

TGA-GC-MS Measurements on Pinewood/Sapwood

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Introduction

Nowadays, biomass is more and more frequently used as an alternative to conventional energy sources. Key benefit is the "CO₂ neutrality". Wood belongs to the most important renewable raw materials.

The main components of wood are cellulose, hemicellulose and lignin.

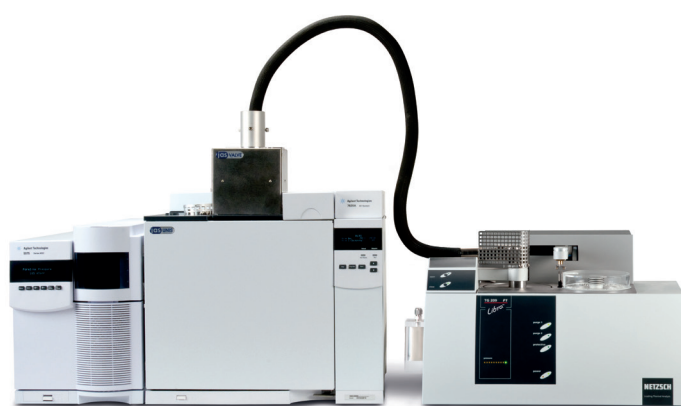


Fig. 1 TG 209 *F1* *Libra*® with GC-MS coupling

Parameters

Temperature range	RT to 500°C
Heating rate	10 K/min
Atmosphere	Helium
Flow rate	65 ml/min
Sample holder	Sample holder for corrosive gases
Crucible	Al ₂ O ₃ (85 µl)
Sample mass	6.9 mg

Tab. 1 TGA measurement parameters

Parameters	Quasi-continuous Mode	Event-controlled Mode
Column		Agilent HP-5ms
Column length		30 m
Column diameter		0.25 mm
Furnace temperature	150°C	100°C to 310°C (10 K/min)
Gas		Helium
Gas flow (split)		20 ml/min (10:1)
Column flow		2 ml/min
Valve	every 2 min	1 x per event

Tab. 2 GC-MS measurement parameters

Quasi-continuous Mode

Simultaneous coupling of the TGA to the GC-MS allows for easy correlation of the outgassing substances with temperature.

First, the cellulose components are decomposed, then the lignin components decompose.

The pyrolysis of pinewood takes place in three steps (figure 2). The first step is the evolution of the water contained. The main decomposition of wood occurs at approx 300°C.

Corresponding to the DTG curve, the main decomposition can be seen at 300°C in the total ion chromatogram.

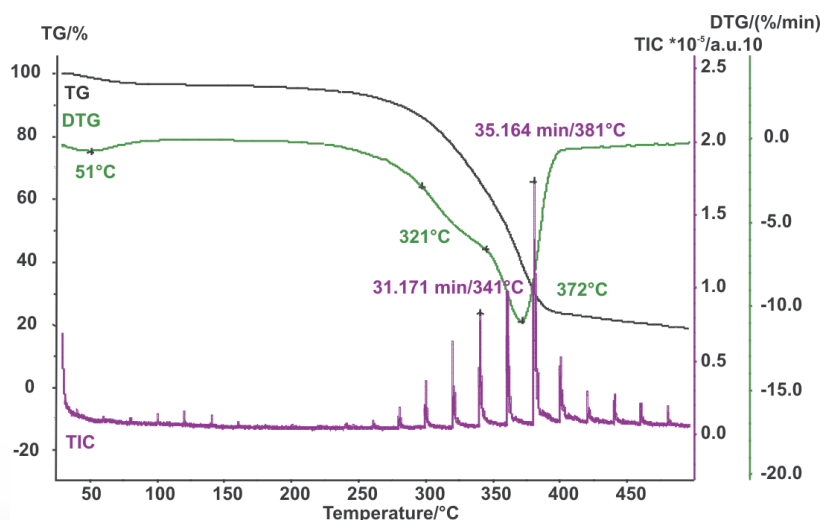


Fig. 2 Pyrolysis of pinewood: TGA (black), DTG (green) and TIC (purple) results versus temperature

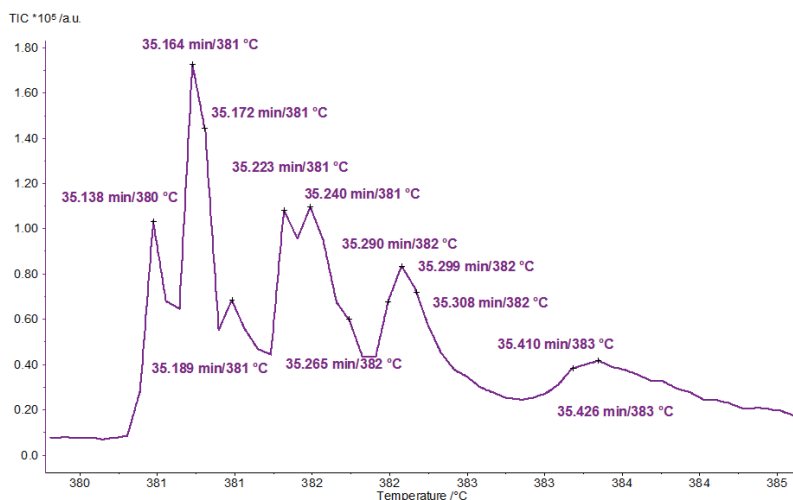


Fig. 3 Pyrolysis of pinewood: TIC results versus temperature (380°C to 385°C)

An enlarged scaling of the TIC in this range is presented in figure 3; the substances detected at the peaks are listed in table 3.

Time/min	Molecule	Molar Mass	Mass Number
35.138	Acetone	58	58
35.164	1,2,3-Thiadiazole	86	58, 86
35.172	2-Methylfuran	82	82, 81, 53
35.189	2-Methyl-Mannomethylpyranosid	178	60, 74
35.223	2-Butenal, 2-Methyl	84	55, 84
35.240	Thiophene	84	84, 58, 45
35.265	Furan, 2,3-Dihydro-5-Methyl	84	84, 55, 69
35.290	Furfural	96	96, 95
35.299	1H-Pyrazol, 1,3-Dimethyl	96	96, 81, 68, 54
35.308	2,5-Dimethylfuran	96	96, 95, 81, 53
35.409	2(5H)-Furanon	84	55, 84, 70
35.426	2H-Pyran, 3,4-Dihydro	84	55, 84, 69

Tab. 3 Detected molecules and their retention times

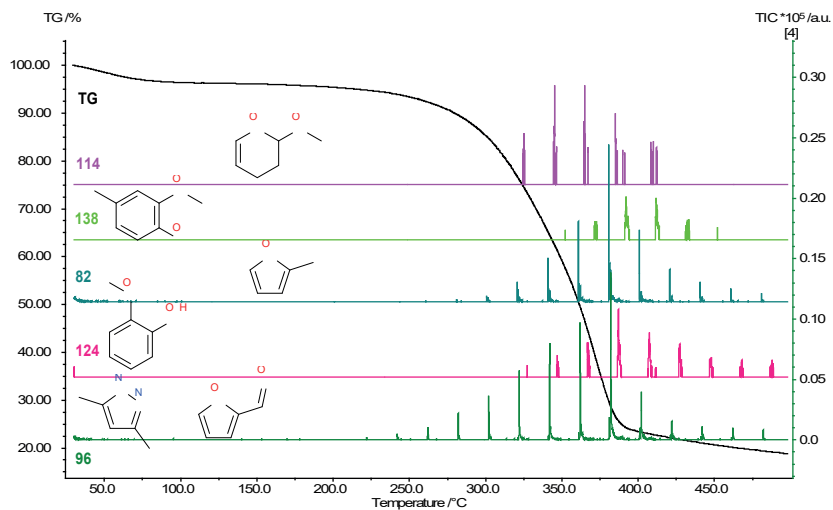


Fig. 4 TGA results (black) and ion chromatograms of mass numbers 82, 96, 114, 125 and 138

The individual mass numbers for pinewood are presented in figure 4 as a function of temperature.

event-controlled mode (figure 5). For this, individual chromatograms at certain temperatures were recorded.

Event-controlled Mode

For a more detailed evaluation of the forming substances, the TGA-GC-MS measurement was carried out in the

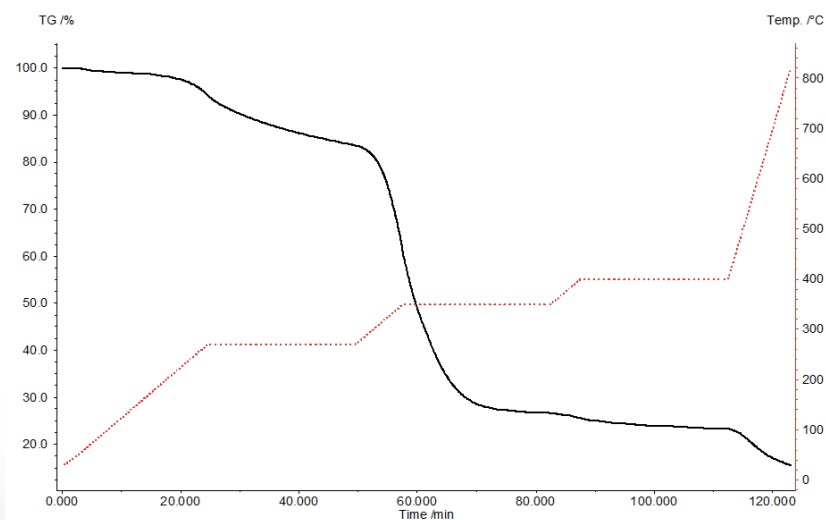


Fig.5 TGA results (black) and temperature (red) versus time

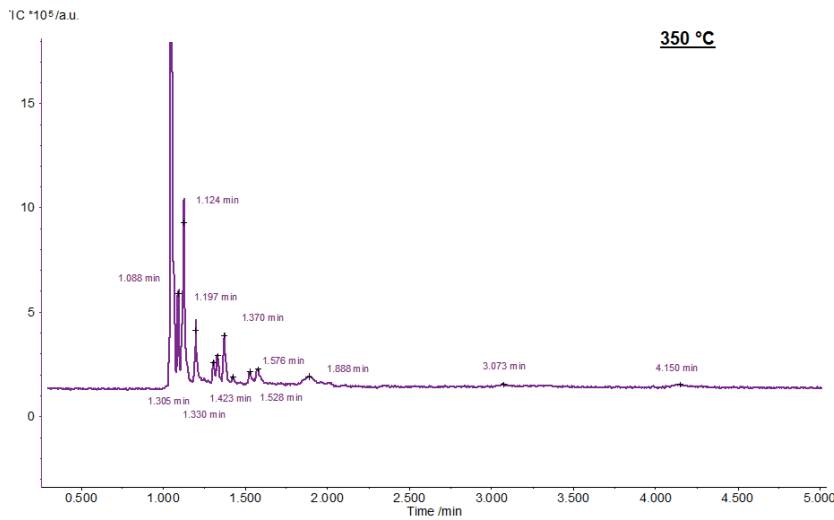


Abb. 6 TIC results versus time at 350°C

Figure 6 shows the chromatogram at 350 °C. The substances measured at the corresponding retention times are presented in table 4.

Retention time/min	Substance
1.047	CO ₂
1.088	3(2H)-Furanon, Dihydro-2-Methyl
1.124	1-Propanol
1.197	1-Hydroxy-2-Propanone
1.305	2(5H)-Furanon
1.330	Acetic Acid, Methylester
1.370	Acetone
1.424	4H-1,2,4-Tiazol, 4-Amino
1.528	Fufural
1.576	2-Furanmethanol
1.888	2(3H)-Furanon, 5-Methyl
3.073	Phenol, 2-Methoxy
4.150	Phenol, 2-Methoxy-4-Methyl

Tab. 4 Detected molecules at 350°C and their retention times