Supplementary Information

Green Catalyst-based cardboard waste conversion into biogas

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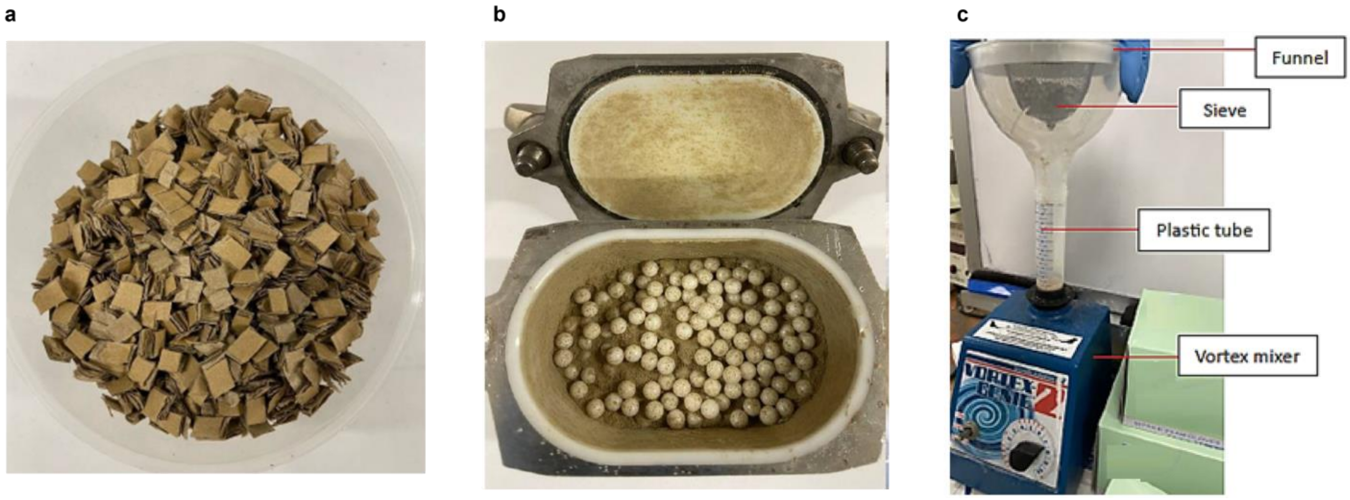


Fig. S1 Cardboard waste (CBW) - (a) square pieces of 4 mm x 4 mm before milling and, (b) after milling with milling ZrO2 balls in ZrO2 jar (c) separation of milled cardboard waste from milling balls in a self-made sieve-vortex device, combining water addition through the funnel cover, swirling action of vortex mixer, and collection of pretreated slurry product directly into a centrifuge plastic tube.

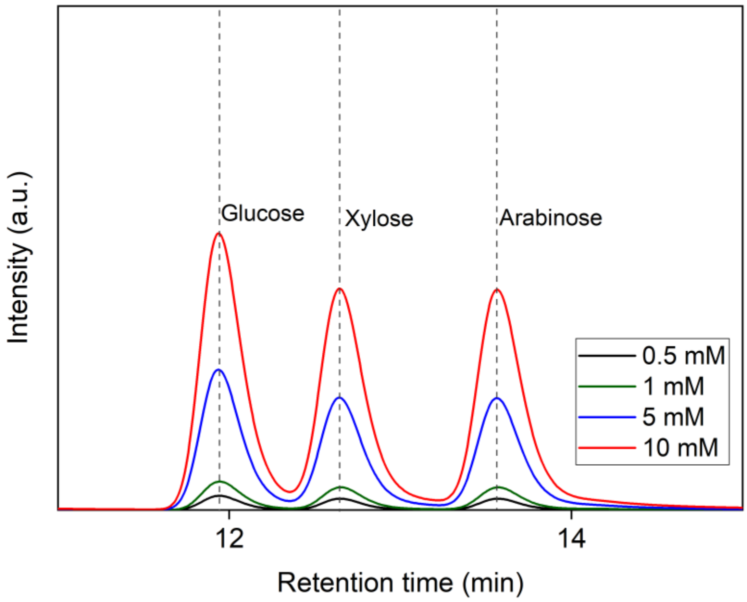


Fig.S2 HPLC chromatogram of mixed sugar standards (Glucose, xylose and arabinose) of different concentrations.

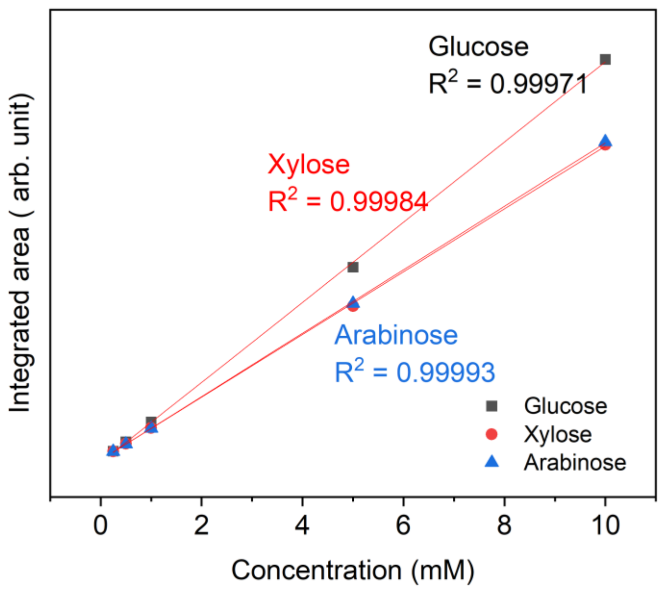


Fig.S3 HPLC calibration curves of sugar standards (Glucose, xylose and arabinose)

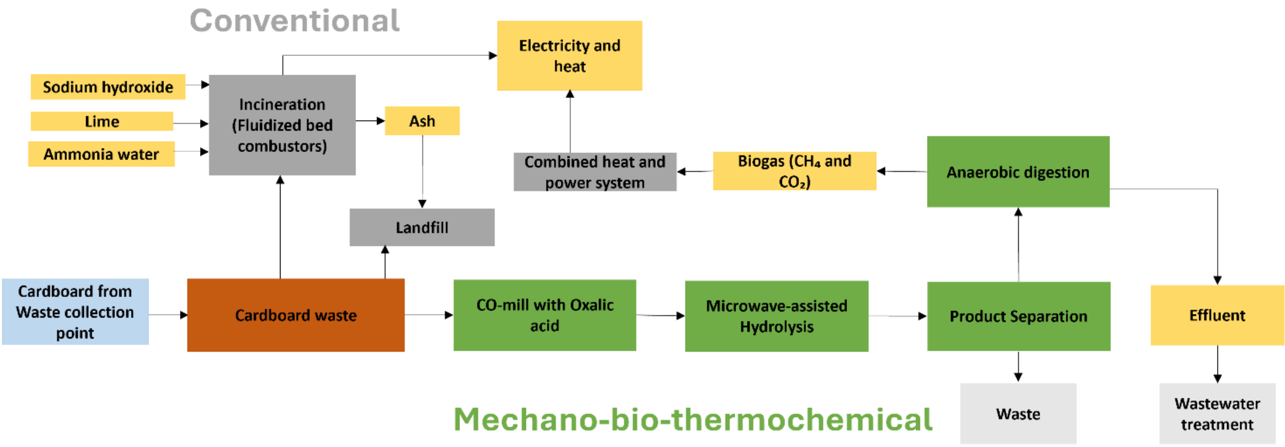


Fig. S4 The process systems of (1) conventional waste treatment and (2) Mechano-bio-thermochemical of CBW showing (grey) the conventional and (green) the novel mechano-bio-thermochemical treatment route. The raw material and products (yellow) considered include sodium hydroxide, lime and ammonia water as pretreatment chemicals for incineration and ash for the waste product. For CBW mechano-bio-thermochemical process, biogas (methane and carbon dioxide are the product after AD process, generating effluent and other residual waste.

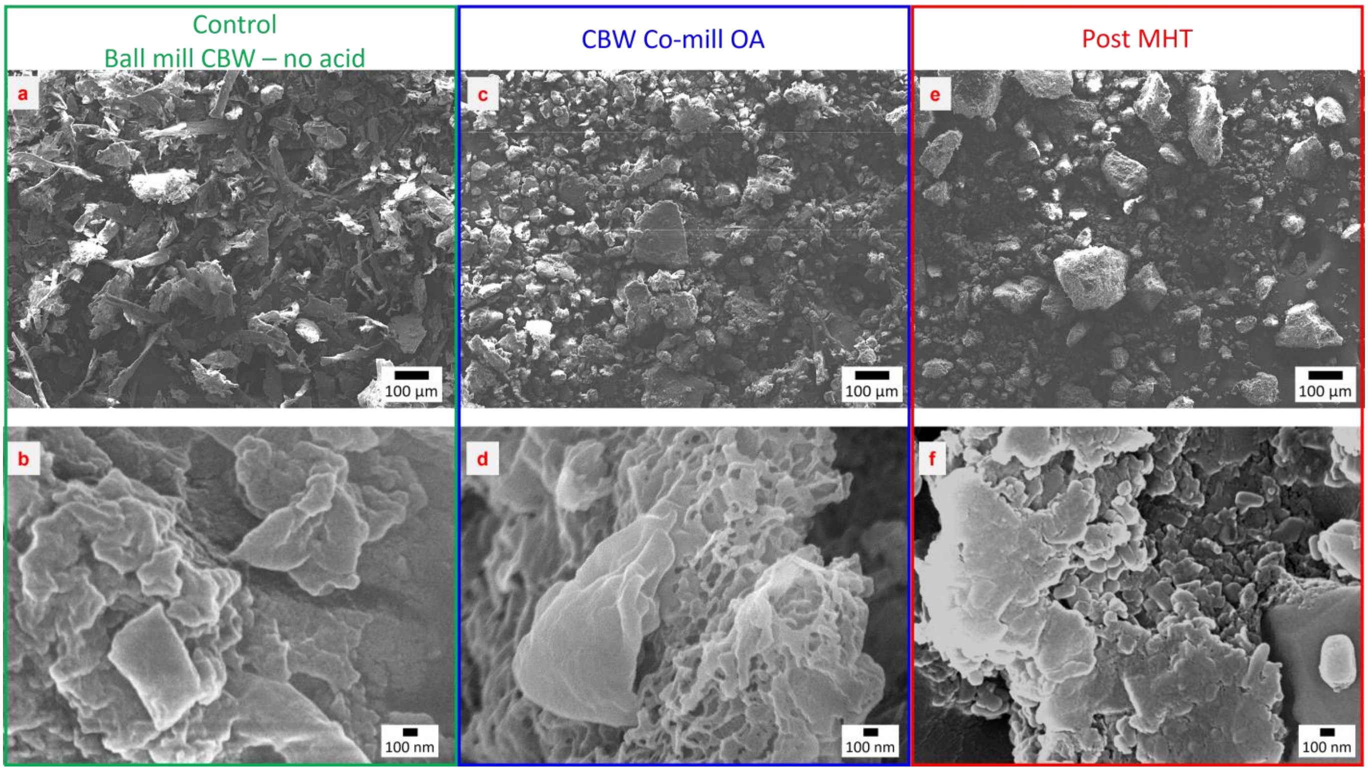


Fig. S5 FE-SEM images of the CBW control sample (milled without catalyst) at different magnifications – (a) 90 ×, (b) ×40,000; and CBW co-mill 47.4 wt% oxalic acid (OA) sample at different magnifications – (c) 90 ×, (d) ×40,000; and post microwave hydrothermal treatment (MHT) of CBW co-mill OA sample at different magnifications – (e) 90 ×, (f) ×40,000.

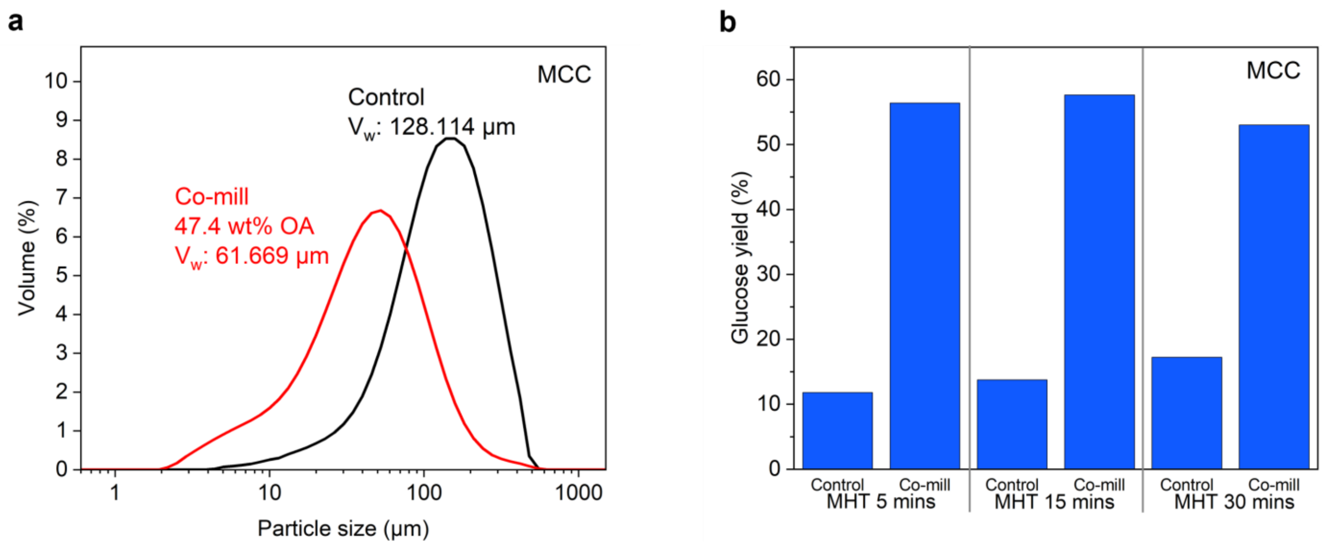


Fig. S6 Co-milled oxalic acid effect on microcrystalline cellulose (MCC) — (a) particles size distribution of control MCC and co-mill MCC (47.4 wt% OA), and (b) Effect of treatment duration on glucose yield from microwave-assisted hydrothermal treatment (MHT) of co-milled MCC at 180 °C after adding water to dilute to 4.11 wt% OA.

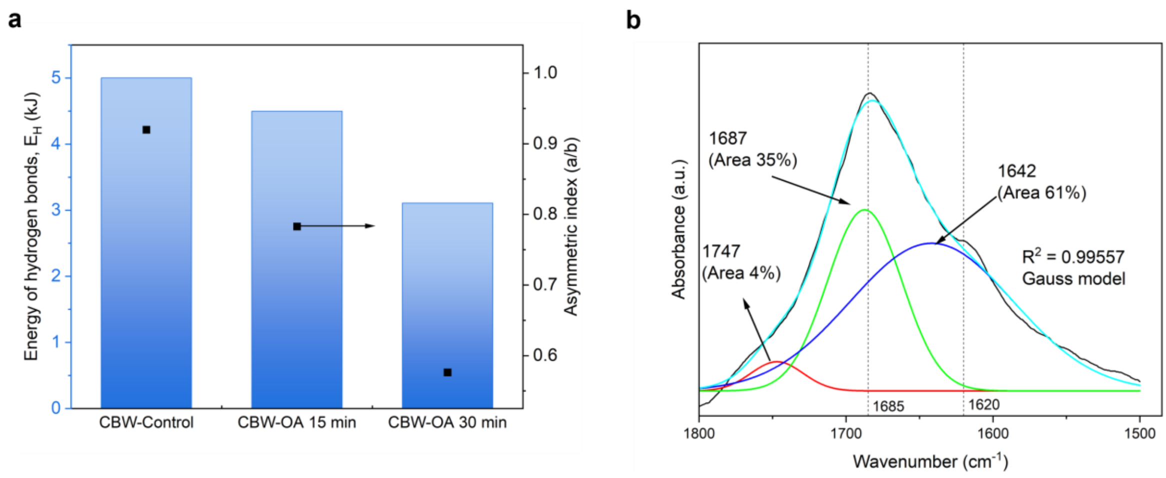


Fig. S7 (a) Hydrogen bond energy and asymmetric index (a/b) derived from the standard frequencies of bonded –OH groups located at 3100–3600 cm^{-1} from FTIR spectra of samples CBW-control, CBW-OA 15 min and CBW-OA 30 min. (b) Curve fit plot of FTIR spectrum of CBW-OA 30 mins using the Gauss model to separate three distinct carboxylate peaks at 1687 cm^{-1}, 1642 cm^{-1}, and a minor ester peak at 1747 cm^{-1}.

Note: Hydrogen bond energy (E_H) = (1/K) × [(v_0-v)/ v_0], where v_0 is the standard frequency corresponding to the free OH groups, (3600 cm^{-1}); v is the measured frequency of the bonded -OH groups (cm^{-1}), and K is 1.6 × 10^{-2} $kcal^{-1}$. Asymmetric index (a/b) is the ratio between segment widths at half height of the OH adsorption band.[1]

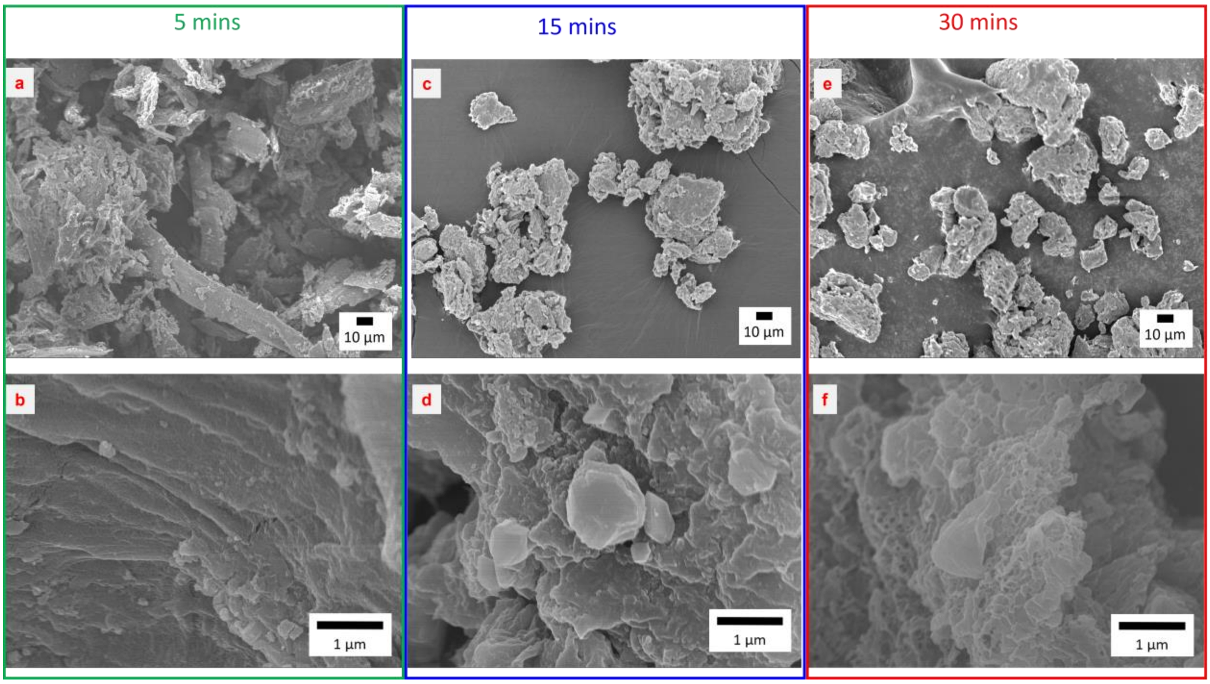


Fig. S8 FE-SEM images of the CBW co-mill 47.4 wt% oxalic acid sample at different milling durations — 5 mins at different magnifications (a) ×500, (b) ×20,000; and 15 mins at different magnifications – (c) 500 ×, (d) ×20,000; and 30 mins at different magnifications – (e) 500 ×, (f) ×20,000.

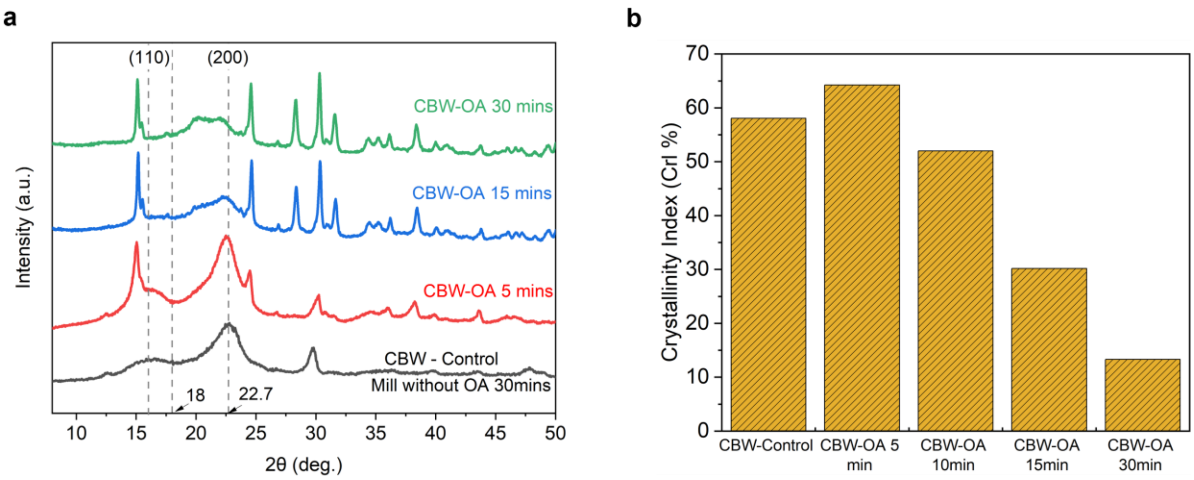


Fig. S9 XRD patterns of (a) CBW control sample (black), CBW co-milled with 47.4 wt% oxalic acid – 5 mins (red), 15 mins (blue) and 30 mins (green), respectively. (110) and (200) represent crystal face of cellulose and other peaks were identified previously to be from calcium carbonate and oxalic acid, and the corresponding (b) crystallinity index (CrI).

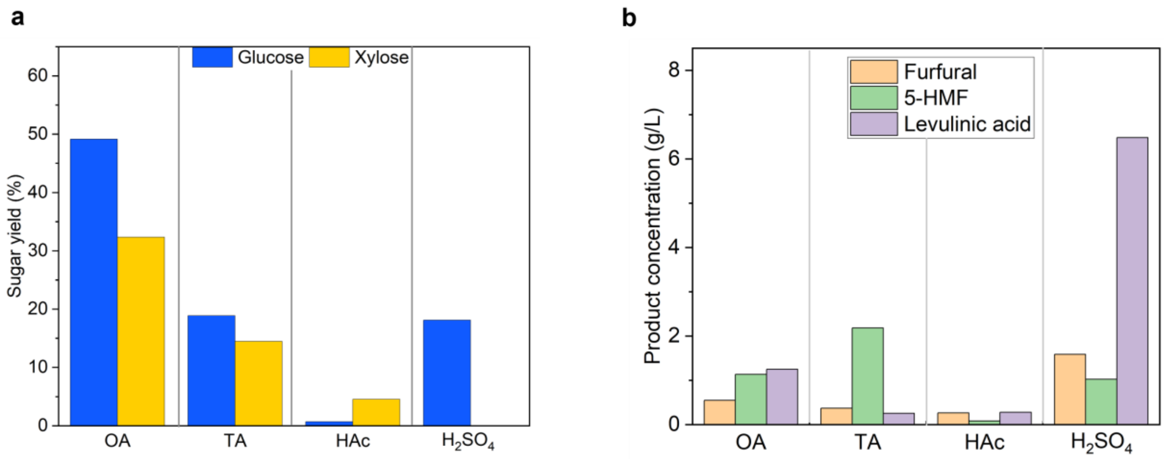


Fig. S10 Comparison of acid effects added during MHT step for hydrolysis of control milled CBW samples. Effect of adding different acids - oxalic acid (OA), tartaric acid (TA), acetic acid (HAc), and sulfuric acid (H_2SO_4) - at equivalent pH levels during MHT (180 °C, 30 min) on: (a) Sugar yield, (b) Sugar degradation product concentration.

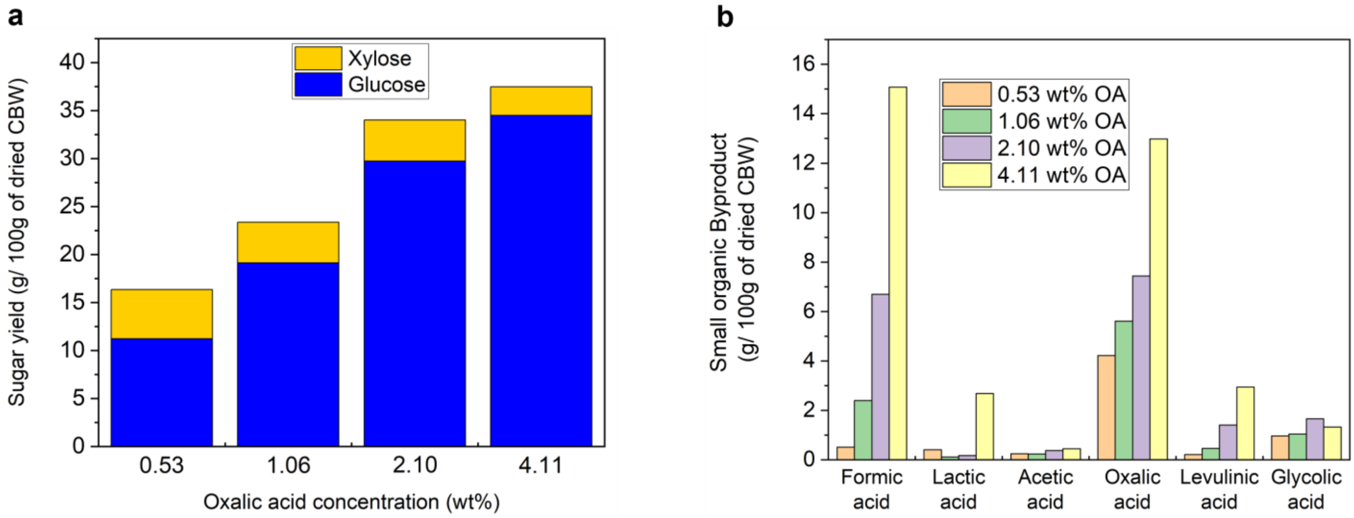


Fig. S11 Effect of varying oxalic acid concentration co-milled with CBW in MHT at 180 °C for 30 min on (a) total sugar yield and, (b) small organic byproduct generation.

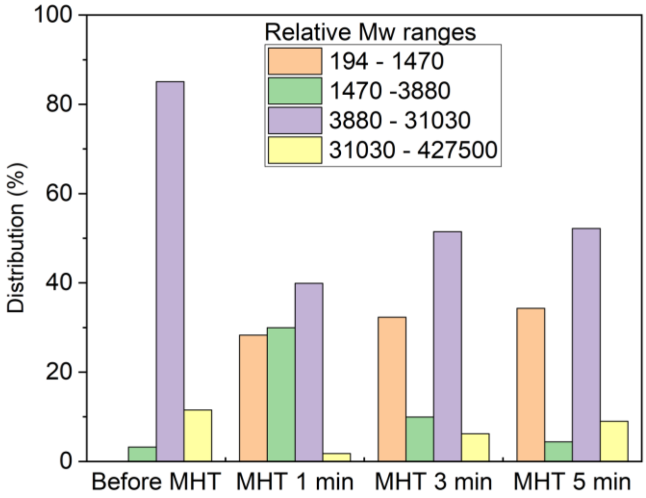
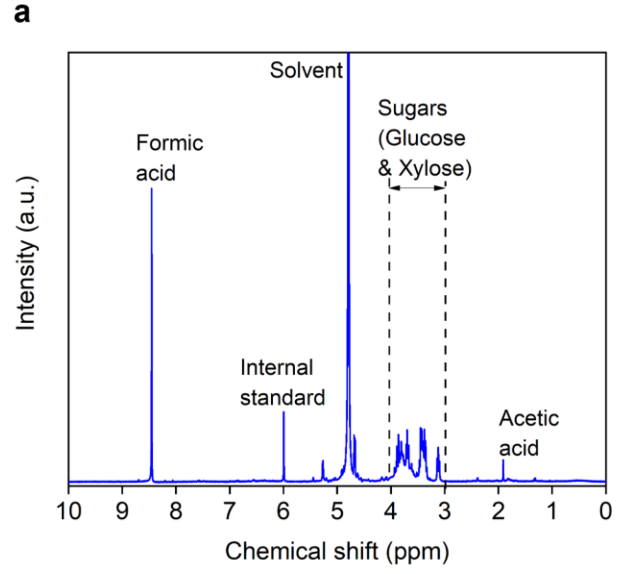


Fig. S12 Effect of different MHT durations on the product size distribution of CBW-OA from GPC analysis.



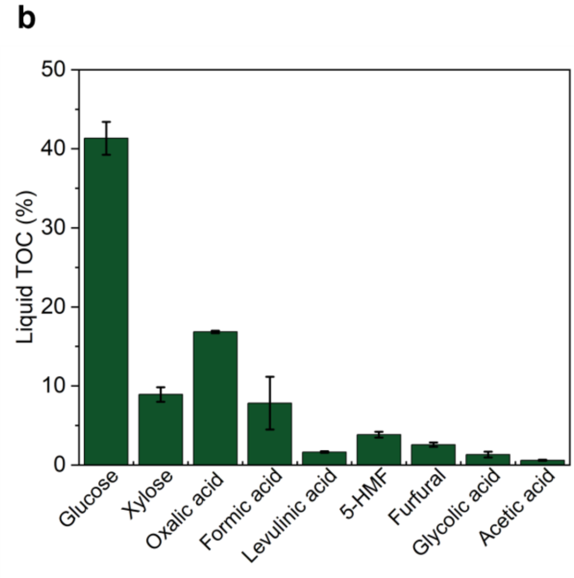


Fig. S13 Liquid products of CBW-OA at optimized conditions (MHT at 180 °C, 5 min and co-milled CBW of 4.11 wt% OA): (a) ^1H NMR spectrum (internal standard used was maleic acid with D_2O as solvent), and (b) TOC analysis.

Table S1

The main infrared peaks identified in the FTIR spectra of CBW samples in Fig. 2e.

Peaks (cm^{-1})	Type of vibration	Related Compounds	Identified in samples	Reference
3500, 1684, 1244	O-H, COOH and C-O	Oxalic acid	COA-30	[2]
3421, 3341, 3312	Stretching of O–H	Polysaccharides	All	[3]
2924, 2900, 2988 2882	Asymmetric vibration $-CH_2$ –C–H stretching	Organic compounds (polysaccharides lignin and hydrocarbon derivatives)	All	[3]
1684, 1619, 1665	Carbonyl stretching C=O	Organic compounds (carboxylate, lignin and hydrocarbon derivatives)	All	[4]
1747	C=O	Ester	COA-30	[5]
1422, 874	Asymmetric vibration of carbonate CO_3^{2-}	Calcium carbonate	C-Ctrl	[6]
1057	Stretching of secondary alcohols and ether C–O–C	Cellulose backbone	All	[7]

COA-30, COA-15 and C-Ctrl represents CBW co-mill OA 30 mins, CBW co-mill OA 15 mins and CBW control samples, respectively.

Table S2
Cardboard waste composition.

Component	**wt.%**
Total organic carbon	51.3
Hemicellulose	*9.75*
Lignin	*15.87*
Cellulose	*55.50*
Ash ($CaCO_3$)	*17.83*

All values are taken from experimental chemical composition analysis done in this work.

Table S3
Life cycle inventory of mechano(*Oxalic acid*)-bio-thermochemical treatment route of CBW.

Process	Operating Conditions/Parameters Description	Type	Flow	Amount	Unit	References or data sources
Knife and ball mill	Conventional ball mill, 0.3 kWh/kg feedstock; 0.5 mol/L Oxalic acid solution, 0.9 kg Oxalic acid/ kg CBW	In	CBW (TOC: 51.3%)	100.00	kg	[8]
		In	Oxalic acid	90.00	kg	
		In	Water	2000.00	kg	
		In	Electricity	30.00	kWh	
		Out	Mixure#1	2190.00	kg	
Microwave hydrolysis	5000 L Heating reactor	In	Mixure#1	2190.00	kg	[8]
		In	Electricity	447.93	kWh	
		Out	Emission to air, CO_2	7.85	kg	
		Out	Mixure#2	2182.15	kg	
Filter	Centrifugal dewatering (45 kWh / ton dry solid); Liquid/Solid ratio: 96%: 4%	In	Mixure#2	2182.15	kg	[8]
		In	Electricity	3.63	kWh	
		Out	Residue	87.29	kg	
		Out	Mixure#3	2094.86	kg	
Landfill	Transport to landfill site; transportation distance (assumed): 20 km	In	Residue	87.29	kg	[9]
		In	Transportation	1.75	tkm	
Anaerobic digestion	Up-flow anaerobic sludge blanket; T=35℃; SRT=20d; TOC degradation rate=40%; Biogas production=650 Nm3/t TOC degraded; Composition of biogas: 60% CH4 and 40% CO2; Biogas loss under normal conditions: 3.0 % of generated biogas.	In	Mixure#3 (18.112% TOC)	2094.86	kg	[8]
		In	Electricity (Stir)	1.26	kWh	
		Out	Emission to air, CH_4 (biogas loss)	1.27	kg	
		Out	Emission to air, CO_2 (biogas loss)	2.34	kg	
		Out	Collected biogas (41.19 kg CH_4; 75.79 kg CO_2)	116.98	kg	
		Out	Effluent	1977.88	kg	
Combined heat and power system	Biogas powered engines; The electricity recovery efficiency is 37.5%; Emission factors: CH4:434 g/GJ, CO:310 g/GJ, N_2O:1.6 g/GJ, NO_X:202 g/GJ. Biogas powered engines; The electricity recovery efficiency is 37.5%; Emission factors: CH_4:434 g/GJ, CO:310 g/GJ, N_2O:1.6 g/GJ, NO_X:202 g/GJ.	In	Collected biogas (67.28 kg CH_4; 3.79 kg CO_2)	116.98	kg	[10]
		Out	Emission to air, CO_2 (biogas combustion)	113.27	kg	
		Out	Emission to air, CH_4 (biogas combustion)	0.99	kg	
		Out	Emission to air, CO (biogas combustion)	0.71	kg	
		Out	Emission to air, N_2O (biogas combustion)	3.66	g	
		Out	Emission to air, NO_X (biogas combustion)	0.46	kg	
		Out	Recovered electricity	238.13	kWh	

Table S4

Life cycle inventory of mechano(H_2SO_4)-bio-thermochemical treatment route of CBW.

Process	Operating Conditions/Parameters Description	Type	Flow	Amount	Unit	References or data sources
Knife and ball mill	Conventional ball mill, 0.3kWh/kg feedstock; 0.2mol/L Sulphuric acid solution	In	CBW (TOC: 51.3%)	100.00	kg	[8]
		In	Sulphuric acid	39.23	kg	
		In	Water	2000.00	kg	
		In	Electricity	30.00	kWh	
		Out	Mixure#1	2139.23	kg	
Microwave hydrolysis	5000 L Heating reactor	In	Mixure#1	2139.23	kg	[8]
		In	Electricity	512.52	kWh	
		Out	Emission to air, CO_2	7.85	kg	
		Out	Mixure#2	2131.38	kg	
Filter	Centrifugal dewatering (45kWh / ton dry solid); Liquid/Solid ratio: 96%：4%	In	Mixure#2	2131.38	kg	[8]
		In	Electricity	3.63	kWh	
		Out	Residue	85.86	kg	
		Out	Mixure#3	2045.52	kg	
Landfill	Transport to landfill site; transportation distance (assumed): 20 km	In	Residue	85.86	kg	
		In	Transportation	1.72	tkm	
Anaerobic digestion	Up-flow anaerobic sludge blanket; T=35 ℃; SRT=20 d; TOC degradation rate=40%; Biogas production=650 Nm^3/t TOC degraded; Composition of biogas: 60% CH_4 and 40% CO_2; Biogas loss under normal conditions: 3.0 % of generated biogas.	In	Mixure#3 (9859 ppm TOC)	2045.52	kg	[9]
		In	Electricity (Stir)	1.26	kWh	[8]
		Out	Emission to air, CH_4 (biogas loss)	0.07	kg	
		Out	Emission to air, CO_2 (biogas loss)	0.12	kg	
		Out	Collected biogas (2.19 kg CH_4; 4.03 kg CO_2)	6.22	kg	
		Out	Effluent	2039.30	kg	
Combined heat and power system	Biogas powered engines; The electricity recovery efficiency is 37.5%; Emission factors: CH_4:434 g/GJ, CO:310 g/GJ, N_2O:1.6 g/GJ, NO_X:202 g/GJ.	In	Collected biogas (2.19 kg CH_4; 4.03 kg CO_2)	6.22	kg	[10]
		Out	Emission to air, CO_2 (biogas combustion)	10.05	kg	
		Out	Emission to air, CH_4 (biogas combustion)	0.05	kg	
		Out	Emission to air, CO (biogas combustion)	0.04	kg	
		Out	Emission to air, N_2O (biogas combustion)	0.19	g	
		Out	Emission to air, NO_X (biogas combustion)	0.02	kg	
		Out	Recovered electricity	12.66	kWh	
		Out	Recovered heat	75.97	MJ	

Table S5
Life cycle inventory of conventional (*incineration*) treatment route of CBW.

Process	Operating Conditions/Parameters Description	Type	Flow	Amount	Unit	References or data sources
Waste incineration power plant	Fluidized bed combustors; 9.5 kWh electricity consumption/ton dry solid; 12.2 kg NaOH, 4.96 kg Lime, 3.72 kg Ammonia water/ton dry solid; The electricity recovery efficiency of the sludge incineration is assumed to be 37.5 %.	In	CBW (TOC: 51.3%)	100.00	kg	[11]
		In	Electricity	0.95	kWh	
		In	NaOH	1.22	kg	
		In	Lime	0.50	kg	
		In	Ammonia water	0.37	kg	
		Out	Emission to air, CO_2	188.10	kg	
		Out	Recovered electricity	152.60	kWh	
		Out	Waste heat	915.63	MJ	
		Out	Ash	9.98	kg	
Safe landfill	Transport to landfill site; transportation distance (assumed): 20 km	In	Residue	9.98	kg	[9]
		In	Transportation	0.20	tkm	

Table S6
Life cycle inventory of conventional (*landfill*) treatment route of CBW.

Process	Operating Conditions/Parameters Description	Type	Flow	Amount	Unit	References or data sources
Landfill	Transport to landfill site; transportation distance (assumed): 20 km; Landfill gas (CO_2:CH_4=1:1); The proportion of CH_4 not oxidized before release: 0.9;	In	CBW (TOC: 51.3%)	100.00	kg	[9]
		In	Electricity	0.02	kWh	
		In	Low density polyethylene	2.7672	g	
		In	Pesticide	1.508	g	
		In	Sand	26.24	kg	
		In	Transportation	2.00	tkm	
		Out	Emission to air, CO_2	94.05	kg	
		Out	Emission to air, CH_4	30.78	kg	

Table S7

Choice of background inventory data.

<table>
<tr><th>External process</th><th>Data source (Ecoinvent 3.5)</th></tr>
<tr><td rowspan="6">Electricity</td><td>Electricity, low voltage (SG)| market for electricity, low voltage | Cut-off, S</td></tr>
<tr><td>Electricity, high voltage (RoW)| electricity production, natural gas, combined cycle power plant | Cut-off, S</td></tr>
<tr><td>Electricity, high voltage (RoW)| electricity production, solar tower power plant, 20 MW | Cut-off, S</td></tr>
<tr><td>Electricity, high voltage (RoW)| electricity production, wind, <1MW turbine, onshore | Cut-off, S</td></tr>
<tr><td>Electricity, high voltage (RoW)| electricity production, hydro, pumped storage | Cut-off, S</td></tr>
<tr><td>Recovered Electricity, low voltage (SG)| market for electricity, low voltage | Cut-off, S</td></tr>
<tr><td>Water</td><td>Water, decarbonised (RoW}| market for water, decarbonised | Cut-off, S</td></tr>
<tr><td>Sulphuric acid</td><td>Sulfuric acid (RoW)| market for sulfuric acid | Cut-off, S</td></tr>
<tr><td>Sodium hydroxide</td><td>Sodium hydroxide, without water, in 50% solution state (GLO)| market for | Cut-off, S</td></tr>
<tr><td>Lime</td><td>Lime, hydrated, packed (RoW)| market for lime, hydrated, packed | Cut-off, S</td></tr>
<tr><td>Ammonia</td><td>Ammonia, liquid (RoW)| market for | Cut-off, S</td></tr>
<tr><td>Polyethylene</td><td>Polyethylene, linear low density, granulate (RoW)| production | Cut-off, S</td></tr>
<tr><td>Pesticide</td><td>Pesticide, unspecified (RoW)| production | Cut-off, S</td></tr>
<tr><td>Sand</td><td>Sand (RoW)| market for sand | Cut-off, S</td></tr>
<tr><td>Transport</td><td>Transport, freight, light commercial vehicle (GLO)| market group for transport, freight, light commercial vehicle | Cut-off, S</td></tr>
</table>

Table S8
Background inventory data of oxalic acid production.[12]

Process	Type	Flow	Amount	Unit
Oxalic acid production	In	Electricity	0.678	kWh
	In	Natural gas	0.807	MJ
	In	Water	5.000	kg
	In	Sugar	0.591	kg
	In	Oxygen	0.510	kg
	In	Sulfuric acid	0.048	kg
	In	Hydrogen peroxide	0.017	kg
	Out	Oxalic acid	1.000	kg

Table S9
Environmental impact categories in ReCiPe 2016 midpoint method and their normalization factors (World (2010) H).

Environmental impact	**Unit**	**Normalization factors**
Environmental impacts related to air quality		
GWP	kg CO_2 eq^{-1}	1.25×10^{-4}
ODP	kg CFC-11 eq^{-1}	1.67×10^{1}
PMFP	kg $PM_{2.5}$ eq^{-1}	3.91×10^{-2}
EOFP	kg NO_x eq^{-1}	5.63×10^{-2}
Environmental impacts related to water quality		
FEP	kg P eq^{-1}	1.54×10^{0}
MEP	kg N eq^{-1}	2.17×10^{-1}
FETP	kg 1,4-DCB eq^{-1}	8.15×10^{-1}
METP	kg 1,4-DCB eq^{-1}	9.69×10^{-1}
WCP	m^3 eq^{-1}	3.75×10^{-3}
Environmental impacts related to soil quality		
TAP	kg SO_2 eq^{-1}	2.44×10^{-2}
TETP	kg 1,4-DCB eq^{-1}	9.65×10^{-4}
LU	m^2a crop eq^{-1}	1.62×10^{-4}
Environmental impacts related to human health		
HCTP	kg 1,4-DCB eq^{-1}	3.61×10^{-1}
HNCTP	kg 1,4-DCB eq^{-1}	6.71×10^{-3}
IR	kBq Co-60 eq^{-1}	2.08×10^{-3}
HOFP	kg NO_x eq^{-1}	4.86×10^{-2}
Environmental impacts related to resource usage		
MDP	kg Cu eq^{-1}	8.33×10^{-6}
FDP	kg oil eq^{-1}	1.02×10^{-3}

Table S10

Effect of Control Milling and Oxalic Acid Co-Milling on Cellulose Molecular Weight and Degree of Polymerization (isolated cellulose of cardboard waste after treatment).

Sample	**Speed (RPM)**	**Time (mins)**	**Molecular weight of CTC (x 10^4)**	**DP of cellulose**
Control	1000	30	14.60	422
Co-mill OA	1000	30	3.54	102

*CTC refers to cellulose tricarbanilate. The average molecular weight (Mw) of CTC was obtained by converting from a universal calibration curve of polystyrene standards [13]. The CTC DP was then calculated by dividing Mw by 519. This value was normalized to DP of cellulose by multiplying by 1.5, correcting for a degree of substitution of 2. [14]

Table S11 Benchmark of dilute acid hydrothermal conversion of different feedstocks to sugar.

Catalyst	Feedstock	Temperature (°C)	Time (h)	Loading (g/ml)	Xylose Yield (%) [a]	Glucose Yield (%) [b]	Total sugar (g/100g dry mass) [c]	Reference
H_2SO_4 (0.25–1.00 wt%)	Aspen	160, 175, and 190	0.53	0.05	85.0	12.0	18.70	[15]
H_2SO_4 (0.25–1.00 wt%)	Basswood	160, 175, and 190	0.53	0.05	80.0	12.0	17.53	[15]
H_2SO_4 (0.25–1.00 wt%)	Red maple	160, 175, and 190	0.53	0.05	82.0	13.0	20.12	[15]
H_2SO_4 (0.25–1.00 wt%)	Switchgrass	160, 175, and 190	0.53	0.05	94.0	10.0	22.69	[15]
H_2SO_4 (0.34 wt%)	Sugarcane bagasse	210	Two-step: 1) 0.17 2) 0.67	0.05	91.0	36.0	37.80	[16]
H_2SO_4 (0.20 wt%)	Paulownia	Two-step *MHT*: 1) 180 2) 210	Two-step: 1) 0.50 2) 0.50 *Ramp	0.05	100.0	53.0	38.10	[17]
H_2SO_4 (1.50 wt%)	Corncob	125	1.50	0.10	83.4	7.8	31.76	[18]
H_2SO_4 (1.00 wt%)	Oil palm empty fruit bunch	190 *MHT*	0.05 *Ramp	0.10	77.5	17.3	20.30	[19]
Weak acid Carbon (0.10 g)	Japanese cedar wood	210	1.00	0.01	34.7	25.3	5.10	[20]
Biochar (0.20 g)	Switch grass	100	6	0.05	28.2	11.2	27.00 [d]	[21]
Oxalic acid (1.10 w/v%)	Oil palm empty fruit bunch	190 *MHT*	0.05	0.10	40.0% of total yield [e]		27.12	[22]
Oxalic acid (0.50 wt%)	Maple wood	160	0.46	0.10	73.9	1.7	19.30	[23]
Oxalic acid (0.73 wt%)	Yellow poplar	158	0.22	0.25	72.0	Not tested	12.25	[24]
H_2SO_4 (10.00 wt%)	Cardboard waste	96	3	0.04	6.09 g/L of reducing sugars		15.22	[25]
Oxalic acid (4.11 wt%)	**Cardboard waste**	**140 *MHT***	**0.08**	**0.05**	**100.0**	**19.4**	**25.42**	**This work**
Oxalic acid (4.11 wt%)	**Cardboard waste**	**180 *MHT***	**0.08**	**0.05**	**84.3**	**69.8**	**52.32**	**This work**

[a] Yield % calculation based on total mass of xylose measured to total mass of xylan in hemicellulose, [b] Yield % calculation based on total mass of glucose measured to total mass of cellulose, [c] g/100g dry mass calculated from total mass of glucose and xylose measured per 100g of dry biomass, [d] g/100g dry mass calculated from total mass of glucose, xylose, galactose, arabinose and mannose measured per 100g of dry biomass, [e] Total reducing sugar yield%.

MHT: Microwave hydrothermal treatment.

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