

Supporting Information

**Improvement of lignin yield and purity from corncob in the presence of steam explosion and liquid hot
pressured alcohol**

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Lignin Acetylation

Acetylation is frequently performed on the residual and cooking liquor lignins. The lignins (dried under vacuum at 40°C for 8-16 hours) were acetylated with acetic anhydride/pyridine (1/1, v/v) at room temperature for 24 hours in 50-ml round-bottom flask. The concentration of the lignin in this solution was approximately 50 mg/ml. After 24 hours, the solution was diluted with ~30 ml of ethanol and stirred for an additional 30 minutes, after which the solvents were removed with a rotary evaporator. Repeated addition and removal of ethanol allowed for the removal of acetic acid and pyridine from the sample. The residue was then dissolved in chloroform, washed twice with filtered deionized water in a separatory funnel, and dried with anhydrous sodium sulfate. The chloroform solution (~10 ml) was added drop-wise to approximately 150 ml of anhydrous ether and the product collected as a precipitate. The precipitate was washed twice with ether, each time being collected by centrifugation. The precipitate was dried under high vacuum at 40°C for 24 hours.

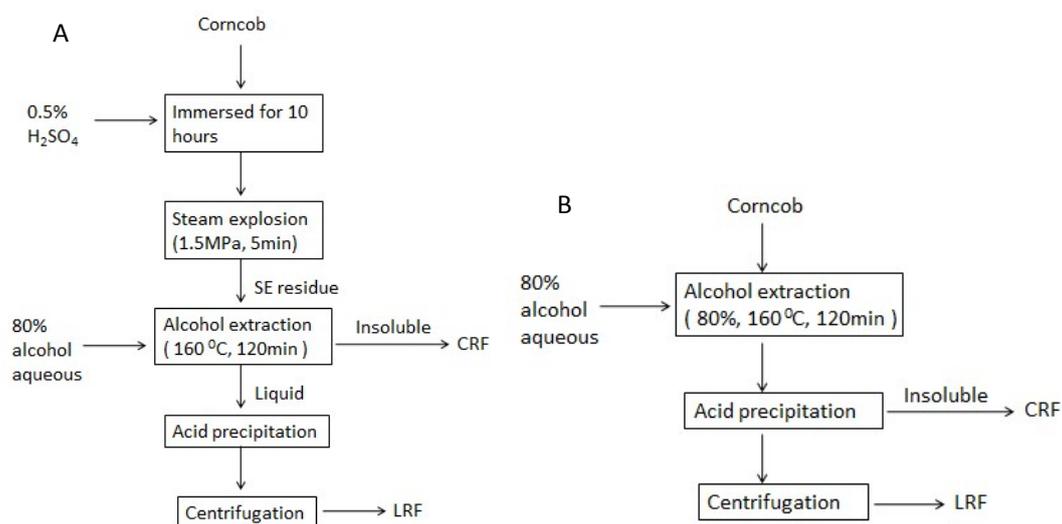


Fig.S1 Schematic representation of the work flow for corncob treatment: (A) the combination of DASE and LHPAD (lignin yield 40.94%); (B) single LHPAD process (lignin yield 6.81%).

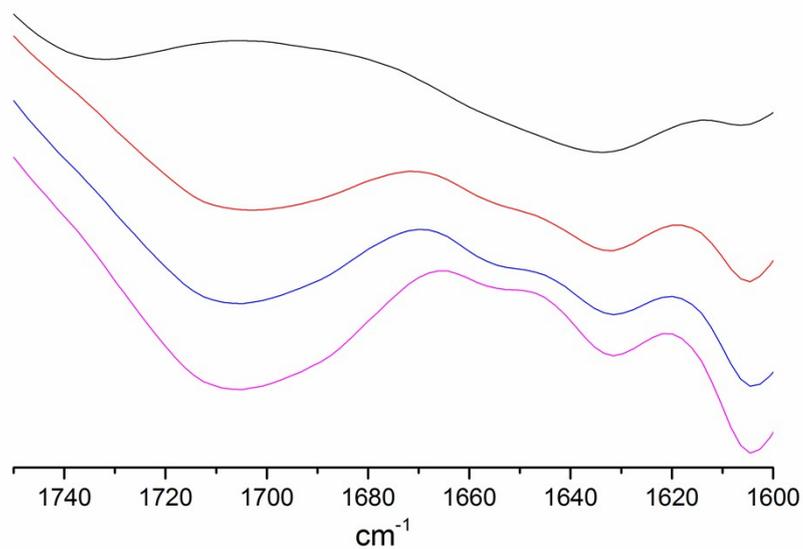


Fig. S2 The FTIR spectrum of UC (black), SER (red), CRF (blue), LRF (pink).

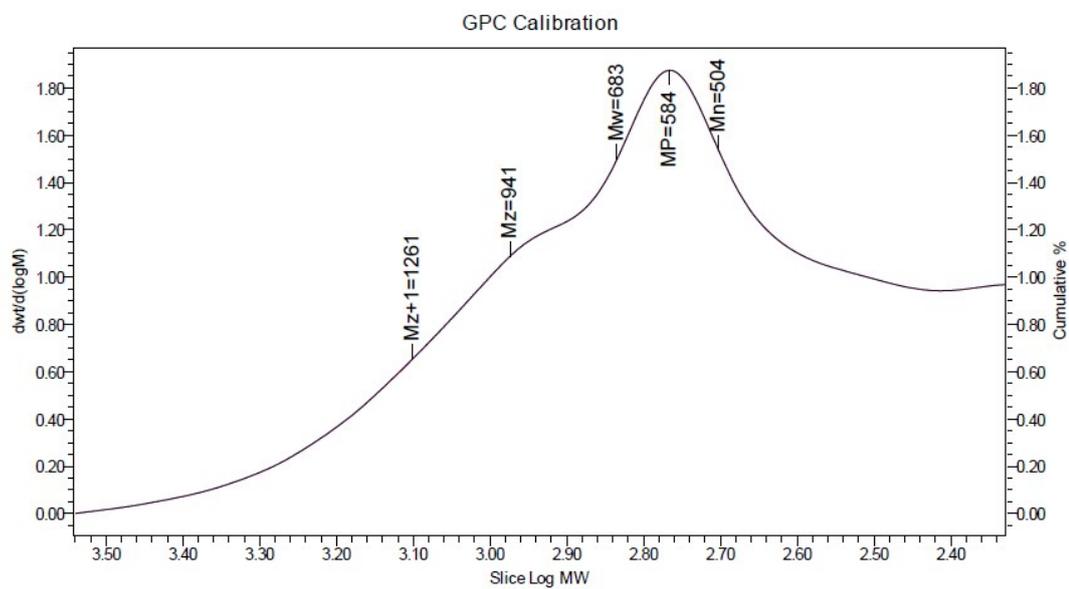


Fig. S3 Gel permeation chromatograms of lignin rich fraction (LRF) in water phase measured with UV detector.

Table. S1. Assignments of ^{13}C - ^1H correlation signals in the HSQC spectrum of the LRF.

Lable	$\delta\text{C}/\delta\text{H}$ (ppm)	Assignment
$-\text{OCH}_3$	56.0/3.72	C-H in methoxyls (MeO)
I_γ	61.4/4.05	$\text{C}_\gamma\text{-H}_\gamma$ in cinnamyl alcohol ending groups
A'_γ	62.7/4.28	$\text{C}_\gamma\text{-H}_\gamma$ in γ -acylated β -O-4 linkages
$\text{S}_{2,6}$	104.1/6.67	$\text{C}_{2,6}\text{-H}_{2,6}$ in syringyl unit(S unit)
G_2	111.3/7.02	$\text{C}_2\text{-H}_2$ in guaiacyl unit (G unit)
G_5	115.4/6.69	$\text{C}_5\text{-H}_5$ in guaiacyl units (G unit)
G_6	119.0/6.79	$\text{C}_6\text{-H}_6$ in guaiacyl units (G unit)
G'_6	123.3/7.60	$\text{C}_6\text{-H}_6$ in oxidized ($\text{C}_\alpha=\text{O}$) guaiacyl unit
P_7	144.1/7.43	$\text{C}_7\text{-H}_7$ in <i>p</i> -coumaroylated substructure
$\text{P}_{2,6}$	130.2/7.48	$\text{C}_{2,6}\text{-H}_{2,6}$ in <i>p</i> -coumaroylated substructure
$\text{P}_{3,5}$	115.4/6.78	$\text{C}_3\text{-H}_3, \text{C}_5\text{-H}_5$ in <i>p</i> -coumaroylated substructure
$\text{H}_{2,6}$	128.2/7.16	$\text{C}_{2,6}\text{-H}_{2,6}$ in <i>p</i> -hydroxyphenyl unit