

APPENDIX A

Carbon materials electrodes

The different carbon materials electrodes as a working electrode including are glass carbon, graphite and SPCE electrodes. The pictures of the working electrode are shown in Figure 5.1 and the design of SPCE shown in Figure 5.2.

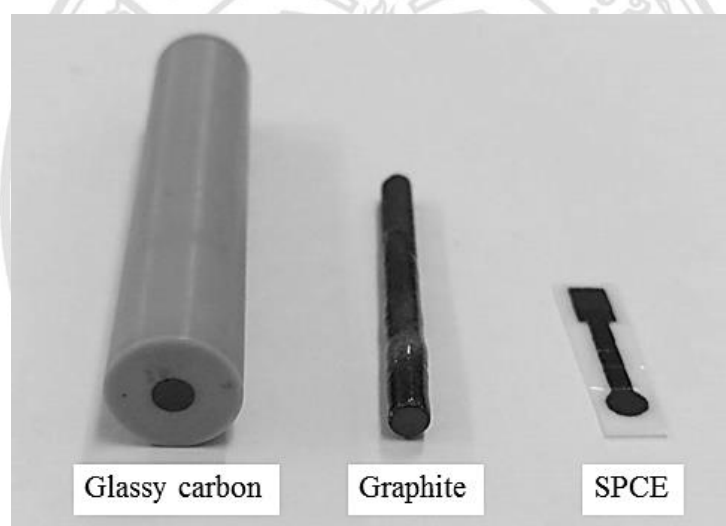


Figure 5.1 The bare glass carbon, graphite and SPCE electrodes as a working electrode

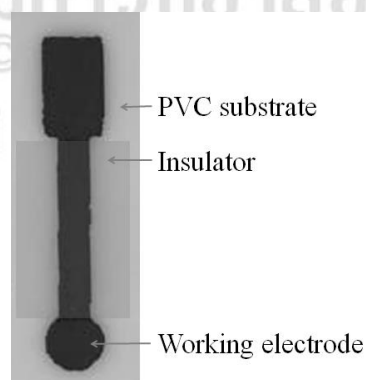


Figure 5.2 The design of SPCE working electrode

APPENDIX B

Detection limit calculation

In this work, the detection limits are determined by reducing the concentration of the analytes to minimum level that give the voltammograms as shown in Figure 5.3 and 5.5. The Excel LINEST function was used to calculate the standard deviation of y-intercept of calibration graph [Rojanarata, 2010]. The calibration graphs for very low concentrations of analyte are shown in Figure 5.4 and 5.6, and the calculation of detection limits are summarized in Table 5.1 and 5.2.

1. The limits of detection of cadmium, lead and zinc in 0.1 M acetate buffer solution (pH 4.5)

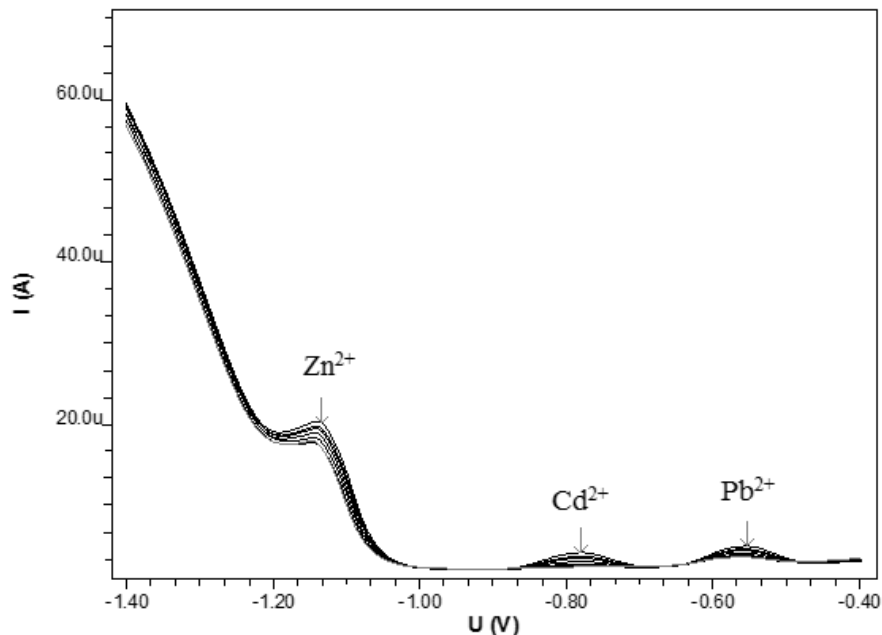


Figure 5.3 The stripping voltammograms for various concentrations (0, 0.1, 0.2, 0.4, 0.6, 0.8 and 1.0 $\mu\text{g L}^{-1}$) of cadmium lead and zinc ions in 0.1 M acetate buffer solution (pH 4.5) on the Bi-SPCE electrode

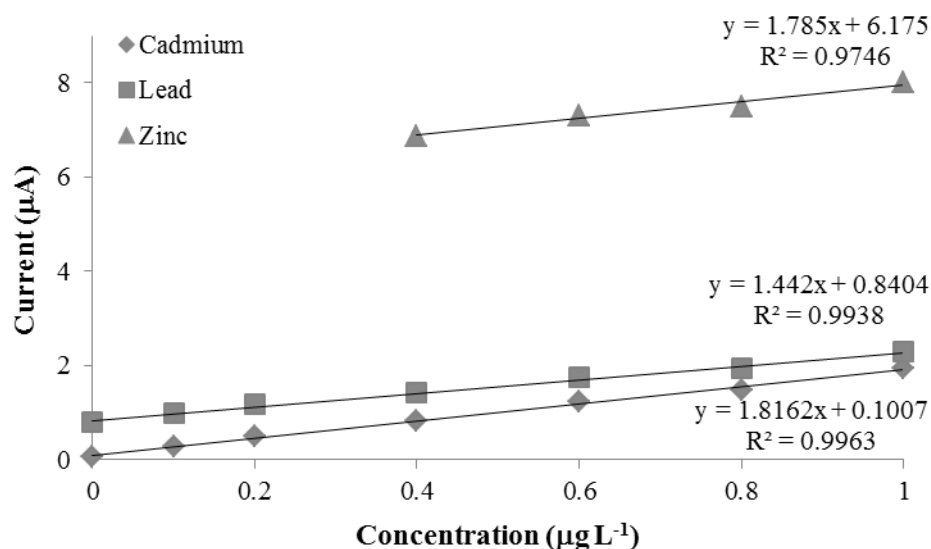


Figure 5.4 The calibration graphs for the simultaneous analysis of cadmium lead and zinc in 0.1 M acetate buffer solution (pH 4.5) on the Bi-SPCE electrode

Table 5.1 Calculation of detection limit of cadmium, lead and zinc by ASV with Bi-SPCE as a working electrode

Concentration ($\mu\text{g L}^{-1}$)	Peak current (μA)		
	Cadmium	Lead	Zinc
0	0.070	0.794	6.213
0.1	0.289	0.979	5.954
0.2	0.491	1.176	5.962
0.4	0.821	1.424	6.876
0.6	1.243	1.759	7.315
0.8	1.482	1.935	7.504
1.0	1.939	2.286	8.003
\bar{x} of blank signal	0.070	0.794	6.213
S.D. of y-intercept	0.05	0.05	0.09
LOD	0.06	0.06	0.17

For example, the detection limit of cadmium can be calculated as following:
the linear regression equation is $y = 1.8162x + 0.1007$

$$\text{LOD} = \frac{[(\bar{X} + 3\text{S.D.})] - c}{m}$$

$$\text{LOD} = \frac{[0.070 + 3(0.05)] - 0.1007}{1.8162}$$

$$\text{LOD} = 0.06$$

Therefore, the detection limit of cadmium is $0.06 \mu\text{g L}^{-1}$

2. The limits of detection of cadmium and lead in 4% (v/v) acetic acid solution (pH 4.5)

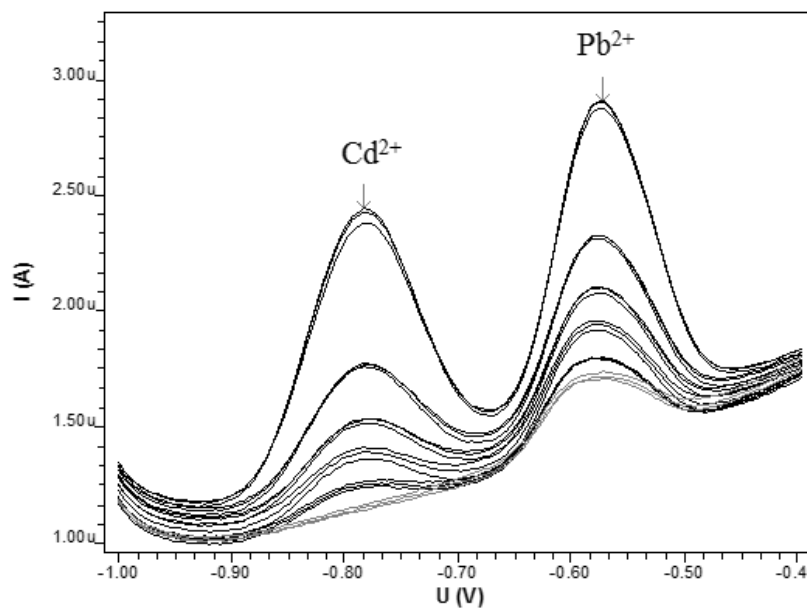


Figure 5.5 The stripping voltammograms for various concentrations (0, 0.1, 0.2, 0.4, 0.6, 0.8 and $1.0 \mu\text{g L}^{-1}$) of cadmium and lead ions in 4% (v/v) acetic acid (pH 4.5) on the Bi-SPCE electrode

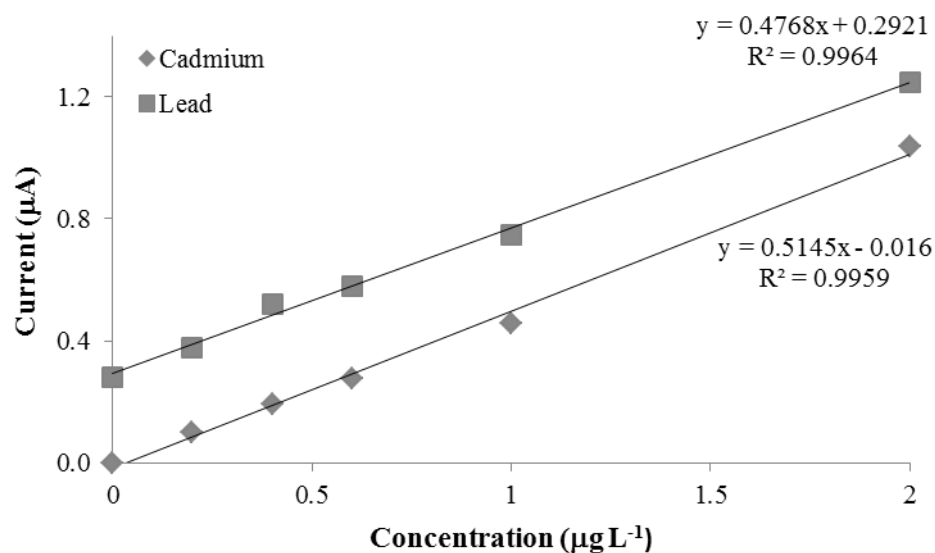


Figure 5.6 The calibration graphs for the simultaneous analysis of cadmium lead and zinc in 4% (v/v) acetic acid (pH 4.5) on the Bi-SPCE electrode

Table 5.2 Calculation of detection limit of cadmium and lead by ASV with Bi-SPCE as a working electrode

Concentration (µg L ⁻¹)	Peak current (µA)	
	Cadmium	Lead
0	0.000	0.280
0.2	0.101	0.377
0.4	0.193	0.521
0.6	0.277	0.580
1	0.458	0.748
2	1.036	1.249
\bar{x} of blank signal	0.000	0.280
S.D. of y-intercept	0.03	0.03
LOD	0.20	0.11

APPENDIX C

Chelex 100 resins column

To prepare the resin column, column is made by packing Chelex 100 resin in a 10 mL hypodermic syringe column for treatment of water and the picture of column is shown in Figure 5.7. Inner diameter and length of column are 1.5 and 8.0 cm, respectively. The cotton was blocked the Chelex 100 resin from loosing with the sample solution passing through the column.

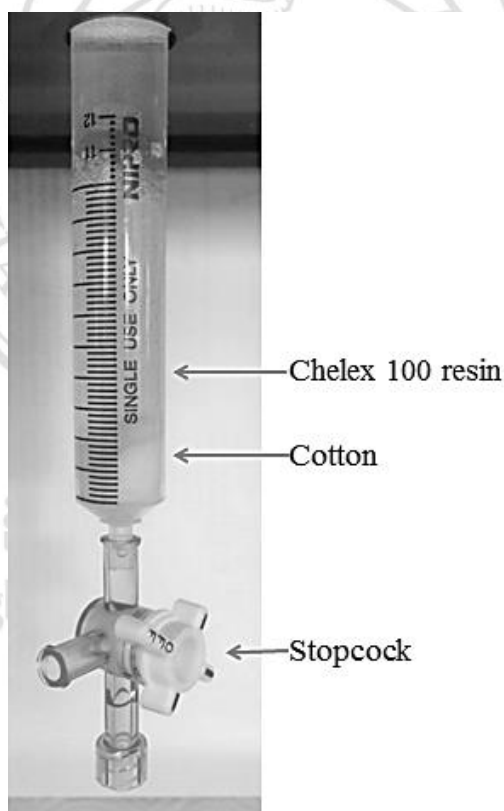


Figure 5.7 The Chelex 100 resin column

APPENDIX D

Calculation of release of cadmium and lead from ceramic wares

In my work, 80 mL of 4% (v/v) acetic acid pH 2.3 was filled in each sample, after 24 h the extracted solution was adjusted to pH 4.5 and made up to a final volume of 100 mL with 4% (v/v) acetic acid pH 4.5. The cadmium and lead concentration ($\mu\text{g L}^{-1}$) were found directly from the X-intercept of standard addition graphs. The cadmium and lead released per unit area ($\mu\text{g dm}^{-2}$) was calculated and the results are summarized in Table 5.3

Table 5.3 Calculation of release amounts of cadmium and lead from ceramic wares

Sample	Found ($\mu\text{g L}^{-1}$) ^a		Surface area of the extracted ceramic (cm^2)	Released amounts ($\mu\text{g dm}^{-2}$) ^a	
	Cadmium	Lead		Cadmium	Lead
No. 1	N.D.	1.97 ± 0.31	110	N.D.	0.179 ± 0.029
No. 2	N.D.	1.90 ± 0.39	98	N.D.	0.194 ± 0.040
No. 3	N.D.	1.81 ± 0.27	98	N.D.	0.185 ± 0.027
No. 4	N.D.	2.21 ± 0.05	106	N.D.	0.208 ± 0.004
No. 5	N.D.	2.52 ± 0.24	106	N.D.	0.237 ± 0.023
No. 6	N.D.	1.38 ± 0.17	110	N.D.	0.125 ± 0.015
No. 7	N.D.	3.28 ± 0.63	98	N.D.	0.334 ± 0.064

^a Average value of two determination \pm standard deviation

N.D. mean not detected

For example in sample No. 1, the lead released per unit area can be calculated as following the steps:

First: calculate the lead concentration ($\mu\text{g L}^{-1}$) in the filling volume of extract solution (80 mL) by

$$C_1V_1 = C_2V_2$$

$$C_1 = \frac{(1.97 \mu\text{g L}^{-1})(100 \text{ mL})}{(80 \text{ mL})}$$

$$C_1 = 2.46 \mu\text{g L}^{-1}$$

Second: calculate the surface area of ceramic ware sample by circle area ($2\pi rh$) combine with cylinder area (πr^2)

When; π is a mathematic constant (3.14)

r is a radius of ceramic ware sample (5.00 cm)

h is a height of extract solution level (1.00 cm)

$$\text{Surface area of ceramic ware} = 2\pi rh + \pi r^2$$

$$\text{Surface area of ceramic ware} = 2(3.14)(5.00 \text{ cm})(1.00 \text{ cm}) + (3.14)(5.00 \text{ cm})^2$$

$$\text{Surface area of ceramic ware} = 110 \text{ cm}^2 = 1.10 \text{ dm}^2$$

Third: calculate the release of lead concentration in milligrams per square centimeter ($\mu\text{g dm}^{-2}$) by

$$\text{Release of lead} = \frac{\text{lead concentration } (\mu\text{g L}^{-1}) \times \text{volume of extraction (L)}}{\text{surface area (dm}^2\text{)}}$$

$$\text{Release of lead} = \frac{2.46 (\mu\text{g L}^{-1}) \times 0.08 (\text{L})}{1.10 (\text{dm}^2)}$$

$$\text{Release of lead} = 0.179 \mu\text{g dm}^{-2}$$

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	2015	T. Sridara and J. Jakmune, "Determination of cadmium and lead in the acetic acid extract of glazed ceramic surfaces by anodic stripping voltammetry with bismuth-modified screen-printed

electrode (Poster Presentation)”, The 41th Congress on Science and Technology of Thailand (STT 41), Suranaree University of Technology, Nakhon Ratchasima, Thailand, 6-8 November 2015.

2016 T. Sridara and J. Jakmune, “Anodic stripping voltammetric method with bismuth-modified screen-printed electrode for determination of cadmium and lead in ceramic samples (Poster Presentation and Full paper Presentation)”, The 42th Congress on Science and Technology of Thailand (STT 42), Centara Grand Hotel, Bangkok, Thailand, 30 November-2 December 2016.

2016 T. Sridara and J. Jakmune, “Anodic stripping voltammetric method with bismuth-modified screen-printed electrode for determination of cadmium and lead in water treatment (Poster Presentation)”, The 13th Asian Conference on Analytical Sciences (ASIANALYSIS XIII), The Empress International Convention Center, Chiang Mai, Thailand, 8-11 December 2016.



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