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Detection of Toxic Metals in Lipsticks Products in Riyadh, Saudi Arabia

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ABSTRACT

Since the dawn of civilization cosmetics have constituted a part of routine body care not only by the upper strata of society but also by middle and low class people. Heavy metals contamination in cosmetic products is becoming an important health problem in both worldwide and locally at the level of the Kingdom of Saudi Arabia (KSA). The aim of this study was to study. Quantitatively estimated heavy metals "lead, cadmium, mercury and Arsenic" using graphite. Total of 21 popular brands used lipstick products sold in Riyadh market samples from 3 different types of lipsticks frequently used among females in Saudi Arabia was digested. The digested samples were analyzed for lead, cadmium, mercury and Arsenic using graphite furnace- atomic absorption spectrometry. The mercury concentration was high followed by arsenic and cadmium, finally lead. The results indicate that the toxic heavy metals in all the samples were below the US FDA and SFDA permissible limits for cosmetic products with the exception the mercury content in some lipstick samples was higher than SFDA. There was no significant difference among the lipsticks in price categories. Continuous use and possible unintended ingestion of these toxic heavy metals, though in low levels in the cosmetics, may pose potential health risk due to their bioaccumulation in body organs.

Keywords: Lipsticks, Toxic metals, Atomic Absorption Spectroscopy.

INTRODUCTION

Cosmetics have come to stay as part of products we use on a daily basis. With its usage also comes the undesirable threat of effects of heavy metals, which may be present in these products in levels exceeding the permissible, on the human body. It is undeniable that at certain tolerated limits, some heavy metals could be of biological importance to man¹. However others (like As, Pb, Cd) have been reported to have no known bio-importance and can be very toxic when consumed even at very low concentrations²⁻⁸. The nature of the effects could be toxic (acute, chronic or sub-chronic), neurotoxic, carcinogenic, mutagenic or teratogenic¹. Cadmium as stated earlier is toxic at extremely low levels. Long term exposure to cadmium leads to renal dysfunction and high exposure can lead to obstructive lung disease and cadmium pneumonitis resulting from inhaled dusts and fumes1. Lead, the most significant toxin of the heavy metals, can be ingested through food and water in its inorganic form which is easily absorbed by the body⁵. Lead poisoning causes inhibition of hemoglobin synthesis, kidney dysfunction reproductive and cardiovascular systems dysfunction9-11. Lead affects the development of the grey matter of the brain in children resulting in poor intelligence quotient (IQ)¹². Mercury compounds are readily absorbed through the skin on topical application and have the tendency to accumulate in the body. They may cause allergic reactions, skin irritation or neurotoxic manifestations¹³. Mercury intoxication from cosmetics has been featured in numerous news stories in recent years. There is evidence suggesting children who had been exposed in-utero from their mother's experienced developmental issues. These children were affected with a range of symptoms including motor difficulties, sensory problems and mental retardation ¹³. Arsenic, cadmium, lead and mercury are described as heavy metals which in their standard state have a specific gravity (density) of more than about 5g/cm3 (Arsenic, 5.7; cadmium, 8.65; lead, 11.34; and mercury, 13.549) while metals like copper, nickel, chromium and iron are essential in very low concentration for the survival of all forms of life, but, when present in higher concentration can cause metabolic anomalies14.

There has been studies and scientific debate on the exposure of the eyes to lead as a result of the use of cosmetics¹⁵⁻¹⁹. Underarm cosmetics are being investigated as a possible cause of breast cancer²⁰ while talcum powders have been observed to contain as bursiform and sizeable concentrations of Ni, Cr and Co²⁰. Eye cosmetics such as Kohl and Surma have been identified as a source of Pb exposure to the ocular system in adults and children²²⁻²⁴. Similar studies, however of traditional make-ups used in Nigeria, have reported very high levels of trace metals in locally sourced eye make-up^{25, 26}. Due to the vast number of cosmetics in Nigerian market, many brands have not been investigated but they are widely used in this study area. The objective of the study therefore was to determine the selected heavy metals content of some lipsticks products in Riyadh market.

MATERIALS AND METHODS

Chemicals and reagents

All reagents must be of analytical grade (Nitric acid (69 %), hydrofluoric acid (70 %), hydrochloric acid (70 %) and Hydrogen peroxide 30% v/v, Reductant: For Hg either, 1.1 % w/v stannous chloride in 3%v/v hydrochloric acid or 0.2 % w/v sodium borohydride in 0.05% sodium hydroxide, 50% w/v Magnesium nitrate, Deionized water, resistivity 18.2 Mohm). Standard calibration solutions: Cd, Pb, As and Hg standard stock solutions conc. 1000 g/ ml. Modifier for graphite furnace - atomic absorption spectrometry (GF-AAS), For Pb and Cd: Mix 1:1 of 0.2% w/v Mg (NO₃) $2.6H_2O$ in 0.5% v/v nitric acid and 0.2% w/v NH₄H₂PO₄ in 0.5% v/v nitric acid.

Instruments

Microwave Digestion – System, High Perfrmance from (ETHOS ONE), Atomic Absorption Spectrometer 240FS AA, from Agilent Technologies with (Graphite Furnace) GTA 120) æPSD 120 Programmable Sample Dispenser, and carrier gas was Argon.

Sample Collection

Lipstick products were purchased from different shops at Riyadh city. The lipstick were categorized according to their price : "cheap" (category I < 60 SR), "intermediate" (category II < 130 SR) and "expensive" (category III > 140 SR). These include red lipstick. In all, 21 brand samples were collected for analysis.

Microwave digestion

Lipstick Samples were extracted using a microwave digester²⁷. The digestion procedure was as follows: 0.25 g of lipstick was weighed into a microwave vessel liner. Subsequently, 8 ml of nitric acid (69 %) purchased from CHEM-LAP (Belgium), 1 ml hydrogen peroxide (35%) purchased from Riedelde Hean , 1 ml hydrofluoric acid (70 %) purchased from SIGMA-ALDRICH and 1 ml hydrochloric acid (70 %) purchased from SIGMA-ALDRICH were added. The liners were placed in vessels, closed with a sealed cap, a put into the microwave oven (Ethos One). The samples were digested applying the following microwave program: (20 min, 180 °C, 1500 w). The extracts were filtered to remove the wax and glitters from the lipsticks using filter paper and then diluted with 20 ml de-ionized water. The lipstick samples were allowed to stand for 24 h in the refrigerator.

Preparation of standard stock solutions and working standards

Stock solutions were prepared from which working standards were freshly prepared by serial dilution. The stock solutions of lead, cadmium, arsenic and mercury were obtained already prepared. Five serial standards of each element were prepared for the calibration. The final acid concentration was maintained at about 1% during serial dilution and subsequent dilution of stock solutions to keep the metal in a free ion state appropriate weighing of metals was done prior to dissolving them in acids to make 1000 ppm of stock solutions. Serial standard solutions were prepared in the following ranges in ppm; Pb (10, 20, 30, 40, 50 µg/L), Cd (0.5, 1, 1.5, 2, 2.5 µg/L), As (10, 20, 30, 40, 50, µg/L) and Hg (5, 10, 20, 30, 40 mg/L). The serial standards were aspirated into the instruments. The absorbance was plotted against their concentrations to obtain calibration curves. The correlation coefficients were calculated to and used to express the performance of the instrument.

Sample Analysis

Clear solutions of the digested samples were analyzed for Pb, Cd, As and Hg using air-acetylene flame atomic absorption spectrophotometer (model: AAS 240FS), Agilent Technologies Company, America) by the standard calibration technique. All measurements were run in triplicates for the samples and standard solutions and the results reported as mean \pm standard deviation. The operational conditions during the analysis of heavy metals are listed in Table 1.

Quality control

For each batch of sample analysis, a method blank was carried throughout the entire sample preparation and analytical process [28]. These blanks are useful in determining if the samples are being contaminated. The limit of detection (LOD) and limit of quantification (LOQ) were calculated with three and ten times the standard deviation of the 10 individually prepared method blank solution [29]. Extraction recovery was evaluated by spiking three replicates of blank matrix (organic lip balm) with heavy metals standard using Eq (1):

Recovery
$$\% = \left(\frac{a-b}{c}\right) \times 100$$
 ...(1)

Where a: is the concentration of the sample after spiking, b: is the concentration of the sample before spiking and c is the concentration of standard used for spiking. The recovery and spiking of microwave digested samples concentration are shown in Table 2.

Statistical analysis

The data was analyzed using Package for the Social Science (SPSS) VERSION 17. Descriptive statistical parameters such as mean and standard deviation (SD) were used to describe the heavy metal concentration in the lipstick samples. One Way Analysis of Variance (ANOVA) were used to determine the difference of the heavy metals concentration among different prince categories of lipsticks at a significance level of p<0.05.

RESULTS AND DISCUSSION

The average of extraction recovery for Cd, Pb, Hg and As was 98.86%, 98.76%, 97.5% and 98.25%, respectively. The seven-point calibration curve showed good linearity, where correlation coefficients (R²) ranged from 0.9917 to 0.9986. The concentration of heavy metals in the lipstick samples of different price categories are summarized in Table 3. The average concentration of mercury, cadmium, lead and arsenic in the lipstick in price categories I (cheap price) were 1.52 ppm, 1.64 ppb, 38.69 ppb and 92.47 ppb, respectively. While the average concentration in the lipstick in price categories II (intermediate price) were 0.36 ppm, 1.03 ppb, 9.96 ppb and 133.13 ppb, respectively. The average concentration in the lipstick in price categories III (expensive price) were 0.25 ppm, 0.55 ppb, 1.4 ppb and 102.44 ppb, respectively Table 4.

The results showed the concentration of mercury, cadmium and lead are high in cheap lipstick samples compared with the intermediate and expensive lipstick samples. The highest concentration 1.52 ppm, 1.94 ppb and 40.30 ppb in the sample No. C2 and C6, respectively. As proved that arsenic concentration is high in cheap, intermediate and expensive lipstick samples; 93.4 ppb, 153.98 ppb and 124.62 ppb in the samples No. C1, I6 and E5. The results showed the mercury concentration was high followed by arsenic and cadmium, finally lead.

Previous studies focused on the lead content in cosmetic samples³⁰⁻³². Lead was previously detected in 25 lipstick samples; the concentrations of lead ranged from 0.11 to 4.48 mg kg-1³¹. The concentration of lead in this study was higher than the concentration reported by Gunduz and Akman³¹. Besides, Al-Saleh et al.³⁰ reported that four brands of lipstick exceeded the United States food and drug Administration (US FDA) lead limit as impurities (20 ppm). The US FDA has approved the use Mica (silicate minerals that provide a glittery and metallic shimmery look) with good manufacturing practice with lead content should not exceed 20 ppm in externally used drugs, dentifrices, and cosmetics³³. On the other hand, based on the guidelines of control of cosmetic products in Saudi Arabia which is prepared in accordance with the ASEAN cosmetic directive, lead, cadmium, mercury and arsenic are included in the list of substances which must not form part of the composition of cosmetic products as described in Annex II³⁴. These heavy metals should not be added to cosmetics during the manufacturing process as an ingredients formula. However, lead, cadmium, mercury and arsenic were found in all of the lipsticks tested in this study. The existence of heavy metals was believed to be due to the natural occurrences of these heavy metals in the color additives as well as contamination in the lipstick manufacturing process. During the manufacturing process, the heavy metals sources might come from solder, leaded paints on manufacturing equipment, and also from lead-contaminated dust from the manufacturing surroundings. Saudi Food and Drug Authority (SFDA) has shown the limits of heavy metals in some cosmetics as shown in the following table35:

However, the lead content in all lipstick samples in this study was below the US FDA limit (20 ppm), SFDA limit (1 ppm). The safe permissible limit for lead and cadmium in cosmetics as suggested in health Canada are 10 ppm and 3 ppm, respectively, while the limit for cadmium has not been determined

Elements	Cd	Pb	Hg	As
Wavelength (nm)	228.8	283.3	253.7	197.2
Slit Width (nm)	0.5	0.5	0.5	0.5
Lamp Current (mA)	4	10	4	10
Sensitivity (ppm) At 0.2 Abs	1.00 ppb	27 ppb	70 ppm	50 ppb
Detection Limit	0.063 ppb	0.951 ppb	0.036 ppm	0.474 ppb
Optimum Working Range Instrument	0.00-5.00 ppb AAS	0.0-50.00 ppb AAS	0.00-30.00 ppm GT-AAS	0.00-150 ppb AAS

Table 1: The Standard Operating Parameters of the Elements Analyzed

Table 2: Recovery and spiking of microwave digested samples concentration "ppb" for Pb, Cd and As , "ppm" for Hg

Heavy Metals	Un-spiked (Mean ± SE)	Standard Added to Sample	Spiked Samples (Mean ± SE)	Correlation coefficient (R ²)	Recovery%
Pb	2.645±0.01	8	10.555±0.02	0.9986	98.76
Cd	0.559 ± 0.00	10	10.445±0.01	0.9973	98.86
As	0.222±0.01	2	2.187±0.01	0.9982	98.25
Hg	0.203±0.01	1	1.178±0.02	0.9917	97.5

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Price Code Country of Samples Category production Brand		Samples Brand	Pd (ppb) Mean± SD	Cd (ppb) Mean± SD	Hg (ppm) Mean± SD	As (ppb) Mean± SD	
	C1	China	Adeem	4.5	0.49	1.04	93.4
	C2	China	TOP LADY	1.94	1.94	1.52	0.93
	C3	China	SaSh	3.16	0.3	0.98	3.52
I	C4	China	Baolishi	38.69	0.34	0.61	27.16
	C5	China	Karite	5.23	0.73	0.12	27.17
	C6	China	OILY	40.3	0.46	N.D	53.35
	C7	China	IN DREEM	5.15	0.4	N.D	13.58
	11	Italian	CLARINS	4.7	0.14	0.16	41.12
	12	Italian	Wojooh	5.31	0.24	0.37	66.84
	13	American	CLINIQUE	3.04	0.31	N.D	50.55
II	14	Italian	MAKE UP FOR EVER	5.28	0.28	N.D	35.06
	15	French	BOURJOIS	2.94	0.27	0.3	41.6
	16	ireland	Max Factor	12.9	1.17	0.63	153.98
	17	French	benefit	3.37	0.14	0.37	20.85
	E1	French	CHANEL	2.99	0.2	N.D	49.38
	E2	French	Dior	2.63	0.68	0.25	22.18
	E3	French	YVES SAINT LAUREN	T 2.58	0.27	N.D	40.46
	E4	American	ESTEE LAUDER	2.57	0.21	N.D	56.52
	E5	French	LANCOME	2.95	0.36	N.D	124.62
	E6	French	GIVENCHY	3.8	0.29	N.D	66.73
	E7	French	GUERLAIN	2.4	0.13	N.D	30.8

Table 3: The concentration of heavy metals in the lipstick samples

 Table 4: Mean, standard deviation, maximum and minimum values of the concentration of metals in lipsticks

Heav meta	y Is	Ν	Range	Minimum	Maximum	Mean deviation	Standard	Variance
Hg	Cheap	7	1.52	N.D	1.52	0.61	0.5960	0.3560
	Intermediate	7	0.36	N.D	0.36	0.26	0.2270	0.0510
	Expensive	7	0.25	N.D	0.25	0.039	0.0930	0.0090
Cd	Cheap	7	1.64	0.30	1.94	0.67	0.5790	0.3350
	Intermediate	7	1.03	0.14	1.17	0.36	0.3620	0.1310
	Expensive	7	0.55	0.13	0.68	0.31	0.1800	0.0330
Pb	Cheap	7	38.69	N.D	38.69	8.38	13.4970	182.1750
	Intermediate	7	9.96	2.94	12.90	5.36	3.4760	12.0820
	Expensive	7	1.40	2.40	3.80	2.85	0.4720	0.2220
As	Cheap	7	92.47	0.93	93.40	31.30	32.6210	1064.145
	Intermediate	7	133.13	20.85	153.98	58.57	44.3540	1967.297
	Expensive	7	102.44	22.18	124.62	55.81	33.8820	1148.002

[36]. The cadmium content in all lipstick samples in this study was below the US FDA limit (5 ppm) and SFDA limit (0.1 ppm). The arsenic content in lipstick samples was below the US FDA limit(3 ppm) and SFDA limit (0.5 ppm) with the exception of two samples is higher than the limit (sample No. E5 and I6). Finally, the mercury content in some lipstick samples in this study was higher than SFDA limit (0.2 ppm) but within the limit according to US FDA (1 ppm) with the exception of one sample is higher than the limit (sample No. C2). The results of Statistical analysis are shown in (Table 5-8). The ANOVA was used to determine the difference of lead, cadmium, arsenic and mercury content between price categories. The results of ANOVA showed that there was no significant difference of mercury content among the lipsticks in price categories I, II and III, where the p value was more than 0.05 [p=0.32, F(2.18)=4.19]. The results in the ANOVA test on cadmium showed that the p value was more than 0.05, indicating there was no significant difference of cadmium content between the lipsticks in price categories I, II and III, p>0.05

	Lead ppm	Mercury ppm	Arsenic ppm	Cadmium ppm	
For eyes products	Nill	Nill	Nill	Nill	
Oral hygiene products	1	0.2	0.5	0.1	
Other cosmetics products	10	3	3	3	

Table 5: One Way Analysis of Variance (ANOVA) of Means for mercury concentration in different price of lipstick samples

		Sum of Squares	degrees of freedom (df)	Mean Square	Ratio of the mean squares(F)	Sig.
lipstick	Between Groups	1.161	2	0.581	4.190	0.32
	Within Groups	2.494	18	0.193		
	Total	3.656	20			

Table 6: One Way Analysis of Variance (ANOVA) of Means for cadmium concentration in different price of lipstick samples

		Sum of Squares	degrees of freedom (df)	Mean Square	Ratio of the mean squares(F)	Sig.
lipstick	Between Groups Within Groups Total	0.522 2.992 3.514	2 18 20	0.261 0.166	1.572	0.235

Table 7: One Way Analysis of Variance (ANOVA) of Means for lead concentration in different price of lipstick samples

		Sum of Squares	degrees of freedom (df)	Mean Square	Ratio of the mean squares(F)	Sig.
lipstick	Between Groups	107.548	2	53.774	0.830	0.452
	Total	1274.429	20	04.027		

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	arsenic concentration in different price of lipstick samples							
		Sum of Squares	degrees of freedom (df)	Mean Square	Ratio of the mean squares(F)	Sig.		
lipstick	Between Groups Within Groups Total	3154.836 25076.667 28231.503	2 18 20	1577.418 1393.148	1.132	0.344		

Table 8: One Way Analysis of Variance (ANOVA) of Means for arsenic concentration in different price of lipstick samples

[p=0.235 , F(2.18)= 1.572]. The results of ANOVA showed that there was no significant difference of lead content among the lipsticks in price categories I, II and III, where the p value was more than 0.05 [p=0.452, F(2.18)=0.83]. On the other hand , the results in the ANOVA test on arsenic showed that the p value was more than 0.05, indicating there was no significant difference of arsenic content between the lipsticks in price categories I, II and III, p>0.05 [p=0.59 , F(2.18)= 3.33]. The results of this study was comparable to the study by Piccinini²⁷.

CONCLUSION

In the present study, arsenic, cadmium, lead, and mercury were determined in various brand of lipsticks in price categories. The results showed the mercury concentration was high followed by arsenic and cadmium, finally lead in the select lipsticks. It is feared how-ever that the continuous use of lipstick products contaminated with such heavy metals may however cause slow release of these metals into the human body and cause harmful effects to the consumers over time. Extensive use of such products should be avoided until the situation is adequately addressed.

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