2$ **Eq. 3.1**

(0.001 M salicylaldehyde) (0.5 mL) = (C_2) (10 mL)

C_2 = 0.00005 M salicylaldehyde

$$\frac{0.02}{0.00005} = 400 \text{ times}$$

- Calculation the Amount of VBC Reacts with 10 wt% PEI

From 10 wt% PEI solution 18 mL, there is amine = 2.1327×10^{-3} mol (The calculation is shown in page 55)

- 1 mol of primary amine reacts with 1 mol of VBC
- Each PEI repeating unit consists of 25% primary amine, 50% secondary amine and 25% tertiary amines

Mole fraction of primary amine = $\frac{1}{4} \times 2.1327 \times 10 - 3$

= 5.327×10^{-4} mol

Mole fraction of secondary amine = $\frac{2}{4} \times 2.1327 \times 10 - 3$

= 1.065×10^{-3} mol

Mole fraction of tertiary amine = $\frac{1}{4} \times 2.1327 \times 10 - 3$

= 5.327×10^{-4} mol

From 1 mol of primary amine reacts with 1 mol of VBC,

- There is primary amine = 5.327×10^{-4} mol
- Hence, need VBC = 5.327×10^{-4} mol

  (VBC Mw = 152.6 g/mol, density = 1.074 g/mL)
- Weight of VBC = Mw x Mole

  = $152.6 \times 5.327 \times 10^{-4}$

  = 0.081 g VBC
- Volume of VBC = Weight / Density

  = 0.081 / 1.074

  = 0.75 mL VBC

Table C3 Calculation the amount of VBC reacts with 10 wt% PEI

Amine in PEI reacted by VBC	Mol of amine	Used VBC (mol)	Weight of VBC (g)	Volume of VBC (mL)
Only primary amine in PEI	5.33×10^{-4}	5.33×10^{-4}	0.081	0.075
Only primary + secondary amine	1.60×10^{-3}	1.60×10^{-3}	0.244	0.225
All amines	2.13×10^{-3}	2.13×10^{-3}	0.325	0.300

Table C3 UV-VIS results calculated from calibration curve

Solution	Absobance at 255 nm	Residual conc. Of salicylaldehyde in diluted fitrate (mg/L)	400X times dilution factor(mg/L)	Used salicylaldehyde (mg/L)	mmol/L	mmol of used salicylaldehyde in 20 mL	mmol of primary amine in polyHIPE	mmol of overall amine in polyHIPE	Initial amine loaded	Percent of amine in polyHIPE
(a) PolyHIPEs with different monomer ratio and 0 wt% PEI										
0.02M Salicylaldehyde	0.577	6.805	2721.94	0.00	0	0	0	0	0	0
100/0, 0% PEI	0.578	6.818	2727.04	-5.10	0	0	0	0	0	0
75/25, 0% PEI	0.575	6.779	2711.73	10.20	0	0	0	0	0	0
50/50, 0% PEI	0.577	6.805	2721.94	0.00	0	0	0	0	0	0
(b) PolyHIPEs with different monomer ratio and 10 wt% PEI										
100/0, 10% PE	0.570	6.716	2686.22	35.71	0.292	0.006	0.006	0.023	2.133	0.11
90/10, 10% PEI	0.468	5.415	2165.82	556.12	4.554	0.091	0.091	0.364	2.133	1.71
80/20, 10% PEI	0.470	5.440	2176.02	545.92	4.470	0.089	0.089	0.358	2.133	1.68
70/30, 10% PEI	0.475	5.504	2201.53	520.41	4.261	0.085	0.085	0.341	2.133	1.60
60/40, 10% PEI	0.468	5.415	2165.82	556.12	4.554	0.091	0.091	0.364	2.133	1.71
50/50, 10% PEI	0.474	5.491	2196.43	525.51	4.303	0.086	0.086	0.344	2.133	1.61
(c) PolyHIPEs with monomer ratio 60/40 and different wt% PEI										
60/40, 5% PEI	0.488	5.670	2267.86	454.08	3.718	0.074	0.074	0.297	1.066	1.39
60/40, 10% PEI	0.462	5.338	2135.20	586.73	4.805	0.096	0.096	0.384	2.133	1.80
60/40, 15% PEI	0.446	5.134	2053.57	668.37	5.473	0.109	0.109	0.438	3.199	2.05
60/40, 20% PEI	0.413	4.713	1885.20	836.73	6.852	0.137	0.137	0.548	4.265	2.57

Appendix D FTIR wavenumber

Table D1 FTIR wavenumber

Origin	Wavenumber (cm^{-1})	Assignment
		Primary amino
N - H	3400–3380	Aliphatic primary amine, NH stretch
	+3345–3325	
N - H	1650–1590	Primary amine, NH bend
C - N	1090–1020	Primary amine, CN stretch
		Secondary amino
>N - H	3360–3310	Aliphatic secondary amine, NH stretch
>N - H	~3450	Aromatic secondary amine, NH stretch
>N - H	3490–3430	Heterocyclic amine, NH stretch
= N - H	3350–3320	Imino compounds, NH stretch
>N - H	1650–1550	Secondary amine, NH bend
C - N	1190–1130	Secondary amine, CN stretch
	1250-1020	
		Tertiary amino
C - N	1210–1150	Tertiary amine, CN stretch
C - Cl	~1200	Aliphatic chloro compounds,
	800–700	C - Cl stretch
O - H	3570–3200 (broad)	Hydroxy group, H-bonded
		OH stretch
C - H	2820 - 2810	C-H stretch

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Proceedings:

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