

Development of a Capillary Electrophoresis Method for the Monitoring of Red Seaweed Galactans-Derivative Oligosaccharides Production

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Red seaweed galactan-derivative oligosaccharides have become important starting materials in the preparation of biologically relevant glycosides as well as in stereoselective synthesis. In this way, detailed studies of the available hydrolytic processes for the production of this kind of oligosaccharide are necessary. Here we developed and validated an efficient analytical capillary electrophoresis (CE) method for the monitoring of oligosaccharide alditols production under partial reductive hydrolysis (PRH). For this purpose, previously isolated *kappa*-carrageenan di-, tetra- and hexasaccharide alditols were utilized as external standards with erytritol as internal standard. The method development led us to utilize the following conditions for its validation: pH 9 borate buffer (10 mM), indirect UV detection with p-toluenesulfonic acid (5 mM) as detectable co-ion at 214 nm, 20 kV (normal polarity) at 45 °C with all analytes being separated within 5 min. Method validation parameters were determined revealing good peak area intra- and inter-day repeatability. Analytical curves for each standard were linear in the 1.0000–0.0652 mg.mL⁻¹ interval. PRH of *kappa*-carrageenan was monitored by taking triplicate aliquots at 0, 0.25, 0.5, 0.75, 1, 2, 4, 8 and 16 h. Each aliquot was evaporated to dryness (removal of TFA) and treated with cationic resin (removal of 4-MMB). The sample processing recoveries were higher than 90%. CE analysis of each aliquot allowed the determination of the best conditions for the preparation of oligosaccharides of different degrees of polymerization. Based on these results, this methodology was considered suitable for the monitoring of sulfated polysaccharide hydrolysis.

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