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Application Data Sheet

No.94

GC-MS
Gas Chromatograph Mass Spectrometer

GC-MS Analysis of Catalytic Reaction Products of Glucose in Biomass Research

When converting biomass resources into energy or raw chemical materials, it is essential to improve the conversion yield and analyze the reaction process. This sort of analysis requires identifying volatile components; therefore, GC-MS systems, with their excellent qualitative capabilities, are useful.
This report presents results obtained by adding a solid acid catalyst to an aqueous glucose solution, heating the solution, and then directly analyzing the resulting reaction solution using GC-MS without pretreatment.
At present, in the utilization of woody biomass, the cellulose that comprises approximately 60 % to 70 % of the material can be converted to glucose. The analysis of the catalytic reaction presented in this report revealed that primarily hydroxymehylfurfural is further generated from glucose used as a raw material. Hydroxymehylfurfural is a compound with considerable added value as a raw material for pharmaceuticals and chemicals. The analysis also revealed the existence of lactic acid and other low-grade organic acid compounds.
In addition, utilizing the GCMS-QP2010 Ultra, which features a differential vacuum system, enabled acquisition of favorable results even when directly injecting the sample aqueous solution into the GC-MS.

Reaction Sequence

Fig. 1 shows how glucose is converted to hydroxymehylfurfural via a solid acid catalyst.

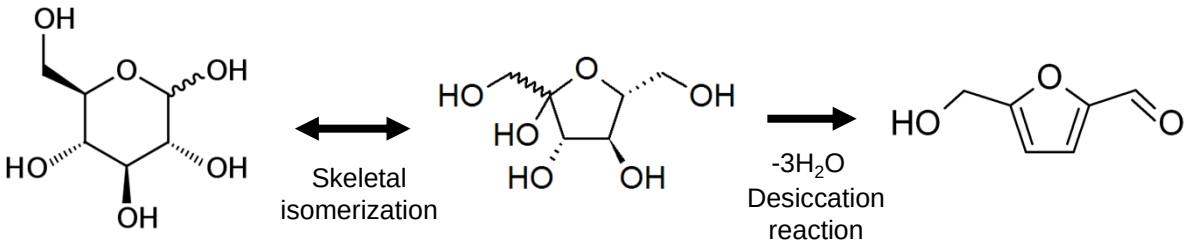

Fig. 1: Catalytic Reaction of Glucose

Analysis Conditions

Table 1: Analysis Conditions

GC-MS:	GCMS-QP2010 Ultra
Column:	Stabilwax (30 m long, 0.25 mm I.D., df = 0.25 μm)
Glass Insert:	Split insert with wool (P/N: 225-20803-01)

[GC]		[MS]	
Injection Unit Temp.:	230 °C	Interface Temp.:	250 °C
Column Oven Temp.:	50 °C (5 min) → (10 °C/min) → 250 °C (10 min)	Ion Source Temp.:	230 °C
Injection Mode:	Split (split ratio 10)	Measurement Mode:	Scan
Carrier Gas Control:	Linear velocity (50.0 cm/sec)	Scan Event Time:	0.30 sec
Injection Volume:	0.5 μL	Scan Mass Range:	*m/z* 15 to 550
		Scan Speed:	2,000 u/sec

Analysis Results

Figs. 2 and 3 show the results obtained by adding a solid acid catalyst to an aqueous glucose solution, heating the solution, and then injecting the resulting reaction solution directly into the GC-MS to measure it without pretreatment. In addition to 5-hydroxymethylfurfural, Peak 10, which is the main reaction product, formic acid, acetic acid, lactic acid, and other low-grade organic acid compounds were also confirmed. Compounds were surmised from the results of a similarity search using the NIST library.

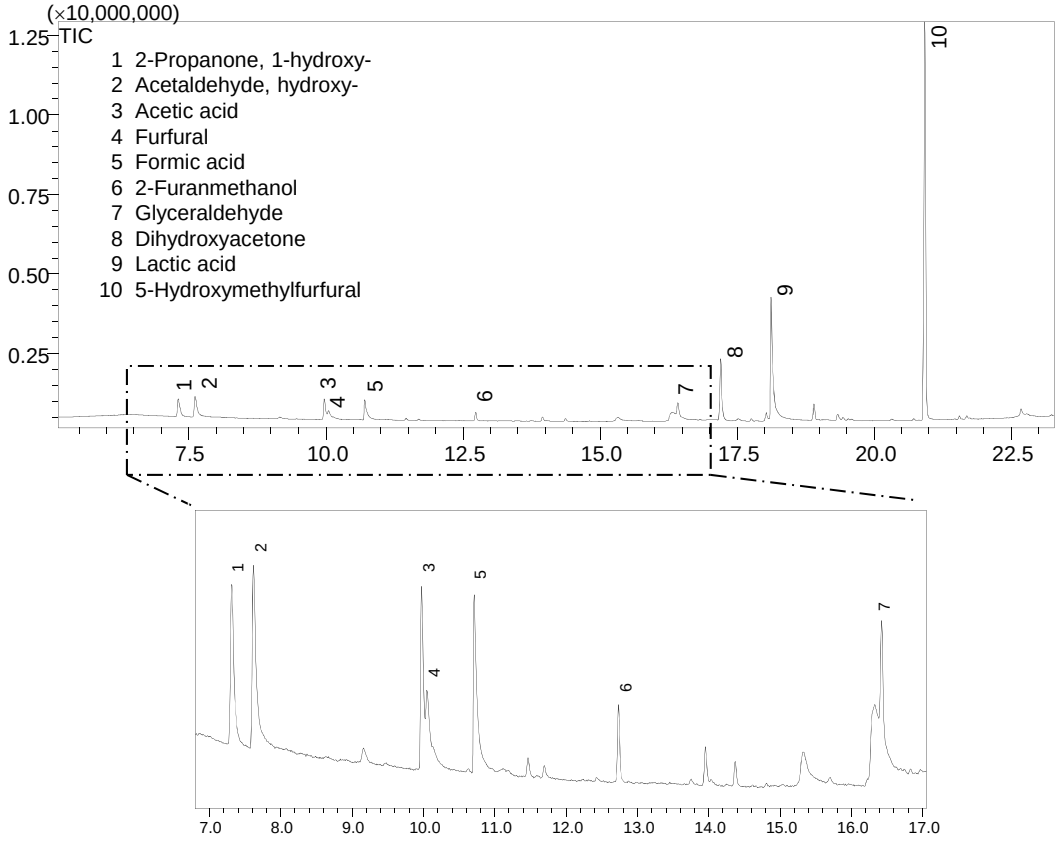

Fig. 2: Total Ion Current Chromatogram for the Reaction Solution

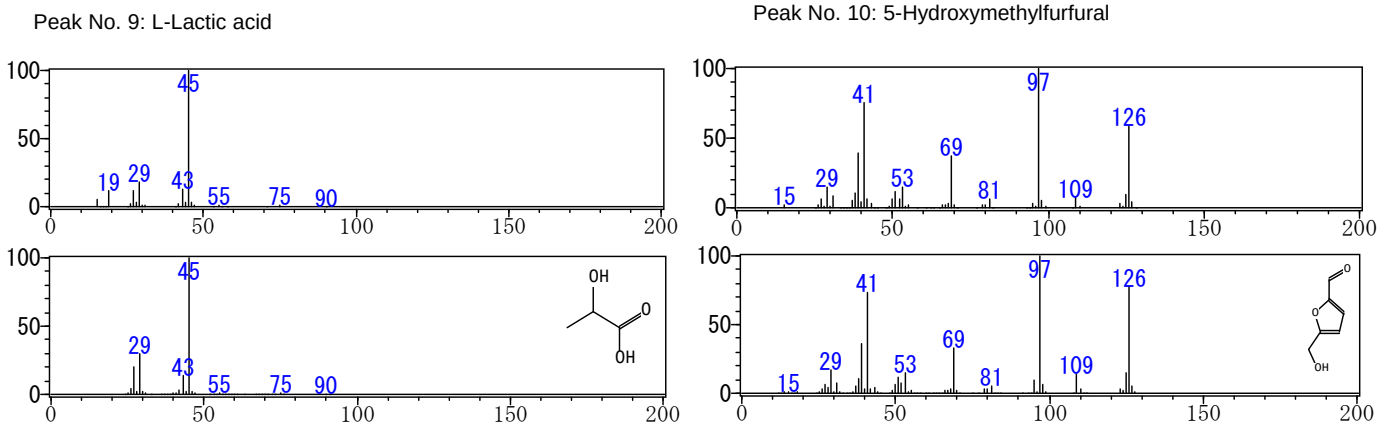

Fig. 3: Mass Spectra (Top: Mass Spectra for Peaks 9 and 10; Bottom: Library Search Results)

Note: Professor Michikazu Hara of the Materials & Structures Laboratory at the Tokyo Institute of Technology provided the sample.

First Edition: December 2013

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