Quality assurance methods for Agave sugar syrups

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Abstract:
As with all sugar rich products there is always a risk that they will be extended with sugars from a cheaper source. This poster describes some of the work that has been conducted by Eurofins on procedures to help control the quality of agave syrups.

Introduction
The use of Agave plants, Agave tequilana & Agave salmiana, as a sugar source for the production of tequila and mescal alcoholic beverages has been carried out for years. However, due to the syrups low glycemic index (GI), and the bad publicity that surrounds high fructose corn syrups (HFCS), these products have recently found another outlet as sugar substitutes.

Inulin, the agave storage carbohydrate (a polyfructan), is hydrolysed almost entirely during the extraction process to fructose, glucose and low levels of sucrose and oligofructoses[1]. Although there is a Mexican standard for Agave syrups[2], which lays down some quality parameters, due to the large price differential between agave syrups ($3800/T) and HFCS ($490/T) for instance, this has not stopped unscrupulous suppliers blending their products with cheaper sugar syrups.

This poster will review some of the data that we have collected over the last few years working with these syrups.

Methodologies:
Agave Syrup Samples: 130 Mexican agave syrups were taken for sugar and oligosaccharide analysis. Originally 51 agave syrups[3] were taken for NMR analysis, but now the database holds in excess of 100 products. 54 other syrups were judged to be adulterated from their oligosaccharide profiles. All reagents were of an appropriate analytical grade.

A QA sample of agave syrup is used with each batch of samples and the data monitored to ensure the reliability of the analytical data.

Sugar and polyol analysis by HPAEC-PAD[4]
Sample preparation: Agave syrups are diluted in HPLC grade water (0.5g/250ml) for the analysis of glucose, sucrose, mannotol and inositol. This solution is further diluted 1:50 for the analysis of fructose. Solutions are filtered (0.45µm membrane) prior to analysis.
HPAEC: Waters Chromatography e2695 pump and 2465 electrochemical detector.
Column: Dionex MA1 (250 x 4.6 mm) with guard (MA1G).
Standard stock solution: Solution contains, fructose (2000 mg/l), glucose (400 mg/l), sucrose (25 mg/l), inositol (40 mg/l) and mannotol (40 mg/l). This is then appropriately diluted to a 4 point standard curve for each analyte.
Injection volume: 10 µl
Solvent A: 0.5M NaOH* in HPLC grade water
Solvent B: 2.0M NaOH* in HPLC grade water
Flow rate: 0.5 ml using 100% A, at the end of data collection period (38mins) the column is washed with solvent B prior to re-equilibration in preparation for the next sample’s analysis.
Total run time: 60 mins
Temperature: Ambient
*Solvents A & B are supplied by Fisher Scientific. However, care has to be taken to inhibit CO2 dissolving in the liquid. NOTE: Solid NaOH should not be used to prepare eluants as it absorbs carbon dioxide from the atmosphere, which is a powerful ‘pusher’ in this procedure and will lead to shifting retention times.

13C-SNIF-NMR[6]
Sample preparation: Agave syrups are diluted to 10 Brix prior to analysis. An aliquot (50 µl) of this solution is freeze dried prior to derivatisation using Tri-Sil TP (500 µl) with heating for 30 mins at 75°C (ca 170°F).
Freeze drier: Thermo Scientific Savent SPD 1010 Speedvac concentrator.
GC: Agilent 6890 with FID. Column DBS (30M, i.d. 0.25 mm, film 0.25 µm)
GC conditions: as defined in references [4 & 5].

Discussion (cont):
Even if the individual sugars are isolated and their δ13C values measured it is our contention that the values are too close to provide a reliable and sensitive method of detection for added syrups derived from a C4 plant to agave.

Conclusions
This means that the oligosaccharide profile and the 13C-SNIF method offer the best approaches to control the authenticity of this type of syrup. The former method is relatively quick and easy to apply and only requires the normal equipment that would be found in most labs. It will detect the addition of syrups derived from starch (high fructose or not) and also cane inverters at low levels. The isotopic method provides an additional “high tech” approach to assess the product and will detect the presence of syrups that have been treated in such a way to remove the disaccharide marker peaks.

References: