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APPENDICES

APPENDIX A

Colorimetric Method for Chromium(VI) Analysis

1. Principle

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

2. Special reagents

2.1 *Stock chromium solution*: dissolve 141.1 mg $K_2Cr_2O_7$ in double distilled water (DDW) and dilute to 1 L; 1.00 mL = 50.0 μg Cr^{6+}

2.2 *Diphenylcarbazide solution*: dissolve 250 mg 1,5-diphenylcarbazide in 50 mL acetone. Store in a brown bottle. Discard when solution becomes discolored.

3. Procedures

a. <u>Preparation of calibration curve</u>:

1. Pipet measured volumes of standard chromium solution (5 μg/mL) ranging from 2.00 to 20.0 mL, to give standards for 10 to 100 μg Cr, into 250-mL volumetric flasks.

2. Transfer solution to a 100-mL

3. Use 0.2 N H_2SO_4 and a pH meter to adjust solution to pH 1.0 ± 0.3, diluted to 100 mL, and mix.

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

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

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

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

b. Sample measurement

1. Transfer 10 mL of solution to a 100-mL

2. Use 0.2 N H_2SO_4 and a pH meter to adjust solution to pH 1.0 ± 0.3, diluted to 100 mL, and mix.

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

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

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

APPENDIX B

Table B-1 Photoreduction of chromium(VI) using TiO_2 thin film obtained from TTiP to ethanol to PEG600 at 1:20:0.5 and 1:20:1.

Time (min)	Chromium concentration (mg/L)	
	1:20:0.5	**1:20:1**
0	25.00	25.00
5	24.82	24.67
10	24.61	24.49
15	24.53	24.01
30	24.16	23.63
60	23.18	23.49
90	21.74	22.38
120	21.07	21.60
150	19.60	20.95
180	18.43	20.01

Table B-2 Photoreduction of chromium(VI) using TiO_2 thin film obtained from TTiP to ethanol to DEG at 1:20:0.5, 1:20:1 and 1:20:1.5.

Time (min)	Chromium concentration (mg/L)		
	1:20:0.5	**1:20:1**	**1:20:1.5**
0	25.00	25.00	25.00
5	24.94	24.89	24.87
10	24.88	24.72	24.76
15	24.45	24.63	24.70
30	23.99	24.29	23.82
60	23.14	23.10	23.11
90	21.97	22.27	22.22
120	20.85	21.27	21.37
150	19.73	19.88	20.33
180	18.57	18.91	18.77

Table B-3 Photoreduction of chromium(VI) using TiO_2 thin film obtained from TTiP to ethanol to PEG600 to DEG at 1:20:0.5:0, 1:20:0:0.5 and 1:20:0.5:0.5.

Time (min)	**Chromium concentration (mg/L)**		
	1:20:0.5:0	**1:20::0:0.5**	**1:20:0.5:0.5**
0	25.00	25.00	25.00
5	24.82	24.94	24.81
10	24.61	24.88	24.53
15	·24.53	24.45	24.33
30	24.16	23.99	23.75
60	23.18	23.14	22.68
90	21.74	21.97	21.52
120	21.07	20.85	20.83
150	19.60	19.73	19.64
180	18.43	18.57	17.81

Table B-4 Photoreduction of chromium(VI) using TiO_2 thin film prepared with different calcined temperature.

Time (min)	Chromium concentration (mg/L)	
	400°C	500°C
0	25.00	25.00
5	24.95	24.81
10	24.82	24.53
15	24.53	24.33
30	24.02	23.75
60	23.18	22.68
90	22.02	21.52
120	21.46	20.83
150	20.94	19.64
180	19.81	17.81

Table B-5 Photoreduction of chromium(VI) using TiO_2 thin film prepared with 1, 3 and 5 coating cycles (calcined at 500°C).

Time (min)	**Chromium concentration (mg/L)**		
	1 cycle	**3 cycles**	**5 cycles**
0	25.00	25.00	25.00
5	25.08	24.81	24.12
10	24.99	24.53	23.83
15	24.69	24.33	23.40
30	24.35	23.75	23.23
60	23.34	22.68	22.11
90	22.06	21.52	20.45
120	20.86	20.83	19.02
150	19.68	19.64	17.38
180	18.51	17.81	15.99

APPENDIX C

Examples of Calculated TiO_2 weight and Crystallite Size

C.1 Calculation of TiO_2 weight

	No.1	No.2	No.3	No.4	No.5	Average
Increasing mass of TiO_2 (g)	0.0006	0.0006	0.0008	0.0007	0.0007	-
Film surface area (cm^2)	6	6	6	6	6	-
Mass of TiO_2 per unit of surface area (10^{-3} g/cm^2)	0.100	0.100	0.133	0.117	0.117	0.113

$$\text{Mass of } TiO_2 \text{ per unit of surface area} = \frac{0.0006}{6} \times 1000 = 0.100 \times 10^{-3}\ g/cm^2$$

C.2 Calculation of crystallite size

X-ray diffraction patterns were used for the crystallite size estimation by line broadening measurements in the Debye-Scherrer equation:

$$L = K\lambda / \beta\cos\theta \qquad (3.1)$$

where,

L	=	the crystallite size (nm)
K	=	the Debye-Scherrer constant (ususlly taken as 0.89)
λ	=	the wavelength of the X-ray radiation (Cu Kα = 0.15418 nm)
β	=	the line width at half-maximum height of the broadened peak
θ	=	the half diffraction angle of the centroid of the peak (degree)

From Figure 4.23, crystallite size of TiO_2 prepared with 5-coating cycles can be calculated as follows:

$$L = \frac{0.89 \times 0.15418}{0.0038 \times \cos(25.28/2)} = 40.2 \text{ nm}$$

BIOGHAPHY

Miss Parichart Amornchat was born on May 1, 1979 in Bangkok, Thailand. She received her Bachelor's degree in Food Science and Technology from faculty of Agro-Industry, Kasetsart University in 2000. After graduation she worked in the field of food processing and quality assurance in private company for three years. Then, she pursued her Master Degree studies in the International Postgraduate Program in Environmental Management (Hazardous Waste Management), Inter-Department of Environmental Management Chulalongkorn University, Bangkok, Thailand on May, 2003. She had a publication with her advisor, Asst. Prof. Puangrat Kajitvichyanukul in the subject of "Effects of Diethylene glycol on TiO_2 Thin film properties prepared by sol-gel process", Proceedings for International Symposium on Nanotechnology in Environmental Protection and Pollution, ISNEPP 2005, January 12-14, 2005