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Short communication

Simultaneous determination of three azo dyes in food product by ion mobility spectrometry

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ABSTRACT

Color is an important property for food evaluation. Synthetic azo dyes are usually used in food product to obtain better appearance because of their stability and low cost. However, such dyes should be strictly controlled because of their potential threat to human health. A simple, rapid and sensitive method has been developed to determine orange II, allura red, and para red simultaneously by ion mobility spectrometry. The three dyes could be separated at the same time and the migration time of orange II, allura red, and para red are 12.070 ± 0.010, 8.180 ± 0.015, and 11.037 ± 0.016 ms, respectively. The effects of different parameters, such as pH, solvent, percentage of water, were investigated to establish the optimal condition. The detection limits were 0.1, 0.05, and 0.2 μg/mL for orange II, allura red, and para red, respectively. The recoveries of the three azo dyes from jellies were all higher than 81%. The developed method is fast and accurate for the detection of the three synthetic dyes.

1. Introduction

Color is one of the most important properties for food evaluation. Color is not only an attractive element to catch people's attention, but also a characteristic that reveals the quality of food. A variety of natural or synthetic food colorants are added to food products to enhance visual aesthetics and show excellent quality. However, most natural dyes are unstable and could be easily degraded during food production, and cost much more than synthetic food colorants. Hence, synthetic dyes are widely used for their stability and low cost. Azo dyes are a class of synthetic food colorants employed in food industry, and they are characterized by azo groups (—N=N—) in their structures. The use of azo dyes is strictly controlled by legislation worldwide because of their potential risk to human health [1,2]. Azo dyes can be reduced by azoreductase enzyme, releasing aromatic amines in intestinal bacteria and liver cells [3]. Considering both the potential threat to health and the need for food production, detection of synthetic dyes in food is becoming increasingly important.

Recent studies have proved the importance of developing fast and accurate methods for detection of synthetic dyes [4]. Several methods can be used to detect dyes by absorbance measurement, including thin-layer chromatography [5,6] and spectrophotometry [7,8],; however, these methods are limited by overlap of absorption spectra and time-consuming sample pretreatment. Capillary electrophoresis is suitable for ionic species but is limited by the small injection volume [9,10]. Surface-enhanced Raman spectroscopy is a rapid detection method suitable for fast-field analysis [11]. The most widely used detection techniques are ion-pair chromatography [12] and high-performance liquid chromatography (HPLC) with ultraviolet/visible (UV/Vis) and diode-array (DAD) [13,14]. Liquid chromatography–mass spectrometry (LC–MS) is a general method for detection of synthetic dyes characterized by reliability and low limit of detection [15]. LC–MS is usually used for analysis of illegal and toxic dyes at trace levels [14].

Ion mobility spectrometry (IMS) is an analytical technique with a wide applicability for volatile and semi-volatile compounds. IMS is characterized by high sensitivity, low cost, and fast response. IMS was first used for detecting chemical warfare agents in military and screening explosives at airports [16]. As a powerful analytical technique, IMS was developed to perform qualitative and quantitative analysis of toxic compounds [16,17], drugs [18], air [19], and food

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[20–22]. However, rapid detection of synthetic dyes with IMS has not been reported.

Para red is a type of industrial dye that is illegally added into some spicy Mexican food, and has promoted panic all over the word in 2005, for it causes irritation to eyes, skin, and respiratory system. Allura red and orange II can be used as food additives, but controlled at a low level. In this study, the three azo dyes (i.e., allura red, para red, and orange II) were detected by IMS under optimized condition. A simple, rapid, and sensitive method for synthetic dyes detecting was developed, with a fast sample preparation.

2. Method

2.1. Reagents and chemicals

The standard solutions of orange II (0.5 mg/mL), para red (1.0 mg/mL), allura red (1.0 mg/mL) were purchased from the National Research Center for Certified Reference Materials (Beijing, China). Methanol and other solvent (HPLC Grade) was purchased from Sinopharm Chemical Reagent Beijing Company (Beijing, China). All solutions prepared for IMS were filtered through 0.45 μm membranes before use.

2.2. Instruments

The IMS used in this work (Excellims GA2100) utilizes electrospray ionization (ESI) as ion source. The carrier and drift gases were both dried air.

2.3. Preparation of standard and sample solutions

A portion (5 mL) of 1 mg/mL standard solutions was diluted in 100 mL of solvent (methanol: water of 90:10) to obtain a 50 μg/mL standard solution.

All samples were obtained from a local market. Jelly was smashed first before adding the same volume of solvent (methanol: water of 90:10). After shaking for 10 min, the tubes were centrifuged for 1 min at 10,000 rpm. The supernatant was transferred to a new tube and filtered through 0.45 μm membranes. A sample solution (1 mL) was diluted in 10 mL of solvent (methanol: water of 90:10) to obtained a diluted sample solution when necessary.

2.4. IMS experiment conditions

The IMS parameters are described below. The ion source was either positive or negative, depending on the characters of the substance to be examined. The suitable source voltage was different for different dyes (Section 3.1). The drift tube voltage, gas inlet temperature, drift tube temperature, gate voltage, gate pulse width, drift gas glow, exhaust pump, spec summing, run time, and spectrum length were set at 7500 V, 180 °C, 180 °C, 45 V, 120 μs, 1.2 L/min, 0.8 L/min, 10, 30 s, and 25 ms, respectively, without any change.

Citric acid (migration time is 8.606 ms) was used to calibrate the drift time of IMS in negative mode.

3. Results and discussion

3.1. Optimization of IMS parameters

Both the positive and negative modes in IMS were tested to detect orange II, allura red, and para red. In positive mode, no signal was detected (data not shown). However, in negative mode, the three azo dyes all showed a significant peak in the IMS spectrum (Fig. 2). Thus, the negative mode of IMS was chosen to detect the azo dyes. Orange II, allura red, and para red all have sulfate groups

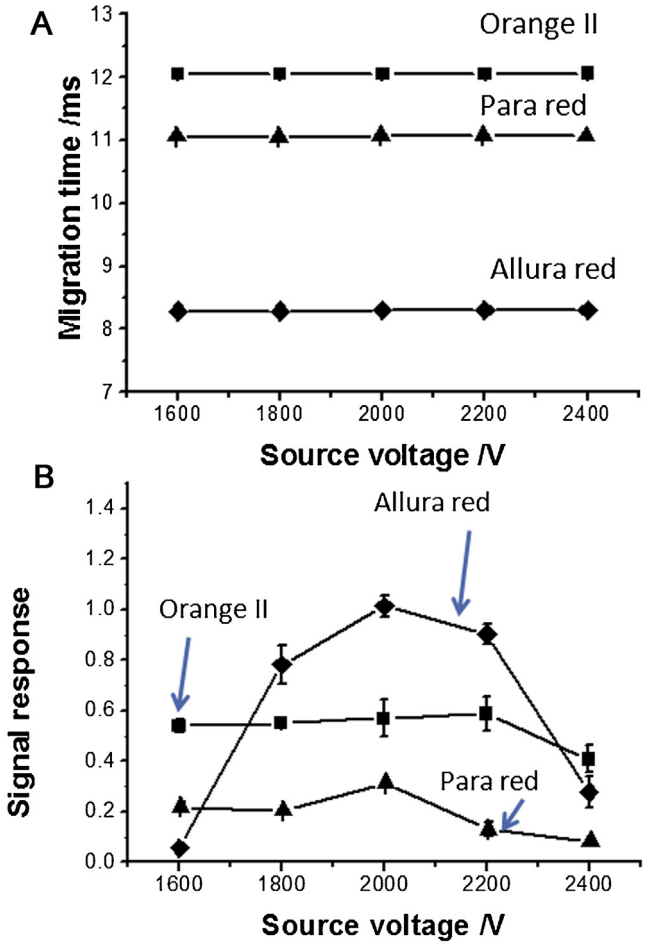

Fig. 1. The response of three azo dyes on different source voltage in IMS. (A) The change of migration time on different source voltage. (B) The change of signal response on different source voltage. Filled square for orange II, filled diamond for allura red and filled triangle for para red.

($-SO_3$) or hydroxyl group (—OH), and thus they could easily lose a hydrogen atom to form a negative ion.

Different source voltages provide different ionization degrees of compounds; Source voltage of 1600 V–2400 V was tested to determine a best response with three replicates (Fig. 1). The migration time of the three azo dyes showed no significant changes in different voltages (Fig. 1A). However, the signal response changed dramatically with the change in voltage (Fig. 1B). At 2000 V, allura red has the greatest response, which slightly changed and at 1800 and 2200 V. By contrast, the response area decreased dramatically at 1600 and 2400 V. The greatest response of orange II and para red were at 2200 and 2000 V, respectively, and the signal responses slightly decreased at 1800, 2000, and 2200 V. Thus, 2000 V of source voltage was chosen for the detection of azo dyes to obtain a stable response.

3.2. Characterization of the three dyes in IMS

Standard solutions (50 mg/mL) of orange II, allura red, and para red were injected into the IMS instrument for characterization. The IMS spectrum of orange II showed only one signal (Fig. 2A), and the migration time was 12.070 ± 0.010 (Table 1). The IMS spectrum of allura red displayed one peak (Fig. 2B), and the migration time was 8.294 ± 0.010 (Table 1). The IMS spectrum of para red showed two peak signals (Fig. 2C); one at 11 ms, and the other at 14 ms. The latter was proposed to be the peak for the para red dimer, whereas