

Rational design of the La-doped CuCoAl Hydrotalcite catalyst for selective hydrogenation of furfuryl alcohol to 1,5-Pentanediol

Jingjing Tan,^{a*} Hailong Huang,^{a,b} Yuanna Zhang,^{a,b} Jinglei Cui,^{c*} Jing Zhang,^d Long Huang,^e Yongzhao Wang^a and Yulei Zhu^{f*}

^a Engineering Research Center of Ministry of Education for Fine Chemicals, Shanxi University, Taiyuan 030006, Shanxi Province, PR China

^b School of Chemistry and Chemical Engineering, Shanxi University, Taiyuan 030006, Shanxi Province, PR China

^c Institute of Resources and Environmental Engineering, Shanxi University, Taiyuan 030006, China;

^d Institute of Applied Chemistry, Shanxi University, Taiyuan, 030006 P. R. China

^e Beijing Key Laboratory of Fuels Cleaning and Advanced Catalytic Emission Reduction Technology, Beijing Institute of Petrochemical Technology, Beijing 102617, China

^f State Key Laboratory of Coal Conversion, Institute of Coal Chemistry, Chinese Academy of Sciences, Taiyuan 030001, PR China, Synfuels China Co. Ltd., Beijing, PR China.

*Corresponding authors:

E-mail addresses: tanjingjing@sxu.edu.cn, cuijl@sxu.edu.cn, zhuyulei@sxicc.ac.cn;

1 Experimental section

1.1 Materials

Cu(NO₃)₂·3H₂O, furfuryl alcohol (Analytical purity, Shanghai Sinopharm Chemical Reagent Co., Ltd.); Co(NO₃)₂·6H₂O, Al(NO₃)₃·9H₂O (Analytical purity, Shanghai Maclin Biochemical Technology Co., Ltd.); La(NO₃)₃·6H₂O (Analytical purity, Shanghai Aladdin Biochemical Technology Co., Ltd.); Ethanol (Analytical purity, China National Medicines Corporation Ltd.); NaOH, Na₂CO₃ (Analytical purity, Tianjin Damao Chemical Reagent Factory).

1.2 Catalyst preparation

1.2.1 Preparation of CuCoAl Hydrotalcite-Like Catalyst Precursors

The CuCoAl hydrotalcite-like catalyst precursors were prepared with a fixed molar ratio of metals (Cu:Co:Al=1:29:10) using a co-precipitation method. A certain mass of Cu(NO₃)₂·3H₂O, Co(NO₃)₂·6H₂O, and Al(NO₃)₃·9H₂O was taken according to the ratio of M²⁺/M³⁺=3:1. These salts were dissolved in water to prepare a 0.5 M mixed salt solution (Solution A). Simultaneously, a certain mass of NaOH and Na₂CO₃ was taken to prepare a mixed alkali solution as the precipitating agent, with a molar ratio of n(Na₂CO₃): n(NaOH)=3:10 (Solution B). Under stirring, Solutions A and B were continuously titrated using a peristaltic pump, maintaining the pH of the solution at approximately 10 throughout the titration process. After titration completion, stirring was continued for a certain period. Subsequently, the reaction mixture was aged, filtered, washed and dried to obtain the CuCoAl hydrotalcite-like precursor.

1.2.2 Preparation of La/CuCoAl-DP by deposition-precipitation method

The catalyst of La/CuCoAl-DP was prepared by the deposition-precipitation method. The content of La was 1.5%. Under stirring, a specified amount of La (NO₃)₃·6H₂O was dispersed and dissolved in 100 mL of deionized water. The prepared CuCoAl hydrotalcite-like precursor was then added and stirred for 10 minutes. Subsequently, 1 M sodium hydroxide solution was added dropwise until the pH of the solution reached 9, followed by stirring for another 30 minutes. The reaction mixture was then aged, filtered, washed and dried. Finally, the obtained catalyst precursor was calcined in air at 500°C for 3 hours to obtain the catalyst denoted as La/CuCoAl-DP.

1.2.3 Preparation of La/CuCoAl-IM by impregnation method

The catalyst of La/CuCoAl-IM was prepared by the incipient wetness impregnation method. The loading of La was 1.5%. A specified amount of lanthanum nitrate hexahydrate was dissolved and dispersed in a certain amount of deionized water. The prepared CuCoAl hydrotalcite-like precursor

was added and quickly stirred to ensure uniform dispersion of La sites in the catalyst. Subsequently, the mixture was aged and dried. Finally, the obtained catalyst precursor was calcined in air at 500°C for 3 hours to obtain the catalyst denoted as La/CuCoAl-IM.

1.2.4 Preparation of La/CuCoAl-CP by Co-precipitation Method

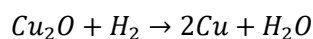
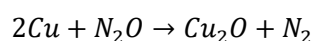
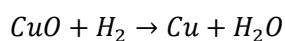
The catalyst of La/CuCoAl-CP was prepared using the co-precipitation method. The fixed molar ratio of metals (Cu:Co:Al:La=1:29:9.77:0.23) was used to prepare the CuCoAlLa hydrotalcite-like catalyst. According to the molar ratio of $M^{2+}/M^{3+}=3:1$, a certain mass of $\text{Cu}(\text{NO}_3)_2 \cdot 3\text{H}_2\text{O}$, $\text{Co}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$, $\text{Al}(\text{NO}_3)_3 \cdot 9\text{H}_2\text{O}$ and $\text{La}(\text{NO}_3)_3 \cdot 6\text{H}_2\text{O}$ was taken to prepare a 0.5 M mixed solution (Solution A). A mixed alkali solution (Solution B) with a ratio of $n(\text{Na}_2\text{CO}_3):n(\text{NaOH})=3:10$ was also prepared. Using a peristaltic pump, Solutions A and B were titrated in a stirred condition, maintaining the pH of the solution at approximately 10 throughout the titration process. After titration completion, stirring was continued for a certain period. The titrated solution was then aged, filtered, washed, and dried. Finally, the obtained catalyst precursor was calcined in air at 500°C for 3 hours to obtain the catalyst denoted as La/CuCoAl-CP.

1.3 Characterization techniques

X-ray diffraction (XRD) analysis was conducted on a Bruker D8 Advance diffractometer using Cu K α radiation to determine the catalyst's crystalline phases. Nitrogen physisorption was carried out on a Micromeritics ASAP 2020 physical adsorption analyzer to obtain the catalyst's textural properties. Carbon dioxide temperature-programmed desorption (CO_2 -TPD) was conducted on a BSD-Chem C200 chemisorption analyzer equipped with a TCD detector to qualitatively and quantitatively analyze the catalyst's surface basic sites. High-resolution transmission electron microscopy (HRTEM) was conducted on a JEOL JEM-2100 high-resolution transmission electron microscope to obtain microstructural information about the catalyst. Elemental composition and content in the catalyst were characterized using inductively coupled plasma atomic emission spectroscopy (ICP-OES) (Thermo-Fisher iCAP 7400). Hydrogen temperature-programmed reduction (H_2 -TPR) and desorption (H_2 -TPD, N_2O -TPD) were measured on an Auto Chem II 2920 chemisorption analyzer equipped with a thermal conductivity detector (TCD). Hydrogen consumption was calibrated using Ag_2O to characterize the active sites, hydrogen storage capacity, and interactions between active metals and supports. For H_2 -TPD: 100 mg of the catalyst was *in situ* reduced in a quartz tube with H_2/Ar at 400°C for 1 h. After cooling down to 50°C in Ar

atmosphere, the furnace temperature was then increased to 800°C at a rate of 5°C/min. N₂O titration tests were performed to ascertain the dispersion and surface area of Cu. 150 mg of catalyst was programmed to 270°C with 5°C/min under the atmosphere of H₂/Ar (10%, V/V) to obtain hydrogen consumption *X*. Then the temperature was cooled to 60°C, purging with He for 1h. Then N₂O/He was switched to (10%, V/V, 50mL/min) purge for 1h, and the Cu⁰ on the surface was oxidized to Cu₂O. After that, the gas was switched to He for purging for 30 minutes to remove excess N₂O. Finally, the gas was switched to H₂/Ar (10%, V/V) and programmed to 270°C for reduction (gas flow rate 50 mL/min, heating rate 5°C/min), then the hydrogen consumption *Y* was obtained.

The reactions that occur throughout the process are as follows: ^[1-3]



*D*_{Cu} calculation formula:

$$D_{Cu} = \frac{2Y}{X} \times 100\% \quad (1)$$

The metal dispersion, metal specific surface area and metal particle size can be calculated from the adsorption volume *V_a* measured by the chemical pulse adsorption method and some parameters.

Cu dispersion (*D*_{cu}, %):

Cu surface area (based on per gram of sample, m²/g_{cat.}):

$$S_{Cu} = \frac{2YN_{av}w_{Cu}}{XM_{Cu} \times 1.4 \times 10^{19}} \approx \frac{1353Yw_{Cu}}{X} \quad (2)$$

N_{av}: Avogadro constant, 6.02×10²³; *M_{Cu}*: Molar mass of metal Cu, 64g/mol; *w_{Cu}*: The loading amount of Cu in the catalyst (wt %), which can be measured by ICP-OES.

X-ray photoelectron spectroscopy (XPS) and X-ray Auger electron spectroscopy (XAES) were performed on an Escalab 250Xi X-ray photoelectron spectrometer using Al Kα radiation (1486.7 eV) with C1s binding energy (284.6 eV) as a reference. Before measurement, the catalyst was reduced *in-situ* at 400°C 1 atm H₂/N₂ mixture gas for 1 h (250°C, 6 h) in an XPS auxiliary pretreatment chamber, and then transferred to an XPS measurement chamber under high vacuum conditions without air exposure. The Cu element was subjected to LMM Auger electron analysis to determine the valence state of Cu.

1.4 Computational parameters

All the computations were implemented with the spin-polarized density functional theory (DFT)

method, as performed by the Vienna ab initio simulation package (VASP). The projector augmented wavefunction (PAW) pseudo-potentials are used to describe ionic potentials. The exchange correlation energy is sculptured by Perdew-Burke-Ernzerhof (PBE) functional within the generalized gradient approximation (GGA). In the course of the geometry relaxation, the energy cut-off was chosen as 500 eV. The electronic energy was considered self-consistent when the energy change was smaller than 10^{-5} eV. In structural optimizations, the Brillouin zone is sampled by $4 \times 4 \times 1$ mesh points in k-space based on the Monkhorst-Pack scheme for the calculated models. The vacuum gap is set as 15Å to preclude interplay between two adjacent surfaces. The force convergence criterion for atomic relaxation is set to be 0.02 eV/Å. The CuCo model used the CoO (110) surface as the substrate, with one Co atom on the CoO surface replaced by a Cu atom. The CuCoLa model was derived from the CuCo model by substituting one Co atom adjacent to the Cu atom on the CoO surface with a La atom. The H₂ decomposition was simulated using the climbing image nudged elastic band (CI-NEB) method, with a force tolerance of 0.05 eV/Å.

The adsorption energy (ΔE_{ads}) of adsorbed molecules on the substrate was defined as:

$$\Delta E_{ads} = E_{sur+mol} - E_{sur} - E_{mol} \quad (3)$$

Where $E_{sur+mol}$ represents the total energy of the molecule adsorbed structure. E_{sur} represents the total energy of the optimized bare surface, E_{mol} is the total energy of adsorbate in vacuum, respectively.

Table S1 The hydrogenation of FFA to 1,5-PeD over different catalysts ^a

Entry	Catalyst	Conv.%	Selectivity%			
			1,5-PeD	1,2-PeD	THFA	Others
1	La/CuCoAl-DP ^b	81.9	50.4	15.7	19.8	14.1
2	La/CuCoAl-DP ^c	83.6	47.4	12.7	27.3	12.6
3	CuCoAl	76.2	46.6	14.6	22.6	16.2
4	1.5%La(OH) ₃ +CuCoAl	86.9	51.4	15.2	18.7	14.8
5	3%La(OH) ₃ +CuCoAl	87.6	52.9	15.1	17.5	14.5
6	5%La(OH) ₃ +CuCoAl	75.6	47.8	12.6	22.5	17.1
7	La/CuCoAl-DP	97.6	54.2	12.7	13.0	20.1

Reaction conditions: FFA 0.5 g, catalyst 0.1 g, ethanol 39.5g; Temperature 160°C, reaction time 2 h, 4 MPa H₂; carbon balance in all reactions $\geq 97\%$. THFA: Tetrahydrofurfuryl alcohol; FFA: furfuryl alcohol; 1,2-PeD:1,2-Pentenediol; 1,5-PeD:1,5-Pentenediol; others: 1-Pentanol, 2-Pentanol, 1-Butanol, 2-Methylfuran, ^b: Temperature 140°C, ^c: La/CuCoAl-DP reduced at 400°C without calcination.

Table S2 The hydrogenation of THFA to 1,5-PeD over different catalysts

Entry	Catalyst	Conv.%	Selectivity%		
			1,5-PeD	1,2-PeD	Others
1	CuCoAl	1.5	0	0	100
2	La/CuCoAl-DP	9.5	46.8	0	53.2

Reaction conditions: THFA 0.5 g, catalyst 0.1 g, ethanol 39.5g; Temperature 160°C, reaction time 6 h, 4 MPa H₂. carbon balance in all reactions $\geq 97\%$. THFA: Tetrahydrofurfuryl alcohol; 1,2-PeD:1,2-Pentenediol; 1,5-PeD:1,5-Pentenediol, others: 2-Methyltetrahydrofuran

Table S3 Effect of water on the hydrogenation of FFA

Entry	Solvent/g	Conv.%	Selectivity%			
			1,5-PeD	1,2-PeD	THFA	Others
1	39.5g water	75.5	14.8	4.2	8.1	72.9 ^a
2	34.5g ethanol+5g water	84.3	53.0	9.0	22.7	15.3 ^b
3	39.5g ethanol	97.6	54.2	12.7	13.0	20.1 ^b

Reaction conditions: FFA 0.5 g, La/CuCoAl-DP catalyst 0.1 g, ethanol 39.5g; Temperature 160°C, reaction time 2 h, 4 MPa H₂; carbon balance in all reactions $\geq 97\%$. THFA: Tetrahydrofurfuryl alcohol; FFA: furfuryl alcohol; 1,2-PeD:1,2-Pentenediol; 1,5-PeD:1,5-Pentenediol; others: ^a: cyclopentanone (20.0%), cyclopentanone (52.9%); ^b: 1-Pentanol, 2-Pentanol, 1-Butanol, 2-Methylfuran.

Table S4 1,5-PeD and 1,2-PeD hydrogenation over La/CuCoAl-DP catalyst

Entry	Substrate	Conv.%	Selectivity%		
			2-MF	1-BuOH	1-PeOH
1	1,5-PeD ^a	31.2	81.3	16.8	1.9
2	1,2-PeD ^a	-	-	-	-
3	1,5-PeD ^b	2.3	-	84.7	15.3
4	1,2-PeD ^b	-	-	-	-

Reaction conditions: Substrate 0.5g, catalyst 0.1 g, 4 MPa H₂; ethanol 39.5g, carbon balance in all reactions \geq 97%. 1,5-PeD:1,5-Pentenediol, 1,2-PeD:1,2-Pentenediol; 1-PeOH: 1-Pentanol, 1-BuOH: 1-Butanol, 2-MF: 2-Methylfuran. a: Temperature 200°C, reaction time 24 h, b: Temperature 160°C, reaction time 6 h

Table S5 XPS Elemental valence state distribution of catalyst

Catalyst	Cu%			Co%			O%		
	Cu ⁰	Cu ⁺	Cu ⁿ⁺	Co ⁰	Co ²⁺	Co ³⁺	O _I	O _{II}	O _{III}
CuCoAl	21.3	58.1	20.6	5.2	37.4	57.4	27.6	43.7	28.7
La/CuCoAl-CP	14.7	45.5	39.8	3.2	34.6	62.2	39.5	39.2	21.3
La/CuCoAl-IM	13.4	49.8	36.8	2.9	30.1	67.0	40.6	38.7	20.7
La/CuCoAl-DP ^a	11.6	46.0	42.4	2.0	35.9	62.1	36.6	40.3	23.1
La/CuCoAl-DP ^b	10.7	40.2	49.1	2.6	44.0	53.4	32.2	47.8	20.0
La/CuCoAl-DP ^c	17.2	47.3	35.5	6.8	38.7	54.5	20.6	44.8	34.6

a: 500°C calcined 3 h, 400°C reduced 1 h; b: 250°C calcined 8 h, 250°C reduced 6 h; c: Used 8th

Table S6 XPS parameters of La 3d in La/CuCoAl catalysts

Catalyst	La 3d ^{3/2} /eV		La 3d ^{5/2} /eV		ΔE_1 /eV	ΔE_2 /eV
La-CuCoAl-CP	852.0	855.5	835.2	838.5	16.8	3.3
La/CuCoAl-IM	850.7	855.1	833.9	838.5	16.8	4.6
La/CuCoAl-DP ^a	851.8	855.4	835.0	838.7	16.8	3.7
La/CuCoAl-DP ^b	851.9	855.4	835.1	839.0	16.8	3.9
La/CuCoAl-DP ^c	852.4	855.7	835.6	838.9	16.8	3.3

a: 500°C calcined 3 h, 400°C reduced 1 h; b: 250°C calcined 8 h, 250°C reduced 6 h; c: Used 8th

Table S7 Desorption amount of H₂ and CO₂ over the different catalysts

Catalyst	n(H) (mmol·g ⁻¹) ^a	CO ₂ (mmol·g ⁻¹) ^b
CuCoAl	0.28	0.10
La/CuCoAl-CP	0.39	0.24
La/CuCoAl-IM	0.37	0.20
La/CuCoAl-DP ^c	0.42	0.27
La/CuCoAl-DP ^d	0.79	0.84

^a: n(H) was calculated by H₂-TPD, ^b: CO₂ volume was calculated by CO₂-TPD, ^c: the catalyst was calcined at 500°C for 3 h and reduced at 400°C for 1 h; ^d: The catalyst was calcined at 250°C for 8 h and reduce at 250°C for 6 h.

Table S8 Desorption amount CO₂ over the different catalysts

Catalyst	CO ₂ (mmol·g ⁻¹) ^a		
	weak basic sites	medium basic sites	strong basic sites
CuCoAl	0.071	-	0.034
La/CuCoAl-CP	0.076	-	0.164
La/CuCoAl-IM	0.053	0.011	0.136
La/CuCoAl-DP	0.053	-	0.217
La/CuCoAl-DP ^b	0.479	0.244	0.134

^a: CO₂ volume was calculated by CO₂-TPD. 50~300°C: weak basic sites; 300~500°C: medium basic sites; 500~800°C: strong basic sites, ^b: The catalyst was calcined at 250°C for 8 h and reduce at 250°C for 6 h.

Table S9 The comparison of the catalytic ability of La/CuCoAl-DP with the non-noble metal catalysts reported in the literature

Entry	Substrate	Catalyst	Reaction conditions	Conversion %	Selectivity of 1,5-PeD %	Yield of 1,5-PeD %	Ref.
1	FFA	Co/TiO ₂	140°C, 2.34MPa WHSV=5.8 h ⁻¹	100	30.3	30.3	[4]
2	FFA	CuMg ₃ AlO _{4.5}	150°C, 1.5 MPa H ₂ , 24 h	100	28.8	28.8	[5]
3	FFA	Cu _{1.8} Mg _{1.2} Al	140°C, 6 MPa H ₂ , 10 h	95.2	15.6	14.9	[6]
4	FFA	Cu-Al ₂ O ₃	140°C, 8 MPa H ₂ , 8 h	85.8	22.3	19.1	[7]
5	FFA	Cu _{0.25} Co _{2.75} Al	160°C, 4 MPa H ₂ , 2 h	98.8	41.6	41.1	[8]
6	FFA	Cu _{0.2} Co _{5.8} /Al ₂ O ₃	140°C, 4MPa H ₂ , 2 h	97.2	45.5	44.7	[9]
7	FFA	Ni ₁ Co ₁₁ Al	160°C, 4 MPa H ₂ , 4 h	100	42.5	42.5	[10]
8	FFA	Ni _{4.5} Co _{3.5} Al	160°C, 3 MPa H ₂ , 6 h	100	48.7	48.7	[11]
9	FFA	Cu-LaCoO ₃	140°C, 6 MPa H ₂ , 2 h	100	40.3	40.3	[12]
10	FFA	Ni-Y ₂ O ₃	150°C, 2 MPa H ₂ , 24 h	100	41.9	41.9	[13]
11	FFA	Ni-La (2.5)-HT	150°C, 2 MPa H ₂ , 72 h	100	55.8	55.8	[14]
12	FFA	40Ni-La	150°C, 3 MPa H ₂ , 24 h	100	56.6	56.6	[15]
13	FFA	Co-MOF	170°C, 3 MPa H ₂ , 1.5 h	97.2	46	44.7	[16]
14	FFA	La/CuCoAl-DP	160°C, 4 MPa H ₂ , 2 h	100	59.5	59.5	This work

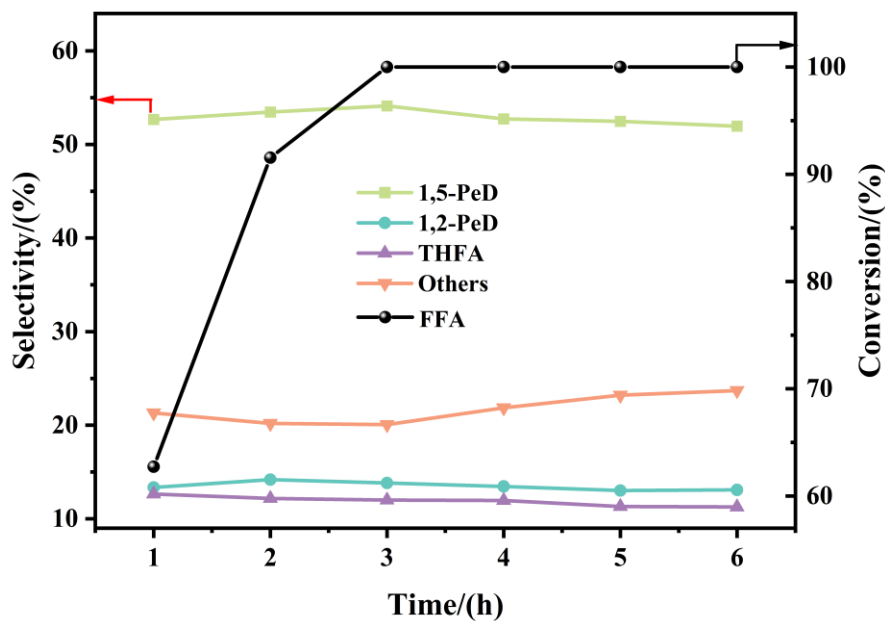


Figure S1 The effect of reaction time on the distribution of the products

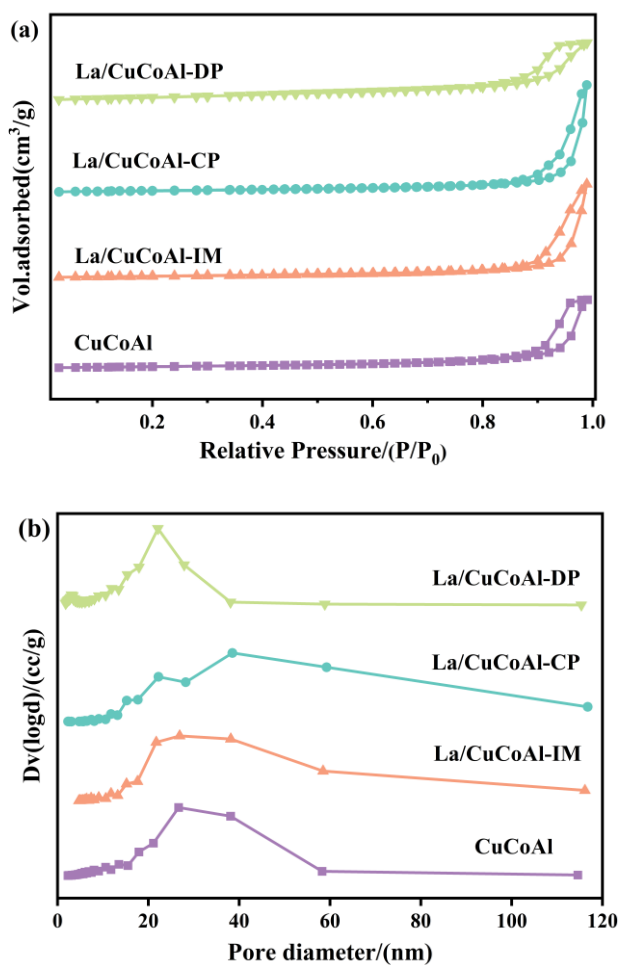


Figure S2 (a) N₂ adsorption-desorption isotherms of reduced catalysts; (b) Pore size distribution of reduced catalysts

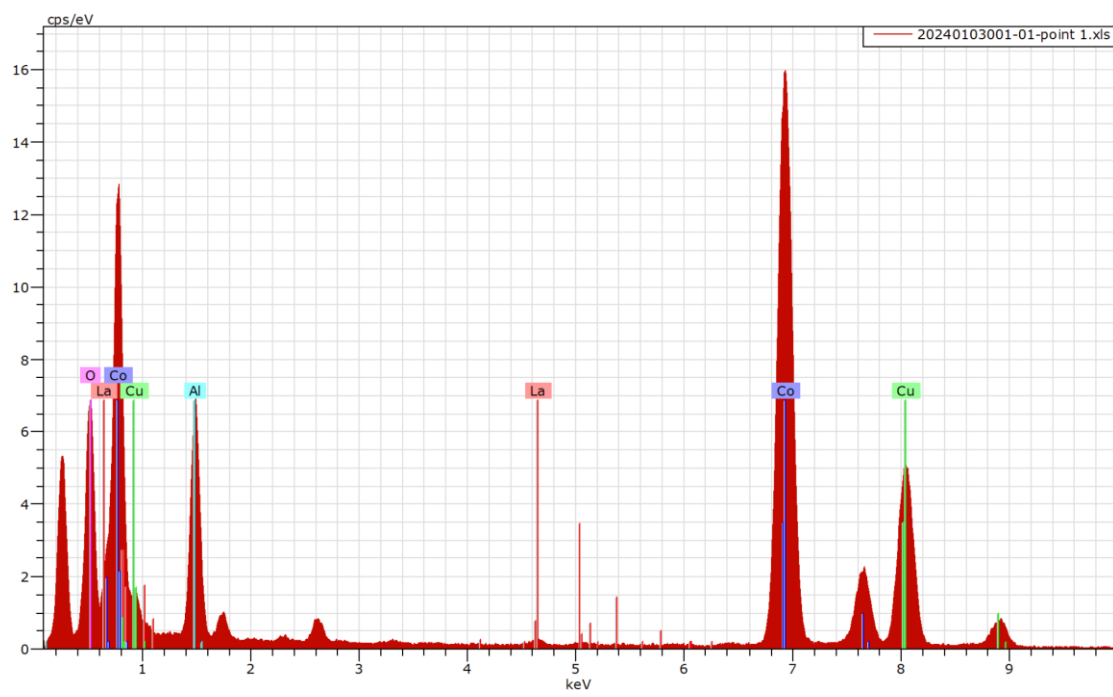


Figure S3 The elemental distribution of La/CuCoAl-DP catalyst after reduction

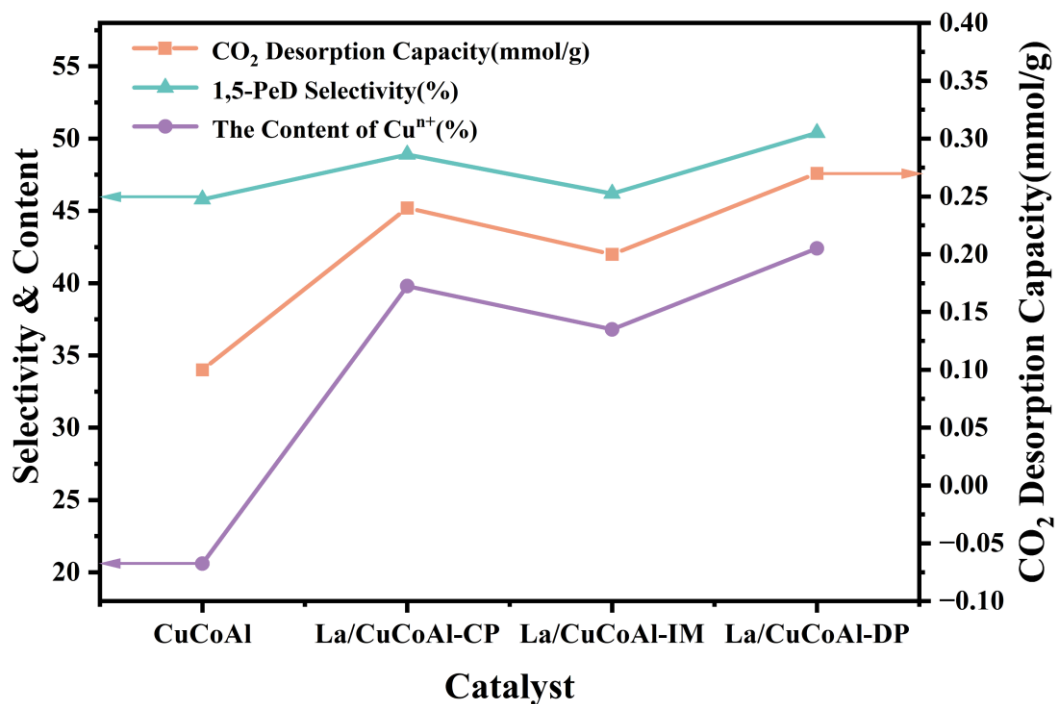


Figure S4 The influence of Cu⁺ content and CO₂ desorption on the selectivity of 1,5-PeD in catalysts

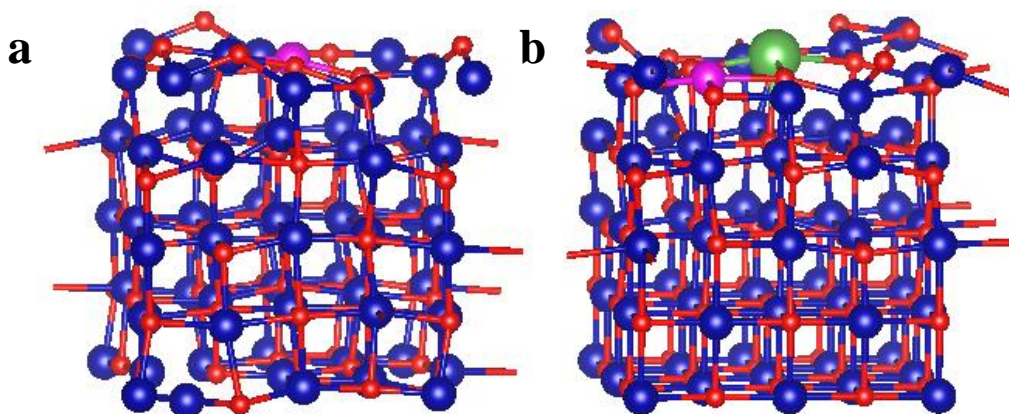


Figure S5 The optimized structures of (a) CuCo model (b) CuCoLa model slabs. Cu: magenta, Co: dark blue, La: green, O: red.

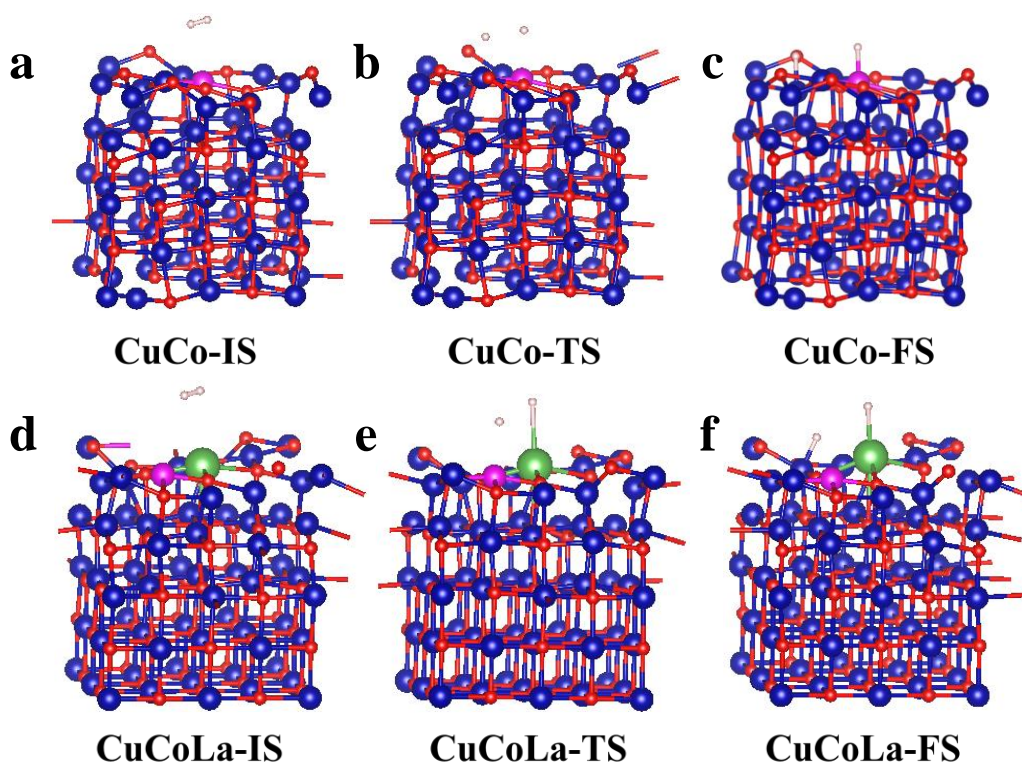


Figure S6 (a-c) Optimized geometries of the reactants, transition states and products dissociated from H_2 molecules to H atoms on CuCo surface; (d-f) Optimized geometries of the reactant, transition state and product of H_2 molecule dissociation to H atom on CuCoLa surface. Cu: magenta, Co: dark blue, La: green, O: red, H: light pink.

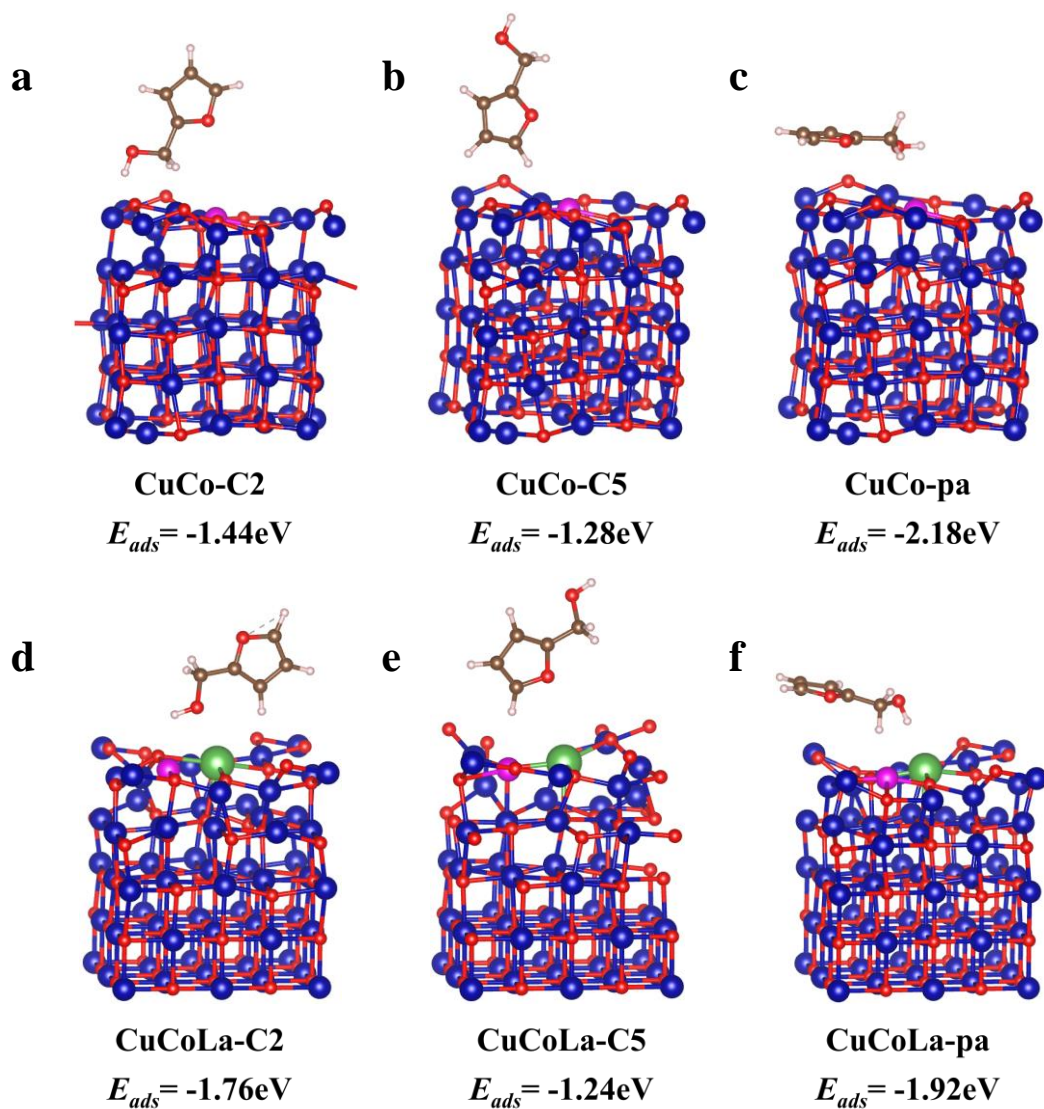


Figure S7 (a-c) Optimized adsorption geometries and adsorption energies of FFA on CuCo surface; (e-f) Optimized adsorption geometries and adsorption energies of FFA on CuCoLa surface. Here, E_{ads} denotes adsorption energy. Cu: magenta, Co: dark blue, La: green, O: red, H: light pink; C: Brown.

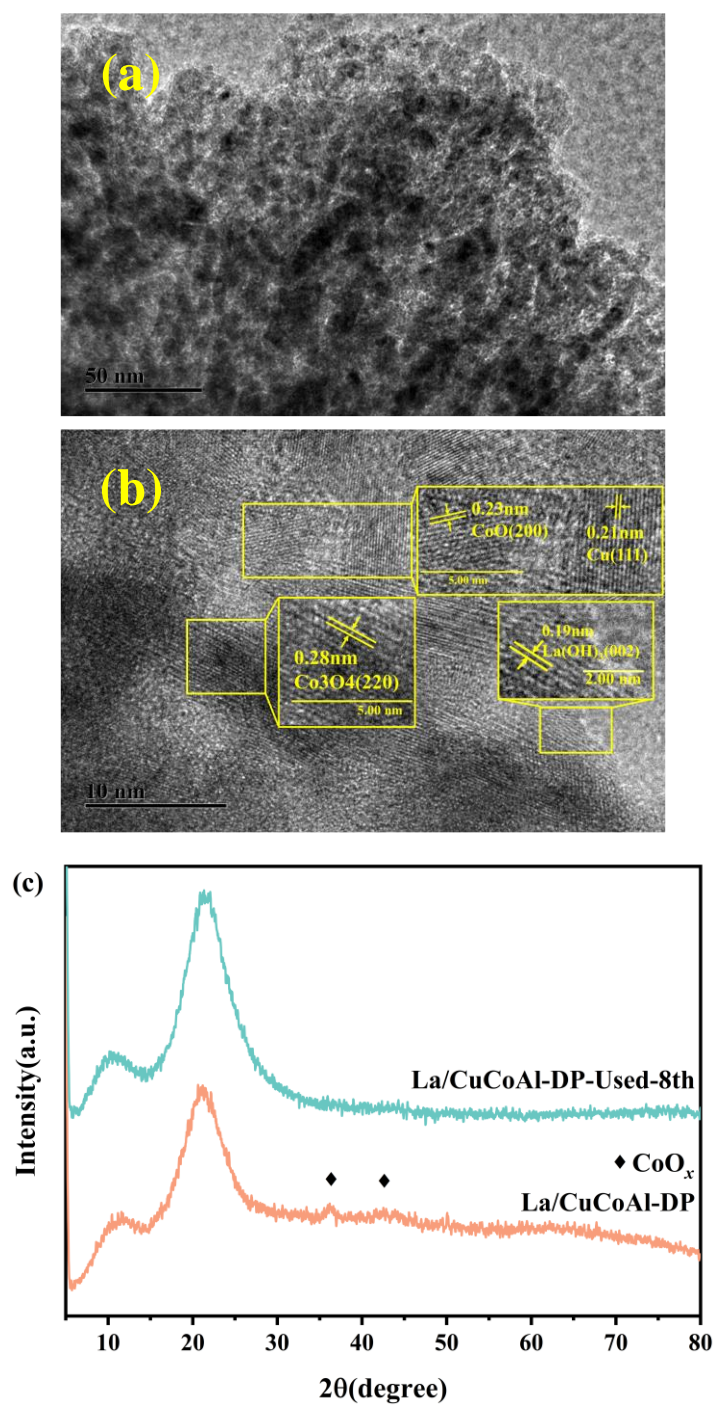


Figure S8 TEM images of the reduced La/CuCoAl-DP-Used-8th catalyst(a~b); (c) XRD patterns of the reduced La/CuCoAl-DP catalysts.

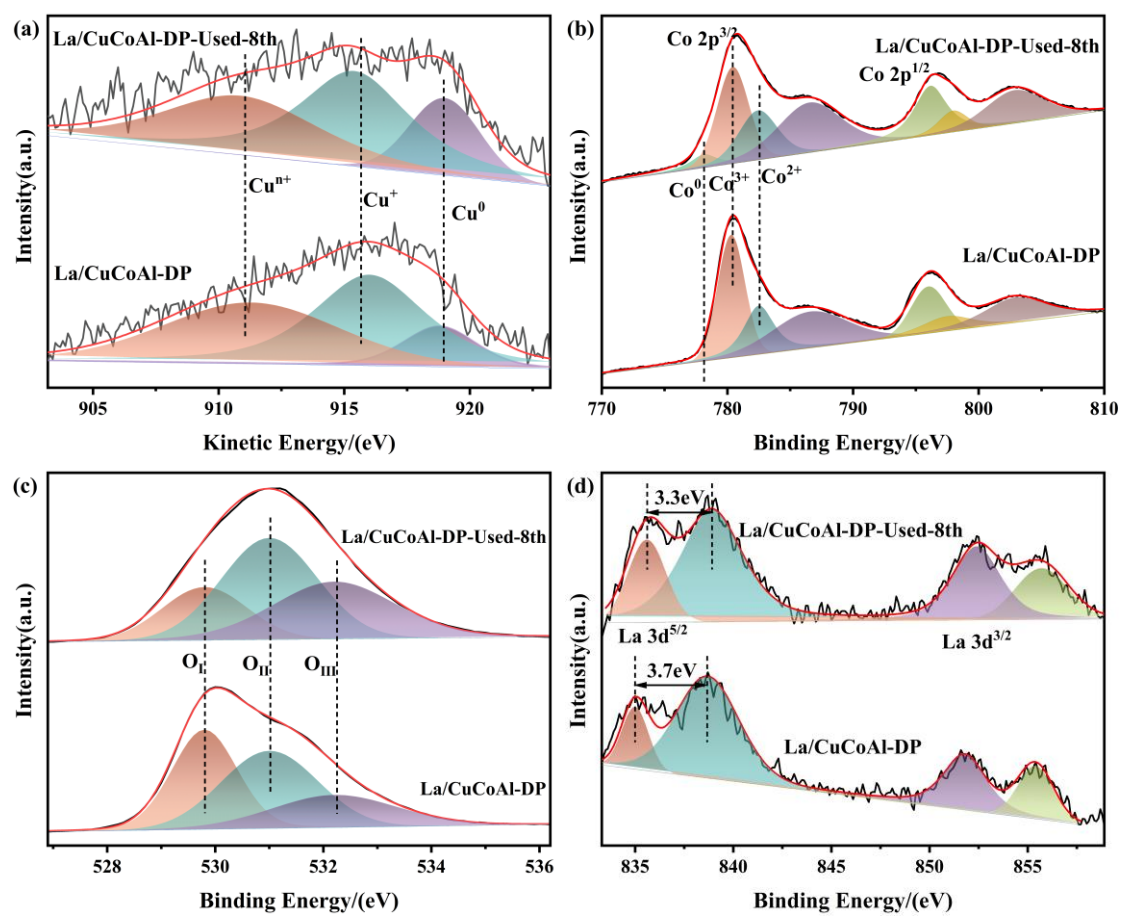


Figure S9 XPS patterns of reduced La/CuCoAl-DP catalysts

(a) Cu LMM XANS; (b) Co2p XPS; (c) O 1s XPS; (d) La 3d XPS

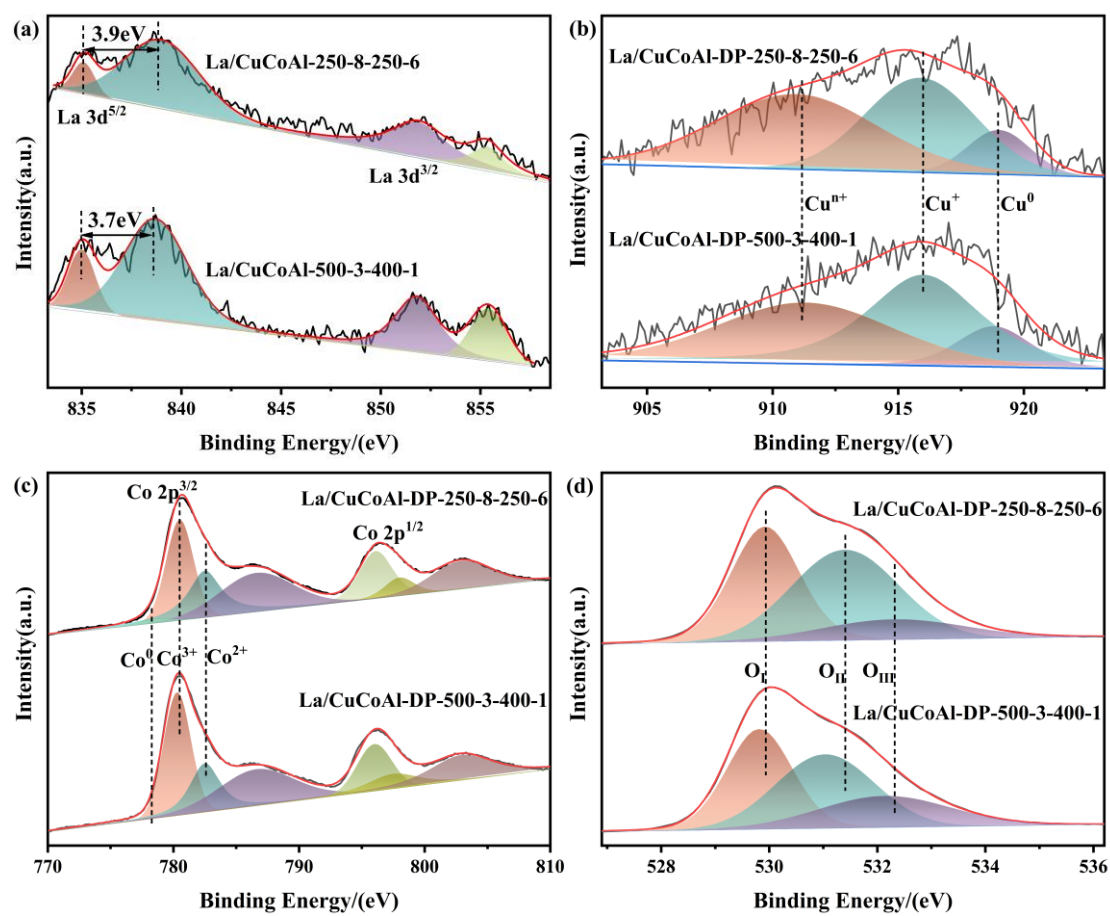


Figure S10 XPS patterns of reduced La/CuCoAl-DP catalysts

(a) La 3d XPS; (b) Cu LMM XANS; (c) Co 2p XPS; (d) O 1s XPS.

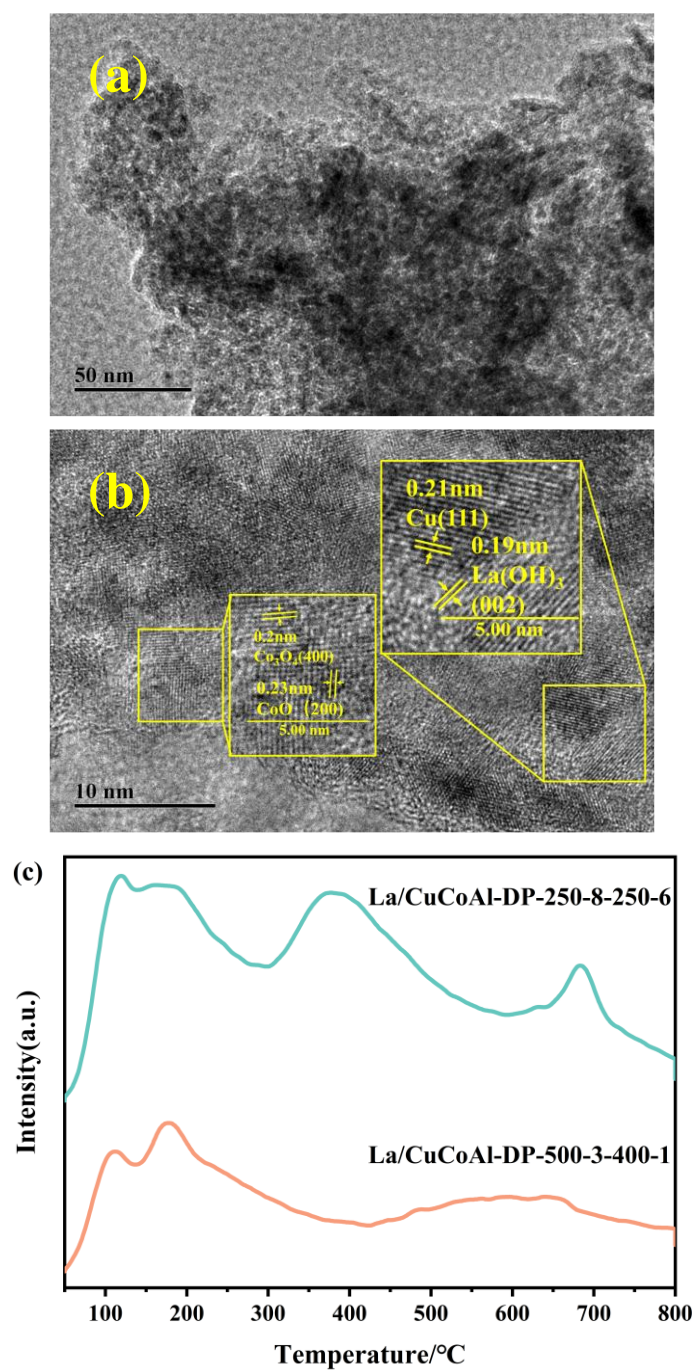


Figure S11 (a~b) TEM images of the reduced La/CuCoAl-DP-250-8-250-6 catalyst; (c) CO₂-TPD patterns of reduced La/CuCoAl-DP catalysts

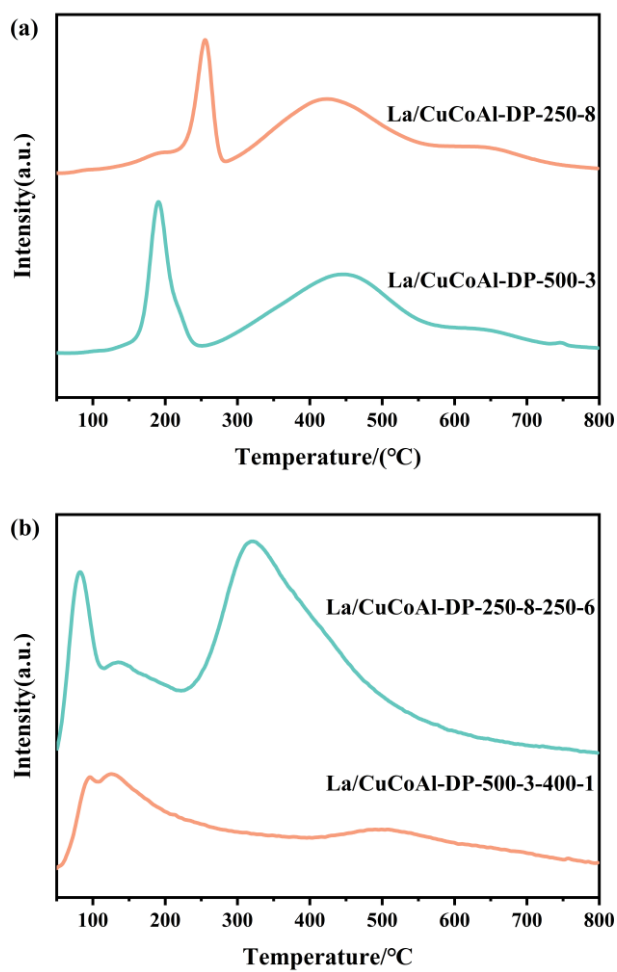


Figure S12 (a) H₂-TPR patterns of calcined catalysts; (b) H₂-TPD patterns of reduced catalysts

Notes and references

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