Research Article

ปริมาณตะกั่วและแคดเมียมในตัวอย่างอายแชโดว์จากจังหวัดขอนแก่นและนครหลวงเวียงจันทน์

อุดสี บุนมีไช[1], วิรัช เรืองศรีตระกูล[2]*

[1]นักศึกษาบัณฑิตศึกษา, คณะเภสัชศาสตร์ มหาวิทยาลัยขอนแก่น อำเภอเมือง จังหวัดขอนแก่น 40002

[2]รองศาสตราจารย์, คณะเภสัชศาสตร์ มหาวิทยาลัยขอนแก่น อำเภอเมือง จังหวัดขอนแก่น 40002

* **ติดต่อผู้นิพนธ์ :** คณะเภสัชศาสตร์ มหาวิทยาลัยขอนแก่น อำเภอเมือง ขอนแก่น 40002 โทรศัพท์ 043-202378 โทรสาร 043-202379 อีเมล: wirat_ru@kku.ac.th

บทคัดย่อ

ปริมาณตะกั่วและแคดเมียมในตัวอย่างอายแชโดว์จากจังหวัดขอนแก่นและนครหลวงเวียงจันทน์

อุดสี บุนมีไช[1], วิรัช เรืองศรีตระกูล[2]*

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วัตถุประสงค์ของการศึกษานี้ ได้ทำตรวจวิเคราะห์ปริมาณตะกั่ว และแคดเมียม ในตัวอย่างอายแชโดว์จำนวน 90 ตัวอย่าง ตัวอย่างเหล่านี้ได้เก็บมาจากตลาดในเขตจังหวัดขอนแก่น ประเทศไทย (45 ตัวอย่าง) และนครหลวงเวียงจันทน์ ประเทศสาธารณรัฐประชาธิปไตยประชาชนลาว (45 ตัวอย่าง) ตัวอย่างได้แบ่งเป็นสามกลุ่มโดยใช้ราคาของตัวอย่างเป็นตัวกำหนด (1-40, 41-80 และ มากกว่า 80 บาท) **วัสดุและวิธีทดลอง:** การเตรียมตัวอย่างสำหรับการวิเคราะห์ปริมาณโลหะใช้เทคนิคการย่อยแบบเปียก ตัวอย่างอายแชโดว์จะนำมาย่อยด้วยกรดไนตริกที่มีความเข้มข้น 50 เปอร์เซ็นต์ ปริมาตรต่อปริมาตร ในหลอดย่อยสารแบบ PTFE และให้ความร้อนด้วยชุดให้ความร้อนเป็นเวลา 2-3 ชั่วโมง ตัวอย่างจากการย่อยนี้จะปล่อยให้เย็นที่อุณภูมิห้องและนำสารละลายใสที่ได้ไปปรับปริมาตรในขวดปรับปริมาตรขนาด 25 มิลลิลิตร สารละลายใสที่ได้จะถูกแยกด้วยเครื่องปั่นเหวี่ยงหนีศูนย์กลาง ที่ความเร็ว 6,000 รอบต่อนาที เป็นเวลา 3 นาที นำสารละลายที่ได้วัดค่าการดูดกลืนแสง ด้วยวิธีอะตอมมิกแอบซอบชั่นสเปกโทรโฟโตเมตรีที่ความยาวคลื่น 283.31 และ 228.80 นาโนเมตร สำหรับตะกั่วและแคดเมียม ตามลำดับ **ผลการศึกษา:** กราฟมาตรฐานของสารละลายมาตรฐานตะกั่วและแคดเมียม มีความเป็นเส้นตรงในช่วงความเข้มข้น เท่ากับ 0.25 – 3.0 ไมโครกรัมต่อมิลลิลิตร และ 0.05 – 3.0 ไมโครกรัมต่อมิลลิลิตร ตามลำดับ ภายในช่วงความเข้มข้นที่ศึกษานี้จะได้สมการสหสัมพันธ์ที่เป็นเส้นตรงระหว่างค่าการดูดกลืนแสง (y) และความเข้มข้น (x) ของตะกั่วและแคดเมียม ได้สมการ $y = 0.0159x - 0.0006$ ($r^2 = 0.9996$, $n = 7$) และ $y = 0.2937x + 0.0172$ ($r^2 = 0.9962$, $n = 7$) ตามลำดับ ค่าเฉลี่ยของปริมาณตะกั่วในกลุ่มราคาต่างๆ (1-40, 41-80 และ มากกว่า 80 บาท) มีค่าเท่ากับ 3.35, 1.41, 0.92 และ 4.92, 2.30, 1.06 ไมโครกรัมต่อกรัม สำหรับตัวอย่างอายแชโดว์จากจังหวัดขอนแก่น ประเทศไทย และนครหลวงเวียงจันทน์ ประเทศสาธารณรัฐประชาธิปไตยประชาชนลาว ตามลำดับ ค่าเฉลี่ยของปริมาณแคดเมียมในกลุ่มราคาต่างๆ (1-40, 41-80 และ มากกว่า 80 บาท) มีค่าเท่ากับ 0.14, 0.11, 0.10 และ 0.19, 0.14, 0.13 ไมโครกรัมต่อกรัม สำหรับตัวอย่างอายแชโดว์จากจังหวัดขอนแก่น ประเทศไทย และนครหลวงเวียงจันทน์ ประเทศสาธารณรัฐประชาธิปไตยประชาชนลาว ตามลำดับ **สรุปผล:** การศึกษานี้พบว่าตัวอย่างอายแชโดว์เกือบทั้งหมดมีการปนเปื้อนด้วยตะกั่ว (93.3 %) และแคดเมียม (88.8 %) มีการพบตัวอย่างเพียงสองตัวอย่างจากจังหวัดขอนแก่น ประเทศไทย และนครหลวงเวียงจันทน์ ประเทศสาธารณรัฐประชาธิปไตยประชาชนลาวที่มีปริมาณตะกั่ว (29.96 และ 40.05 ไมโครกรัมต่อกรัม) ซึ่งสูงเกินกว่าระดับที่อนุญาตให้พบได้ตามประกาศของกระทรวงสาธารณสุข 2559 (2016) ประเทศไทย การวิเคราะห์ข้อมูลในเชิงสถิติโดยใช้ One-Way ANOVA ไม่พบความแตกต่างกันอย่างมีนัยสำคัญทางสถิติของความสัมพันธ์ระหว่างช่วงราคากับปริมาณตะกั่วและแคดเมียมในตัวอย่างอายแชโดว์

คำสำคัญ: อายแชโดว์, ตะกั่ว, แคดเมียม, อะตอมมิกแอบซอบชั่นสเปกโทรโฟโตเมตรี

Lead and Cadmium Contents in Eye Shadows from Khon Kaen Province and Vientiane Capital

Aoutsy Bounmysay[1], Wirat Ruengsitagoon[2]*

[1]Graduate student, Faculty of Pharmaceutical Sciences, Khon Kaen University, Khon Kaen 40002, Thailand
[2]Associate Professor, Faculty of Pharmaceutical Sciences, Khon Kaen University, Khon Kaen 40002, Thailand
*** Corresponding author :** Faculty of Pharmaceutical Sciences, Khon Kaen University, Tel. +66 43 202 378;
Fax: +66 43 202 379. E-mail : wirat_ru@kku.ac.th

Abstract

Lead and Cadmium Contents in Eye Shadows from Khon Kaen Province and Vientiane Capital

Aoutsy Bounmysay[1], Wirat Ruengsitagoon[2]*

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The purpose of this study was the determination of lead (Pb) and cadmium (Cd) contents in 90 eye shadow samples which were collected from flea markets in Khon Kaen province, Thailand (45 samples) and Vientiane Capital, Lao PDR (45 samples). These samples were categorized into three ranges of the price (1-40, 41-80 and >80 Thai Baht). **Materials and methods:** Sample preparation for metal analysis was done by a wet digestion method. The sample was digested with 50 % v/v nitric acid in the PTFE vessel placed in a heat block apparatus for 2-3 h. The digestion was followed by cooling at room temperature and transferred the supernatant into 25 ml graduate volumetric flasks. The supernatant was separated by using the centrifuge apparatus at 6,000 RPM for 3 minutes. Atomic absorption spectrophotometry was measured the absorption values at 283.31 and 228.80 NM for the determination of Pb and Cd, respectively. **Results:** The calibration curves of Pb and Cd standard were linear in the rages of 0.25 –3.0 µg mL^{-1} and 0.05 – 3.0 µg mL^{-1}, respectively. Within these concentration ranges, linear regressions of the absorbance (y) and concentrations (x) of Pb and Cd expressed the equation $y = 0.0159x - 0.0006$ (r^2 = 0.9996, n=7) and $y = 0.2937x + 0.0172$ (r^2 = 0.9962, n = 7), respectively. It was found that the average concentration level of Pb in each price group (1-40, 41-80 and >80 Thai Baht) was 3.35, 1.41, 0.92 and 4.92, 2.30, 1.06 µg g^{-1} in eye shadow samples from Khon Kaen province and Vientiane Capital, respectively. The average concentration level of Cd in each price group (1-40, 41-80 and >80 Thai Baht) was shown to be 0.14, 0.11, 0.10 and 0.19, 0.14, 0.13 µg g^{-1} in eye shadow samples from Khon Kaen province and Vientiane Capital, respectively. **Conclusion:** The studies were found that almost eye shadow samples were contaminated with Pb (93.3 %) and Cd (88.8 %). These were found that only two eye shadow samples from Khon Kaen province, Thailand and Vientiane Capital, Lao PDR, gave concentration levels of Pb (29.96 and 40.05 µg g^{-1}) higher than the permissible limits from the Announcement 2559 (2016) of the Ministry of Public Health, Thailand. The statistical analysis using One-Way ANOVA, the result did not show a significant difference between ranges of price and amount of Pb or Cd in eye shadow samples.

Keywords: Eye shadows, Lead, Cadmium, Atomic absorption spectrophotometry

Introduction

Nowadays, rapid economic changed, industrialization was increased in many countries. Environmental, food, cosmetics and living area may be contaminated with pollutants including heavy metals which is one of the toxicant groups which strongly threaten human health through direct toxicity. According to the International Agency for Research on Cancer (IARC) classification, cadmium (Cd) and its compounds are in class I (carcinogenic to humans), lead (Pb) and its inorganic compounds are in class 2A (probably carcinogenic to humans), (Sah *et al.*, 2019). Heavy metal was the main target for investigating that have known as toxic substances. Interestingly, lead is a typical carcinogenic agent. Chronic poisoning with lead metal leads to a condition known as Saturnism, characterized by severe anemia, digestive disorder, nervous and renal failure. Cadmium is also a highly toxic substance that can cause cancer and disorders of bone, cardiovascular, renal, nervous, digestive, reproductive and respiratory systems (Malvadi and Sancholi, 2018).

Eye make-up includes a wide range of cosmetic products such as eye shadows, pencils, mascaras and eyeliners, each for a specific use. The eye shadows are preparations designed to enhance the depth of eyes thanks to a colored background that gives contour to the eyelid. The choice of shade depends upon the eye color we wish to compliment. These products exist in different forms, such as compact powders, which are the most commonly used. They are conditioned in cases with one shade only or a selection of several. The colored product is applied with a flexible brush or foam applicator (Salvador and Chisvert, 2007).

Eye shadow products are the wide variety of colors, which are produced by the addition of pigments, which may be mineral or organic and may contain Pb or Cd as impurities in the pigment formulation (Batista *et al.*, 2016). These metals may undergo retention and effect directly in the skin or be absorbed through the skin into the system, accumulate in the body, and exert to toxic effects on human organs. Pb and Cd have been described as wide-ranging toxic effects on many bodily systems, including the nervous, renal, musculoskeletal and cardiovascular systems (Salama, 2016). For Thailand, the permissible limits of Pb and Cd contamination in cosmetic should not be higher than 20 and 3 μg g^{-1} (The Ministry of Public Health of Thailand, 2016), respectively. Several methods have been reported previously for determined Pb and Cd in eye shadows such as atomic absorption spectrophotometry (Nkansah *et al.*, 2018), graphite furnace atomic absorption spectrophotometry (Ahmed *et al.*, 2017), inductively couple plasma – optical emission spectrophotometry (Alnuwaiser, 2018), inductively couple plasma –mass spectrophotometry (Volpe *et al.*, 2012) and capillary electrophoresis (Chen *et al.*, 2017). Atomic absorption spectrophotometric method is a simply used for metal analysis because this method was related with simple preparation processes, low-cost expense and high throughput rate for analysis samples (Butcher, 2013).

The aim of the study was to determine the concentration of Pb and Cd using flame atomic absorption spectrometry in eye shadow samples which were collected from Khon Kaen province, Thailand and Vientiane Capital, Lao PDR. The statistical analysis was studied the relationship between the price ranges of sample and amount of Pb or Cd level.

Materials and methods

Instrumentations

1. Flame atomic absorption spectrophotometer, PinAAcle 900F, PerkinElmer®, USA
2. Electrodeless discharge lamp (EDL) for lead, PerkinElmer®, USA
3. Electrodeless discharge lamp (EDL) for cadmium, PerkinElmer®, USA
4. Sample preparation heat block, SPB 100-12, PerkinElmer®, USA
5. Deionized distillated water unit (18.2 MΩ·cm), Millipore® Milli-Q; USA

Chemicals

1. Lead standard solution(Pb), Spectrosol grade, BDH; UK
2. Cadmium standard solution(Cd), Spectrosol grade, BDH; UK
3. Nitric acid(HNO_3), AR grade, Merck, Germany

Procedure

Sample collection

The 90 eye shadow samples were collected from Khon Kaen province, Thailand (45 samples) and Vientiane Capital, Lao PDR (45 samples). These samples were categorized into three ranges of price (1-40, 41-80 and >80 Thai Baht) in order to study the relationship among ranges of price and metal content in eyes shadow samples. All of eye shadow samples were purchased from a flea market or grocery stores in Khon Kaen province and Vientiane Capital (Figure 1).

Figure 1. Example of eye shadow samples collected from Khon Kaen province, Thailand and Vientiane Capital, Lao PDR.

Standard of lead and cadmium solution preparation

A stock standard solution of 50 µg mL^{-1} Pb or Cd was prepared by pipette 5 ml 1000 µg mL^{-1} commercial Pb or Cd into 100 ml graduate volumetric flasks. Each standard solution was adjusted volume to 100 ml with deionized distillated water. The calibration curve of the Pb standard solution was prepared by diluting 50 µg mL^{-1} stock Pb solution to working concentration of 0.25, 0.50, 0.75, 1.0, 2.0 and 3.0 µg mL^{-1}. The Pb stock solution was transferred to 50 ml graduate volumetric flasks in volume of 0.25, 0.50, 0.75, 1.0, 2.0 and 3.0 ml according to the working concentration, respectively, added deionized distilled water to the graduate mark. Cd standard solution in concentrations of 0.05, 0.1, 0.25, 0.5, 1.0, 2.0 and 3.0 µg mL^{-1} were prepared with pipet 50 µg mL^{-1} Cd stock standard solutions in the volume of 0.05, 0.1, 0.25, 0.5, 1.0, 2.0 and 3.0 ml and made up the solution to 50 ml with deionized distilled water which were given the required standard Cd solution.

All laboratory glasswares were soaked in 10% v/v nitric acid (Kazai et al., 2009) for 24 h; washed with distilled water and finally with deionized distilled water and dried in such a manner to ensure that no any metal contamination from glasswares.

Sample preparation

For analysis of Pb and Cd, 3 g of each eye shadow sample was placed in a PTFE vessel. Nitric acid (50% v/v) was added into a PTFE vessel of each eye shadows sample in 40 ml and heated by digestion block apparatus at 110 °C for 2-3 h. Afterward, these PTFE vessels were left to cool down at room temperature and transferred the supernatant into 25 ml graduate volumetric flasks. Then, sample solution was made up volume to graduated mark with deionize distillated water and transferred the adequate volume to the centrifuge tube. The clear solution was separated by centrifuge at 6,000 RPM for 3 minutes, then this supernatant was determined Pb and Cd concentration by using flame atomic absorption spectrophotometer (FAAS).

Atomic absorption spectrophotometric method

The analysis of Pb and Cd contents were determined in eye shadow samples from Khon Kaen province, Thailand and Vientiane Capital, Lao PDR. Flame atomic absorption spectrometry (FAAS) was carried out by the titanium burner head with 100 mm in length of single slot which were applied with air and acetylene gas. The electrodeless discharge lamp (EDL) for Pb and Cd were used as light source in order to increase the sensitivity of

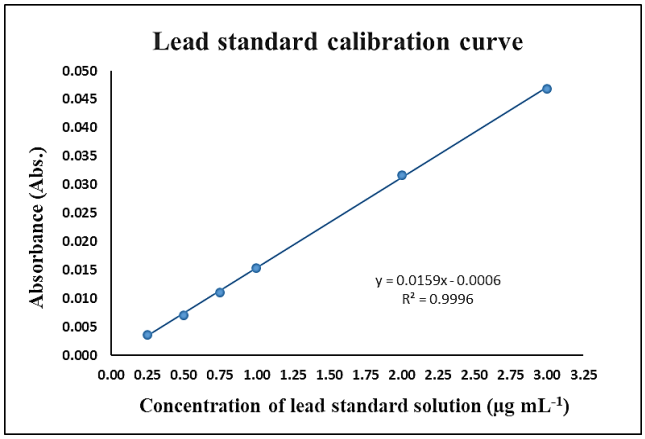
the analysis method. The appropriate wavelength for determination of Pb and Cd are 283.31 and 228.80 nm, respectively. The absorption value was then normalized using mathematical deconvolution to achieve delegate data represent the concentration scale of each standard. The delegate data were then plotted against concentration to obtain calibration curve where the suspected samples were correlated to predict its unknown concentration.

Results

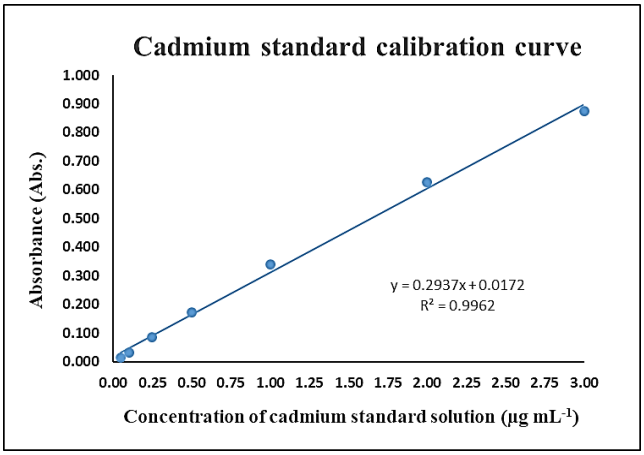
Analytical characteristics

The linear calibration curve of Pb and Cd were investigated in concentration ranges of 0.25 – 3.0 µg mL^{-1} and 0.05 – 3.0 µg mL^{-1}, respectively. Within these concentration ranges, linear regressions of the absorbance (y) and concentrations (x) of Pb and Cd expressed the equation $y = 0.0159x - 0.0006$ ($r^2 = 0.9996$, n=7) and $y = 0.2937x + 0.0172$ ($r^2 = 0.9962$, $n = 7$), respectively (Figure 2). The detection limit was defined as the concentration of analyte that gave the signal that was different from the bank by an amount equal to three times the standard deviation of the bank signal. These were found to be 0.10 µg mL^{-1} for Pb and 0.02 µg mL^{-1} for a Cd standard solution. The quantification limit is defined as the analyst producing a signal at least ten times the standard deviation of the blank signal, and were shown to be 0.33 and 0.07 µg mL^{-1} for Pb and Cd standard solution, respectively.

The precision of the proposed method was studied through the repeatability by investigating seven replicated of two concentrations (0.3 and 1.0 µg mL^{-1}) of Pb and Cd standard solution. The relative standard deviation (RSD) of these studies was found to be less than 0.03 % of each. Accuracy was tested by means of recovery determination. The percentage recoveries of two concentrations (0.3 and 1.0 µg mL^{-1}) of Pb and Cd standard solutions (n=7) were found to be in the range of 97.21% – 104.71% (Table 1).



(a)



(b)

Figure 2. Calibration curve, **(a)** Lead standard solution, **(b)** Cadmium standard solution

Table 1. Analytical characteristics of proposed method for determination of Pb and Cd.

Parameters	Optimum value for Pb determination	Optimum value for Cd determination
Linearity of calibration curve	0.25 – 3.0 µg mL^{-1}	0.05 – 3.0 µg mL^{-1}
Linear regression equation	y = 0.0159x - 0.0006	y = 0.2937x + 0.0172
Correlation coefficient, r^2	0.9996	0.9962
Limit of detection, LOD	0.10 µg mL^{-1}	0.02 µg mL^{-1}
Limit of quantitation, LOQ	0.33 µg mL^{-1}	0.07 µg mL^{-1}
Repeatability (n=7); RSD.		
0.30 µg mL^{-1}	0.0004 %	0.0295 %
1.00 µg mL^{-1}	0.0005 %	0.0388 %
Percentage recoveries (n=7)		
0.30 µg mL^{-1}	97.73 %	97.21 %
1.00 µg mL^{-1}	103.91 %	104.71 %

Table 2. Concentration level of Pb in eye shadow samples from Khon Kaen province, Thailand and Vientiane Capital, Lao PDR.

Eye shadows sample No	Pb level in eye shadow samples from Khon Kaen, Thailand (µg g^{-1}), n=45*			Pb level in eye shadow samples from Vientiane Capital, Lao PDR (µg g^{-1}), n=45*		
	Price ranges of collected sample (Thai Baht)			Price ranges of collected sample (Thai Baht)		
	1-40	41-80	>80	1-40	41-80	>80
1	0.31	ND	ND	0.53	2.83	2.21
2	1.15	0.46	0.71	0.79	14.47	ND
3	1.20	1.31	ND	4.99	0.74	1.58
4	1.15	0.49	1.59	ND	3.27	0.21
5	0.27	0.29	1.26	ND	1.10	1.30
6	0.74	0.80	0.34	3.35	0.64	0.12
7	0.41	0.84	0.65	0.51	2.86	0.34
8	29.96	2.07	1.05	0.59	0.92	0.41
9	0.76	3.36	0.62	0.47	0.81	0.58
10	2.20	1.98	0.72	1.01	1.31	1.32
11	0.68	4.87	0.32	0.62	2.10	0.36
12	0.72	1.12	1.09	40.05	0.85	2.31
13	3.65	1.18	2.10	5.83	1.33	2.16
14	6.37	0.69	0.65	3.57	0.45	1.42
15	0.70	0.28	0.81	1.65	0.81	0.57
Average ±sd	3.35±7.53	1.41±1.30	0.92±0.50	4.92±10.71	2.30±3.48	1.06±0.79

ND: None detected

*: Triplicate analysis

Table 3. Concentration level of Cd in eye shadow samples from Khon Kaen province, Thailand and Vientiane Capital, Lao PDR.

Eye shadows sample No	Cd level of eyeshadow samples from Khon Kaen, Thailand (μg g^{-1}), *n*=45*			Cd level of eye shadow samples from Vientiane Capital, Lao PDR (μg g^{-1}), *n*=45*		
	Price ranges of collected sample (Thai Baht)			Price ranges of collected sample (Thai Baht)		
	1-40	41-80	>80	1-40	41-80	>80
1	0.11	0.08	ND	0.13	0.17	0.13
2	0.08	0.13	0.07	0.12	0.33	0.18
3	0.09	0.09	ND	0.33	0.19	0.13
4	ND	0.07	0.12	0.07	0.15	0.10
5	ND	ND	0.08	0.29	0.09	0.18
6	0.08	0.07	0.23	0.04	0.19	0.17
7	0.06	0.09	0.06	0.23	0.06	0.04
8	0.32	0.12	0.06	0.26	0.06	0.05
9	0.22	0.05	0.09	0.10	0.19	ND
10	0.24	ND	0.08	0.08	0.11	ND
11	0.09	0.25	0.09	0.21	0.07	0.12
12	0.08	0.16	ND	0.60	0.09	0.13
13	0.07	0.14	0.13	0.10	0.15	ND
14	0.21	ND	0.14	0.25	ND	0.18
15	0.17	0.09	0.06	0.05	0.07	0.18
Average ±sd	0.14±0.08	0.11±0.05	0.10±0.04	0.19±0.14	0.14±0.07	0.13±0.04

ND: None detected

*: Triplicate analysis

Table 4. Concentrations of Pb and Cd in eye shadow samples.

Eye shadows sample	Pb level found in ranges, (μg g^{-1})	Average, *n*=45 (μg g^{-1})	Number of sample higher than the permissible limits* (*n*)	Cd level found in ranges, (μg g^{-1})	Average, *n*=45 (μg g^{-1})	Number of sample higher than the permissible limits** (*n*)
Khon Kaen, Thailand, *n*=45	ND –29.96	1.82	1 (2.2 %)	ND – 0.32	0.10	–
Vientiane, Lao PDR, *n*=45	ND –40.05	2.51	1 (2.2 %)	ND – 0.59	0.14	–

ND: None detected

* The permissible limits for Pb; 20 μg g^{-1} (The Ministry of Public Health of Thailand, 2016).

**The permissible limits for Cd; 3 μg g^{-1} (The Ministry of Public Health of Thailand, 2016).

The 90 eye shadow samples were examined Pb and Cd contents. Eye shadow samples were collected from Khon Kaen province, Thailand and Vientiane Capital, Lao PDR, which were categorized into three ranges of price (1-40, 41-80 and >80 Thai Baht). It was found that the average concentration level of Pb in each price group (1-40, 41-80 and >80 Thai Baht) were 3.35, 1.41, 0.92 and 4.92, 2.30, 1.06 μg g^{-1} for eye shadow samples from Khon Kaen province and Vientiane Capital, respectively. The average concentration level of Cd in each price group (1-40, 41-80 and >80 Thai Baht) were shown to be 0.14, 0.11, 0.10 and 0.19, 0.14, 0.13 μg g^{-1} for eye shadow samples from Khon Kaen province and Vientiane Capital, respectively (Table 2, 3). Interestingly, the studied did not show significant difference among ranges of price and amount of Pb or Cd in eye shadow samples by using One-Way ANOVA for independent measure at 95% confidence.

The average concentration levels of Pb were found to be 1.82 and 2.51 μg g^{-1} for 45 eye shadow samples which were collected from Khon Kaen province, Thailand and Vientiane Capital, Lao PDR, respectively. The average concentration levels of Cd were shown to be 0.10 and 0.14 μg g^{-1} for eye shadow samples collected from Khon Kaen province, Thailand and Vientiane Capital, Lao PDR, respectively (Table 4). These were found that almost all of the samples gave the Pb and Cd concentrations not higher than the permissible limits which was noticed by The Ministry of Public Health of Thailand.

Discussion and Conclusions

Pb and Cd were investigated from eye shadow samples which were collected from Khon Kaen province, Thailand and Vientiane Capital, Lao PDR. These samples were categorized to three price ranges of samples (1-40, 41-80 and >80 Thai Baht). The studies were found that almost all of the samples were contaminated with Pb (93.3 %) and Cd (88.8 %). Interestingly, the samples with none fluorescence color, fine and good look packaging may be safer than others because all of these were found low levels of Pb and Cd contamination. These were found that only two eye shadow samples from Khon Kaen province, Thailand and Vientiane Capital, Lao PDR, gave a Pb level (29.96 and 40.05 μg g^{-1}) higher than the permissible limits from the Announcement 2559 (2016) of the Ministry of Public Health, Thailand. All of Cd levels from Khon Kaen province, Thailand and Vientiane Capital, Lao PDR, are not higher than the notice the permissible limits. The statistical analysis using One-Way ANOVA, the result did not show significant difference between the range of price and amount of Pb or Cd in eye shadow samples. For the benefit of consumers, quality and safety products of eye shadow should be concerned.

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