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Supplementary Data

Integration of Hydrothermal Carbonisation with Anaerobic Digestion; Opportunities for Valorisation of Digestate

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Table S1. Yields of products following hydrothermal carbonisation of digestate.

Feedstock and HTC process conditions	Yields (wt% db)		
	Solid	Liquid	Gas
AGR digestate			
150 °C at 20%	82.7 ± 2.2	15.2 ± 1.0	2.1 ± 1.2
200 °C at 10%	60.0 ± 2.6	36.6 ± 0.3	3.4 ± 2.2
200 °C at 20%	67.8 ± 1.5	26.9 ± 4.6	5.3 ± 3.1
200 °C at 30%	80.1 ± 1.7	16.8 ± 1.0	3.1 ± 0.7
250 °C at 10%	47.2 ± 0.1	50.1 ± 0.5	2.8 ± 0.4
250 °C at 20%	51.1 ± 2.9	45.7 ± 3.7	3.2 ± 0.8
250 °C at 30%	49.4 ± 1.1	45.6 ± 0.6	4.9 ± 0.5
MSW digestate			
150 °C at 20%	90.6 ± 2.1	8.1 ± 2.2	1.4 ± 0.1
200 °C at 10%	85.0 ± 0.2	10.2 ± 2.5	4.9 ± 2.3
200 °C at 20%	87.1 ± 0.2	9.5 ± 2.1	3.3 ± 1.9
200 °C at 30%	85.7 ± 0.8	11.1 ± 0.8	3.3 ± 0.1
250 °C at 10%	82.0 ± 0.0	15.0 ± 0.9	2.9 ± 0.9
250 °C at 20%	84.1 ± 1.3	12.1 ± 3.0	3.8 ± 1.8
250 °C at 30%	83.5 ± 1.0	5.4 ± 5.4	2.4 ± 2.4
SS digestate			
150 °C at 20%	87.1 ± 1.9	11.6 ± 2.0	1.3 ± 0.1
200 °C at 10%	72.6 ± 1.3	23.0 ± 1.8	4.4 ± 0.5
200 °C at 20%	76.1 ± 0.3	19.2 ± 0.4	4.7 ± 0.7
200 °C at 30%	78.0 ± 1.2	17.3 ± 4.3	4.7 ± 3.1
250 °C at 10%	65.6 ± 0.3	31.4 ± 0.3	3.0 ± 0.0
250 °C at 20%	67.9 ± 0.4	27.4 ± 1.2	4.7 ± 0.7
250 °C at 30%	69.5 ± 0.1	25.4 ± 0.2	5.1 ± 0.3
VGF digestate			
150 °C at 20%	89.0 ± 0.2	9.8 ± 0.2	1.2 ± 0.0
200 °C at 10%	79.5 ± 0.1	19.1 ± 0.2	1.4 ± 0.2
200 °C at 20%	80.9 ± 1.0	16.6 ± 0.2	2.5 ± 0.8
200 °C at 30%	79.8 ± 1.3	15.3 ± 1.6	4.9 ± 2.9
250 °C at 10%	71.1 ± 0.5	20.5 ± 5.8	8.4 ± 5.3
250 °C at 20%	71.4 ± 1.4	25.5 ± 0.7	3.1 ± 0.7
250 °C at 30%	73.7 ± 0.1	21.4 ± 1.1	4.9 ± 1.2

Table S2. Ultimate, proximate and heating analysis of feedstock and corresponding hydrochar.

Material	Ultimate analysis (wt% db)					Proximate analysis (wt% db)			HHV	H/C	O/C
	C	H	N	S	O[a]	VM	FC	Ash[a]	(MJ/kg, db)	(daf)	(daf)
AGR digestate	44.1 ± 0.1	5.1 ± 0.0	3.2 ± 0.0	0.3 ± 0.0	31.3 ± 0.3	70.2 ± 0.3	13.8 ± 0.0	16.0 ± 0.3	17.8	1.38	0.53
150 °C at 20%	44.2 ± 1.9	4.8 ± 0.3	3.1 ± 0.1	0.0 ± 0.0	27.5 ± 0.5	62.2 ± 3.2	17.5 ± 0.6	20.4 ± 2.7	17.9	1.29	0.47
200 °C at 10%	52.0 ± 0.4	6.9 ± 1.4	3.1 ± 0.5	0.1 ± 0.1	23.1 ± 3.5	69.4 ± 1.5	15.9 ± 2.8	14.7 ± 1.4	21.6	1.59	0.33
200 °C at 20%	50.8 ± 0.8	6.0 ± 0.7	3.3 ± 0.5	0.1 ± 0.1	24.4 ± 3.3	67.4 ± 0.5	17.3 ± 1.9	15.3 ± 1.4	20.7	1.40	0.36
200 °C at 30%	51.2 ± 1.2	6.1 ± 0.5	3.5 ± 0.2	0.0 ± 0.0	22.8 ± 2.5	66.7 ± 0.3	17.0 ± 1.3	16.3 ± 1.0	20.9	1.42	0.33
250 °C at 10%	57.3 ± 1.4	6.3 ± 1.1	3.6 ± 0.3	0.2 ± 0.0	11.5 ± 1.9	56.7 ± 0.1	22.3 ± 0.9	21.0 ± 0.8	24.0	1.32	0.15
250 °C at 20%	57.1 ± 1.4	6.6 ± 1.5	3.9 ± 0.3	0.2 ± 0.0	12.0 ± 3.1	58.2 ± 0.0	21.6 ± 0.1	20.2 ± 0.1	24.2	1.39	0.16
250 °C at 30%	56.7 ± 0.4	5.8 ± 0.4	3.8 ± 0.0	0.3 ± 0.0	12.7 ± 0.7	56.2 ± 0.8	23.1 ± 0.9	20.7 ± 0.1	23.3	1.21	0.17
MSW digestate	24.1 ± 0.0	1.7 ± 0.0	1.5 ± 0.0	0.2 ± 0.0	16.9 ± 0.0	36.2 ± 0.1	8.3 ± 0.0	55.5 ± 0.1	15.6	0.83	0.53
150 °C at 20%	23.8 ± 1.7	2.1 ± 0.3	1.2 ± 0.1	0.0 ± 0.0	19.0 ± 4.5	39.4 ± 6.8	6.6 ± 0.5	54.0 ± 6.3	15.2	1.04	0.60
200 °C at 10%	22.6 ± 0.1	1.6 ± 0.0	0.7 ± 0.0	0.1 ± 0.1	11.7 ± 1.7	32.0 ± 2.3	4.7 ± 0.9	63.4 ± 1.5	15.5	0.85	0.39
200 °C at 20%	21.4 ± 0.2	1.6 ± 0.0	0.9 ± 0.0	0.1 ± 0.0	13.9 ± 0.1	32.3 ± 0.8	5.6 ± 0.7	62.1 ± 0.1	15.6	0.87	0.49
200 °C at 30%	24.0 ± 1.4	1.8 ± 0.1	0.9 ± 0.0	0.1 ± 0.0	12.2 ± 0.4	32.4 ± 1.0	6.5 ± 0.2	61.1 ± 1.2	15.4	0.90	0.38
250 °C at 10%	23.0 ± 2.8	1.6 ± 0.2	0.7 ± 0.1	0.2 ± 0.2	9.7 ± 2.0	28.3 ± 4.6	6.9 ± 0.7	64.9 ± 5.3	15.6	0.83	0.32
250 °C at 20%	21.7 ± 1.5	1.6 ± 0.1	0.8 ± 0.0	0.0 ± 0.0	7.9 ± 0.3	25.7 ± 3.1	6.3 ± 1.2	68.0 ± 2.0	15.6	0.88	0.28
250 °C at 30%	23.4 ± 0.7	1.7 ± 0.0	0.9 ± 0.0	0.0 ± 0.0	7.8 ± 0.9	26.9 ± 0.2	6.8 ± 0.4	66.3 ± 0.2	15.5	0.85	0.25
SS digestate	28.6 ± 0.3	3.1 ± 0.1	3.4 ± 0.0	1.5 ± 0.0	16.4 ± 0.3	51.0 ± 0.1	2.1 ± 0.1	46.9 ± 0.0	14.9	1.30	0.43
150 °C at 20%	33.4 ± 1.8	4.4 ± 0.6	3.2 ± 0.2	0.3 ± 0.1	15.0 ± 2.3	50.5 ± 4.7	5.7 ± 0.2	43.8 ± 4.9	15.0	1.56	0.34
200 °C at 10%	34.0 ± 1.2	4.2 ± 0.2	2.1 ± 0.0	0.8 ± 0.1	13.0 ± 1.0	47.9 ± 0.5	6.1 ± 0.0	46.0 ± 0.4	15.0	1.48	0.29
200 °C at 20%	34.0 ± 1.1	4.2 ± 0.2	2.4 ± 0.1	0.9 ± 0.1	14.0 ± 0.6	49.1 ± 0.9	6.5 ± 0.2	44.4 ± 0.7	15.1	1.47	0.31
200 °C at 30%	35.2 ± 0.7	4.4 ± 0.1	2.9 ± 0.0	1.2 ± 0.0	12.7 ± 0.1	49.4 ± 0.8	6.9 ± 0.1	43.7 ± 0.9	15.4	1.48	0.27
250 °C at 10%	34.4 ± 0.3	4.0 ± 0.2	2.2 ± 0.2	0.8 ± 0.0	9.2 ± 0.7	44.3 ± 0.6	6.2 ± 0.3	49.5 ± 0.9	15.2	1.39	0.20
250 °C at 20%	34.7 ± 0.4	4.1 ± 0.0	2.4 ± 0.1	0.7 ± 0.3	10.6 ± 0.6	45.9 ± 0.5	6.6 ± 0.2	47.5 ± 0.3	15.3	1.42	0.23
250 °C at 30%	36.4 ± 0.8	4.3 ± 0.1	2.8 ± 0.0	0.9 ± 0.3	9.0 ± 0.7	46.6 ± 0.6	6.8 ± 0.0	46.6 ± 0.6	15.7	1.40	0.19
VGF digestate	29.5 ± 0.1	3.0 ± 0.1	2.0 ± 0.0	0.3 ± 0.0	21.3 ± 0.6	47.2 ± 1.1	9.0 ± 0.2	43.8 ± 0.8	14.9	1.22	0.54
150 °C at 20%	29.7 ± 1.8	3.0 ± 0.1	1.9 ± 0.1	0.0 ± 0.0	12.2 ± 1.2	37.6 ± 0.7	9.3 ± 0.0	53.1 ± 0.7	15.0	1.22	0.31
200 °C at 10%	26.3 ± 1.6	2.7 ± 0.2	1.1 ± 0.0	0.2 ± 0.0	14.4 ± 3.2	37.5 ± 0.7	7.2 ± 0.7	55.3 ± 1.4	14.8	1.22	0.41
200 °C at 20%	32.2 ± 1.9	3.3 ± 0.2	1.5 ± 0.1	0.3 ± 0.0	13.3 ± 0.0	41.7 ± 1.5	9.0 ± 0.7	49.4 ± 2.3	15.1	1.23	0.31
200 °C at 30%	30.4 ± 1.5	3.1 ± 0.1	1.4 ± 0.1	0.2 ± 0.0	12.5 ± 1.3	39.3 ± 1.8	8.4 ± 1.1	52.3 ± 3.0	14.9	1.23	0.31
250 °C at 10%	26.4 ± 0.6	2.6 ± 0.1	1.2 ± 0.1	0.1 ± 0.1	8.3 ± 0.1	30.4 ± 0.6	8.2 ± 0.1	61.3 ± 0.7	14.8	1.17	0.24
250 °C at 20%	27.8 ± 0.7	2.7 ± 0.0	1.4 ± 0.0	0.1 ± 0.1	9.0 ± 1.6	31.7 ± 1.7	9.2 ± 0.7	59.0 ± 2.4	14.9	1.14	0.24
250 °C at 30%	29.1 ± 2.1	2.9 ± 0.4	1.5 ± 0.2	0.1 ± 0.1	5.4 ± 1.5	30.4 ± 0.4	8.6 ± 0.7	61.0 ± 1.1	15.0	1.17	0.14

[a] calculated by difference; [b] calculated according to Equation (1)

Table S3. Inorganic analysis and slagging and fouling indices of feedstock and corresponding hydrochar.

Material	Inorganic analysis (wt % db)							Slagging and fouling indices					
	Na	Mg	P	K	Ca	Fe	Si	AI	BAI	R b/a	SI	FI	SVI
AGR digestate	0.25	0.66	1.16	1.57	1.34	2.27	1.78	1.25	1.46	2.1	0.6	29.2	38
150 °C at 20%	0.23	0.68	1.08	2.03	1.48	0.55	1.81	1.54	0.29	1.7	0.1	32.2	49
200 °C at 10%	0.27	0.50	1.17	1.13	1.71	0.68	2.27	0.80	0.56	1.2	0.1	13.6	54
200 °C at 20%	0.24	0.59	1.15	1.75	1.60	0.60	2.21	1.18	0.35	1.3	0.2	20.9	54
200 °C at 30%	0.26	0.65	1.24	2.03	1.72	0.66	2.17	1.34	0.34	1.5	0.0	25.5	51
250 °C at 10%	0.22	1.14	1.98	0.75	2.83	1.09	2.97	0.50	1.29	1.3	0.3	7.4	46
250 °C at 20%	0.14	0.99	1.69	1.02	2.34	0.95	3.33	0.59	0.95	1.0	0.2	7.3	53
250 °C at 30%	0.10	1.09	1.81	1.04	2.47	0.90	3.36	0.60	0.93	1.1	0.3	7.1	52
MSW digestate	0.92	1.40	0.73	1.63	10.38	3.23	10.15	2.05	1.44	0.9	0.2	5.3	50
150 °C at 20%	0.55	1.48	0.95	0.91	14.85	3.60	7.03	1.21	2.80	1.5	0.0	5.2	35
200 °C at 10%	0.58	1.65	0.96	1.41	13.91	3.62	10.93	1.60	2.09	1.0	0.1	4.0	46
200 °C at 20%	0.52	1.69	1.11	1.11	13.53	3.85	10.33	1.31	2.70	1.0	0.1	3.4	45
200 °C at 30%	0.57	1.63	1.05	1.12	13.42	3.84	10.01	1.37	2.60	1.1	0.1	3.7	44
250 °C at 10%	0.47	1.75	1.18	0.86	17.57	4.10	8.92	1.07	3.52	1.4	0.3	3.6	36
250 °C at 20%	0.32	1.57	3.62	1.01	5.43	4.46	12.15	1.06	3.88	0.5	0.0	1.2	61
250 °C at 30%	0.92	1.65	0.90	1.34	11.92	3.42	13.46	1.84	1.72	0.8	0.0	3.3	54
SS digestate	0.50	0.89	2.75	0.73	4.61	3.26	7.56	1.04	3.00	0.6	1.0	2.1	56
150 °C at 20%	0.38	0.96	2.12	0.70	3.26	2.48	8.40	0.90	2.62	0.4	0.1	1.3	65
200 °C at 10%	0.22	0.99	2.25	0.72	3.32	2.78	8.90	0.78	3.40	0.4	0.3	1.1	65
200 °C at 20%	0.25	0.94	2.31	0.67	3.37	2.73	8.40	0.75	3.43	0.4	0.4	1.1	64
200 °C at 30%	0.38	0.95	2.16	0.72	3.22	2.58	8.32	0.89	2.68	0.4	0.5	1.4	65
250 °C at 10%	0.58	1.19	0.73	0.93	9.45	2.59	9.72	1.25	1.94	0.8	0.7	3.2	52
250 °C at 20%	0.41	1.07	2.47	0.74	3.66	2.84	8.81	0.95	2.81	0.5	0.3	1.4	63
250 °C at 30%	0.23	1.07	2.38	0.73	3.52	2.75	8.77	0.75	3.32	0.4	0.4	1.1	64
VGF digestate	0.67	0.74	0.58	1.18	4.70	1.38	12.05	1.55	0.85	0.4	0.1	2.2	73
150 °C at 20%	0.62	1.08	1.03	1.05	7.15	1.66	13.51	1.41	1.13	0.5	0.0	2.0	67
200 °C at 10%	0.26	0.83	0.96	0.72	5.83	1.67	16.12	0.83	1.95	0.3	0.1	0.8	74
200 °C at 20%	0.22	0.84	0.93	0.83	5.73	1.58	13.45	0.86	1.73	0.4	0.1	1.1	71
200 °C at 30%	1.23	0.88	0.83	0.91	5.56	1.43	14.25	1.84	0.74	0.4	0.1	2.2	73
250 °C at 10%	0.50	0.99	0.94	0.87	5.88	1.79	18.21	1.16	1.49	0.3	0.0	0.9	76
250 °C at 20%	0.29	1.03	0.92	0.78	6.38	1.82	17.06	0.89	1.96	0.4	0.1	0.8	73
250 °C at 30%	0.60	1.05	1.01	0.92	6.42	1.90	17.26	1.28	1.42	0.4	0.0	1.2	73

Table S4. Liquid analysis and experimental biochemical methane potential comparison of HTC process waters.

HTC process waters	pH	TN (g/L)	TOC (g/L)	C:N ratio	COD (g/L)	Total VFA (g/L)	Total phenols (g/L)	BMPexp ($NmlCH_4/gCOD$)
AGR digestate								
150 °C at 20%	6.7	3.1	13.9	4.5	45.5	1.8	1.4	100.0
200 °C at 10%	6.9	1.6	9.2	5.6	25.2	1.3	1.1	136.0
200 °C at 20%	6.2	1.9	14.9	8.0	42.2	2.1	1.6	180.7
200 °C at 30%	5.8	3.3	18.8	5.8	54.6	2.0	2.2	178.6
250 °C at 10%	5.7	1.4	10.9	7.6	29.1	2.5	0.8	139.5
250 °C at 20%	6.1	2.2	16.5	7.4	46.3	4.2	0.8	155.5
250 °C at 30%	6.5	2.9	17.3	5.9	56.6	6.1	0.9	166.3
MSW digestate								
150 °C at 20%	6.5	1.1	3.4	3.1	22.6	0.7	0.3	84.6
200 °C at 10%	7.2	0.9	3.1	3.4	10.7	0.9	0.2	129.0
200 °C at 20%	7.1	2.4	5.7	2.4	18.1	0.9	0.4	137.7
200 °C at 30%	7.4	2.4	8.7	3.7	23.4	1.5	0.5	157.2
250 °C at 10%	7.1	1.0	2.7	2.7	9.4	1.3	0.3	154.0
250 °C at 20%	7.8	1.7	6.0	3.6	16.4	1.3	0.6	134.6
250 °C at 30%	7.5	3.4	6.1	1.8	21.2	1.9	0.6	151.8
SS digestate								
150 °C at 20%	5.6	2.4	16.6	6.9	31.0	1.1	0.2	100.2
200 °C at 10%	5.2	2.6	7.8	3.0	21.0	1.4	0.3	163.5
200 °C at 20%	6.2	4.5	17.1	3.8	38.9	1.8	0.9	181.7
200 °C at 30%	5.6	6.2	21.0	3.4	48.7	2.8	0.9	196.9
250 °C at 10%	7.2	2.4	7.1	3.0	23.4	3.5	0.5	146.3
250 °C at 20%	7.6	4.7	18.4	3.9	43.6	5.3	0.8	151.9
250 °C at 30%	7.5	6.5	20.3	3.1	51.6	5.5	0.9	163.2
VGF digestate								
150 °C at 20%	6.5	1.0	5.7	5.9	11.9	0.9	0.3	121.4
200 °C at 10%	6.1	1.1	4.2	3.8	12.0	1.2	0.4	142.0
200 °C at 20%	5.7	1.4	8.5	6.3	22.5	1.1	0.9	158.6
200 °C at 30%	6.0	1.7	10.3	5.9	31.5	1.3	1.2	144.4
250 °C at 10%	6.4	0.9	4.2	4.7	16.1	1.4	0.7	128.0
250 °C at 20%	6.4	1.5	9.8	6.5	27.8	2.2	1.2	125.1
250 °C at 30%	6.7	2.1	10.6	5.0	36.9	2.6	1.5	113.0

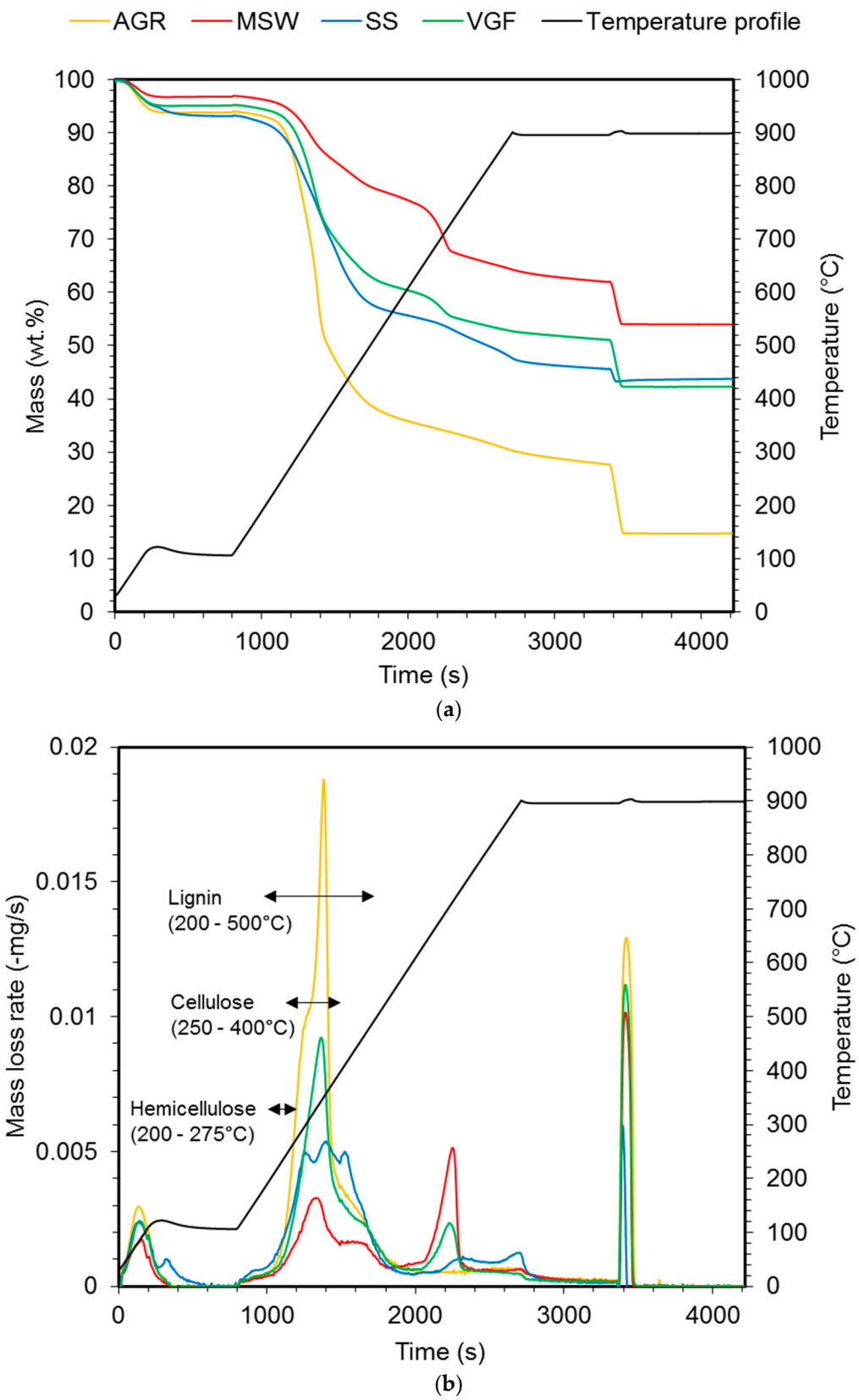

Figure S1. Thermal decomposition behaviour of the digestate feedstocks shown by (**a**) thermogravimetric analysis (TGA) and (**b**) derivative thermogravimetric (DTG) curves.