

Supplementary Information

Production of Syringic Acid by Direct CO₂ Insertion into Syringol via Kolbe-Schmitt Type Reaction

Omar Mohammad^[a], Jude A. Onwudili^{*[a,b]}, Qingchun Yuan,^[a,b]

[a] O. Mohammad, J.A. Onwudili, Q. Yuan. Energy and Bioproducts Research Institute, Aston University, Aston Triangle, Birmingham, B4 7ET, West Midlands, United Kingdom

[b] J.A. Onwudili, Q. Yuan. Department of Chemical Engineering and Applied Chemistry, Aston University, Aston Triangle, Birmingham, B4 7ET, West Midlands, United Kingdom

*Corresponding Author's E-mail: j.onwudili@aston.ac.uk

Figure S1: Calibration curve for syringol in a water-acetone (50% v/v) solution.

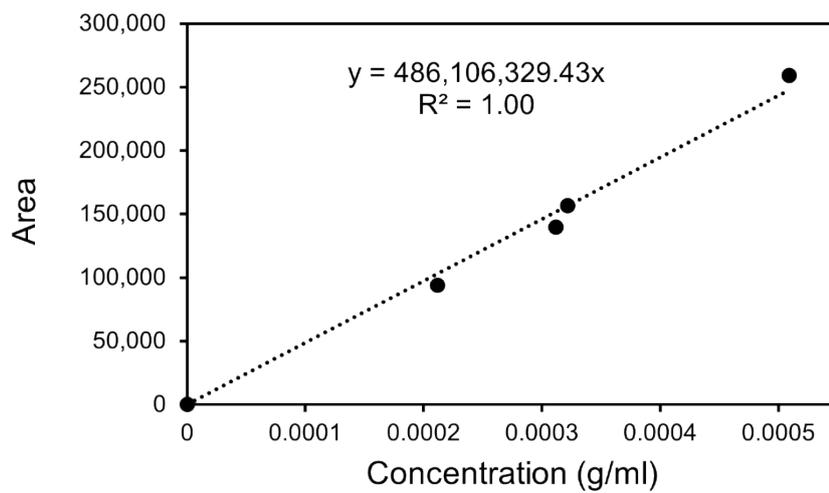


Table S1: Effect of pressure on product distribution, showing normalised peak areas for the solid and toluene fractions. The reaction involved adding 50 wt% guaiacol to the sodium salt of syringol (SyONa).

Compounds	Normalised Peak Areas (%)			
	Solid Fraction			
	5 bar	10 bar	30 bar	50 bar
Syringol	58.8	59.9	40.1	60.7
Syringic acid	0.13	24.0	48.8	24.3
3-Methoxysalicylic acid	0	7.77	1.88	7.87
Vanillic acid	0	4.77	6.88	4.83
1,2,3-Trimethoxybenzene	2.79	1.23	0.44	0.85
3-methoxycatechol	0	0.87	1.31	0.22
Methyl syringate	0	0.52	0.31	0.33
Benzoic acid, 3,4,5-trimethoxy-	0	0.32	0.44	0.33
Guaiacol	37.4	0.29	8.03	0.3
2,6-Dimethoxyhydroquinone	0	0.18	0.28	0.18
Benzoic acid, 4-hydroxy-3-methoxy-, methyl ester	0	0.06	0.05	0.06
Catechol	0.84	0.04	0.11	0.04
Benzoic acid, 3,4-dimethoxy-	0	0	0.64	0
Benzoic acid, 2,3-dihydroxy-	0	0	0.08	0
2,3-Dimethoxybenzoic acid	0	0	0.05	0
	Toluene Fraction			
Syringol	66.8	38.19	49.41	42.91
Guaiacol	31.6	51.82	41.43	52.31
1,2,3-Trimethoxybenzene	1.18	7.14	6.35	3.67
Benzene, 1,2-dimethoxy-	0.42	2.85	2.81	1.11

Table S2: Effect of pressure on product distribution, showing normalised peak areas for the solid and toluene fractions. The reaction involved adding 50 wt% K₂CO₃ to the sodium salt of syringol (SyONa).

Compounds	Normalised Peak Areas (%)			
	Solid Fraction			
	5 bar	10 bar	30 bar	50 bar
3-Methoxycatechol	0	0.17	0.95	0.21
1,2,3-Trimethoxybenzene	7.00	0.17	0.91	0.12
Syringol	88.4	66.35	54.95	66.97
Benzoic acid, 3,4,5-trimethoxy-	0	0.19	0.52	0.09
Methyl syringate	0	0.14	0.34	0.29
Syringic acid	4.65	32.98	42.33	32.32
	Toluene Fraction			
1,2,3-Trimethoxybenzene	4.53	5.47	6.68	4.85
Syringol	95.5	94.53	93.32	95.15

Table S3: Effect of reaction time on product distribution, showing normalised peak areas for the solid and toluene fractions. The reaction involved adding 50 wt% guaiacol to the sodium salt of syringol (SyONa).

Compounds	Normalised Peak Areas (%)		
	Solid Fraction		
	2h	4h	6h
Syringol	59.91	26.57	23.78
Syringic acid	24.03	63.48	64.91
3-Methoxysalicylic acid	7.77	0.79	1.72
Vanillic acid	4.77	5.94	5.27
1,2,3-Trimethoxybenzene	1.23	0.55	0.83
3-Methoxycatechol	0.87	0.78	0.43
Methyl syringate	0.52	0	0
Benzoic acid, 3,4,5-trimethoxy-	0.32	0.58	0.99
Phenol, 2-methoxy-	0.29	0.05	0.07
2,6-Dimethoxyhydroquinone	0.18	0.16	0.37
Benzoic acid, 3,4-dimethoxy-	0	0.66	0.11
Benzoic acid, 4-hydroxy-3-methoxy-, methyl ester	0.06	0	0
Methyl syringate	0	0.44	0.98
4-Methoxy-3,5-dihydroxybenzoic acid	0	0	0.53
Catechol	0.04	0	0
	Toluene Fraction		
Syringol	49.41	51.18	49.35
Phenol, 2-methoxy-	41.43	39.62	32.22
1,2,3-Trimethoxybenzene	6.35	7.05	14.19
Benzene, 1,2-dimethoxy-	2.81	2.15	4.23

Table S4: Effect of reaction time on product distribution, showing normalised peak areas for the solid and toluene fractions. The reaction involved adding 50 wt% K_2CO_3 to the sodium salt of syringol (SyONa).

Compounds	Normalised Peak Areas (%)		
	Solid Fraction		
	2h	4h	6h
3-Methoxycatechol	0.95	1.01	1.35
1,2,3-Trimethoxybenzene	0.91	1.21	0.11
Syringol	44.95	43.8	11.71
Benzoic acid, 3,4,5-trimethoxy-	0.52	0.27	0.07
Methyl syringate	0.34	0.35	0.96
Syringic acid	52.33	53.36	85.80
	Toluene Fraction		
1,2,3-Trimethoxybenzene	6.68	12.16	14.63
Syringol	93.32	87.84	85.37