

Supplementary Information

Boosting the value-added aromatics synthesis directly from syngas via Cr_2O_3 and Ga doped capsule zeolite

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Experimental Section

(1) Catalyst Preparation

Synthesis of High-Temperature Methanol Synthesis Catalyst Cr₂O₃. Cr₂O₃ was prepared by a precipitation method with (NH₄)₂CO₃ as a precipitation agent. Typically, Cr(NO₃)₃·9H₂O (1 M) and (NH₄)₂CO₃ (1 M) aqueous solutions were simultaneously dropped into an empty beaker at 70 °C with the help of magnetic stirring. The pH of the suspension liquid was controlled at 7.0~8.0 by adjusting the injection speed of the (NH₄)₂CO₃ aqueous solution. After static aging at 70 °C for 3 h, the precipitate was collected by filtration and washed with distilled water 3 times. The product was dried overnight at 120 °C in an oven and calcined at 500 °C in air for 3 h (heating rate of 5 °C/min) in a muffle furnace to obtain the green Cr₂O₃ catalyst.

Synthesis of H/M-ZSM-5 and H/M-ZSM-5@SiO₂ (M = Zn, Ga, La, and Mg). The parent H-ZSM-5 zeolite was prepared by a hydrothermal method, during which tetraethyl orthosilicate (TEOS), Al(NO₃)₃·9H₂O, and tetrapropylammonium hydroxide (TPAOH) were employed as Si sources, Al sources, and organic template, respectively. Typically, TEOS, Al(NO₃)₃·9H₂O, 25% TPAOH, and ethanol were dissolved into distilled water with a molar ratio of SiO₂ : Al₂O₃ : H₂O : TPAOH : EtOH = 1 : 0.0125 : 50 : 0.24 : 4. After magnetic stirring for 4 h, the clear solution was transferred into a Teflon-sealed autoclave and maintained at 180 °C for 24 h. The white product was washed with distilled water, dried at 120 °C overnight, and calcined at 550 °C in air for 5 h to remove the organic template in the microporous of the zeolite.

M-ZSM-5 zeolites were prepared by an ion-exchange method. Typically, H-ZSM-5 of 2 g was immersed into an aqueous solution of metallic nitrate (100 mL, 0.1 M). After magnetic stirring for 15 h at 80 °C, the resultant white product was collected by centrifugation, washed by distilled water 3 times, and calcined at 500 °C in an air atmosphere to obtain the ion-exchanged zeolite M-ZSM-5.

Capsule zeolites were prepared by the chemical liquid deposition method. The bare zeolite (H-ZSM-5 or M-ZSM-5) of 1 g was immersed into n-hexane with the desired amount of tetramethoxysilane (TMOS). After magnetic stirring for 4 h, the sample was dried at 80 °C and calcined at 550 °C for 3 h. The amount of SiO₂ in H/M-ZSM-5@SiO₂ was controlled by varying the dosage of TMOS and the deposition times.

The Fabrication of Bifunctional Catalysts. The bifunctional catalysts were fabricated by physically mixing. Cr₂O₃ and capsule zeolite with the mass ratio of 1 : 1 were mixed in an agate mortar for 10 min,

and then pressed, crushed, and sieved to 20-40 meshes.

(2) Catalyst Characterization

The XRD patterns of the catalysts were recorded by a Rigaku RINT 2400 X-ray diffractometer with Cu K α radiation. The morphology of the obtained catalysts was observed by a scanning electron microscopy (SEM, JSM-6700F, JEOL) and a transmission electron microscopy (TEM, JEM-2100UHR, JEOL). Scanning transformation electron microscopy (STEM) was employed to observe the elemental distribution in the zeolites. The chemical state of Ga species in the zeolites was characterized by the X-ray photoelectron spectroscopy (XPS, Thermo Fischer Scientific ESCALAB 250Xi). The amount of Ga in the zeolite component was determined by inductive coupled plasma-atomic emission spectroscopy (ICP-AES). The specific surface area and pore size distribution of the samples were measured by a Micromeritics 3Flex 2MP instrument.

NH₃/CO/CO₂ temperature-programmed desorption (NH₃/CO/CO₂-TPD) profiles were performed on a MicrotracBEL BELCATII-T-SP instrument. For NH₃/CO-TPD, the catalyst was pretreated at 150 °C for 1 h to remove the H₂O in the microporous of the zeolite. Then the sample was saturated by NH₃ or CO at 50 °C. After removing the physisorbed NH₃ or CO by He flow, the NH₃/CO-TPD profiles were recorded under He flow with a heating rate of 10 °C min⁻¹. For CO₂-TPD, the catalyst was first pretreated at 400 °C for 2 h in a flow of pure H₂. After cooling down to 50 °C, the catalyst was saturated with pure CO₂ for 1 h, and then flushed with He flow to remove the physisorbed molecules. Afterwards, the TPD experiment was started with a heating rate of 10 °C min⁻¹ under He flow.

Pyridine and 2,6-di-tert-butylpyridine adsorbed Fourier transform infrared (FTIR) spectra (Pyridine/DTBPy-FTIR) were recorded by a Bruker Tensor 27 FTIR spectrometer. The zeolite sample was first pressed into a self-supported wafer. After degassing at 400 °C and 10⁻⁴ Pa for 1 h, the catalyst wafer was exposed in the pyridine or DTBPy vapor for probing molecule adsorption (150 °C, 30 min). Then the FTIR spectrum was recorded after evacuation at 150 °C for 30 min. The in-situ diffuse reflectance infrared Fourier transform (DRIFT) spectrum of methanol to aromatics process under CO atmosphere was collected using a FTIR spectrometer (Thermo Nicolet NEXUS 470 FT-IR). Before the measurement, Ga-ZSM-5@SiO₂ catalyst was pretreated at 400 °C for 1 h under a N₂ atmosphere. After the temperature cooling down to 380 °C under N₂ flow, CO was fed into the chamber through a container with methanol in it. The spectrum was recorded by collecting 64 scans at 4 cm⁻¹ resolution with the catalyst under 380 °C and 2 MPa.

(3) Computational methods

All of spin-polarized calculations based on density functional theory (DFT) were performed by utilizing DMol³ package.^[1] The generalized gradient approximation (GGA) in the Perdew-Burke-Ernzerhof form and Semicore Pseudopotential method (DSPP) with the double numerical basis sets plus the polarization functional (DNP) were adopted.^[2,3] A (3 × 3 × 1) supercell were used. The Brillouin zones were sampled by using Monkhorst-Pack scheme with a 3 × 3 × 3 k-point grid and the double numerical basis sets plus the polarization functional (DNP) were adopted. A DFT-D correction with Grimme scheme was used to account for the dispersion interaction.^[4] The SCF convergence for each electronic energy was set as 1.0 × 10⁻⁵ Ha, and the geometry optimization convergence criteria were set up as follows: 1.0 × 10⁻⁵ Ha for energy, 0.004 Ha Å⁻¹ for force, and 0.01 Å for displacement, respectively. Energy barriers were examined by linear and quadratic synchronous transit methods in combination with the conjugated gradient (CG) refinement.

(4) Catalytic performance evaluation

The tandem reaction was performed in a fixed-bed reactor with an internal diameter of 8 mm. Before the reaction, the catalyst was reduced by pure H₂ at 400 °C for 2 h. After cooling down to room temperature, the reactant gas mixture (gas composition as 3.0% Ar, 29.6% CO, 4.87% CO₂, H₂ balance) was fed into the reactor. The reaction pressure and temperature were 3.0 MPa and 380 °C, respectively. The reaction effluents were quantitatively analyzed online with two gas chromatographs (GC-8A, Shimadzu), one equipped with a thermal conductive detector (TCD) by an active charcoal column for Ar, CO, CH₄, and CO₂ analysis; while another with a flame ionization detector (FID) by a HP-PLOT/Q column for hydrocarbons analysis. An off-line gas chromatograph (GC-2010, Shimadzu) equipped with an FID and two separated capillary columns (DB-1 and DB-WAX) was employed to further identify the xylene isomers in the collected liquid products. In order to suppress the possible condensation during the reaction process, the pipeline from the catalyst bed to the gas chromatographs was heated at 200 °C. For the methanol to aromatics (MTA) reaction on zeolite, the liquid methanol (0.005 mL min⁻¹) was fed into the reactor by a TOSOH DP 8020 pump with the help of CO gas flow (15 mL min⁻¹). The MTA process was performed under 380 °C and 2 MPa. The CO conversion and hydrocarbons distribution were calculated according to the following equations:

$$CO\ Conversion = \frac{CO_{inlet} - CO_{outlet}}{CO_{inlet}} \times 100\%$$

Where CO_{inlet} and CO_{outlet} represent moles of CO at the inlet and outlet, respectively.

$$Sel_{C_nH_m} = \frac{nC_nH_m \text{ outlet}}{\sum_1^n nC_nH_m \text{ outlet}} \times 100\%$$

The presence of CO_2 (4.87%) in the feed gas could suppress the water gas shift reaction ($CO + H_2O \rightarrow CO_2 + H_2$) and CO_2 conversion was calculated according to:

$$CO_2 \text{ conversion} = \frac{CO_{2 \text{ inlet}} - CO_{2 \text{ outlet}}}{CO_{2 \text{ inlet}}} \times 100\%$$

Where $CO_{2 \text{ inlet}}$ and $CO_{2 \text{ outlet}}$ represent moles of CO_2 at the inlet and outlet, respectively.

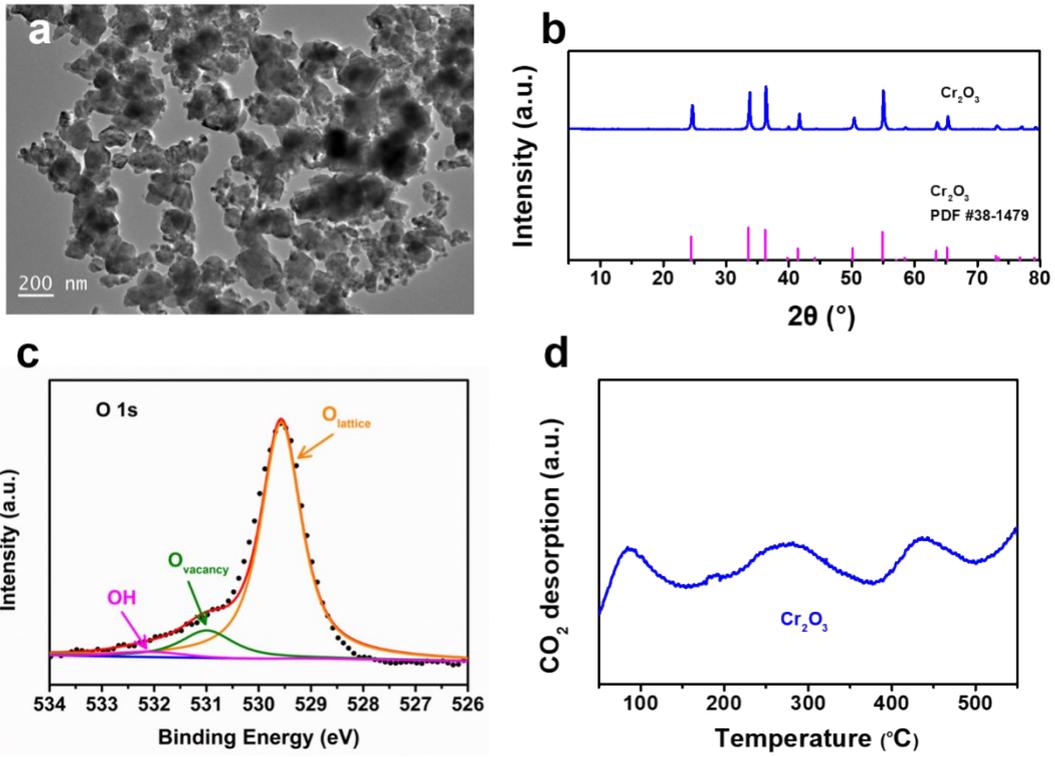


Figure S1. TEM image (a), XRD pattern (b), XPS (c), and CO₂-TPD (d) profile of Cr_2O_3 .

Table S1. The effect of SiO₂ membrane thickness on the products distribution of tandem catalysis of syngas.

Catalyst	CO Conv. (%)	CO ₂ Conv. (%)	Hydrocarbon distribution (%)					
			CH ₄	C ₂₋₄ ⁰	C ₂₋₄ ⁼	CH ₃ OH+DME	C ₅₊	Aromatics
Cr ₂ O ₃ /H-ZSM-5@SiO ₂ -13.8%	20.6	13.0	10.7	16.0	3.7	0	1.6	68.0
Cr ₂ O ₃ /H-ZSM-5@SiO ₂ -39.0%	20.8	13.1	4.1	19.3	5.1	0.1	2.1	69.3
Cr ₂ O ₃ /H-ZSM-5@SiO ₂ -56.1%	18.7	12.5	5.2	17.2	5.3	0.1	4.0	68.2

^aReaction conditions: 380 °C, 3 MPa, (Ar 3%, CO 29.6%, CO₂ 4.87%, H₂ balance), GHSV = 1200 mL g_{cat}⁻¹ h⁻¹, time on stream (TOS) = 8 h, metal oxide/zeolite mass ratio = 1, 0.5 g catalyst.

Products distribution in aromatics.

Catalyst	Selectivity of aromatics (%) ^a								STY of p-X (g _c h ⁻¹ kg _{cat} ⁻¹)	STY of A (g _c h ⁻¹ kg _{cat} ⁻¹)
	B	T	E	<i>m</i> -X	<i>o</i> -X	<i>p</i> -X	A(C ₉)	A(C ₁₀₊)		
Cr ₂ O ₃ /H-ZSM-5@SiO ₂ -13.8%	0.7	4.3	0.8	0.5	6.1	22.9	25.7	7.0	9.9	29.4
Cr ₂ O ₃ /H-ZSM-5@SiO ₂ -39.0%	0.7	4.5	0.7	0.6	5.8	33.0	20.0	4.0	14.4	30.3
Cr ₂ O ₃ /H-ZSM-5@SiO ₂ -56.1%	0.6	4.7	0.9	0.4	6.0	31.5	19.8	4.3	12.4	26.9

^aThe selectivity of B, T, *o*-X, *m*-X, *p*-X, A(C₉), and A(C₁₀₊) in all of the products, in which B = Benzene, T = Toluene, *o*-X = *ortho*-xylene, *m*-X = *meta*-xylene, and *p*-X = *para*-xylene.

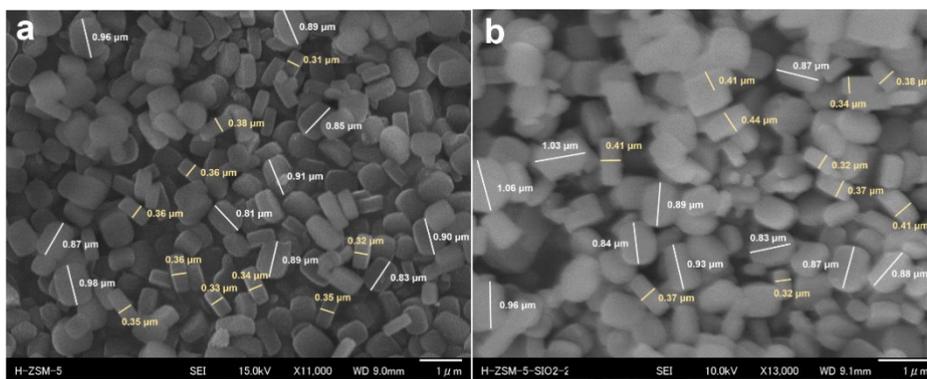


Figure S2. SEM images of H-ZSM-5 (a) and H-ZSM-5@SiO₂-39.0% (b).

Table S2. Texture properties of the parent H-ZSM-5 and some modified zeolites.

Catalyst	S_{BET} ($\text{m}^2 \text{g}^{-1}$) ^a	V ($\text{cm}^3 \text{g}^{-1}$) ^b	D (nm) ^c
H-ZSM-5	385.4	0.27	2.8
Ga-ZSM-5	350.3	0.25	2.9
H-ZSM-5@SiO ₂ -13.8%	347.3	0.24	2.7
H-ZSM-5@SiO ₂ -39.0%	322.7	0.22	2.7
H-ZSM-5@SiO ₂ -56.1%	304.1	0.19	2.5
Ga-ZSM-5@SiO ₂ ^d	306.7	0.20	2.6

^aSpecific surface area calculate by BET formula.

^bPore volume calculated by BJH formula.

^cAverage pore diameter calculated by BJH formula.

^dSiO₂ deposition amount of 39.0% was employed in this capsule zeolite.

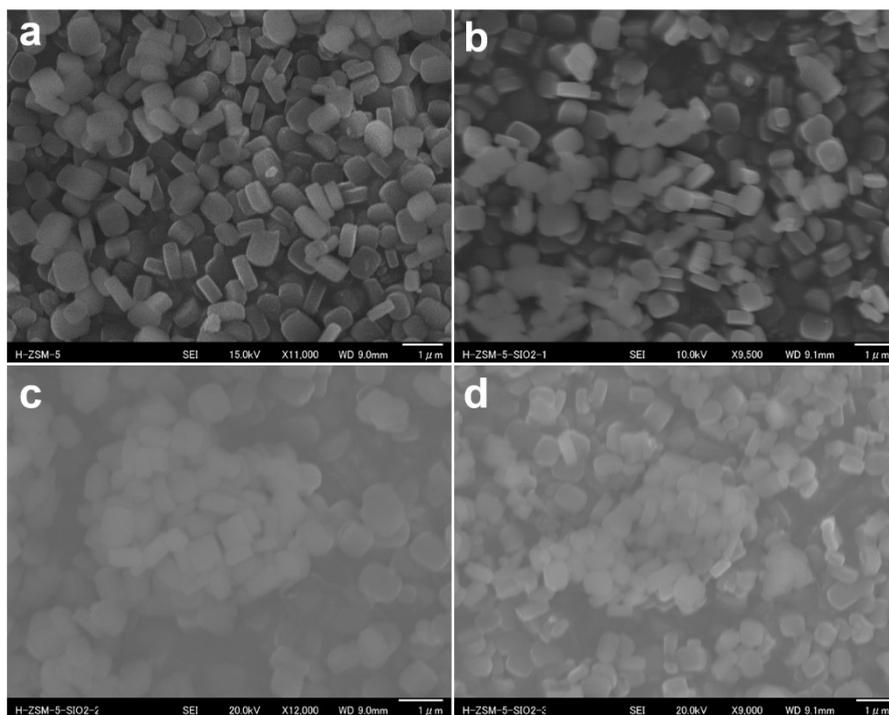


Figure S3. SEM images of H-ZSM-5 (a), H-ZSM-5@SiO₂-13.8% (b), H-ZSM-5@SiO₂-39.0% (c), and H-ZSM-5@SiO₂-56.1% (d).

Table S3. Catalytic performances of Cr₂O₃/M-ZSM-5@SiO₂ (M = H, Zn, Ga, La, Mg) catalysts for syngas direct conversion into aromatics.^a

Catalyst	CO Conv. (%)	CO ₂ Conv. (%)	Hydrocarbon distribution (%)					
			CH ₄	C ₂₋₄ ⁰	C ₂₋₄ ^F	CH ₃ OH+DME	C ₅₊	Aromatics
Cr ₂ O ₃ /H-ZSM-5@SiO ₂	20.8	13.1	4.1	19.3	5.1	0.1	2.1	69.3
Cr ₂ O ₃ /Zn-ZSM-5@SiO ₂	25.8	4.4	6.5	17.2	2.9	0	2.0	71.4
Cr ₂ O ₃ /Ga-ZSM-5@SiO ₂	28.4	1.5	4.4	14.6	2.3	0	2.3	76.4
Cr ₂ O ₃ /La-ZSM-5@SiO ₂	18.5	11.3	5.7	16.5	3.8	0.1	1.7	72.2
Cr ₂ O ₃ /Mg-ZSM-5@SiO ₂	18.3	11.7	11.6	18.3	3.5	0	1.7	64.9

^aReaction conditions: 380 °C, 3 MPa, (Ar 3%, CO 29.6%, CO₂ 4.87%, H₂ balance), GHSV = 1200 mL g_{cat}⁻¹ h⁻¹, time on stream (TOS) = 8 h, metal oxide/zeolite mass ratio = 1, 0.5 g catalyst.

Products distribution in aromatics.

Catalyst	Selectivity of aromatics (%) ^a								STY of p-X (g _C h ⁻¹ kg _{cat} ⁻¹)	STY of A (g _C h ⁻¹ kg _{cat} ⁻¹)
	B	T	E	m-X	o-X	p-X	A(C ₉)	A(C ₁₀₊)		
Cr ₂ O ₃ /H-ZSM-5@SiO ₂	0.7	4.5	0.7	0.6	5.8	33.0	20.0	4.0	14.4	30.3
Cr ₂ O ₃ /Zn-ZSM-5@SiO ₂	0.7	5.5	0.9	0.7	6.7	35.8	17.0	4.1	18.1	36.0
Cr ₂ O ₃ /Ga-ZSM-5@SiO ₂	0.9	6.5	0.9	0.8	7.3	38.6	17.8	3.6	21.0	41.6
Cr ₂ O ₃ /La-ZSM-5@SiO ₂	0.8	5.3	0.7	0.7	6.8	35.2	19.0	3.7	13.7	28.0
Cr ₂ O ₃ /Mg-ZSM-5@SiO ₂	0.9	5.8	0.7	0.7	5.3	33.1	15.6	2.8	12.7	25.0

^aThe selectivity of B, T, o-X, m-X, p-X, A(C₉), and A(C₁₀₊) in all of the products, in which B = Benzene, T = Toluene, o-X = *ortho*-xylene, m-X = *meta*-xylene, and p-X = *para*-xylene.

Table S4. Comparison of the space time yield (STY) of aromatics obtained in this work with other literature.

Catalyst	T (°C)	P (MPa)	CO conv. (%)	CO ₂ Sel. (%)	Aromatics Sel. (%) in total HCs	GHSV (ml g _{cat} ⁻¹ h ⁻¹)	STY of A (g _C kg _{cat} ⁻¹ h ⁻¹)	Reference
CZS/0.5%Cu-Z5-Si2	450	3.6	40.8	40.9	72.1	600	27.9	[5]
ZnCr ₂ O ₄ /H-ZSM-5	350	2.0	37.0	40.0~50.0	83.3	300	11.9~14.3	[6]
MnO/H-ZSM-5	350	2.0	21.0	44.0	83.4	333	5.6	[7]
ZnCrO _x -ZSM-5-2.8	350	4.0	18.3	49.0	69.0	1500	24.6	[8]
ZnO-ZrO ₂ /H-ZSM-5 ^a	380	3.0	11.0	0	73.0	3000	31.0	[9]
Zn-ZrO ₂ /H-ZSM-5	430	3.0	21.0	41.0	73.0	1500	23.3	[10]
Ce _{0.2} Zr _{0.8} O/H-ZSM-5	380	2.0	5.1	18.7	83.2	3500	31.0	[11]
Mo-ZrO ₂ /H-ZSM-5	400	4.0	22.0	41.0	76.0	3000	50.7	[12]
ZnCrO _x -ZSM-5-533	350	4.0	16.0	46.9	73.9	1500	24.0	[13]
Cr ₂ O ₃ /Ga-ZSM-5@SiO ₂	380	3.0	28.4	-1.5	76.4	1200	41.6	This work

^aCO₂/(CO+CO₂) = 0.25.

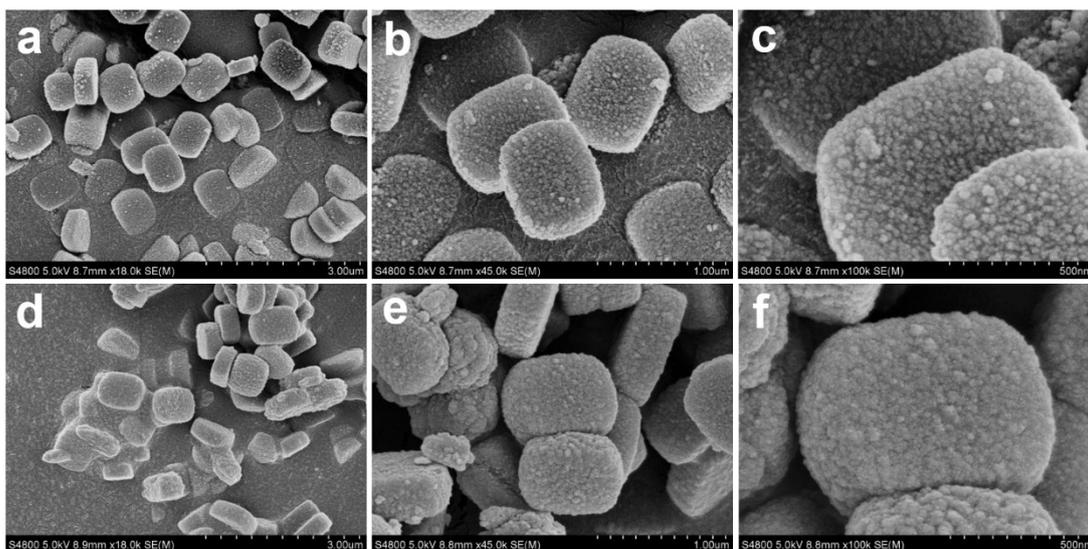


Figure S4. SEM images of Ga-ZSM-5 (a-c) and Ga-ZSM-5@SiO₂ (d-f).

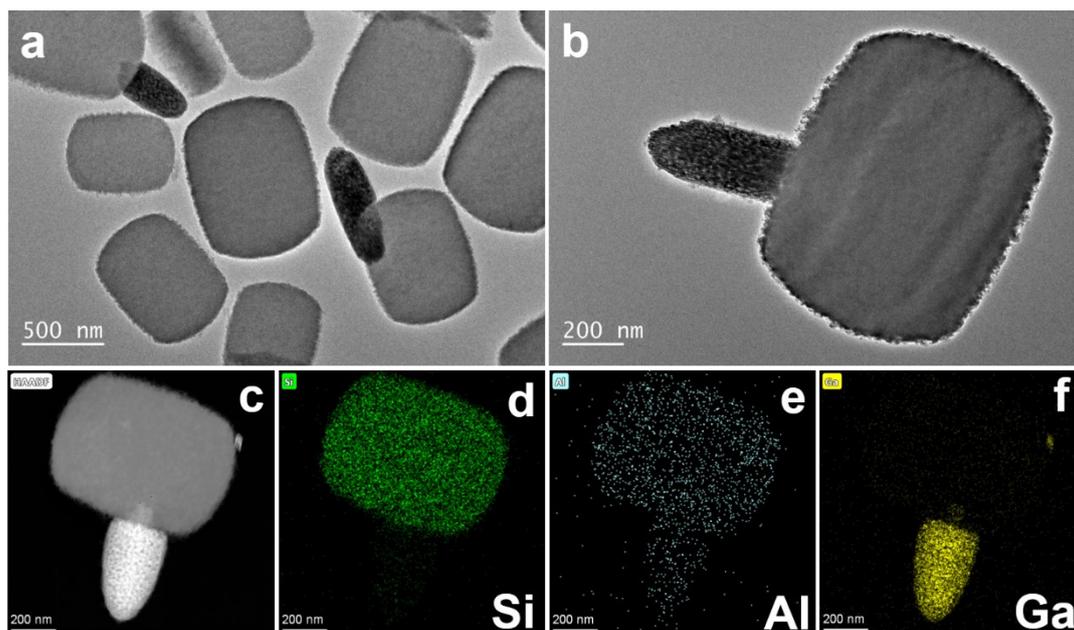


Figure S5. TEM images of Ga-ZSM-5 (a, b), STEM image of Ga-ZSM-5 (c) and the corresponding elemental mapping of Si, Al, and Ga (d-f).

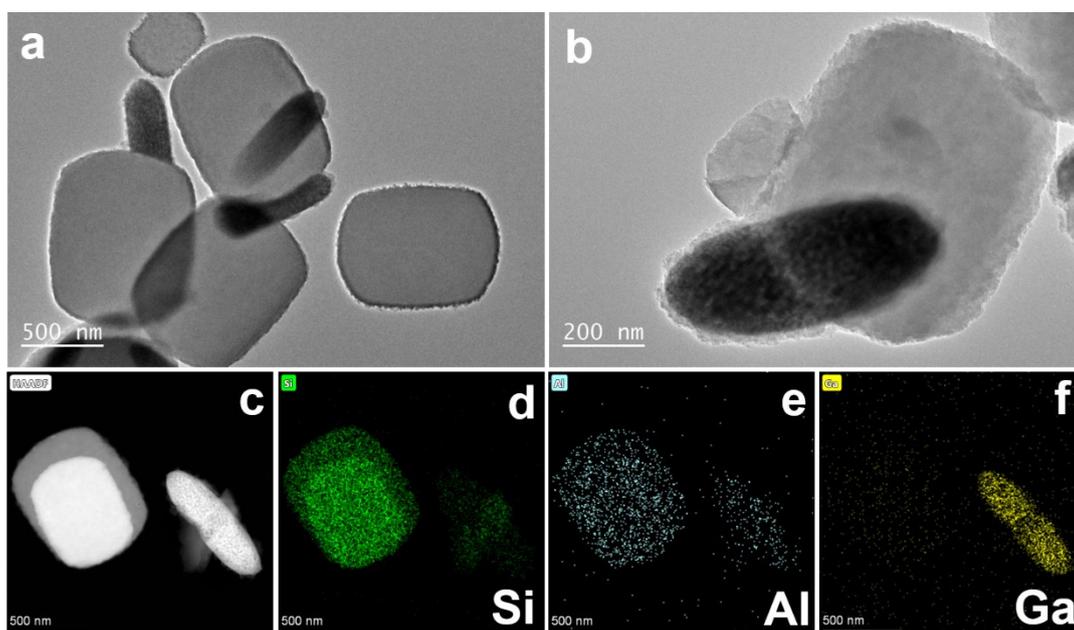


Figure S6. TEM images of Ga-ZSM-5@SiO₂ (a, b), STEM image of Ga-ZSM-5@SiO₂ (c) and the corresponding elemental mapping of Si, Al, and Ga (d-f).

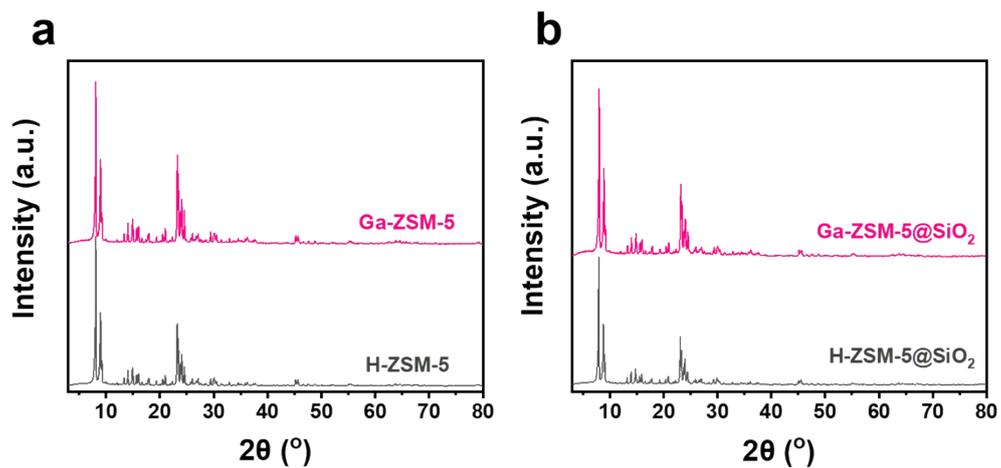


Figure S7. XRD patterns of the bare zeolites (a) and the capsule zeolites (b).

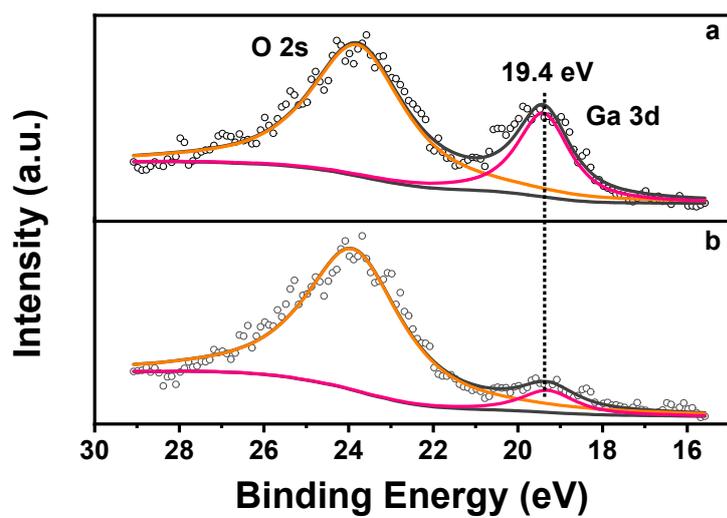


Figure S8. XPS results of Ga 3d spectra of Ga-ZSM-5 (a) and Ga-ZSM-5@SiO₂ (b).

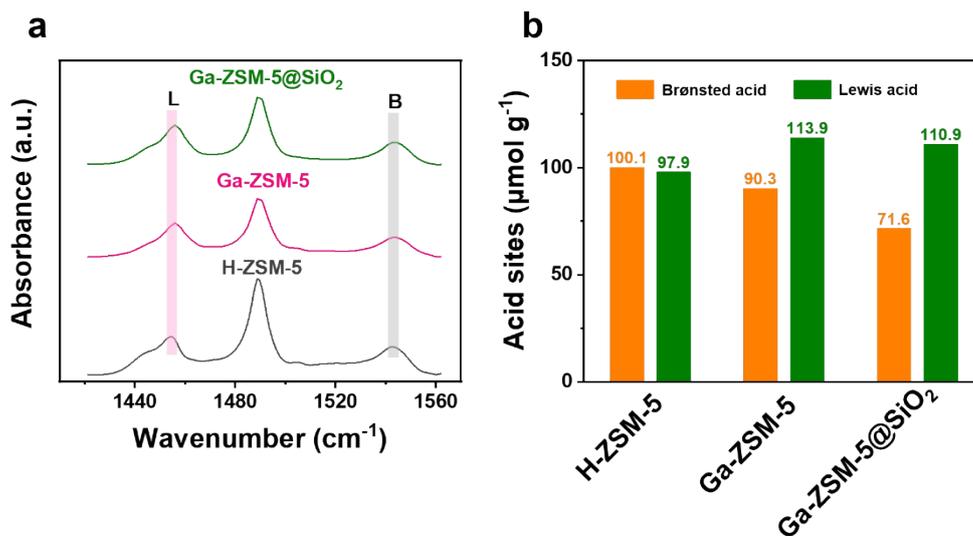


Figure S9. (a) Pyridine-FTIR spectra of H-ZSM-5, Ga-ZSM-5, and Ga-ZSM-5@SiO₂ recorded after evacuation at 150 °C. (b) Densities of B and L acid sites on H-ZSM-5, Ga-ZSM-5, and Ga-ZSM-5@SiO₂.

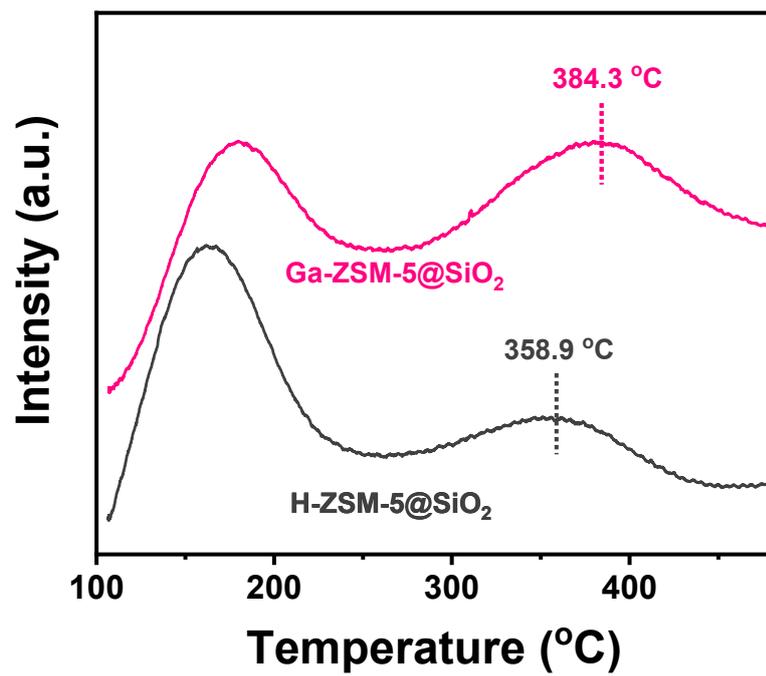


Figure S10. NH₃-TPD profiles of H-ZSM-5 and Ga-ZSM-5.

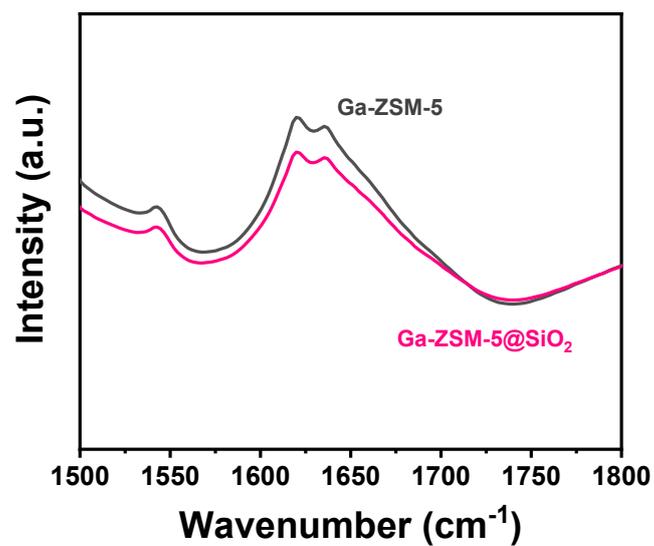


Figure S11. DTBPy-FTIR spectra of Ga-ZSM-5 and Ga-ZSM-5@SiO₂.

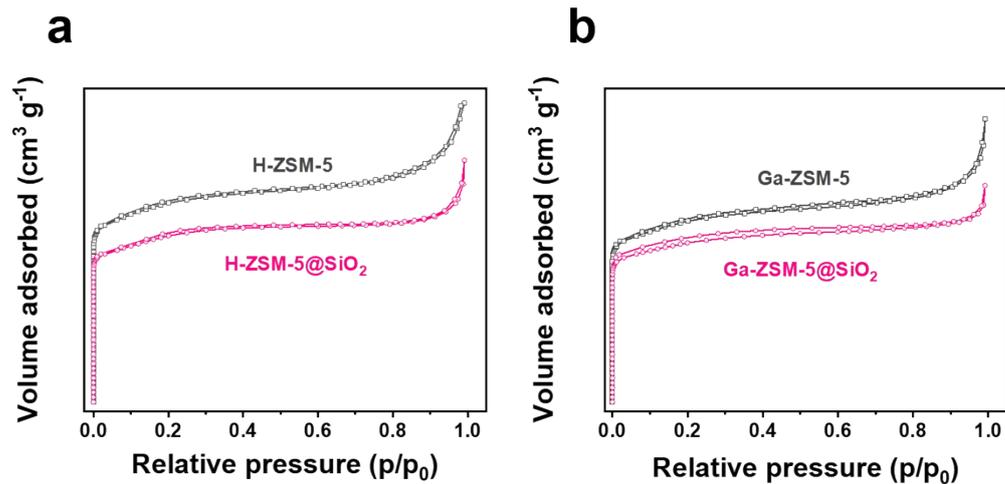


Figure S12. N₂ adsorption-desorption isotherms of H/Ga-ZSM-5 and H/Ga-ZSM-5@SiO₂.

Table S5. Catalytic performances of Cr₂O₃/H-ZSM-5@SiO₂, Cr₂O₃/Ga₂O₃/H-ZSM-5@SiO₂, and Cr₂O₃/Ga-ZSM-5@SiO₂.^a

Catalyst	CO Conv. (%)	CO ₂ Conv. (%)	Hydrocarbon distribution (%)						STY of A (g _C kg _{cat} ⁻¹ h ⁻¹)
			CH ₄	C ₂₋₄ ⁰	C ₂₋₄ ⁼	CH ₃ OH +DME	C ₅₊	Aromatics	
Cr ₂ O ₃ /H-ZSM-5@SiO ₂	20.8	13.1	4.1	19.3	5.1	0.1	2.1	69.3	30.3
Cr ₂ O ₃ /Ga ₂ O ₃ /H-ZSM-5@SiO ₂	16.2	6.5	3.9	15.6	4.5	0	3.5	72.5	23.7
Cr ₂ O ₃ /Ga-ZSM-5@SiO ₂	28.4	1.5	4.4	14.6	2.3	0	2.3	76.4	41.6

^aReaction conditions: 380 °C, 3 MPa, (Ar 3%, CO 29.6%, CO₂ 4.87%, H₂ balance), GHSV = 1200 mL g_{cat}⁻¹ h⁻¹, time on stream (TOS) = 8 h, metal oxide/zeolite mass ratio = 1, 0.5 g catalyst. Cr₂O₃/Ga₂O₃/H-ZSM-5@SiO₂ was prepared by physically mixing of Cr₂O₃ (0.25 g), H-ZSM-5@SiO₂ (0.25 g), and Ga₂O₃ (0.0125 g).

Table S6. CO conversion and products distribution of methanol conversion under CO-rich atmosphere via M-ZSM-5@SiO₂ (M = H, Zn, Ga, La, Mg) catalysts.^a

Catalyst	CO Conv. (%)	Hydrocarbon distribution (%)				
		CH ₄	C ₂₋₄ ⁰	C ₂₋₄ ⁼	C ₅₊	Aromatics (<i>p</i> -X)
H-ZSM-5@SiO ₂	-1.8	0.9	6.4	0	29.9	62.8 (28.4)
Zn-ZSM-5@SiO ₂	0.2	0.2	6.2	0.2	18.9	74.5 (37.0)
Ga-ZSM-5@SiO ₂	2.1	0	7.2	0.2	11.3	81.3 (32.8)
La-ZSM-5@SiO ₂	-2.8	0	8.0	0.2	17.4	74.4 (28.7)
Mg-ZSM-5@SiO ₂	-3.5	0.3	5.6	0.2	17.7	76.2 (34.6)

^aReaction conditions: 380 °C, 2 MPa, liquid methanol flow rate 0.005 mL min⁻¹, gas flow (CO 90%, Ar balance) rate 15 mL min⁻¹, TOS = 8 h, 1.0 g zeolite catalyst.

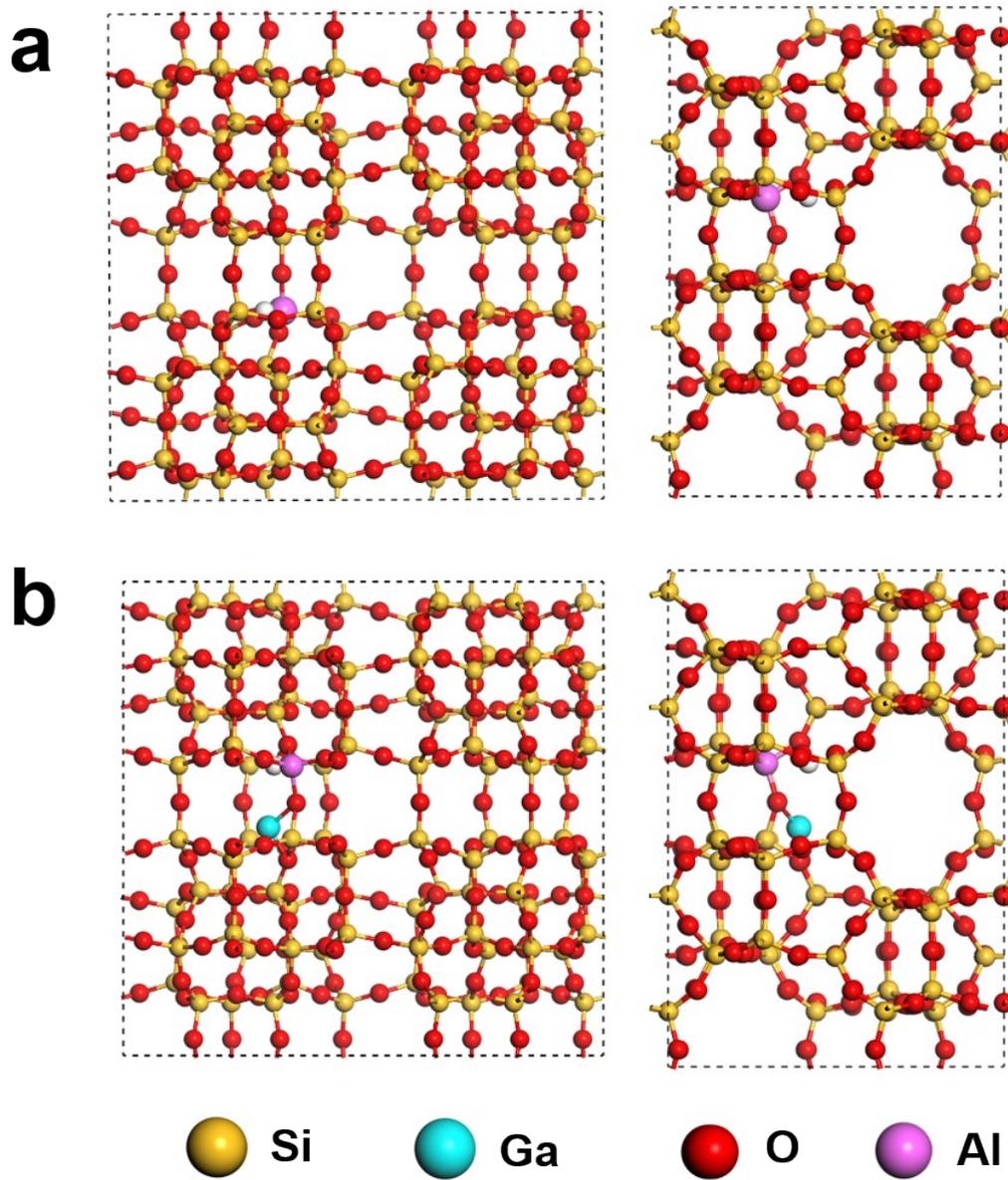


Figure S13. The modules of H-ZSM-5 (a) and Ga-ZSM-5 (b).

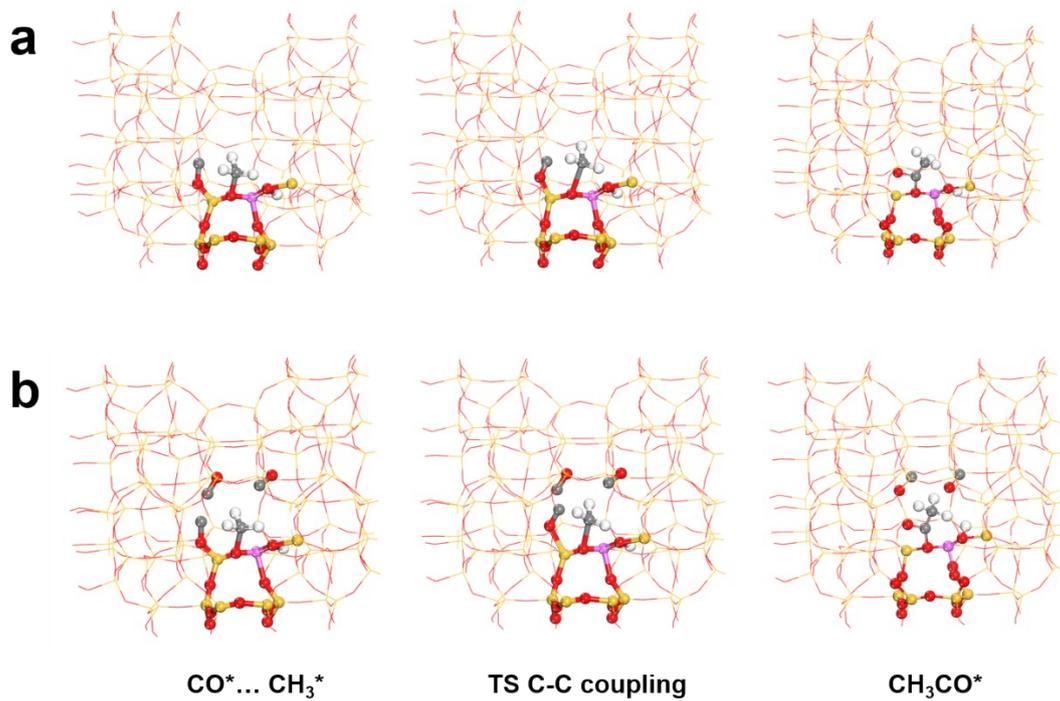


Figure S14. The reaction schemes of the first C-C bond formation on H-ZSM-5 under CO-free (a) or CO-rich (b) atmosphere (yellow: Si, red: O, pink: Al, white: H, grey: C).

Reference

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