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Application News

No.C110

Liquid Chromatography Mass Spectrometry

Application of Direct Analysis in Real Time (Part 3) Rapid Analysis of Fatty Acids in Rice Bran Using LCMS-2020

The DART (direct analysis in real time) method can be used to directly ionize samples. Application News No. C109 describes an example of rapidly analyzing the lipids in foods without pretreatment.
This article describes an example of analyzing the lipids in rice bran, which is known to involve an extremely tedious and time-consuming pretreatment process. Rice bran contains about 18 to 20 % oil, which is used as rice oil. A key characteristic of rice oil is its stability with respect to oxidation, due to its low content of linolenic acid, which is a polyunsaturated fatty acid. The rice germ portion of rice bran, which is known for its high nutritional value, contains high levels of rice oil. However, extracting it requires using hexane or other organic solvents and is time-consuming. Therefore, we evaluated the use of the DART method to analyze the lipids by simply exposing the rice bran to direct analysis, without involving solvent extraction or any other pretreatment process.

Analytical Conditions for Rice Bran

A rice bran containing known fatty acids, mostly listed in Table 1, was used for measurements.

Fig. 1 Rice Bran Used for Measurements

Table 1 Primary Fatty Acids in the Rice Bran Used for Analysis

Myristic acid	0.3 %
Linolenic acid	1.1 %
Palmitic acid	17 %
Linoleic acid	33.4 %
Oleic acid	44 %
Stearic acid	1.7 %

A DART-SVP ion source (from IonSense Inc., in MA, USA) and an LCMS-2020 single quadrupole mass spectrometer were used for analysis. Due to its ultra fast scan speed capability up to 15000 u/sec and ultra fast polarity switching time of 15 msec, the LCMS-2020 is able to perform multiple scans within the 50 to 1500 *m/z* range using both positive and negative polarity modes, all within one second. Using this functionality, we were able to simultaneously detect spectra for both triglycerides (detected using the positive ion mode) and fatty acids (detected using the negative ion mode).

Table 2 Analytical Conditions

DART Heater Temperature	: 200, 300, 400 °C
Scan Type	: *m/z* 50 – 1500 (Positive / Negative)
Neburizing Gas Flow	: 1.5 L/min.
Drying Gas Flow	: 5.0 L/min.
DL Temperature	: 250 °C
Block Heater Temperature	: 400 °C

Analysis of Lipids in Rice Bran

A mass spectrum of rice bran is shown in Fig. 2. At a DART heater temperature of 200 ˚C, peaks were detected for linoleic acid and other fatty acid in the negative mode mass spectrum. An enlargement of the *m/z* range from 170 to 320 is shown in Fig. 3. It shows that oleic acid, linoleic acid, and then palmitic acid are detected based on prominent peaks. A peak for a sugar is also shown at *m/z* 269.

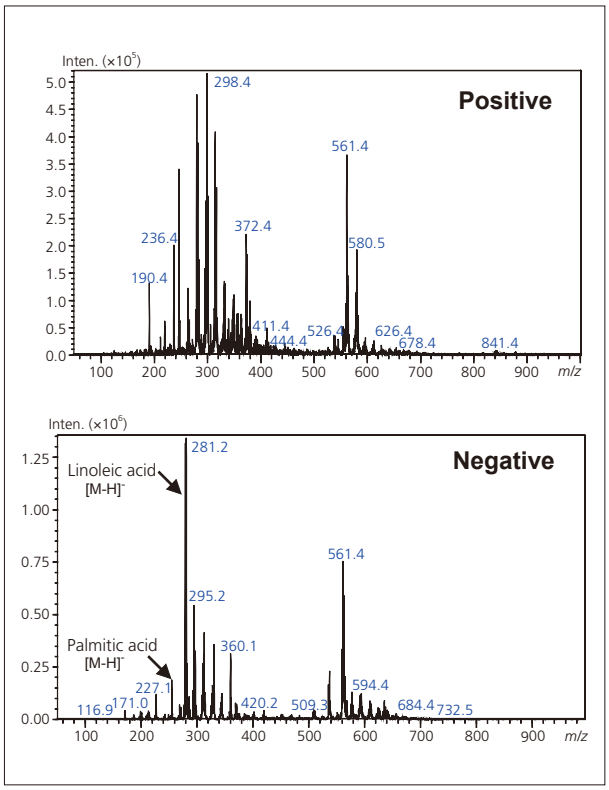

Fig. 2 Mass Spectra of Rice Bran (200 ˚C DART heating temperature)

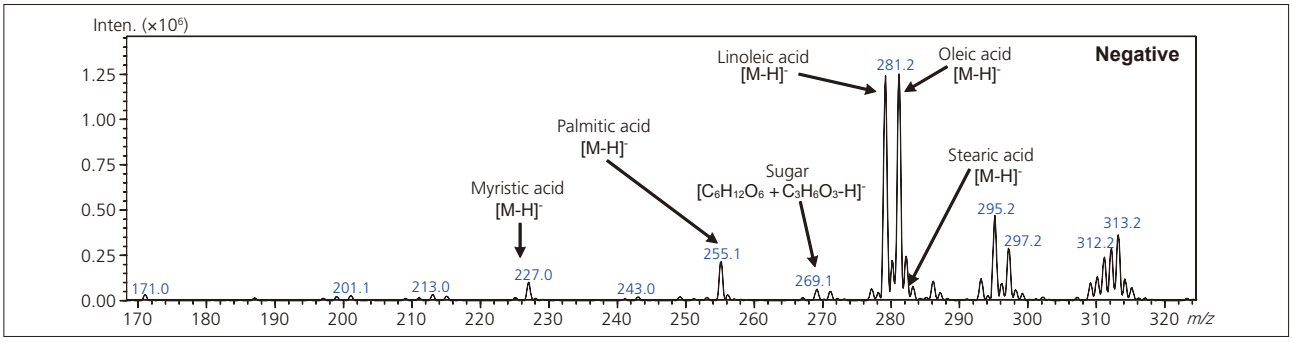

Fig. 3 Mass Spectra of Rice Bran (200 ˚C DART heating temperature)

MS spectra for a heating temperature of 400 ˚C are shown in Fig. 4. This shows peaks for lipid such as diglycerides, that were not visible in the positive-mode MS spectra for a 200 ˚C heating temperature. (The triglyceride fatty acids in the figure are displayed only in the combined state.) Enlarged views of the positive-mode results between *m/z* 540 and 650 and between 800 and 920 are shown. They show diglycerides and triglycerides, including oleic acid and linoleic acid. These spectra also show correlation with the signal intensity ratio of fatty acids detected by negative mode shown in Fig. 3.

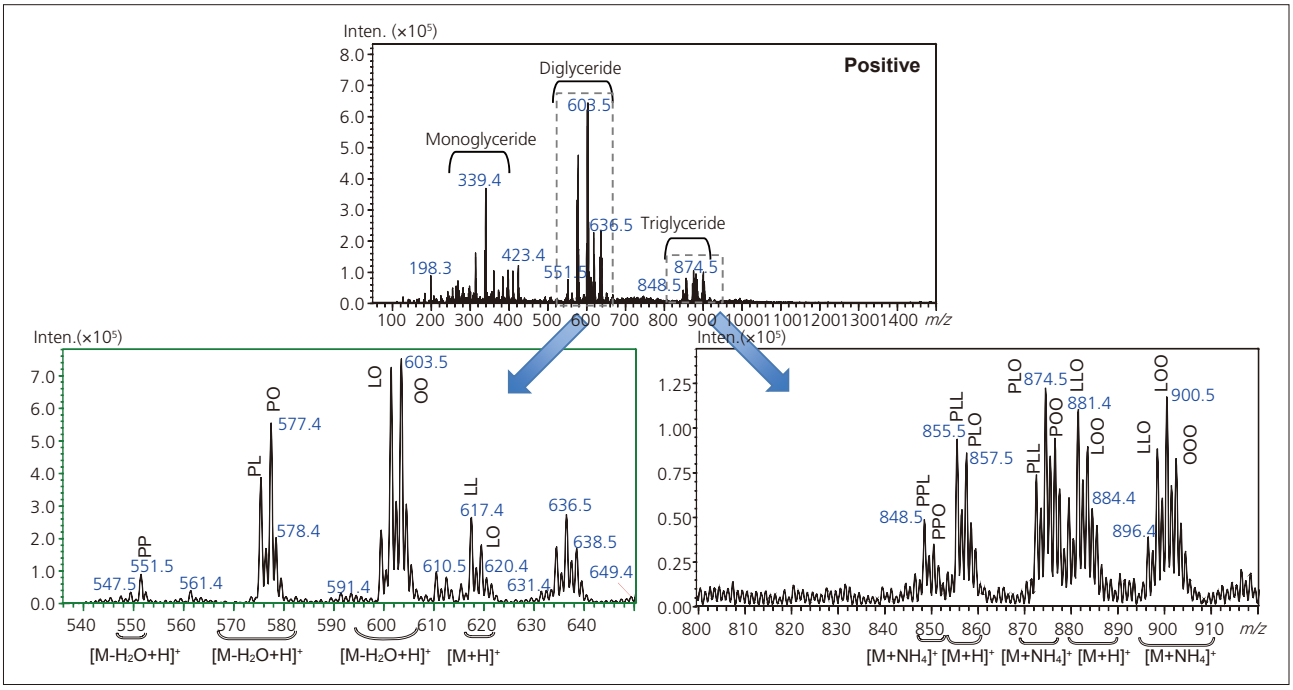

Fig. 4 Mass Spectra of Rice Bran (400 ˚C heating temperature)

To evaluate the optimal heating temperature and reproducibility for fatty acids, sugars, and lipids, samples were measured consecutively at three different heating temperatures. Extracted ion chromatograms of linoleic acid, monosaccharide, and triglyceride (LOO) are shown in Fig. 5. This confirmed that fatty acids are detected more favorably at a lower temperature, between 200 and 300 ˚C, and sugars and triglycerides are detected more favorably at a higher temperature of 400 ˚C or higher. Reproducibility of signal intensity was also achieved.

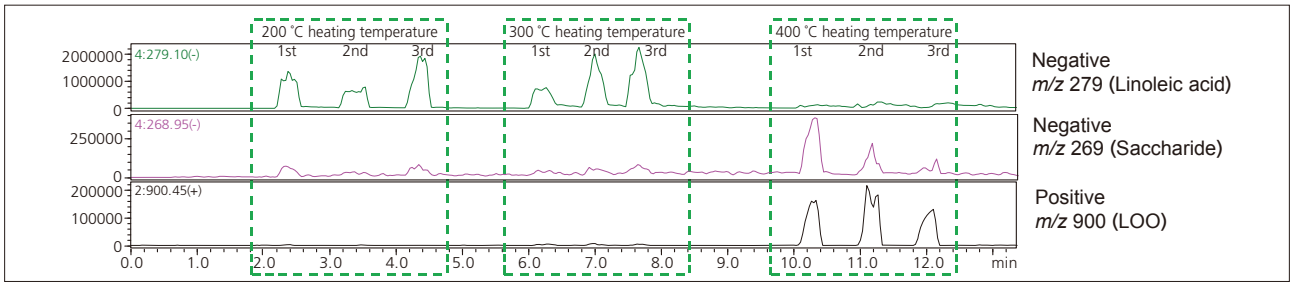

Fig. 5 Extracted Ion Chromatogram (DART heating temperature between 200 and 400 ˚C)

We wish to express our sincere gratitude to Shun Wada (professor emeritus at Tokyo University of Marine Science and Technology) and the Japan Inspection Institute of Fats & Oils for the rice bran samples, in addition to their kind cooperation in the analysis of the data.
DART is a product of IonSense Inc. (http://www.ionsense.com/).

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