

Simultaneous determination of acid-soluble biomass-derived compounds using high performance anion exchange chromatography coupled with pulsed amperometric detection

N. Anders^{a*}, H. Humann^a, B. Langhans^a, A. C. Spieß^{a,b}

^a AVT-Enzyme Process Technology, RWTH Aachen University, Worringer Weg 1, 52074 Aachen, Germany

^b DWI – Leibniz Institute for Interactive Materials, Forckenbeckstraße 50, 52074 Aachen, Germany

Table of content:

Table S-1: HPAEC-PAD results for each measured carbohydrate and each biomass in g/L including molecular formula. The results were obtained after a two-step acid hydrolysis according to NREL/TP-510-42618 procedure.¹ The hydrolysis led to liquid solutions which were diluted tenfold and measured with the method described in HPAEC-PAD instrumentation, software and method.

Table S-2: HPAEC-PAD results for each measured uronic acid, sugar degradation product as well as the lignin model compound and each biomass in g/L including molecular formula. The results were obtained after a two-step acid hydrolysis according to NREL/TP-510-42618 procedure.¹ The hydrolysis led to liquid solutions which were diluted tenfold and measured with the method described in HPAEC-PAD instrumentation, software and method.

Table S-3: Precision of the HPAEC-PAD method. Based on a tenfold measurement of melon peel hydrolyzate, the standard deviation and the relative standard deviation were estimated. n.d. = not detectable, n.q. = not quantifiable.

Figure S-1: Superposition of chromatograms of commercially available soluble biomass standards. These chromatograms show the separation of all compound classes, alcoholic acid soluble lignin compounds, sugar degradation product, monosaccharides, oligosaccharides, uronic acids and phenolic acid soluble lignin compounds.

Figure S-2: Overlay of chromatograms from different hydrolyzates. Diverse biomasses were hydrolyzed with a two-step acid hydrolysis. The obtained liquid was analyzed using the HPAEC-PAD method described here. A and C: full chromatogram. B and D: chromatograms of the biomass derived phenols.

References:

(1) Sluiter A., Hamas B., Ruiz R., Scarlata C., Sluiter J., Templeton D. and Crocker D.: Determination of structural carbohydrates and lignin in biomass, Laboratory Analytical Procedures (LAP), Technical Report NREL/TP-510-42618, 2008

Table S-1: HPAEC-PAD results for each measured carbohydrate and each biomass in g/L including molecular formula. n.d. = not determinable

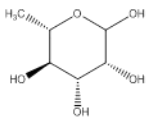
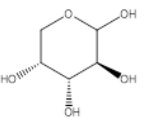
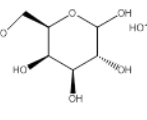
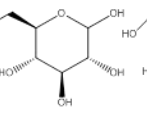
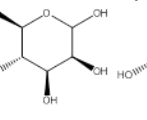
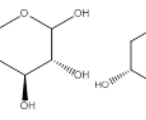
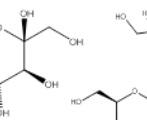
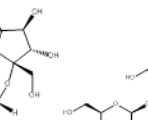
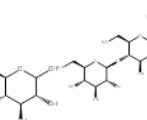
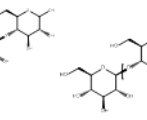
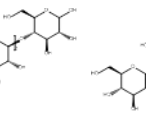
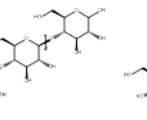
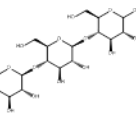
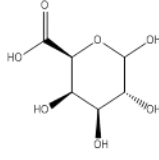
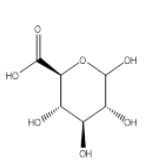
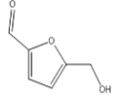
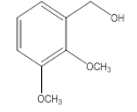
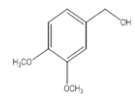
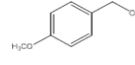
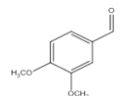
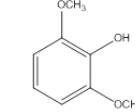
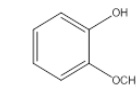
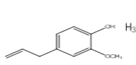
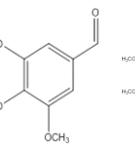
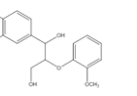
													
	Rhamnose	Arabinose	Galactose	Glucose	Mannose	Xylose	Fructose	Sucrose	Cellobiose	Cellotriose	Cellotetraose	Cellopentaose	Cellohexaose
Avicel	n.d.	n.d.	n.d.	2.570	0.060	0.054	n.d.	n.d.	0.034	n.d.	n.d.	n.d.	n.d.
Banana peel	n.d.	0.093	0.055	0.416	0.063	0.064	0.015	n.d.	0.008	0.003	n.d.	n.d.	n.d.
Barley awn	n.d.	0.099	n.d.	0.900	n.d.	0.598	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
Barley straw	n.d.	0.080	n.d.	0.958	n.d.	0.461	n.d.	n.d.	0.001	n.d.	n.d.	n.d.	n.d.
Coconut peel	0.007	0.016	n.d.	0.300	n.d.	n.d.	n.d.	n.d.	0.001	0.001	n.d.	n.d.	0.003
Honey dew melon peel	0.017	0.052	0.082	0.243	0.008	0.024	0.013	n.d.	0.001	0.001	n.d.	0.001	n.d.
Lawn grass	n.d.	0.117	0.061	0.658	n.d.	0.302	n.d.	n.d.	0.014	0.003	n.d.	n.d.	n.d.
Lignin, alkali	n.d.	n.d.	0.015	n.d.	n.d.	0.088	n.d.	n.d.	0.001	n.d.	n.d.	n.d.	n.d.
Lime peel	0.014	0.316	0.098	0.441	0.056	0.058	n.d.	n.d.	0.003	0.002	n.d.	n.d.	n.d.
News paper	n.d.	0.010	n.d.	1.588	0.212	0.192	n.d.	n.d.	0.003	n.d.	n.d.	n.d.	n.d.
orange peel	0.039	0.384	0.242	0.290	0.100	0.067	0.031	n.d.	0.015	0.001	n.d.	0.002	0.001
Organocat spruce wood	0.003	0.067	0.061	0.440	0.595	0.179	n.d.	n.d.	0.003	0.001	n.d.	0.001	n.d.
Organosolv spruce wood	n.d.	n.d.	n.d.	0.407	0.013	0.048	n.d.	n.d.	0.001	0.001	0.002	n.d.	n.d.
Pectin	0.052	0.034	0.247	0.230	n.d.	0.006	n.d.	n.d.	0.025	0.001	n.d.	n.d.	n.d.
Spruce wood	n.d.	0.026	0.066	0.998	0.309	0.115	n.d.	n.d.	0.027	0.004	n.d.	n.d.	n.d.
Water melon peel	0.006	0.019	0.072	0.184	0.031	0.070	0.014	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
Xylan	0.002	0.004	0.025	0.024	n.d.	1.116	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.

Table S-2: HPAEC-PAD results for each measured uronic acid, sugar degradation product as well as the lignin model compound and each biomass in g/L including molecular formula. n. s. = not specified, n.d. = not determinable.

	Galacturonic acid	Glucuronic acid	5-HMF	2,3-dimethoxy-benzyl alcohol	3,4-dimethoxy-benzyl alcohol	4-methoxy-benzyl alcohol	3,4-dimethoxy-benzaldehyde	2,6-dimethoxy-phenol	2-methoxy-phenol	4-allyl-2-methoxy-phenol	3,5-dimethoxy-4-hydroxy-benzaldehyde	LMC	AIL
													n.s.
Avicel	n.d.	n.d.	0.043	0.054	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	0.015
Banana peel	0.014	0.008	0.042	0.041	n.d.	n.d.	n.d.	n.d.	0.085	n.d.	n.d.	n.d.	0.866
Barley awn	n.d.	n.d.	0.035	0.046	n.d.	n.d.	n.d.	n.d.	0.119	n.d.	n.d.	n.d.	0.756
Barley straw	n.d.	n.d.	0.035	0.046	n.d.	n.d.	n.d.	n.d.	0.083	n.d.	n.d.	n.d.	0.827
Coconut peel	0.024	0.002	n.d.	0.008	n.d.	n.d.	n.d.	n.d.	n.d.	0.023	0.009	n.d.	1.839
Honey dew melon peel	0.104	0.006	n.d.	0.023	0.019	n.d.	0.022	n.d.	n.d.	0.024	0.002	n.d.	n.d.
Lawn grass	0.104	0.008	0.040	0.043	n.d.	n.d.	n.d.	n.d.	0.092	n.d.	n.d.	n.d.	0.699
Lignin, alkali	n.d.	n.d.	0.031	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	2.040
Lime peel	0.381	0.005	0.038	0.044	0.003	n.d.	n.d.	n.d.	0.097	0.016	0.001	n.d.	0.272
News paper	n.d.	n.d.	0.037	0.048	n.d.	n.d.	0.023	n.d.	n.d.	n.d.	n.d.	n.d.	0.631
orange peel	0.657	0.004	n.d.	0.009	n.d.	n.d.	n.d.	n.d.	n.d.	0.017	n.d.	n.d.	0.237
Organo cat spruce wood	n.d.	0.004	n.d.	0.011	0.007	n.d.	0.026	n.d.	n.d.	0.064	0.002	n.d.	1.154
Organosolv spruce wood	n.d.	n.d.	n.d.	0.011	0.036	n.d.	n.d.	n.d.	n.d.	0.006	0.001	n.d.	1.075
Pectin	1.619	0.007	n.d.	0.004	0.038	n.d.	n.d.	n.d.	0.006	0.035	0.001	n.d.	n.d.
Spruce wood	n.d.	0.004	0.035	0.045	n.d.	n.d.	0.031	n.d.	n.d.	n.d.	n.d.	n.d.	0.955
Water melon	0.010	n.d.	0.017	0.005	n.d.	n.d.	n.d.	n.d.	n.d.	0.000	0.000	n.d.	0.495
xylan	n.d.	n.d.	0.031	0.039	n.d.	n.d.	n.d.	n.d.	0.172	n.d.	n.d.	n.d.	0.115

Tab. S-3: Precision of the HPAEC-PAD method. Based on a tenfold measurement of water melon peel hydrolyzate, the standard deviation and the relative standard deviation were estimated. n.d. = not detectable, n.q. = not quantifiable.

Compound	standard		RSD
	average	deviation	
	[mg/L]	[mg/L]	[%]
Rhamnose	5.08	0.18	3.54
Arabinose	19.25	0.20	1.03
Galactose	71.68	0.61	0.85
Glucose	184.46	1.65	0.89
Mannose	31.12	1.26	4.04
Xylose	69.76	1.44	2.06
Fructose	14.10	0.09	0.63
Sucrose	n.d.		
Cellobiose	n.q.		
Cellotriose	n.q.		
Cellotetraose	n.q.		
Cellopentose	n.q.		
Cellohexaose	n.q.		
Galacturonic acid	10.37	0.23	2.22
Glucuronic acid	n.q.		
5-HMF	17.30	0.18	1.04
2,3-dimethoxybenzyl alcohol	5.53	0.069	1.25
3,4-dimethoxybenzyl alcohol	n.q.		
4-methoxybenzyl alcohol	n.q.		
3,4-dimethoxybenzaldehyde	n.q.		
Lignin model compound	n.d.		
2,6-dimethoxyphenol	n.q.		
2-methoxyphenol	n.q.		
4-allyl-2-methoxyphenol	0.28	0.06	21.43
3,5-dimethoxy-4-hydroxybenzaldehyde	0.15	0.00	0.02

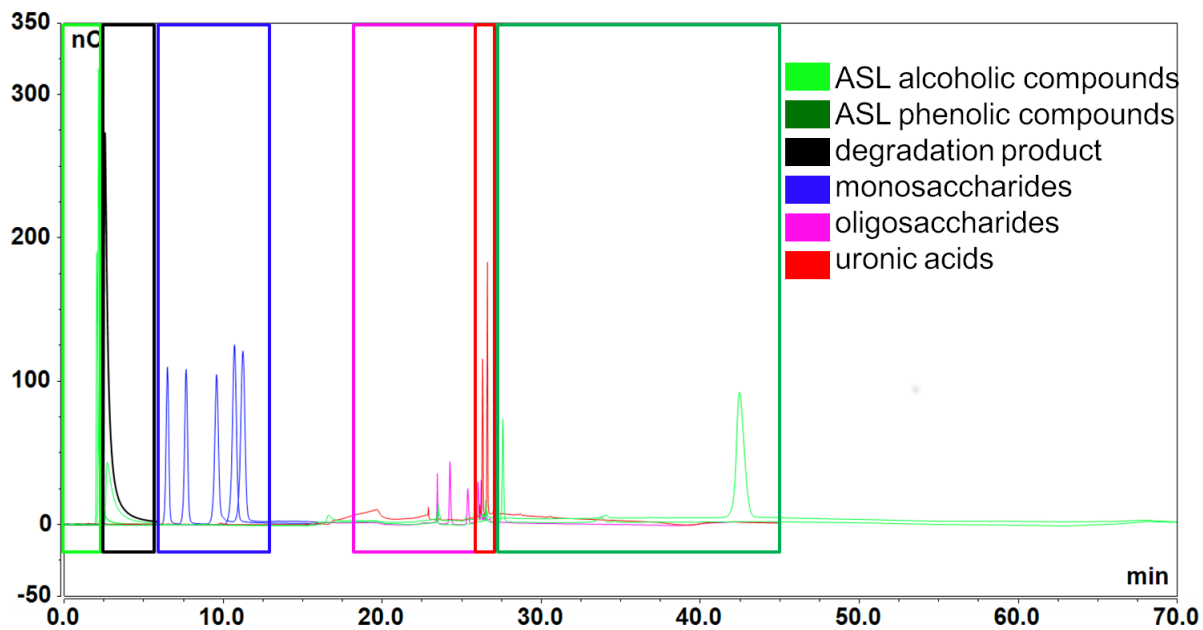


Fig. S-1: Superposition of chromatograms of commercially available soluble biomass standards. The green box shows the acid-soluble lignin (ASL) compounds, the black box the sugar degradation product 5-HMF, the blue box the monosaccharides, the pink box the soluble oligosaccharides and the red box the uronic acids.

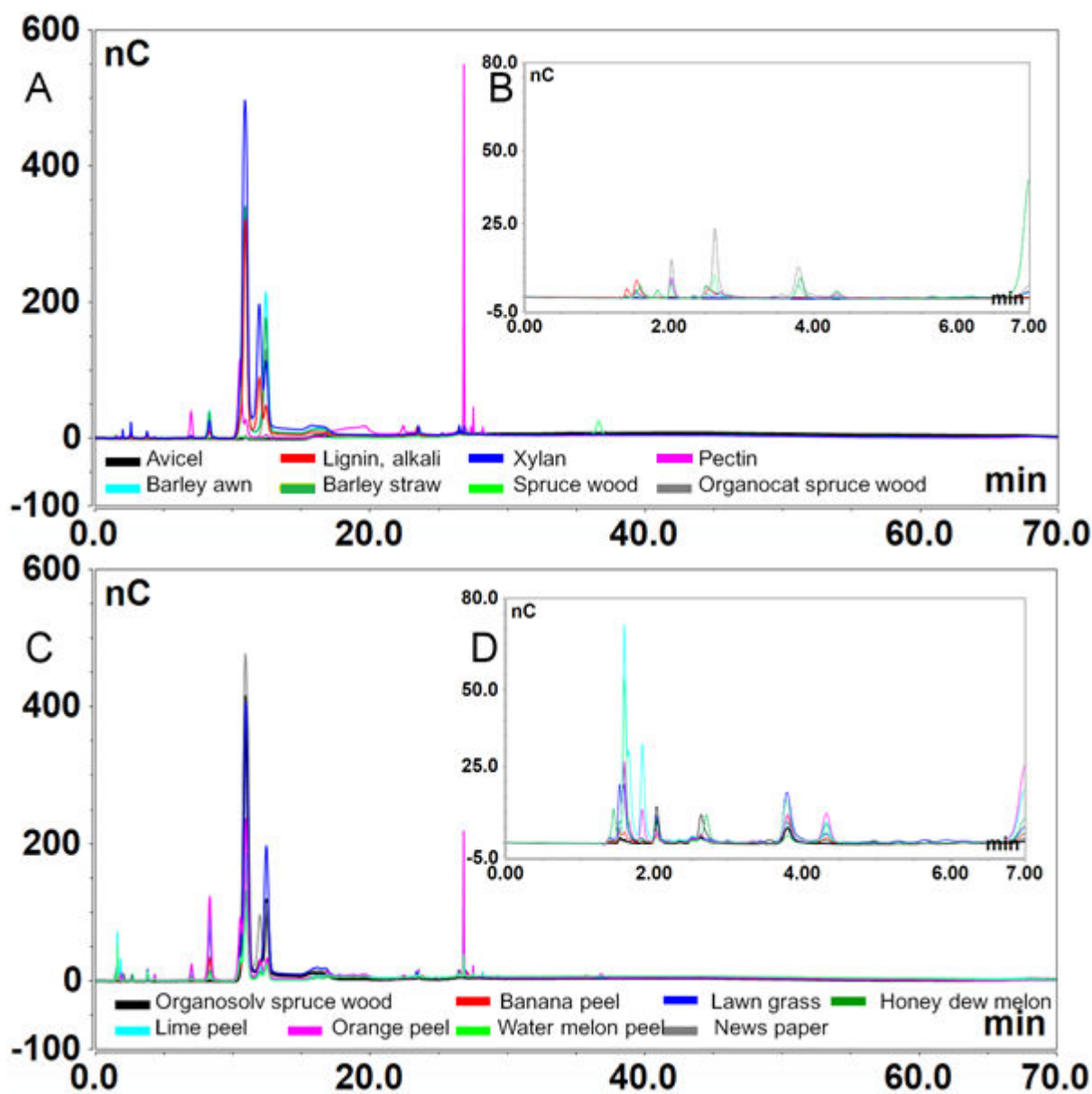


Fig. S-2: Overlay of chromatograms from different hydrolyzates. Diverse biomasses were hydrolyzed with a two-step acid hydrolysis. The obtained liquid was analyzed using the HPAEC-PAD method described here. A and C: full chromatogram. B and D: chromatograms of the biomass derived phenols.