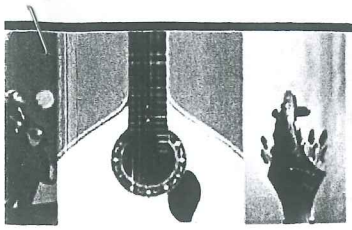

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6TH INTERNATIONAL MEETING OF THE PORTUGUESE CARBOHYDRATE GROUP

3RD IBERIAN CARBOHYDRATE MEETING

11-15 SEPTEMBER 2005

HOTEL D. LUIS - COIMBRA - PORTUGAL

Headspace solid phase microextraction-gas chromatography as a simple and clean methodology for determination of methylesterification and acetylation of polysaccharides

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A simple and solvent-free methodology was developed for the simultaneous determination of methanol (degree of methylesterification - DM) and acetic acid (degree of acetylation - DA) released by saponification of polysaccharides. A headspace solid-phase microextraction (HS-SPME) methodology with a DVB/Carboxen/PDMS fibre using external calibration curves was used for the quantification of both analytes. Methanol and acetic acid were separated by gas chromatography and detected using a flame ionization detector (GC-FID). In order to evaluate the effect of a possible interference of one analyte on the amount estimated of the other, calibration curves of each compound were also constructed in the presence of different concentrations of the other analyte. A linear relationship between the concentration of methanol and acetic acid and their GC peak area was observed (R^2= 0.987 for methanol and 0.988 for acetic acid) with a reproducibility bellow 10% for both analytes, expressed as a percentage of the mean.

The analyses of different pectic polysaccharides and cell wall extracts (DM 40-85% and DA 5-20%) were compared with the DM and DA determined by GC-FID direct injection of the samples after saponification and internal standard addition [2]. Therefore, HS-SPME-GC-FID revealed to be a clean and reliable methodology for the determination of DM and DA of polysaccharides. This method is simpler than the isotope internal calibration proposed for the use of HS-SPME-GC-MS [3] and automatic HS-GC [4].

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