Challenges in sample preparation of lipstick for further toxic elements determination by ICP-MS: is the use of complexing acids necessary?

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Abstract

Complexing acids combined with HNO₃ were evaluated for lipstick digestion and toxic elements determination by ICP-MS.

Introduction

Toxic elements can be present in raw materials used in cosmetics, especially in mineral pigments and can be transferred to the final product.¹ In the case of lipstick, the effects caused by toxic elements may be more significant, due to the higher risk of oral ingestion. Many analytical techniques have been used, especially for Cd, Cr, Ni and Pb in lipstick. However, in general, a sample preparation step must be carried out before analysis to convert the sample to a suitable solution for analyses.¹ In this context, conventional or microwave-assisted acid digestion using concentrated acids has been widely used for digestion of lipstick. Moreover, in most studies, a mixture containing concentrated HNO₃ and HF has been used for sample preparation.²,³ Usually, HNO₃ solutions are used for digestion of organic compounds, and HF is used due to its good complexation capacity, when compared to other acids. However, when HF is used, sample introduction systems of some equipment may be damaged due to high reactivity of this acid with glass and quartz materials. The HF also does not allow the application of methods based on diluted acids, in which the systems used are usually composed of quartz. Furthermore, HF has high toxicity and it is difficult to purify. Thus, in this work, the use of complexing acids (HF and HCl) combined with HNO₃ for digestion of varied types of lipstick was carefully evaluated for further Cd, Cr, Ni and Pb determination by ICP-MS.

Results and discussion

Samples of creamy, shimmering and opaque lipstick were purchased in a local market (Pelotas-RS). Before analysis, the samples were homogenized in a water bath at 70 °C for 20 min. Samples (500 mg) were digested in closed vessels using concentrated acids, including 6 ml of HNO₃, a mixture of HNO₃ (6 ml) and HF (0.5 or 2 ml), or HNO₃ (6 ml) and HCl (0.5 or 2 ml). Digestions were carried out using a microwave oven (Multiwave 3000TM, Anton Paar), and the microwave heating program applied was as follows: (i) 750 W for 10 min (ramp of 20 min) and (ii) 0 W for 20 min. Determination of $^{111}$Cd, $^{53}$Cr, $^{58}$Ni and $^{208}$Pb was performed by ICP-MS. The results for Cd, Cr and Ni after digestion of creamy lipstick using only HNO₃, HNO₃ and HF (0.5 or 2 ml), or HNO₃ and HCl (0.5 or 2 ml) did not present a statistical difference (ANOVA, 95% of confidence level). For Pb no statistical difference was observed between the results obtained using HNO₃ (272 ± 14 ng g⁻¹) or HNO₃ and HCl (0.5 ml: 271 ± 12 ng g⁻¹ or 2 ml: 299 ± 18 ng g⁻¹). However, the obtained values for Pb in creamy lipstick samples were lower than the limit of detection (298 ng g⁻¹) when the mixture of HNO₃ and HF was used for digestion. For the shimmering lipstick, results for Cr and Pb after digestion using only HNO₃ were lower than results obtained for other evaluated conditions. Probably, this behavior is related to the presence of aluminosilicates in shimmering lipstick, which can difficult the elements extraction without complexing acids, due to their interaction with metals. However, according to the obtained results, the use of HNO₃ and HCl (0.5 ml) was suitable for solubilization of both elements during digestion of shimmering lipstick, with the use of HF not being necessary. Moreover, for this condition, the RSDs were lower than 4%. Obtained results for Cd, Ni and Pb in opaque lipstick after digestion using all evaluated conditions did not present statistical difference (ANOVA, 95% of confidence level). Furthermore, for Cr no statistical difference was verified between concentrations obtained using HNO₃ (2903 ± 178 ng g⁻¹) or HNO₃ + HF (2979 ± 125 ng g⁻¹). Thus, the results indicate that, probably, the use of complexing acids is only necessary for Cr and Pb determination in shimmering lipstick.

Conclusion

The use of complexing acids is essential for Cr and Pb determination in shimmering lipsticks, which present significant amounts of silicates in their constitution. Moreover, the HCl allowed the solubilization of Cr and Pb without totally solubilizing the inorganic residue obtained after digestion, which can interfere in the determination step.

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References