

Title:**Seaweed Polysaccharides Studied by Solution- and Solid-State NMR and Chemometrics****Authors & affiliations:**

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Solution-state ¹H nuclear magnetic resonance (NMR) spectroscopy is a reference analytical technique in research and industry for the qualitative and quantitative analysis of alginates^{1,2} - linear polysaccharides extracted from brown seaweed (*Phaeophyceae*) consisting of a mixture of mannuronic acid (M) and guluronic acid (G) units. The technique is especially used for the determination of alginate monomer composition (M/G-ratio), which to a large degree determines the functionality of the alginates in numerous applications within the food and pharmaceutical industries. However, at the concentrations convenient for ¹H solution-state NMR analysis, alginate solutions are too viscous to provide well-resolved spectra. Thus, the viscosity must be reduced and this is typically done by a relatively time-consuming and labour intensive acid hydrolysis. Moreover, the spectra obtained by solution-state NMR can be affected by the presence of residual amounts of cationic and paramagnetic ions and thus introduce artefacts that might lead to wrong conclusions about the sample composition.³ For this reason we have investigated the potential of using the ¹H high resolution (HR) magic angle spinning (MAS) NMR of alginates suspended in D₂O and ¹³C cross-polarisation (CP) MAS NMR of powders as alternative methods for determination of alginate composition. These methods require minimum sample preparation and are not affected by the presence of different amounts of cations to the same extend as the solution-state NMR method. A total of 42 different sodium alginate samples were analysed and it was found that the M/G ratio derived from the ¹H HR MAS and ¹³C CP/MAS spectra were comparable to the M/G ratio calculated from the solution-state spectra. However, the M/G ratios of samples with high calcium ion content were closer to the reference value when using the ¹³C CP/MAS spectra for the prediction. It can be concluded that ¹³C CP/MAS NMR in combination with multivariate data analysis is an excellent candidate for accurate rapid analysis of the M/G ratio of alginates.

References:

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