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## APPENDICES

### Appendix A Calculation the Amount of the Carbon by Temperature Programmed Oxidation (TPO)

The temperature programmed oxidation technique was used to determine the amount of carbon. It can be calculated from the area under peak of the TPO profile. The carbon dioxide gas was used as the reference peak (so-called A) which was 100  $\square$ l. On the other hand, the sample peak was called B.

$$\text{From } PV = nRT \quad (1)$$

As, P = Pressure (atm), 1 atm

V = Volume ( $\text{cm}^3$ ), 100  $\mu$ l ( $0.1 \text{ cm}^3$ )

n = Number of mole (mole)

R = Gas constant ( $\text{cm}^3 \cdot \text{atm} / \text{gmole} \cdot \text{K}$ ),  $82.058 \text{ cm}^3 \cdot \text{atm} / \text{gmole} \cdot \text{K}$

T = Temperature (K),  $31^\circ\text{C}$  (304 K)

$$\text{Therefore, } n = 4 \times 10^{-6} \text{ g. mole}$$

The carbon dioxide 1 mole gave 1 mole of carbon. Consequently, the carbon dioxide  $4 \times 10^{-6}$  mole presents the equal mole of carbon.

Let, Area A has the amount of carbon  $4 \times 10^{-6}$  mole. Therefore, the area B has the amount of carbon

$$\text{The amount of sample's carbon } 4 \times 10^{-6} \cdot B/A$$

Example: Carbon nanotubes were oxidized at  $250^\circ\text{C}$  for 12 hours.

Area under pulse peaks of carbon dioxide were

1 <sup>st</sup> peak	2 <sup>nd</sup> peak	3 <sup>rd</sup> peak
$8.50 \times 10^5$	$8.83 \times 10^5$	$8.40 \times 10^5$
Average (A)	$8.57 \times 10^5$	

Area under peak of sample, which has weight 2.16 mg, was  $1.64 \times 10^7$  (B).

Then, the amount of sample's carbon was  $7.65 \times 10^{-5}$  mole,  $9.18 \times 10^{-4}$  grams.

The percentage of carbon was  $(9.18 \times 10^{-4} / 0.0216) * 100 = 4.25$  wt%.

## Appendix B Metal Digestion

**Table B1** Comparison of Catalyst Removal Between With and Non Oxidative Pre-Treatment

Cocentration of HCl (M)	Initial existed		Oxidative Pretreatment		% Removal		Non-oxidative Pretreatment		% Removal	
	Co (ppm)	Mo (ppm)	Co (ppm)	Mo (ppm)	Co (ppm)	Mo (ppm)	Co (ppm)	Mo (ppm)	Co (ppm)	Mo (ppm)
0.5	9.356	45.694	8.4	25.23	89.780	55.215	-	-	-	-
			8.42	25.32	89.994	55.412	-	-	-	-
			8.45	25.6	90.314	56.025	-	-	-	-
			8.43	24.66	90.101	53.968	-	-	-	-
1	9.337	45.602	9.12	33.56	97.672	73.593	0.705	3.138	7.550	6.881
			9.15	33.43	97.993	73.308	0.703	2.973	7.529	6.519
			9.21	33.31	98.636	73.045	0.709	3.163	7.593	6.936
			9.19	32.8	98.421	71.926	0.707	2.979	7.572	6.533
5	9.319	45.511	9.3	39.79	99.800	87.430	1.088	4.254	11.676	9.347
			9.29	39.62	99.693	87.056	1.087	4.238	11.665	9.312
			9.29	39.47	99.693	86.727	1.111	4.271	11.922	9.385
			9.31	37.96	99.907	83.409	1.109	4.012	11.901	8.815
8	9.244	45.144	9.31	40.47	100.718	89.646	-	-	-	-
			9.24	40.46	99.960	89.623	-	-	-	-
			9.22	40.49	99.744	89.690	-	-	-	-
			9.23	40.37	99.852	89.424	-	-	-	-
12	9.450	46.152	9.42	41.69	99.684	90.332	1.137	4.496	12.032	9.742
			9.43	41.05	99.789	88.946	1.129	4.250	11.947	9.209
			9.45	41.51	100.001	89.942	1.129	4.457	11.947	9.657
			9.43	41.54	99.789	90.007	1.121	4.207	11.863	9.116

**Table B2** Effects of Concentration of HCl

Cocentration of HCl (M)	Initial existed		% Removal	
	Co (ppm)	Mo (ppm)	Co (ppm)	Mo (ppm)
0.5	9.356	45.694	8.40	25.23
			8.42	25.32
			8.45	25.6
			8.43	24.66
1	9.337	45.602	9.12	24.66
			9.15	24.66
			9.21	24.66
			9.19	24.66
5	9.319	45.511	9.30	24.66
			9.29	24.66
			9.29	24.66
			9.31	24.66
8	9.244	45.144	9.31	24.66
			9.24	24.66
			9.22	24.66
			9.23	24.66
12	9.450	46.152	9.42	24.66
			9.43	24.66
			9.45	24.66
			9.43	24.66

**Table B3** Effects of Sonication Time

Time dissolved (hr)	Initial existed on cat.		Ion detection		% Removal	
	Co (ppm)	Mo (ppm)	Co (ppm)	Mo (ppm)	Co (ppm)	Mo (ppm)
1	10.238	50.002	8.625	31.42	84.243	62.838
			8.620	32.73	84.194	65.458
			8.214	31.35	80.229	62.698
			8.216	32.76	80.248	65.518
3	10.299	50.301	9.12	36.54	88.549	72.643
			9.15	36.03	88.840	71.629
			9.21	36.87	89.423	73.299
			9.19	35.12	89.228	69.820
6	10.197	49.803	9.62	42.37	94.337	85.076
			9.71	44.2	95.220	88.750
			9.86	44.98	96.691	90.317
			9.77	44.69	95.808	89.734
12	10.238	50.002	9.97	46.71	97.380	93.417
			10.22	44.81	99.822	89.617
			9.84	44.64	96.110	89.277
			10.2	46.75	99.627	93.497

**Table B4** Effect of Temperature

Temperature (°C) dissolution	Initial existed on cat.		Ion detection		% Removal	
	Co (ppm)	Mo (ppm)	Co (ppm)	Mo (ppm)	Co (ppm)	Mo (ppm)
RT ( 30 °C)	10.095	49.305	9.357	30	92.685	60.846
			9.579	31.29	94.884	63.463
			9.305	29.88	92.170	60.603
			9.539	32.76	94.488	66.444
40	10.238	50.002	9.572	34.47	93.493	68.938
			9.834	36.09	96.052	72.177
			9.639	34.16	94.147	68.318
			9.887	35.84	96.570	71.677
65	10.259	50.101	9.467	42.98	92.283	85.786
			9.74	42.37	94.945	84.569
			9.55	41.93	93.092	83.690
			9.808	42.21	95.607	84.249
80	10.218	49.902	9.605	45.12	94.002	90.417
			9.858	44.11	96.478	88.393
			9.595	44.42	93.905	89.014
			9.824	46.09	96.146	92.361

**Table B5** Effects of Number Cycle for Dissolution

Number of cycle	Initial existed on catalyst		Ion detection		% Removal	
	Co (ppm)	Mo (ppm)	Co (ppm)	Mo (ppm)	Co (ppm)	Mo (ppm)
1	10.259	50.101	9.506	45.32	92.664	90.457
			9.557	44.3	93.161	88.421
			9.252	44.28	90.188	88.381
			9.421	46.28	91.835	92.373
2	10.259	50.101	0.344	1.012	3.353	2.020
			0.354	1.072	3.451	2.140
			0.348	1.013	3.392	2.022
			0.358	1.091	3.490	2.178
3	10.259	50.101	0.156	0.522	1.521	1.042
			0.16	0.574	1.560	1.146
			0.155	0.578	1.511	1.154
			0.163	0.578	1.589	1.154
4	10.259	50.101	0.025	0.074	0.244	0.148
			0.026	0.0737	0.253	0.147
			0.024	0.074	0.234	0.148
			0.026	0.073	0.253	0.146

**Appendix C Determination of Contact Angle****Table C. Determination of Contact Angle**

Number of Measurement	Contact Angle Theta[deg]		
	Silica	Carbon Black	As-SWNTs
1	15.1	63.7	40.80
2	15.2	61.4	39.20
3	14.8	60.2	41.70
4	15.2	58.7	40.00
5	16.2	58.7	38.30
6	15.6	58.2	37.50
7	17.2	60.2	40.80
8	15.7	59.8	39.20
9	17.3	57.6	37.50
Average	15.81	59.83	39.44

### Appendix D Determination of Point of Zero Charge (PZC)

**Table D** Determination Point of Zero Charge (PZC)

Initial pH	Final pH (Equilibrium pH)								
	SiO <sub>2</sub>			Pure Carbon			SWNTs		
	1 <sup>st</sup>	2 <sup>nd</sup>	Ave.	1 <sup>st</sup>	2 <sup>nd</sup>	Ave.	1 <sup>st</sup>	2 <sup>nd</sup>	Ave.
1	0.85	0.83	0.84	0.95	0.94	0.945	1.1	1.2	1.15
2	1.9	1.91	1.905	1.97	1.96	1.965	1.97	2	1.985
3	3.63	3.63	3.63	2.93	2.93	2.93	3.22	3.23	3.225
4	4.13	4.14	4.135	4.12	4.12	4.12	3.98	3.99	3.985
5	4.69	4.7	4.695	5.34	5.38	5.36	6.33	6.32	6.325
6	5.87	5.75	5.81	5.39	5.44	5.415	5.35	5.36	5.355
7	6.17	6.18	6.175	6.44	6.46	6.45	6.65	6.75	6.7
8	6.83	6.86	6.845	6.56	6.61	6.585	6.6	6.58	6.59
9	7.57	7.58	7.575	7.24	7.27	7.255	7.09	7.12	7.105
10	7.97	7.96	7.965	7.38	7.37	7.375	7.01	7.02	7.015
11	9.48	9.48	9.48	8.24	8.23	8.235	8.81	8.82	8.815
12	10.01	10.01	10.01	10.16	10.15	10.155	10.37	10.36	10.365
13	13.08	13.12	13.1	12.77	12.8	12.785	12.78	12.8	12.79
14	13.98	13.92	13.95	13.98	13.92	13.95	13.98	13.92	13.95

## Appendix E Froth Flotation Results

**Table E** Froth flotation results

pH	Concentration (CMC)	Weight		% Purity	
		Nonionic	Anionic	Nonionic	Anionic
5	0.25	0.01	0.0104	56.86	58.31
	0.5	0.01	0.0104	56.37	60.89
	0.75	0.0102	0.0102	60.95	57.20
	1	0.0103	0.0104	55.23	50.46
3	0.25	0.0102	0.01	56.40	61.90
	0.5	0.0102	0.0105	57.32	69.32
	0.75	0.01	0.0105	64.20	60.42
	1	0.01	0.01	55.87	58.48
7	0.25	0.0104	0.0102	57.85	58.84
	0.5	0.0104	0.0096	60.59	67.03
	0.75	0.01	0.0103	64.81	109.81
	1	0.0101	0.0104	56.28	55.35
9	0.25	0.0103	0.0105	54.98	57.05
	0.5	0.0105	0.0103	58.59	62.19
	0.75	0.0103	0.0103	61.11	56.89
	1	0.01	0.01	54.89	54.84

## Appendix F XPS Results

**Table F1** XPS of Cobalt Reduce

Name	Start BE	Peak BE	End BE	Height Counts	FWHM eV	Area (P) CPS.eV	Area (N)
C1s	293.2	286.7	282.6	19472.34	3.41	69095.18	9617.79
C1s	293.2	289.97	282.6	6291.05	3.12	20426.5	2844.07
O1s	542.4	538.73	530.7	120714.55	2.74	344079.92	16736.26
O1s	542.4	534.6	530.7	18347.85	3.5	66861.54	3250.77
Si2p	112.4	109.09	104.9	22741.35	2.7	63888.2	10735.73
Co2p3	795.51	786.85	773.71	6341.35	3.49	23090.87	268.8
Co2p3	795.51	782.77	773.71	3354.22	3.48	12138.03	141.22
Co2p1	810.8	802.53	796.3	1968.62	3.34	6843.07	154.06
Co2p1	810.8	808.29	796.3	920.51	2.56	2454.67	55.31

**Table F2** XPS of Molybdenum Reduce

Name	Start BE	Peak BE	End BE	Height Counts	FWHM eV	Area (P) CPS.eV	Area (N)
C1s	295.2	285.85	280.9	19339.76	3.5	70457.1	9806.67
C1s	295.2	289	280.9	4812.58	3.43	17195.96	2394.07
Mo3d5	241	232.39	226.2	44091	3.5	161201.18	3975
Mo2d3	241	235.8	226.2	19843.85	2.58	53455.55	7409.96
O1s	541	533.08	527.9	82008.58	3.5	299832.82	14575.37
O1s	541	530.55	527.9	38175.82	1.94	77343.23	3758.79
Si2p	111.5	103.5	99.5	11403.04	2.57	30653.15	5148.85
Si2p	111.5	106.94	99.5	2092.9	3.48	7588.57	1274.98

**Table F3** XPS of Co:Mo/SiO<sub>2</sub> Reduce

Name	Start BE	Peak BE	End BE	Height Counts	FWHM eV	Area (P) CPS.eV	Area (N)
C1s	295	286.88	279.9	19424.78	3.49	70625.88	9831
C1s	295	290.04	279.9	7040.16	3.46	25424.12	3539.93
O1s	542.5	538.58	530.6	162403.06	2.71	459317.93	22341.13
O1s	542.5	535.16	530.6	23523.28	3.5	85969.05	4180.01
Si2p	112.2	108.81	102.8	29823.71	2.69	83726.28	14069.02
Co2p3	797.4	786.24	779.8	3642.43	3.5	13304.76	154.87
Co2p3	797.4	790.4	779.8	1672.4	3.5	6097.83	71.02
Co2p1	808.4	802.06	798.71	1032.34	3.33	3582.66	80.65
Mo3d5	244.6	237.44	231.7	7110.2	3.18	23503.3	579.79
Mo3d5	244.6	234.35	231.7	4183.58	2.72	11844.74	292.12
Mo3d3	244.6	240.43	231.7	2793.12	2.71	7876.8	281.52

**Table F4** XPS of Co:Mo/SiO<sub>2</sub> After Calcined

Name	Start BE	Peak BE	End BE	Height Counts	FWHM eV	Area (P) CPS.eV	Area (N)
C1s	295	286.15	281.3	20625.33	2.99	64112.66	8923.83
C1s	295	289.58	281.3	8272.58	3.5	30139.75	4196.35
Co2p3	813.71	786.28	764.4	5164.76	3.5	18852.72	219.44
C4_Co820-	813.71	781.57	764.4	2883.5	3.5	10542.04	1547.55
Mo3d5	245.6	237.3	233.8	7634.84	2.5	19970.92	492.65
Mo3d3	245.6	240.46	233.8	5740.92	3.08	18494.27	660.98
O1s	542.7	538.66	530.4	161160.55	2.79	468777.18	22801.42
O1s	542.7	534.72	530.4	18627.97	3.5	67865.54	3299.63
Si2p	112.5	108.9	104.1	29777.09	2.75	85450.81	14358.9

**Table F5** XPS of Reduce and Oxidize samples

Name	Start BE	Peak BE	End BE	Height Counts	FWHM eV	Area (P) CPS.eV	Area (N)
Cl <sub>s</sub>	295	286.15	281.3	20625.33	2.99	64112.66	8923.83
Cl <sub>s</sub>	295	289.58	281.3	8272.58	3.5	30139.75	4196.35
Mo3d <sub>5</sub>	245.6	237.3	233.8	7634.84	2.5	19970.92	492.65
Mo3d <sub>3</sub>	245.6	240.46	233.8	5740.92	3.08	18494.27	660.98
O <sub>1s</sub>	542.7	538.66	530.4	161160.55	2.79	468777.18	22801.42
O <sub>1s</sub>	542.7	534.72	530.4	18627.97	3.5	67865.54	3299.63
Si2p	112.5	108.9	104.1	29777.09	2.75	85450.81	14358.9
Co3d <sub>5</sub>	797.11	786.67	780.61	4495.19	3.5	16380.44	2406.36
Co3d <sub>5</sub>	797.11	791.55	780.61	2275	3.5	8285.17	1217.98
Co3d <sub>3</sub>	811.11	802.8	798.71	2137.87	3.5	7801.23	1148.71
Co3d <sub>3</sub>	811.11	808.48	798.71	1149.46	3.5	4188.83	617.31

**Table F6** After Acid treatment step

Name	Start BE	Peak BE	End BE	Height Counts	FWHM eV	Area (P) CPS.eV	Area (N)
C1s	295.2	286.4	280.5	36561.77	3.45	131272.13	18272.13
C1s	295.2	290	280.5	6464.84	2.54	17078.68	2377.94
Mo3d5	247.4	232.67	226.5	743.35	3.48	2701.92	66.63
Mo3d3	247.4	235.93	226.5	381.92	2.8	1112.66	39.75
O1s	541.4	536.56	528.4	74638.82	3.07	238391.29	11592.84
O1s	541.4	533.89	528.4	48626.45	2.89	146080.61	7101.83
O1s.	541.4	531.03	528.4	13064.05	2.55	34674.57	1685.23
Si2p	111.4	107.06	100.4	12007.6	3.01	37672.39	6329.51
Si2p	111.4	104.61	100.4	3424.22	2.91	10398.07	1746.72
Co2p3	793.8	782.47	776.51	2143.45	3.43	7667.59	89.2
C6_Co820-755_Asource A	793.8	780.67	776.51	2131.15	0.53	1181.62	173.44
Co3d5	774.71	768.42	765.8	719.45	0.18	2545.79	373.03
C6_Co820-755_Asource B	793.8	785.47	776.51	1387.96	1.77	2558.33	375.76

**CURRICULUM VITAE**

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**Presentations:**

1. L. Nattapong, N. Chaninat, W. Ladawan, and P. Thirawudh (2005). Recycling of Silane-Crosslinked Polyethylene (Si-XLPE) by the Extruder: Effect of Active and Non-active Additives. 15<sup>th</sup> Annual Conference of Chemical Engineering and Applied Chemistry.