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Development of a fast and selective separation method to determine histamine in tuna fish samples using capillary zone electrophoresis

Luciano Vitali [a], Andressa Camargo Valese [a], Mônia Stremel Azevedo [a], Luciano Valdemiro Gonzaga [a], Ana Carolina Oliveira Costa [a], Marcel Piovezan [b], Jacqueline Pereira Vistuba [b], Gustavo A. Micke [b,*]

[a] Department of Science and Food Technology, Federal University of Santa Catarina, 88040-900 Florianopolis, SC, Brazil
[b] Department of Chemistry, Federal University of Santa Catarina, 88040-900 Florianopolis, SC, Brazil

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ABSTRACT

This paper reports on the development of a fast and selective separation method by capillary zone electrophoresis (CZE) for the determination of histamine in tuna fish samples. The background electrolyte was composed of 60 mmol L^{-1} hydroxyisobutyric acid and 10 mmol L^{-1} sodium hydroxide at pH 3.3. The internal standard used was imidazole. Separations were performed in a fused uncoated silica capillary (32 cm total length, 8.5 cm effective length and 50 μm internal diameter) with direct UV detection at 210 nm. The samples and standards were injected hydrodynamically (50 mbar, 3 s) from the outlet capillary end (nearest to the detector) and the electrophoretic system was operated under normal polarity and constant voltage conditions of 30 kV (positive polarity on the injection side). The migration time of histamine in the proposed method was only 0.34 min. The method was then validated and different tuna fish samples were analyzed. Good linearity ($R^2 > 0.999$), a limit of detection 0.14 mg L^{-1}, intra-day precision better than 3.5% (peak area of sample), and recovery in the range of 94–108% were obtained. The results of the histamine concentration determined in the samples by the CZE method were compared with the LC–MS/MS method.

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1. Introduction

Histamine is a biogenic amine present in various levels in many foods, such as cheese, vegetables, fish, and others. It forms in food by decarboxylation of the amino acid histidine catalyzed by L-histidine decarboxylase in the presence of decarboxylase-positive microorganisms, and by conditions that allow bacterial growth and decarboxylase activity. Free histidine can be found naturally in foods or may be liberated by proteolysis during processing or storage. Therefore, high concentrations of histamine in foods are related to microbial fermentation. Thus, histamine can be used as an indicator of hygienic food quality [1]. Furthermore, foods containing high levels of histamine are related to food-borne illness. In case of fish consumption, according to the Food and Drug Administration (FDA), histamine levels above 200 mg kg^{-1} can cause the development of an illness called scombroid poisoning. Some symptoms of this illness include tingling or burning in or around the mouth or throat, rash or hives on the upper body, drop in the blood pressure, headache, dizziness, itching of the skin, nausea, vomiting, diarrhea, asthmatic-like constriction of the air passage, heart palpitation, and respiratory distress [2,3]. In United States, the FDA regulates a maximum limit of 50 mg kg^{-1} (5 mg per 100 g) of histamine for fresh and canned fish [2]. In Brazil, the Brazilian Ministry of Agriculture and Livestock (Portuguese acronym MAPA) has established a maximum limit of 100 mg kg^{-1} of histamine in the muscles of fresh and frozen fish, as well as for canned fish [4–6]. Papers have reported that in addition to the conditions of hygiene, other factors of processing methods for fish, including their storage temperature and storage time, influence in the histamine concentration [7–11].

Several methods were proposed in the literature for the determination of histamine in fish samples. These include high-performance liquid chromatography with fluorescence or UV detection and the use of derivatization [12], gas chromatography with flame ionizing detector [13], ultra-performance liquid chromatography with UV detection [14], high-performance liquid chromatography with fluorescence detection using the pre-column derivatization procedure [15], high-performance liquid chromatography with fluorescence and UV detection [16], ion-exchange chromatography with conductivity detection [17], capillary electrophoresis and high-performance liquid chromatography with UV detection [18], and liquid chromatography–tandem mass spectrometry using a solid phase extraction procedure [19]. In the most of these methods, the separation time of

* Corresponding author. Tel.: +55 48 3721 6852x215; fax: +55 48 3721 6852.
E-mail address: gustavomicke@qmc.ufsc.br (G.A. Micke).

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the histamine varies over a wide range (3–12 min), and in some cases, is too long. The complexity of the sample matrix may contribute to these long separation times observed due to the appearance of the interfering peaks in the separation. In some cases, sample preparation (clean up) procedures are used in order to reduce these interference effects. However, these effects can be minimized while developing the method of analysis. Capillary electrophoresis (CE) is a separation technique that has the potential to allow the selection of appropriate conditions for the separation method in order to minimize the number of peaks that can interfere in the separation of the analytes. Likewise, the "cleaning of the electropherogram" may contribute to rapid separations with a duration less than 1 min, which increases the instrumental throughput of the analysis.

In this context, the aim of this study was to develop a fast and selective method for histamine determination using capillary zone electrophoresis (CZE) with UV detection. The proposed method was applied in the analysis of tuna fish samples and the results were compared with the LC–MS/MS method.

2. Experimental section

2.1. Chemicals and solutions

α-Hydroxyisobutyric acid (HIBA) purchased from Sigma-Aldrich (Sao Paulo, SP, Brazil) and sodium hydroxide purchased from Tedia Brazil (Rio de Janeiro, RJ, Brazil) were used in the background electrolyte (BGE). Ethanol (EtOH, content 99%) acquired from Synth (Diadema, SP, Brazil) was used to prepare the tuna fish samples. The standards of histamine and imidazole (internal standard, IS) were purchased from Sigma-Aldrich (Sao Paulo, SP, Brazil) and MERCK (Darmstadt, Germany), respectively. The standard stock solution of histamine (600 mg L^{-1}) was prepared in EtOH. Imidazole (30 mg L^{-1}) was prepared in deionized water. The calibration solutions of histamine were prepared by diluting the stock solution with EtOH, and were diluted in the ratio of 1:1 with IS solution before the injection for CZE (IS injected—15 mg L^{-1}).

2.2. Preparation of the sample and the CE system

Six samples of tuna fish were purchased at the local market: three samples of canned fish, one sample of fresh fish, one sample of old fish and one sample in the form of sushi. The canned samples were analyzed immediately after opening. The fresh fish and sushi samples were purchased and analyzed immediately. One sample of fish (frozen tuna fish) was analyzed after storage for three months in a freezer at -15 °C. About 10 g of the samples were weighed in a flask (volume ~150 mL) and 25 mL of EtOH was added. The samples were triturated using a processor (Philips-Wallita) for 1 min (for the formation of a homogeneous emulsion). A portion of the emulsion was inserted in a flask and centrifuged at 10,000 rpm for 3 min. An aliquot of the centrifuged solution was removed, diluted at 1:1 with the IS solution and injected in the CE instrument. Some samples were diluted with EtOH 5 or 10 times before dilution with the IS solution due to high levels of ions present in the matrix. The samples were prepared and injected in triplicate.

The experiments to optimize the separation were conducted on a CE system (7100 Capillary Electrophoresis System, Agilent Technologies, Palo Alto, United States) equipped with a diode array detector set at 210 nm, a temperature control device (set at 25 °C) and data treatment software (HP ChemStation). Samples and standards were injected hydrodynamically (50 mbar, 3 s) from the outlet capillary end (nearest to the detector) and the electrophoretic system was operated under normal polarity and constant voltage conditions of 30 kV (positive polarity on the injection side). For all experiments, a fused-silica capillary obtained from Polymicro Technologies (Phoenix, United States) measuring 32 cm (8.5 cm effective length) × 50 μm internal diameter × 375 μm outside diameter was used. The BGE was composed of HIBA 60 mmol L^{-1} and sodium hydroxide 10 mmol L^{-1} at pH 3.3 prepared in deionized water (Milli-Q, Millipore, Bedford, MA, United States) with a resistivity of 18.2 M Ω cm. Between runs, the capillary was rinsed for 30 s with the BGE.

2.3. Comparative method by LC–MS/MS

The comparative method, using the LC–MS/MS analysis, was performed on a chromatographic equipment consisting of a high-performance liquid chromatography system (Agilent Technologies, Germany). The separation was performed on a Synergi Polar-RP 80A C-18 column (150 mm, 2.0 mm ID, 4 μm particle size) purchased from Phenomenex. The runs were performed by isocratic mode using a mobile phase composed of 95% solvent A (H_2O + 0.1% formic acid) and 5% solvent B (95:5 acetonitrile/H_2O). The flow rate was set at 200 μL min^{-1}. In all instances, the injection volume was 5.0 μL. The column temperature was set at 30 °C. The LC system was coupled with a mass spectrometer system consisting of a hybrid triplequadrupole/linear ion trap mass spectrometer Q Trap 3200 (Applied Biosystems/MDS Sciex, Concord, Canada). The Analyst software version 1.5.1 was used for the LC–MS/MS system control and data analysis. The mass spectrometry was tuned in to the negative and positive modes by the infusion of polypropylene glycol solution. The experiments were performed using the TurboIonSprayTM source (electrospray-ESI) in positive ion mode. The capillary needle was maintained at 4500 V. The MS/MS parameters were curtain gas, 10 psi; temperature, 450 °C; gas 1, 45 psi; gas 2, 45 psi; CAD gas, medium. Other parameters of the mass spectrometer for the cone and collision energy were precursor ion (mz 112.1), fragment quantitative transition (68.1), declustering potential (21), entrance potential (7), collision cell entrance potential (10), collision energy (27 and 19), and collision cell exit potential (4). The histamine was monitored and quantified using multiple reaction monitoring (MRM). The optimization of the mass spectrometer was performed by the direct infusion of an aqueous solution containing the analyte investigated. The tuna fish samples injected in LC–MS/MS needed clean-up before the injections. An aliquot of the centrifuged solution (same solution of the sample centrifuged prepared for CZE–UV analysis) was removed and diluted with a solution of trifluoroacetic acid (final concentration 10 mmol L^{-1}) and deionized water. After the dilution, 1.0 mL of the sample was inserted in a flask containing 0.25 mL of chloroform. This mixture was agitated and centrifuged, and the aliquot of the supernatant was reserved for LC–MS/MS analysis. The same procedure (partition with chloroform) was performed with the standards of the analytical curve.

3. Results and discussion

3.1. Choosing BGE components and internal standard

The chemical structure of histamine (Fig. 1-a) shows an imidazole ring capable of absorbing UV radiation, allowing the direct detection of the analyte in the CE instrument with a UV detector. Another structural characteristic of histamine in relation to acid–base dissociation constants is the existence of two values of pK_a, 6.0 and 9.8 [20], which confers to this molecule the ability to contain two positive charges, depending on the pH of the medium, as shown

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