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Assessment of trace elements concentration in cosmetic foundation using X-ray fluorescence technique

Nogueira [a*], C. S.; Zamboni[a], R. S.; Paschuk[a], S. A.; Correa[a], J. N.

[A] Federal University Of Technology – Paraná, 80230-901, Curitiba, Parana, Brazil.

*Correspondence: camilanogueira@alunos.utfpr.edu.br

Abstract: Facial foundations play a significant role in beauty routines. However, the presence of heavy metals and toxic elements in cosmetics is a concern due to potential health risks. This study investigated the chemical composition of 52 facial foundation samples obtained in Curitiba and São Paulo, Brazil, using X-ray fluorescence for elemental identification. Quantification was performed using the standard addition method and calibration curves. The results indicated the presence of Cu, Zn, Br, Zr, and Ba in concentrations exceeding the limits established by the Brazilian Health Regulatory Agency (ANVISA). Cd and Ni, elements prohibited in cosmetic formulations, exhibited maximum concentrations of 277 ± 51 μg/g and 130 ± 10 μg/g, respectively. The highest concentration of Zr reached 589 ± 57 μg/g, approximately six times the regulatory limit. In contrast, Ba concentrations exceeded 14,000 μg/g, approximately 60 times the limit, with the highest concentration recorded at 30,867 ± 1,500 μg/g. These findings underscore the need for rigorous monitoring of cosmetic products in Brazil and highlight discrepancies between the marketed cosmetics and ANVISA regulations. Additionally, this study contributes to the growing knowledge regarding potentially harmful elements in facial foundations.

Keywords: facial foundation; trace elements; heavy metals; X-ray fluorescence.

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Avaliação da concentração de elementos traço em bases cosméticas por fluorescência de raios X

Resumo: As bases faciais desempenham um papel significativo na rotina de beleza. Contudo, a presença de metais pesados e elementos tóxicos em cosméticos é uma preocupação, devido aos potenciais riscos à saúde. Este estudo investigou a composição química de 52 amostras de bases faciais adquiridas em Curitiba e São Paulo, Brasil, utilizando a técnica de fluorescência de raios X para identificação dos elementos. A quantificação foi realizada pelos métodos de adição de padrão e curvas de calibração. Os resultados indicaram a presença de elementos como Cu, Zn, Br, Zr e Ba em concentrações acima dos limites estabelecidos pela Agência Nacional de Vigilância Sanitária (ANVISA). O Cd e o Ni, elementos cuja composição é proibida na formulação de cosméticos, apresentaram concentração máxima de 277 ± 51 µg/g e 130 ± 10 µg/g, respectivamente. A maior concentração determinada para o Zr atingiu 589 ± 57 µg/g, aproximadamente seis vezes acima do limite estabelecido. Em contrapartida, as concentrações de Ba ultrapassaram 14 mil µg/g, aproximadamente 60 vezes acima do limite, com a maior concentração atingindo 30.867 ± 1.500 µg/g. Estes resultados ressaltam a necessidade de monitoramento dos produtos cosméticos no Brasil e apontam discordâncias entre os cosméticos comercializados no país e as resoluções proposta pela ANVISA, além de contribuir para a ampliação do conhecimento acerca da presença de elementos potencialmente nocivos à saúde em bases faciais.

Palavras-chave: bases faciais; elementos traço; metais pesados; fluorescência de raios X.

1. INTRODUCTION

Facial foundations play a significant role in beauty routines and personal care. These products are developed through complex formulations, typically combining emulsions of water, oil, and silicone, ensuring adhesion, smoothness, and durability upon application. The diverse coloration of foundations is achieved through metallic pigments derived from oxides, allowing for a wide range of shades that adapt to different skin types [1,2].

In the regulatory context, both the Brazilian Health Regulatory Agency (ANVISA) and the European Parliament and of the Council of the European Union set guidelines regarding permissible ingredients in cosmetics, given that certain chemical elements and heavy metals in makeup products can be classified as impurities, presenting traces of contamination in the chemical substances used as dyes and pigments in cosmetics. Therefore, for a cosmetic product to be marketed in Brazil, manufacturers must meet several technical requirements, including detailed information about the formulation, documents certifying product safety, and assurance that it does not pose a health risk when used appropriately [3,4].

Despite this, several studies conducted in recent years indicate the presence of potentially toxic elements in cosmetic products, such as Cr, Ni, Hg, Sb, and others in makeup [5], Pb, Ni, Cu, Zn, and Fe in powder foundations [6], Cd, Cr, Fe, Ni, and Pb in fluid foundations [7], Al, Ba, Pb, and others in powder foundations [8], among others.

Since ANVISA sets the maximum permissible levels for contamination of artificial organic dyes and a list of substances not permitted in cosmetic products, it is scientifically relevant to conduct research to determine the concentration of such elements to ensure that cosmetic products marketed in Brazil comply with regulations. Therefore, this article aims to contribute to scientific knowledge by offering an analysis of the chemical composition of facial foundations, with an emphasis on potentially harmful substances and their implications for consumer safety.

In this study, X-ray fluorescence was employed for data collection and the identification of elements present in the samples. Quantitative analysis methods such as the standard addition method and calibration curve were used to quantify the elements found.

1.1. Heavy Metals and Toxic Elements

Metals in cosmetics can be present deliberately, such as using pigments, dyes, and UV filters, or due to contamination, whether during the cosmetic production process or through contamination of the raw materials used in manufacturing. Heavy metals, such as Hg, Cd, and Pb, can be absorbed through the skin and transported to organs, potentially causing health damage. Other metals, such as Ni, Co, and Cu, may not be absorbed but can accumulate on the outer layer of the skin, causing allergic dermatitis [9].

Research reports adverse reactions associated with heavy metals in cosmetics, e.g., allergic dermatitis, itching, scaling, and facial eczema due to Ni contamination in eye pencils [9]. Although zinc oxide (ZnO) is generally considered safe, a recent study indicated that its use, associated with UVB exposure, can cause severe skin inflammation and other health problems [10].

Metals in cosmetics may be intentionally present, such as in the use of pigments, dyes, and UV filters, or they may result from contamination, either during the cosmetic production process or due to the contamination of raw materials used in manufacturing.

1.2. Analytical Methods

The standard addition method allows for determining the concentration of an unknown element in the sample by successive additions of known concentrations of the element in the sample [11]. This method adds an appropriate amount of analyte to the sample of interest, increasing the analyte concentration in the sample.

The ratio between the analyte line's original intensity and the analyte's intensity plus the additive equals the ratio between the original analyte and the analyte plus additive concentrations. The relation between the original intensity and the intensity added analyte is shown in the following equation:

$$\frac{I_X}{I_{X+S}} = \frac{X}{X+S}$$

Where X and I_X correspond to the initial concentration and intensity of the analyte, respectively, and S to the concentration of the additive that, together with the analyte, generates an intensity response of I_{X+S} [11,12].

Once the initial concentration of the analyte in the sample of interest is known, the calibration curve method can be employed using the sample, now with a known concentration, as a standard for the quantification of the other samples.

In practice, standard samples with different concentrations of the analyte of interest are measured. The intensity of the analytical line is collected for each of the standards, and subsequently, a graph of analytical line intensity versus analyte concentration is constructed. From the calibration curve, quantification is done by measuring the intensity of the analytical line in the samples of interest and applying this intensity to the calibration curve equation to obtain the concentration of the analyte in the samples of interest [11].

2. MATERIALS AND METHODS

In this study, 52 samples of facial foundations were analyzed and acquired from cosmetic and perfumery stores in Curitiba and São Paulo, Brazil. Brand popularity or product specifics were not considered. There was no initial price limitation on the samples, with the price range of the acquired foundations between 15 BRL and 125 BRL in 2023. No chemical treatment was performed on the samples.

The samples were cataloged: letter A followed by a number identifying the product/brand analyzed, and the letters C for fair and E for dark classify the foundation shades. Thus, samples of the same brand with the C and E designations were analyzed in fair and dark shades.

Data was collected using equipment composed of an X-ray tube with an Ag target (Mini-X Model) and a silicon semiconductor detector (X-123SDD Model - Silicon Drift Detectors), both marketed by Amptek Inc.®. The silicon drift detector (SDD) is composed of a fully depleted, high-resistivity silicon crystal with a thickness of 500 μm and an ultrathin dead layer of 0.15 μm. It features a beryllium (Be) window with a thickness of 0.5 mil, allowing minimal X-ray attenuation. The active detection area is 25 mm², ensuring reliable signal collection.

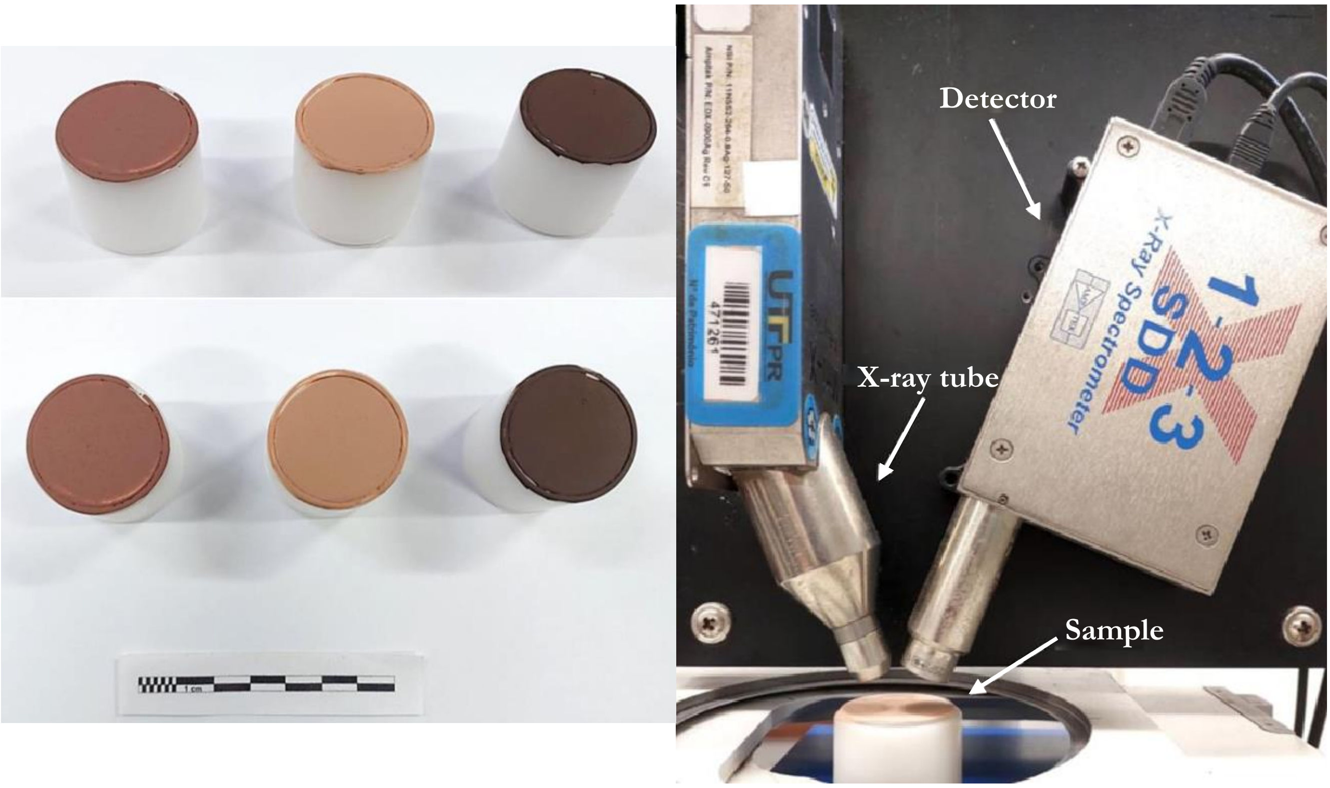
Data acquisition was performed using a potential of 40 kV and a current of 10 μA with a 1 mm collimator at the exit of the X-ray tube beam. The dead time variation for all collected spectra was 2% to 8%. Furthermore, during spectrum acquisition, the multichannel analyzer and the digital pulse processor were operated using 4,096 channels with an energy resolution of 9.9 eV per channel and a total gain of 31.61x. Spectrum analysis was performed using the open-source software PyMCA [13], where the elemental composition of the samples was identified, and peak area information was extracted for subsequent quantification using the standard addition and calibration curve methods.

2.1. Standard Samples

The aqueous solutions containing known concentrations of the elements of interest were used to prepare the standard samples.

A small concentration of those solutions was added into a container containing a fixed amount of facial foundation. The solution and facial foundation were manually mixed, as the foundation contains a large amount of emulsifier, which facilitated homogeneity. The mixture of facial foundation and solution was then poured onto a support with an external diameter of 44.7 mm, an internal diameter of 39.9 mm, and a height of 29.5 mm, from the PANalytical®, along with a plastic film approximately 10 μm thick from the Bompack®, and exposed to the irradiation and detection kit (Mini-X and X-123SDD) under the same parameters used for the samples without the addition, as shown in Fig. 1.

Figure 1: Prepared sample and acquisition setup.



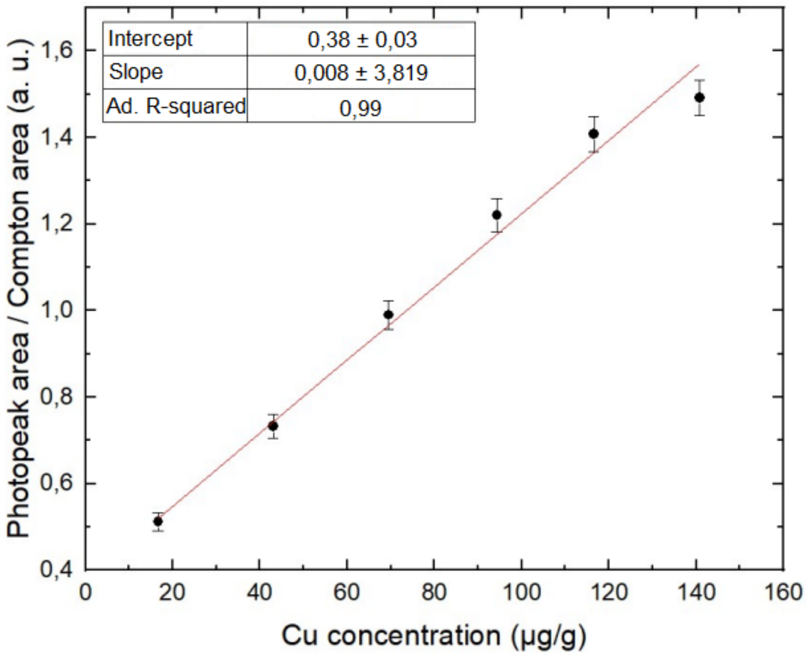
Source: Authors (2025).

This procedure was repeated five times, with different concentrations of the compound added to the foundation each time. In total, six measurements of the standard sample were obtained: five corresponding to the added concentrations and one with the untreated foundation, which was used as the zero point.

These samples were then subjected to the standard addition method, and the initial concentration of the elements of interest in the standard samples was determined. Once the initial concentration was known, a calibration curve graph was constructed using the

OriginPro® software for each element of interest. The calibration curve for the copper element is presented in Fig. 2.

Figure 2: Calibration curve for Cu.



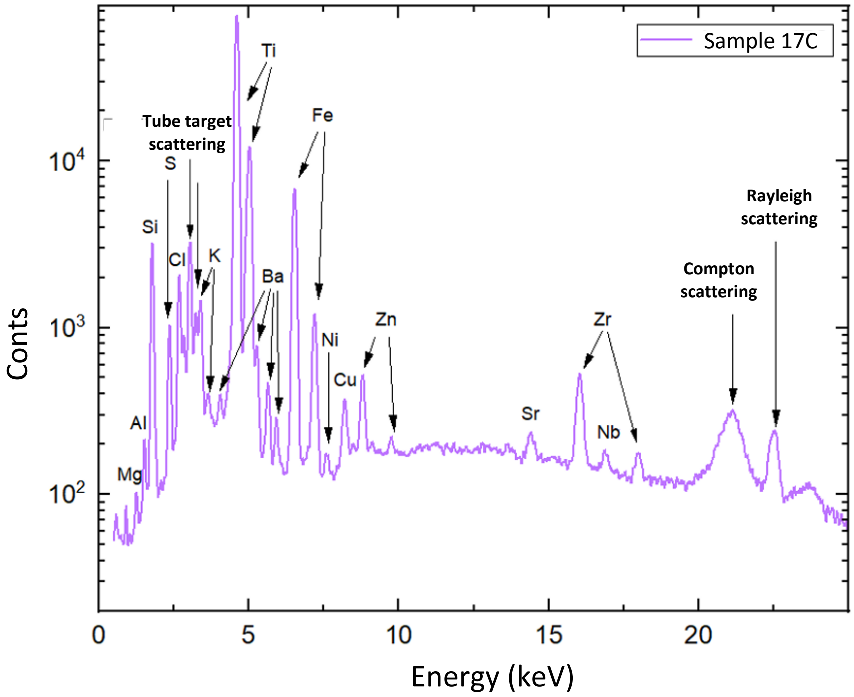
Source: Authors (2024).

3. RESULTS AND DISCUSSIONS

The qualitative analysis allowed the identification of the elements Mg, Al, Si, P, S, Cl, K, Ca, Ti, Cr, Mn, Fe, Ni, Cu, Zn, As, Br, Rb, Sr, Zr, Nb, Cd, and Ba.

In Fig. 3, the spectrum of a sample containing trace elements and the identification of the elements in this sample can be observed.

Figure 3: Spectrum sample 17 fair on a logarithmic scale.



Source: Authors (2024).

Of these chemical elements, Mg, Al, Si, S, K, Ca, Ti, and Fe were not quantified since chemical compounds of these elements are deliberately present in the formulation and are included in the list of ingredients in the products.

Calibration curves were constructed for the remaining elements. The equation of the curve provided by the linear fit of the experimental data allowed for quantifying the 52 analyzed samples. A curve similar to that shown in Fig. 2 was created for each analyzed element.

Table I presents the mean and maximum concentration values, as well as the detection limit for each element.

Table 1: Mean and maximum concentrations and limit of detection of trace elements found on facial foundation.

Element	CAS number	Mean (µg/g)	Maximum (µg/g)	Limit of detection (µg/g)
Cr	7440-47-3	11	25.4 ± 4.2	0.12
Ni	7440-02-0	12	130.3 ± 1.0	0.11
Cu	7440-50-8	37	142.0 ± 9.9	0.38
Zn	7440-66-6	1730	33288 ± 2736	0.45
Br	7726-95-6	103	143 ± 18	0.09
Rb	7440-17-7	4	5.3 ± 4.1	0.11
Sr	7440-24-6	7	22.1 ± 3.7	0.07
Zr	7440-67-7	118	589 ± 57	0
Ba	7440-39-3	22579.2	30867 ± 1486	0.13
Cd	7440-43-9	101.5	277 ± 51	0

According to ANVISA's RDC No. 529 resolution, Cr, Ni, Br, Cd, and Sr compounds are prohibited in cosmetic products at any concentration [14]. These elements were found in quantities ranging from tens to hundreds of parts per million. Noteworthy are the concentrations of Br, Cd, and Ni in samples A15C (143 ± 18 µg/g), A36E (277 ± 51 µg/g), and A36C (130 ± 10 µg/g), respectively.

Regarding restrictive elements, concentrations exceeding the limits established by ANVISA [15] of 100 µg/g for heavy metals and 500 µg/g for the element Ba were identified. The highest concentration determined for Zr was approximately six times above the limit (589 ± 57 µg/g), while Ba was obtained approximately 60 times above the limit (30867 ± 1500 µg/g).

However, it is essential to note that in another resolution (RDC No. 628), ANVISA determines that insoluble lacquers, salts, and pigments of Ba, Zr, and Sr are permitted as long as their insolubility is proven [15]. Since the analyses in this report do not constitute adequate tests to verify the insolubility of these elements, it was not possible to infer these characteristics of the compounds, and, therefore, the concentrations obtained in this study may be aligned with ANVISA norms.

For Cu, only two samples, A36C and A37C, presented concentration values above the limit, being 108.7 ± 8.4 μg/g and 142.0 ± 9.9 μg/g, respectively.

Zn, in turn, presented elevated concentration numbers concerning the established limit, the highest of which was 33287 ± 2700 μg/g in sample A36C, approximately 330 times above the limit. However, it should be noted that Zn is an element commonly used in cosmetics to prevent damage caused by solar radiation and is permitted with a maximum concentration of 25% in cosmetic formulations [16].

Rb was observed in four samples in low concentrations, being 5.3 ± 4.1 μg/g in sample A17C and 2.36 ± 0.69 μg/g in sample A21C. In samples A8E and A26C, the concentration was below the detection limit.

The element As was detected in one sample. However, it was not possible to quantify this element. When obtaining the calibration curve equation for As, the peak intensity of As in the sample of interest was below the detection limit. Therefore, it was not possible to determine whether it complied with the limits proposed by ANVISA.

When comparing the results obtained in this study with recent literature, the concentrations are comparable for some elements. Table 2 presents this comparison.

Table 2 : Maximum concentrations in μg/g of the elements found in facial foundations: in this study with other studies.

Element	This study	Saah et al. (2022) [17]	Abdel-Ghany e Ragab (2016) [18]	Shomar e Rashkeev (2021) [8]
Cr	25.4 ± 4.2	83,7	153 ± 72	6741 ± 1261
Ni	130.3 ± 1.0	-	318 ± 145	-
Cu	142.0 ± 9.9	-	1001 ± 722	8124 ± 2149
Zn	33288 ± 2736	7,17x10^4	673 ± 433	8909 ± 2102
Br	143 ± 18	-	-	-
Rb	5.3 ± 4.1	-	25 ± 13	-
Sr	22.1 ± 3.7	-	27 ± 12	-
Zr	589 ± 57	-	25 ± 18	-
Ba	30867 ± 1486	-	-	32 ± 11
Cd	277 ± 51	-	-	25 ± 4

The elements Cr, Ni, Cu, Rb, and Sr presented statistically similar results to those of Abdel-Ghany and Ragab (2016), with a 95.4% confidence limit.

The discrepancies in the concentration values may be related to the type of product analyzed, whether to the texture of the cosmetic, the raw material used in manufacturing, or the brand. Different products, especially those developed in different regions, are also expected to present different contamination levels.

4. CONCLUSIONS

This study aimed to determine the concentrations of trace elements in fluid foundation samples acquired from cosmetic stores in Curitiba and São Paulo. The X-ray fluorescence technique was employed for the analyses, and the elements were quantified using the standard addition and calibration curve methods.

The analysis of the element concentrations revealed the presence of health-hazardous elements, such as Ni and Cd, in considerable amounts. Although in most samples the Ni concentrations were below 10 μg/g, two samples recorded concentrations of 77.7 ± 5.8 μg/g and 130 ± 10 μg/g. Conversely, Cd exhibited concentrations above 100 μg/g in 6 of the 24 samples where this element was identified, with the highest concentration being approximately 277 ± 51 μg/g.

Additionally, high concentrations of Ba were found in two analyzed samples. These values exceed 14,000 μg/g, with the maximum allowable value by ANVISA for this element being 500 μg/g [15].

Despite the caveats in the regulations established by the national regulatory agency, all quantified elements presented concentrations above the limits proposed by ANVISA in at least one sample, except for Rb. Furthermore, elements prohibited by ANVISA in the formulation of cosmetics, including Cr, Ni, Br, Cd, and Sr, were identified.

This research contributes to expanding the understanding of potentially harmful elements in cosmetics. Moreover, it highlights discrepancies between the marketed products in the country and the resolutions proposed by the Brazilian Health Regulatory Agency.

Finally, it is recommended that this study be extended to include makeup and cosmetics in general, as well as other personal care products frequently used in Brazilians' daily routines.

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CONFLICT OF INTEREST

The authors declare the following academic commitments, which may be considered potential competing interests: This report results from a master's dissertation presented to the Graduate Program in Electrical Engineering and Industrial Informatics (CPGEI) of the Federal University of Technology – Paraná – Brazil (UTFPR).

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