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Hidden Metals in Several Brands of Lipstick and Face Powder Present on Polish Market

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Abstract: Cosmetics still retain their brilliant effect, but public concern about their toxicity has become a hot issue. Trace amounts of toxic heavy metals can be either intentionally added to cosmetics or present as impurities in the raw materials. We therefore assessed the levels of lead, nickel, copper, zinc and iron in six brands of lipstick and six brands of cosmetic powder that are widely available in local Polish markets. The cosmetics were digested and analyzed for the metals using flame atomic absorption spectrophotometry. Lead and nickel were found in some powders, but none in lipstick samples. This study revealed that the levels of these metals were higher than the specifications reported in the literature data. On the other hand, the copper content was determined at the level of 435 mg/kg in one sample of powder and at 75.92 mg/kg in one lipstick. Iron levels ranged from 0 to 12,168.57 mg/kg depending on the brand of powder or lipstick, and were generally higher in powders. Zinc was detected in the range of 1.73–488.31 mg/kg in all 12 samples. The results lead to the conclusion that constant control of metallic content in lipsticks, powders and other facial cosmetics should be seriously considered.

Keywords: lipstick; cosmetic powders; heavy metals; facial cosmetics

1. Introduction

Heavy metals occurring in the natural environment (water, soil, rocks) are found in trace amounts in raw materials used in the cosmetic industry. They can be desirable ingredients (e.g., dyes) or undesirable in many cosmetics and dietary supplements. Metallic impurities posing a threat to the health of consumers are, inter alia, particularly harmful elements such as lead (Pb) and nickel (Ni). Their content in cosmetic products is prohibited or restricted by the regulations of some countries, but, in many countries, there are no regulations in this regard. Apart from these toxic trace metals, elements such as iron (Fe), copper (Cu), and zinc (Zn) are necessary, but dangerous in excessive amounts, may also be present in cosmetics. Contamination of cosmetic preparations with heavy metals occurs in the process of production, or as a result of inadequate purification of the natural raw materials used as ingredients [1]. Despite numerous controls in the manufacturing process, production in accordance with the principles of Good Manufacturing Practice (GMP), and campaigns for the safe use of cosmetics, these elements are still present even in the best quality products. The single use of a cosmetic containing a toxic element is not usually associated with the appearance of side effects. However, due to the way they are used, they can pose a certain risk. One of the main problems is the presence of lead in lipsticks and in coloring agents. Some of the metals found in cosmetics, such as nickel and copper, cause allergic reactions [2,3]. The effect of long-term exposure to heavy metals is their accumulation in the body, and as a consequence, the increased risk of various health problems. In this work we present the results of a survey of metallic impurities in several lipsticks and face powders present on the Polish market.

The aim of the study has been to evaluate concentrations of lead, nickel, copper, zinc and iron. This choice of metals was imposed by the fact that Pb and Ni belong to compounds prohibited in cosmetics. Some Zn compounds are limited by special restrictions, and the presence of Cu and Fe is allowed in cosmetics according to Regulation No. 1223/2009 of the European Parliament [4]. Therefore, it was interesting to find out the presence and the levels of these metals in the chosen cosmetic products in order to draw consumers' awareness to their exposure to these hidden metals.

2. Materials and Methods

2.1. Samples

The cosmetic products were chosen by randomized sampling. The samples were chosen with consideration of variety of color, type, and form of product. The twelve cosmetic samples used in our studies, including five different brands of lipstick (two of them from one manufacturer) and six various brands of face powder, were present on the Polish market. Of the six face powders, half were Polish brands; and, one of them was available only on the Polish market. In case of the lipsticks, only one of them belonged to a Polish brand; nevertheless, this cosmetic is exported to European countries (such as Great Britain and Italy), and to the United States.

2.2. Reagents and Solutions

Analytical reagent-grade chemicals (HNO_3 (65%) and H_2O_2 (30%)) (J.T. Baker, Deventer, The Netherlands) were used for the preparation of all solutions. Ultrapure water from an HLP5 system (Hydrolab Polska, Gdansk, Poland) was used for preparation of working reagents and for sample dilution. Metal standards were prepared each day. Known amounts of the certified standard stock solution (1000 ppm, Merck, Darmstadt, Germany) were added to the three probes to give final concentrations in the range of 0.5–20 ppm for the selected metal. The concentration levels were driven by apparatus prescriptions, i.e., for Zn we used the following concentrations: 0.5, 1, and 2 μg/mL; for Fe and Ni, 2.5, 5, and 10 μg/mL; for Pb, 5, 10, and 20 μg/mL; and for Cu, 1.25, 2.5, and 5.0 μg/mL. Zero point for calibration was not used. There was a good linear relationship between absorbance and standard concentration of the metals used. The linear correlation coefficients (R^2) for the metals were in the range of 0.9822–1.000 (Table 1). The limit of detection (LOD) values were determined by apparatus prescriptions, which means that the lowest concentration in calibration for a selected metal was ascribed as the LOD value, whereas the limit of quantification (LOQ) values was defined as three times of the LOD value. Due to the unavailability of certified material, the accuracy of the method was determined by measuring the recovery of metal added to face powder matrices. These samples were run along with the test samples using the same analytical procedure. The analytical recovery from face powder samples with 0.006, 0.015, and 0.022 mg kg^{-1} for all metals used was 88.7–99.1%, 92.1–100%, and 95.9–101%, respectively. All the solutions were prepared in double-distilled water. Dilution correction was applied for samples diluted or concentrated during analysis. The glassware was cleaned and immersed in 6% nitric acid for 24 h. Teflon beakers were treated with 6% nitric acid and washed with ultrapure water.

2.3. Preliminary Treatment of Samples

Samples were homogenized by stirring them vigorously. To avoid metallic contamination, nonmetallic tools were used during sample preparation. When samples were not analyzed immediately, they were stored in thoroughly cleaned polyethylene containers. Solid samples (~1.0 g) were weighed in Teflon vessels and a mixture of 6 mL of 65% nitric acid and 2 mL of 30% hydrogen peroxide was added. The vessels were heated in a microwave digestion system according to the manufacturer's instructions (Magnum V2, Ertec, Wrocław, Poland). After mineralization, the liquid residues were diluted in a small amount of ultrapure water and then made up to 25 mL in volumetric flasks. A blank mineralization was carried out in the same way.

2.4. Measurements

For the hidden metals assays, a 932 AA spectrophotometer from GBC (Dandenong, VIC, Australia) with deuterium background correction was used. The system was controlled by a Data Station running the GBC Avanta Version 1.33 (GBC, 932 AA spectrophotometer, Dandenong, VIC, Australia). The instrument settings were as follows in Table 1.

Table 1. Atomic absorption spectrometer settings and validation data for analyzed metals.

	Instrument Settings			Flame Composition		Percent Recovery			Linearity	LOD	LOQ
	Wavelength (nm)	Slit Width (nm)	Lamp Current (mA)	Air (L/min)	Acetylene (L/min)	[a] 6.0 μg/L	[a] 15.0 μg/L	[a] 22.0 μg/L	R^2	(μg/L)	(μg/L)
Pb	217.0	1.0	5.0	7.0	2.0	93.4	92.1	98.8	0.9967	5.00	15.14
Ni	232.0	0.2	4.0	7.0	2.0	88.7	97.2	95.9	1.0000	2.50	7.50
Cu	324.7	0.2	3.0	7.0	2.0	91.8	99.6	99.7	0.9968	1.25	3.81
Zn	213.9	0.2	5.0	7.0	2.0	94.6	94.8	99.6	0.9884	0.50	1.50
Fe	248.0	0.2	5.0	7.0	2.0	99.1	100	101	0.9822	2.50	7.58

[a] amount added.

All samples were injected three times. Samples that gave a peak higher than that of the highest value of standard were automatically diluted and measured again. The calibration curve was obtained using three points, including the blank. Each sample was enriched with three standard solutions and re-analyzed to assess the accuracy of the total procedure of metals' analysis in cosmetics.

2.5. Statistical Methods

Results of the research were analyzed for statistical significance using the OriginPro 8.0 program (OriginLab Corporation, version 8.0 of OriginPro, Northampton, MA, USA). Values in the text are presented in tabulated form as mean ± SD with ND as not detectable. Experiments were carried out in triplicate.

3. Results and Discussion

The available literature data show that metals such as Pb, Ni, Cu, Fe and Zn are present in lipsticks and powders produced and used in various parts of the world. Metallic impurities can easily penetrate through the particularly delicate and sensitive skin of the face [5–8]. Lipsticks and powders are frequently used as daily facial cosmetics. Therefore, there is a serious need to find out what profiles Pb, Ni, Cu, Zn and Fe have in these cosmetic products. The results of the determination of the metal concentrations in several brands of the aforementioned face-care products available on the Polish market are presented in Table 2.

Most personal care ingredients and products enter into contact with human skin, occasionally resulting in local adverse effects. In addition, depending on their chemical and physical properties, skin penetration may occur, leading to human systemic exposure. The safety of ingredients for personal care products is regulated under cosmetic and/or pharmaceutical regulations within the EU, and under Code of Federal Regulations (CFR) Title 21, part 700 in the US issued by the US Food and Drug Administration (FDA).

The total content of Pb, Ni, Cu, Zn and Fe was estimated in randomly chosen lipsticks and cosmetic powders. Few brands had lead content above 20 mg/kg, which is the US FDA's limit for lead as impurities. This level might put consumers at the risk of lead poisoning. It is acknowledged that heavy metal impurities in cosmetic products are unavoidable due to the ubiquitous nature of these elements, but also that lead should be removed wherever technically feasible. Cosmetics in general can have a high concentration of trace-metal elements. Given the significant and relatively uncontrolled human exposure to cosmetics and their ingredients, these products must be thoroughly evaluated for their safety prior to marketing.

Table 2. Descriptive statistical summary of heavy metal concentration (mean ± SD) in different cosmetic products in mg/kg (ND = not detectable) ($p < 0.05$) (* $p < 0.01$; ** $p < 0.001$).

Sample ID	Type; Color	Pb	Ni	Cu	Zn	Fe
Powder 1	Mineral compact powder; beige	ND	ND	459.54 ± 18.84	488.31 ± 12.21	4654.79 ± 51.20
Powder 2	Hypoallergenic compact powder; white	ND	ND	ND	1.73 ± 0.002	9825.56 ± 49.13
Powder 3	Mineral fluid; beige	34.84 ± 0.69	ND	ND	25.4 ± 0.23	12,168.57 ± 243.37
Powder 4	Matte compact powder; beige	ND	ND	ND	3.79 ± 0.022	4183.18 ± 25.10
Powder 5	Powder pearls; beige	23.60 ± 0.21	9.95 ± 0.029 **	ND	3.70 ± 0.070	4829.33 ± 251.12
Powder 6	Matte compact powder; beige	17.43 ± 0.52	12.47 ± 0.55 **	ND	392.96 ± 16.11	5023.37 ± 185.86
Lipstick 1	Cream; beige	ND	ND	ND	3.14 ± 0.047	7830.51 ± 438.51
Lipstick 2	Cream; red	ND	ND	ND	2.14 ± 0.055	31.41 ± 1.41
Lipstick 3	Cream; purple	ND	ND	ND	4.63 ± 0.32	8046.42 ± 482.78
Lipstick 4	Matte; red	ND	ND	ND	2.25 ± 0.083	8.00 ± 0.44
Lipstick 5	Gloss; red	ND	ND	ND	4.47 ± 0.018	5373.81 ± 279.44
Lipstick 6	Cream; violet	ND	ND	75.92 ± 2.66 *	98.96 ± 0.74 *	160.57 ± 0.78

The concentrations of the studied metals varied significantly ($p < 0.05$) from one brand to another, and also varied between the categories of product. These differences may be associated with differences in raw material types, sources of raw materials, and sorting and manufacturing processes. The beige color of face powders did not have a significant effect on the metal content in the samples, but consistency, especially that of mineral fluid (No. 6), revealed an impact on lead and iron levels. In the case of lipsticks, there was no significant relationship between the color of the sample and the content of the metals determined. Sample No. 6 stood out as the only one containing copper. The type of raw materials, and especially color and mineral additives, probably plays the most important role in these metal concentrations. The data presented in Table 2 showed similarity with the global data reported in the literature (see Table 3).

Table 3. Selected literature data of heavy metal concentrations in lipsticks and powders (references are given in square brackets).

Metal	[mg/kg]	
	Lipsticks	**Powders**
Lead	0.04–3.75 [9] 0.3–2.44 [10] <0.025–1.32 [11] 0.23–3740.0 [12] 0.77–15.44 [13]	<2.3 [19] 0–61.86 [14] 0.0132–8.0610 [20] 5.9–3399. [16]
Nickel	0.14–4.24 [12] 0–22.8 [14] <0.012–6.27 [11] 0.0721–1.0626 [15]	<1.8–6.52 [19] 0.72–214.54 [14] 5.9–3399 [16]
Copper	0–118.6 [14] 0.0498–7.0782 [15] <0.01–7.38 [11] 1.1–135.4 [16]	0–9.69 [21] <1.2–3.92 [15] 1.4–23.4 [16]
Zinc	<0.03–18.00 [17] 9.2–33.0 [16]	<0.1–1874 [15] 8.0–3300 [16]
Iron	0–6839.7 [14,18] 421.6–44,070 [16]	0–1067 [14] 3591–40,835 [15] 157.3–47,098 [16]

3.1. Lead

Lead concentrations in the cosmetics tested in this study varied from <LOQ (below limit of detection) to 34.84 mg/kg. Powder 3 had an exceptionally high concentration compared to the other products. However, all the lipsticks contained lead below the limit of detection. The maximum allowable limit for Pb as an impurity in cosmetic products is set at 20 mg/kg by the US FDA [22], but according to Regulation No. 1223/2009 of the European Parliament [4], the presence of Pb is not allowed at all. In this study, 2 out of the 12 brands investigated exceeded the US FDA limits, and all of them were beyond European regulations. Cosmetic products with high concentrations of Pb, whether applied once or a number of times per day, could lead to human exposure to Pb [23].

3.2. Nickel

The concentration of Ni found in our samples falls within the concentration ranges reported in the literature (Table 3). The highest concentration of Ni (12.47 mg/kg) was observed in one brand of powder (No. 6). The presence of Ni in lipsticks was not observed (<LOQ). A Ni concentration of about 0.5 mg/kg is sufficient to cause contact dermatitis in skin [24,25]. The levels of Ni found in our cosmetic products could trigger contact dermatitis in people with hypersensitivity. The transport of Ni across the stratum corneum is slow, and is limited to <1%, but it depends on a number of factors including the counter ion (e.g., acetate, chloride, nitrate, or sulfate), the oxidizing capacity of sweat, the anatomical site, gender of the skin, dosage, and exposure time. Contact allergies associated with Ni exposure arise due to the ability of nickel to bind to amino acid residues to form Ni-complexed proteins [2].

3.3. Copper

The mean concentrations of Cu in these facial cosmetics ranged from ND to 459.54 mg/kg, and are in the range of the data reported in the literature (Table 3). The higher concentration of Cu in the powders could be due to the fact that copper-containing compounds might have been used as pigment in these types of facial cosmetics. Although Cu is rarely a skin sensitizer [26], there are reported cases of immune reactions due to exposure to Cu [3].

3.4. Zinc

Zinc is not a metal of significant toxicological importance. Cosmetics containing zinc fulfill different functions depending on the type of compound present. Zinc can act protectively, or give conditioning effects or antibacterial properties. Zinc salts increase adhesion and improve the application of the cosmetic [27]. An excess of zinc, similarly to other metals from the group of micronutrients, is harmful. Exposure to high levels of Zn is known to cause neurological and gastrointestinal disorders [28]. The results in this study showed that the highest concentrations of Zn were observed in the powders No. 1 and No. 6 (488.31 mg/kg and 392.96 mg/kg, respectively) and in one of lipsticks (98.96 mg/kg in No. 6). The Zn concentrations in other samples were much lower, and were similar to each other. These zinc contents fall within the scope of data presented elsewhere (Table 3).

3.5. Iron

Iron, like zinc, is considered to be a metal of insignificant toxicological effect, but a long exposure from cosmetic products may cause cellular death or colorectal cancer as a result of cumulative effects [29]. Iron in cosmetic products supports skin regeneration [19]. It takes part in the synthesis of collagen, and it has moisturizing properties. Iron compounds have a well-established role in dyes in many cosmetic products. Dyes containing iron are used in all mineral cosmetics in which the aim is to obtain a color similar to that of flesh shades. The concentrations of Fe in the studied products varied significantly between face powders (4183.18–12,168.57 mg/kg) and between lipsticks (8.00–8046.42 mg/kg). The concentrations of Fe in these cosmetic products were higher than the other elements studied. Our results do not differ from the values given in the literature (Table 3).

Based on this survey, the mean concentrations of metals can be set in the following order: Fe > Cu > Zn > Pb > Ni, which is in accordance with some available literature data. The values for Pb and Ni are a cause for alarm, because their presence is prohibited in cosmetic products in the countries of the EU according to Regulation No. 1223/2009 [4]. However, according to the Scientific Committee on Consumer Safety (SCCS), limits of lead and nickel in oral and non-cutaneous absorption were established as 20 mg/kg for Pb and 200 mg/kg for Ni [30]. These values could be considered safer in cosmetics, as skin absorption is much lower than oral absorption. The limits calculated according to the SCCS if respected, might protect consumers from systemic toxicity, but a different discussion relates to local reactions, which manifest themselves as irritation or allergy. The metals considered have irritant properties, but behave differently depending on the matrix they are in. Furthermore, the concentration values of metals would still have different meanings depending on the cosmetic product, because the type of exposure may be different leading to differing bioavailability of the metals. Therefore, it can be assumed that if the concentration "c" of the metal (Pb, Ni) is irritating in a lipstick, the same concentration could be harmless in a powder, or in any other cosmetic product, and vice versa.

Cosmetic products and ingredients are not subject to the US FDA pre-market approval authority, with the exception of color additives. The US FDA lays the responsibility on the cosmetic firms for checking the safety of their products and ingredients before introducing them into the market. Regarding Cu, Zn and Fe, there is no regulation of the levels in lipsticks or powders. There is only consideration about the metals' uses and restrictions. [31]. The amounts of these metals in cosmetics should be consistent with good manufacturing practice, and should not exceed the minimum reasonably required to accomplish the intended coloring effect.

Despite the fact that iron, copper, and zinc are essential in the body, the values obtained in this study raise safety concerns due to the cumulative effect arising from incessant exposure. The elevated levels of these metals in some of the products tested are very likely due to the use of some natural or inorganic pigments, such mica, or iron oxides, or the use of metal-coated apparatuses during the production of cosmetics.

4. Conclusions

The application of the flame atomic absorption spectroscopy (FAAS) technique allowed the quantification of heavy metals in facial cosmetics (lipsticks and powders). In some samples, lead and nickel were present at level prohibited by European regulation.

It should be emphasized that although Pb, Ni and Cu were not detected in some samples due to the detection limit of the analytical procedure, this does not mean that these metals were totally absent. However, from our results one can infer that a lower amount of Pb, Ni or Cu is more beneficial for consumers, especially for those with hypersensitivity to contact allergies.

The overall results indicate that powders with high amounts of lead and nickel can be harmful due to accumulation of these metals in the body. According to our studies, greater attention should be paid, and safer limits for metal concentrations in facial cosmetics should be seriously considered, especially for those metals causing allergic contact dermatitis. In consequence of the growing usage of cosmetics, it is necessary to pay special attention to these problems. Therefore, quality controls should be recommended for products designed to enter into long-term contact with the skin.

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