Electronic Supporting Information for

Feedstock-agnostic reductive catalytic fractionation in alcohol and alcohol-water mixtures

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Table S1. Compositional analysis of parent and post-RCF pulp samples in methanol or methanol/water mixture.

Sample		Water	Ash	Extractives	Protein	Free sugars	Lignin	Glucan	Xylan	Galactan	Arabinan	Mannan	Acetyl	Total	Isolated sample	Delignification[b]	Glucan retention rate[c]	Xylan retention rate[d]
Poplar	Native (As-received)	3.0%	0.6%	3.7%	0.4%	0.0%	25.2%	43.9%	12.8%	1.3%	0.1%	2.7%	3.7%	97.4%	2.70 g	-	-	-
	Native (Dried)[a]	-	0.6%	3.8%	0.4%	0.0%	26.0%	45.3%	13.2%	1.3%	0.1%	2.8%	3.8%	97.3%	2.62 g	-	-	-
	MeOH	0.0%	0.8%	0.0%	0.0%	0.0%	13.7%	65.4%	19.7%	1.4%	0.0%	3.4%	0.1%	104.5%	1.78 g	64.0%	98.4%	101.4%
	MeOH/H_2O	0.0%	8.4%	0.0%	0.0%	0.0%	4.2%	83.3%	4.3%	0.8%	0.0%	2.7%	0.0%	103.7%	1.23 g	92.5%	86.1%	15.2%
Switchgrass	Native (As-received)	5.7%	4.4%	7.7%	0.0%	3.4%	16.1%	31.8%	20.4%	1.3%	3.0%	0.0%	2.2%	96.0%	2.70 g	-	-	-
	Native (Dried)[a]	-	4.6%	8.1%	0.0%	3.6%	17.1%	33.8%	21.6%	1.3%	3.1%	0.0%	2.4%	95.6%	2.55 g	-	-	-
	MeOH	0.0%	5.5%	0.0%	0.0%	0.0%	8.6%	48.6%	30.7%	1.6%	3.8%	0.0%	0.0%	98.8%	1.80 g	64.6%	101.7%	100.2%
	MeOH/H_2O	0.0%	6.1%	0.0%	0.0%	0.0%	2.5%	79.7%	10.0%	0.8%	0.4%	0.0%	0.1%	99.6%	0.96 g	94.5%	88.5%	17.4%
Corn stover	Native (As-received)	3.0%	1.9%	8.3%	0.0%	0.4%	17.5%	37.0%	21.4%	1.0%	2.1%	0.0%	2.1%	94.7%	2.70 g	-	-	-
	Native (Dried)[a]	-	1.9%	8.5%	0.0%	0.4%	18.0%	38.2%	22.1%	1.0%	2.1%	0.0%	2.2%	94.4%	2.62 g	-	-	-
	MeOH	0.0%	2.8%	0.0%	0.0%	0.0%	8.0%	54.2%	31.1%	1.7%	3.4%	0.0%	0.1%	101.3%	1.72 g	70.9%	93.6%	92.9%
	MeOH/H_2O	0.0%	9.3%	0.0%	0.0%	0.0%	2.9%	81.3%	10.2%	0.8%	0.4%	0.0%	0.1%	105.0%	1.24 g	92.5%	101.0%	21.9%
Pine	Native (As-received)	3.9%	0.8%	4.7%	0.0%	0.1%	32.2%	36.0%	6.6%	2.4%	2.3%	10.9%	1.2%	101.1%	2.70 g	-	-	-
	Native (Dried)[a]	-	0.9%	4.9%	0.0%	0.1%	33.5%	37.4%	6.8%	2.5%	2.4%	11.4%	1.2%	101.1%	2.60 g	-	-	-
	MeOH	0.0%	0.7%	0.0%	0.0%	0.0%	29.2%	50.9%	9.6%	2.4%	1.5%	11.5%	0.0%	105.8%	2.00 g	32.9%	104.9%	108.1%
	MeOH/H_2O	0.0%	5.5%	0.0%	0.0%	0.0%	16.3%	72.3%	4.8%	0.9%	0.2%	5.6%	0.0%	105.6%	1.18 g	77.9%	88.0%	31.9%

[a]Sample after drying in a 120°C overnight. [b]Calculated using **Equation 3**. [c]Calculated using **Equation 4**. [d]Calculated using **Equation 5**.

Table S2. Phenolic monomer and lignin oil yields from RCF of poplar, pine, switchgrass, and corn stover.

Temp.	Biomass	Solvent	Oil yield[a] (wt% total lignin)	Monophenolic monomer yield (wt% total lignin)[b]													
				MG	EG	PG	P(ene)G	P(OH)G	PS	P(ene)S	P(OH)S	Phenol	E(OH)	HC[c,d]	HF		total
															HFA[c]	MHF/EHF[c]	
230°C	Poplar	MeOH	65.1±8.7	0.0	0.0	8.3	2.5	2.1	14.1	0.7	3.7	1.2	0.0	0.0	0.0	0.0	32.6±2.7
		EtOH	60.8±3.3	0.0	0.5	5.2	0.4	3.7	10.3	0.3	4.8	1.8	0.0	0.0	0.0	0.0	27.0±1.7
		MeOH/H_2O	71.4±8.8	0.0	0.4	8.3	0.6	3.1	14.8	0.4	3.6	2.2	0.0	0.0	0.0	0.0	33.4±0.7
		EtOH/H_2O	73.8±0.8	0.0	0.4	4.9	0.3	6.9	8.6	0.2	9.8	1.5	0.0	0.0	0.0	0.0	32.6±0.6
	Switchgrass	MeOH	81.4±11.6	0.0	0.8	6.6	0.0	2.3	6.3	0.0	2.8	0.0	2.0	5.7	0.0	5.1	31.6±1.6
		EtOH	98.8±5.8	0.0	0.7	6.2	0.0	2.9	5.7	0.0	2.9	0.0	1.7	5.1	1.7	3.4	30.3±0.1
		MeOH/H_2O	96.7±13.3	0.3	1.6	4.9	0.0	3.6	6.4	0.0	3.4	0.0	2.6	5.3	2.2	3.3	33.5±1.8
		EtOH/H_2O	119.9±21.3	0.0	0.8	3.9	0.0	4.6	5.2	0.0	4.9	0.0	1.8	4.7	2.9	1.7	30.5±0.4
	Corn stover	MeOH	82.8±3.2	0.0	0.4	3.5	0.0	1.5	5.5	0.0	1.9	0.0	4.2	8.5	0.0	6.6	32.1±1.5
		EtOH	74.7±16.1	0.0	0.7	3.1	0.0	1.5	4.4	0.0	2.0	0.0	4.1	9.0	0.0	5.6	30.4±1.7
		MeOH/H_2O	104.5±24.2	0.0	1.4	2.9	0.0	2.6	6.1	0.0	2.0	0.0	4.5	6.7	1.8	3.0	31.0±0.6
		EtOH/H_2O	114.0±12.1	0.0	1.0	2.0	0.0	3.2	5.6	0.0	2.3	0.0	3.6	7.4	3.0	2.4	30.5±3.3
	Pine	MeOH	42.5±2.3	0.0	0.3	10.4	0.3	1.7	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	12.7±0.2
		EtOH	41.0±3.2	0.0	0.3	8.3	0.2	1.8	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	10.6±0.6
		MeOH/H_2O	76.2±1.1	0.2	1.2	5.9	0.0	9.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	16.3±0.7
		EtOH/H_2O	74.1±5.0	0.3	0.7	9.4	0.0	6.9	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	17.3±1.7

[a]Calculated using **Equation 1**. [b]Calculated using GC-FID analysis and **Equation 2**. [c]Monomer yield was calculated using NMR analysis with 1,3,5-tri-*tert*-butylbenzene as an internal standard. [d]A combined yield of dihydrocoumaric acid and methyl (or ethyl) dihydrocoumarate due to significant overlap of their NMR peaks.

MG (methyl-guaiacol). EG (ethyl-guaiacol). PG (propyl-guaiacol). P(ene)G (propylene-guaiacol). P(OH)G (propanol-guaiacol). PS (propyl-syringol). P(ene)S (propylene-syringol). P(OH)S (propanol-syringol). HC (dihydrocoumaric acid + methyl-dihydrocoumarate + ethyl-dihydrocoumarate). HFA (dihydroferulic acid). MHF (methyl-dihydroferulate). EHF (ethyl-dihydroferulate).

Table S3. Phenolic monomer and lignin oil yields on a biomass basis from FT-RCF of poplar, pine, switchgrass, and corn stover.

Biomass	Solvent	Oil yield[a] (wt% total lignin)	Monophenolic monomer yield (wt% total lignin)[b]													
			MG	EG	PG	P(ene)G	P(OH)G	PS	P(ene)S	P(OH)S	Phenol	E(OH)	HC[c,d]	HF		total
														HFA[c]	MHF	
Poplar	MeOH	68.2±7.0	0.1	0.2	2.8	1.6	5.3	4.2	0.0	11.0	0.0	0.0	0.0	0.0	0.0	25.2±1.7
	MeOH/H_2O	75.7±3.2	0.0	0.0	0.2	0.1	5.4	1.4	0.7	7.2	0.0	0.0	0.0	0.0	0.0	15.0±1.1
Switchgrass	MeOH	88.9±12.4	0.0	0.0	1.6	0.3	3.6	1.6	0.0	5.4	0.0	1.2	6.2	0.0	7.1	27.0±1.4
	MeOH/H_2O	104.9±7.0	0.0	0.0	0.0	0.0	3.6	1.0	0.0	4.3	0.0	2.1	8.6	3.3	5.4	28.3±1.6
Corn stover	MeOH	94.2±7.0	0.0	0.0	1.3	0.5	3.0	2.2	0.0	5.0	0.0	3.4	11.0	0.0	9.4	35.8±1.6
	MeOH/H_2O	96.7±5.5	0.0	0.0	0.0	0.0	2.0	0.6	0.0	2.4	0.0	1.2	15.0	4.9	5.4	31.5±0.7
Pine	MeOH	36.1±1.6	0.0	0.0	2.6	0.0	3.9	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	6.5±0.1
	MeOH/H_2O	55.9±8.1	0.0	0.0	0.2	0.0	6.1	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	6.3±0.5

[a]Calculated using **Equation 1**. [b]Calculated using GC-FID analysis and **Equation 2**. [c]Quantified by comparing with the NMR peak area of MHF which was pre-quantified with GC-FID and used as an internal standard. [d]A combined yield of dihydrocoumaric acid and methyl-dihydrocoumarate due to significant overlap of their NMR peaks.
EG (ethyl-guaiacol). PG (propyl-guaiacol). P(ene)G (propylene-guaiacol). P(OH)G (propanol-guaiacol). PS (propyl-syringol). P(ene)S (propylene-syringol). P(OH)S (propanol-syringol). HC (dihydrocoumaric acid + methyl-dihydrocoumarate). HFA (dihydroferulic acid). MHF (methyl-dihydroferulate).

Table S4. Summary of delignification, glucan/xylan retention, monomer yield, and oil yield.

Solvent	Biomass	Delignification	Glucan retention	Xylan retention	Oil yield[a] (wt% total lignin)	Oil yield (wt% dried biomass)	Pulp yield (wt% dried biomass)	Monomer yield (wt% total lignin)[b]
MeOH	Poplar	64.0%	98.4%	101.4%	68.2	17.7	67.9	25.2
	Switchgrass	64.6%	101.7%	100.2%	88.9	15.2	70.5	27.0
	Corn stover	70.9%	93.6%	92.9%	94.2	17.0	65.6	35.8
	Pine	32.9%	104.9%	108.1%	36.1	12.1	76.9	6.5
MeOH/H_2O	Poplar	92.5%	86.1%	15.2%	75.7	19.7	46.9	15.0
	Switchgrass	94.5%	88.5%	17.4%	104.9	17.9	37.6	28.3
	Corn stover	92.5%	101.0%	21.9%	96.7	17.4	47.3	31.5
	Pine	77.9%	88.0%	31.9%	55.9	18.7	45.4	6.3

[a]Calculated using **Equation 1**. [b]Calculated using **Equation 2**.

Table S5. Summary of monomer yield, oil yield, and pulp yield of batch and FT-RCF in literature and this study.

Entry	Reaction type	Biomass	Solvent	Conditions	Monomer yield (wt% total lignin)[a]	Oil yield (wt% total lignin)[a]	Oil yield (wt% biomass)	Pulp yield (wt% biomass)	Ref.
1	Batch	Poplar	MeOH	30 bar H_2, 215°C, 15wt% Ni/C	20.8	60.7	15.8	-	[1]
2			MeOH	30 bar H_2, 200°C, 5 wt% Pd/C	28.2[b]	52.0[c]	11.2	-	[2]
3			MeOH	30 bar H_2, 230°C, 5 wt% Ru/C	32.6	65.1	16.9	-	This study
3			MeOH/H_2O (1:1 v/v)	30 bar H_2, 200°C, 5 wt% Pd/C	41.6[b]	80.0[c]	17.3	-	[2]
4			MeOH/H_2O (1:1 w/w)	30 bar H_2, 230°C, 5 wt% Ru/C	33.4	71.4	18.5		This study
5			2-PrOH/H_2O (7:3 v/v)	220°C, Raney Ni	-	-	26	52	[3]
6		Pine + spruce	MeOH	30 bar H_2, 250°C, 5 wt% Ru/C	21.0	56.0[c]	15.2	-	[4]
7		Pine	MeOH	30 bar H_2, 220°C, Ru/CNT	11.1	78.8[d]	-	-	[5]
8			MeOH	30 bar H_2, 230°C, 5 wt% Ru/C	12.7	42.5	14.2	-	This study
9			EtOH/H_2O (1:1 v/v)	210°C, Pd/C (5 mol%)	9.3	84.6[d]	-	41.1	[6]
10			MeOH/H_2O (1:1 w/w)	30 bar H_2, 230°C, 5 wt% Ru/C	16.3	76.2	25.5	-	This study
11		Corn Stover	MeOH	30 bar H_2, 200°C, 5 wt% Ni/C	28.6	59.0	8.0	73.0	[7]
12			MeOH	30 bar H_2, 230°C, 5 wt% Ru/C	32.1	82.8	14.9	-	This study
13			MeOH/H_2O (1:1 w/w)	30 bar H_2, 230°C, 5 wt% Ru/C	31.0	104.5	18.8	-	This study
14		Switchgrass	MeOH	40 bar H_2, 250°C, Ru/C	41.5	-	-	31.0	[8]
15			MeOH	30 bar H_2, 230°C, 5 wt% Ru/C	31.6	81.4	13.9		This study
16			MeOH/H_2O (1:1 w/w)	30 bar H_2, 230°C, 5 wt% Ru/C	33.5	96.7	16.5		This study
17		Miscanthus	MeOH	30 bar H_2, 220°C, Ru/CNT	26.0	84.6[d]	-	-	[5]
18	FT	Poplar	MeOH	110 bar H_2 200 sccm, feed 2 mL/min, 225°C, 15 wt% Ni/C	22.0	54.0	14.0	67.3	[9]
19			MeOH	110 bar H_2 200 sccm, feed 2 mL/min, 225°C, 5 wt% Ru/C	25.2	68.2	17.7		This study
20			MeOH/H_2O (1:1 v/v)	110 bar H_2 200 sccm, feed 2 mL/min, 225°C, 5 wt% Ru/C	19.0	92.9[d]	-	48.6	[9]
21			MeOH/H_2O (1:1 w/w)	110 bar H_2 200 sccm, feed 2 mL/min, 225°C, 5 wt% Ru/C	15.0	75.7	19.7		This study
22		Birch	MeOH/H_2O (7:3 v/v), 2.8 g/L H_3PO_4	Feed 0.3 mL/min, 180°C (biomass bed), 200°C (catalyst bed), 5 wt% Pd/C	39.0	51.9	14.0	39.0	[10]

[a]Calculated based on total lignin [b,c]Calculated based on Klason lignin [d]Delignification calculated using lignin residual in pulp

Quantification of hydroxycinnamate products

Hydroxycinnamate products from batch RCF reactions with switchgrass and corn stover were quantified via ^{1}H NMR using 1,3,5 tri-*tert*-butylbenzene (δ = 7.28, 3H, TTB) as an internal standard. From the RCF reaction mixture, 5 mL were dried using rotary evaporation to yield the RCF oil. Then, 1 mL of 1 g/L TTB was added to resolubilize, and the solution was filtered with a 0.2 μm filter and transferred to an NMR tube. A ^{1}H spectrum was acquired on a Bruker Neo Avance 400 MHz instrument equipped with a nitrogen cooled Prodigy cryoprobe using 32 scans, a sweep width of 20 ppm, and a delay of 3 seconds. Processing was performed on TopSpin 3.61 using a line broadening of 0.3 Hz. Figures were created with MestreNova.

The absence of double bond-containing products such as *p*-coumaric acid, ferulic acid, and esterified analogues was verified by the lack of resonances at δ = 7.54 ppm (coumarates) and δ = 7.33 ppm (ferulates). This indicates the complete hydrogenation of double bonds in hydroxycinnamates during RCF. Significant overlap between the dihydrocoumaric acid and the corresponding ester was observed, and therefore a single integration for both products is performed to quantify the combined yield. Dihydroferulic acid and its corresponding ester are well enough resolved to integrate both separately. The yield was calculated using the molecular weights of coumaric and ferulic acid so as not to bias the yields based on esterification/hydrogenation.

For RCF reactions in methanol, only the methyl esters were observed. However, for reactions in a methanol/water mixture, both the ester and acid products were present. Switchgrass and corn stover behaved differently ethanol reactions: only ethyl esters were observed for corn stover, but the switchgrass samples showed that the acid was present. In ethanol/H_2O reactions, acids were present in an even greater amount than in the MeOH/H_2O reactions. The following table shows the resonances we used:

Table S6. Resonances used for ^{1}H quantification of hydroxycinnamate products.

Analyte	^{1}H NMR Resonance (ppm)
Dihydrocoumaric acid	7.05
Methyl-dihydrocoumarate	7.07
Ethyl-dihydrocoumarate	7.07
Dihydroferulic Acid	6.87
Methyl-dihydroferulate	6.85
Ethyl-dihydroferulate	6.85

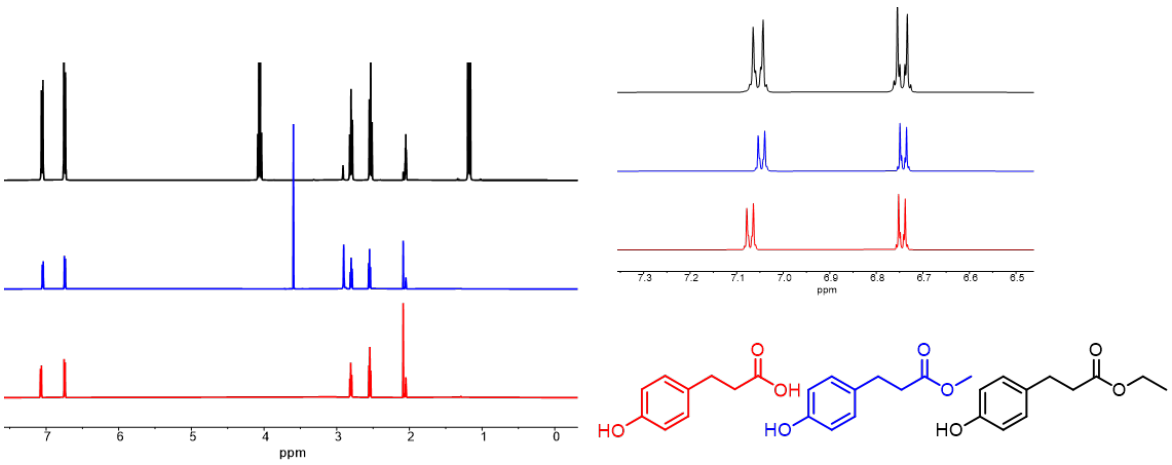

Figure S1. ^{1}H NMR spectra of dihydrocoumaric acid (red), methyl-dihydrocoumarate (blue) and ethyl-dihydrocoumarate (black).

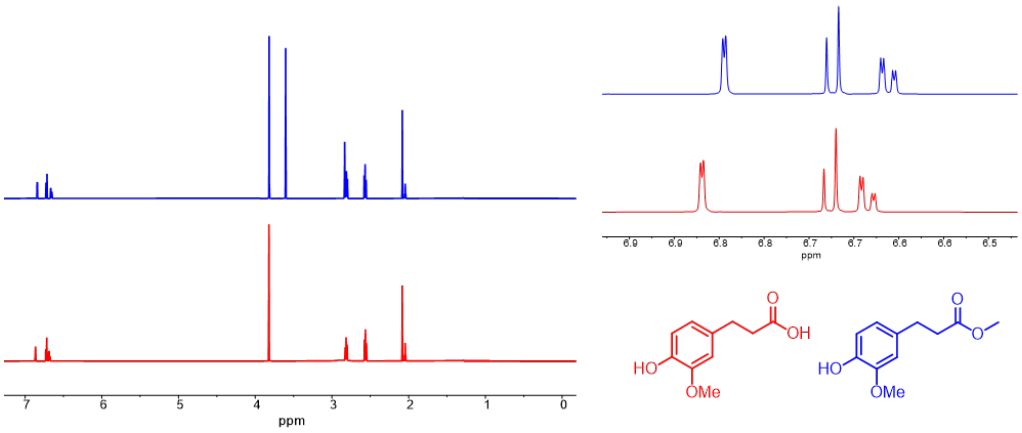

Figure S2. ^{1}H NMR spectra of dihydrocoumaric acid (red) and methyl-dihydrocoumarate (blue).

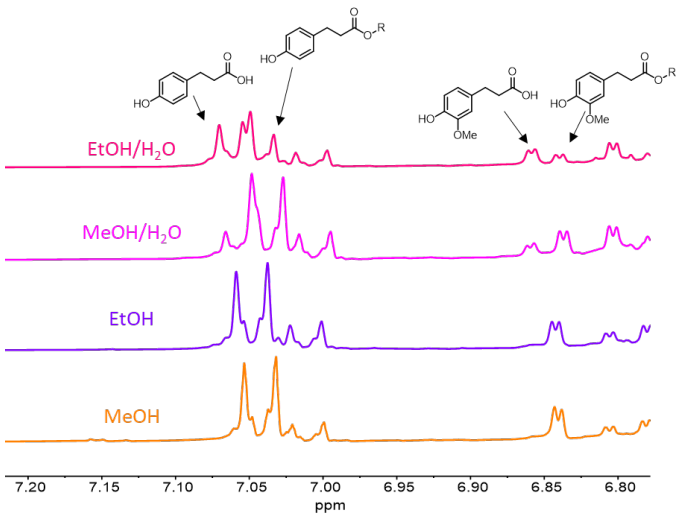

Figure S3. ^{1}H NMR spectra of corn stover samples from batch RCF reactions.

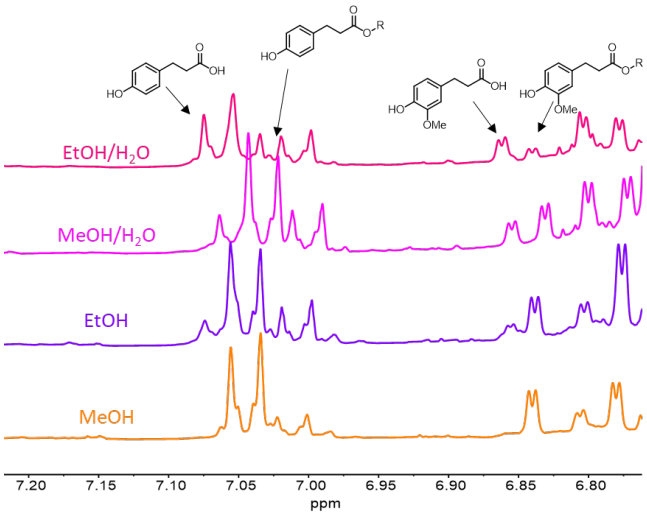

Figure S4. ^{1}H NMR spectra of switchgrass samples from batch RCF reactions.

Determination of water content in lignin oils by volumetric Karl Fischer titration

The lignin oils obtained from batch RCF of switchgrass and corn stover in methanol/water and L-L extraction were analyzed to determine their water content using volumetric Karl Fischer titrations, following the Laboratory Analytical Procedure (LAP) of the National Renewable Energy Laboratory entitled "Determination of Water Content in Bio-Oils by Volumetric Karl Fischer Titration" (2021) with slight modifications.[11] In short, the one-component titrant CombiTitrant 5 Keto was used to titrate in methanol (titration medium) the corresponding RCF oil sample previously dissolved in methanol to help its gravimetric addition; the process was followed electrochemically using a Metrohm 870 KF Titrino plus instrument, and the results reported with a confidence level of 95% (confidence interval was calculated as two times the standard deviation of 3 replicates).

Water content in lignin oils by volumetric Karl Fischer titration

Substrate	Water content (wt %)
Switchgrass	0.55 ± 0.16
Corn stover	0.46 ± 0.08

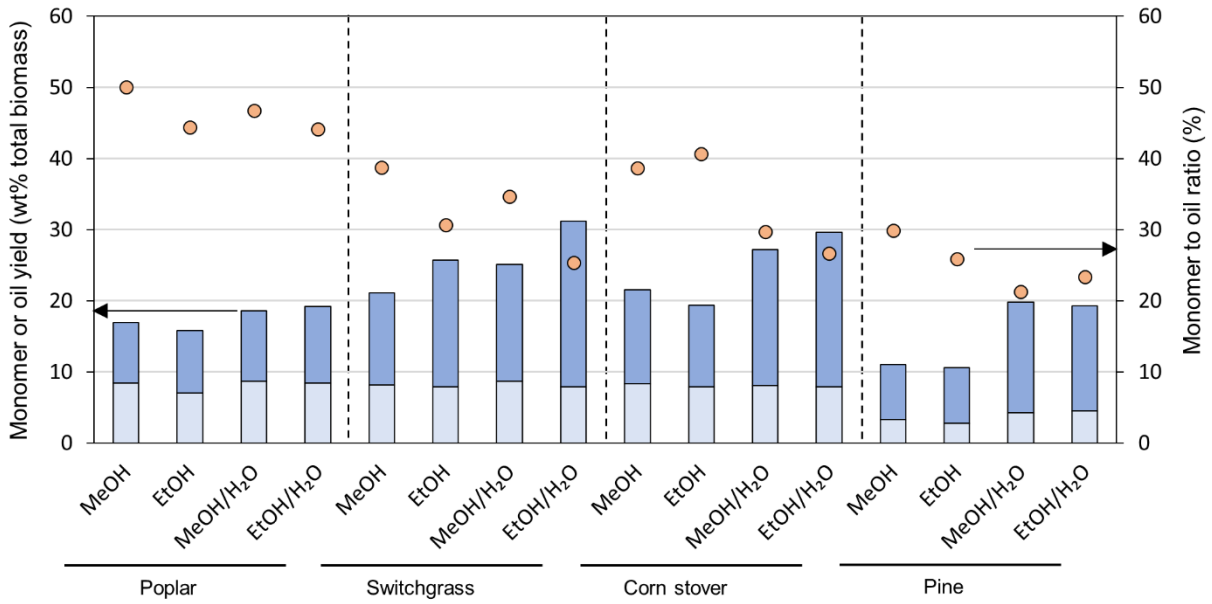

Figure S5. Monomer and oil yields and the resulting the monomer-to-oil ratios of batch RCF with different feedstocks and solvents. Circle dots represent the monomer-to-oil ratio. Light bars are monomer yields and oil yields are the sum of light and dark bars.

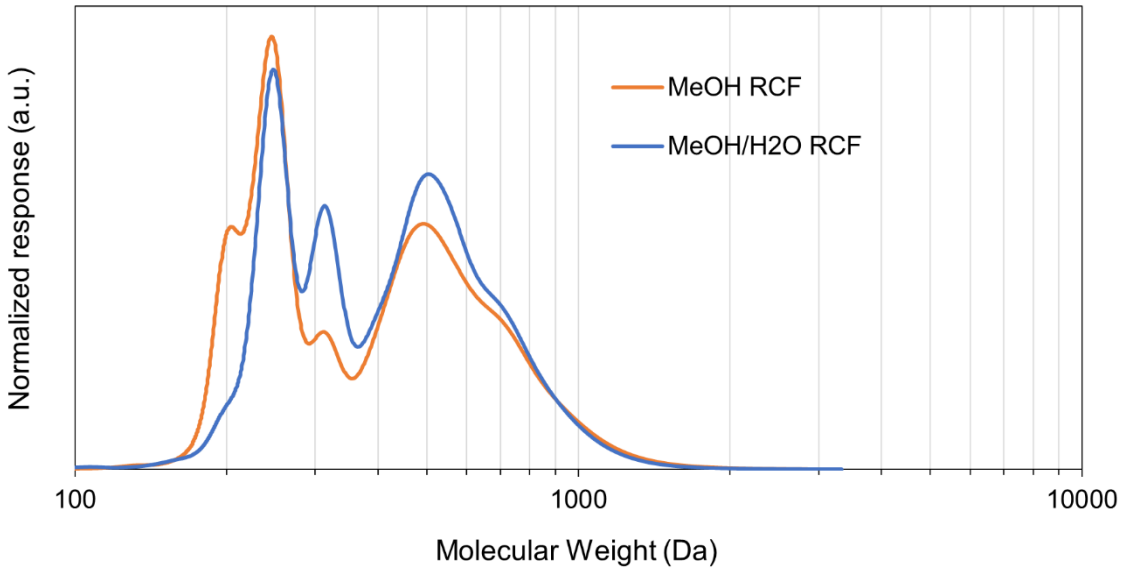

Figure S6. GPC traces of lignin oils from batch RCF of poplar with methanol and 50:50 w/w methanol/water.

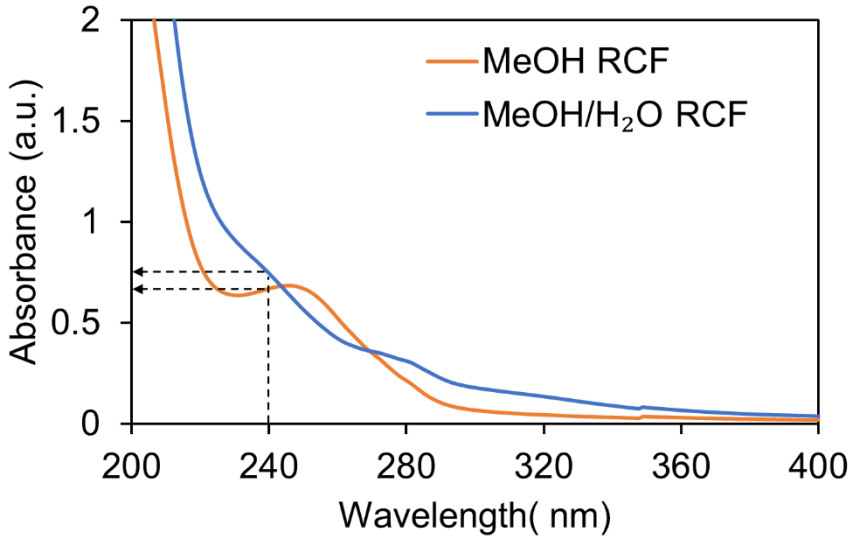

Figure S7. UV-Vis spectra of the aqueous phase obtained after FT-RCF, solvent removal, and liquid-liquid extraction. 240 nm was used for the quantification of lignin. MeOH RCF sample was diluted 100 times with water and MeOH/H_2O RCF sample was diluted 25 times with water. FT-RCF conditions: 2 mL/min feed solvent, 2.7 g poplar, 0.9 g 5 wt% Ru/C (diluted with 2.1 g of fused silica), 1,600 psig, 200 sccm H2, 225°C, and 1 h heating ramp and 3 h run. The absorptivity, ε, was measured to be 19.5 L g^{-1} cm^{-1} using FT-MeOH RCF oil.

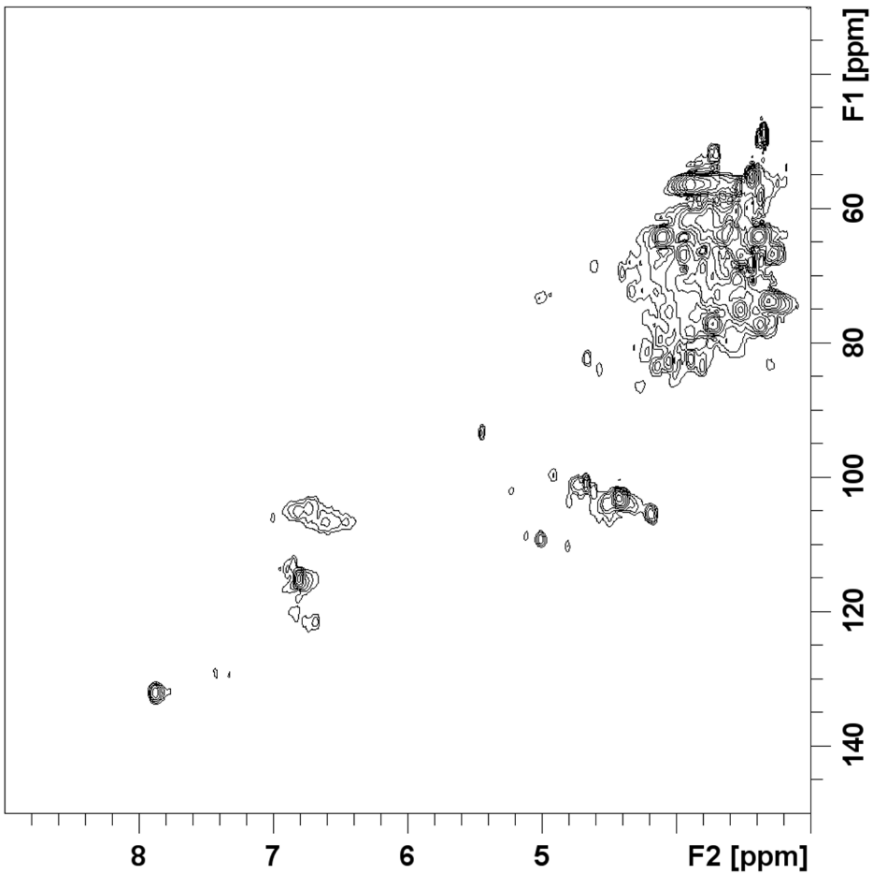

Figure S8. HSQC spectra of the isolated products from the aqueous phase after liquid-liquid extraction of RCF oil from methanol/water RCF of poplar.

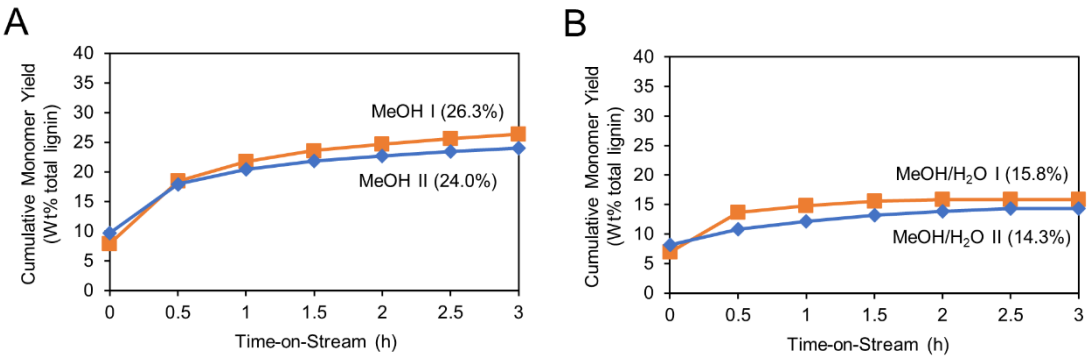

Figure S9. Reaction profiles of FT-RCF. Two different reactions were conducted with two biomass beds. Reaction conditions: 2 mL/min feed solvent, 2.7 g poplar, 0.9 g 5 wt% Ru/C (diluted with 2.1 g of fused silica), 1,600 psig, 200 sccm H_2, 225°C, and 1 h heating ramp and 3 h run. **(A)** poplar-methanol. **(B)** poplar-methanol/water.

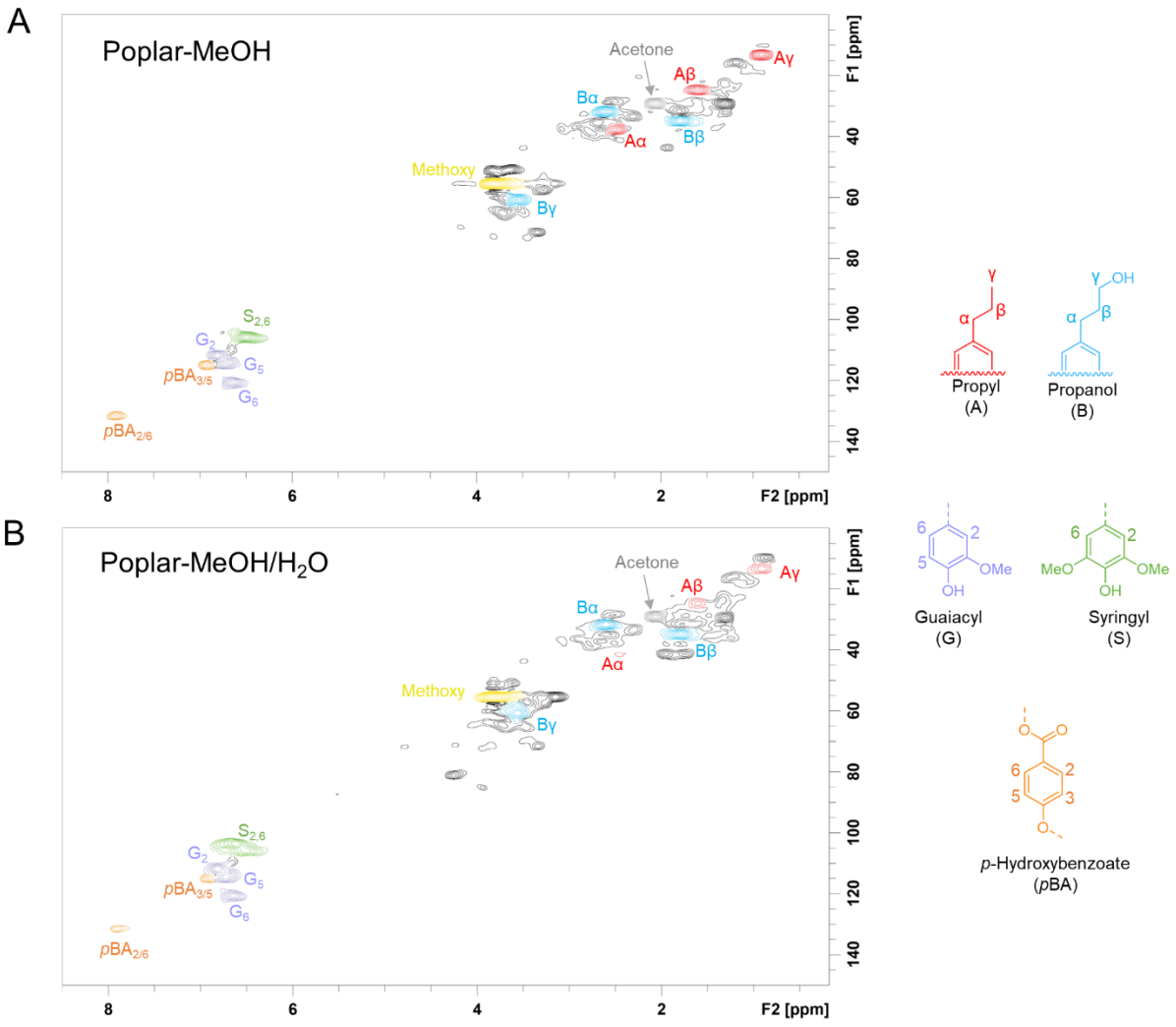

Figure S10. HSQC spectra of the isolated lignin oil from the organic phase after liquid-liquid extraction of RCF oil from RCF of poplar in **(A)** methanol **(B)** methanol/water mixture.

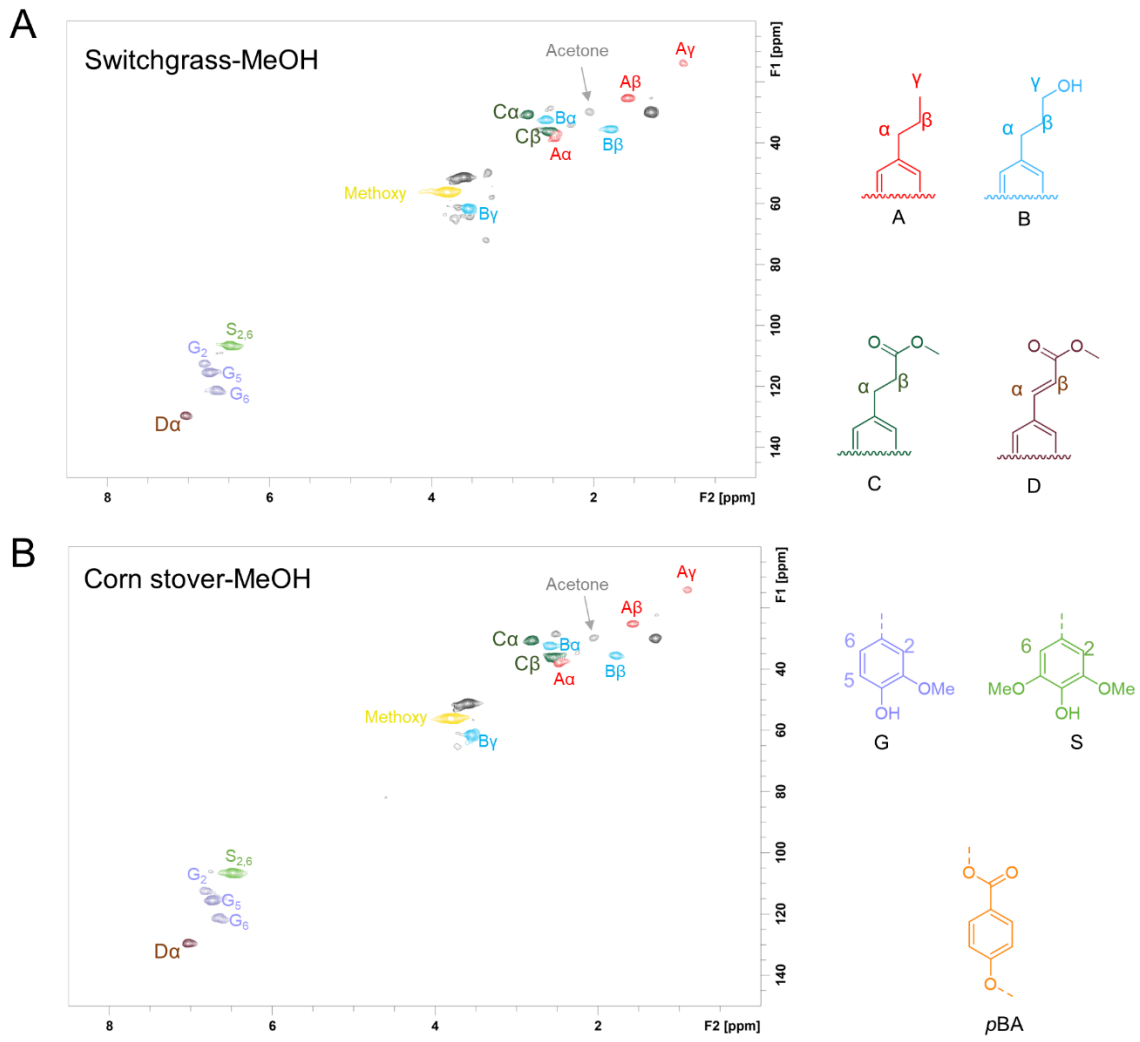

Figure S11. HSQC spectra of the lignin oil from methanol RCF with **(A)** switchgrass, **(B)** corn stover, and (C) pine.

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