

Supplementary materials

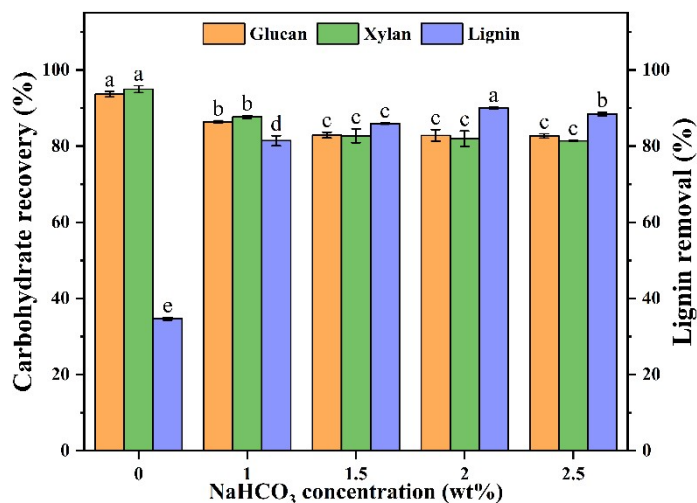


Fig. S1. Composition recoveries of DES pretreatment with different additions of NaHCO₃.

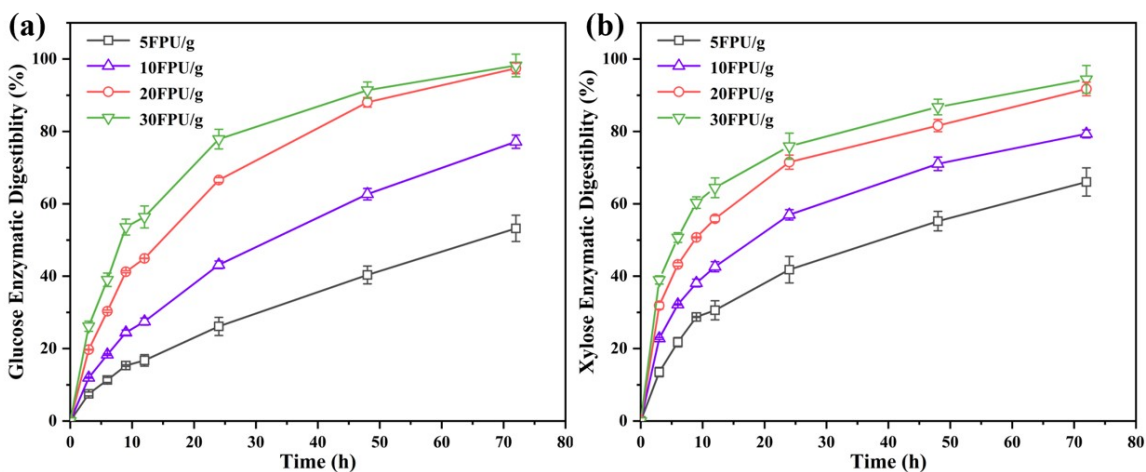


Fig. S2. Effects of different enzyme loads on the yield of glucose and xylose enzymatically hydrolyzed by pretreated CS.

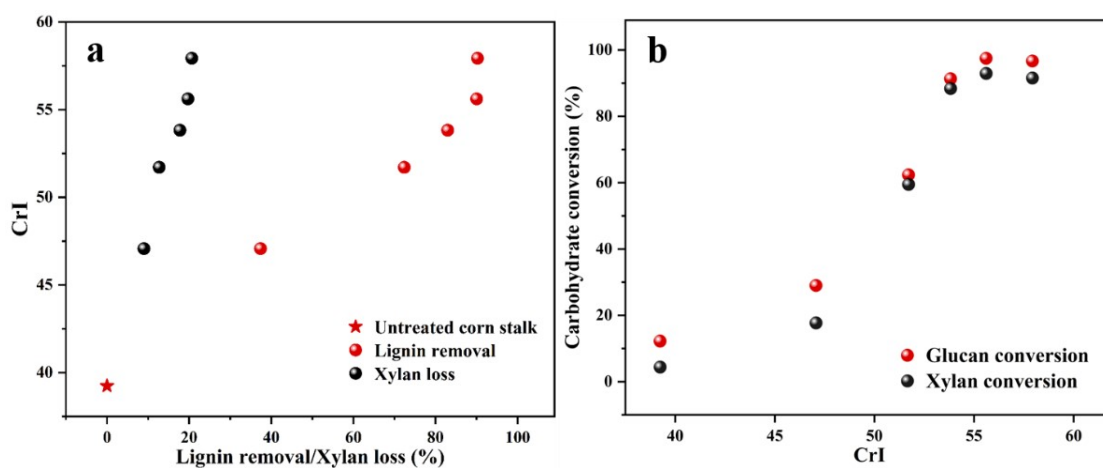


Fig. S3. Correlations (a) between CrI and lignin removal/xylan loss, (b) between CrI and Carbohydrate conversion.

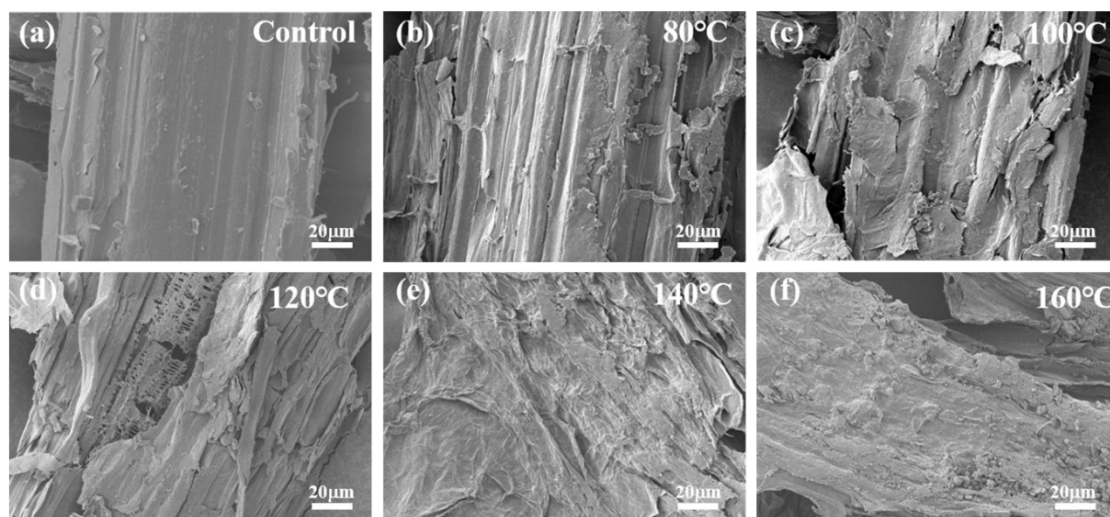


Fig. S4. (a) SEM images of untreated CR, samples treated at (b) 80°C, (c) 100°C, (d) 120°C, (e) 140°C, and (f) 160°C at different temperatures.

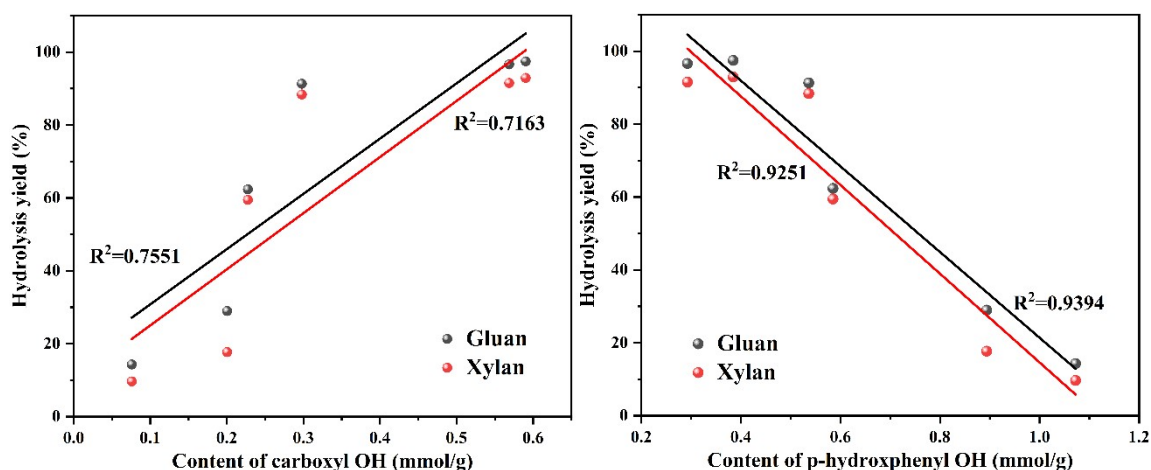


Fig. S5. Correlation between p-hydroxyphenyl OH(a), and carboxyl OH content (b) in lignins and the corresponding 72 hydrolysis yield.

Table S1. Thermal parameters of the lignin

Sample	$T_{\text{peak}}(^{\circ}\text{C})$		$\text{DTG}_{\text{max}}(\%/^{\circ}\text{C})$		Solid(%)
	P1	P2	P1	P2	
DEL	238	350	-0.33	-0.29	32.61
L80	224	337	-0.29	-0.31	34.99
L100	253	345	-0.24	-0.31	40.83
L120	254	343	-0.27	-0.31	41.54
L140	253	327	-0.18	-0.24	44.95
L160	254	343	-0.16	-0.26	44.53

File S1. Preparation of double enzymatic lignin (DEL)

The CS raw material was ball-milled using a planetary ball mill at 450 rpm for 5 h. Then, 5 g of the prepared CS sample was added to 100 mL of sodium citrate buffer (0.05 M, pH 4.80), shaken well, and then 60 FPU/g of cellulase was added. The following enzymatic hydrolysis process was carried out in a thermostatic incubation oscillator (50 °C, 120 rpm) for 48 h. The final mixture was centrifuged, and the residual solid was adequately washed with sodium citrate buffer (0.05 M, pH 4.80) and then freeze-dried. The dried residual solid was ball-milled again for 2 h and enzymatically digested. After the purification and freeze-drying process, DEL samples were obtained.