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APPENDICES

APPENDIX A

Colorimetric method for chromium (VI) analysis

1. Principle

This process measures only hexavalent chromium. It is determined colorimetrically by reaction with diphenylcarbazide in acid solution. The complex red-violet color was produced that can be measured with 540 nm.

2. Special reagents

2.1 Diphenylcarbazide solution: dissolve 250 mg 1,5-diphenylcarbazide in 50 ml of acetone. Then, store diphenylcarbazide solution in a brown bottle. Discard when solution becomes discolored.

2.2 Stock chromium solution: dissolve 1.411 g of $K_2Cr_2O_7$ in double distilled water (DDW) and dilute to 1 l; 1.00 ml = 500.0 µg Cr⁶⁺

3. Procedures

(Standard Methods for the examination of water and wastewater, 1998).

3.1 Preparation of calibration curve:

1) Pipet measured volumes of standard chromium solution (500 μ g/ml) ranging from 1.00 to 25.0 ml, to give standards for 0.50 to 12.50 mg Cr (VI), into 250 ml volumetric flasks. Then, get standard chromium solution 2 to 50 mg/l or ppm.

2) Take 5 ml of solution to a bigger

3) Add 0.25 ml H₃PO₄

4) Use 0.2 N H₂SO₄ and a pH meter to adjust solution to pH 1.0 ± 0.3

5) Transfer solution to a 100 ml volumetric flask, dilute to 100 ml and mix.

6) Add 2.0 ml diphenycarbazide solution, mix and allow 5 to 10 min for full color development.

8) Transfer an appropriate portion to a 1-cm absorption cell and measure its absorbance at 540 nm. Use distilled water as reference.

9) Correct absorbance reading of sample by subtracting absorbance of a blank carried through the method.

10) Construct a calibration curve by plotting corrected absorbance against micrograms of chromium.

3.2 Sample measurement

- 1) Take 5 ml of sample in the bigger
- 2) Add 0.25 ml H₃PO₄
- 3) Use 0.2 N H₂SO₄ for adjust pH to be 1.0 ± 0.3
- Transfer solution to a 100 ml volumetric flask, dilute to 100 ml and mix.

5) Add 2.0 mL diphenycarbazide solution, mix and allow 5 to 10 min for full color development.

6) Transfer an appropriate portion to a 1-cm absorption cell and measure its absorbance at 540 nm. Use distilled water as reference.

7) Correct absorbance reading of sample by subtracting absorbance of a blank carried through the method.

APPENDIX B

Calculations

Example experimental data: Wastewater flow rate 20 ml/s, reaction period time 143 min, reaction time for completely remove is 208 min, volume of wastewater 20 litter and volume capacity of reactor is 10.092 litter.

1. Calculation of contact time, min/cycle

- Contact time	= <u>10.092 lite</u>	<u>r = 8.41 min</u>
	20 ml/s	

2. Calculation of treating cycle, cycle

- Treating cycle	=	<u>143 min</u>	=	17
		8.41 min		

3. Calculation of Cr (VI) removal percentage, %

- Cr (VI) removal percentage = $\frac{143 \text{ min}}{208 \text{ min}} *100 = 68.75 \%$

Example experimental data; Rotating speed disc 10 rpm, reaction period time 105 min, reaction time for completely remove is 182 min, volume of wastewater 20 litter and volume capacity of reactor is 10.092 m³.

4. Calculation of rotating disc cycle, round

- Total rotating cycle in 105 min = 10 rpm* 105 min = 1050 rounds

5. Calculation of reaction time for one round of rotating disc, min

- Reaction time for one round =
$$1_{10}$$
 = 0.1 min
10 rpm

Example experimental data: Outer diameter of disc plate 12 cm, inner diameter of disc plate is 1.27 cm, wastewater level in the reactor is 5 cm, disc submerge in the wastewater is 4.5 cm, in the reactor has 12 discs plate, amount of TiO_2 is approximate 0.226 mg/cm² and the experimental time is 105 min.



6. Calculation of TiO₂ coating surface area which contract wastewater, m²

- No TiO₂ coating surface area can calculate by

$$A = \left(\frac{\Pi . r^2 . \theta_2}{360}\right) - \left(\frac{1}{2} . r^2 - \sin \theta_2\right)$$
$$A = \left(\frac{\Pi . 6^2 . (77.364)}{360}\right) - \left(\frac{1}{2} . 6^2 - \sin 77.364\right) = 7.281 \text{ cm}^2$$

- All area of disc plate:

$$A = \left(\frac{\Pi . (12)^2}{4}\right) - \left(\frac{\Pi . (1.27)^2}{4}\right) = 111.83 \text{ cm}^2$$

- Total TiO₂ coating surface area on one side of disc plate

 $A = 111.83 - 7.281 = 104.549 \text{ cm}^2$

- TiO₂ coating surface area which contract wastewater

$$A = 104.549 - \left(\frac{\prod .3^2}{4}\right) = 97.48 \text{ cm}^2$$

- 7. Calculation of total TiO₂ coating surface area which contract wastewater in the experimental time, m²
 - If experimental time is 105 min, TiO₂ coating surface area which contract wastewater can calculate:

$$A = 97.48 \text{ cm}^2 / \text{round} + 12 \times 2 \times 1050 \text{ round} = 245.65 \text{ m}^2$$

8. Calculation of amount of TiO2 use in one experiment, mg

 \rightarrow amount of TiO₂ used can calculate:

 $0.226 \text{ mg/cm}^{2*} 97.48 \text{ cm}^{2*} 12* 2 = 528 \text{ mg}$

APPENDIX C

EXPERIMENTAL DATAS

Table C-1 Photoreduction of chromium (VI) was using RDPR in the operating condition of initial concentration 25 ppm, wastewater flow rate 90 ml/s, rotating speed disc 200 rpm and TiO₂ coating surface area 0.234 m² in the initial pH of 3, 7 and 11.

		Residual chromium (VI) concentration, (ppm)								
Time (min)		pH 3			pH 7			pH 11		
	1	2	Average	1	2	Average	1	2	Average	
0	25.00	25.00	25.00	25.00	25.00	25.00	25.00	25.00	25.00	
15	20.34	20.30	20.32	24.67	24.64	24.65	24.69	24.73	24.71	
25	18.97	19.03	19.00	24.33	24.33	24.33	24.98	25.02	25.00	
35	15.22	14.84	15.03	24.06	24.04	24.05	24.66	24.72	24.69	
45	13.01	13.09	13.05	23.70	23.68	23.69	24.68	24.67	24.68	
60	9.77	9.87	9.82	23.42	23.43	23.42	25.03	25.02	25.02	
75	5.93	6.07	6.00	23.21	23.21	23.21	24.90	24.96	24.93	
105	0.07	0.07	0.07	23.09	23.09	23.09	24.66	24.84	24.75	
135	0	0	0	22.77	22.77	22.77	25.01	24.72	24.86	
165				22.26	22.27	22.27	24.86	24.60	24.73	
195				21.54	21.55	21.55	24.98	24.91	24.94	

Table C-2.1 Photoreduction of chromium (VI) was using RDPR in the operating condition of initial concentration 25 ppm, rotating speed disc50 rpm, initial pH of wastewater was 3 and TiO2 coating surface area were 0.234 m² in the wastewater flow rate of 20, 40, 60 ml/s.

			Residua	l chromiun	(VI) conce	entration, (ppm)			
Time (min)	Flow rate 20 ml/s			Flo	Flow rate 40 ml/s			Flow rate 60 ml/s		
	1	2	Average	1	2	Average	1	2	Average	
0	25.00	25.00	25.00	25.00	25.00	25.00	25.00	25.00	25.00	
15	22.42	22.41	22.42	21.67	22.06	21.87	22.54	22.63	22.59	
25	20.22	21.78	21.00	18.51	20.56	19.54	20.07	20.11	20.09	
35	18.80	20.88	19.84	17.35	19.24	18.29	19.02	19.10	19.06	
45	17.68	19.63	18.66	16.58	17.23	16.91	17.59	17.63	17.61	
60	15.81	18.89	17.35	14.94	15.06	15.00	15.02	15.00	15.01	
75	14.78	17.21	15.99	12.89	13.06	12.98	13.56	13.76	13.66	
105	12.04	13.77	12.91	10.66	10.75	10.71	9.45	9.53	9.49	
135	8.69	10.13	9.41	7.12	7.23	7.18	6.12	6.02	6.07	
165	4.52	5.91	5.22	3.25	3.27	3.26	1.89	2.03	1.96	
195	1.17	1.68	1.42	0.00	0.00	0.00	0.00	0.00	0.00	
225	0.00	0.00	0.00							

Table C-2.2 Photoreduction of chromium (VI) was using RDPR in the operating condition of initial concentration 25 ppm, rotating speed disc50 rpm, initial pH of wastewater was 3 and TiO2 coating surface area was 0.234 m² in the wastewater flow rate of 80, 90 ml/s.

	F	Residual ch	romium (V	I) concentr	ation, (pp	m)		
Time (min)	Flo	ow rate 80 n	nl/s	Flo	Flow rate 90 ml/s			
	1	2	Average	1	2	Average		
0	25.00	25.00	25.00	25.00	25.00	25.00		
15	21.95	22.07	22.01	22.09	22.15	22.12		
25	20.32	20.19	20.26	20.07	20.09	20.08		
35	17.94	18.21	18.07	18.66	18.77	18.71		
45	16.49	16.54	16.52	17.24	17.54	17.39		
60	14.96	15.06	15.01	15.09	15.42	15.25		
75	12.17	12.63	12.40	12.28	12.36	12.32		
105	8.02	8.11	8.07	5.48	5.58	5.53		
135	3.26	3.19	3.22	1.49	1.36	1.42		
165	0.00	0.00	0.00	0.00	0.00	0.00		

			Residua	l chromiu	m (VI) co	ncentratio	n, (ppm)			
Time (min)	Rpm 10				Rpm 50			Rpm 100		
	1	2	Average	1	2	Average	1	2	Average	
0	25.00	25.00	25.00	25.00	25.00	25.00	25.00	25.00	25.00	
15	21.13	21.49	21.31	22.09	22.07	22.08	22.52	22.56	22.54	
25	19.48	19.54	19.51	20.07	20.13	20.10	19.62	19.72	19.67	
35	18.27	18.15	18.12	18.66	18.70	18.68	17.07	17.11	17.09	
45	16.23	17.73	16.98	17.24	17.60	17.42	14.84	14.86	14.85	
60	13.90	15.42	14.66	15.08	15.14	15.11	12.23	12.43	12.33	
75	11.81	14.61	13.21	12.28	12.32	12.30	9.44	9.72	9.08	
105	9.30	9.30	9.30	6.98	7.26	7.12	4.26	4.34	4.30	
135	6.22	6.40	6.31	2.36	2.54	2.45	0.72	0.34	0.53	
165	3.30	3.20	3.25	0.05	0.15	0.10	0.00	0.00	0.00	
195	0.02	0.04	0.03							

Table C-3.1 Photoreduction of chromium (VI) was using RDPR in the operating condition of initial concentration 25 ppm, wastewater flow rate90 ml/s, initial pH of wastewater was 3 and TiO2 coating surface area were 0.234 m² in the rotating disc speed of 10, 50 and 100 rpm.

Table C-3.2 Photoreduction of chromium (VI) was using RDPR in the operating condition of initial concentration 25 ppm, wastewater flow rate90 ml/s, initial pH of wastewater was 3 and TiO2 coating surface area were 0.234 m² in the rotating disc speed of 150 and 200 rpm.

	Res	Residual chromium (VI) concentration, (ppm)								
Time (min)		Rpm 150			Rpm 200					
	1	2	Average	1	2	Average				
0	25.00	25.00	25.00	25.00	25.00	25.00				
15	22.00	21.98	21.99	20.34	20.30	20.32				
25	20.01	19.81	19.91	18.97	19.03	19.00				
35	17.86	17.78	17.82	15.22	14.84	15.03				
45	15.66	15.48	15.57	13.01	13.09	13.05				
60	11.60	11.64	11.62	9.77	9.87	9.82				
75	7.50	7.38	7.44	5.93	6.07	6.00				
105	2.76	2.84	2.80	0.07	0.07	0.07				
135	0.00	0.00	0.00	0.00	0.00	0.00				

Table C-4.1 Photoreduction of chromium (VI) was using RDPR in the operating condition of initial concentration 50 ppm, wastewater flow rate90 ml/s, rotating disc speed 200 rpm and initial pH of wastewater was 3 in the TiO2 coating surface area of 0.1170, 0.1754 and0.2340 m²

			Residua	l chromiui	m (VI) cor	icentration	, (ppm)		
Time (min)		0.1170 m	2		0.1754 m	2		0.2340 m	2
	1	2	Average	1	2	Average	1	2	Average
0	50.00	50.00	50.00	50.00	50.00	50.00	50.00	50.00	50.00
15	49.07	49.19	49.13	49.17	49.03	49.10	47.60	47.30	47.45
25	48.32	48.42	48.37	48.15	48.03	48.09	45.15	44.99	45.07
35	47.57	47.73	47.65	47.40	47.28	47.34	44.31	44.29	44.30
45	46.39	46.89	46.64	45.88	45.76	45.82	42.59	42.87	42.73
60	45.84	46.02	45.93	44.22	44.10	44.16	41.17	41.03	41.10
75	45.34	45.62	45.48	42.87	42.75	42.81	39.76	39.88	39.82
105	43.76	43.98	43.87	40.59	40.49	40.54	36.59	36.51	36.55
135	41.83	41.85	41.84	37.10	37.44	37.27	33.94	33.62	33.78
165	40.21	40.75	40.48	34.30	34.70	34.50	30.58	30.06	30.32
195	38.28	38.54	38.41	31.58	32.10	31.84	27.39	27.45	27.42
225	36.56	37.48	37.02	29.81	30.15	29.98	24.34	24.34	24.34
255	35.54	35.54	35.54	27.50	27.74	27.62	21.31	21.49	21.40
285	33.85	34.13	33.99	25.08	25.66	25.37	17.56	17.70	17.63
315	32.88	33.18	33.03	23.30	23.66	23.48	14.31	14.73	14.52
345	31.45	31.87	31.66	20.96	21.00	20.98	10.59	11.13	10.86
375	31.06	29.66	30.36	17.50	17.66	17.58	7.57	8.21	7.89
405	29.65	28.95	29.30	15.35	15.33	15.34	2.25	4.05	3.15



		Residual c	hromium (VI) c	oncentrat	ion, (ppm)		
Time (min)		0.2630	m ²		0.2924 m ²			
	1	2	Average	1	2	Average		
0	50.00	50.00	50.00	50.00	50.00	50.00		
15	49.37	48.63	49.00	49.22	49.04	49.13		
25	47.30	46.58	46.94	47.87	47.17	47.52		
35	46.17	45.57	45.87	47.21	46.19	46.70		
45	44.84	44.14	44.49	45.49	44.43	44.96		
60	43.43	42.71	43.07	43.23	42.43	42.83		
75	41.92	41.04	41.48	40.48	39.52	40.00		
105	38.28	37.76	38.02	36.26	35.44	35.85		
135	35.00	34.46	34.73	33.01	32.03	32.52		
165	31.21	30.83	31.02	29.60	28.84	29.22		
195	27.02	26.58	26.80	25.90	25.24	25.57		
225	22.89	22.91	22.90	20.32	19.8	20.06		
255	18.60	17.98	18.29	15.22	14.84	15.03		
285	15.86	15.68	15.77	11.66	11.36	11.51		
315	12.33	12.23	12.28	8.84	8.62	8.73		
345	7.90	7.7	7.80	5.62	5.46	5.54		
375	3.79	3.47	3.63	2.05	0.95	1.50		
405	0.00	0.00	0.00	0.00	0.00	0.00		

Table C-4.2 Photoreduction of chromium (VI) was using RDPR in the operating condition of initial concentration 50 ppm, wastewater flow rate90 ml/s, rotating disc speed 200 rpm and initial pH of wastewater was 3 in the TiO2 coating surface area of 0.2630 and 0.2924 m².

Table C-5.1 Photoreduction of chromium (VI) was using RDPR in the operating condition of wastewater flow rate 90 ml/s, rotating disc speed200 rpm, initial pH of wastewater was 3 and TiO2 coating surface area were 0.2340 m² in the initial concentration of 25, 40 and 50 ppm.

	Residual chromium (VI) concentration, (ppm)								
Time (min)		25 ppm			40 ppm			50 ppm	
	1	2	Average	1	2	Average	1	2	Average
0	25.00	25.00	25.00	40.00	40.00	40.00	50.00	50.00	50.00
15	20.34	20.30	20.32	37.48	37.56	37.52	47.37	47.81	47.59
25	18.97	19.03	19.00	36.01	35.89	35.95	45.19	45.11	45.15
35	15.22	14.84	15.03	34.45	34.37	34.41	77.33	11.29	44.31
45	13.01	13.09	13.05	32.98	33.06	33.02	42.57	42.61	42.59
60	9.77	9.87	9.82	32.06	31.9	31.98	41.14	41.20	41.17
75	5.93	6.07	6.00	29.81	29.71	29.76	39.71	39.81	39.76
105	0.07	0.07	0.07	25.69	25.67	25.68	36.54	36.64	36.59
135	-	-	-	21.30	21.22	21.26	33.87	34.01	33.94
165	-	-	-	18.06	17.92	17.99	30.55	30.61	30.58
195	-	-	-	13.41	12.89	13.15	27.38	27.40	27.39
225	-	-	-	9.61	9.39	9.50	24.33	24.35	24.34
255	-	-	-	6.09	5.91	6.00	21.21	21.41	21.31
285	-	-	-	2.49	2.61	2.55	17.62	17.50	17.56
315	-	-	-	0.00	0.00	0.00	14.36	14.26	14.31
345	-	-	-	-	-	-	10.57	10.61	10.59
375	-	-	-	-	-	-	7.53	7.61	7.57
405	-	-	-	•	-	-	2.19	2.31	2.25
435	-	-	-	-	-	-	0.10	0.18	0.14

Table C-5.2 Photoreduction of chromium (VI) was using RDPR in the operating condition of wastewater flow rate 90 ml/s, rotating disc speed200 rpm, initial pH of wastewater was 3 and TiO2 coating surface area were 0.2340 m² in the initial concentration of 80, 100 and150 ppm.

	Residual chromium (VI) concentration, (ppm)								
Time (min)		80 ppm			100 ppm			150 ppm	
	1	2	Average	1	2	Average	1	2	Average
0	80.00	80.00	80.00	100.00	100.00	100.00	150.00	150.00	150.00
15	77.01	76.31	76.66	97.43	97.71	97.57	146.71	146.63	146.67
25	75.91	75.79	75.85	95.74	95.80	95.77	144.60	144.56	144.58
35	75.10	74.12	74.61	95.2	95.32	95.26	141.32	141.18	141.25
45	73.22	73.08	73.15	94.44	94.52	94.48	140.38	140.24	140.31
60	69.67	69.65	69.66	93.43	94.01	93.72	137.15	137.11	137.13
75	66.33	66.13	66.23	89.91	90.11	90.01	132.56	132.42	132.49
105	63.08	62.98	63.03	84.65	84.89	84.77	127.81	127.79	127.80
135	58.11	57.97	58.04	78.83	78.85	78.84	121.30	120.34	120.82
165	52.23	52.21	52.22	72.6	72.88	72.74	115.82	115.40	115.61
195	46.51	46.25	46.38	68.46	70.00	69.23	109.71	109.63	109.67
225	42.92	42.08	42.50	61.53	61.63	61.58	102.36	103.02	102.69
255	38.11	38.05	38.08	57.1	57.32	57.21	93.55	93.59	93.57
285	34.67	34.47	34.57	52.85	53.13	52.99	89.32	89.18	89.25
315	30.53	30.23	30.38	49.74	49.78	49.76	83.44	83.28	83.36
345	25.77	25.67	25.72	44.09	44.25	44.17	78.28	77.92	78.10
375	21.63	21.39	21.51	37.23	37.41	37.32	70.01	69.83	69.92
405	17.29	17.21	17.25	33.13	33.15	33.14	63.09	63.21	63.15
435	13.52	13.26	13.39	26.72	26.78	26.75	55.03	54.27	54.65
465	7.85	7.71	7.78	20.87	21.09	20.98	50.32	50.86	50.59
495	3.12	3.02	3.07	14.3	14.36	14.33	45.42	45.34	45.38
525	0.00	0.00	0.00	9.84	10.10	9.97	45.19	44.73	44.96
555				3.38	3.18	3.28	44.96	44.76	44.86
585				0.03	0.07	0.05	45.10	45.04	45.07

Table C-5.3 Photoreduction of chromium (VI) was using RDPR in the operating condition of wastewater flow rate 90 ml/s, rotating disc speed 200 rpm, initial pH of wastewater was 3 and TiO₂ coating surface area was 0.2340 m² in the initial concentration of 250, 300 and 500 ppm.

			Res	idual chrom	ium (VI) con	centration, (p	pm)		
Time (min)	250 ppm 300 ppm 500 ppm								
	1	2	Average	1	2	Average	1	2	Average
0	250.00	250.00	250.00	300.00	300.00	300.00	500.00	500.00	500.00
15	246.11	_245.71	245.91	295.07	295.20	295.13	498.80	499.00	498.90
25	244.01	243.64	243.83	292.10	292.16	292.13	491.10	492.10	491.60
35	241.36	241.24	241.30	291.19	291.41	291.30	489.40	490.80	490.10
45	237.85	237.72	237.78	290.90	290.05	290.47	489.50	489.50	489.50
60	233.91	233.85	233.88	290.42	290.11	290.27	485.70	486.10	485.90
75	227.76	227.87	227.81	287.11	287.21	287.16	477.60	480.20	478.90
105	221.35	221.26	221.30	286.20	286.25	286.23	476.80	476.20	476.50
135	213.18	212.96	213.07	279.07	279.09	279.08	472.10	472.50	472.30
165	205.22	206.76	205.99	279.06	279.10	279.08	468.60	468.80	468.70
195	197.53	197.50	197.51	273.98	276.31	275.15	464.30	464.50	464.40
225	190.41	190.36	190.38	2207.41	2752.32	272.45	459.00	461.00	460.00
255	183.26	182.30	182.78	269.01	268.24	268.62	455.50	455.10	455.30
285	175.08	175.06	175.07	263.18	263.30	263.24	450.60	451.60	451.10
315	169.05	168.61	168.83	256.31	256.50	256.40	446.90	447.50	447.20
345	164.00	162.77	163.38	249.58	249.35	249.47	442.60	443.20	442.90
375	158.10	156.83	157.46	245.59	245.68	245.63	438.80	439.00	438.90
405	155.32	154.67	155.00	240.70	240.63	240.66	433.20	434.00	433.60
435	152.10	152.17	152.14	234.82	234.91	234.86	428.20	430.00	429.10
465	149.23	150.08	149.66	226.98	227.21	227.10	423.00	423.60	423.30
495	147.76	147.80	147.78	224.13	224.26	224.20	417.20	419.00	418.10
525	147.51	147.60	147.55	218.25	218.55	218.40	411.60	412.80	412.20
555				213.28	220.41	216.85	409.40	408.40	408.90
585				211.75	215.52	213.63	401.70	401.30	401.50
615				209.34	211.30	210.32	402.50	402.50	402.50
645				207.10	207.12	207.11	402.40	401.20	401.80
675				204.23	204.81	204.52	402.40	402.80	402.60
705				205.62	205.50	205.56	404.50	401.50	403.00

BIOGHAPHY

Miss Pattama Paksaharn was born on June 29, 1982 in Udonthani, Thailand. She received her Bachelor's degree in Environmental Engineering from faculty of Engineering, King Mongkut's University of Technology Thonburi (KMUTT) in 2004. At KMUTT, she has studied in the topic of "Heavy metal wastewater treatment using modifies egg shell" as her senior project which was publication in the topic of "Chicken's Egg Shell Technology for Pretreatment of Waste Solution from Acid Copper Electroplating Bath" in International Conference Hazardous Waste Management for a Sustainable Future, January 10-12, 2006, Bangkok, Thailand.

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