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ACCEPTED MANUSCRIPT

Miniaturized matrix solid-phase dispersion followed by liquid chromatography-tandem mass spectrometry for the quantification of synthetic dyes in cosmetics and foodstuffs used or consumed by children

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Highlights

- MSPD-LC-MS/MS developed for dyes analysis in products mostly consumed by children
- LC-MS/MS optimized using a very low ionic strength mobile phase (3mM)
- Miniaturized MSPD optimized for rapid, low cost, and easy sample preparation
- Method performance demonstrated in a broad range of real cosmetics and confectionery
- Dyes in samples were \geq 989 µg g⁻¹, and a prohibited dye was found in a candy

ABSTRACT

Miniaturized matrix solid-phase dispersion (MSPD) followed by liquid chromatography tandem mass spectrometry (LC-MS/MS) has been proposed for the simultaneous analysis of different classes of synthetic dyes in confectionery and cosmetics intended for or mostly consumed by children. Selected compounds include most of the permitted dyes as food additives as well as some of the most frequently used to color cosmetic products in accordance with the respective European directives. MSPD procedure was optimized by means of experimental design, allowing an effective, rapid and simple extraction of dyes with low sample and reagents consumption (0.1 g of sample and 2 mL of elution solvent). LC-MS/MS was optimized for good resolution, selectivity and sensitivity using a low ionic strength mobile phase (3 mM NH₄Ac-methanol). Method performance was demonstrated in real samples showing good linearity (R ≥ 0.9928) and intra- and inter-day precision (%RSD \leq 15%). Method LODs were \leq 0.952 µg g⁻¹ and \leq 0.476 µg g⁻¹ ¹ for confectionery and cosmetic samples, respectively. Recoveries of compounds from nine different matrices were quantitative. The validated method was successfully applied to 24 commercial samples (14 cosmetics and 10 foods) in which 9 of the selected dyes were found at concentrations up to 989 µg g⁻¹, exceeding in some cases the regulated maximum permitted limits. A non-permitted dye, Acid Orange 7, was found in one candy.

Keywords: synthetic dyes; cosmetics; confectionery; matrix solid-phase dispersion; liquid chromatography-tandem mass spectrometry

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