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Electrolyte-assisted microemulsion breaking in vortex-agitated solidified floating organic drop microextraction for preconcentration and analysis of Sudan dyes in chili products†

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For the extraction and determination of trace amounts of Sudan I–IV dyes in chili product samples, a simple and efficient liquid phase microextraction technique was developed using electrolyte-assisted microemulsion breaking in vortex-agitated solidified floating organic drop microextraction (VA-SFODME-EAMB) and high performance liquid chromatography. Under optimal conditions, 5.0 mL of sample solution yielded a pre-concentration factor of 62. Linearity (0.5–2500 ng mL^{-1}, R^2 > 0.99), the limit of detection (0.16–0.24 ng mL^{-1}), and the limit of quantification (0.53–0.80 ng mL^{-1}) were used to validate this method. The precision, expressed as relative standard deviation (% RSD), was calculated using seven and five experiments with mixed Sudan dyes at a concentration of 500 ng mL^{-1}. Intra- and inter-day analyses validated the % RSD precision data (<3.5 and 7.0%, respectively). For the analyzed real samples, the recoveries of these dyes ranged between 90.1% and 109%. This newly proposed method is simple, rapid, and environmentally friendly without using both a disperser solvent and a centrifugation step to separate the aqueous and organic phases. In particular, it is applicable for food analysis as a useful screening test of illegal adulteration with Sudan dyes.

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Introduction

Sudan I–IV dyes (ESI, Table S1†), phenyl-azoic derivatives, are extensively used as colorants in chemical industries and various household products. They have widely been used for making plastics, printing inks, waxes, leather, fabrics, and floor polishes.[1–3] Despite being banned as food additives, they are quite often detected in food products, such as chili powder, chili products, curry, curcuma, red pepper, and virgin palm oil, because they enhance their visual aesthetics and promote sales. Sudan dyes belong to the azo-dyes group, and therefore, are stable under food preparation conditions. However, they can be enzymatically transformed into aromatic amines in the human body,[4] possibly leading to an abnormal headache in adults,[5] neurotoxicities,[6] genotoxicity,[7] and carcinogenicity.[8] They posed an increased risk of cancer in humans and were classified as category 3 carcinogens by the IARC (International Agency for Research on Cancer). As a result, Sudan dyes are illegal as additives in foodstuffs destined for human consumption according to both the FSA (Food Standards Agency) and the European Union[9] and their presence at any level is not safe for humans.[10] In recent years, events impacting on the security of food supply have caused wide food scarcity around the world. Therefore, it is essential to develop a sensitive and accurate method to detect Sudan dyes in food. However, prior to the analysis by high performance liquid chromatography (HPLC), these dyes need to be extracted and pre-concentrated by techniques such as solid-phase extraction,[11] magnetic solid-phase extraction,[12] film microextraction,[13] molecularly imprinted polymer extraction,[14] matrix solid-phase dispersion extraction,[15] ultrasound-assisted liquid–liquid extraction,[16] ionic liquid extraction,[17] cloud point extraction,[18] gel permeation chromatography,[19] microwave-assisted ionic liquid microextraction[20] and organic solvent-free air-assisted liquid–liquid microextraction (LLME).[21]

The development of dispersive liquid-phase microextraction with solidification of floating organic droplets (SFO-DLPME) was reported in 2014 by Chen and Huang.[22] In this method, 1-

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