

Supporting Information

Reaction of Methane with MO_x/CeO₂ (M= Fe, Ni, and Cu) Catalysts: In Situ Studies with Time-Resolved X-ray Diffraction

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Table S1. Linear combination fitting (LCF) of XANES spectrum for FeO_x/CeO₂ sample.

	Fe ₂ O ₃ (%)	Fe ₃ O ₄ (%)	R-factor	Chi-square
Fitting results	85.3+/-9.3	14.7+/-9.3	0.0038	0.0447

Table S2. EXAFS fitting of MO_x/CeO₂ (M = Fe, Co, Ni, and Cu) catalysts.

Samples	Shells	Bond length	Coordination number	σ^2 (Å ²)	E0 shift (eV)	S ₀ ²
CuO _x /CeO ₂	Cu-O	1.95+/-0.01	3.5+/-1.2	0.004	1.0	0.85
	Cu-Cu	2.84+/-0.01	0.4+/-0.2	0.005		
NiO _x /CeO ₂	Ni-O	2.08+/-0.01	4.2+/-1.0	0.003	-0.4	0.76
	Ni-Ni	2.98+/-0.01	9.5+/-2.0	0.006		
CoO _x /CeO ₂	Co-O	1.91+/-0.01	5.9+/-0.6	0.003	0.7	0.64
	Co-Co	2.86+/-0.01	5.7+/-0.9	0.005		
	Co-Co	3.37+/-0.01	8.0+/-1.6	0.006		
FeO _x /CeO ₂	Fe-O	1.99+/-0.02	4.2+/-0.8	0.010	0.5	0.71
	Fe-Fe	3.04+/-0.02	4.7+/-2.2	0.019		
	Fe-Fe	3.34+/-0.02	4.3+/-2.4	0.019		

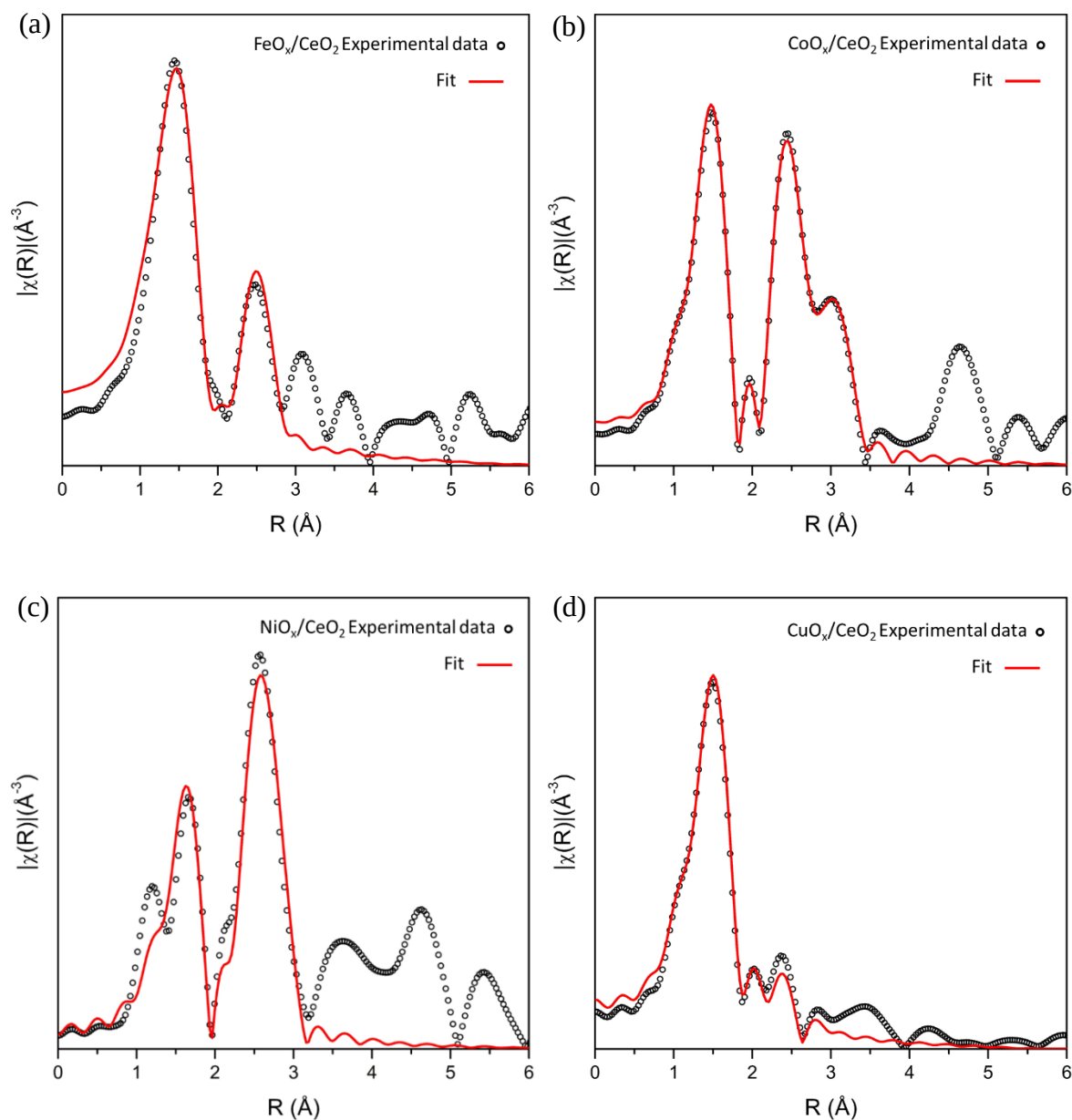


Figure S1. EXAFS fittings of the MO_x/CeO_2 ($\text{M} = \text{Fe}, \text{Co}, \text{Ni}, \text{and Cu}$) catalysts from (a) Fe K-edge, (b) Co K-edge, (c) Ni K-edge, (d) Cu K-edge. Red solid lines represent for the calculated fits for (a) Fe_2O_3 , (b) Co_3O_4 , (c) NiO , (d) CuO while circles in figure are from the experimental results.

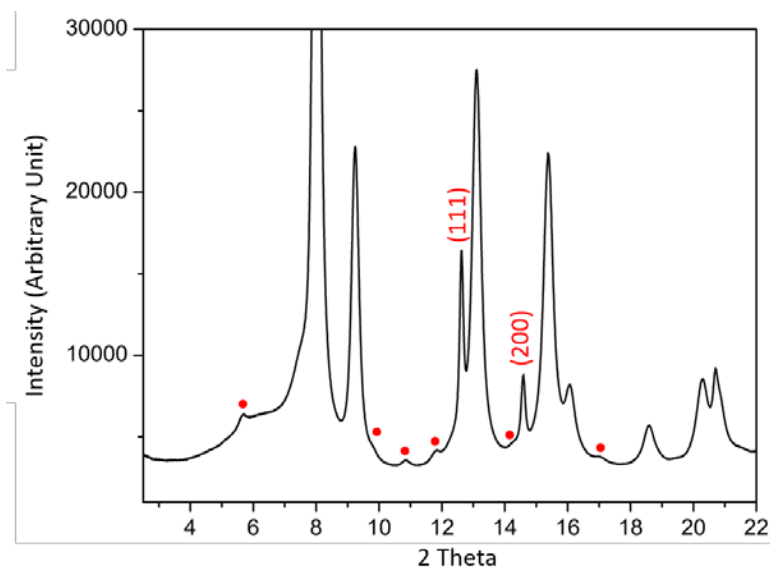


Figure S2. XRD pattern of 10 wt% NiO_x/CeO₂ catalysts at 700 °C during CH₄-TPR reaction. Characteristic peaks of the reduced metallic Ni phase are labelled in red and diffraction peaks marked with the red dots come from the Ce₂O₃ (Ia-3) phase.

Table S3. ICP-OES Results of Metal Phase Composition in the Prepared MO_x/CeO₂ Samples.

Metals	Fe	Co	Ni	Cu
Intended wt%	10	10	10	10
Measured wt%	9.94+/-0.03	10.7+/-0.02	9.69+/-0.01	9.58+/-0.01