

Effect of autohydrolysis and ionosolv treatments on eucalyptus fractionation and recovered lignin properties

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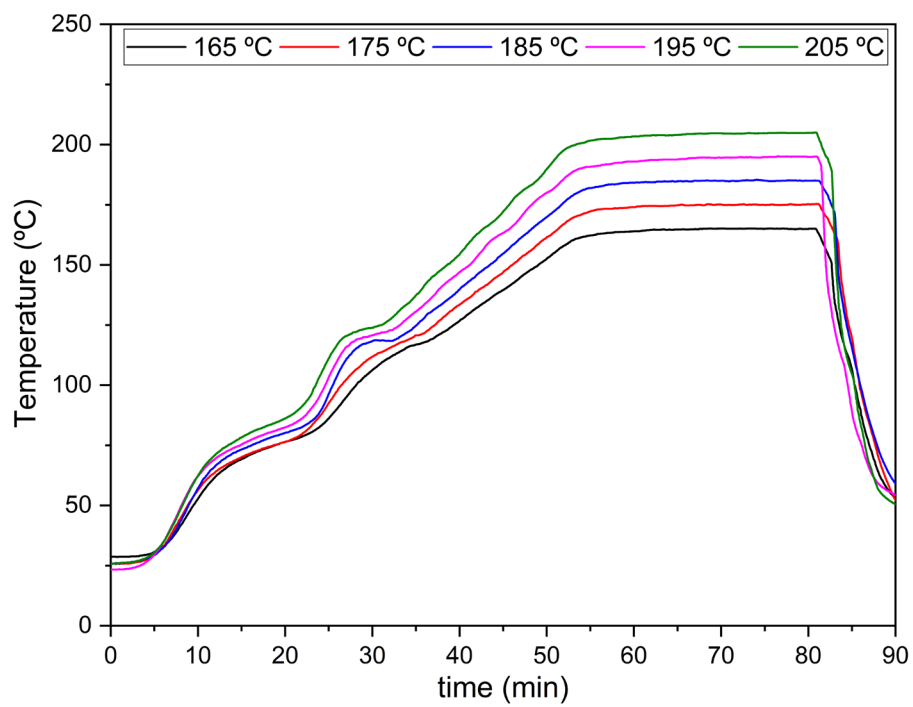


Figure S1. Temperature profiles of the autohydrolysis step.

Table S1. ANOVA table of the fractions' recovery responses. Values in red indicate not significant effects.

Effect	Sum of squares	Degrees of freedom	Mean of squares	<i>P</i>
Hemicelluloses recovery				
T_{AH}	2708.48	1	2708.48	0.0064
T_{AH}^2	1555.98	1	1555.98	0.0168
Lack of fit	33.8113	2	16.9056	0.9151
Total error	398.31	4	99.578	
Total	4662.77	6		
Lignin recovery				
T_{AH}	38.4300	1	38.4300	0.0925
t_{iono}	674.0214	1	674.0214	0.0130
T_{AH}^2	0.0576	1	0.0576	0.9639
t_{iono}^2	18.7226	1	18.7226	0.4412
$T_{AH} \cdot t_{iono}$	16.5649	1	16.5649	0.4661
Lack of fit	77.3738	3	25.7913	0.3521
Total error	71.6598	3	23.8866	
Total	831.9779	9		
CRF recovery				
T_{AH}	5.8559	1	5.8559	0.0981
t_{iono}	123.8044	1	123.8044	0.0107
T_{AH}^2	6.0972	1	6.0972	0.5417
t_{iono}^2	51.4547	1	51.4547	0.0510
$T_{AH} \cdot t_{iono}$	0.1549	1	0.1549	0.9198
Lack of fit	20.2946	3	6.7649	0.1039
Total error	39.4082	3	7.8816	
Total	242.4996	9		
Lignin content in CRF				
T_{AH}	0.5613	1	0.5613	0.6902
t_{iono}	1.9503	1	1.9503	0.0929
T_{AH}^2	3.8446	1	3.8446	0.3336
t_{iono}^2	26.3314	1	26.3314	0.0473
$T_{AH} \cdot t_{iono}$	0.1849	1	0.1849	0.8172
Lack of fit	7.2293	2	3.6147	0.0631
Total error	8.7260	3	2.9087	
Total	54.3224	9		

Table S2. Post autohydrolysis solid recovery and composition.

T _{AH} (°C)	t _{iono} (h)	Solid recovery ^a	Lignin ^b	Glucan ^b	Hemicelluloses ^b
175	1	75.45	35.17±0.01	58.84±0.26	5.48±0.38
175	5	75.45	35.17±0.01	58.84±0.26	5.48±0.38
185	3	72.43	34.84±2.85	60.17±0.48	4.77±1.45
185	3	72.43	34.84±2.85	60.17±0.48	4.77±1.45
185	3	72.43	34.84±2.85	60.17±0.48	4.77±1.45
195	1	70.17	35.66±0.69	63.74±0.51	1.77±0.09
195	5	70.17	35.66±0.69	63.74±0.51	1.77±0.09
170.9	3	77.64	32.23±0.50	57.25±0.23	8.88±0.16
199.1	3	69.23	34.91±0.32	69.16±0.18	0.83±0.07
185	0.17	72.43	34.84±2.85	60.17±0.48	4.77±1.45
185	5.83	72.43	34.84±2.85	60.17±0.48	4.77±1.45

a: g recovered solid/100 g of eucalyptus

b: g compound/100 g recovered solid

Table S3. ANOVA table of the lignin properties responses. Values in red indicate not significant effects.

Effect	Sum of squares	Degrees of freedom	Mean of squares	<i>P</i>
Mw				
T _{AH}	1.38·10 ⁸	1	1.38·10 ⁸	0.13602
t _{iono}	1.83·10 ⁹	1	1.83·10 ⁹	0.00517
T _{AH} ²	3.59·10 ⁷	1	3.59·10 ⁷	0.04738
t _{iono} ²	3.86·10 ⁸	1	3.86·10 ⁸	0.03295
T _{AH} · t _{iono}	2.02·10 ⁸	1	2.02·10 ⁸	0.04157
Lack of fit	1.3541·10 ⁸	3	4.5136·10 ⁷	0.1727
Total error	1.01·10 ⁸	3	3.37·10 ⁷	
Total	2.74·10 ⁹	9		
PDI				
T _{AH}	161.2021	1	161.2021	0.3430
t _{iono}	1775.6972	1	1775.6972	0.0055
T _{AH} ²	25.5346	1	25.5346	0.6851
t _{iono} ²	391.7245	1	391.7245	0.0404
T _{AH} · t _{iono}	53.8022	1	53.8022	0.5626
Lack of fit	374.6401	2	187.3200	0.1516
Total error	383.1062	3	127.7021	
Total	2795.8832	9		
T _{D10%}				
T _{AH}	1354.1656	1	1354.1656	0.0131
t _{iono}	355.8491	1	355.8491	0.0428
T _{AH} ²	0.0286	1	0.0286	0.0421
t _{iono} ²	3016.4461	1	3016.4461	0.0042
T _{AH} · t _{iono}	676	1	676	0.0333
Lack of fit	433.037	3	433.037	0.0131
Total error	144.7106	3	144.7106	
Total	5944.1	9		

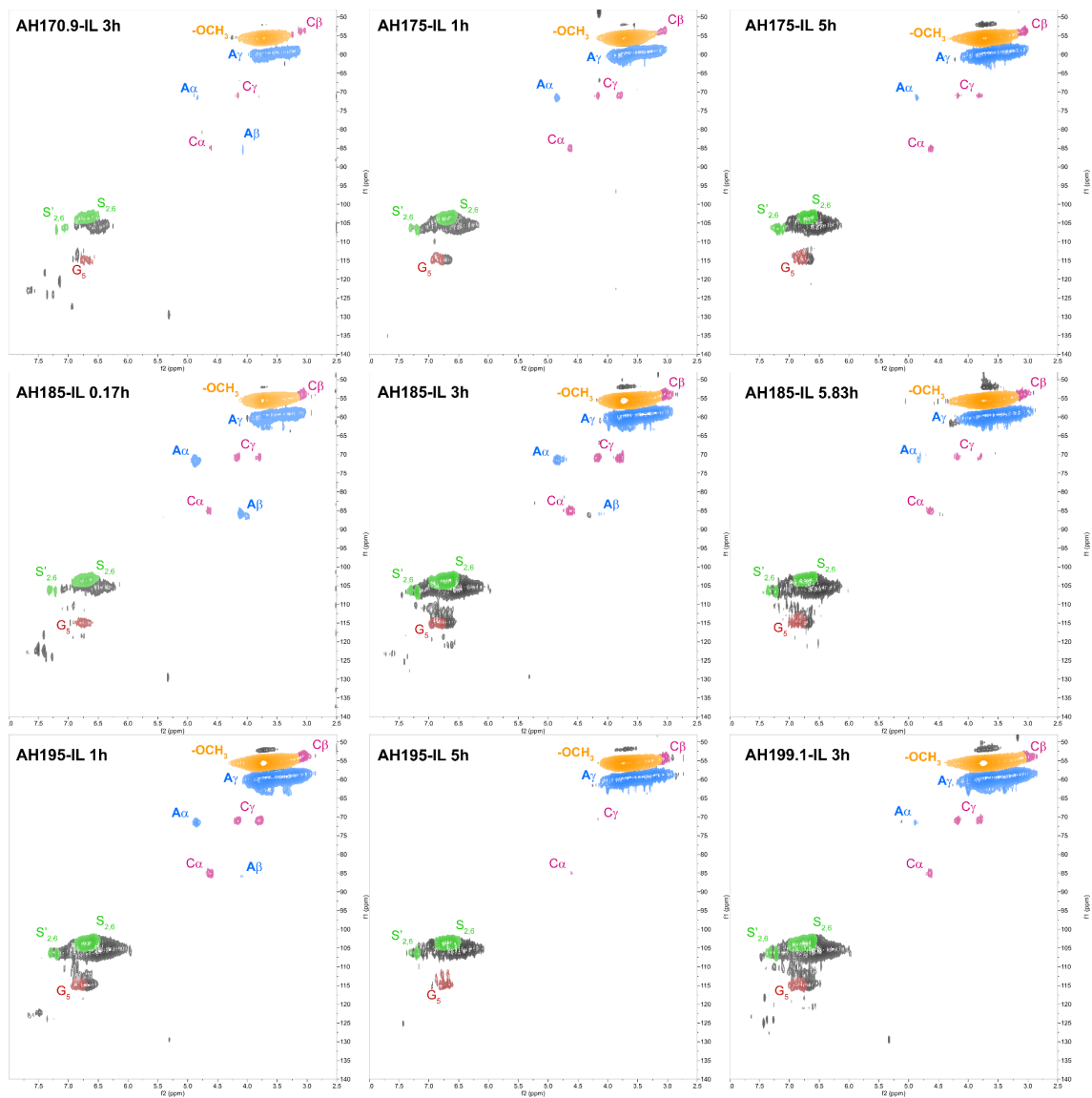


Figure S2. HSQC-NMR spectra of the recovered lignins by the autohydrolysis+ionosolv process combination.