

## Assessment of Permissible Limits for Heavy Metals in Various Inspired and Authentic Lipsticks

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Heavy metals in lipsticks is a safety threat to the health of consumers. The analytical information on heavy metals in lipsticks is crucial to increase the awareness of users. This study measured and compared the concentration of lead (Pb), cadmium (Cd), chromium (Cr), and copper (Cu) in authentic and inspired lipsticks. A total of 24 lipsticks (12 authentic and 12 inspired) were digested using wet acid digestion method and the elements were analyzed by using atomic absorption spectroscopy (AAS). Based on the permissible levels in lipstick set by the United States Food and Drug Administration (US FDA), all the heavy metals analyzed were detected in three brands of inspired lipsticks. These results revealed that the inspired lipsticks may not be safe to be used by the public.

**Key words:** Heavy metal; health; inspired lipstick; authentic lipstick; atomic absorption spectroscopy

*Received: July 2019; Accepted: April 2020*

Lipstick is a cosmetic product applied on the lips for the users to promote their appearance, attractiveness, and beauty in the modern lifestyle. Generally, lipsticks are composed of wax, oil, and coloring agents, and also added with many other auxiliary components to enhance their desired effects depending on the manufacturers [1,2]. Consequently, the end products reach users with a wide variety of colors arising from the intentional addition of pigments in the stage of production. The pigment formulations are believed to contain metals, such as cadmium (Cd), chromium (Cr), lead (Pb), and copper (Cu) as impurities [3,4,5]. Cd, Cr, Pb, and Cu are considered as heavy metals, which are the concern of lipstick users due to their potential toxicity that may cause detrimental effects on the human health, particularly on the nervous, digestive, reproductive, respiration, and integumentary systems [2,5 6,7]. Previous studies recorded the concentration of Cd, Pb, Cr, and Cu with the range of 0.06 – 10.6 mg/kg [4,8], 0.11 - 20 mg/L [9,10], 0.48 – 39.4 mg/kg [4,8] and 19.71 µg/g - 118.9 mg/kg [11,12] in lipstick samples, respectively.

Several countries have implemented regulations to control and monitor the permissible levels of heavy metal contents in cosmetic products. Based on the Guidelines of Control of Cosmetic Products in Malaysia in accordance to the ASEAN Cosmetic Directive, Pb, Cd, and Cr are prohibited in cosmetic products [4]. Recently, some inspired lipsticks were smuggled into the local market. The inspired lipsticks might cause harm to the health of consumers, due to unknown materials or chemicals used to produce them. Nevertheless, they were welcomed by consumers due to the lower price compared with the authentic lipsticks [13,14]. According to Alias *et al.* [14], most of inspired lipsticks had different packaging compared to authentic lipsticks. In addition, Zakaria and Ho [4] found that most of inspired cosmetics have higher concentrations of heavy metals compared to authentic cosmetics. Thereby, the determination of the concentration of heavy metals in lipsticks, particularly for inspired lipsticks is crucial to increase the awareness of consumers.

The elemental information of lipstick samples

had been explored by several authors using various instrumental techniques, including atomic absorption spectroscopy [3,6], inductively coupled plasma-mass spectrometry [15, 16, 17], inductively coupled plasma-optical emission spectroscopy [4,5], and x-ray fluorescence [18]. These previous studies have successfully measured the heavy metal contents in authentic lipsticks; however, to our knowledge, there was no study conducted to assess and compare the heavy metal levels in both authentic and inspired lipsticks. Therefore, in this study, authentic and inspired lipsticks were physically and chemically examined to detect and compare the heavy metal contents in the samples.

## MATERIALS AND METHODS

### Chemicals and Reagents

Trace metal grade 65% nitric acid (HNO<sub>3</sub>) and 30% hydrogen peroxide (H<sub>2</sub>O<sub>2</sub>) were obtained from HMBG and Merck, respectively. The element stock solutions, copper (Cu), chromium (Cr), cadmium (Cd), and lead (Pb), were obtained from Merck, each with the concentration of 1000 mg/L.

### Preparation of Calibration Standards

Each of the element standard solutions (Cu, Cd, Cr, and Pb) was prepared into five different concentrations by

serial dilution from 1000 mg/L of the standard stock solution. Copper and chromium were diluted to obtain a calibration range from 0.3125 mg/L to 5.0 mg/L. Cadmium was diluted from 1000 mg/L stock solution into the range of 0.125 mg/L to 2.0 mg/L, while lead was diluted from 1000 mg/L stock solution into the range of 1.25 mg/L to 20 mg/L.

### Lipstick Sample Collection

A total of twenty-four lipstick samples, twelve authentic lipsticks and twelve inspired lipsticks, were purchased from the drugstore and local markets. Based on the labels, the twelve authentic lipsticks were imported from three different countries, i.e., United State, United Kingdom, and South Korea, while the origins of the inspired lipsticks were unknown. Table 1 lists the authentic and inspired lipsticks based on the brands, codes, and country of origin.

### Visual Examination

A total of twenty-four lipstick samples were smeared on a white A4 paper to facilitate the visual examination. The authentic lipsticks were used as the control materials with only red color code was considered in this study. The lipstick stain showed different shades with the control materials even had similar code which indicated that sample as inspired lipstick. The visual comparison was conducted under white light.

**Table 1.** Information on the lipstick samples

Authentic lipstick (n= 12)				Inspired Lipstick (n=12)			
Sample	Brands	Red Color Code	Origin	Sample	Brands	Red Color Code	Origin
A1		Please Me		1A		Please Me	-
A2	Mac	Russian Red	United State	2A	Mac	Russian Red	
A3		Ruby Woo		3A		Ruby Woo	
B1		Kiss Woo		1B		02	-
B2	3CE	Hypnotic	South Korea	2B	3CE	11	
B3		Keen		3B		04	
C1		Tilt		1C		13	-
C2	Urban Decay	Broadcast	United State	2C	Urban Decay	07	
C3		Wired		3C		18	
D1		Ma'damn		1D		11	-
D2	Fenty beauty	PMS	United Kingdom	2D	Fenty Beauty	07	
D3		Spanked		3D		01	

### Lipstick Sample Preparation

The sample preparation was conducted using wet acid digestion according to the method used by Al-Saleh and Al-Enazi [3]. 200 mg ( $\pm 0.1$  mg) of lipstick sample was weighed using an analytical balance in a clean 100 mL beaker, and subsequently added with 4 mL of concentrated  $\text{HNO}_3$ . The beaker was covered with a watch glass and left for 4 hours at room temperature, followed by heating in an oven overnight at  $77^\circ\text{C}$ . After the first stage digestion, the sample was cooled at room temperature. Subsequently, the beaker was added with 1 mL of 30%  $\text{H}_2\text{O}_2$  and subjected to oven heating for another hour at  $77^\circ\text{C}$  to completely oxidize the organic matter of the residues. The digested sample was filtered using 55 mm filter paper and diluted with deionized water to 10 mL. A reagent blank solution was also used as a negative control. This study could not be validated by certified reference material (CRM) since lipstick is not available as CRM for heavy metal composition as reported by Edgar *et al.* [17].

### Instrumental Analysis

Perkin Elmer AAnalyst800 Atomic Absorption Spectrometry (AAS) available in the Analytical Laboratory, Universiti Sains Malaysia was utilized in this study. Prior to sample analysis, the instrument was calibrated with all the target elements at a five calibration points. Upon generation of the calibration curve, the lipstick samples prepared in solutions were aspirated into the AAS system. For the quantitative analysis, the correlation coefficient ( $r^2$ ) of the calibration curves of Cd, Cr, Pb, and Cu were 0.9996, 1.0000, 0.9998, and 0.9999, respectively.

### Data Analysis

The concentrations of the metals in the lipstick samples

were compared with the permissible levels set by the United States Food and Drug Administration (US FDA) listed in Table 2 [19]. However, Hostynek [20] reported that there was no specific rules or permissible limits for Cu in lipstick. This was due to the fact that humans can tolerate higher amounts of Cu as claimed by Lenntech [21]. Data interpretation was carried out using response surface methodology (RSM). Box-Behnken design (BBD) of RSM was used to determine the differences of the heavy metal concentrations between the authentic and inspired lipsticks at a significance level of  $p < 0.05$ . The concentrations of heavy metals (Cu, Cd, Pb, and Cr) in different brands of authentic and inspired lipsticks were compared and reported in this study.

## RESULTS AND DISCUSSION

### Visual Examination

The color of the authentic and inspired lipstick samples was examined using visual examination. Table 3 demonstrates the differences in the shades of color between the authentic and inspired lipsticks.

In general, the color of the inspired lipstick samples was lighter than the authentic lipsticks. Most of the authentic lipsticks were dark in color and less oily while the inspired lipsticks were light in color. Two of the inspired lipstick samples (1C and 2C) were shimmering when examined by visualization. The shimmer effect may due to pearlescent pigments, such as mica (aluminosilicates), included during the manufacturing process [16,22]. However, the concentrations of heavy metals cannot be measured based on the physical examination only. There must be chemical analyses conducted in order to detect and confirm the presence of the heavy metals in the lipsticks.

**Table 2.** Permissible limits for concentrations of Cd, Cr, Pb, and Cu set by the US FDA

Heavy metal element	Permissible limit (mg/L)
Cd	3
Pb	20
Cr	5

**Table 3.** Differences in shades of color between authentic and inspired lipstick samples

<b>Brand</b>	<b>Authentic</b>	<b>Inspired</b>
<b>Mac</b>	A1	1A
	A2	2A
	A3	3A
<b>3CE</b>	B1	1B
	B2	2B
	B3	3B
<b>Urban Decay</b>	C1	1C
	C2	2C
	C3	3C
<b>Fenty Beauty</b>	D1	1D
	D2	2D
	D3	3D

**Table 4.** Concentrations of heavy metals in authentic and inspired lipsticks

Authentic Lipstick					Inspired Lipstick				
Sample	Cd (mg/kg)	Cr (mg/kg)	Pb (mg/kg)	Cu (mg/kg)	Sample	Cd (mg/kg)	Cr (mg/kg)	Pb (mg/kg)	Cu (mg/kg)
A1	1.35 ±0.17	5.48 ±0.44	11.15 ±3.5	18.46 ±0.15	1A	1.41 ±0.19	8.93 ±0.17	20.39 ±1.47	17.17 ±0.36
A2	1.71 ±0.29	6.95 ±0.75	9.76 ±1.85	22.1 ±0.29	2A	1.76 ±0.13	20.48 ±0.5	19.14 ±0.57	23.95 ±0.20
A3	1.47± 0.12	5.69 ±0.35	8.02 ±3.72	10.41 ±0.05	3A	0.80 ±0.24	8.75 ±0.25	24.20 ±1.18	19.65 ±0.03
B1	2.42 ±0.21	9.95 ±0.12	19.13 ±3.61	31.11 ±0.41	1B	2.14 ±0.28	8.61 ±0.29	20.7 ±3.24	40.00 ±0.44
B2	1.62 ±0.12	4.71 ±0.31	9.66 ±3.5	15.69 ±0.25	2B	11.90 ±0.17	4.5 ±0.02	6.5 ±0.95	11.90 ±0.10
B3	2.38 ±0.12	9.80 ±0.03	21.53 ±1.82	41.93 ±0.44	3B	2.50 ±0.71	7.79 ±0.65	8.04 ±1.32	21.86 ±0.33
C1	0.43 ±0.14	ND <sup>a</sup>	ND <sup>a</sup>	4.23 ±0.03	1C	1.00 ±0.11	BDL <sup>b</sup>	BDL <sup>b</sup>	4.19 ±0.13
C2	0.60 ±0.08	ND <sup>a</sup>	ND <sup>a</sup>	4.15 ±0.07	2C	0.55 ±0.22	85.8 ±0.8	355.20 ±1.78	8.20 ±0.15
C3	0.84 ±0.34	ND <sup>a</sup>	ND <sup>a</sup>	4.51 ±0.19	3C	0.86 ±0.12	2.58 ±0.50	ND <sup>a</sup>	9.29 ±0.09
D1	ND <sup>a</sup>	ND <sup>a</sup>	4.98 ±2.38	ND <sup>a</sup>	1D	0.76 ±0.41	6.52 ±0.20	76.86 ±1.04	11.67 ±0.11
D2	ND <sup>a</sup>	ND <sup>a</sup>	7.46 ±1.96	ND <sup>a</sup>	2D	0.59 ±0.14	1.23 ±0.46	16.36 ±1.2	1.32 ±0.09
D3	0.15 ±0.41	ND <sup>a</sup>	8.69 ±1.66	ND <sup>a</sup>	3D	1.11 ±0.41	7.09 ±0.18	62.56 ±0.83	10.85 ±0.13

<sup>a</sup> ND: not detected.

<sup>b</sup> BDL: below detection limit.

### Heavy Metal Analysis

The concentration of each heavy metal in the lipstick samples is given in Table 4. The results are reported as the mean ±SD for each metal. Based on Table 4, the highest concentration of Cd was found in inspired lipstick 2B at 11.9±0.17 mg/kg. Cadmium is one of inorganic impurities which is present in the coloring agents of cosmetic products such as lipstick and face powder [4,5,17]. The highest concentration of Cd and Pb in inspired lipstick 2C were 85.8±0.8 and 355.2±1.78 mg/kg, respectively. Chromium may present as chromium(III) oxide, chromium(III) hydroxide, or chromium(IV) in the color ingredient of lipstick [2], while lead acts as the synthetic pearlescent pigment which increases the shimmer effect of lipstick [22]. Copper was shown at the highest concentration at 41.93±0.44 mg/kg in authentic lipstick B3. Copper is the nanoparticle introduced into cosmetic products to replace synthetic preservatives [2]. Cadmium, chromium, and lead are metals which are prohibiting or restricting with exception in the cosmetic regulation.

Based on the Guidelines of Control of Cosmetic Products in Malaysia in accordance with the ASEAN Cosmetic Directive, lead, cadmium, and chromium are metals which are not allowed in cosmetic products [4,13]. In accordance to the Regulation No. 1223/2009 of the European Parliament, Cd, Pb, and Cr(IV) are banned in cosmetic products, while Cu, Cr<sub>2</sub>O<sub>3</sub>, and Cr(OH)<sub>3</sub> are allowed as colorants in cosmetics [2,23]. However, the presence of heavy metals in lipstick is unavoidable even under the condition of good manufacturing practice (GMP) due to their persistence and natural availability in the environment, particularly in rocks, soil, and water [2,3,16,17]. Owing to their ubiquitous properties in the environment, the US FDA has only restricted mercury (Hg) with exception and set the permissible levels for Pb and arsenic in cosmetic products [2]. Therefore, the presence of heavy metals (Pb, Cd, and Cr) in the authentic lipsticks can be explained. Nevertheless, the higher concentrations of heavy metals (Pb, Cd, and Cr) in the inspired lipsticks may due to uncontrollable or intentional addition of inorganic pigments into the lipsticks by manufacturers

without implementing the manufacturing regulation of cosmetic products in order to enhance the quality of the products [9,24].

### Comparison of Concentrations of Heavy Metals in lipsticks against US FDA

The concentrations of all the metals detected in all brands (Table 4) were compared with the US FDA limits (Table 2) to ensure the safety of the lipsticks for consumer use. Although lipstick users may only be exposed to small amounts of heavy metals when using lipsticks, the accumulation of heavy metals in the body of users over prolonged period of time may cause chronic health effects [4].

Both authentic and inspired lipsticks under the brand of Mac showed the concentration Cu and Cd under the permissible limits, but Cr was found to be exceeding the permissible limit of the US FDA. However, for inspired lipsticks labelled with this brand, the concentration of Pb exceeded the permissible limit provided by the US FDA.

Table 4 shows that the concentration of Cr in several of the red color codes registered under authentic brand of 3CE exceeded the permissible limit that was set by the US FDA, while only one of the red color code samples, B3 recorded a higher amount of Pb than the US FDA limit. Conversely, for the inspired lipstick samples under the same brand, all were proved that the amounts of heavy metals in these samples were higher than allowed limits by the US FDA.

The concentration of all heavy metals in the samples under authentic brand of Urban Decay was found to be within the permissible limits. Thus, it can be said that they are safe to be used by consumers. But, for inspired lipsticks under the same brand, the content of Pb and Cr in inspired lipstick sample 2C exceeded the permissible standard provided by the US FDA.

Among all the brands tested in this study, both authentic and inspired lipsticks labelled under the brand of Fenty beauty proved to be safe since some of the heavy metals were not detected, which might be due to the trace amounts of these metals in the samples. Although Pb was detected in both groups of samples, its concentration was still in the safe limit to be used by consumers.

All of the inspired lipsticks which were labelled under three brands, MAC, 3CE, and Urban Decay are discouraged to be used due to the samples contained higher amounts of Pb which exceeded the permissible levels of the US FDA. Lead is one of the carcinogenic elements which potentially threatens the health safety of lipstick users [5]. After a lipstick which contains a high level of Pb is applied to the lips, Pb will be absorbed

from the gastrointestinal tract into the blood circulation. Subsequently, it will distribute and accumulate in the internal organs which then cause systematic effects such as blood systematic, teratogenicity, and neurological disorder [2,4,6,25].

In addition, the data from BBD of RSM showed that there were statistically significant differences between the authentic and inspired lipsticks in terms of the concentration of the heavy metals analyzed ( $p < 0.05$ ) for three brands. However, p-value more than 0.05 was obtained for Fenty Beauty brand, indicating there was no significant difference of heavy metal contents for both groups of lipsticks.

### CONCLUSION

This study successfully determined the levels of Cr, Cd, Pb, and Cu in the authentic and inspired lipsticks. This study revealed that all the heavy metals analyzed were detected in the inspired lipsticks for three brands (MAC, 3CE, and Urban Decay). Therefore, the inspired lipsticks which were smuggled into the market are discouraged to be purchased and used by consumers due to the potentially high levels of heavy metal contents in the cosmetic products which could cause detrimental health effects for consumers.

### ACKNOWLEDGEMENTS

The authors greatly appreciate the financial support received from the Universiti Sains Malaysia (USM) under the Short Term Grant Scheme (304/PPSK/6315125).

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