Analysis of Furfurals in Glycosylated Woody Biomass with the Agilent 1260 Infinity LC system

Application Note

Biofuels and Alternative Energy

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Abstract

Although bioethanol is currently produced mainly from edible plants, research is underway into production methods based on non-edible plants, for example, wood. When wood is used as a raw material, furfural and 5-hydroxymethyl furfural (5-HMF) are produced as over-decomposition products of sugars during its glycosylation and fermentation. This is an example of analysis of furfurals in wood sugar solutions obtained by low environmental impact hydrothermal treatment and mechanochemical treatment followed by enzyme-based glycosylation. The samples were kindly provided by Mr. Shigeki Sawayama, Head of the Research Team, and Mr. Katsuji Murakami, Chief Researcher, from the Biomass Research Center of the National Institute of Advanced Industrial Science and Technology.

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The chromatograms of the reference solutions are shown in Figure 1.

Configuration

Agilent 1260 Infinity LC System

- Agilent 1260 Infinity Series Binary Pump SL (G1312B)
- Agilent 1260 Infinity Series High Performance Autosampler (G1367C)
- Agilent 1260 Infinity Series Thermostatted Column Compartment (G1316B)
- Agilent 1260 Infinity Series Diode Array Detector VL Plus (G1315C)

Analytical Conditions

Column:	Agilent ZORBAX RRHT SB-C18 3.0 mm × 50 mm,1.8 µm
Mobile phase:	0.1% formic acid/acetonitrile 3/97 (0–3 min)→ 10/90 (3–5 min)
Flow rate:	0.5 mL/min
Column temperature:	40 °C
Sample quantity:	2 µL
Detection:	Signal = 280 nm, bandwidth 4 nm, reference = 380 nm, bandwidth 80 nm

Figures 2–7 show analytical assay results of wood sugar solutions obtained using different pre-treatment methods and raw materials. The wood sugar solutions were diluted 10-fold with ultrapurified water and centrifuged, the supernatant was used for samples.

Conclusion

The amount of furfural and 5-HMF, which interfere with glycosylation and fermentation of biomass, depends on hydrothermal treatment process. The Agilent 1260 Infinity Series is suitable for the analysis in glycosylated woody biomass because of good sensitivity and good usability.

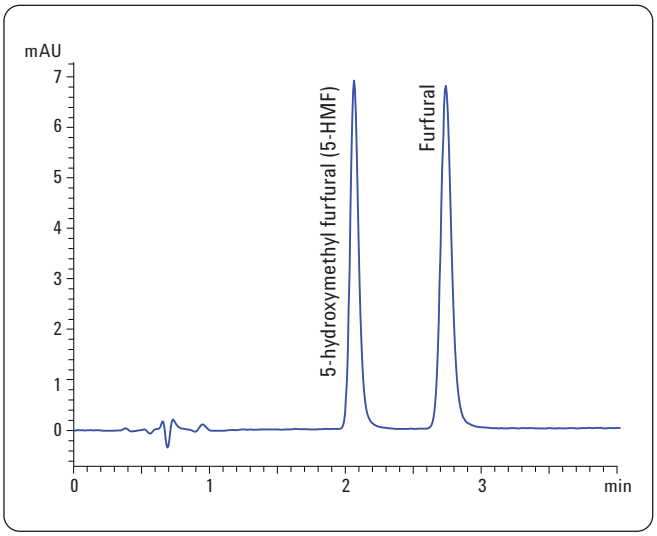

Figure 1
Chromatogram of Reference Solutions (1 mg/mL each).

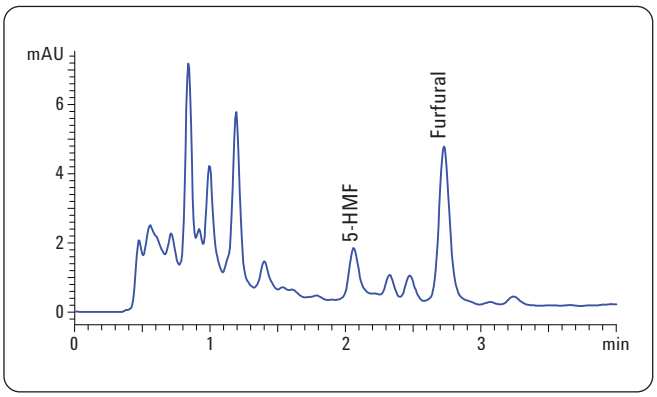

Figure 2
Eucalyptus, ball mill.

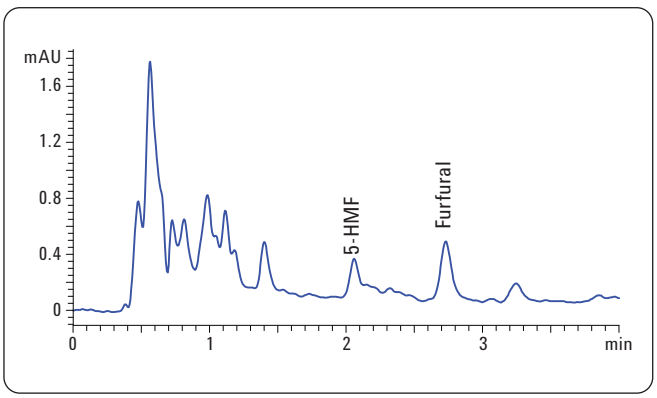

Figure 3
Bagasse, ball mill.

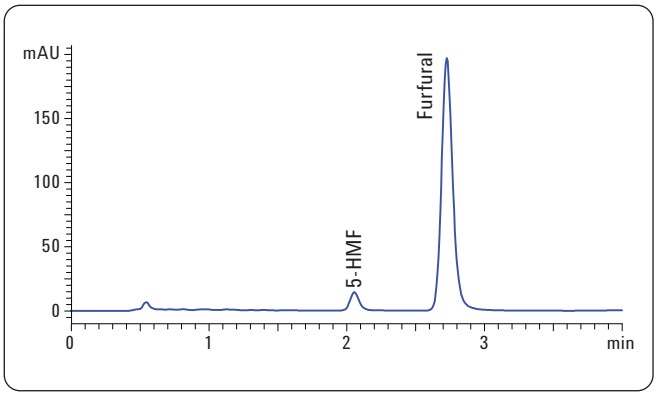

Figure 4
Bagasse, hydrothermal, 180 °C 5 min.

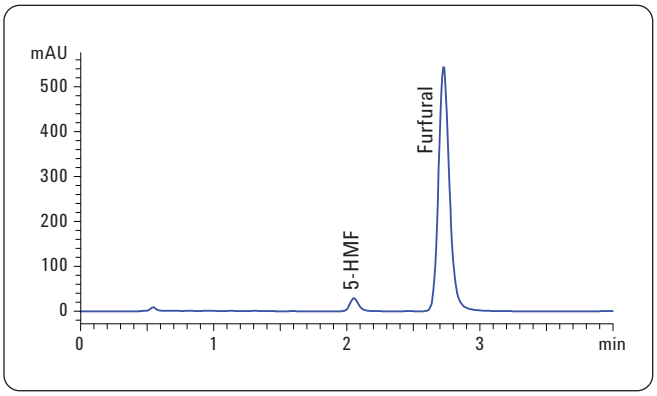

Figure 6
Bagasse, hydrothermal, 180 °C 30 min.

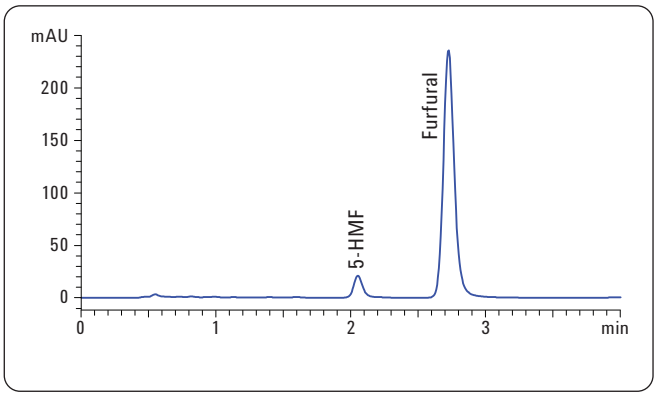

Figure 5
Bagasse, hydrothermal, 160 °C 15 min.

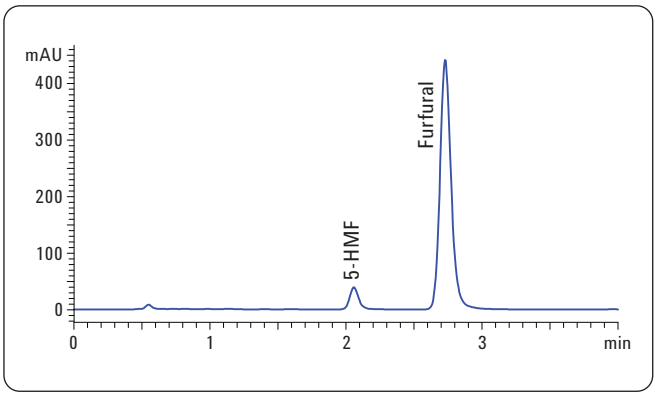

Figure 7
Bagasse, hydrothermal, 160 °C 30 min w/phosphoric acid.

Sample		5-HMF assay results [mg/L]	Furfural assay results [mg/L]
Eucalyptus	Ball mill	2.3	6.7
Bagasse	Ball mill	14.8	62.7
Bagasse	Hydrothermal treatment, 180 °C 5 min	22.6	298
Bagasse	Hydrothermal treatment, 160 °C 15 min	32.2	357
Bagasse	Hydrothermal treatment, 180 °C 30 min	45.5	826
Bagasse	Hydrothermal treatment, 160 °C 30 min w/phosphoric acid added	60.0	669

Table 1
Furfural assay results for samples (wood sugar solutions).

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