

Supplementary Materials:

Ammonia–Mechanical Pretreatment of Wheat Straw for the Production of Lactic Acid and High-Quality Lignin

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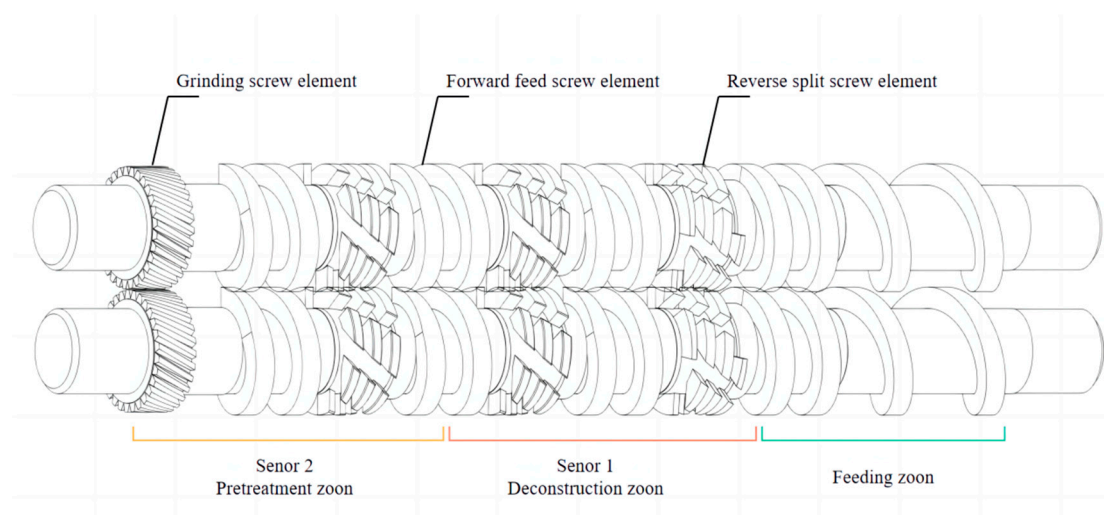


Figure S1. Twin-screw element structure diagram.

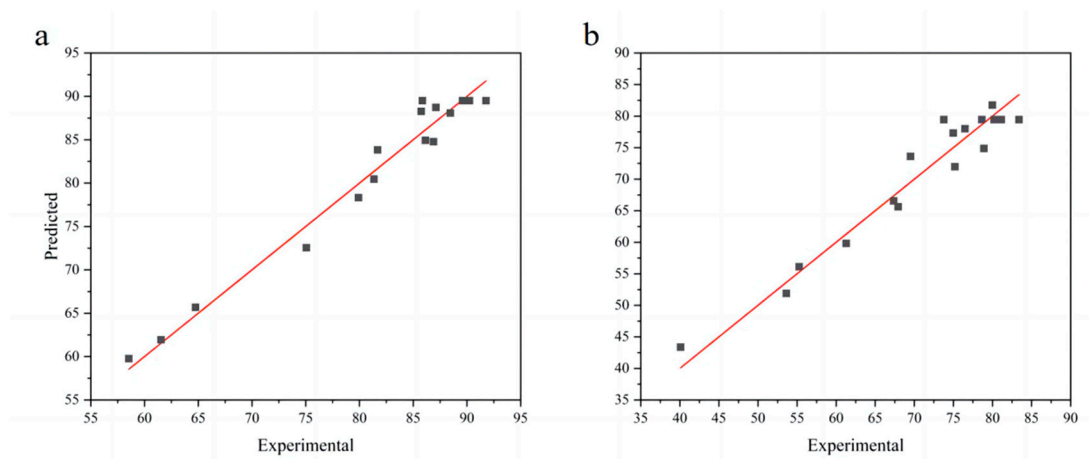


Figure S2. Predicted vs experimental of conversion. (a) Cellulose; (b) Hemicellulose.

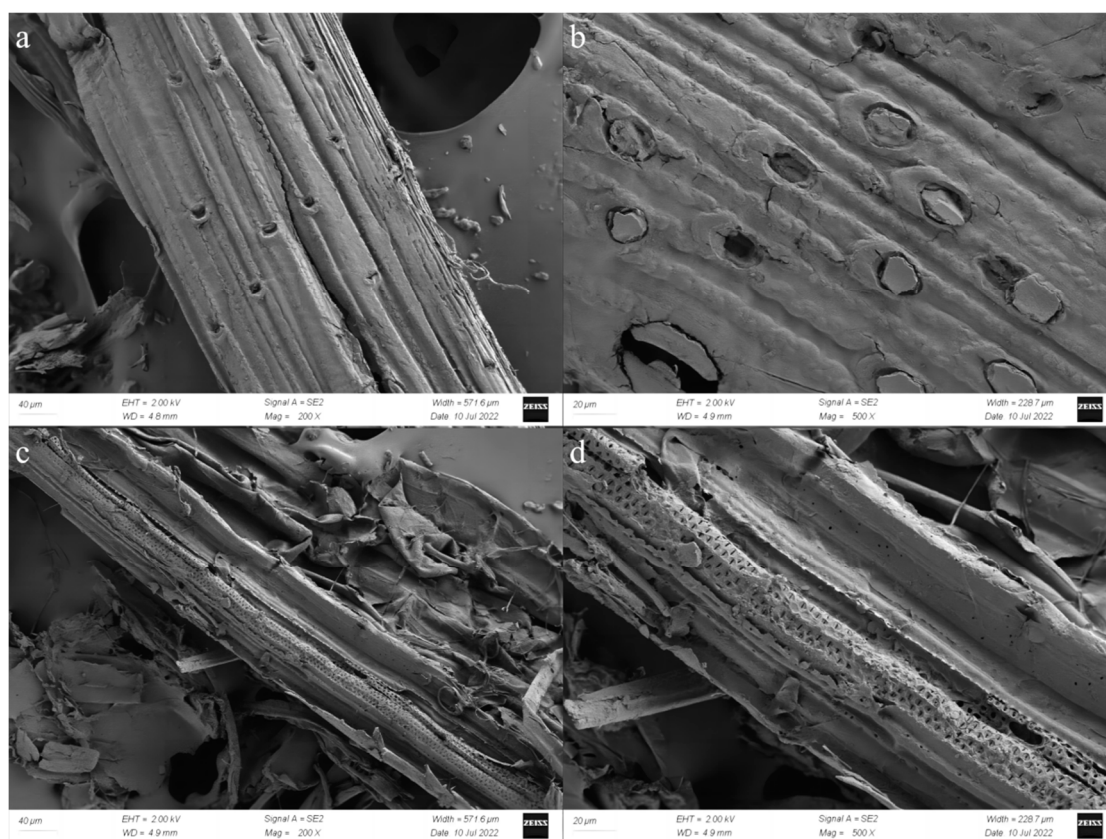


Figure S3. SEM micrographs of wheat straw and pretreatment residue (at optimal pretreatment conditions). (a-b) wheat straw, (c-d) pretreatment residue.

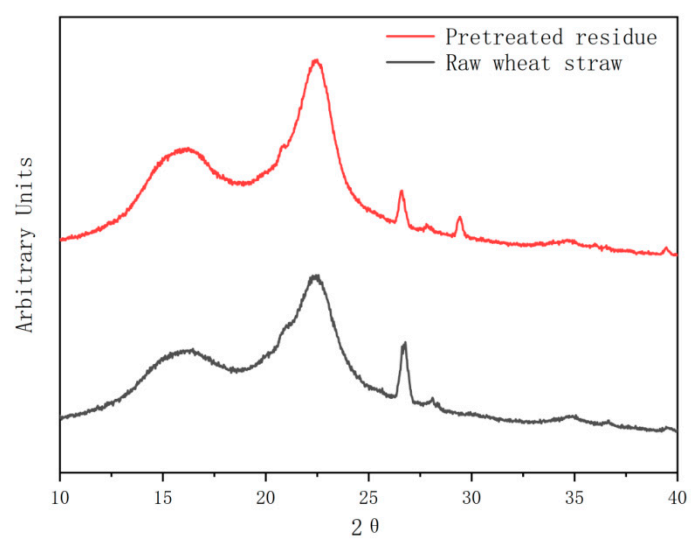


Figure S4. XRD of wheat straw and pretreatment residue (at optimal pretreatment conditions).

Table S1. Box-Behnken Design (BBD) for optimization of process parameters affecting the cellulose conversion (Y_{Glucose}) and hemicellulose conversion (Y_{Xylose}) during ammonia-mechanical pretreatment of wheat straw

Entry	X_1^c	X_2^d	X_3^e	$Y_{\text{Cellulose-end}}\%$		$Y_{\text{Hemicellulose-end}}\%$	
				Experimental	Predicted	Experimental	Predicted
1 ^a	15	3:1	6	87.12	88.72	76.50	77.98
2 ^a	25	2:1	6	88.46	88.07	79.99	81.74
3 ^a	25	3:1	4	86.14	84.93	75.20	71.96
4 ^a	15	2:1	4	91.78	89.50	78.65	79.44
5 ^a	15	3:1	2	81.69	83.82	69.52	73.59
6 ^a	15	1:1	2	79.92	78.32	61.29	59.80
7 ^a	25	2:1	2	81.36	80.45	67.37	66.54
8 ^a	25	1:1	4	85.75	88.27	74.99	77.30
9 ^a	15	2:1	4	85.86	89.50	73.79	79.44
10 ^a	5	2:1	6	64.75	65.67	55.28	56.11
11 ^a	5	3:1	4	75.06	72.54	67.94	65.62
12 ^a	5	2:1	2	61.54	61.93	53.64	51.88
13 ^a	5	1:1	4	58.54	59.75	40.11	43.35
14 ^a	15	2:1	4	89.60	89.50	80.22	79.44
15 ^a	15	2:1	4	90.24	89.50	81.13	79.44
16 ^a	15	2:1	4	90.03	89.50	83.40	79.44
17 ^a	15	1:1	6	86.89	84.77	78.92	74.85
18 ^b	19	2.1:1	4.8	93.47	92.45	82.61	83.39

^a The pretreatment residue under BBD conditions. ^b The pretreatment residue under the optimal condition. ^c Aqueous ammonia concentration (w/w). ^d Liquid-solid ratio (w/w). ^e Holding time (h).

The second-order polynomial model of the cellulose and hemicellulose conversion was fit with the following equation (1) and (2):

$$Y_{\text{cellulose-end}} = 4.76 + 5.08X_1 + 15.68X_2 + 9.02X_3 - 0.11X_1^2 - 1.63X_2^2 - 0.99X_3^2 - 0.40X_1X_2 + 0.05X_1X_3 - 0.19X_2X_3 \quad (1)$$

$$Y_{\text{hemicellulose-end}} = -33.66 + 5.19X_1 + 34.693X_2 + 11.40X_3 - 0.11X_1^2 - 3.69X_2^2 - 1.05X_3^2 - 0.69X_1X_2 + 0.14X_1X_3 - 1.33X_2X_3 \quad (2)$$

Table S2. ANOVA table of linear, quadratic and interactive terms of ammonia-mechanical pretreatment process variables on responses (F-value)

Source	Cellulose conversions		Hemicellulose conversion	
	F Value	<i>p</i> -value	F Value	<i>p</i> -value
Model	25.70	0.0001	12.72	0.0015
A	114.74	< 0.0001	44.13	0.0003
B	6.13	0.0425	7.79	0.0269
C	8.84	0.0207	10.27	0.0150
AB	8.92	0.0203	10.37	0.0146
AC	0.52	0.4947	1.64	0.2413
BC	0.081	0.7838	1.54	0.2543
A ²	76.40	< 0.0001	28.68	0.0011
B ²	1.53	0.2565	3.14	0.1195
C ²	9.10	0.0195	4.03	0.0847
Lack of Fit	not significant		not significant	
C.V.%	3.32		6.09	
R ²	0.9706		0.9424	
Adjusted R ²	0.9329		0.8683	

Table S3. Composition of wheat straw enzymatic hydrolysate used for lactic acid fermentation

Component	Concentration (g/L)
Glucose	60.90
Xylose	25.16
Cellobiose	0.80
Arabinose	2.99
Furfural	/
5-hydroxymethyl furfural	/
Acetic acid	/
Formic acid	/

Table S4. The crystallinity index of wheat straw and pretreatment residue (at optimal pretreatment conditions)

Name	I₀₀₂	I_{am}	CrI
Wheat straw	13848	7244	47.69%
Pretreatment residue	16612	7713	53.57%

Table S5. Ultimate analysis of MWL and P-lignin (extracted at optimal pretreatment conditions)

Name	C (%)	H (%)	N (%)	S (%)
MWL	57.13	6.235	0.215	0.08
P-lignin.	48.735	6.075	2.755	0.05

Table S6. Assignment in main lignin ^{13}C - ^1H cross-signals in the HSQC spectra

Label	$\delta_{\text{C}}/\delta_{\text{H}}$	Assignments
Methoxyl group	56.1/3.73	C-H in methoxyls
B $_{\beta}$	53.30/3.56	C $_{\beta}$ -H $_{\beta}$ in phenylcoumarane substructures (B)
B $_{\gamma}$	62.5/3.73	C $_{\gamma}$ -H $_{\gamma}$ in phenylcoumaran substructures (B)
A $_{\alpha}$	71.6/4.88	C $_{\alpha}$ -H $_{\alpha}$ in β -O-4' substructures (A)
A $_{\beta}'$ (S)	84.0/4.42	C $_{\beta}$ -H $_{\beta}$ in β -O-4' substructures linked to S' (A)
A $_{\beta}$ (S)	86.1/4.14	C $_{\beta}$ -H $_{\beta}$ in β -O-4' substructures linked to S units (A)
A $_{\gamma}$	60.6/3.68	C $_{\gamma}$ -H $_{\gamma}$ in β -O-4' substructures (A)
A' $_{\gamma}$	62.4/4.13	C $_{\gamma}$ -H $_{\gamma}$ in γ -acylated β -O-4' (A)
T $_{2,6}$	104.1/7.45	C $_{2,6}$ -H $_{2,6}$ in T units (T)
T $_6$	99.1/6.23	C $_6$ -H $_6$ in T units (T)
T $_8$	94.5/6.59	C $_8$ -H $_8$ in T units (T)
T $_3$	104.8/7.05	C $_3$ -H $_3$ in T units (T)
S $_{2,6}$	104.3/6.71	C $_{2,6}$ -H $_{2,6}$ in etherified syringyl units (S)
G $_2$	110.7/6.96	C $_2$ -H $_2$ in guaiacyl units (G)
G $_5$	115.4/6.79	C $_5$ -H $_5$ in guaiacyl units (G)
G $_6$	119.2/6.78	C $_6$ -H $_6$ in guaiacyl units (G)
H $_{2,6}$	128.4/7.23	C $_{2,6}$ -H $_{2,6}$ in H units (H)
FA $_2$	111.6/7.33	C $_2$ -H $_2$ in ferulic acid (FA)
FA $_{\beta}$	115.5/6.53	C $_{\beta}$ -H $_{\beta}$ in ferulic acid (FA)
FA $_{\alpha}$	145.2/7.58	C $_{\alpha}$ -H $_{\alpha}$ in ferulic acid (FA)
FAM $_2$	111.3/7.32	C $_2$ -H $_2$ in feruloyl amide (FAM)
FAM $_6$	120.9/7.07	C $_6$ -H $_6$ in feruloyl amide (FAM)
<i>p</i> CA $_{\beta}$	114.0/6.29	C $_{\beta}$ -H $_{\beta}$ in <i>p</i> -coumaric acid (<i>p</i> CA)
<i>p</i> CA $_{\alpha}$	144.8/7.26	C $_{\alpha}$ -H $_{\alpha}$ in <i>p</i> -coumaric acid (<i>p</i> CA)
<i>p</i> CA $_{3,5}$	115.4/6.75	C $_{3,5}$ -H $_{3,5}$ in <i>p</i> -coumaric acid (<i>p</i> CA)
<i>p</i> CA $_{2,6}$	130.3/7.47	C $_{2,6}$ -H $_{2,6}$ in <i>p</i> -coumaric acid (<i>p</i> CA)
<i>p</i> CAM $_{3,5}$	115.1/6.89	C $_{3,5}$ -H $_{3,5}$ in <i>p</i> -coumaroyl amide (<i>p</i> CAM)
<i>p</i> CAM $_{2,6}$	128.4/7.26	C $_{2,6}$ -H $_{2,6}$ in <i>p</i> -coumaroyl amide (<i>p</i> CAM)