Journal of Public Health Policy (2022) 43:54–64
https://doi.org/10.1057/s41271-021-00324-8

ORIGINAL ARTICLE

Analysis of lead, cadmium, and arsenic in colored cosmetics marketed in Pakistan

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Accepted: 29 November 2021 / Published online: 17 January 2022

Abstract

Heavy metal contamination of the cosmetic products poses potential harm for consumers' health. We aimed to determine the concentration of Arsenic (As), Lead (Pb), and Cadmium (Cd) in locally available colored cosmetics such as lipsticks, foundation cream, facial powder, and eye shadows. We determined concentration of As, Pb, and Cd in 40 samples of colored cosmetics by Inductively Coupled Plasma Optical Emission Spectroscopy (ICP-OES) and determined significant differences in As and Cd concentrations across the colored cosmetics. We detected a high concentration of Pb and As in lipstick and eye shadow samples. Concentration of Pb and Cd in the samples of foundation creams was at safe level. Most of the cosmetic samples contained heavy metals above safe levels posing a threat to the health of female consumers who use them for long periods. Regulatory bodies in Pakistan must adopt and enforce international standards for colored cosmetics.

Keywords Heavy metals · Colored cosmetics · ICP-OES · Lead · Arsenic · Cadmium

Introduction

Women use cosmetics products, particularly coloring cosmetics such as lipsticks, eye shadows, talc powder, and facial tone foundations, to accentuate appearance, hide, or conceal the flaws and freckles. Cosmetics may impart a smoothening and shining effect on the skin, making it more attractive. Chemical ingredients are present in cosmetics owing to their special properties and functions. These include coloring agents, emollients, sun screening agents, surfactants, antioxidants, preservatives, and perfuming agents [1]. Coloring cosmetics contain

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inorganic pigments or dyes, talc, kaolin, mica, calcium carbonate, magnesium carbonate, zinc oxide, and titanium oxide. These raw materials come from mineral origins and are vulnerable to inclusion of heavy metals that may contaminate the final formulation [2, 3]. Heavy metals such as Lead (Pb), Arsenic (As), Cadmium (Cd), Cobalt (Co), Nickel (Ni), and Copper (Cu) may enter cosmetic products from low-quality raw materials or metallic equipment used during the manufacturing process, or both. Continuous and long-term application of such contaminated cosmetics to sensitive facial regions such as eyes and lips increases the chances of exposure to heavy metals.

Researchers have contributed to wide discussion of the safety and toxicity of heavy metals [4–7]. Some have reported that absorption and retention of heavy metals caused severe skin disorders [8, 9]. Heavy metal toxicity may be acute or chronic, depending upon the route, dose, and duration of exposure. When bound to the biological macromolecules, they may trigger oxidation by free radical formation leading to excessive damage and deterioration at the cellular level [10]. Human bodies may absorb and retain metals such as Ni, Co, and Cr in the stratum corneum that may cause allergic contact dermatitis [2]. Metals such as Pb, Hg, Cd, and As can be absorbed into the bloodstream and accumulate in body organs. Lead may inhibit steps in the biosynthesis of hemoglobin and cause neurological and renal problems over a prolonged exposure [11]. Researchers also report that lead causes sterility in males and miscarriages or spontaneous abortions in females [12]. Infants and children are prone to Pb toxicity even at low concentration that ultimately causes a decrease in their IQ level and learning abilities [13]. Dermal exposure to a high concentration of Cd results in skin dermatitis and its absorption and retention in the body causes serious renal problems and metabolic disorders [14]. *Ullmann's encyclopedia of industrial chemistry* documents concerns about chronic exposures to As [15]. Arsenic causes carcinogenic effects at high concentration and adversely effects skin and keratin body structures like hair and nails [16]. Simultaneous exposure to more than one heavy metal such as Pb and Cd in a cosmetic product causes synergistic effects on toxicity.

Determination of heavy metal concentrations in cosmetics is very important. Commercially available coloring beauty products must be monitored to assess if the levels of heavy metals are safe or hazardous. A critical review sets out results from investigation of heavy metals contents of cosmetic products manufactured and marketed in various countries [17]. Researchers have used analytical techniques, such as Atomic Absorption Spectroscopy and ICP-MS and ICP-OES, to quantify the heavy metal contents of cosmetic products [18, 19].

High-resource countries have guidelines to prohibit use of certain heavy metals in cosmetics and define the permissible levels of heavy metals allowed in cosmetic products. European countries have banned used of metals such as Pb, Cd, As, Ni, and Hg in cosmetics. They allow, with restrictions, other metals such as Zn, Ag, and Sr. According to European regulations No. 1223/2009, the presence of trace amounts of heavy metal contaminants in cosmetics is technically unavoidable, and unfortunately, safe limits are not described for certain heavy metals. The Food and Drug Administration (FDA) of United States (US) amended a rule about color additives in 2019 to prohibit use of lead acetate in hair coloring cosmetics [20].

Pakistan is a lower resource country with a relatively small domestic cosmetics industry. Instead, Pakistan imports most of the products sold in country from India, China, Gulf States, Europe, and the US. The local industry lacks essential awareness about the safety of ingredients and chemicals used. Various news outlets have reported substandard cosmetics in the market [21]. Pakistan needs to establish safety standards for colored cosmetic products. Determination of heavy metal concentration in cosmetics and establishing safety standards is crucial, as is monitoring of locally available coloring beauty products. To the best of our knowledge, no previous research reports analysis of As, Cd, and Pb by ICP-OES in colored cosmetics available in the Pakistani market. We designed the current work to examine the heavy metal contents of coloring cosmetics in the local market. We compared the results against international standards. This work forms part of our efforts to build an indigenous dataset and to boost consumer awareness. The results may also stimulate discussion among regulatory bodies to enforce strict quality controls for manufacturers and marketers.

Materials and methods

Samples

We purchased all the cosmetic samples from the local market in Faisalabad. We selected the brands and color shades frequently used by female consumers. Details of samples are as follows:

(a) Two imported and two locally manufactured lipstick brands with three color shades of each brand making a total of 12 lipstick samples.
(b) Two imported brands of eye shadows with a total of 10 eye shadows samples.
(c) Ten and eight imported brands samples of foundation cream and facial compact powers, respectively.

We stored all the samples at room temperature in a dry place for analysis.

Reagents and solutions

We purchased all the chemicals from Sigma Aldrich. We used analytical grade Nitric acid (HNO_3) 65%, Perchloric acid ($HClO_4$) 70%, and Hydrogen peroxide (H_2O_2) 30% for acid digestion of all the cosmetic samples. We soaked all pieces of glassware in 10% HNO_3 overnight, then washed them with distilled water, and dried them. We used only double distilled water throughout the analysis.

Sample digestion

Each sample was digested by microwave-assisted acid digestion using nitric acid (HNO_3) and perchloric acid ($HClO_4$) according to the method reported in the literature [22]. The digestion proceeded as follows. The research team took a sample (0.2 g) in a Teflon vessel and added 7.0 mL of (HNO_3/$HClO_4$ 5:2); then heated it using microwave power 300 W for 5 min and then 350 W for 2 min in a conventional microwave oven; then added Hydrogen peroxide (1.0 mL); and heated the sample again for 2 min.

After completion of digestion, we analyzed the samples for presence of Pb, Cd, and As. For each type of colored cosmetic, we used a blank sample (consisting of a sample matrix with all the chemical reagents, except the analyte) throughout the sample preparation. The instrument was calibrated with a standard solution of each metal having a concentration range of 0.005–5.00 μg/g. There was a linear correlation between the concentration of standards and their absorbance. We determined the extraction recovery (%) of all the metals by spiking the lipstick matrix with heavy metals standard (known concentration of metals) and measuring the concentration of each metal after complete digestion. We treated these samples with the same analytical procedure under the similar conditions of digestion as the test samples of cosmetics. Limit of quantification (LOQ), Linear correlation coefficients (R^2), and percent recovery for Pb, As, and Cd appear in Table 1.

Inductively Coupled Plasma Optical Emission Spectroscopy (ICP-OES) analysis

The amount of Pb, As, and Cd were determined by ICP-OES in the Central High-Tech Laboratory, Government College University Faisalabad. The instrument specifications are Prodigy 7, Teledyne Leemans Labs. The RF power was 1.0 KW, Auxiliary gas flow rate 0.3 LPM, Nebulizer gas flow rate 1.0 LPM, and coolant flow at 14 LPM.

Statistical analysis

We analyzed all the samples in triplicate. We calculated mean (m), standard deviation (SD), and standard error (SE) for the metals in each sample using SPSS statistics software. We compared the brands using multivariate analysis (MANOVA) for each product.

Table 1 Percent recovery, LOQ, and R^2 for Pb, As, and Cd

Heavy metal	Percent recovery (%)	Limit of quantification (LOQ) ppb	Correlation coefficient (R^2)
Pb	89	23	0.965
As	92	18	0.991
Cd	95	21	0.984

Results and discussion

Although most of the heavy metals cannot be used as active constituents in the production of cosmetics, detectable amounts in cosmetic products are unavoidable. We found contamination with Pb, Cd, and As in all forty samples, with higher concentrations of Pb and As in some of the colored cosmetic samples. Lead was the most frequently detected metal, present in 39 samples (97%). We found cadmium in 38 out of 40 samples (95%). The concentration of Pb was highest of all metals analyzed with a range of 0.13–74.61 µg/g. We found arsenic in 62% of the samples with a maximum concentration of 18.0 µg/g in facial compact powder.

The concentrations of Pb, As, and Cd in 12 samples of lipsticks appear in Table 2. The amount of Pb in the lipstick samples varied from 1.10 to 74.61 µg/g. We detected an alarmingly high level of Pb 74.61 ± 2.10 µg/g in the brown shade of lipstick (brand 2) and also the highest concentration among lipstick brands. Previous studies have also reported high levels of Pb in lipstick products marketed in China, Saudi Arabia, Malasia, and other counties [23–26]. In 5 out of 12 samples analyzed, we found As in a range of 1.86–15.13 µg/g. The brown color shade demonstrated the highest level of As. The concentration of Cd ranged from 0.79 to 12.85 µg/g.

Multivariate analysis of results showed that at 0.05 significance level, each of the four brands of the lipsticks were significantly different from each other when considered jointly for the presence of Pb, Cd, and As. We conducted a separate ANOVA for each metal that showed that no significant difference between lipstick brands for the concentration of Pb. For As and Cd, however, we found a significant difference across all four brands of lipsticks. Brand 2 contained a significantly higher concentration of Pb compared to the other three.

We found contamination with Pb, As, and Cd in varying concentrations in all 10 samples of eye shadow. We summarize the results in Table 3. The concentration range of Pb in all the samples of eye shadow is 1.91–57.01 µg/g. Three

Table 2 Quantity of Pb, Cd, and As in the lipstick samples (ND = Not detected)

Brand	Lipsticks	Color Shade	Cd (µg/g) (mean ± SD)	Pb (µg/g) (mean ± SD)	As (µg/g) (mean ± SD)
B1	L1	Red	12.85 ± 0.50	1.10 ± 0.10	3.50 ± 0.10
	L2	Pink	10.14 ± 0.50	5.21 ± 0.10	ND
	L3	Dark Brown	11.60 ± 0.10	1.33 ± 0.08	4.91 ± 0.20
B2	L4	Pink	0.79 ± 0.08	9.77 ± 0.03	10.83 ± 0.04
	L5	Red	2.39 ± 0.02	61.35 ± 5.40	1.86 ± 0.05
	L6	Brown	3.89 ± 0.02	74.61 ± 2.10	15.1 ± 0.30
B3	L7	Red	11.82 ± 0.3	10.27 ± 0.02	ND
	L8	Pink	6.81 ± 0.40	33.85 ± 0.90	ND
	L9	Brown	11.13 ± 1.80	16.03 ± 3.50	ND
B4	L10	Pink	5.60 ± 0.05	2.70 ± 0.10	ND
	L11	Red	1.79 ± 0.06	6.54 ± 0.30	ND
	L12	Brown	1.21 ± 0.30	16.61 ± 01.00	ND

Table 3 Quantity of Pb, Cd, and As in the eye shadow samples

Brand	Color shades	Sample Name	Cd (µg/g) (mean ± SD)	Pb (µg/g) (mean ± SD)	As (µg/g) (mean ± SD)
1	Dark Brown	ESI	18.9 ± 0.20	57.01 ± 0.20	0.35 ± 0.09
	Dark Blue	ES2	16.82 ± 0.50	16.50 ± 0.80	8.70 ± 2.30
	Rose Red	ES3	3.30 ± 0.30	13.41 ± 0.4	3.50 ± 0.40
	Dark Gray	ES4	0.95 ± 0.11	41.73 ± 0.90	5.72 ± 0.30
	Green	ES5	2.70 ± 0.10	26.60 ± 0.35	0.41 ± 0.02
2	Dark Brown	ES6	13.47 ± 0.28	18.23 ± 0.07	1.02 ± 0.10
	Dark Gray	ES7	7.70 ± 0.34	10.52 ± 0.3	12.14 ± 0.63
	Purple	ES8	6.55 ± 0.50	4.01 ± 0.20	0.65 ± 0.10
	Violet	ES9	0.90 ± 0.09	1.91 ± 0.16	0.04 ± 0.03
	Green	ES10	5.23 ± 0.11	19.85 ± 0.52	0.10 ± 0.03

samples contained Pb levels above safe limits set by the United States Food and Drug Administration (FDA). Dark brown (ES1) and gray color (ES4) shades of brand 1 showed higher Pb levels. We detected the concentration of Cd in the range of 0.90–18.91 µg/g. The dark brown color shade (ES1) contained high levels of Cd (18.91 µg/g) and of Pb (57.01 µg/g), making it the most contaminated color of eye shadow. All samples of eye shadows showed the presence of As; only 4 of 10 samples contained this metal above 3 µg/g (level by FDA). The concentration of As varied from 0.04 to 12.14 µg/g in the eye shadow samples. Multivariate analysis showed differences among eye color shades of two brands. For the concentration of Pb, we found a significant difference between the two brands of eye shadows: all the samples of brand one contained high concentrations of Pb compared to the samples in brand 2. We did not find significant differences in concentration of As and Cd in the two eye shadow brands.

Foundation cream is a kind of face paint to enhance complexion color. Foundation creams in the form of water in oil type emulsion contain various kinds of filler and inorganic pigments [27]. Table 4 shows results of the heavy metals (Pb, Cd, and As) in 10 samples of locally available but imported foundation creams in Pakistan. Lead and Cd appeared in all samples with the concentration ranging from 0.20–15.4 µg/g to 0.08–11.6 µg/g, respectively. Only four samples showed the presence of As (0.93–11.91 µg/g). We detected a high level of As (11.91 µg/g, 7.83 µg/g) in the two samples (FN8, FN9). One (FN 8) that showed a higher concentration of As also contained Cd (11.63 µg/g) above the threshold level. When we compared all the ten cream samples, we found significant differences in the concentration of Pb and Cd.

For compact facial powder, 6 of the 8 samples contained Pb, Cd, and As as shown in Table 5. The amount of Pb in all these samples was 0.93–29.48 µg/g, with only one containing a very high quantity of Pb, i.e., 29.48 µg/g. The concentration for Cd ranged from 0.05 to 7.63 µg/g; for As, we found considerably higher levels in approximately 90% of the samples, in the range of 0.91–26.43 µg/g.

Table 4 Quantity of Pb, Cd, and As in the foundation creams

Sample name	Cd (µg/g) (mean ± SD)	Pb (µg/g) (mean ± SD)	As (µg/g) (mean ± SD)
FN1	0.08 ± 0.01	1.43 ± 0.10	ND
FN2	0.20 ± 0.02	0.20 ± 0.07	ND
FN3	6.20 ± 0.04	5.93 ± 0.05	ND
FN4	2.90 ± 0.12	10.61 ± 0.15	ND
FN5	1.90 ± 0.03	12.87 ± 0.04	0.93 ± 0.03
FN6	0.50 ± 0.04	15.43 ± 0.12	3.48 ± 0.13
FN7	3.60 ± 0.01	2.10 ± 0.04	ND
FN8	11.63 ± 0.06	1.91 ± 0.06	7.83 ± 0.03
FN9	1.12 ± 0.01	0.33 ± 0.01	11.91 ± 0.12
FN10	0.23 ± 0.01	0.71 ± 0.30	ND

Previously published data about the heavy metal contents of facial compact powders support our results [28, 29].

Various regulatory bodies have specified usage or maximum threshold limits for heavy metals in formulation and application [30]. The US FDA, a Canadian agency, and other regulatory bodies have specified maximum permissible limits for metals in color cosmetics and other beautify products. Regulators define the maximum permissible limit according to nature of the product, application dosage, and time the product would remain on the skin, and the site of application. Our study found that most of the colored cosmetics contained levels of metals significantly higher than the limits described in Table 6. This is a matter of great concern and requires urgent attention.

The great variation in concentration or quantity of heavy metals in colored cosmetic samples may reflect differences in the variety and quantity of mineral ingredients used in their formulation and in manufacturing practices. The maximum concentration of Pb allowed by the FDA in cosmetic products applied externally is 20 µg/g. Our results show some samples of lipsticks (L5, L6) and eye shadows (ES1, ES4, and ES5) to contain a concentration of Pb much higher than the limits defined by the FDA. Continuous application of such cosmetics could aggravate toxic effects

Table 5 Quantity of Pb, Cd, and As in the compact powders (ND = Not detected)

Powders	Cd (µg/g) (mean ± SD)	Pb (µg/g) (mean ± SD)	As (µg/g) (mean ± SD)
P1	2.38 ± 0.01	8.80 ± 0.14	0.91 ± 0.05
P2	4.12 ± 0.03	1.30 ± 0.03	7.67 ± 0.07
P3	7.63 ± 0.25	0.81 ± 0.03	26.43 ± 1.10
P4	3.76 ± 0.38	0.95 ± 0.26	17.24 ± 0.14
P5	ND	0.93 ± 0.03	13.47 ± 0.16
P6	0.47 ± 0.04	15.92 ± 0.21	12.15 ± 0.06
P7	ND	29.48 ± 0.8	18.43 ± 2.11
P8	0.05 ± 0.03	ND	5.81 ± 0.22

Table 6 International standards for metals (Pb, Cd, and As) in cosmetics

Metals	European standards Conc. (μg/g)	FDA Conc. (μg/g)	Canadian standards Conc. (μg/g)
Arsenic	03	03	05
Lead	10	20	10
Cadmium	03	–	05

of Pb. Lead toxicity may cause serious effects on body systems, such as renal dysfunctions, damage to the central nervous and reproductive systems. Pregnant women and their fetuses could be seriously adversely affected by the toxic effects of Pb accumulation in the body [16].

We found concentration of Cd in some of the lipsticks and most shades of eye shadow samples in our study above the threshold limits defined by Canadian and European regulatory bodies. Cadmium produces carcinogenic effects from continuous exposure. Accumulation of high levels of Cd in the body causes oxidative stress that ultimately affects kidneys and liver. At the cellular level, Cd causes mitochondrial dysfunction, resulting in less energy production and increased release of reactive oxygen species (ROS) [31]. Some components of lipstick formulation, such as waxes and oils, can interact and penetrate the lipid layer of the skin. Since users apply lipsticks directly and retain them on their lips for a long time, the product can be easily ingested by licking, eating, or drinking. Lipstick formulations contain significant levels of organic and inorganic pigments. We believe the source of contamination for these metals may be these pigments. Sub-standard pigments in developing countries are a serious cause for concern. Metal ions enter the products during planned manufacturing processes or due to poor manufacturing conditions.

Compact powders contain inorganic pigments and talc, and the possibility of heavy metal contamination comes from the mineral origin of the raw materials. Comparing heavy metals among all brands of compact powders showed As to be present at high concentration in all the samples. Arsenic appeared in a few samples of lipstick and eye shadows. Research reports have shown that As contamination of the products mostly occurs through use of low-quality mineral ingredients because As reaches the natural resources by leaching into aquifers or through mining processes. Absorption of As takes place through ingestion and rarely through the skin or respiratory system. Once accumulated in the body, arsenic toxicity results in inactivation of enzymes involved in the cell's energy production pathway and DNA synthesis or repair. Arsenic is reported to have carcinogenic effects [16]. Research has shown certain heavy metals, such as Pb, Cd, Zn, and Hg, when studied together for joint toxicity, cause enhanced generation of free radical oxygen species, apoptosis, and arresting of the cell cycle, ultimately causing severe cell damage [32]. Another study found that lead and cadmium together can induce high blood pressure and enhanced cardiovascular disease mortality rates as a consequence of the synergistic effect of Pb and Cd toxicity [33].

The cosmetic industry in Pakistan is growing. More research is needed to help it develop good quality cosmetic products that meet international standards. China is the biggest exporter of cosmetic products to Pakistan, and most of the low-priced cosmetic products available in the Pakistani market are Chinese imports. Lack of public awareness means that consumers do not read product labels that list cosmetic product ingredients. Therefore, we recommend that Pakistan adopt strict quality control regulations for import of such products to Pakistan.

Conclusions

This research presents important information about the potential contamination with heavy metals of colored cosmetics available in Pakistan. All the colored cosmetic samples in our study contained detectable concentrations of heavy metals (Pb, Cd, and As) and some samples contained levels of heavy metals far above the safe limits set by FDA. High levels of Pb and As contaminated were detected in samples of lipsticks and eye shadows. Most imported brands of cosmetics, we tested could not meet international standards due to high levels of Pb and As. Repeated applications of such products on skin could be harmful and cause severe damage at the local and systematic levels. To ensure the public safety and well-being of consumers, regulatory bodies in Pakistan must adopt and enforce international standards.

Acknowledgements We are thankful to Dr. Abullah Ijaz, Director High-tech laboratory, Government College University Faisalabad, for his support for heavy metal analysis by ICP-OES.

Declarations

Conflict of interest On behalf of all authors, the corresponding author states that there is no conflict of interest.

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