New lignin streams derived from heteropoly acids-enhanced neutral deep eutectic solvent fractionation: Towards structural elucidation and antioxidant performance

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Fig. S1. Photographs of lignin solubility in ethanol, tetrahydrofuran, dimethylacetamide solvents: (a) low solubility, (b) moderate solubility, (c) good solubility.

Table S1

The solubility of lignin fractions.

Solvents	HPA-DES lignin		Alkali Lignin		Lignosulphonate	
	Solubility (mg/mL)	Solubility (wt %)	Solubility (mg/mL)	Solubility (wt %)	Solubility (mg/mL)	Solubility (wt %)
Methanol	<1	<0.13	<1	<0.13	<1	<0.13
Ethanol	< 1	<0.13	<1	<0.13	<1	<0.13
Acetone	< 1	<0.13	<1	<0.13	<1	<0.13
Deionized water	< 1	<0.10	<3	<3.00	> 100	> 10
Ethylacetate	< 1	<0.11	<1	<0.11	<1	<0.11
Cyclohexane	< 1	<0.13	<1	<0.13	<1	<0.13
Phenylcarbinol	< 1	<0.10	<1	<0.10	<1	<0.10
Dichloroethane	< 1	<0.08	<1	<0.08	<1	<0.08
1,4-dioxane	< 1	<0.10	<1	<0.10	<1	<0.10
Tetrahydrofuran	4	0.45	4	0.45	3	0.34
Dimethyl sulfoxide	> 100	> 9.1	> 70	> 6.4	> 50	> 4.5
Dimethylacetamide	> 100	> 10.7	> 80	> 8.5	> 60	> 6.4

Table S2

Assignments of ^{13}C-^{1}H correlated signals in the HSQC spectra of the lignin from bamboo.

Labels	δ_C/δ_H (ppm)	Assignment
C_β	53.8/3.71	C_β-H_β in phenylcomaran structures(C)
B_β	53.8/3.07	C_β-H_β in resinol substructures(B)
-OCH_3	55.2/3.59	C-H in methoxyls
A_γ	59.5/3.62	C_γ-H_γ in *β*-*O*-4 substructures(A)
I_γ	62.3/4.09	C_γ-H_γ in cinnamyl(sinapyl/coniferyl) alcohol end groups(I)
X_5	62.8/3.25	C_5-H_5 *β*-D-xylopyranoside substructures(X)
B_γ	71.1/3.70 and	C_γ-H_γ in resinol substructures(B)

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A_{α}	71.3/4.86	C_{α}-H_{α} in *β-O*-4 substructures(A)
X_2	73.0/3.10	C_2-H_2 β-D-xylopyranoside substructures(X)
X'_2	73.1/4.46	C_2-H_2 in 2-O-Ac-β-D-xylopyranoside substructures(X)
X_3	74.1/3.26	C_3-H_3 β-D-xylopyranoside substructures(X)
X_4	76.4/3.63	C_4-H_4 β-D-xylopyranoside substructures(X)
D'_{α}	79.4/4.01	C_{α}-H_{α} in spirodienone substructures(D')
A'_{β}(G)	80.8/4.61	C_{β}-H_{β} in *β-O*-4 linked to a G (H)
B'_{α}	83.0/4.91	C_{α}-H_{α} in *β* - *β* (B′, tetrahydrofuran)
A_{β}(G)	83.2/4.34	C_{β}-H_{β} in *β-O*-4 linked to a G (H)
B_{α}	84.8/4.62	C_{α}-H_{α} in resinol substructures(B)
A_{β}(S)	85.7/4.10	C_{β}-H_{β} in *β-O*-4 linked to a S unit(A)
C_{α}	87.5/5.48	C_{α}-H_{α} in phenylcomaran structures(C)
$S_{2,6}$	103.7/6.59	$C_{2,6}$-$H_{2,6}$ in syringyl units(S)
$T'_{2,6}$	105.4/7.34	$C_{2,6}$-$H_{2,6}$ in tricin (T)
G_2	111.1/6.94	C_2-H_2 in guaiacyl units(G)
FA_2	111.3/7.32	C_2-H_2 in ferulate (FA)
PCA_{β}/FA_{β}	113.6/6.23	C_{β}-H_{β} in p-coumarate (PCA) and ferulate (FA)
G_5	114.5/6.70	C_5-H_5 in guaiacyl units(G)
$PCA_{3,5}$	115.8/6.68	$C_{3,5}$-$H_{3,5}$ in p-coumarate (PCA)

G_6	119.1/6.78	C_6-H_6 in guaiacyl units(G)
FA_6	122.5/7.13	$C_{2,6}$-$H_{2,6}$ in ferulate (FA)
J_β	126.1/6.72	C_β-H_β in cinnamyl aldehyde end-group (J)
$H_{2,6}$	128.0/7.22	$C_{2,6}$-$H_{2,6}$ in *p*-hydroxyphenyl units(H)
$PCA_{2,6}$/$PCA_{2,6}$	130.1/7.48	$C_{2,6}$-$H_{2,6}$ in *p*-coumarate (PCA)
PCA_α/FA_α	144.6/7.46	C_α-H_α in p-coumarate (PCA) and ferulate (FA)

Fig. S1. Photographs of lignin solubility in ethanol, tetrahydrofuran, dimethylacetamide solvents: (a) low solubility, (b) moderate solubility, (c) good solubility.