Sample Preparation and Analysis of Cosmetics Using Sequential Microwave Digestion

Abstract

Elemental analysis of cosmetics is necessitated by both governmental regulation and guidance, as well as safety of potential consumers. It is important that there is a safe, rapid, and efficient way to digest these cosmetics to achieve a sensitive and accurate analysis via Inductively Coupled Plasma – Optical Emission Spectroscopy (ICP-OES). The BLADE™ microwave digestion system from CEM Corporation can safely operate at the temperatures and pressures required to provide a rapid and efficient digestion of these materials. A variety of cosmetics were digested using the BLADE for analysis by ICP-OES. Following FDA's guidance for testing, arsenic (As), cadmium (Cd), chromium (Cr), cobalt (Co), lead (Pb), mercury (Hg), and nickel (Ni) were analyzed. In addition, silicon (Si) and titanium (Ti) were also analyzed, as they are commonly used in cosmetics. Concentrations of these elements are reported in lip gloss, lipstick, powder foundation, eyeshadow, mascara, and liquid foundation.

Introduction

Cosmetics and beauty products have been used by humans throughout our history; eyeshadow and blush have been present since BCE in both Egyptian and Greek cultures. Human use of cosmetics has only increased since, with an estimated current global market size of 299.77 billion[1]. From the everyday application of sunscreen or lotion to the use of makeup for beauty enhancement or special effects, cosmetics and topicals are regularly used in daily life. Metals have a long history of use as colorants in cosmetics, with iron oxides being common in concealers and aluminum in lipsticks[2]. Other uses for the metals include as preservatives, antiperspirants, and UV filters[3]. Many heavy metals such as arsenic, cadmium, lead, and mercury can be toxic to humans and, as such, must be analyzed and regulated in consumer products. Regular use, coupled with the method of application, makes accurate heavy metals analysis of special concern in cosmetics.

In this study, cosmetics were digested using the BLADE microwave digestion system and analyzed on an Agilent ICP-OES. Traditional heavy metals of concern such as Pb, Hg, As, and Cd, as well as other metals tested by the FDA[4], and ones commonly used in cosmetics were analyzed. Titanium and silicon were specifically examined due to their historic uses in makeup.

Materials and Methods

Instrumentation

The samples discussed were all digested using the BLADE. Designed for sequential digestion, the BLADE utilizes automation for loading and unloading samples using a robotic arm. A simple quartz vessel, with the option of Teflon® liners, and a two-piece snap cap are used to contain each digestion sample. Samples are heated to temperature using microwave energy and stirred using magnetic stir bars to ensure even digestion. After digestion, samples are cooled, unloaded from the cavity, and the system automatically loads the next sample until the entire queue is finished. The sequential format of the BLADE allows for a variety of acids and different sample matrices to be used in a single batch. During digestions, the samples can be observed using a built-in camera.

Sample Preparation

Standards PT-CS-20 Lip Gloss and PT-CS-21 Powdered Cosmetics were purchased from LGC Standards. Different types of cosmetics, including lip gloss, lipstick, powder foundation, eyeshadow, mascara, plant-based mascara, and liquid foundation, were purchased from a local retailer. A 0.1 g sample size of each sample was weighed onto filter paper for simple transfer into the digestion vessel. Filter paper was tested to be free of contaminants and was included in acid blanks.

GFS Veritas® Trace Metals Grade nitric acid (HNO_3), hydrochloric acid (HCl), and hydrofluoric acid (HF) were used. The acid mixtures detailed in **Table 1** (page 2) were utilized, and each sample was heated to 220 °C and held for 5 minutes. The lipstick was digested with and without the use of HF for comparison.

Samples that needed HF required a second digestion step using boric acid to dissolve acid-insoluble fluorides such as CaF_2. After cooling below 70 °C, the vessels were opened and solid boric acid + H_2O were added as detailed in **Table 1** (page 2). The vessels were sealed and the solutions were heated to 160 °C and held at that temperature for 2 minutes. Digests were diluted to 50 g with DI water prior to analysis.

Table 1. Samples with Acid Mixtures

	HNO_3 (mL)	HCl (mL)	HF (mL)	H_3BO_3 (g)	H_2O (mL)
Lip Gloss Standard (PT-CS-20)	3.5	0.5	2	1	10
Powdered Cosmetics Standard (PT-CS-21)	3.5	0.5	2	1	10
Lip Gloss	3.5	0.5	2	1	10
Lipstick (HF)	3.5	0.5	2	1	10
Lipstick (No HF)	3.5	0.5	0	0	0
Powder Foundation	3.5	0.5	2	1	10
Eyeshadow	3.5	0.5	2	1	10
Mascara	3.5	0.5	1	0.5	10
Plant-Based Mascara	3.5	0.5	1	0.5	10
Liquid Foundation	3.5	0.5	1	0.5	10

Analysis

Samples were analyzed using an Agilent 5110 ICP-OES. The conditions are listed in **Table 2**. All samples were analyzed for a range of trace and percent level elements, based upon their composition and associated contaminant concerns.

Table 2. Instrument Conditions of Agilent 5110 ICP-OES

Parameters	Values
RF Power (kw)	1.20
Viewing Mode	SVDV
Viewing Height (mm)	8
Read Time (sec)	20
Nebulizer Flow (L/min)	0.70
Plasma Flow (L/min)	12.00
Aux Flow (L/min)	1.00
Rinse (sec)	30.00
Internal Standard	Yttrium (377.433 nm)
Background Correction	Fitted

Results

Elemental concentrations and percent recovery values for the lip gloss and powdered cosmetics standards are presented in **Table 3**. Recoveries for both standards were found to be within the 97- 105% range for the certified elements, well within the accepted criteria.

Table 3. Average Elemental Concentrations (ppm) and Percent Recovery Values for the Certified Elements in the Standards

Avg (n=3)	As	Cd	Hg	Pb	Cr	Ni
Lip Gloss	17.01	38.88	40.83	46.35	11.41	25.31
Expected	17.08	39.10	40.83	46.01	11.50	24.40
% Recovery	99.57	99.44	99.93	100.7	99.22	99.63
Powdered Cosmetic	19.03	21.17	12.49	38.99	39.78	22.44
Expected	18.13	21.65	12.46	39.69	39.84	22.84
% Recovery	105.0	97.76	100.2	98.24	99.86	88.26

The average elemental concentration of the elements tested by the FDA of the store-bought cosmetics is presented in **Table 4**.

Table 4. Elemental Concentration (ppm) in Cosmetics

Avg (n=3)	As	Cd	Co	Cr	Hg	Ni	Pb
Lip Gloss	5.39	0.82	5.92	13.7	2.03	0.570	n.d
Lipstick	6.29	0.86	8.37	11.8	1.85	0.860	1.28
Powder Foundation	8.55	0.65	54.6	65.8	0.620	2.70	n.d
Eyeshadow	8.37	0.99	2.14	101	1.42	3.99	1.87
Mascara	2.50	1.26	2.41	2.28	1.00	2.04	2.60
Plant-Based Mascara	2.65	1.13	2.80	4.27	0.780	4.71	2.28
Liquid Foundation	5.04	0.36	128	13.3	1.56	0.204	0.380

Silicon and titanium content of the cosmetics are presented in **Table 5**. These elements were found to be abundant at the percent level, which was expected due to their known use in cosmetics. Furthermore, it can be seen that when lipstick was digested without the use of HF, the abundance of both Si and Ti was significantly less than when digested with HF. This data supports when analyzing for these elements it is important that HF is used.

Table 5. Si and Ti concentration (ppm) in Cosmetics

Avg (n=3)	Si	Ti
Lipstick (No HF)	1390	178
Lipstick (with HF)	22900	3160
Lip Gloss	29000	3190
Powder Foundation	157000	41300
Eyeshadow	210000	842
Mascara	1100	2.93
Plant-Based Mascara	3040	18.4
Liquid Foundation	24800	77100

Conclusion

Proper monitoring and analysis of elemental impurities in cosmetics is imperative to ensuring consumer health. Furthermore, analyzing elements commonly used in the production of cosmetics is important for quality assurance. By utilizing the BLADE microwave digestion system, various types of cosmetics were fully digested and analyzed for their elemental content, thus providing a safe, rapid, and efficient way to digest these cosmetics to achieve a sensitive and accurate analysis using ICP-OES.

References

[1] Cosmetics Market Size, Share | Global Industry Trends [2030]. https://www.fortunebusinessinsights.com/cosmetics-market-102614 (accessed January 25, 2024).

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[3] Burger P., Landreau A., Azoulay S., Michel T., Fernandez X. Skin Whitening Cosmetics: Feedback and Challenges in the Development of Natural Skin Lighteners. *Cosmetics*. [Online] **2016**, 3(4), 36. https://www.mdpi.com/2079-9284/3/4/36 (accessed January 25, 2024).

[4] US Food and Drug Administration. FDA's Testing of Cosmetics for Arsenic, Cadmium, Chromium, Cobalt, Lead, Mercury, and Nickel Content. https://www.fda.gov/cosmetics/potential-contaminants-cosmetics/fdas-testing-cosmetics-arsenic-cadmium-chromium-cobalt-lead-mercury-and-nickel-content (accessed January 25, 2024).

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