

Impact of ammonium sulfite-based sequential pretreatment combinations on two distinct saccharification of wheat straw

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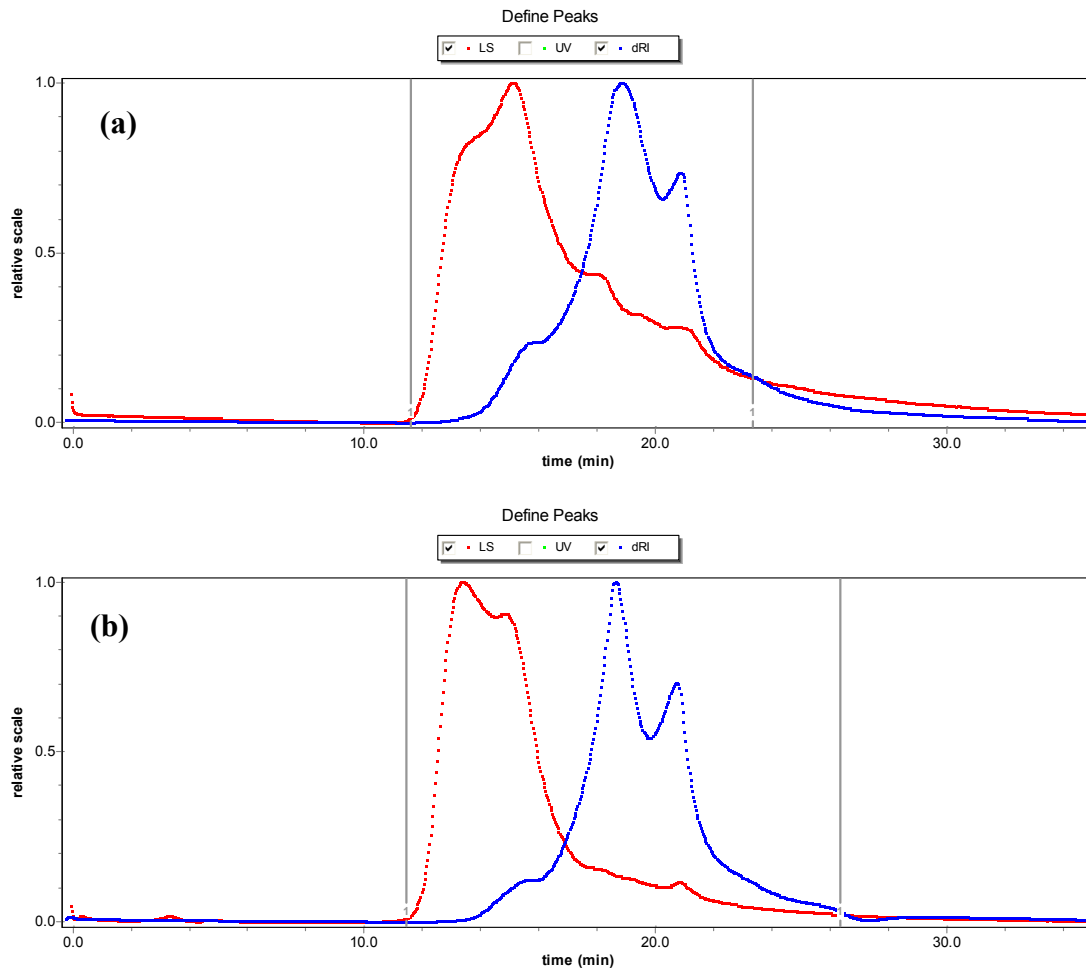


Figure S1 GPC spectra of lignin in spent liquor from AS pretreatment (a. the lignin from the first step of AS+HT pretreatment; b. the lignin from the second step of HT+AS pretreatment)

Table S1 Assignments of ^{13}C - ^1H correlation signals in the HSQC spectra of the lignin fractions in AS pretreatment spent liquor

Label	$\delta_{\text{C}} / \delta_{\text{H}}$	Assignment
B $_{\beta}$	53.28/3.09	C $_{\beta}$ -H $_{\beta}$ in resinol substructures (B)
-OCH $_3$	56/3.73	C-H in methoxyls
A $_{\gamma}$	59.86-60.97/3.31-3.65	C $_{\gamma}$ -H $_{\gamma}$ in β -O-4' substructures (A)
X $_5$	62.8/3.23	C $_5$ -H $_5$ in β -D-xylopyranoside
A' $_{\gamma}$	62.8/3.96	C $_{\gamma}$ -H $_{\gamma}$ in γ -acylated β -O-4' substructures (A')
A'' $_{\gamma}$	63.11/4.18	C $_{\gamma}$ -H $_{\gamma}$ in γ -acylated β -O-4' substructures (A'')
B $_{\gamma}$	71/3.8, 4.2	C $_{\gamma}$ -H $_{\gamma}$ in resinol substructures (B)
A $_{\alpha}$	71.8/4.6	C $_{\alpha}$ -H $_{\alpha}$ in β -O-4' substructures (A)
X $_2$	72.79/3.13	C $_2$ -H $_2$ in β -D-xylopyranoside
X $_4$	76.35/3.63	C $_4$ -H $_4$ in β -D-xylopyranoside
X $_3$	77.46/3.76	C $_3$ -H $_3$ in β -D-xylopyranoside
D $_{\beta'}$	80.8/4.01	C $_{\beta'}$ -H $_{\beta'}$ in spirodienone substructures (D)
D $_{\alpha}$	80.8/5.07	C $_{\alpha}$ -H $_{\alpha}$ in spirodienone substructures (D)
A $_{\beta(\text{G/H})}$	82.9/4.38	C $_{\beta}$ -H $_{\beta}$ in β -O-4' substructures linked to G and H units (A)
A $_{\beta(\text{S})}$	84.6/4.13	C $_{\beta}$ -H $_{\beta}$ in β -O-4' substructures linked to S units (A)
S $_{2,6}$	103.6/6.37	C $_{2,6}$ -H $_{2,6}$ in etherified syringyl units (S)
S' $_{2,6}$	106.9/6.64	C $_{2,6}$ -H $_{2,6}$ in oxidized (C $_{\alpha}$ =O) syringyl units (S')
G $_2$	107.5/6.48	C $_2$ -H $_2$ in guaiacyl units (G)
G' $_2$	113.2/6.92	C $_2$ -H $_2$ in oxidized (C $_{\alpha}$ =O) guaiacyl units (G')
G $_5$	115.3/6.74	C $_5$ -H $_5$ in guaiacyl units (G)
G $_6$	122.3/6.79	C $_6$ -H $_6$ in guaiacyl units (G)
H $_{2,6}$	130.5/7.17	C $_{2,6}$ -H $_{2,6}$ in p-hydroxyphenyl units (H)

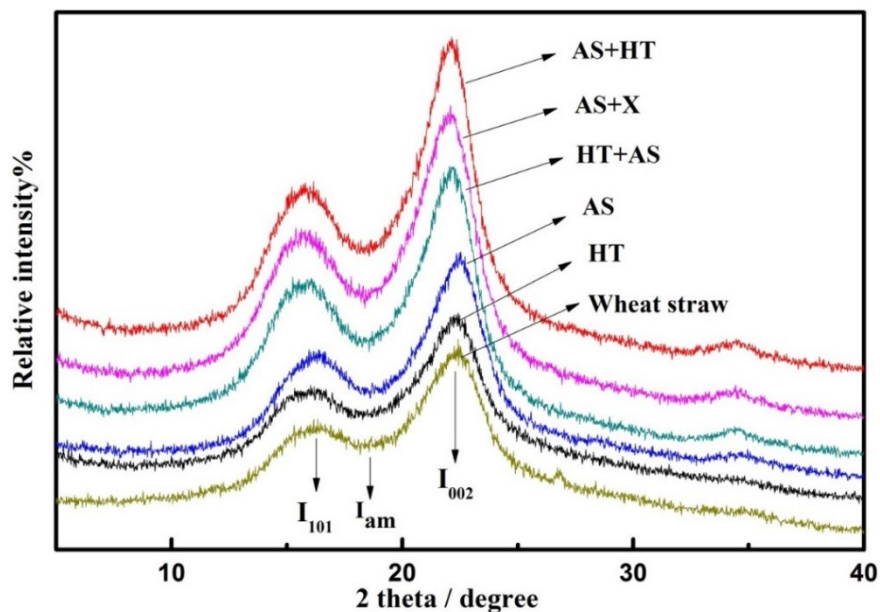


Figure S2 X-ray diffraction images of raw and pretreated wheat straw

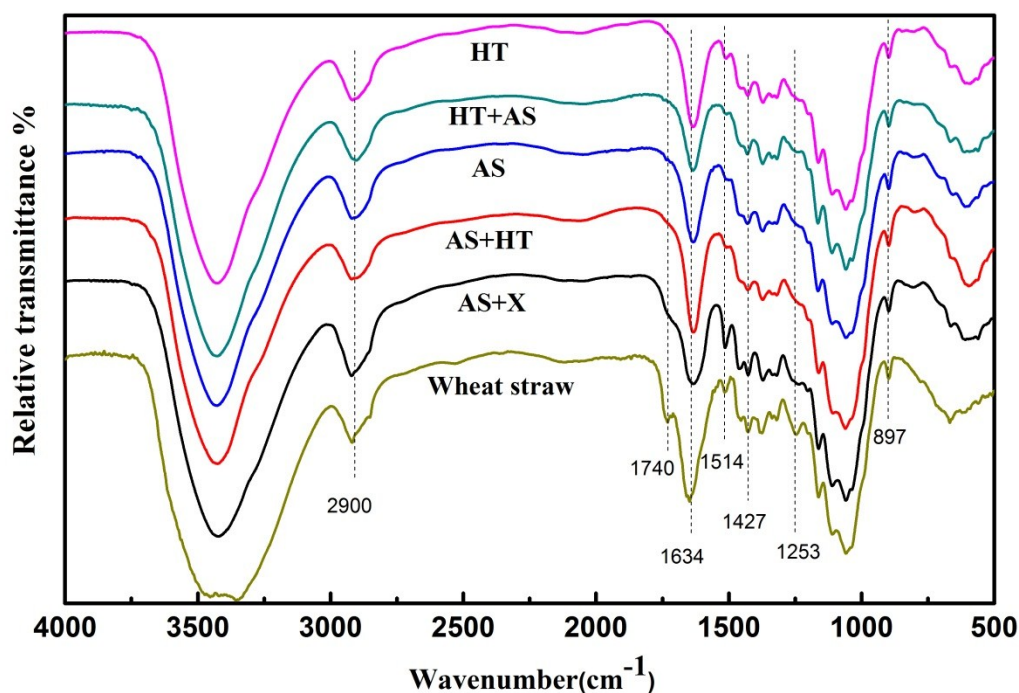


Figure S3 FTIR spectra of raw and pretreated wheat straw

Table S2 FTIR peak assignments of raw and pretreated wheat straw

Wavenumber (cm ⁻¹)	Peak assignment	Components
897	Anomeric vibration at β -glycosidic linkage	Polysaccharides
1253	C—O stretching vibration	Aryl ring of lignin
1427	CH bending vibration of CH ₃	Phenolic lignin
1514	Aromatic skeletal vibration	Lignin
1634	Aromatic skeletal vibration	Lignin
1740	C=O stretching in acetyl groups and carboxylic acids	Acetyl groups on heteromannans and heteroxylans
2900	Asymmetric stretching vibration of methylene groups	Lignin and Polysaccharides
3400	Stretch of —OH groups	alcoholic hydroxyl group of lignin and Polysaccharides

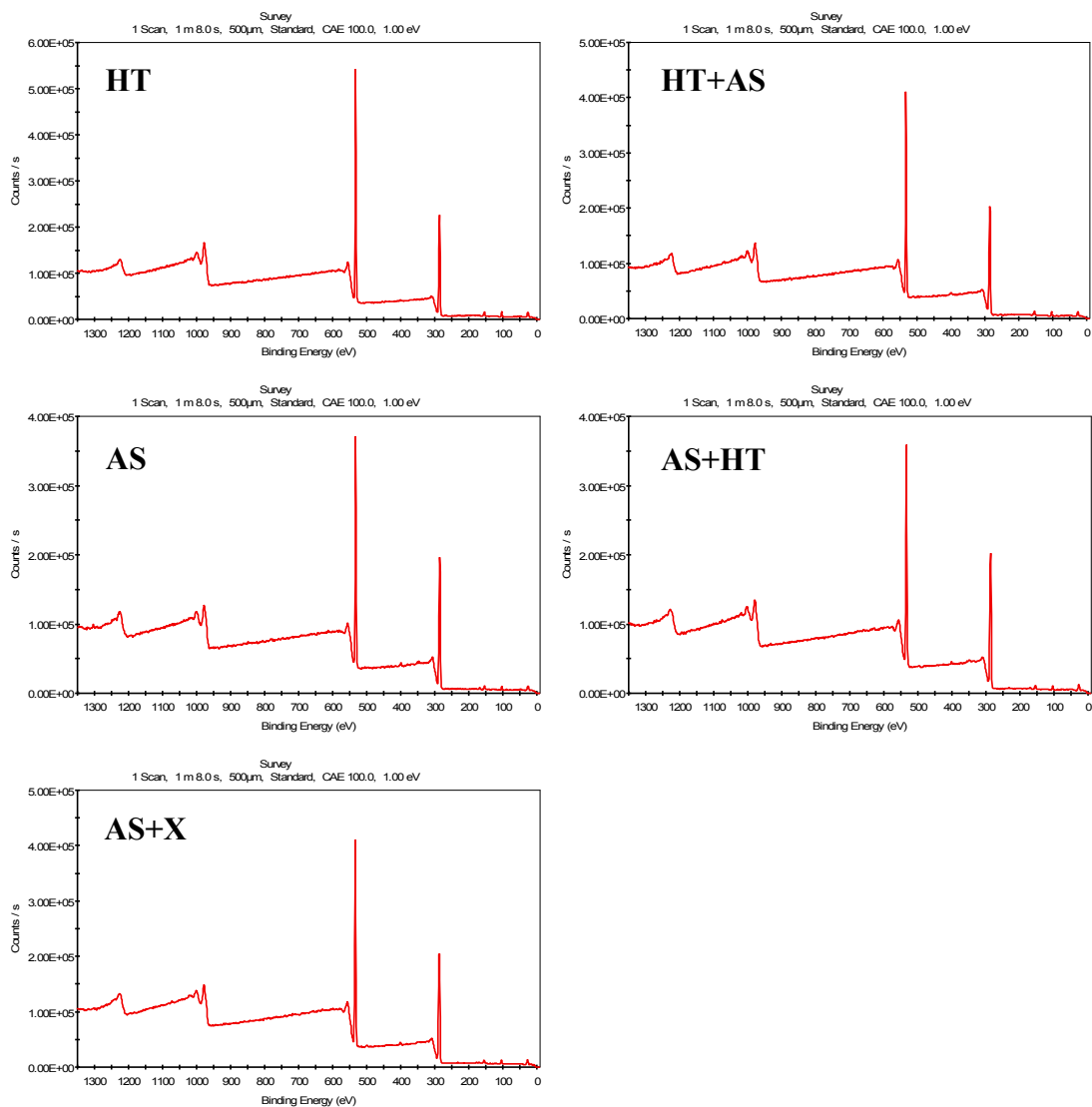


Figure S4 XPS spectra of pretreated wheat straw