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Separation and determination of acrylamide in potato chips by micellar electrokinetic capillary chromatography

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Abstract

A simple and rapid method using micellar electrokinetic capillary chromatography (MEKC) was developed for the separation and determination of acrylamide in potato chips at low levels for the first time. The experimental conditions for the separation and quantification of acrylamide were optimized at first. The optimized conditions were: 50 mmol/L $Na_2B_4O_7$ and 40 mmol/L SDS at pH 10.0, 12 kV applied voltage, 76 cm total length (67 cm effective length) and 75 μm i.d. capillary, 198 nm wavelength, 15 cm high 25 s hydrodynamics sample injection, 20 °C air-cooling. The linear response of acrylamide concentration ranges from 0.5 to 100 μg/mL with high correlation coefficient ($r = 0.9986$, $n = 9$). The LOD and LOQ were estimated to be 0.1 and 0.33 μg/mL based on S/N = 3 and 10. The precision values (expressed as R.S.D.) of intra- and inter-day were 0.86–4.35% and 2.61–9.65%, respectively. Recoveries spiked at levels 2, 20, 60 μg/mL ranged between 90.86% and 99.6% with R.S.D. less than 6.5%. Finally, the developed method has been applied to the analysis of real samples and has achieved satisfactory results. All of these indicated that it was a reliable method for the quantification of acrylamide in potato chips.

Keywords: Acrylamide; MEKC; Potato chips; Quantification; Capillary electrophoresis

1. Introduction

In April 2002, researchers from the University of Stockholm and the Swedish National Food Administration (NFA) reported the presence of acrylamide (2-propenamide) in a wide range of fried and oven-cooked foods [1]. These findings have attracted considerable attention worldwide because acrylamide has been classified as "probably carcinogenic to humans" by the International Agency for Research on Cancer (IARC) [2]. Recent studies have shown that acrylamide (see Fig. 1) was formed during the Maillard reaction, and that the major reactants leading to the release of acrylamide were sugars and asparagine [3,4]. The potential health risk of acrylamide in food has been considered by a number of government agencies and national authorities [5]. Thus, simple and sensitive analytical methods for the separation and determination of acrylamide are of great interest.

So far, methods based on gas chromatography (GC)–mass spectrometry (MS) [6–10] and liquid chromatography with tandem mass spectrometry (LC–MS/MS) [11–18] techniques have been reported. Although acknowledged as the most useful and authoritative method for the determination of acrylamide [19], these methods have some connatural disadvantages, such as complex procedure, expensive consume of instrument and reagents, pollution of organic reagents in laboratory, etc. [20]. Capillary electrophoresis (CE) has been successfully applied to the analysis of the complex matrices [21–23] because of its advantages of excellent separation efficiency, rapid analysis and minimal use of the samples and solvents [24,25]. Micellar electrokinetic capillary chromatography (MEKC) is an important separation mode of CE, and it can be employed for the separation of not only charged but also neural compounds by means of its capacity to partition molecules between the aqueous phase and the pseudo-stationary micellar phase [26,27]. It has been proven that MEKC can compete with HPLC with regard to the efficiency and selectivity [28,29], and several MEKC methods have been developed for the analysis of natural products [30,31].

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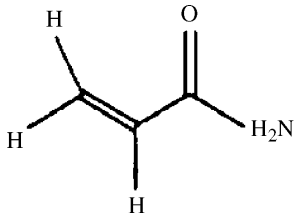

Fig. 1. Chemical structure of acrylamide. 71. 08 g/mol, CAS: 79-06-1.

In this paper, we had an innovative try and took the lead in developing a simple, fast and low-cost analytical method for the determination of acrylamide in potato chips by MEKC. Our work was based on the systematic investigation of the influences of pH value, concentration of buffer and SDS, applied voltage and the validation of method.

2. Experimental

2.1. Apparatus

The analysis of acrylamide was performed by an ACS 2000 HPCE apparatus (Beijing Cailu Scientific Inc., Beijing, China). The apparatus were comprised of a digital power supply (up to voltage 30 kV), a HW-2000 chromatography workstation and a UV–vis detector that could perform wavelength scanning from 190 to 740 nm. A fused-silica capillary (Factory of Yongnian Optical Fiber, Heibei, China), which was with 75 μm i.d. × 76 cm (67 cm effective length) was used. An Ultra-pure Water System (SG Ultra Clear system, Wasseraufbereitung und Regenerierstation Gmbh, Germany) was used to produce ultra pure water. pH meter (METTLER TOLEDO, Switzerland) was used for the pH measurement.

2.2. Chemicals

Acrylamide and sodium dodecyl sulfate (biochemical reagent, BC) were purchased from the Shanghai Shisheng Cell and Bio-technology Company (Shanghai, China). Sodium tetraborate (guarantee reagent, GR) was purchased from the Shanghai Chemistry Reagent Company (Shanghai, China). Methacrylamide (≥98% purity) and hexane (analytical reagent, AR) were obtained from the Sinopharm Chemical Reagent Co., Ltd. (Shanghai, China). Methanol (analytical reagent, AR) was purchased from the First Chemical Plant of Zhenxing (Shanghai, China).

2.3. Standards and reagents

$Na_2B_4O_7$-SDS solutions were chosen as the background electrolyte in our experiment. The pH value of the buffers ranged from 8.5 to 10.5 and the concentrations of $Na_2B_4O_7$ changed from 10 to 60 mmol/L as well as the SDS was from 10 to 50 mmol/L.

Stock solution of acrylamide (1 mg/mL) and methacrylamide (1 mg/mL) were prepared by dissolving in MeOH. Working standards for HPCE analysis were prepared according to the following procedures. 200, 120, 80, 40, 20, 10, 4, 2 and 1 μL of acrylamide solution (1 mg/mL) and 100 μL methacrylamide solution (1 mg/mL) were added into blank sample, respectively. Corresponding volume of MeOH were added afterwards and then the working standards were conducted in accordance with the procedures in Section 2.4. The final concentrations of working standards were 100, 60, 40, 20, 10, 5, 1 and 0.5 μg/mL.

2.4. Sample preparation

Finely ground potato chips (4 g) were weighed into a 50 mL centrifuge tube, and then 20 mL MeOH [32] and 100 μL internal standard (methacrylamide, 1 mg/mL) were added into the tube which would be strongly vibrated with a vibrator for 2 min later. After that, the mixture in the tube was centrifuged at 10,000 rpm (11,180 × *g*) and 10 °C for 10 min. The extraction was repeated for three times and the clear supernatants were combined together and were transferred into a beaker placed in a water bath at 70 °C for evaporation. The residue was dissolved in 1 mL ultra-pure water, and then the same volume hexane was added for defatting for two times. Before injection the sample solution was diluted two times with background electrolyte. All solutions were stored at 4 °C in a refrigerator until use.

2.5. Analytical procedures

HPCE was carried out with the following procedures. Before using, the new capillary was conditioned by rinsing with 1 mol/L NaOH for 20 min, ultra-pure water for 20 min, 1 mol/L HCl for 20 min, ultra-pure water for 20 min, and equilibrated for 30 min with the background electrolyte, in order. Between injections, the capillary was rinsed with the background electrolyte for 3 min. Since acrylamide is a derivative of carboxylic acid, the detection wavelength was set at 198 nm, which is the maximum absorbance wavelength of acrylamide [32]. Hydrodynamics injection (15 cm high, 25 s) was chosen to load samples. The working voltage was 12 kV. Temperature control of capillary was carried out with 20 °C air-cooling. Detection data were collected and processed with the HW-2000 Chromatography Workstation Software.

3. Results and discussions

3.1. Optimization of conditions

3.1.1. Effect of running voltage

Applied voltage had a great important effect on migration time, current strength and resolution. It indicated that with the increase of applied voltage, the migration time decreased due to the increasing of electroosmotic flow (EOF). However, it would induce a poor resolution of peaks as well as a deteriorated baseline due to the much higher Joule's heating and electric current at higher voltage. In order to obtain better resolution and faster detection of acrylamide, 12 kV was chosen as the working voltage.