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Simultaneous Determination of Propionate and Sorbate in Bread by Capillary Electrophoresis with Capacitively Coupled Contactless Conductivity Detection.

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Abstract

A method using capillary electrophoresis with capacitively coupled contactless conductivity detection was proposed for determination of preservatives in bread. Sample preparation required only an ultrasonic-assisted aqueous extraction of the analytes. The proposed method demonstrated to be simple, fast, and reliable for quantification of propionate and sorbate in commercial samples of bread.

Key words: *Preservatives, eletrochemical detection, food analysis.*

Introduction

Calcium propionate and sodium sorbate are food preservatives widely used in breads as antimicrobial agents. The Brazilian Health Regulatory Agency (ANVISA) establish that the amount of these preservatives added to bread must be lower than 0.1 % (w/w) for sorbate and propionate. Gas chromatography (GC)[1] is the most used analytical technique for determination of these preservatives in food, particularly propionate. However, the reported GC methods require a prior derivatization step of these analytes.

In this work, by the first time a simple and fast method for determination of propionate and sorbate using capillary electrophoresis (CE) with capacitively coupled contactless conductivity detection (C^4D) is proposed.

Results and Discussion

CE separation of the preservatives was conducted in a home-made CE-C^4D system[2], operating at 628 kHz and 2 V (peak to peak) exciting signal. A bare fused silica capillary with 80 cm total length (71.5 cm effective) and 75 µm i.d. was used. The background electrolyte (BGE) was composed by 10 mmol L^{-1} Histidine(His), 20 mmol L^{-1} 2-(N-morpholino)ethanesulfonic acid (MES), and 0.2 mmol L^{-1} Cetyltrimethyl ammonium bromide (CTAB) as an electroosmotic flow inverter. Separation voltage was -27 kV. The preservatives were extracted from 1.0000 g of bread samples, using 50 mL of water, under sonication for 10 min. The aqueous extraction solution was then diluted with water before injection in the CE-C^4D system.

The CE separation with baseline resolution between sorbate and propionate was achieved in less than 7 min (Figure 1). Calibration curves at a concentration range from 10 to 30 mg L^{-1} and 10 to 50 mg L^{-1} for sorbate and propionate, respectively, showed good determination coefficients (R^2) of 0.9992 to sorbate and 0.998 fo propionate. The compound sodium hexanoate at a concentration of 20 mg L^{-1} was used as an internal standard, because this compound was not found in the samples and do not co-migrate with the analytes. The limits of quantification (LOQ) were 2.27 and 1.55 mg L^{-1} for propionate and sorbate, respectively.

By using the CE-C^4D, commercial samples of bread were analyzed and concentrations ranging of 0.14 to 0.38 % to propionate and 0.073 to 0.091 % of sorbate were found.

Table 1 shows some analytical parameters that were evaluated.

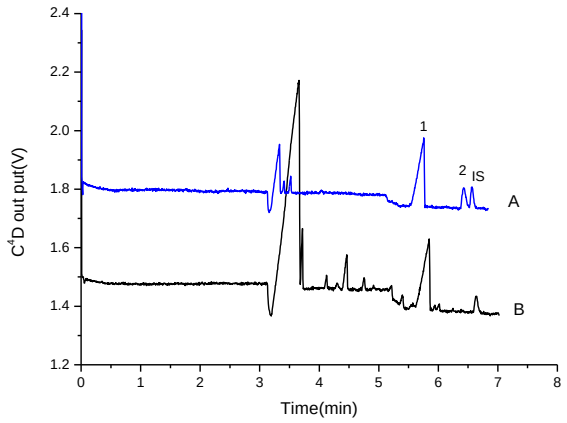

Figure 1. Electropherograms of standard solution of sorbate and propionate (A) and extract of bread (B) respectively. BGE: 10 mmol L^{-1} MES, 20 mmol L^{-1} His and 0.2 mmol L^{-1} CTAB. Separation voltage of -27 kV; hydrodynamic sample injection at 11 kPa for 5 s. C^4D detection operating at 628 kHz. Peaks: (IS) hexanoate, (1) propionate, (2) sorbate.

Table 1. Analytical parameters of the method.

	Sorbate	Propionate
Regression equation	Y=0.058x-0.025	Y=0.191x-0.098
R^2	0.9992	0.998
LOD (mg L^{-1})	0.68	0.46
LOQ (mg L^{-1})	2.27	1.55

Conclusions

The proposed CE-C^4D demonstrated to be simple, fast, and efficient for determination of propionate and sorbate in bread samples.

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