

Application News

No. B72

MALDI-TOF Mass Spectrometry

Surface Analysis of PET Film Using a Benchtop MALDI-TOF Mass Spectrometer

MALDI-TOF mass spectrometers are a mass spectrometer type that is used in a wide range of fields likewise LCMS, in terms of high through-put and high sensitivity. These instruments have recently been utilized more and more for simple molecular weight measurement and profiling of synthesized products and high-molecular compounds. This is because instruments of this type have several features: singly-charged ions are generated so molecular weights can be recognized easily, the mass range is wide, and there are many solvent options because the sample is dried before measurement.

On the other hand, due to changes in social conditions in these several years, government offices, universities, and private enterprises strongly request the reduction of costs for both introduction and running of instruments used for such applications. The benchtop "MALDI-8020" MALDI-TOF mass spectrometer is a new instrument that can sufficiently meet such market needs. The noteworthy point of this instrument is that it has a shorter flight tube, which is the key feature of its small size, while retaining the performance equal to or higher than that of a conventional model.

In recent years, the usability of MS imaging using a MALDI-TOF mass spectrometer has become widely recognized and various techniques are being developed. On the other hand, needs for examining the compounds that exist on surfaces, rather than its "microscopic structure", are increasing in association with degradation and durability tests.

This article introduces an example of analyzing a PET film surface using the benchtop MALDI-TOF mass spectrometer "MALDI-8020".

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■ Benchtop MALDI-TOF MS: MALDI-8020

The MALDI-8020 (Fig. 1) is a compact-design, minimal-space linear mode-only MALDI-TOF mass spectrometer. Its performance in linear mode (positive ion) is comparable to the same mode of a conventional MALDI-TOF mass spectrometer. Equipped with a 200 Hz solid-state laser and a load-lock chamber mechanism that enables a target plate change while maintaining the degree of vacuum at the measuring position, the instrument ensures rapid measurements.

The flight tube of this instrument is shortened to 0.85 m long; however, the mass resolving power is as good as that of a conventional model. The instrument has a practical resolving power that enables isotope separation even near m/z 4000.

■ Preparation of Thermally Treated PET Film

Polyethylene terephthalate (PET) is a general-purpose engineering plastic material and is used for extensive applications.

It is generally known that oligomers in polymers are deposited in the process of molding or drawing involving heating and which cause defective lots and trouble in processes. In particular, it is known that cyclic trimer oligomers of PET are apt to move to the resin or film surface and produce deposits on it through thermal treatment.

A sheet of commercially available PET film was separated into two: one was heated at a temperature of 100 °C, and the other was left for several hours at room temperature. Fig. 2 shows optical microscopic images of these two films after the treatment. We can see that the film subjected to thermal treatment is slightly whitish. We secured this film on the stainless steel MALDI plate with conductive double-sided tape and sprayed a matrix (20 mg/mL, dithranol, 3 mg/mL Na-TFA in THF) for use as a MALDI sample.



Fig. 1 Benchtop MALDI-TOF MS: MALDI-8020

Facile Surface Measurement of PET Film

Normal MS measurement was performed for the area marked within the red line in Fig. 2 using MALDI-8020. As a result, regardless of thermal treatment, irganox1010 molecular-related ions (Na adduct) were detected at m/z 1199 (Fig. 3). In addition, ions were detected at m/z 599 only from the surface subjected to thermal treatment. This corresponds to the molecular weight of PET cyclic trimer (Na adduct).

Next, using irganox1010 and cyclic trimer signals, mapping images were obtained as shown in Fig. 4. These mapping images enable graphical representation of surface differences before and after thermal treatment.

Conclusion

We performed simple surface analysis using the benchtop MALDI-TOF mass spectrometer "MALDI-8020", and identified the differences between polymer film surfaces before and after thermal treatment.

In recent years, development of MALDI-TOF mass spectrometers has led to larger instruments with high specifications and, due to their size and initial/running costs, there has been a barrier to introducing the instruments for some more routine applications.

The MALDI-8020, the world's smallest commercially available MALDI-TOF mass spectrometer, satisfies the needs of chemical product analysis in linear mode, and its future dissemination in material analysis field is expected.

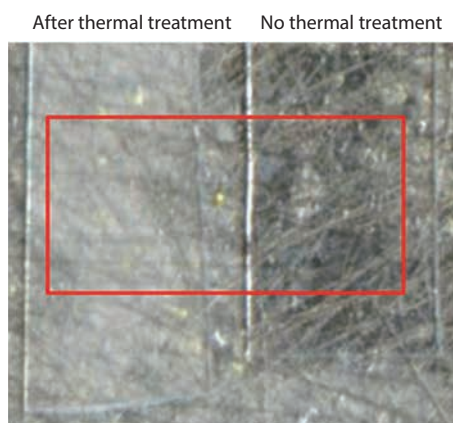


Fig. 2 Optical Microscopic Images of PET Film

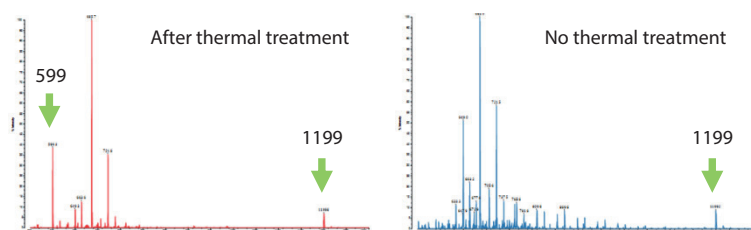


Fig. 3 Mass Spectrum of PET Film Surface

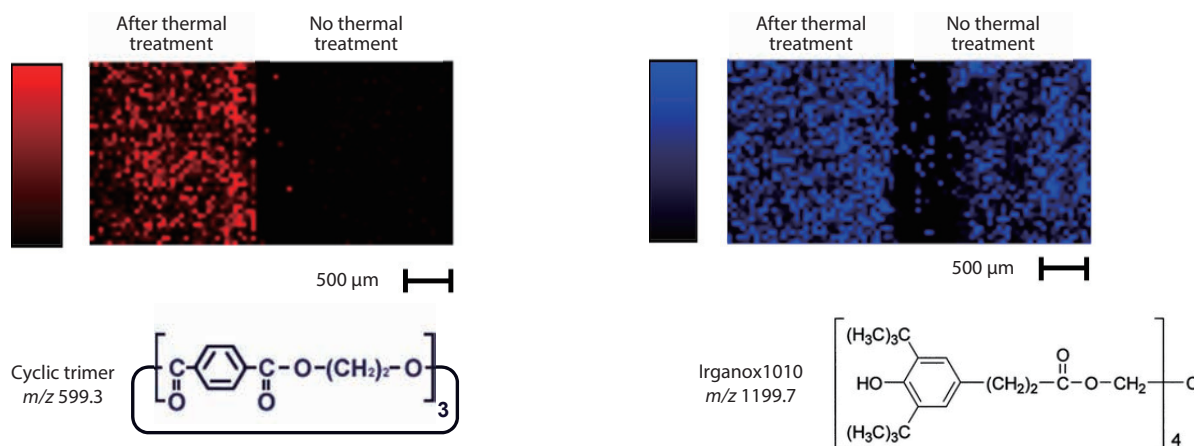


Fig. 4 Mapping Images of m/z 599 and 1199 (Fig. 2, in the red line)