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APPENDICES

APPENDIX A
EXPERIMENTAL DATA

Table A-1 TCE degradation using heat activated persulfate oxidation at various persulfate/TCE molar ratios (Initial TCE concentration of 10.95 ppm)

No.	Persulfate/TCE molar ratio	Duration time (hr)	TCE conc. (ppm)			
			1	2	3	Average
1	5:1	1	8.30	8.55	9.13	8.66
2	5:1	2	5.89	5.24	5.74	5.62
3	5:1	3	3.81	9.77	2.25	3.28
4	5:1	4	1.28	1.30	1.50	1.36
5	5:1	5	0.95	1.04	1.11	1.03
6	10:1	1	7.82	8.03	7.95	7.93
7	10:1	2	5.37	5.84	5.27	5.49
8	10:1	3	3.26	3.34	3.39	3.33
9	10:1	4	1.32	1.52	1.73	1.53
10	10:1	5	0.54	1.00	0.66	0.73
11	15:1	1	4.88	5.69	5.77	5.45
12	15:1	2	2.30	2.18	1.70	2.06
13	15:1	3	0.28	0.36	0.50	0.38
14	15:1	4	0.01	0.02	0.01	0.02
15	15:1	5	0.01	0.01	0.01	0.01

Table A-2 TCE removal percentage using the optimal condition of heat activated persulfate oxidation system for degrading 50 ppm and 100 ppm TCE

No.	Persulfate/TCE molar ratio	Initial TCE conc. (ppm)	Duration time (hr)	TCE conc. (ppm)			
				1	2	3	Average
1	5:1	53.7	1	31.2	32.3	32.9	32.1
2	5:1	53.7	2	12.4	13.1	15.0	13.5
3	5:1	53.7	3	4.7	3.3	8.5	5.5
4	5:1	53.7	4	0.1	0.1	0.1	0.1
5	5:1	53.7	5	0.1	0.1	0.1	0.1
6	10:1	104.2	1	53.8	52.2	55.0	53.6
7	10:1	104.2	2	31.2	32.3	26.7	30.1
8	10:1	104.2	3	12.8	12.9	14.4	13.4
9	10:1	104.2	4	1.0	0.8	1.6	1.1
10	10:1	104.2	5	0.1	0.1	0.1	0.1

Table A-3 TCE degradation using UV activated persulfate oxidation at various persulfate/TCE molar ratios

No.	Persulfate/TCE molar ratio	Initial TCE conc. (ppm)	Duration time (hr)	TCE conc. (ppm)			
				1	2	3	Average
1	5:1	10.20	1	9.12	8.36	9.01	8.83
2	5:1	10.20	2	6.46	5.50	5.56	5.84
3	5:1	10.20	3	3.89	3.52	2.85	3.42
4	5:1	10.20	4	1.97	2.17	1.58	1.91
5	5:1	10.20	5	1.32	0.69	0.60	0.87
6	10:1	9.80	1	5.48	5.25	5.38	5.36
7	10:1	9.80	2	1.38	0.78	1.08	1.08
8	10:1	9.80	3	0.27	0.02	0.04	0.11
9	10:1	9.80	4	0.05	0.02	0.04	0.04
10	10:1	9.80	5	0.01	0.02	0.02	0.02
11	15:1	10.10	1	6.25	6.14	5.99	6.12
12	15:1	10.10	2	3.00	3.32	2.68	3.00
13	15:1	10.10	3	0.24	0.07	0.38	0.22
14	15:1	10.10	4	0.03	0.02	0.09	0.05
15	15:1	10.10	5	0.02	0.01	0.02	0.02

Table A-4 TCE degradation using the optimal condition of UV activated persulfate oxidation for degrading 50 ppm and 100 ppm TCE

No.	Initial TCE conc. (ppm)	Duration time (hr)	TCE conc. (ppm)			
			1	2	3	Average
1	51.1	1	42.2	38.2	38.9	39.8
2	51.1	2	29.2	25.8	25.9	27.0
3	51.1	3	20.4	15.2	16.6	17.4
4	51.1	4	7.6	10.0	10.9	9.5
5	51.1	5	5.9	2.4	6.3	4.8
6	102.4	1	75.5	85.4	71.6	77.5
7	102.4	2	57.0	58.9	57.0	57.6
8	102.4	3	42.8	31.5	34.2	36.2
9	102.4	4	16.7	19.0	20.8	18.8
10	102.4	5	3.8	3.9	5.4	4.4

Table A-5 TCE degradation by persulfate oxidation without heat and UV activated condition at various persulfate/TCE molar ratios (Initial TCE concentration of 9.35 ppm)

No.	Persulfate/TCE molar ratio	Duration time (hr)	TCE conc. (ppm)			
			1	2	3	Average
1	5:1	1	8.05	7.18	7.18	7.47
2	5:1	2	7.55	7.46	7.65	7.55
3	5:1	3	6.93	7.91	7.42	7.42
4	5:1	4	7.12	6.43	6.86	6.80
5	5:1	5	6.23	6.49	6.17	6.30
6	10:1	1	9.05	8.46	8.16	8.56
7	10:1	2	7.62	7.29	8.55	7.82
8	10:1	3	7.26	8.30	7.89	7.82
9	10:1	4	7.22	7.32	7.38	7.31
10	10:1	5	5.90	6.48	6.51	6.30
11	15:1	1	8.68	7.62	9.05	8.45
12	15:1	2	8.27	8.01	9.27	8.52
13	15:1	3	7.02	7.28	7.76	7.35
14	15:1	4	9.42	5.90	7.11	7.48
15	15:1	5	6.29	6.31	5.16	5.92

Table A-6 TCE remaining percentage of blanks at different reaction time

Sample Name	% TCE remaining at different treatment time				
	1 hr	2 hrs	3 hrs	4 hrs	5 hrs
X _{1,1}	99.9	98.7	98.2	94.5	93.3
X _{1,2}	99.8	98.7	99	98.1	96.3
X _{1,3}	98.9	95.9	93.5	92.9	92.1
X _{2,1}	100.0	99.4	95.8	95.6	93.8
X _{2,2}	99.8	97.6	96.5	94.1	93.2
X _{2,3}	99.7	98.5	93.4	95.2	92.8

(X_{a,b} ; a = persulfate oxidation method ; 1= activated by heat ; 2 = activated by UV

, b = concentration of TCE ; 1= 10 ppm, 2 = 50 ppm ; 3 = 100 ppm)

Chromatogram of CO₂ standards and blank

R_f of CO₂ is approximately 1.130



Figure A-1 Chromatogram of blank (air) in the analysis of CO₂



Figure A-2 Chromatogram of 4.205 ppm CO₂



Figure A-3 Chromatogram of 33.604 ppm CO₂



Figure A-4 Chromatogram of 67.208 ppm CO₂



Figure A-5 Chromatogram of 100.812 ppm CO₂

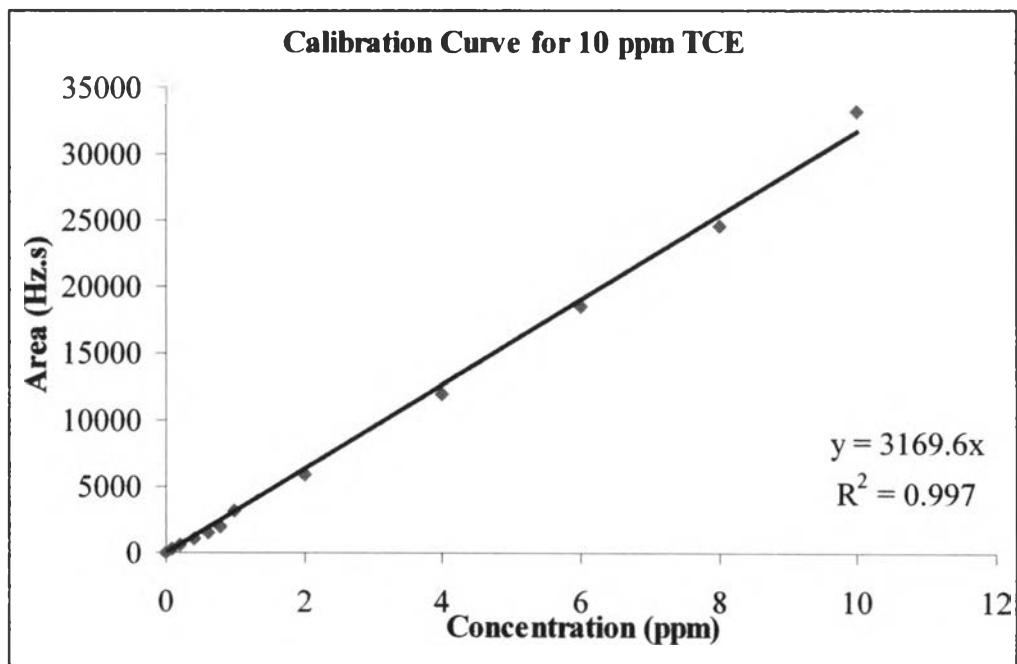


Figure A-6 Chromatogram of 134 ppm CO₂

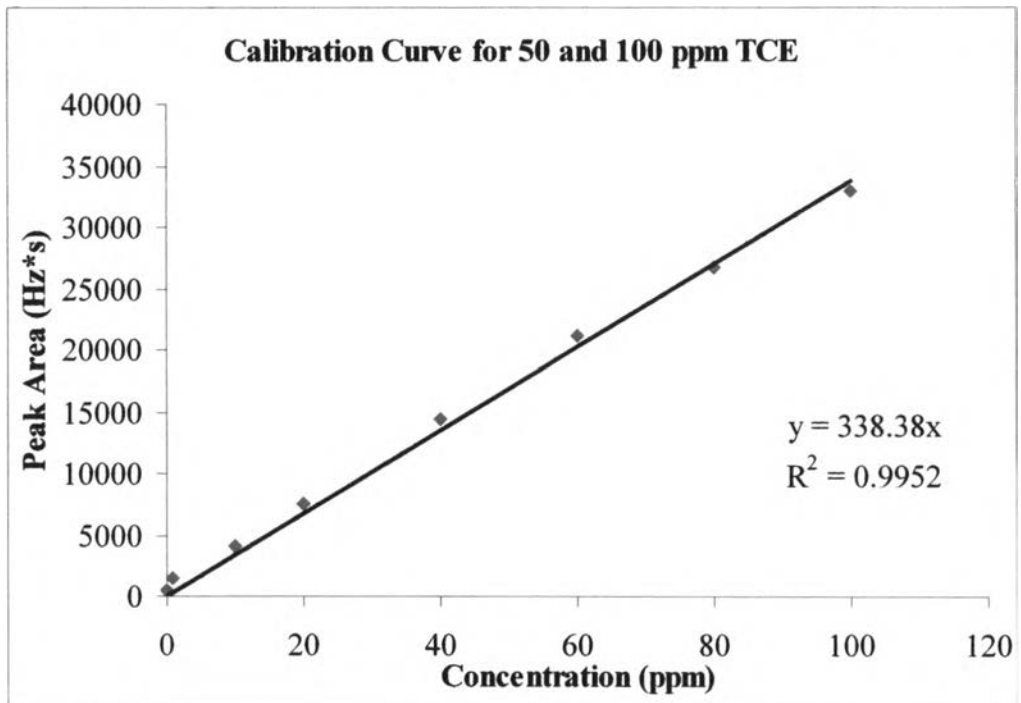
APPENDIX B
CALIBRATION CURVE

Calibration curve of TCE

A calibration curve was developed for 10 ppm TCE and 100 ppm TCE, then the standards were diluted to obtain the desired concentration and extracted like samples (triplicate per each). The calibration standards were analyzed similar to sample procedures. Calibration curve was shown in FigureB-1 and Figure B-2.



FigureB-1 Calibration curve for 10 ppm TCE (Detection limit = 0.01 ppm)



FigureB-2 Calibration curve for 50 and 100 ppm TCE (Detection limit = 0.1 ppm)

APPENDIX C
UV INTENSITY MEASUREMENT

UV intensity measurement

UV intensities were measured to determine potential energy of the UV lamp configuration (See Figure C-1) for scale-up reactor design. UV intensities were measured by ferrioxalate actinometer at every sample position. Average intensity of this system was 10.96 μ Einstein/s (SD = 0.94, CV = 9 %). UV intensities at different sample positions (Figure C-1 and Table C-1) were grouped as in Figure C-2 to C-5. In groups of X, Y, and Z, UV intensities of group X was the lowest and those of group Z was the highest showing that intensities depended on the distance from samples to center of UV lamps. Farther distance from lamp center provided lower intensity.

Due to the fact that UV intensities were different depending on the distance from lamp center, experiment for group A, B, C, and D was set to determine its effects. Sets of 3 samples were experimented at 4 sets of positions (A, B, C, and D). From Table C-2, concentrations of TCE of each set were not different so much according to the values of CV which were less than 13% (the value of CV, which was less than 15 %, indicated that there was no significant difference in this measurement). Hence, the variation of intensities did not significantly affect the oxidation processes due to relatively constant concentrations of residual TCE.

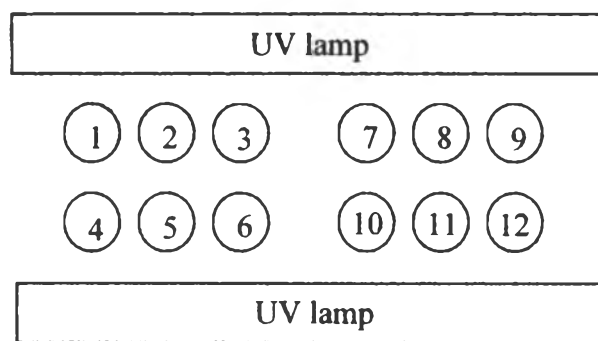


Figure C-1 Position of samples in the UV activated persulfate oxidation system

Table C-1 UV intensity of UV persulfate oxidation system at every sample position

Sample Position (Group of sample)	UV intensity (μEinstein/s)
1 (X, A)	10.06
2 (Y, A)	10.92
3 (Z, A)	12.90
4 (X, B)	9.54
5 (Y, B)	10.42
6 (Z, B)	13.03
7 (Z, C)	12.95
8 (Y, C)	10.14
9 (X, C)	9.81
10 (Z, D)	12.40
11 (Y, D)	9.90
12 (X, D)	9.48

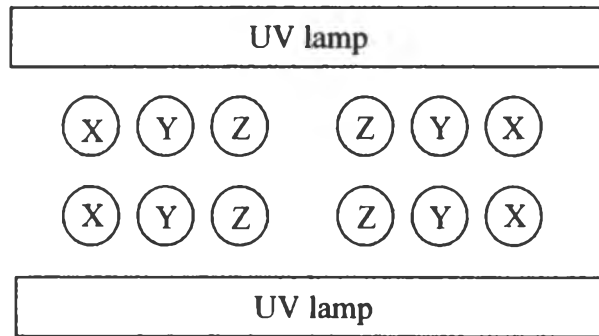


Figure C-2 Position of groups of similarity

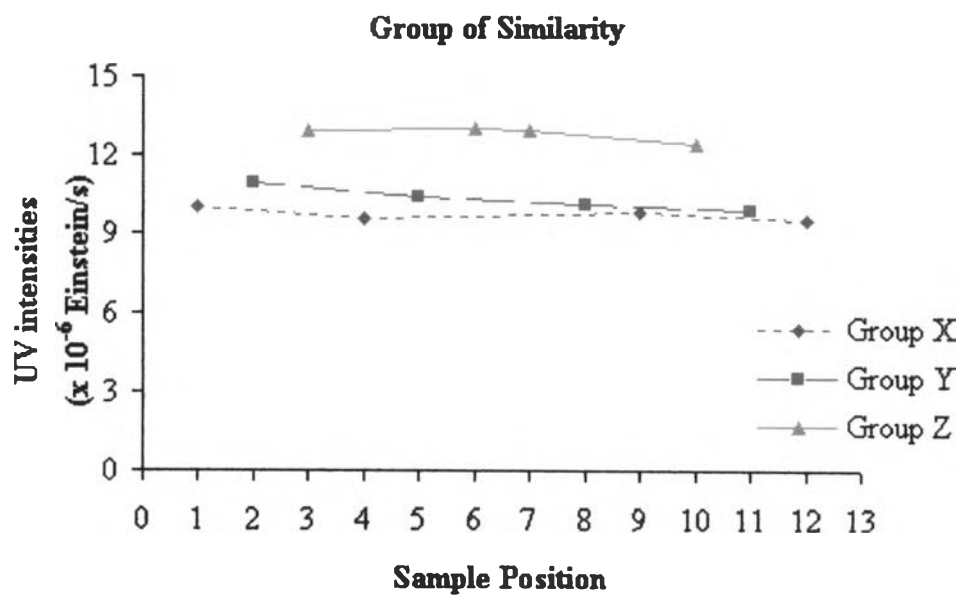


Figure C-3 UV intensities of groups of similarity

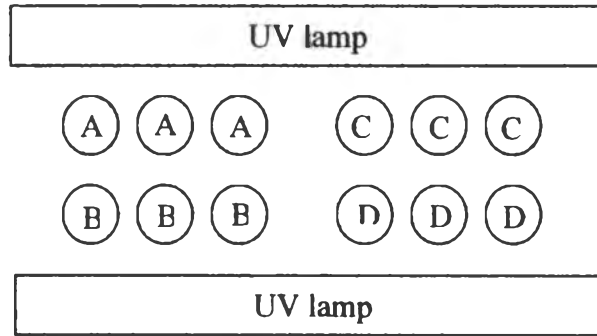


Figure C-4 Position of groups of difference

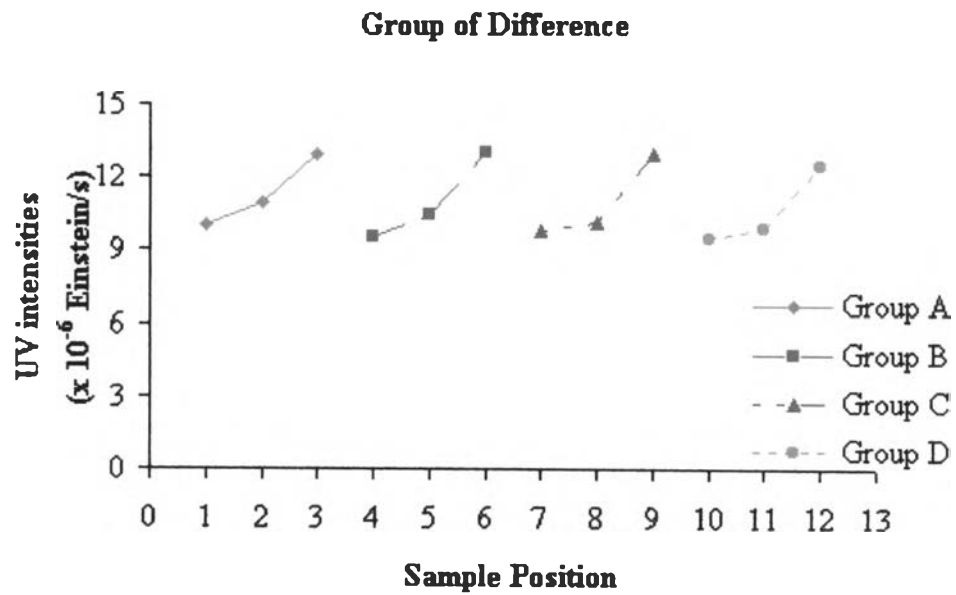


Figure C-5 UV intensities of groups of difference

Table C-2 Normalized concentrations of TCE (C/C_0) in UV persulfate oxidation system at each group of difference

Group	C/C_0				SD	CV
	Sample 1	Sample 2	Sample 3	Average		
A	0.894	0.820	0.884	0.866	0.033	3.8 %
B	0.634	0.540	0.545	0.573	0.043	7.5 %
C	0.381	0.345	0.279	0.335	0.042	12.6 %
D	0.193	0.213	0.155	0.187	0.024	12.7 %

APPENDIX D
CALCULATIONS

Preparation of synthetic samples

Given data

Molecular weight : $C_2HCl_3 = 131.39$

Density : $C_2HCl_3 = 1.465 \text{ g/mL @ } 20^\circ\text{C}$

1) Volume of TCE in 50 mL of 500 ppm TCE stock solution

$$= (500 \text{ mg/L} * 0.05 \text{ L}) * 1 \text{ mL}/1465 \text{ mg}$$

$$= 0.01706 \text{ mL}$$

$$= 17.06 \mu\text{L}$$

2) Volume of TCE in 250 mL of 500 ppm TCE stock solution

$$= (500 \text{ mg/L} * 0.25 \text{ L}) * 1 \text{ mL}/1465 \text{ mg}$$

$$= 0.085324 \text{ mL}$$

$$= 85.32 \mu\text{L}$$

3) Volume of 500 ppm TCE stock solution in 50 mL of 10 ppm TCE

$$C_1V_1 = C_2V_2$$

$$(10 \text{ ppm})(50 \text{ mL}) = (500 \text{ ppm})(V_2) ;$$

$$V_2 = 1 \text{ mL}$$

4) Volume of 500 ppm TCE stock solution in 50 mL of 50 ppm TCE

$$V_2 = (50 \text{ ppm} * 50 \text{ mL})/500 \text{ ppm}$$

$$V_2 = 5 \text{ mL}$$

5) Volume of 500 ppm TCE stock solution in 50 mL of 100 ppm TCE

$$V_2 = (100 \text{ ppm} * 50 \text{ mL})/500 \text{ ppm}$$

$$V_2 = 10 \text{ mL}$$

For 10 ppm TCE, a synthetic sample was prepared by adding 1 mL of 500 ppm TCE in 50 mL vial with 48.5 mL of DI water. For 50 ppm TCE, a synthetic sample was prepared by adding 5 mL of 500 ppm TCE in 50 mL vial with 44.5 mL of DI water. For 100 ppm TCE, a synthetic sample was prepared by adding 10 mL of 500 ppm TCE in 50 mL vial with 39.5 mL of DI water. Then, 0.5 mL of persulfate solution was added to react with TCE.

Preparation of persulfate and thiosulfate solutions for 10 ppm TCE

Given data

Molecular weight : $C_2HCl_3 = 131.39$, $Na_2S_2O_8 = 238.10$, $Na_2S_2O_3 \cdot 5H_2O = 248.18$

Water solubility : $Na_2S_2O_8 = 556$ g/L @ 20°C , $Na_2S_2O_3 = 790$ g/L @ 4°C

1) Amount of 10 ppm TCE in a 50 ml vial

$$= (10 \text{ mg/L} * 0.05 \text{ L}) * 1 \text{ g/1000 mg}$$

$$= 0.0005 \text{ g} = 0.0005 \text{ g} * 1 \text{ mol/} 131.39 \text{ g} = 3.8 * 10^{-6} \text{ mol}$$

2) Amount of persulfate

➤ The molar ratio of persulfate and contaminant = 5:1

- Amount of persulfate in a 50 ml vial

$$= 5 * 3.8 * 10^{-6} \text{ mol} * 238.1 \text{ g/mol} * 1000 \text{ mg/1 g} = 4.53 \text{ mg}$$

$$[\text{Persulfate}] = 90.61 \text{ ppm}$$

- Adding 0.5 ml from persulfate stock solution

$$C_1V_1 = C_2V_2$$

$$(4.53 \text{ mg}) = (C_2)(0.0005 \text{ L}) ; C_2 = 9,061 \text{ mg/L}$$

Therefore, preparing 9,061 ppm persulfate stock solution was done by adding 453 mg in 50 mL

- The molar ratio of persulfate and contaminant = 10:1
 - Amount of sodium persulfate in a 50 ml vial = 9.06 mg
 - [Persulfate] = 181.2 ppm
 - Adding 0.5 ml of 18,122 ppm persulfate stock solution
 - Adding 906.1 mg in 50 mL
- The molar ratio of persulfate and contaminant = 15:1
 - Amount of sodium persulfate in a 50 ml vial = 13.59 mg
 - [Persulfate] = 271.8 ppm
 - Adding 0.5 ml of 27,182 ppm persulfate stock solution
 - Adding 1.3591 g in 50 mL

3) Amount of sodium thiosulfate for quenching reaction

Stoichiometric reaction between persulfate and thiosulfate is as followed;



Adding 3 moles of sodium thiosulfate in excess for quenching a mole of sodium persulfate

- The molar ratio of thiosulfate, persulfate and contaminant = 15 : 5 : 1
 - Amount of sodium thiosulfate in a 50 ml vial
 - = $15 * 3.8 * 10^{-6} \text{ mol} * 248.18 \text{ g/mol} * 1000 \text{ mg/1 g} = 14.16 \text{ mg}$
- The molar ratio of thiosulfate, persulfate and contaminant = 30 : 10 : 1
 - Amount of sodium thiosulfate in a 50 ml vial = 28.33 mg
- The molar ratio of thiosulfate, persulfate and contaminant = 45 : 15 : 1
 - Amount of sodium thiosulfate in a 50 ml vial = 42.50 mg

Table C-1 Calculation for preparation of thiosulfate solution quenching 10 ppm TCE

Amount of Na ₂ S ₂ O ₃ in a 50 ml vial (mg)	Concentration (ppm) of stock solution when adding			Stock solution for 0.1mL	
	1 μ L	10 μ L	100 μ L / 0.1 mL	g in 50 mL	g in 25 mL
14.16	14,166,603	1,416,660	141,666	7.0833	3.54165
28.33	28,333,206	2,833,321	283,332	14.1666	7.0833
42.50	42,499,810	4,249,981	424,998	21.2499	10.62495

Adding 0.1 ml of 141.7, 283.3 and 425.0 g/L sodium thiosulfate stock solutions for ratio 15:5:1, 30:10:1 and 45:15:1, respectively was the best solution for adding less volume as possible due to water solubility of sodium thiosulfate.

For 50 ppm TCE and 100 ppm TCE, preparation of persulfate and thiosulfate solutions were calculated with the same pattern.

Energy consumption of heat and UV activated persulfate oxidation system

In heat activated persulfate oxidation system, the amount of heat energy was calculated from temperature change and heat capacity as follows (Tipler and Mosca, 2003).

$$Q = C\Delta T = mc\Delta T \dots\dots\dots(1)$$

(Q= the amount of heat energy (kJ), C = the heat capacity (kJ/K), m = the mass of substance (kg), c = the specific heat or heat capacity per unit mass (kJ/(kg.K)))

Because wastewater used in this study was mixture of water and TCE, equation for calculation of energy will be as followed (Beckermann and Viskanta, 1987).

$$Q = C\Delta T = m\bar{c}\Delta T \dots\dots\dots(2)$$

$$\bar{c} = \frac{\epsilon\rho_1c_1 + (1-\epsilon)\rho_2c_2}{\bar{\rho}} \dots\dots\dots(3)$$

$$\bar{\rho} = \epsilon\rho_1 + (1-\epsilon)\rho_2 \dots\dots\dots(4)$$

(\bar{c} = the average specific heat, ρ = density (g/L), $\bar{\rho}$ = the average density (g/L), ϵ = liquid volume fraction)

Since liquid volume fraction of TCE was much less than that of water (6.83×10^{-5} for 100 ppm TCE and 0.9999, respectively), c in this case should be c_{water} which is 4.184 kJ/kg.K (Tipler and Mosca, 2003).

The amount of energy of each sample is as follows;

$$\begin{aligned} Q &= (0.05 \text{ kg})(4.184 \text{ kJ/kg.K})(40-25\text{K}) \\ &= 3.138 \text{ kJ} \end{aligned}$$

Anyhow, in the contaminated site, volume of water should not be only 0.05L. 1000 L of water was assumed to compare with UV persulfate oxidation method .

$$\begin{aligned} Q &= (1000 \text{ kg})(4.184 \text{ kJ/kg.K})(40-25\text{K}) \\ &= 62,760 \text{ kJ} \end{aligned}$$

In UV activated persulfate oxidation system, the amount of required energy was E_{EO} (electric energy per order, $\text{kWh order}^{-1} \text{ m}^{-3}$) (Oppenländer, 2003).

$$\begin{aligned} E_{EO} &= P_{el}t10^3/V\log([C]_{t=0}/[C]_t) \\ &= (0.02\text{kW} \times 5 \text{ hrs} \times 1000)/(1000 \times \log (100/1)) \\ &= 0.05 \text{ kWh order}^{-1} \text{ m}^{-3} \\ &= 180 \text{ kJ order}^{-1} \text{ m}^{-3} \end{aligned}$$

(P_{el} = electric energy supplied (kW), t = time (hr), V = volume of water treated in the time t (L), $[C]_{t=0}$ = initial TCE concentration, and $[C]_t$ = TCE concentration at time t , Assumption : $P_{el} = 20 \text{ W}$, $t = 5 \text{ hrs}$, % removal = 99% (according to the experiment), $V = 1000 \text{ L}$)

From the study of effects of concentration of TCE on UV activated persulfate oxidation system, at higher temperature, 20 W of lamp power was not enough to degrade TCE effectively compared with heat persulfate oxidation method. If lamp power is increased to 1 kW, E_{EO} will be $9,000 \text{ kJ order}^{-1} \text{ m}^{-3}$.

Concentration of 10 ppm TCE in gas and aqueous phase in a sample vial

Given data

Molecular weight (C_2HCl_3) = 131.39

Henry's law constant of TCE (H_{TCE}) = $9.09 \times 10^{-3} \text{ atm}\cdot\text{m}^3/\text{mol}$ @ 25°C

= $17.82 \times 10^{-3} \text{ atm}\cdot\text{m}^3/\text{mol}$ @ 40°C

$R = 8.205 \times 10^{-5} \text{ atm}\cdot\text{m}^3/\text{mol}\cdot\text{K}$, Liquid volume = 50 mL, Gas volume = 7 mL

The unchanging number of moles of TCE in the system is

$$n_{\text{tot,TCE}} = 3.8 \times 10^{-6} \text{ mol}$$

At equilibrium, this mass of TCE is distributed between the gas and aqueous phases in accordance with Henry's law (Benjamin, 2002):

$$n_{\text{TCE,tot}} = n_{\text{TCE,gas}} + n_{\text{TCE,solution}} = \frac{P_{\text{TCE(g)}} V_{\text{gas}}}{RT} + \frac{P_{\text{TCE(g)}} V_{\text{solution}}}{H_{\text{TCE}}}$$

$$P_{\text{TCE(g)}} = n_{\text{TCE,tot}} \times RT H_{\text{TCE}} / (H_{\text{TCE}} V_{\text{gas}} + V_{\text{solution}} RT) \quad \dots\dots\dots(1)$$

$$[\text{TCE}_{(\text{ag})}] = P_{\text{TCE(g)}}/H_{\text{TCE}} \quad \dots\dots\dots(2)$$

At 25°C

$$P_{\text{TCE(g)}} = \frac{3.8 \times 10^{-6} \text{ mol} \times 8.205 \times 10^{-5} \text{ atm}\cdot\text{m}^3/\text{mol}\cdot\text{K} \times 298 \text{ K} \times 9.09 \times 10^{-3} \text{ atm}\cdot\text{m}^3/\text{mol}}{(9.09 \times 10^{-3} \text{ atm}\cdot\text{m}^3/\text{mol} \times 7 \times 10^{-6} \text{ m}^3) + (50 \times 10^{-6} \text{ m}^3 \times 8.205 \times 10^{-5} \text{ atm}\cdot\text{m}^3/\text{mol}\cdot\text{K} \times 298 \text{ K})}$$

$$= 6.58 \times 10^{-4} \text{ atm}$$

$$[\text{TCE}_{(\text{ag})}] = 6.58 \times 10^{-4} \text{ atm} / 9.09 \times 10^{-3} \text{ atm}\cdot\text{m}^3/\text{mol}$$

$$= 0.072 \text{ mol/m}^3 \times 131.39 \times 10^3 \text{ mg/mol} \times \text{m}^3/1000\text{L} = 9.50 \text{ mg/L}$$

$$[\text{TCE}_{(\text{g})}] = (10 \text{ mg/L} - 9.50 \text{ mg/L}) \times 0.05 \text{ L} / 0.007 \text{ L} = 3.54 \text{ mg/L}$$

At 40°C

$$P_{\text{TCE(g)}} = \frac{3.8 \times 10^{-6} \text{ mol} \times 8.205 \times 10^{-5} \text{ atm} \cdot \text{m}^3 / \text{mol} \cdot \text{K} \times 313 \text{ K} \times 17.82 \times 10^{-3} \text{ atm} \cdot \text{m}^3 / \text{mol}}{(17.82 \times 10^{-3} \text{ atm} \cdot \text{m}^3 / \text{mol} \times 7 \times 10^{-6} \text{ m}^3) + (50 \times 10^{-6} \text{ m}^3 \times 8.205 \times 10^{-5} \text{ atm} \cdot \text{m}^3 / \text{mol} \cdot \text{K} \times 313 \text{ K})}$$

$$= 12.36 \times 10^{-4} \text{ atm}$$

$$[\text{TCE}_{(\text{ag})}] = 12.36 \times 10^{-4} \text{ atm} / 17.82 \times 10^{-3} \text{ atm} \cdot \text{m}^3 / \text{mol} = 0.069 \text{ mol} / \text{m}^3 = 9.11 \text{ mg} / \text{L}$$

$$[\text{TCE}_{(\text{g})}] = (10 \text{ mg} / \text{L} - 9.11 \text{ mg} / \text{L}) \times 0.05 \text{ L} / 0.007 \text{ L} = 6.33 \text{ mg} / \text{L}$$

For 50 ppm TCE and 100 ppm TCE, volatilization of TCE were calculated with the same pattern.

BIOGRAPHY

Miss Trakarn Prapasongsa was born on September 19, 1981 in Bangkok, Thailand. She attended Navaminthrachinutit Bodindecha School in Bangkok and graduated in 1996. She received Bachelor's degree of Environmental Engineering from Chulalongkorn University in 2002. She pursued her Master Degree study in the International Postgraduate Programs in Environmental Management, Inter-Department of Environmental Management, Chulalongkorn University, Bangkok, Thailand in May 2003. She finished her Master Degree of Science in Environmental Management May 2005.

