Determination of Heavy Metal Impurities in Facial Makeup Cosmetic Products in Sudanese Markets, using Inductively Coupled Plasma – Mass Spectrometry (ICP –MS)

Abdalla M. Abdalla Mustafa * • Mohamed El Mukhtar A. Aziz

Department of Chemistry, University of Science and Technology, Sudan
halawani2003@hotmail.com

Abstract: Recently facial makeup cosmetic products have become very popular among Sudanese women in spite of the fact that they might contain toxic materials including Pb, Hg, Cd, As, Ni and Zn, and their use as ingredients in cosmetics is prohibited as they cause skin problems. Owing to the absence of control standards of these products sold in Sudanese markets, the present study aimed at developing a method for the determination of these metals by inductively coupled plasma – mass spectrometry (ICP-MS). Eighty-six samples of different facial makeup cosmetic products were collected randomly from the local Sudanese markets. A microwave- assisted digestion with a mixture of concentrated nitric acid and hydrochloric acid was applied for sample preparation. The concentration of each metal was determined in triplicate by ICP-MS. For eye shadow, in a wide range of concentrations, all analyzed elements were detected at high levels in the imported samples, but Pb, Hg, and Cd were detected at low levels in the locally prepared not only eye shadow but also lipstick samples. Although Pb was present at low concentrations in all samples, locally prepared whitening creams, lipstick, and eye shadow showed highest concentration levels of 33.63 µg /g, 42.42 µg /g, and 52.02 µg /g, respectively. The highest concentration levels for Hg in locally prepared whitening creams and eye shadow samples were found to be 42.09 µg /g, and 21.30 µg /g, respectively. The highest concentration levels for Cd, however, were found to be 5.21 µg /g, 22.53µg /g, and 7.06µg /g, in imported lipstick, eye shadow, and local whitening creams, respectively. High concentration levels of Zn were not obtained in all analyzed samples except in the imported eye shadow, (72.46µg /g). The determination of heavy metals in cosmetic products sold in Sudanese markets increase the needs to set laws to regulate their preparation locally and to set standards to control their quality and safety.

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1. Introduction:

Cosmetics can be defined as many preparations to be applied to the human body for the purpose of cleansing, beautifying, promoting, making attractive or altering the appearance without affecting the body's structure and function. They can also be used to repair or hide skin imperfections, to cleanse, adorn, protect and treat the human body (Al-Dayel et al., 2011). In Sudanese markets, there are plenty of cosmetic products which are prepared locally without any control. The most alarming issue in Sudan is the large number of people practicing skin-lightening techniques. Similar practices are also employed by black ladies in all over the world (Adebajo, 2002).

The challenging problem of the cosmetic industry is to design safe products that help dark- skin woman looks "Lighter". Facial attractiveness, however, is associated with positive evaluation by others, and many Sudanese ladies believe that with the lighter skin they are more competent. Interestingly, the level of education, social class, marital and employment status seem to have less influence on the skin lightening practice. The toxicity of metals and its curie are well documented (Chukwuma, 1997). Since the issue of heavy metals, as deliberate cosmetic ingredients have been addressed, attention is focused on the presence of these substances as impurities, (Health Canada, 2011).

Heavy metals are not intentionally added to cosmetics as constituent ingredients; they are simply trace impurities in the product and are thus not required to be listed on the labels (Reed, 2007). Minute traces may be carried into the product during preparation. The metals of primary toxically concern in cosmetics are: Pb, Hg, Cd, As, Ni and Zn. Dermal exposure is expected to be the most significant route for cosmetic products since the majority of cosmetics are applied on the skin.

Dermal absorption of heavy metals is fairly minimal, with the absorption of individual elements influenced by a number of factors including physical and chemical properties of the mixture, (Dwivedi, (1996). The present study was carried out to develop a method for the determination of heavy metal impurities in the facial makeup cosmetic products in the local market by inductively coupled plasma – mass spectrometry (ICP-MS), (Lee, et al., 2008).

2. Materials and Methods:

2.1. Sample Collection:

Eighty-six different samples, commonly used as facial makeup, were collected from the local markets in Khartoum, Khartoum North, and Omdurman cites and were classified into four different groups marked with different letters and numbers.

Lipstick 25 samples: 20 imported (L1 – L20) and 5-local (LL21 – LL25).

Face Powder 25 local samples: (P26 – P50).

Creams, 30 samples: 15 local skin – lightening creams (LC 51 – LC 59), 15 imported skin – lightening cream (C 66 – C 80) and 6 - imported (F&L 60 – F&L 65).

Eye Shadow: 6 samples (ES91 – ES 96).

2.2. Sample Digestion:

Strict care was taken to ensure complete decomposition of the sample and to avoid contamination. Ultra-pure acids and distilled deionized water were used, (Volpe, 2012). All plastic and glassware were washed many times with tap water, soaked in 5% HNO3, solution for a minimum of 24 hours and then rinsed with deionized water. Solid samples were dried in an oven at 105^{o}C to constant weight and then stored in desiccators. About 3.5g of dried sample was weighed accurately in a porcelain crucible and dry-ashed in a muffle furnace by a stepwise increase of temperature up to 550 ^{o}C, for one hour.

The ashed samples were digested with a few cubic centimeters of 1M nitric acid, evaporated nearly to dryness on a hot plate, in fuming hood, cooled, filtered through Whatman No 42 , and were diluted up to the 100 cm^{3} mark in a volumetric flask, Samples such as cream and lotion, which could not be conveniently processed by dry ashing, were wet digested, with a 4:1mixture of nitric acid (65%) and perchloric acid (70-72%) on a hot plate in fuming hood, nearly to dryness by slowly increasing the temperature, for 2-3 hours because oily compounds are exothermic and burn with flame.

In case that brown or black color appeared then again the same procedure was repeated by adding the mixture of concentrated acids by slow and continuous heating until the evolution of white fumes (marking the end of digestion process) and then, nearly to dryness, alternatively, sample solutions acidified with concentrated HNO3 and HCl, were digested in a programmed microwave. The solutions were allowed to cool and filtered into a 100 Cm^{3} volumetric flask by Whatman No 42 and diluted up to the volumetric mark, (Al-Ashban, 2004).

Like the sample solutions, blank solutions were prepared similarly including the addition of all reagents but excluding the presence of the samples.

3. Heavy metal analysis:

Cosmetic sample solutions were analyzed for the determination of the concentration of Pb, Hg, Cd, As, Ni and Zn. Using Agilent 7700X ICP – MS instrument, (Lee, et al., 2008).

4. Results and Discussion:

Table 1 shows the concentration of heavy metals in, twenty samples of imported lipstick, indicating Pb, Cd and As concentration ranged from 4.18 µg/g to 42.42 µg/g, 0.00 µg/g to 12.00 µg/g and from 0.00 µg/g to 10.21 µg/g, respectively. However, the results of determination of the concentration of heavy metals in, five samples of local lipstick were shown in Table 2.

The concentration of Pb, Hg, Ni and Zn metals ranged from 11.45 µg/g to 29.0 µg/g, 2.35 µg/g to 9.24 µg/g, 0.16 µg/g to 9.41 µg/g and 1.00 µg/g to 10.23 µg/g respectively. Table 3 shows that the concentration of, Pb and Zn metals in twenty-five samples of face - powder ranged from 0.04 µg/g to 20.02 µg/g and from 2.47 µg/g to 9.52 µg/g, respectively.

Table 4 shows the concentration of heavy metal impurities of six fair & lovely samples (F&L), and nine samples of local skin-lightening cream (LC). The concentration of Pb in F&L and LC ranged from 2.71 µg/g to 7.27 µg/g and from as much as 14.49 µg/g to as much as 33.36 µg/g, respectively. The concentration of Zn ranged from 1.19 µg/g to 4.91 µg/g and from as much as 2.41 µg/g to as much as 11.04 µg/g, respectively.

Table 5 shows the concentration of heavy metals in fifteen samples of imported skin-lightening

cream (C). The concentration of Pb, Hg, and Zn metals ranged from 0.09µg/g to 4.49µg/g, from 0.08µg/g to 6.64 µg/g and from 1.19 µg/g to 10.35 µg/g respectively. Table 6 shows that the concentration of heavy metals, Pb, Hg, Cd, Ni and Zn, in six eye shadow samples ranged from 17.22 µg/g to 52.02 µg/g, from 8.10µg/g to 21.30 µg/g, from 10.52 µg/g to 22.53 µg/g, from 12.25 µg/g to 73.94 µg/g and from 9.90 µg/g to 72.46 µg/g respectively.

Lead and cadmium are well known as potentially harmful metals that have aroused considerable interest. Particularly, lead has been described as the most harmful environmental contaminant recognized in human civilization and has shown renal impairment in patients with methyl-malonic aciduria and in the nervous system as stated in different reports linking it to a deficiency in cognitive functioning (Piccinini et al., 2013).

The metals analyzed in this study were not listed as ingredients in any of the products. Due to the lack of manufacturer testing and regulatory oversight, it is possible that the companies themselves were not even aware that their products were contaminated with these toxic metals (Nnorom, et al., 2005). These contaminants most likely entered into the cosmetic products when poor-quality ingredients were used. It is possible that the source of these toxic metals could have been from one or more of the inorganic base material used in manufacturing processes, (Omar, et al., 2001). In spite of the absence of strict control of cosmetic products, those of local lipstick (Table 2), face powder (Table 3) and imported skin- lightening cream (Table 5) samples were laden poisonous metals of Pb, Hg, Cd, As, Ni and Zn.

Similarly, imported lipstick (Table 1) and local skin – lightening cream (Table 4) samples revealed high levels of Pb, as well as Hg respectively. Clinically, Pb poisoning from use of Pb- based eye cosmetics is presented with abdominal cramped pain, encephalopathy (manifested as anxiety and irritability), and anemia (Chauhan, et al., 2010). In Asia, Africa, and the Middle East, they had been identified as suspected source of Pb exposure to the ocular system, (Guy, et al., 1999).

A recurrent theme in research on childhood Pb poisoning has been discovered time and again over the past four decades that Pb is toxic to the developing nervous system at levels previously thought to be safe. The observation that these facial cosmetics are used on neonates and by pregnant women should elicit concerns. Studies have reported that breastfeeding enhances the release of Pb from the bones and that Pb freely crosses the placenta, (Shannon, 2003).

Consequently, gestational Pb poisoning is not only harmful to the woman, but also to the developing fetus, invariably producing congenital Pb poisoning. Epidemiological studies confirm an association between Pb exposure and prevalence of dental carries in school-age children, (Lansdown, 2001). The applications of local preparations containing Pb and Hg, in the treatment of ophthalmic infections and as an eye cleanser and in cosmetic have been identified as a major source of Pb intake in Africa, (Dwivedi, 1996).

The present study indicated that the use of facial cosmetics sold in Sudanese markets exposes users to low levels of heavy metals of which Pb, Cd, and Hg are of most toxicological concern. Understanding the consequences of low-level human Pb and Cd poisoning will depend on upon an accurate assessment of the pervasiveness of toxicity in the global population.

This will require that toxicity thresholds be determined as well as an understanding of the mechanisms underlying toxicity, (International Union of P.A.C, 1976). Education of parents and childcare workers regarding the risks of administering heavy metals-based substances to children and themselves needs to be incorporated into health and healthcare framework systems in developing nations, (Campbell, et al., 2000). Keeping in view their toxicology, the estimation of toxic metals in cosmetic products has prompted us to carry out this study.

Unfortunately, there are no current international standards for impurities like heavy metals in cosmetics except 20µg/g for Lead and 5µg/g for cadmium, (Al-Trabulsy et al., 2013). The Canadian regularity limits for certain metals in cosmetic products are 10 for Pb, 3 for As, Cd, and Hg.

Health Canada but FDA recently allows being not more than (1ppb) in cosmetic products (Brown, 2013). Hg is toxic and rapid absorbed through the skin and can affect people neurologically causing blurred vision or trouble walking. The use of mercury in skin creams have been well documented in other countries and should be avoided. The continuous use of cosmetic products contaminated with such heavy metals may, however, cause a slow release of these metals into the human body and cause harmful effects to the consumer over time. Extensive use of such products should be avoided until the solution is adequately addressed. Removal of heavy metals from personal care products after manufacture is not possible. Nevertheless, careful selection of raw materials that are almost free from heavy metals can improve the quality of the products and save the beauty of the environment.

Finally, this study had revealed that continuous use of these cosmetics could result in an increase in the trace metal levels in the human body beyond acceptable limits. Efforts should be made at

enlightening the users and the general public on the dangers involved.

Table 1: *Concentration of heavy metals in imported lipstick samples collected from Sudanese markets*

Sample ID	Avg. conc. of Pb (µg/g)	Avg. conc. of Hg (µg/g)	Avg. conc. of Cd (µg/g)	Avg. conc. of As (µg/g)	Avg. conc. of Ni (µg/g)	Avg. conc. of Zn(µg/g)
L1	9.83	0.43	0.11	ND	ND	2.09
L2	12.5	ND	2.05	ND	0.95	1.32
L3	14.28	0.06	5.21	0.08	ND	2.17
L4	11.04	ND	0.74	ND	ND	1.04
L5	21.04	ND	0.69	ND	ND	5.06
L6	10.03	ND	1.82	ND	1.95	0.04
L7	16.93	0.22	1.77	5.14	ND	0.86
L8	8.47	1.33	2.11	4.40	3.16	ND
L9	42.42	0.25	0.43	2.71	ND	1.07
L10	12.34	0.61	ND	10.21	ND	2.35
L11	15.25	1.44	ND	5.01	ND	ND
L12	4.97	2.04	7.26	ND	0.20	0.69
L13	16.18	6.02	ND	3.00	ND	0.23
L14	22.10	ND	4.05	0.53	ND	0.84
L15	4.1 8	ND	12.00	0.08	ND	ND
L16	12.67	1.09	5.62	ND	ND	ND
L17	19.11	0.88	4.33	ND	ND	0.94
L18	11.75	ND	ND	ND	0.58	0.51
L19	6.08	ND	ND	ND	0.24	ND
L20	10.18	0.25	0.57	0.79	ND	ND

Where: ND, not detected, and L, for lipstick.

Table 2: *Concentration of heavy metals in local lipstick samples collected from Sudanese markets*

Sample ID	Avg. conc. of Pb (µg/g)	Avg. conc. of Hg (µg/g)	Avg. conc. of Cd (µg/g)	Avg. conc. of As (µg/g)	Avg. conc. of Ni (µg/g)	Avg. conc. of Zn (µg/g)
L L21	23.29	3.07	4.02	ND	2.35	10.23
L L22	28.59	8.96	ND	ND	9.41	5.20
L L23	11.45	8.01	0.66	0.24	5.80	1.00
L L24	17.41	2.35	1.50	ND	0.59	3.84
L L25	29.00	9.24	6.43	ND	0.16	7.47

Where ND, not detected, and LL, local lipstick.

Table 3: *Concentration of heavy metals in face powder samples collected from local Sudanese markets*

Sample ID	Avg. conc. of Pb (µg/g	Avg. conc. of Hg (µg/g	Avg. conc. of Cd(µg/g)	Avg. conc. of As(µg/g)	Avg. conc. of Ni (µg/g	Avg. conc. of Zn (µg/g)
P26	2.15	1.73	0.52	ND	0.84	4.30
P27	0.04	0.30	1.50	ND	1.32	6.23
P28	16.20	ND	0.37	ND	0.42	2.47
P29	8.64	2.10	0.80	0.51	0.08	9.52
P30	3.25	ND	0.53	0.09	ND	5.81
P31	14.07	1.08	2.06	1.21	ND	7.04
P32	16.31	ND	0.73	ND	1.68	2.47
P33	8.12	ND	3.11	0.84	0.92	5.88
P34	2.48	1.06	0.64	ND	1.30	3.30
P35	6.07	0.94	0.39	ND	0.37	2.56
P36	3.37	ND	0.87	0.24	ND	7.11
P37	9.51	ND	1.25	ND	0.89	4.38
P38	0.73	0.05	ND	ND	1.24	6.01
P39	2.04	1.06	ND	1.07	0.51	2.67
P40	20.02	2.11	0.56	1.25	ND	7.81
P41	5.47	ND	0.43	0.06	ND	3.22
P42	6.13	ND	1.72	ND	2.50	6.30
P43	10.48	ND	0.84	ND	0.77	9.21
P44	8.09	ND	ND	ND	1.80	8.63
P45	1.55	0.77	ND	ND	0.08	5.00
P46	2.93	2.01	ND	ND	0.85	9.52
P47	0.05	ND	1.20	0.54	1.35	3.30
P48	7.15	ND	0.61	ND	0.87	2.61
P49	4.28	1.09	0.72	ND	0.99	6.82
P50	6.54	ND	1.46	ND	2.01	7.43

Where: ND; not detected and P face powder.

Table 4: *Concentration of heavy metals in local skin-lightening cream samples (preparation) collected from Sudanese markets.*

Sample ID	Avg. conc. of Pb (µg/g)	Avg. conc. of Hg (µg/g	Avg. conc. of Cd (µg/g)	Avg. conc. of As (µg/g)	Avg. conc. of Ni (µg/g)	Avg. conc. of Zn (µg/g)
LC51	28.90	14.62	7.06	0.04	ND	2.41
LC52	14.49	14.10	ND	0.19	2.27	11.04
LC53	33.63	20.08	ND	0.05	0.54	2.92
LC54	17.13	42.09	1.09	0.96	ND	6.86
LC55	19.30	24.50	0.22	ND	0.88	1.18
LC56	30.63	16.05	ND	ND	1.36	3.20
LC57	23.68	21.63	2.79	0.29	1.26	6.27
LC58	17.20	13.02	ND	1.02	2.01	8.21
LC59	28.36	13.98	2.66	ND	ND	6.01
F&L 60	3.10	2.93	0.06	0.01	1.82	3.76
F&L 61	2.71	4.01	0.08	ND	0.65	7.63
F&L 62	3.80	2.51	ND	ND	0.27	2.40
F&L 63	7.27	1.67	0.03	0.05	0.51	5.16
F&L 64	6.51	2.04	0.09	ND	1.44	1.19
F&L 65	2.94	3.98	0.61	0.03	2.04	4.91

Where, ND not, detected, F&L fair & lovely and LC, local skin- lightening creams (local preparation).

Table 5: *Concentration of heavy metals in imported skin - lightening cream samples collected from Sudanese markets*

Sample ID	Avg. conc. of Pb(µg/g)	Avg. conc. of Hg(µg/g)	Avg. conc. of Cd(µg/g)	Avg. conc. of As(µg/g)	Avg. conc. of Ni (µg/g	Avg. conc. of Zn (µg/g)
C66	1.04	2.93	0.06	0.01	1.82	3.67
C67	0.53	4.01	0.08	ND	0.65	7.63
C68	0.09	2.51	ND	ND	0.27	2.40
C69	0.41	1.67	0.03	ND	0.51	5.16
C70	2.37	2.04	0.09	0.05	1.44	1.19
C71	0.34	3.98	0.61	ND	2.04	4.91
C72	2.73	2.86	0.14	0.03	1.12	4.14
C73	3.44	0.08	ND	0.15	ND	10.35
C74	0.65	2.21	ND	ND	3.09	4.40
C75	2.47	1.08	0.77	ND	2.23	3.11
C76	1.07	6.64	0.80	ND	0.91	6.20
C77	0.18	3.21	ND	ND	1.55	8.62
C78	4.49	1.84	ND	ND	8.21	1.36
C79	1.55	0.96	0.21	ND	7.35	6.24
C80	2.53	0.88	1.50	ND	7.12	4.58

Where, ND, not detected, and C, for skin lightening creams (imported).

Table 6: *Concentration of heavy metals in different brands of Eye Shadow samples collected from Sudanese market.*

Sample ID	Avg. conc. of Pb (µg/g)	Avg. conc. of Hg (µg/g)	Avg. conc. of Cd (µg/g)	Avg. conc. of As (µg/g)	Avg. conc. of Ni (µg/g)	Avg. conc. of Zn (µg/g
ES 91	24.48	12.73	10.52	0.07	73.94	14.30
ES 92	33.06	21.30	11.37	0.17	54.37	61.23
ES 93	40.07	19.21	18.72	ND	68.42	72.46
ES 94	17.22	8.10	22.53	ND	12.81	9.90
ES 95	52.02	9.74	12.13	ND	17.66	19.85
ES 96	29.95	11.12	10.56	ND	12.25	32.8

Where, ND: not detected, ES; eye shadow.

5. Conclusion:

Almost all imported and locally prepared facial makeup cosmetic samples were found to be contaminated with heavy metals. Results of ICP –MS analysis showed that the concentration ranged from 2.70µg/g to 52.02 µg/g and from 1.09 µg/g to 72.46 µg/g for lead and zinc respectively, however, the concentration ranged from 11.04 µg/g to 22.53 µg/g and 73.94 µg/g for Cd, As, and Ni, respectively. Moreover, the concentration of Hg, determined by using ICP-MS technique as well, ranged from 0.00 µg/g to 22.53 µg/g.

6. Recommendation:

Cosmetic products should be frequently analyzed for safety purposes, not only for toxic metals but also for preservatives, colors, and any other additives. Urgent efforts are needed for the establishment of limits of toxic metals, permissible preservatives, and colors to use in cosmetic products in Sudanese markets.

Corresponding Author:
Abdalla. M. Abdalla Mustafa,
Department of Chemistry, University of Science and Technology, Sudan.
E-mail: halawani2003@hotmail.com

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