A Simple and Fast Method for Determination of Propionate and Sorbate in bread by Capillary Electrophoresis with diode array detection.

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Abstract
In this work a simple and fast method is proposed for determination of preservatives in bread using capillary electrophoresis with UV detection. The proposed method was successfully applied for quantification of sorbate and propionate in commercial samples of bread.

Key words: Preservatives, food safety, food analysis.

Introduction
Bread is a foodstuff produced by cooking a mixture of flour obtained from grains (especially wheat), water, salt, and yeast. Bread is a carbohydrate source widely consumed worldwide. Calcium propionate and sodium sorbate are food conservatives used in bakery products as antimicrobial agents. The Brazilian Health Regulatory Agency (ANVISA) establish that the amount of these preservatives added to bread has to be lower than 0.1 % (w/w) for sorbate and propionate. In this work, a simple and fast method for determination of propionate and sorbate using capillary electrophoresis with diode array detection (CE-DAD) is proposed.

CE analyses were performed in a 7100 Agilent CE system. Separations were conducted in a bare fused silica capillary of 70 cm total length (62 cm effective) and 75 μm internal diameter. The sample solutions were hydrodynamically injected into the capillary using 50 mbar pressure for 5 s. The separation voltage was -25 kV and the UV detection was performed at 235 nm to propionate and 250 nm to sorbate. The background electrolyte (BGE) was composed by 10 mmol L⁻¹ benzoic acid with pH adjusted to 5.8 with L-Histidine. Cetyltrimethyl ammonium bromide (CTAB) was added (0.2 mmol L⁻¹) to the BGE as an electroosmotic flow inverter. Sodium acetate was added to all samples and standards solutions as an internal standard. Commercial samples of bread were acquired in the local market (Campinas – SP-Brazil). A mass of 1.000 g of bread sample was weighted in an erlenmeyer and 50 mL of ultra-pure water was added. The suspension was sonicated for 10 min. The aqueous extract were filtered through a 0.22 μm PVDF membrane filter and diluted 10-fold before the injection in the CE system. The separation of the preservatives with baseline resolution was achieved in less than 6 minutes (Fig.1). The Table 1 shows some important analytical parameters of the proposed method. Calibration curves were obtained by the injection of aqueous standard solutions of the analytes with concentrations ranging from 2 to 10 mg L⁻¹ for sorbate and 20 to 100 mg L⁻¹ for propionate. The linearity of the method was evaluated and the coefficients of determination (R²) were higher than 0.99 for both preservatives. Sodium acetate demonstrated to be a suitable internal standard.

Results and Discussion
The linearity of the method was evaluated and the coefficients of determination (R²) were higher than 0.99 for both preservatives. Sodium acetate demonstrated to be a suitable internal standard.

Table 1. Analytical parameters of the proposed method.

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<tr>
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<th>Sorbate</th>
<th>Propionate</th>
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<tr>
<td>Intercept</td>
<td>0.12827</td>
<td>0.5143</td>
</tr>
<tr>
<td>Slope</td>
<td>0.24412</td>
<td>0.08309</td>
</tr>
<tr>
<td>R²</td>
<td>0.997</td>
<td>0.998</td>
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<tr>
<td>LOD (mg L⁻¹)</td>
<td>0.12</td>
<td>0.38</td>
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<tr>
<td>LOQ (mg L⁻¹)</td>
<td>3.3</td>
<td>1.26</td>
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</tbody>
</table>

By using the proposed method, concentrations of 0.03 to 0.075 % of sorbate and 0.19 to 0.34 % of propionate were determined in commercial samples of bread.

Conclusions
The CE-DAD method demonstrated to be fast, simple, and reliable for determination of propionate and sorbate in breads.

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