Supporting Information

Sol-gel hydrothermal synthesis of microstructured CaO-based adsorbents for CO₂ capture

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Fig. S1 XRD pattern of limestone (CaCO₃).



Fig. S2 FTIR spectrum of 3D hierarchical CaCO₃ hollow microspherical adsorbent composed of 1D spikeshaped nanorods



Fig. S3 EDX spectrum of 3D hierarchical CaCO₃ hollow microspherical adsorbent composed of 1D spikeshaped nanorods



Fig. S4 FESEM image of the commercial limestone (CaCO₃) from Malaysia.



Fig. S5 TG–DTA curves of 3D hierarchical CaCO₃ hollow microspherical adsorbent composed of 1D spikeshaped nanorods

Adsorbents	BET (m ² /g)	Crystallite sizes (nm)
CaCO ₃ (2 M NaOH)	44.85	76.75
Ca(OH) ₂ (6 M NaOH)	19.49	44.69
CaC ₂ O ₄ .H ₂ O (10 M NaOH)	7.87	37.12

Table S1 BET surface areas and crystallite sizes of synthesized adsorbents under different NaOH concentrations.



Fig. S6 FTIR spectra of Ca(OH)₂ and CaC₂O₄.H₂O adsorbents synthesized with (a) 6 M and (b) 10 M NaOH.



Fig. S7 EDX spectra of Ca(OH)₂ and (b) CaC₂O₄.H₂O adsorbents synthesized with (a) 6 M and (b) 10 M NaOH.



Fig. S8 TG-DTA curves of Ca(OH)₂ and CaC₂O₄.H₂O adsorbents synthesized with 6 and 10 M NaOH.

	TG Weight loss (%)		
Adsorbents	Step I (100–190 °C)	Step II (370–510 °C)	Step III (560–830°C)
Ca(OH) ₂ (6 M NaOH)	2.1	15.3	17.7
CaC ₂ O ₄ .H ₂ O (10 M NaOH)	10.5	16.3	30.0

Table S2 TG data of $Ca(OH)_2$ and CaC_2O_4 . H_2O adsorbents.



Fig. S9 Multi cycles carbonation conversion curves of 3D hierarchical CaCO₃ hollow microspherical adsorbent composed of 1D spike-shaped nanorods



Fig. S10 FESEM images of the calcined 3D hierarchical CaCO₃ hollow microspherical adsorbent composed of 1D spike-shaped nanorods (a) after 1cycle (b) after 29 cycles.



Fig. S11 FESEM images of the calcined adsorbents after 15 cycles (a) $Ca(OH)_2$, (b) CaC_2O_4 .H₂O and (c) limestone.