

Determination of Toxic Elements in Lipstick by ICP-MS after Oxygen Pressurized Microwave-Assisted Digestion Using Diluted Nitric Acid

Vanize C. Costa¹ (PG), Rochele S. Picoloto² (PQ), Alessandra C. Teotonio¹ (IC), Thais Mazzetti¹ (IC), Rodrigo M. Pereira¹ (PG), Marcia Foster Mesko^{1*} (PQ) (marcia.mesko@pq.cnpq.br)

¹ Centro de Ciências Químicas, Farmacêuticas e de Alimentos, Universidade Federal de Pelotas, RS, Brazil.

² Núcleo de Química, Universidade Tecnológica Federal do Paraná, PR, Brazil.

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Introduction

Nowadays, the use of cosmetics has increased mainly due to the pursuit of beautification. However, some commercially available cosmetics can be contaminated with toxic elements, which damage human health.¹ These elements typically are used in cosmetics as coloring agents. In the case of lipstick, the effects caused by toxic elements may be more significant, as there is a higher risk of direct oral ingestion. In this sense, the determination of toxic elements such as As, Cd, Cr, Ni and Pb is very important. Many analytical techniques have been used to determine these elements in lipstick,¹ and in general, a sample preparation step must be carried out before analysis. In this way, conventional or microwave-assisted acid digestion using concentrated acids have been widely used for digestion of lipstick.^{2,3} However, the use of concentrated acids could increase blank values, resulting in higher limits of detection (LODs), and it can cause interferences during the determination step. Consequently, an additional step could be necessary to remove the excess of acid, which could result in higher LODs making the determination of elements at low concentrations difficult. In this work, microwave-assisted digestion in closed vessels using only diluted nitric acid within oxygen-pressurized atmosphere (MW-O₂-AD) was evaluated regarding the digestion of lipstick for subsequently As, Cd, Cr, Ni and Pb determination by ICP-MS.

Results and Discussion

Lipstick samples (labeled as "A to C") were purchased in a local market (Pelotas-RS). Before analysis, the samples were homogenized in a water bath at 70 °C for 20 min. Initially, lipsticks samples (500 mg) were digested by microwave-assisted acid digestion (MW-AD) using 6 ml of concentrated HNO₃. Additionally, for the digestion of lipstick samples by the proposed MW-O₂-AD method, 6 ml of 3, 5 or 7 mol l⁻¹ HNO₃ solutions and pressures of 5, 10 or 15 bar of O₂ were evaluated (experiments were performed using lipstick sample "A"). Digestions were carried using a microwave oven (Multiwave 3000™, Anton Paar), and the microwave heating program applied was as follows: (i) 1000 W for 10 min (ramp of 5 min) and (ii) 0 W for 20 min. Determination of ⁷⁵As, ¹¹¹Cd, ⁵³Cr, ⁵⁸Ni and ²⁰⁸Pb was performed by ICP-MS.^{2,3} The accuracy of the proposed MW-O₂-AD method was evaluated by recovery tests and by comparison with results

obtained by ICP-MS after lipstick sample digestion using the MW-AD method. Recoveries between 99 and 107% for Cd, Cr, Ni and Pb using 5 mol l⁻¹ HNO₃ solution and 10 bar of O₂ were obtained (relative standard deviations were lower than 10% for all analytes). Moreover, the results obtained for Cd, Cr, Ni and Pb by ICP-MS after digestion using the proposed MW-O₂-AD method (5 mol l⁻¹ HNO₃ solution and 10 bar of O₂) did not present a statistical difference (t-test, 95% of confidence level) with results obtained by ICP-MS after the samples digestion using the conventional MW-AD method. On the other hand, recoveries of As were above 115% when lipstick sample "A" was digested by MW-O₂-AD, probably due to interference caused by the presence of carbon in digests (2156 ± 65 mg l⁻¹ of C). Nevertheless, this interference can be eliminated by the dilution of the final solution (up to 8 times). Afterward, lipstick samples "B" and "C" were also digested by proposed MW-O₂-AD method using 6 ml of 5 mol l⁻¹ HNO₃ solution and 10 bar of O₂. The results obtained for As, Cd, Cr, Ni and Pb by ICP-MS in lipstick samples ranged from 26 to 35, 258 to 331, 202 to 2673, 157 to 2331 and 51 to 184 µg kg⁻¹, respectively. It is important to mention that concentrations of analytes in these samples were lower than limits established by some regulatory agencies.²

Conclusion

The proposed MW-O₂-AD method, using 6 ml of 5 mol l⁻¹ HNO₃ solution and 10 bar of O₂, was suitable for the decomposition of 500 mg of lipstick for subsequent As, Cd, Cr, Ni and Pb determination by ICP-MS. By using the proposed method, it was possible to utilize only diluted nitric acid solutions for lipstick digestion, minimizing the generation of acid residues, according to the recommendations of green chemistry.

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