Supplementary Information (SI) for Energy & Environmental Science. This journal is © The Royal Society of Chemistry 2025

Supplementary Materials for

Catalytic Ultrasound-driven Synthesis of Syngas from CO₂ Saturated Water

Lina Chen,^a Yi Qin,^b Claire T. Coulthard,^a Zoë R. Turner,^a Chunping Chen,^{*a} James Kwan^{*b} and Dermot O'Hare^{*a}

- ^{a.} Department of Chemistry, University of Oxford, Chemistry Research Laboratory, Oxford, OX1 3TA, UK
- ^{b.} Department of Engineering Science, University of Oxford, Oxford, OX1 3PJ, UK

Contents

Methods Supporting Figures Supporting Tables Future prospect for CO₂ sonocatalysis

Methods

Materials.

Sodium hydroxide, sodium carbonate, CuCO₃.Cu(OH)₂, Cu(NO₃)₂·3H₂O, Zn(NO₃)₂·6H₂O, Al(NO₃)₃·9H₂O, dimethyl sulfoxide (DMSO), AgNO₃, and 5,5-Dimethyl-1-Pyrroline-N-Oxide (DMPO) were purchased from Sigma-Aldrich. All chemicals and materials were used without purification. 70% nitric acid and 25 wt% tetrapropylammonium hydroxide (TPAOH) was purchased from Thermo Fisher Scientific. The standard gases for calibration are ordered from BUSE. The pure CO₂ (99.999%), nitrogen (99.999%), Argon (99.999%), and 5% CO₂ in Ar were ordered from BOC. Deionized water (18.2 M Ω cm⁻²) was used in the synthesis and the CO₂ sonocatalysis. Degassed DI water was to fill the sonochemical cylindrical reactor and prepared using a degas unit, which consists of a membrane contractor (3 M Liqui-CeITM G541), vacuum pump (Welch MPC 090 E, type 412021) and a water pump (Flojet LFP, 24 V RLFP222202D), to degas the DI water with the oxygen concentration lower than 20% (Duo pH/Ion/DO meter SG98, Mettler Toledo).

Synthesis of CuZnAl-LDHs.

Cu_{0.8}ZnAl-LDH: CuCO₃.Cu(OH)₂ (0.27g, 1.29 mmol), Zn(NO₃)₂.6H₂O (1.1 g, 3.70 mmol), and Al(NO₃)₃.9H₂O (1.4 g, 3.73 mmol) were dissolved in DI water (256 mL) with 70% HNO₃ (0.82 mL, 9.1 mmol). In a separate beaker deionised water (240 mL) was adjusted to pH 10 with TPAOH. The metal salt solution was added dropwise at 152 mL h⁻¹ under stirring at 500 rpm whilst pH was kept constant at 10 with TPAOH. Once all metal salt solution had been added into the solution, the mixture was then aged at room temperature for 1 hour at 500 rpm. The LDH was collected by filtration, washed with water until pH 7, and dried overnight at 30 °C under vacuum to afford Cu_{0.8}ZnAl-LDH.

For preparing samples with different copper loading, only the relative molar proportions of metal salts are adjusted as appropriate, the rest of the procedure remains the same and the samples are named as Cu_xZnAl-LDH, where x is the stoichiometry of Cu in the sample. The sample ZnAl-LDH is LDH without Cu.

Calcination of CuZnAl-LDHs.

CuZnAl-LDHs were calcined at 500 °C for 5 hours at a 2 °C min⁻¹ ramp rate under constant air flow at 50 mL min⁻¹ to obtain Cu_xZnAl-LDOs, following with the reduction of Cu_xZnAl-LDOs at 290 °C for 2 hours at a ramp rate of 2 °C min⁻¹ under constant gas flow of H₂ at 50 stp mL min⁻¹ (stp = standard temperature and pressure: P = 101.3 kPa, T = 298 K). The sample after reduction treatment was denoted as r-Cu_x/ZnAl-LDO, e.g. r-Cu_{0.8}/ZnAl-LDO.

Acoustic setup.

A cylindrical sonochemical reactor was employed in all acoustic experiments detailed in our previous research.¹⁵ In brief, a function generator (Keysight Technologies, 3600A) delivered a 1 MHz tone burst that was amplified with a fixed gain 55 dB RF power amplifier (Electronics & Innovation, 1040L) to a bespoke 1 MHz ultrasound tube transducer. The tone burst pulse sequence consisted of 100 cycles with a burst period of 1 ms.

This ultrasound transducer created a radially diverging pulse that was reflected 180° to form a converging acoustic pressure wave within a 2.5 mL volume. The pulsed ultrasound, rather than the conventional continuous wave excitations, was employed to minimise heating and prevent second order bubble effects that reduce sonochemistry, such as coalescence and non-spherical bubble motion. The duty cycle (*D*) is calculated as follows:

$$D = \frac{Nc}{T_{\text{burst}} \times f}$$

where *D* represents the duty cycle (dimensionless), Nc represents the number of cycles (dimensionless), T_{burst} represents the burst period (s), *f* represents the frequency (Hz).

CO2 sonocatalysis experiment

To run the CO₂ sonocatalysis experiments, 10 mg catalyst was dispersed in 2.5 mL DI water in a 5 mL polypropylene vial. Pure CO₂ was bubbled into the suspension for 15 min to obtain a CO₂-saturated system before being sealed. Once sealed, the vial was placed in the centre of the sonoreactor (Supplementary Fig. 8) before exposure to ultrasound irradiation for a given time. All reactions were performed at ambient pressure and room temperature. The acoustic parameters of sonoreactor for CO₂ sonocatalysis have been provided in Supplementary Table 2. After the sonication, the gas product was collected and directly analysed by GC (SHIMADZU GCMS 2030) equipped with Carboxen®-1010 PLOT Capillary GC column. using the barrier discharge ionization (BID) as the detector; liquid products were analysed by ¹H NMR spectroscopy. ¹H NMR spectra were recorded on a Brucker AVIII HD 500 spectrometer with a pre-saturation technique to suppress the water signal, in which 0.5 mL filtered solution was mixed with 0.1 mL DMSO (internal standard, diluted to 100 ppm (v/v) by deuterated water). ¹H spectrum was recorded at 298 °K and referenced internally to the residual protio-solvent resonance of the deuterated water used. The ¹³C isotopic labelling experiment performed with similar procedures except for replacing the CO₂ by ¹³CO₂.

Cavitation detection and analysis

The in-situ detection of cavitation was conducted within the sonoreactor; the transmitting transducer used had a 1.17 MHz centre frequency. Acoustic responses during the sonochemical reactions were detected using a 3.5 MHz centre frequency passive cavitation detector (PCD) (Olympus, V384) aligned orthogonal to the incident acoustic wave transmitted by the ultrasound transducer. The PCD signal was filtered with a 1.8 MHz high-pass filter (Thorlabs, EF509) before 20 dB amplification by a broadband amplifier (Edgerton, Germeshausen, and Grier, Model 5185). The amplified signal was captured for subsequent analysis (Supplementary Fig. 18) using a data acquisition device (Tiepie Engineering, HS5) and monitored in real-time with an oscilloscope (Keysight Technologies, DSOX2024A). Detailed acoustic parameters of the sonoreactor for cavitation detection have been provided in Supplementary Table 5.

To identify cavitation events, the recorded noise signals were post-processed using a power Fast Fourier Transform (FFT) to generate a power spectral density (PSD) curve for each individual ultrasound burst^{11,36}. A metric proportional to the power of the noise from cavitation, simply referred to as cavitation power henceforth, was calculated by summing across the frequency spectrum. For each burst, the cavitation power was compared to that of degassed water exposed to ultrasound under identical conditions. A cavitation event was defined as a cavitation power from the sample being 6 dB higher than the cavitation power from the degassed water control. The probability of cavitation was determined as the percentage of bursts that recorded a cavitation event out of the total number of bursts at a given driving voltage. Cavitation threshold was defined here as the drive voltage at which 50% of the bursts resulted in cavitation.

The calculation on the productivity and selectivity of products (CO and H₂)

The number of mol of CO produced is calculated according to following equation:

$$n_{\rm co} = \frac{P \times V_{co}}{R \times T}$$

P is the pressure in the atmosphere (1 atm), V_{CO} is the total volume of CO produced, R is the gas constant (0.082 L atm K⁻¹ mol⁻¹), and T is the temperature based on the laboratory condition (293 K).

The V_{co} was evaluated from the GC calibration data (Supplementary Fig. 16). Similar calculation is conducted to the number of the mol of H₂ produced.

The productivity of CO (p_{co}) is calculated as followings:

$$p_{\rm co} = \frac{n_{co}}{m_{cat} \times t_{irradiation}}$$

 n_{co} is the number of mol of CO produced (µmol), m_{cat} is the mass of catalyst used (g); and $t_{irradiation}$ is the ultrasound irradiation time (h). Similar calculation is performed to get the productivity of H₂ (p_{H2}). Both amount of CO and H₂ in the liquid phase is not counted in the calculations. Note that:

$$t_{irradiation} = D \times t_{total}$$

The selectivity of the CO (S_{CO}) and H₂ (S_{H2}) is calculated as followings:

$$S_{co} = \frac{n_{co}}{n_{total \, products}} \times 100\% = \frac{n_{co}}{n_{co} + n_{H2}} \times 100\%$$

 $S_{\rm H2} = \frac{n_{H2}}{n_{total \, products}} \times 100\% = \frac{n_{H2}}{n_{co} + n_{H2}} \times 100\%$

 $n_{total products}$, n_{co} , and n_{H2} is the number of mol of total products, CO and H₂ produced (µmol) from CO₂ sonocatalysis, respectively.

Sonochemical efficiency (SE) calculation

The sonochemical efficiency for CO₂ sonocatalysis was based on the following equation:

$$SE = \frac{Yield_{product}}{(PD \times t_{irradiation}) \times m_{cat} \times V_{reactor}} = \frac{n_{H2} + n_{co}}{(PD \times t_{irradiation}) \times m_{cat} \times V_{reactor}}$$

PD represents the power density (kW), which is determined as below:

$$PD = V_{rms} \times I_{rms} \times \cos\theta$$
$$PD = \frac{V_{pk-pk} \times I_{pk-pk} \times \cos\theta}{8}$$

Where *Yield*_{product} represents the amount of CO and H₂ produced (µmol), *PD* represents the power density (kW), and $t_{irradiation}$ is the ultrasound irradiation time (s), m_{cat} represents the mass of catalyst (g) and $V_{reactor}$ represents the volume of sonoreactor vessel (L) with exposure to the irradiation. The following SE calculation example is based on the data from CO₂ sonocatalysis in 5%CO₂/Ar with r-Cu_{0.8}ZnAl-LDO catalyst for irradiation time for 1 h: $t_{irradiation} = 1 h = 3600 \text{ s}, PD = 0.212 \text{ kW}, m_{cat} = 0.01 \text{ g}, V_{reactor} = 0.0025 \text{ L}, n_{H2} = 1.501 \text{ µmol and } n_{co} = 2.527 \text{ µmol}$. Thus, 2.527 + 1.501

the SE = $\overline{(0.212 \times 3600) \times 0.01 \times 0.0025}$ = 211.1 µmol kJ⁻¹ g⁻¹ L⁻¹.

Characterisation.

X-ray diffraction (XRD) measurements were recorded on Bruker D8 diffractometer (40 kV and 30 mA) with Cu K α radiation (λ 1 = 1.544 Å and λ 2 = 1.541 Å). XRD characterizations in Figure 1 and Figure S1 used the PMMA holder whereas in Figure 6 used the holder for air-sensitive holder.

Transmission electron microscopy (TEM) images and EDX were obtained from a JEOL 2100 electron microscope with an accelerating voltage of 200 kV using a single tilt specimen at DCCEM within the Department of Materials, University of Oxford. HR-TEM were obtained from JEOL ARM300CF instrument with an accelerating voltage of 300 kV at the electron Physical Sciences Imaging Centre (ePSIC).

Inductively coupled plasma optical emission spectrometry (ICP-OES) was used to analyse the Cu, Zn and Al elements using a Themo Fisher Scientific iCAP 7400 Duo ICP Spectrometer in the University of Cambridge.

XPS data was collected on K-alpha XPS X-ray photoelectron spectrometer (Thermo Fisher Scientific) in the Department of Materials, University of Oxford.

XAFS spectra was recorded at the Cu K absorption edge, in transmission mode in B18, Diamond light source. The Demeter software package (Athena)³⁸⁻⁴⁰ was used for XAFS data analysis for the Cu data. The spectra were calibrated with foils as a reference. To ascertain the reproducibility of the experimental data, at least two scan sets were collected.

Electron paramagnetic resonance (EPR) data was recorded on a Bruker Biospin EMXmicro with continuous wave EPR (CW-EPR) spectrometer operating at X-band (9.1–9.9 GHz), in the Centre for Advanced ESR (CAESR) of the Department of Chemistry, University of Oxford. All EPR data was processed with EasySpin.⁴¹

Supplementary Figures



Fig. S1. XRD paterns. XRD patterns of Cu_xZnAl-LDHs (**a**) and r-Cu_x/ZnAl-LDOs (**b**).



Figure S2. IR characterization of catalyst precursors.



Fig. S3. TEM and HRTEM images. TEM and HRTEM images of r-ZnAl-LDO (**a**, **b**), r-Cu_{0.4}/ZnAl-LDO (**c**, **d**), r-Cu_{1.4}/ZnAl-LDO (**e**, **f**) and r-Cu_{2.8}/ZnAl-LDO (**g**, **h**).



Fig. S4. STEM images with elemental mapping results. STEM images of $r-Cu_{0.4}/ZnAl-LDO$ (**a**), $r-Cu_{1.4}/ZnAl-LDO$ (**b**) and $r-Cu_{2.8}/ZnAl-LDO$ (**c**) with its elemental mapping results of Cu, Zn, Al and O.



Fig. S5. XPS surveys. XPS surveys of r-Cu_{0.4}/ZnAl-LDO (a), r-Cu_{1.4}/ZnAl-LDO (b) and r-Cu_{2.8}/ZnAl-LDO (c).



Fig. S6. High-resolution XPS spectra. High-resolution XPS spectra of Cu 2p (a), Zn 2p (b), Al 2s (c) and Al 2p (d) spectra of r-Cu_x/ZnAl-LDOs catalysts.



Supplementary Fig. 7. The Cu K-edge XANES first-order differential of Cu foil, CuO and r-Cu_{0.8}/ZnAl-LDO.



Fig. S8. EXAFS k space curves of Cu foil, CuO and r-Cu_{0.8}/ZnAl-LDO.



Fig. S9. A photograph of sonochemical cylindrical reactor design, along with the home-made chamber as the reactor.



Fig. S10. ¹H NMR (500 MHz, 298 K, D₂O) spectrum of liquid after CO₂ sonocatalysis over r-Cu_{0.8}/ZnAl-LDO catalyst. ¹H chemical shifts, δ , are given in parts per million (ppm).



Fig. S11. The GC signals patterns of gaseous products determined by BID detector after CO₂ sonocatalysis over r-Cu_{0.8}/ZnAl-LDO catalyst.



Fig. S12. GC results from sonocatalysis in pure N_2 and Ar. The gaseous products from the sonocatalysis conducting in pure N_2 (a) and pure Ar (b) without the introduction of CO_2 over the r-Cu_{0.8}/ZnAl-LDO catalyst. Note: the retention time of gaseous products changed due to the upgradation of GC and all calibration curves were redone accordingly for data analysis.



NO		CO peak area				
	Source	m/z = 28	m/z=29			
1	CO ₂	42020	639			
2	CO ₂	42260	686			
3	CO ₂	41282	625			
4	CO ₂	35458	588			
5	CO ₂	24522	510			
6	CO ₂	24622	482			
7	CO ₂	24090	481			
8	CO ₂	21876	412			
9	CO ₂	22871	439			
10	CO ₂	27412	447			
11	CO ₂	21530	408			
12	CO ₂	15908	290			



NO	CO ₂	CO peak intensity				
	Source	m/z = 28	m/z=29			
1	¹³ CO ₂	95543	6044			
2	¹³ CO ₂	100351	6882			
3	¹³ CO ₂	37428	8734			
4	¹³ CO ₂	36289	8281			
5	¹³ CO ₂	13188	10434			
6	¹³ CO ₂	124826	13010			
7	¹³ CO ₂	111647	10388			
8	¹³ CO ₂	33311	6404			
9	¹³ CO ₂	32964	7373			
10	¹³ CO ₂	26487	8998			
11	¹³ CO ₂	25860	8487			
12	¹³ CO ₂	21260	4910			



Fig. S13. ${}^{13}CO_2$ isotopic experiment to determine the carbon source of CO. The m/z analysis of CO peak in GC with the gas supply of non-labbeling CO₂ (**a**) and labelling ${}^{13}CO_2$ (**b**). (**c**) The GC signals of CO peaks under the ${}^{12}CO_2$ gas supply.



Fig. S14. The determination of the source of CH_4 . Heating the catalyst in pure CO_2 . Insets are the enlarged GC signals for H2 and CO. (a) and repeated labbelling experiments of CO_2 sonocatalysis by using ¹³CO₂ as gas source(**b**, **c**).(**d**) The GC signals of CH₄ peaks with D₂O as solvent, along with its m/z signals.

The confimation of the origin of CH₄: First, the signal of CH₄ is detected when the catalyst was heated at 40 °C in water without the ultrasound (Figure S13a), suggesting CH₄ is released from catalyst itself. Second, CH₄ signal was still observed when sonocatalysis was performed under pure Ar or pure N₂ without the introduction of CO₂ (Figure S11), further confirming that CH₄ is not derived from CO₂ conversion. Third, isotope-labelling experiments of ¹³CO₂ and D₂O were conducted, respectively. In the ¹³CO₂ labelling experiment, the m/z = 17 signal corresponds to OH from water rather than ¹³CH₄, as verified by multiple experiments where m/z =17/18 ratio remained constant, (Figure S13b and S13c), aligning to the water signal. As a result, there is no ¹³CH₄ generated with the ¹³CO₂ gas supply. Similarly, in the D₂O labelling experiment, the observed signals from methane peak (16, 17 and 18) were attributed to CH₄ and water with no evidence of CD₄ or CD_xH_(4-x) species (Figure S13d). Taking all these investigation into account, CH₄ is from catalyst itself.



Fig. S15. The comparison of CO productivity among the catalysts of r-ZnAl-LDO without Cu incorporation, r-Cu_{0.8}/ZnAl-LDO and r-CuO without the incorporation of ZnAl-LDO.



Fig. S16. The XPS spectra of Cu 2p for r-CuO and commercial CuO.



Fig. S17. The standard curves showing the correlation of gaseous products concentration with the areas of GC peak. Experimental GC calibration for H_2 (a) and CO (b).



Fig. S18. The comparison of CO and H₂ productivity between the r-Cu_{0.8}/ZnAl-LDO and physical mixtures.



Fig. S19. The schematic diagram of the data acquisition for cavitation detection.



Fig. S20. The comparison of the gaseous productivities from sonocatalysis among 5% CO_2/Ar without catalyst, pure CO_2 with catalyst and 5% CO_2/Ar with catalyst.



Fig. S21. The characterisations of TEM and elemental mapping of catalyst after CO_2 sonocatalysis. TEM image (a) and element mapping result (b) of r-Cu_{0.8}/ZnAl-LDO.

Supplementary Tables

	wt	% in r-Cu _x /ZnAl-l	LDO	stoichiometry in r-Cu _x /ZnAl-LDO			
	Cu	Zn	AI	Cu	Zn	AI	
r-ZnAl-LDO	0	47.4	17.1	0	1.1	1	
r-Cu _{0.4} /ZnAl-LDO	14.9	43.1	14.7	0.4	1.2	1	
r-Cu _{0.8} /ZnAl-LDO	24.9	35.8	13.3	0.8	1.1	1	
r-Cu _{1.4} /ZnAl-LDO	38.4	27.7	11.8	1.4	1.0	1	
r-Cu _{2.8} /ZnAl-LDO	53.6	18.3	8.2	2.8	0.9	1	

Table S1. The ICP-OES results of r-Cu_x/ZnAl-LDOs.

Note: Calibration was performed by construction of a standard curve using ICP standards from Sigma-Aldrich. Samples were run in ~2% nitric acid (Fisher TraceMetal grade). Samples were weighed on a Mettler UMT2 balance and dissolved in 5 mL nitric acid, diluted with 5 mL water and a 0.5 mL aliquot was diluted to 10 mL with water. There is a total error of <5% on the values obtained (<2% measurement error, <3% sample preparation error)

Table S2. Acoustic parameters of sonoreactor for CO₂ sonocatalysis.

Sonoreactor parameters							
Frequency (MHz)	1.059						
Number of cycles	100						
Burst period (ms)	1						
peak-to-peak voltage (V)	242.1						
peak-to-peak current (A)	7.26						
Phase angle (degree)	14.4						

Sonoreactor parameters for cavitation detection								
Frequency (MHz)	Number of cycles	Burst period (ms)	peak-to-peak voltage (V)	peak-to-peak current (A)	Phase angle (degree)			
1.17	100	1	10.8	0.2	19.8			
			21.3	0.3	19.4			
			31.6	0.5	19.8			
			42.4	0.7	18.9			
			53.5	0.8	18.5			
			65.6	1	18.1			
		Ī	77.9	1.2	17.7			
			90.4	1.4	17.7			
			103	1.6	17.3			
			116.2	1.8	17.3			
			129	2	16.8			
			142.3	2.2	16.9			
			155.9	2.4	17.3			
			169.7	2.6	16.8			
			183	2.9	16.8			
			194.7	3	16.9			
			211.1	3.3	16.8			
			223.9	3.5	16.8			
			237.2	3.7	16.8			
			251.4	3.9	16.4			

Table S3. Acoustic parameters of sonoreactor for cavitation detection.

Table S4. The calculation of CO/H_2 ration among several individual tests for r-Cu_{0.8}ZnAl-LDO catalyst in 5% CO₂/Ar.

No	H ₂ productivity (µmol g ⁻¹ h ⁻¹)	CO productivity (µmol g ⁻¹ h ⁻¹)	CO/H ₂ ratio
1	140	244	1.7
2	166	191	1.2
3	144	323	2.2

Table S5.	Comparison	the	performance	of	$\rm CO_2$	conversion	of	r-Cu _{0.8} /ZnAl-LDO	with	other	ultrasound-triggered	catalytic
systems.												

Catalysis	Catalyst	Looding	Pegeter	Dowor	<u> </u>	Other products	°E	Pof
Catalysis	Catalyst	Loading	Reactor	Power	broductivity		OE	Rei.
		(mg)	size	(KVV)	productivity	productivity		
			(mL)		(µmorg ' n ')	(µmorg 'n ')		
	r-Cu _{0.8} /ZnAl- LDO	10	2.5	0.212	252.7	150.1 (H ₂)	211.1	This work
CO ₂ Sonocatalysis	H ₂ Ti ₃ O ₇	5	5	-	8.3	-	-	Appl. Phys. Lett. 2022, 121, 263901.
	Raney Ni-Ru/C	2500	500	0.205	0.894	0.79 (H ₂)	0.0046	A. Koblov, PhD Thesis, Curtin University, 2011.
CO ₂ sonolysis	-	-	20	-	92.04 (µmol h ⁻¹)	-	-	Zeitschrift für Naturforschun, 1985, B, 40(1), 100-107.
	Nb-doped lead zirconate titanate	-	10	-	789	~100 (H ₂)	-	Nanoscale Adv.,2021, 3,1362–1374
piezocatalysis	Lead-free lithium-doped potassium sodium niobate	10	10	-	438	-	-	Nano Energy, 2022, 95, 107032
Contact-electro- catalytic	Cu-PCN	0.68	-	-	33	-	-	Nat. Commun. 2024, 15(1), 5913.
Sonophotocata- lysis	TiO ₂ photocatalyst	200	40	0.2+0.5	375	-	3.72	J. Photochem. Photobiol. A: Chem. 2002, 160, 11–17

Table S6. Comparison the performance of CO_2 conversion of r-Cu_{0.8}/ZnAl-LDO with those of recently representative photocatalysts without the involvement organic solvent and cocatalyst.

Catalysis	Catalyst	Loading (mg)	CO productivity (µmol g ⁻¹ h ⁻¹)	Other products productivity (µmol g ⁻¹ h ⁻¹)	Ref.
CO ₂ Sonocatalysis	r-Cu _{0.8} /ZnAl- LDO	10	252.7	150.1 (H ₂)	This work
	Van Der Waals Gap-Rich BiOCl Atomic Layer	50	188.2	<5 (H ₂)	Nat. Commun. 2021,12, 5923.
	Ag₁@PCN	2	160	10.5 (CH ₄)	Angew. Chem. Int. Ed. 2023, 62, e202304585.
	FeCoNiCuMn HEA	15	235.2	19.9 (CH ₄)	Adv. Mater. 2024, 2313209.
	Co/BNF SAC	10	32	9.13 (CH ₄ +H ₂)	Adv. Mater. 2024, 36, 2303287.
	Co-Bi ₃ O ₄ Br atomic layer	30	107.1	~0.164 (CH ₄)	Nat. Commun. 2019, 10, 2840.
	Bi ₁₂ O ₁₇ Cl ₂ NTs	30	48.6	-	Angew. Chem. Int. Ed. 2018, 130, 1.
	BiOBr-OVs Atomic Layers	100	87.4	-	Angew. Chem. Int. Ed. 2018, 130, 8855.
	Co tuned Au nanoclusters	10000	3.45	1.84 (CH ₄ +H ₂)	J. Am. Chem. Soc. 2018, 140, 16514.
	Ni doped CdS quantum dots	-	~9.5	-	Angew. Chem. Int. Ed. 2018, 57, 16447.
CO ₂	0.3-Cu/TiO ₂ -AG	12.6	10.9	490 (H ₂)	Chem, 2019, 57, 1818- 1833.
Filotocatalysis	Cu/TiO ₂ -3	-	15.3	0.65 (CH ₄)	Angew. Chem. Int. Ed. 2022, 61, e202207600
	Mn ₁ Co ₁ /CN	50	47	-	Angew. Chem. 2022, 134, e202206579.
	Au _{SA} /Cd _{1-x} S	30	32.2	11.3 (CH ₄) 7.9 (H ₂)	Nat. Commun. 2021, 12, 1675.
	Ni@6MOF/BVO	20	44.5		Adv. Mater. 2022, 34, 2205303.
	Cu-SAEB	1	236	<2.38 (CH ₄)	Angew. Chem. 2023, 135, e202218460.
	BiOCI-B-OV	50	83.64	<1.71	Adv. Mater. 2021, 33, 2100143.
	TMOF-10-NH ₂ (I) 10 78		<5 (CH ₄)	Nat. Commun. 2022, 13, 4592.	
	Cu ₁ N ₃ @ PCN-1	Cu ₁ N ₃ @ PCN-1 10 49.8		-	Angew. Chem. 2022, 61, e202207677.
	Cs ₃ Bi ₂ I ₉	6	7.76	1.49 (CH ₄)	J. Am. Chem. Soc. 2019, 141, 20434.
	QS-Co ₃ O ₄ HoMSs (ZIF-67)	5	46.3	-	J. Am. Chem. Soc. 2019, 141, 2238.

Future prospect for CO₂ sonocatalysis

The integration of CO_2 catalysis with sonochemistry presented here makes a significant contribution to the emerging field of sonocatalysis. We believe it highlights substantial new opportunities to further develop the technology by advancing sonochemical reactors, optimising operating conditions, and designing more efficient and robust catalysts to upscale CO production while minimizing the energy input. Given the positive correlation of CO production to cavitation noise, reducing the cavitation threshold is a key factor for improving CO yield. This can be achieved by reducing the drive frequency, optimizing gas compositions, and structuring the catalysts to trap gas and facilitate cavitation. Additionally, designing a larger reactor vessel that more efficiently delivers ultrasound to larger volumes presents another promising route for scaling up CO production. Future catalyst design would not only focus on prioritizing high CO production efficiency but also ensuring structural stability and operational robustness for long-term operation. Moreover, CO_2 sonocatalysis holds great potential to produce higher-value products beyond CO by developing catalysts with different types of active sites. Since we are still in the early stages of development, advancements in acoustic technology—such as those listed above—are anticipated to significantly improve ultrasound-to-fuel conversion efficiency in the future.