

Supplementary materials

Study of Purified Cellulosic Pulp and Lignin Produced by Wheat Straw Biorefinery

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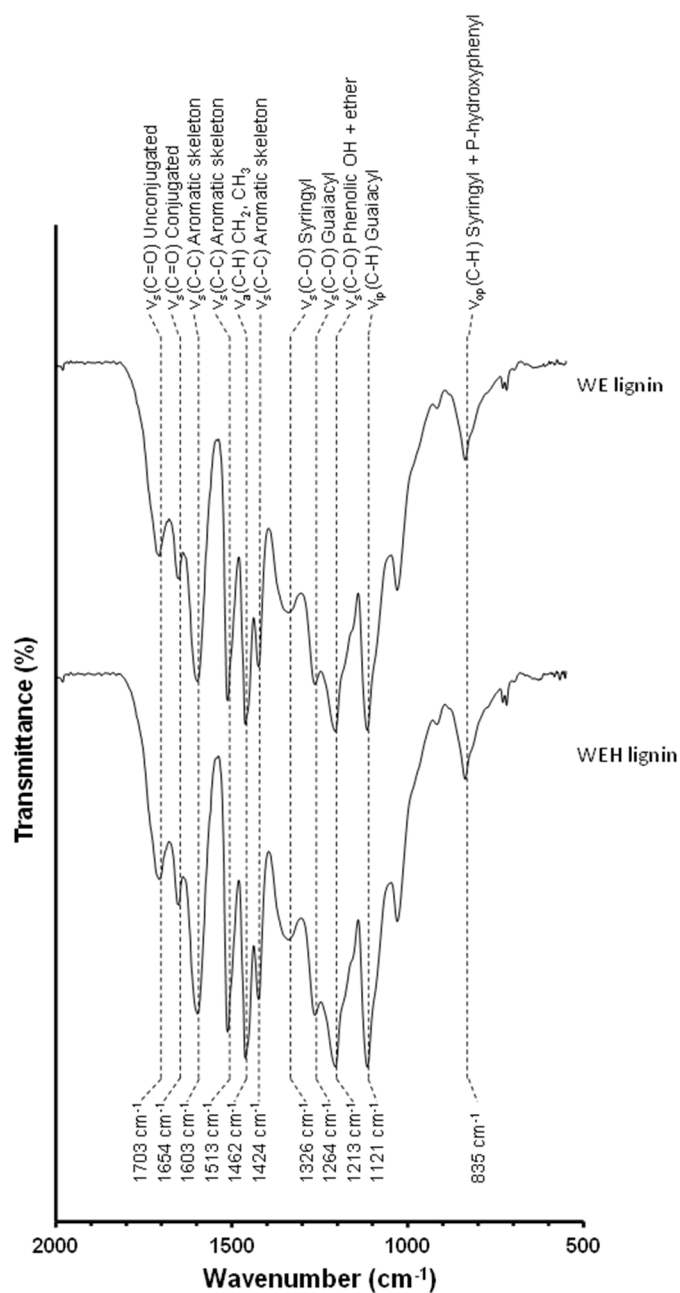


Figure S1 Fourier transform infrared (FTIR) spectra of organosolv lignins isolated from pre-extracted and hydrolyzed wheat straw (WEH) and extractive-free wheat straw (WE): stretching of the C=O bonds within the unconjugated and conjugated carbonyl and carboxyl groups (1703 cm^{-1} and 1654 cm^{-1}), stretching of the C-C bonds within the aromatic skeleton (1603 cm^{-1} , 1513 cm^{-1} and 1424 cm^{-1}), asymmetric deformation of the C-H bonds in CH_2 and CH_3 (1462 cm^{-1}), stretching of C-O bonds within syringyl and guaiacyl units (1326 cm^{-1} and 1264 cm^{-1}), stretching of C-O bonds of phenolic OH and aromatic ether (1213 cm^{-1}), in plane deformation of C-H within syringyl and guaiacyl units (1121 cm^{-1}), out of plane deformation of C-H within syringyl and p-hydroxyphenyl units (836 cm^{-1})

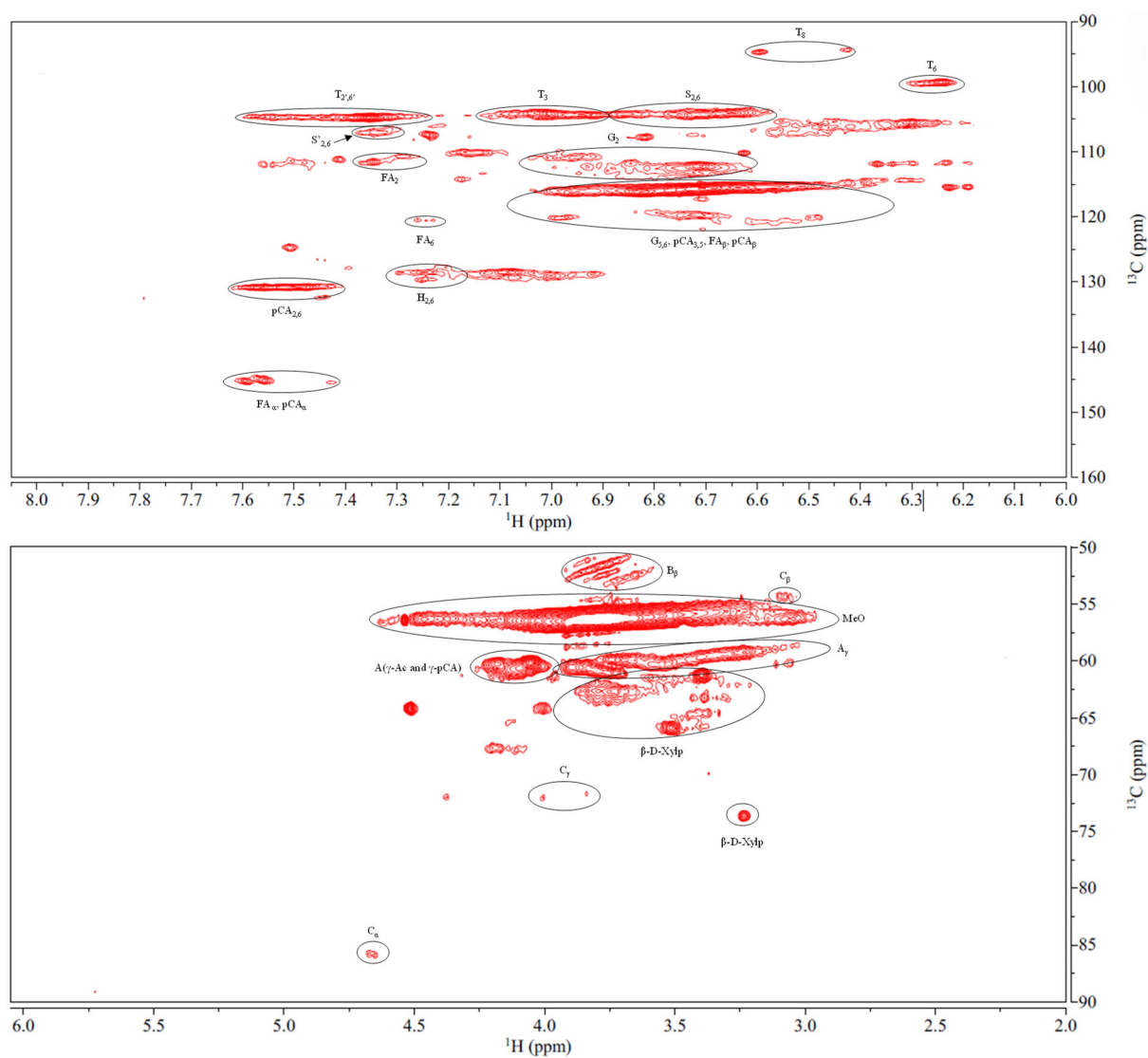


Figure S2 2D NMR spectra of organosolv lignin isolated from WEH: Aromatic regions ($\delta\text{C}/\delta\text{H}$ 90–160/6.0–8.0 ppm) and side chains ($\delta\text{C}/\delta\text{H}$ 50–90/2.0–6.0 ppm)

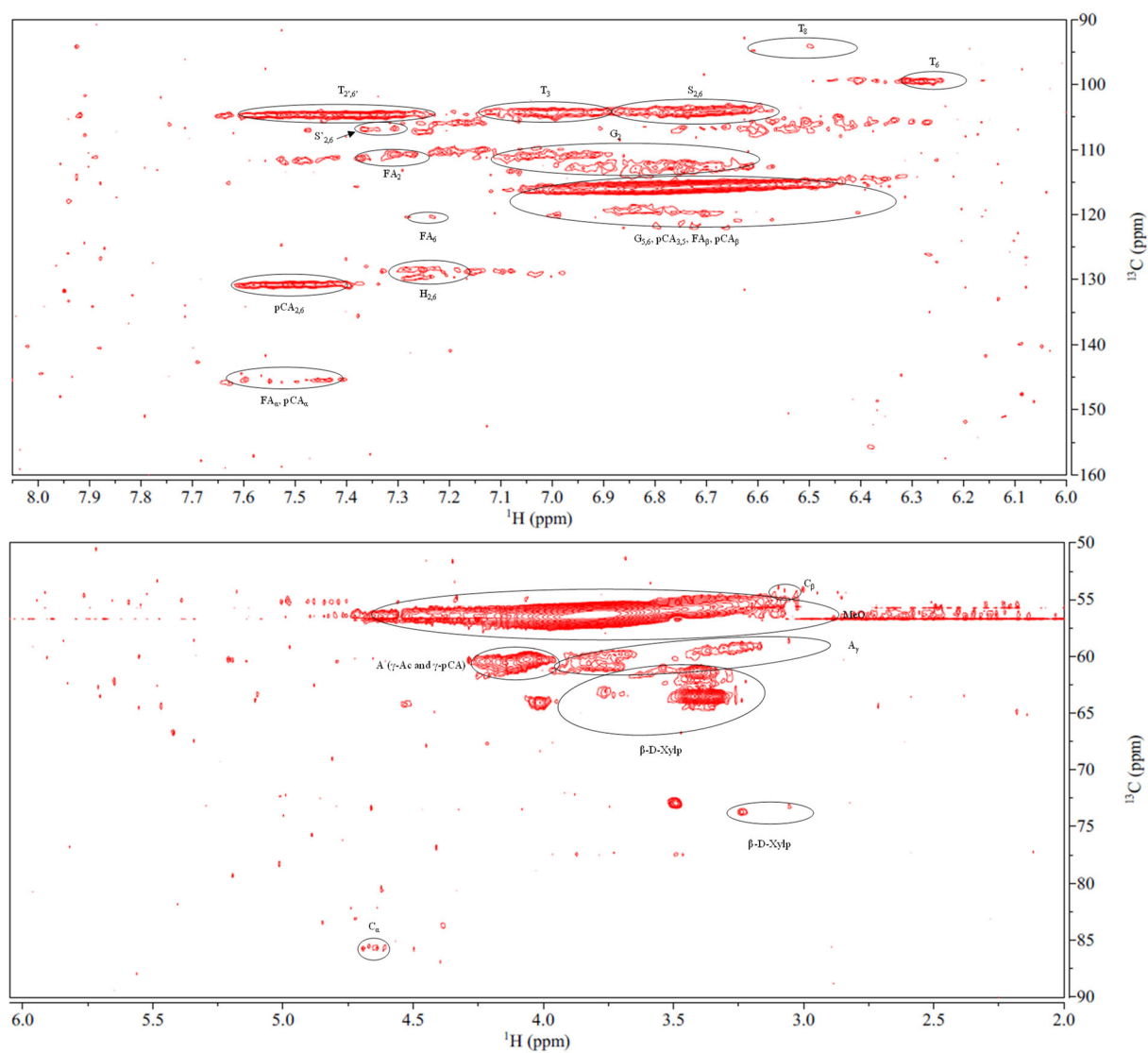


Figure S3 2D NMR spectra of organosolv lignin from WE: Aromatic regions ($\delta\text{C}/\delta\text{H}$ 90-160/6.0-8.0 ppm) and side chains ($\delta\text{C}/\delta\text{H}$ 50-90/2.0-6.0 ppm)

Table S1 Assignment of $^1\text{H}/^{13}\text{C}$ correlation signals in the 2D HSQC spectra of organosolv lignin from WEH

Legend	$\delta\text{C}/\delta\text{H}$ (ppm)	Signal assignment
C_β	54.35/3.10	$\text{C}_\beta\text{-H}_\beta$ in $\beta\text{-}\beta'$ pino/resinols (C)
A_γ	60.31/3.58	$\text{C}_\gamma\text{-H}_\gamma$ in $\beta\text{-O-4}$ β -aryl ether
B_β	52.30/3.76	$\text{C}_\beta\text{-H}_\beta$ in $\beta\text{-5'}$ phenylcoumarans (B)
MeO	56.31/3.76	C-H in methoxyls
A ($\gamma\text{-Ac}$ and $\gamma\text{-pCA}$)	60.12/4.05	$\text{C}_\gamma\text{-H}_\gamma$ in γ -acylated $\beta\text{-O-4}$ substructures (acetate and pCA)
C_γ	71.54/4.22	$\text{C}_\gamma\text{-H}_\gamma$ in $\beta\text{-}\beta'$ pino/resinols (C)
C_α	85.80/4.67	$\text{C}_\alpha\text{-H}_\alpha$ in $\beta\text{-}\beta'$ pino/resinols (C)
T_6	99.37/6.24	$\text{C}_6\text{-H}_6$ in Tricin units (T)
G_6	120.48/6.55	$\text{C}_6\text{-H}_6$ in Guaiacyl units (G)
T_8	94.78/6.60	$\text{C}_8\text{-H}_8$ in Tricin units (T)
$\text{S}_{2,6}$	104.16/6.68	$\text{C}_2\text{-H}_2$ and $\text{C}_6\text{-H}_6$ syringyl units (S)
$\text{pCA}_{3,5}$	119.69/6.71	$\text{C}_3\text{-H}_3$ and $\text{C}_5\text{-H}_5$ in <i>p</i> -coumarate (pCA)
G_2	112.17/6.72	$\text{C}_2\text{-H}_2$ in Guaiacyl units (G)
G_5	115.68/6.73	$\text{C}_5\text{-H}_5$ in Guaiacyl units (G)
FA_β , pCA_β	120.08/6.97	$\text{C}_\beta\text{-H}_\beta$ in Ferulate (FA) and <i>p</i> -coumarate (pCA)
T_3	104.16/7.01	$\text{C}_3\text{-H}_3$ in Tricin units (T)
$\text{H}_{2,6}$	128.48/7.08	$\text{C}_2\text{-H}_2$ and $\text{C}_6\text{-H}_6$ in <i>p</i> -hydroxyphenyl units (H)
$\text{S}'_{2,6}$	107.48/7.24	$\text{C}_2\text{-H}_2$ and $\text{C}_6\text{-H}_6$ in C_α -oxidized ($\text{C}_\alpha=\text{O}$) phenolic, syringyl units (S')
FA_6	120.47/7.26	$\text{C}_6\text{-H}_6$ in Ferulate (FA)
$\text{T}_{2',6'}$	104.75/7.35	$\text{C}_{2'}\text{-H}_{2'}$ and $\text{C}_{6'}\text{-H}_{6'}$ in Tricin units (T)
FA_2	111.58/7.35	$\text{C}_2\text{-H}_2$ in Ferulate (FA)
$\text{pCA}_{2,6}$	130.82/7.56	$\text{C}_2\text{-H}_2$ and $\text{C}_6\text{-H}_6$ in <i>p</i> -coumarate (pCA)
FA_α , pCA_α	145.18/7.56	$\text{C}_\alpha\text{-H}_\alpha$ in Ferulate (FA) and <i>p</i> -coumarate (pCA)

Table S2 GPC analysis data of wheat straw lignin samples

Sample	Mn (g/mol)	Mw (g/mol)	PDI
WEH ^I	702 ± 37	1924 ± 256	2.7 ± 0.2
WEH ^{II}	689 ± 82	1908 ± 545	2.7 ± 0.4
WEH ^{III}	498 ± 27	1181 ± 84	2.37 ± 0.04
WEH ^{IV}	405 ± 14	984 ± 61	2.43 ± 0.07

^I Organosolv lignin isolated from extractives-free hydrolyzed wheat straw (WEH^I) obtained at 160°C, ^{II} Organosolv lignin isolated from extractives-free hydrolyzed wheat straw (WEH^{II}) obtained at 180°C (standard parameters established by Koumba-Yoya and Stevanovic (2016), ^{III} Organosolv lignin isolated from extractives-free hydrolyzed wheat straw (WEH^{III}) obtained at 200°C, ^{IV} Extractives-free hydrolyzed wheat straw lignin (WEH^{IV}) n obtained at 220°C

Table S3 Characterization of cotton, Avicel®, and FiloCell® cellulose samples

Characterization	Cotton	Avicel®	FiloCell®
DP (-)	2062 ± 356	157.5 ± 0.8	593 ± 9
Mw (Da)	334408 ± 57808	25530 ± 126	96163 ± 1406
Crystallinity (¹³ C)	33%	56%	32%
Crystallinity (XRD)	-	80%	92%
Crystallite size (Å)	-	36 ± 3	45 ± 2
Iα (FT-IR)	-	-	-
Iβ (FT-IR)	100%	100%	100%

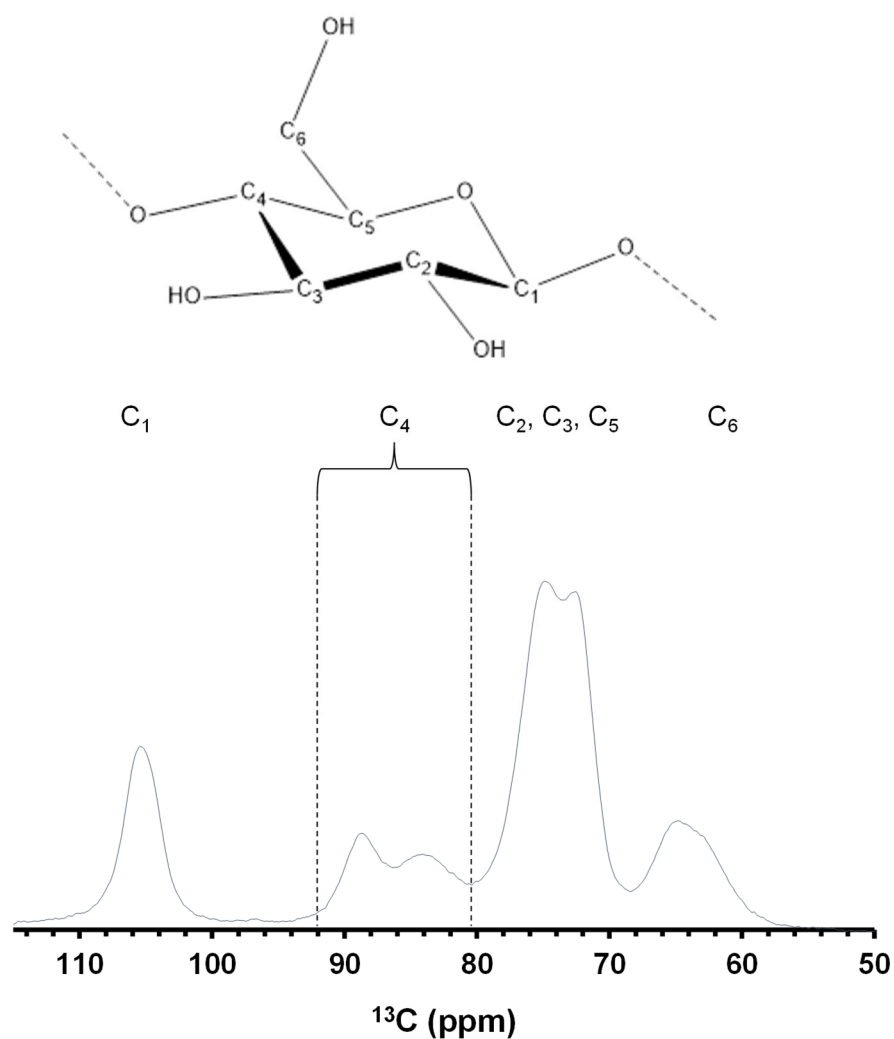


Figure S4 MAS/ ^{13}C NMR spectra of bleached organosolv cellulosic pulp from pre-hydrolyzed wheat straw

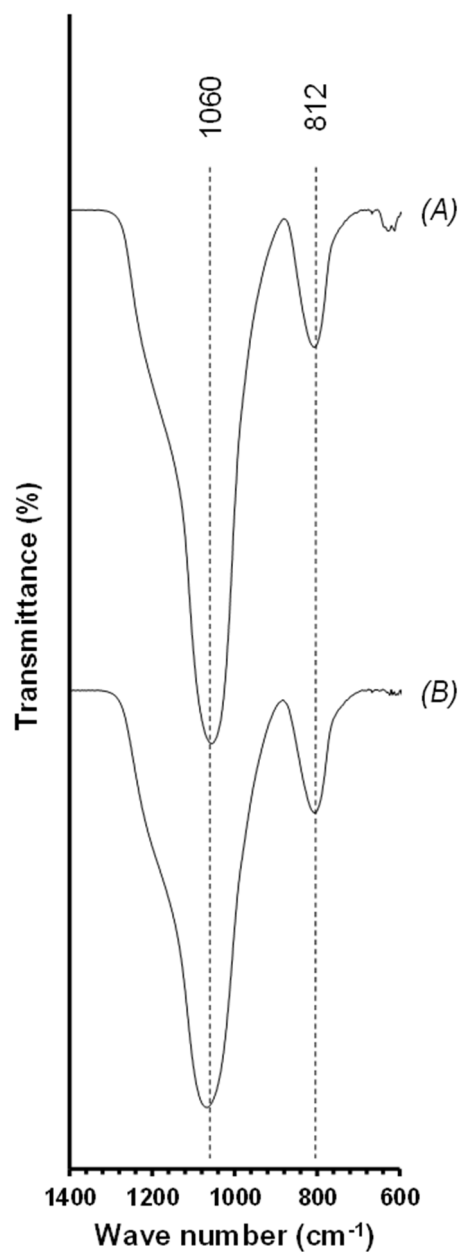


Figure S5 Fourier transform infrared (FTIR) spectrum of silica extracted from wheat straw pulp with the asymmetric stretching vibration of Si-O-Si (1060 cm^{-1}) and symmetric bending vibration of Si-O (812 cm^{-1}): (A) silica produced from sodium silicate solution extracted from wheat straw pulp and (B) commercial silica from Sigma-Aldrich