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Application News

LCMS-2050 High Performance Liquid Chromatograph Mass Spectrometer

Using a Single Quadrupole Mass Spectrometer to Profile Plant-Derived Oligosaccharides and Polysaccharides

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User Benefits

- A single quadrupole LC/MS system can be used for the simultaneous analysis of oligosaccharides and polysaccharides with up to 40 monosaccharide units.
- The wide mass range of the LCMS-2050 system permits comprehensive analysis of oligosaccharides and polysaccharides as well as profiling based on degree of polymerization.

■ Introduction

Food metabolomics is the application of metabolomic techniques in food science. In recent years, food metabolomics has started to be put to a variety of uses, including food quality assessment, predicting food quality, improving manufacturing and storage processes, and evaluating functional properties. Although food contains a huge number of metabolites, many of the metabolites associated with flavor, quality, and functional properties have been identified by prior research. Because of this, food metabolomics normally targets predetermined components of interest. Ignoring unimportant components and performing an exhaustive analysis of these important components is an efficient approach for achieving useful results. Application News 01-00420-JP describes a food metabolomics case study in which a single quadrupole LC/MS system was used to profile the oligosaccharides and polysaccharides present in alcoholic beverages. Oligosaccharides are not only a source of nutrition and flavor, but are also known to increase beneficial bacteria and improve the intestinal environment.

This Application News describes an example comprehensive analysis and profiling of plant-derived oligosaccharides and polysaccharides using the same analytical conditions as Application News 01-00420-JP.

■ Samples and Sample Preparation

Samples were prepared from commercially available isomaltooligosaccharide syrup, fructooligosaccharide syrup, and yacon powder. Samples of the oligosaccharide syrups were prepared by diluting by a factor of 200 with ultrapure water. Yacon powder samples were prepared by the following method. 928 mL of ultrapure water was added to 9.28 mg of yacon powder, mixed with a vortex mixer (1 min), subjected to ultrasonic extraction (10 min), then separated by centrifugation (14,000 rpm, 10 min, 4 °C). Protein was eliminated from the sample by adding 800 µL of acetonitrile to 200 µL of supernatant, mixing with a vortex mixer (1 min), then separating by centrifugation (14,000 rpm, 10 min, 4 °C). 100 µL of ultrapure water was then added to 100 µL of deproteinated supernatant and used for analysis.

■ Analytical Conditions

Analysis was performed using the Nexera™ XR HPLC system and the LCMS-2050 single quadrupole mass spectrometer (Fig. 1). The LCMS-2050 single quadrupole mass spectrometer is compact but affords excellent ease of use and performance. The LCMS-2050 is equipped with a heated DUIS™ dual ion source that combines the benefits of electrospray ionization (ESI) and atmospheric-pressure chemical ionization (APCI), and supports a mass range of *m/z* 2 to 2,000. These features are especially useful in metabolomics, which typically analyzes metabolites with a wide range of physical properties.

HPLC and MS analytical conditions are shown in Table 1. Using the same experimental conditions as in Application News 01-00420-JP, oligosaccharides and polysaccharides composed of hexose monosaccharides were targeted and the selected ion monitoring (SIM) mode was configured for simultaneous analysis of molecules with up to 40 monosaccharide units. Due to measurable mass range and sensitivity considerations, oligosaccharides with a degree of polymerization (DP) of up to 10 were analyzed as monovalent ions, and polysaccharides with a DP of 11 or higher were analyzed as divalent or trivalent ions.

Fig. 1 Nexera™ XR and LCMS-2050

Table 1 Analytical Conditions

HPLC Conditions (Nexera XR)	
Column:	Shodex Asahipak NH2P-40 3E (250 mm × 3.0 mm I.D., 4.0 µm)
Flowrate:	0.3 mL/min
Mobile Phases:	A) 2.5 mmol/L Ammonium bicarbonate aq. B) 25 mmol/L Ammonium bicarbonate aq. / Acetonitrile = 10:90
Time Program:	70 %B (0 min)→40 %B (25 min) → 70 %B (25.01-30 min)
Mixer:	20 µL
Column Temp.:	40 °C
Injection Volume:	5 µL
MS Conditions (LCMS-2050)	
Ionization:	ESI/APCI (DUIS), Negative mode
Mode:	SIM (40 events)
Nebulizing Gas Flow:	3.0 L/min
Drying Gas Flow:	5.0 L/min
Heating Gas Flow:	7.0 L/min
Desolvation Temp.:	400 °C
DL Temp.:	150 °C

■ Analysis of Oligosaccharide Syrups and Yacon Powder

Fig. 2 shows mass chromatograms for isomaltooligosaccharide syrup and fructooligosaccharide syrup. Oligosaccharides and polysaccharides (max. 24 units) believed to be derived from isomaltooligosaccharide were detected in the isomaltooligosaccharide syrup and oligosaccharides (max. 7 units) believed to be derived from fructooligosaccharide were detected in the fructooligosaccharide syrup.

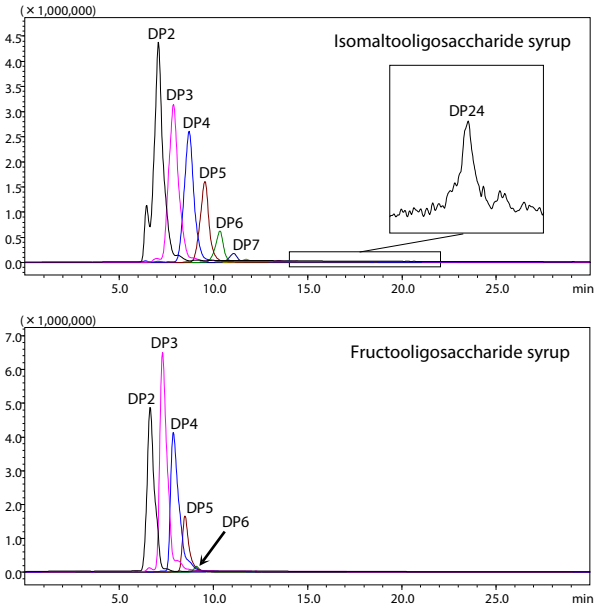

Fig. 2 Mass Chromatograms of Oligosaccharide Syrups

Yacon, which is native to the Andean highlands, contains large amounts of fructooligosaccharides in its root tuber. Fig. 3 shows a mass chromatogram for the yacon powder liquid extract. Oligosaccharides and polysaccharides (max. 17 units) believed to be derived from fructooligosaccharide were detected in the yacon powder liquid extract.

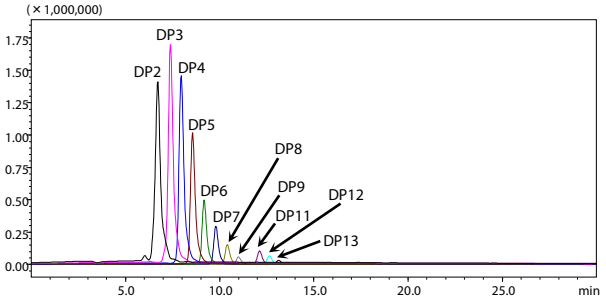

Fig. 3 Mass Chromatogram for Yacon Powder Liquid Extract

■ Proportion of Oligosaccharides and Polysaccharides by Degree of Polymerization

Monosaccharide and oligosaccharide sugars normally taste sweet, but this sweetness tends to decrease with increasing degree of polymerization. Oligosaccharides are also a well-known probiotic that increase beneficial bacteria in the gut, though their effect varies depending on the type of oligosaccharide and the degree of polymerization. Calculating the proportion of oligosaccharides (DP2 to DP10) and polysaccharides (DP11 to DP24) in each oligosaccharide syrup and the yacon powder liquid extract based on degree of polymerization revealed different proportions in each sample, as shown in Fig. 4. Specifically, yacon powder liquid extract contained a larger proportion of large oligosaccharides with a DP of 6 or higher than either oligosaccharide syrup.

■ Conclusion

This article described using a single quadrupole LC/MS system to profile plant-derived oligosaccharides and polysaccharides. The LCMS-2050 single quadrupole mass spectrometer is compact, but its wide mass range enabled the analysis of oligosaccharides and polysaccharides with high degrees of polymerization and the profiling of oligosaccharides and polysaccharides according to their degree of polymerization. The increasing adoption of single quadrupole LC/MS systems promises greater technological advancements and product development in the food sector.

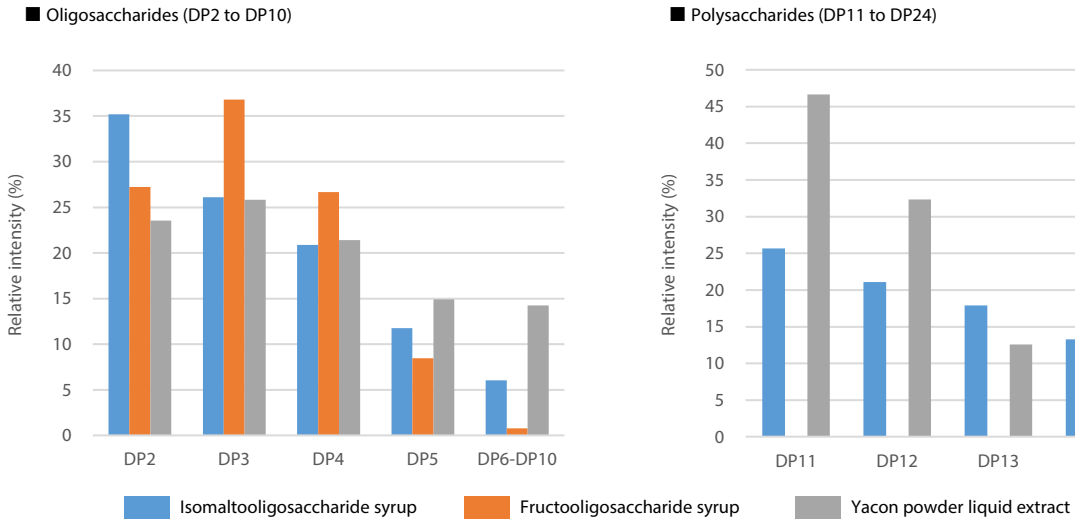

Fig. 4 Proportion of Oligosaccharides and Polysaccharides by Degree of Polymerization

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