Supporting Information

Overcoming Activity/Stability Tradeoffs in CO Oxidation Catalysis by Pt/CeO_2

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1.- Materials synthesis

1.1.- Preparation of the CeO_2-Derived_Pt-Imp

The as-synthesized UiO-66(Ce) was calcined at 450°C (5°C/min) for 4 h in flowing air to obtain the resulting CeO_2-derived. Pt has been introduced by incipient wetness impregnation: 62 mg of an aqueous solution of chloroplatinic acid (8 wt.%) was added dropwise to 1 g of CeO_2-derived. Then, the mixture was dried at 100°C for 4 h. The resulting CeO_2-Derived_Pt-Imp sample was calcined at 450°C (5°C/min) for 5 h in flowing air to obtain high Pt dispersions with a final Pt amount of 0.49 wt% Pt

1.2.- Preparation of the supported Pt/CeO_2-LS (low-surface). This material has been prepared by incipient wetness impregnation: 96 mg of an aqueous solution of chloroplatinic acid (8 wt.%) was added dropwise to 1.5 g of CeO_2 (Sigma-Aldrich, 544841-25G, Lot #MKCK1143). Then, the mixture was dried at 100°C for 2 h. The resulting Pt/CeO_2-LS sample was calcined at 450°C (5°C/min) for 5 h in flowing air to obtain high Pt dispersions with a resultant Pt content of 0.51 wt% Pt.

1.3.- Preparation of the supported Pt/CeO_2-HS (high-surface). This material has been prepared by incipient wetness impregnation: 102 mg of an aqueous solution of chloroplatinic acid (8 wt.%) was added dropwise to 1.5 g of CeO_2 (Solvay, ITQ 3A, Lot # 0633984). Then, the mixture was dried at 100°C for 4 h. The resulting Pt/CeO_2-HS sample was calcined at 450°C (5°C/min) for 5 h in flowing air to obtain high Pt dispersions with a final Pt amount of 0.54 wt% Pt.

2.- Post-synthetic activations in CO

The previous calcined Pt/CeO_2-X (X = HS/LS) samples were reduced at 300°C for 40 min with 50 mL/min (4% CO and balanced with N_2). The resultant reduced-samples were named as Pt/CeO_2-X-CO. For the catalytic tests and DRIFT-based characterization, Pt/CeO_2-X were calcined *in situ* directly before the CO-treatment.

Supplementary Figures

Figure S1. Physicochemical properties of Pt/CeO$_2$-HS sample. **a)** Powder XRD pattern. **b)** HAADF-STEM image on zone axis [111]. **c)** N$_2$ adsorption and desorption isotherms. **d)** TEM image and **e)** HR-FESEM image.

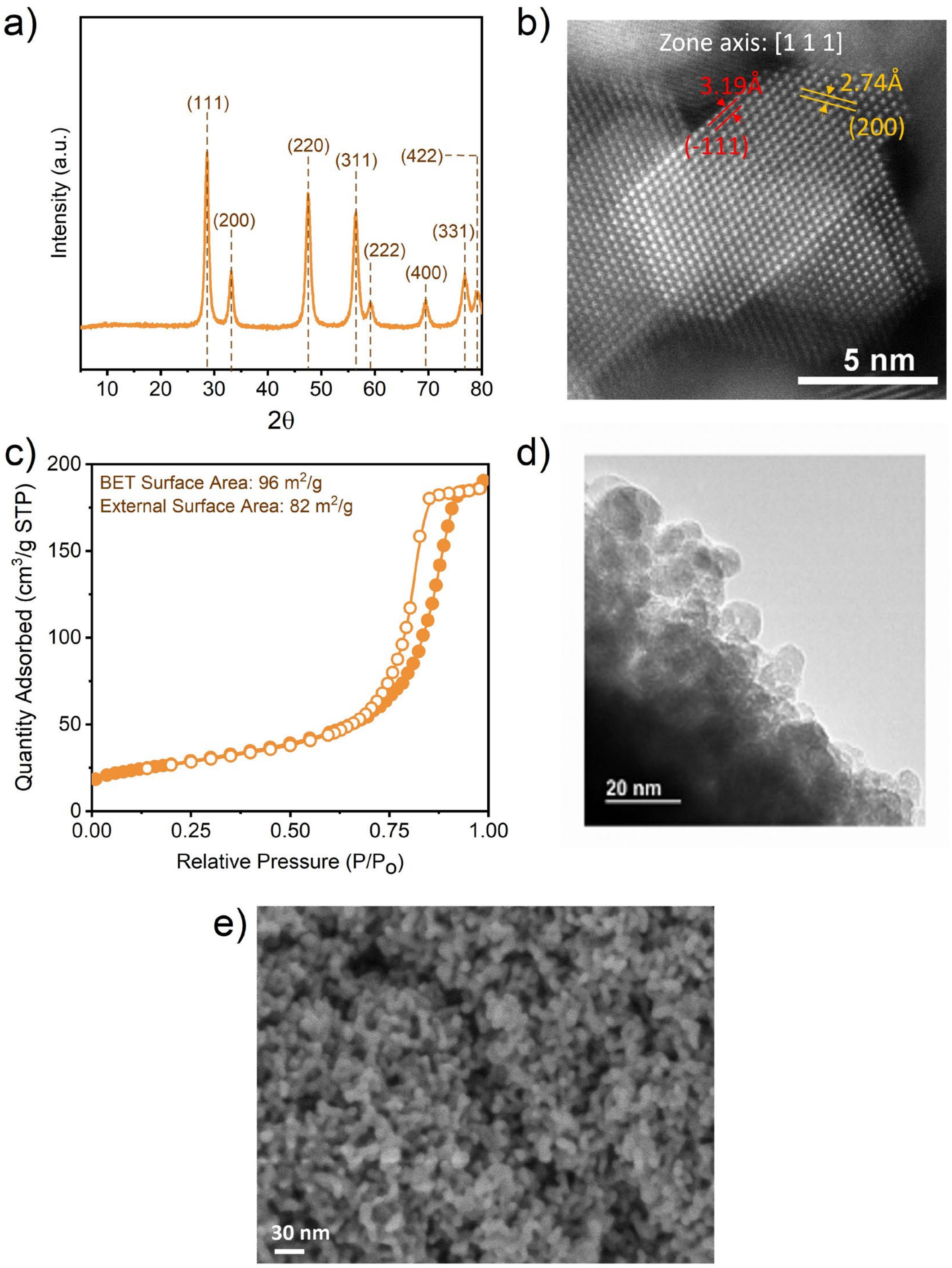


Figure S2. DRIFT spectra of CO adsorbed at room temperature of Pt/CeO_2-HS after calcination in O_2 at 450°C (dark orange) and after consecutive reduction in CO at 300°C (light orange).

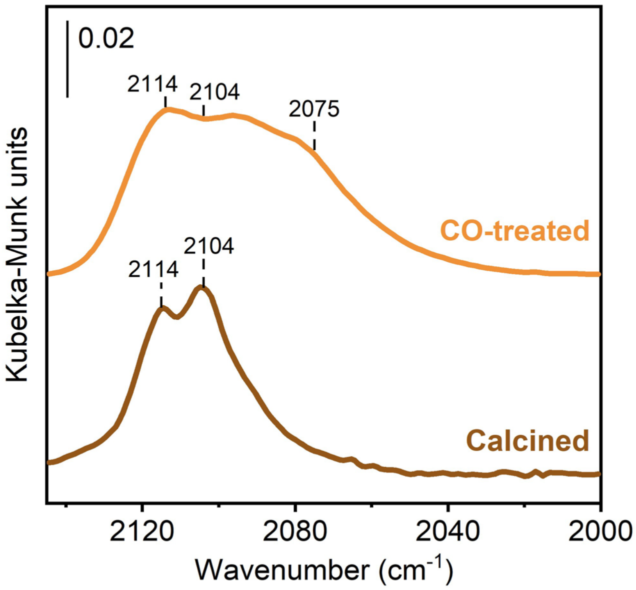


Figure S3 Experimental spectra (black line) and best fit curve (red line) of a,c,e) Pt/CeO$_2$-HS and b,d,f) Pt/CeO$_2$-derived in the a,b)EXAFS, c,d) FT-EXAFS magnitude and e,f) imaginary parts.

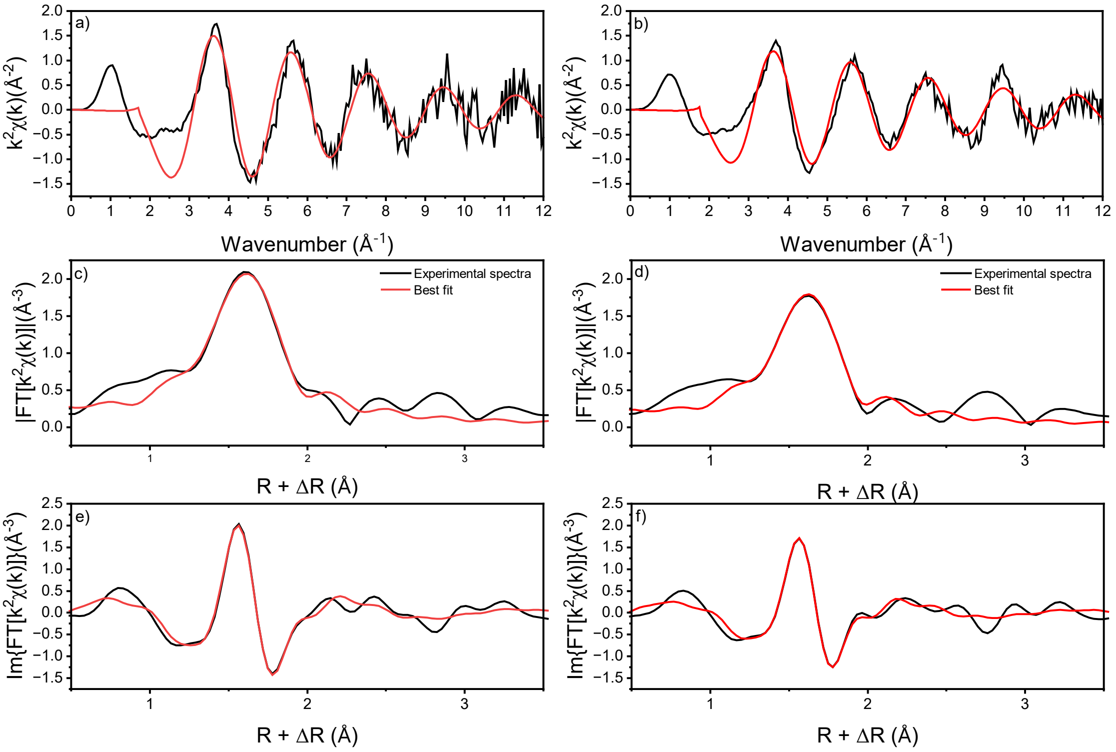


Figure S4. Catalysts aged at 300°C. Light-off conversion curves for the CO oxidation reaction for Pt/CeO_2-HS (orange, **a**), Pt/CeO_2-LS (grey, **b**), 3.5%Pt/CeO_2-HS (red, **c**) and Pt/CeO_2-Derived (blue, **d**) in their calcined forms (full symbol), after CO activation (empty symbol) and followed by a subsequent cycle after reaching 500°C (half full symbol). Reaction conditions: 1.2% CO, 9.7% O_2, 25 ml/min, normalized Pt content and WHSV = 5000 L/g_{Pt}·h.

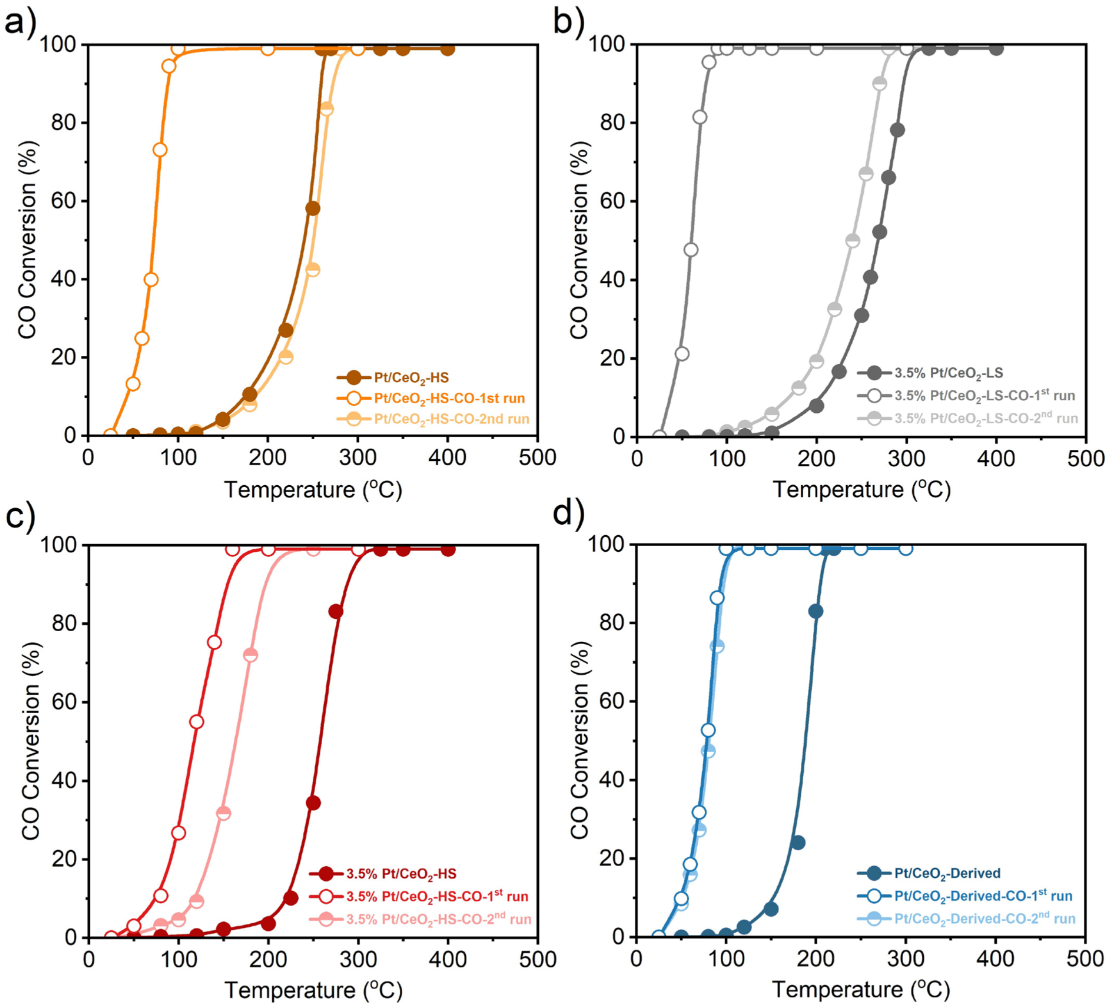


Figure S5. Physicochemical properties of Pt/CeO_2-LS sample. **a)** Powder XRD pattern. **b)** TEM image. **c)** N_2 adsorption and desorption isotherms and **d)** HR-FESEM image.

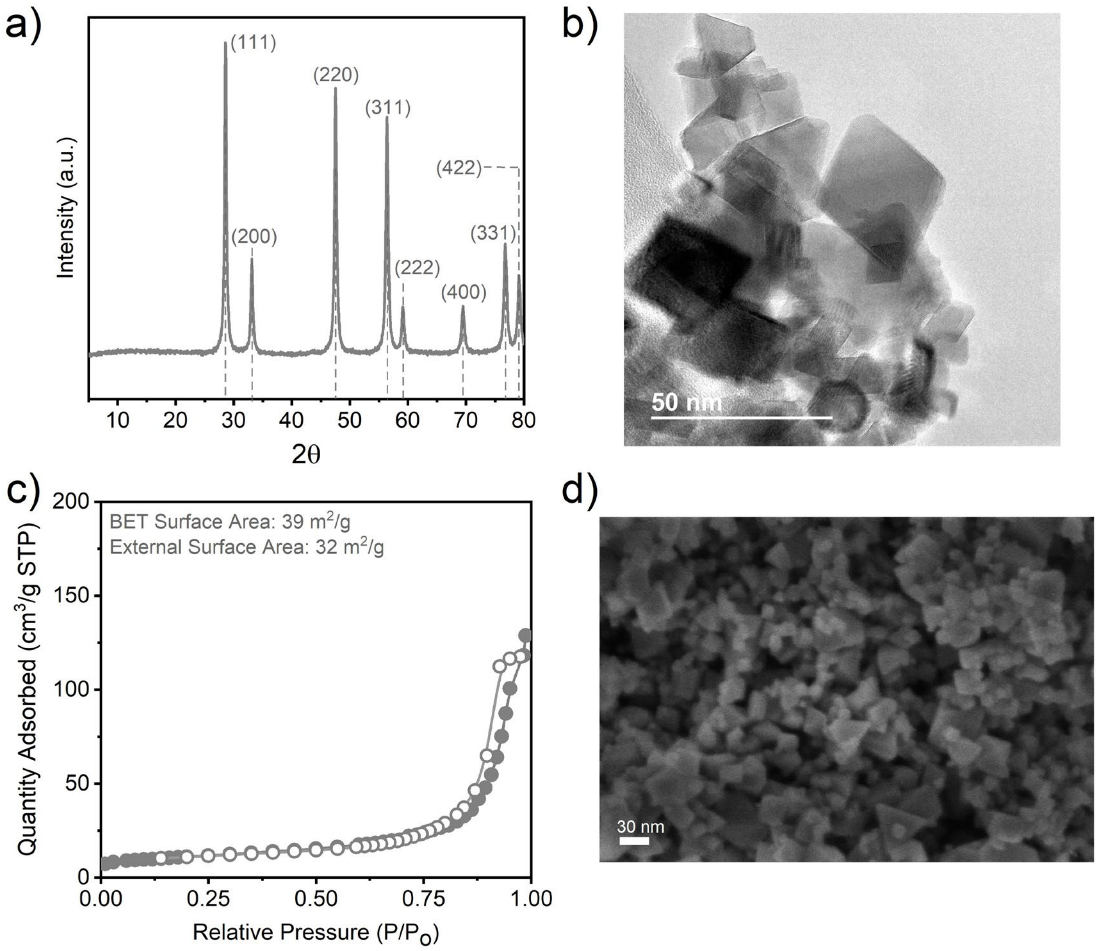


Figure S6. Pt/CeO_2-LS aged at 500°C. Light-off conversion curves for the CO oxidation reaction for the calcined Pt/CeO_2-LS material (full symbol), after CO activation (empty symbol) followed by a subsequent cycle after reaching 500°C (half full symbol). Reaction conditions: 1.2% CO, 9.7% O_2, 25 ml/min, normalized Pt content and WHSV = 5000 L/g_{Pt}·h.

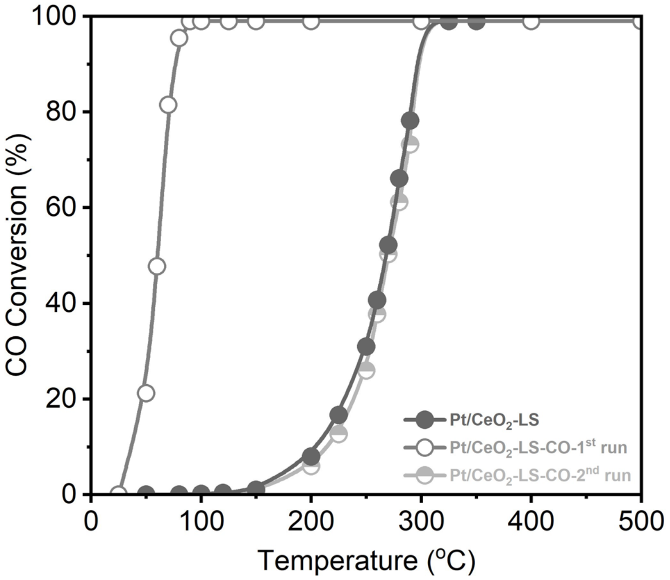


Figure S7. Physicochemical properties of Pt/CeO_2-Derived sample. **a)** Powder XRD pattern. **b)** HAADF-STEM image. **c)** N_2 adsorption and desorption isotherms. **d)** TEM image and **e)** HR-FESEM image.

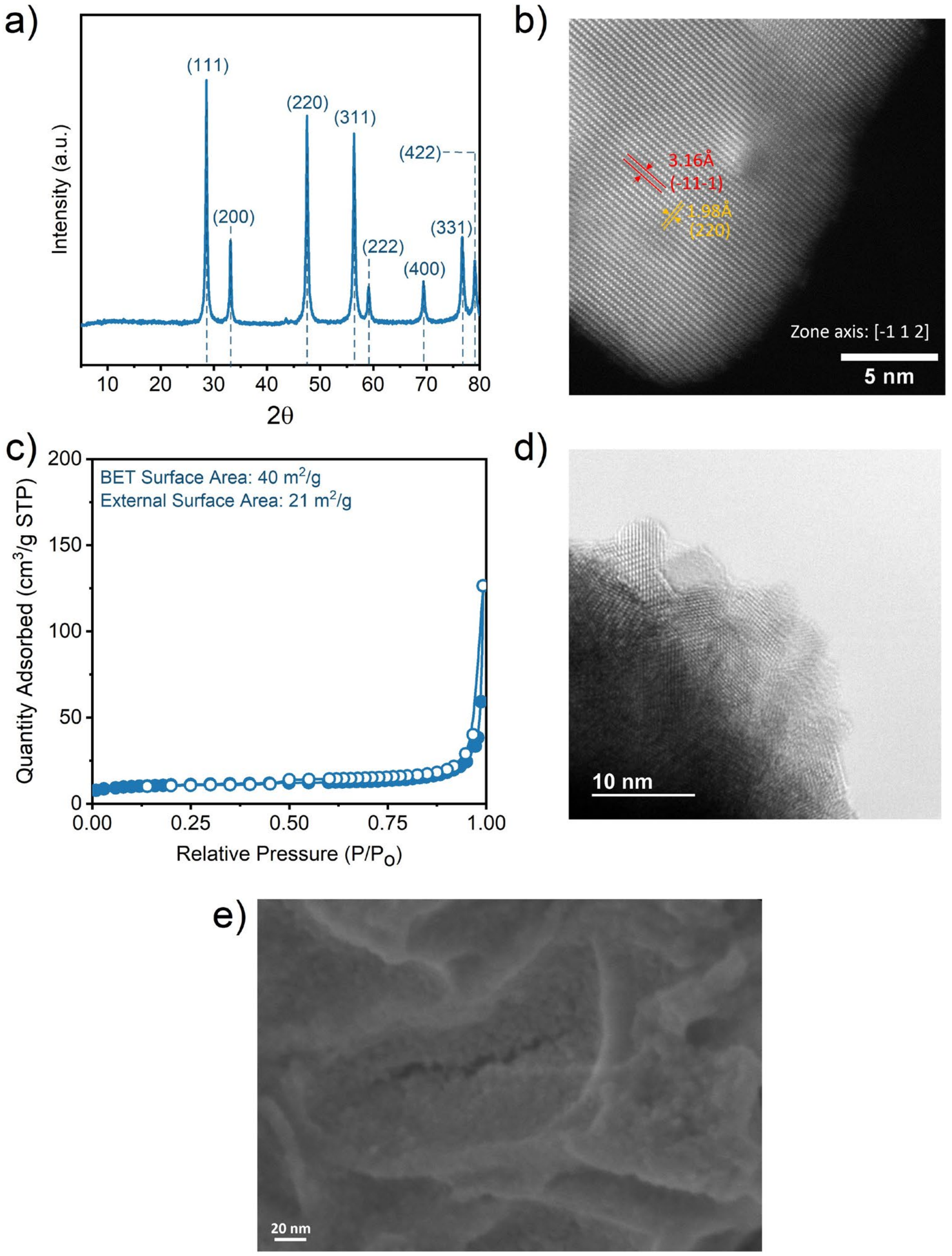


Figure S8. DRIFT spectra of CO adsorbed at room temperature of Pt/CeO_2-Derived after calcination in O_2 at 450°C (dark blue) and after consecutive reduction in CO at 300°C (light blue).

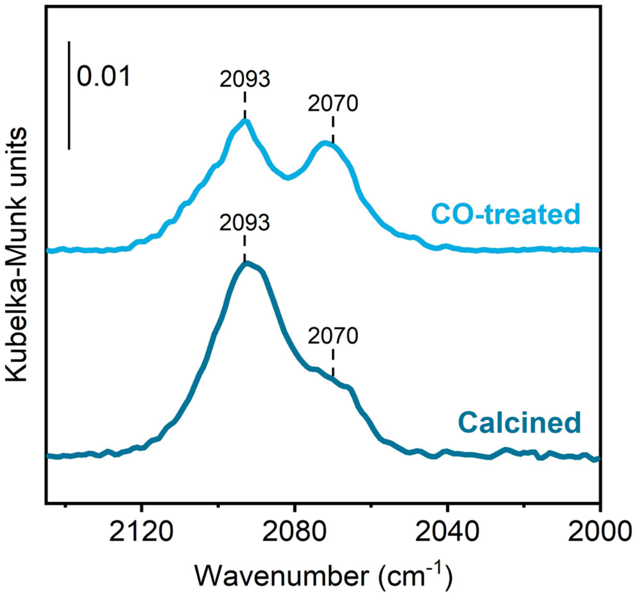


Figure S9. HAADF-STEM images of Pt/CeO_2-Derived directly after its calcination treatment at 450°C.

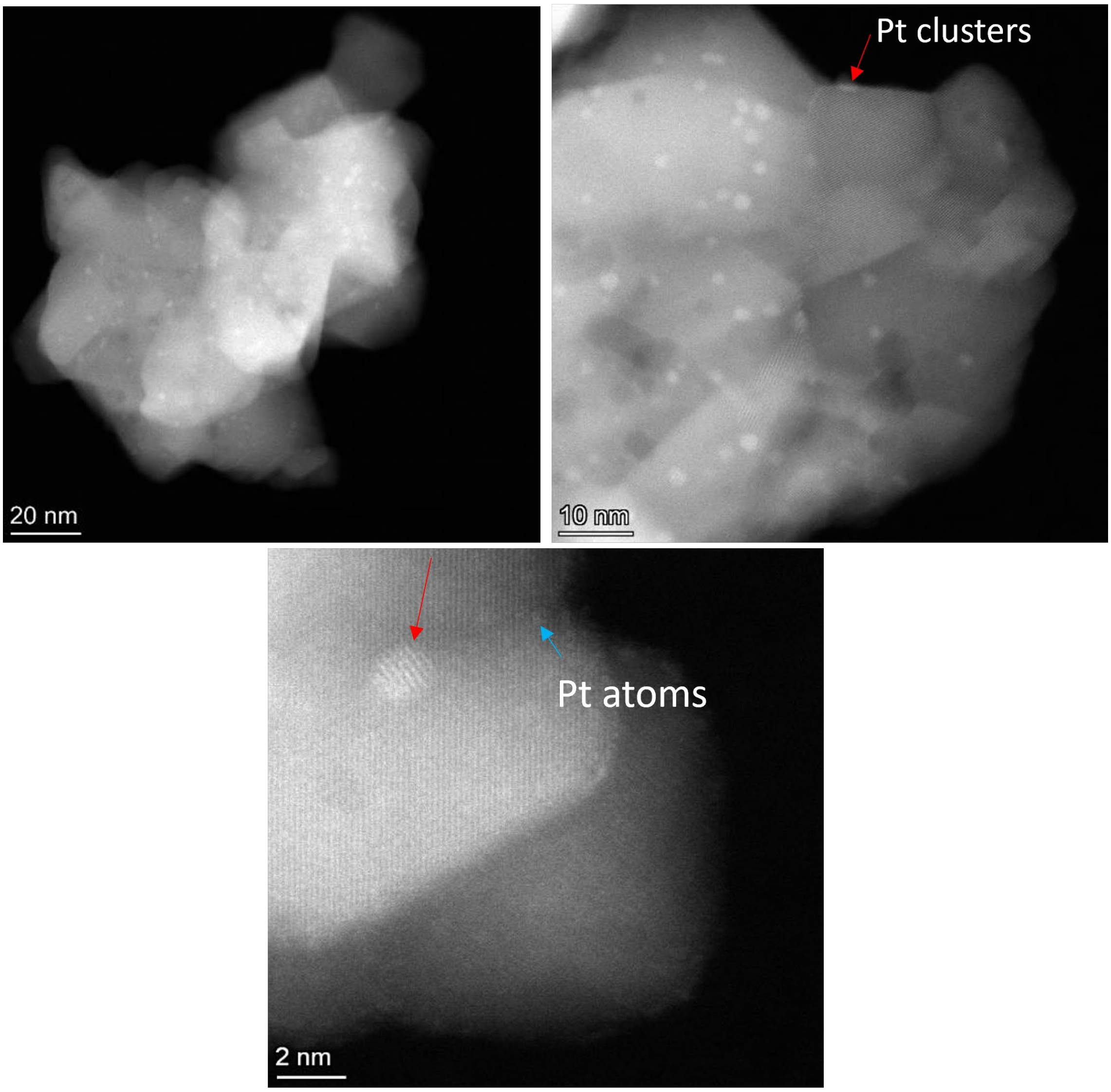


Figure S10. *d*-spacing of large Pt particles at calcined (A) and reduced (B) Pt/CeO_2-Derived sample (CO-treatment at 300°C).

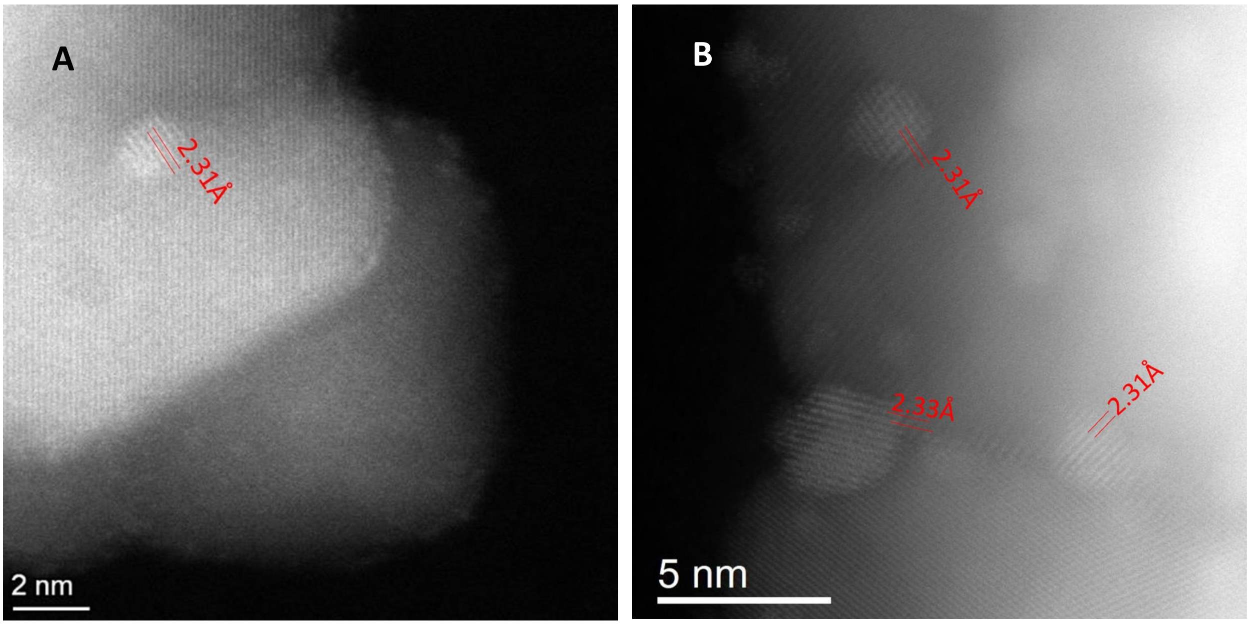


Figure S11. Light-off conversion curves for the CO oxidation reaction for Pt/CeO_2-Derived catalyst using higher WHSV, in its calcined form (full symbol), after CO activation (empty symbol) and followed by a subsequent cycle after reaching 500^{o}C (half full symbol). Reaction conditions: 1.2% CO, 9.7% O_2, 25 ml/min, normalized Pt content and WHSV = 20000 L/g_{Pt}·h.

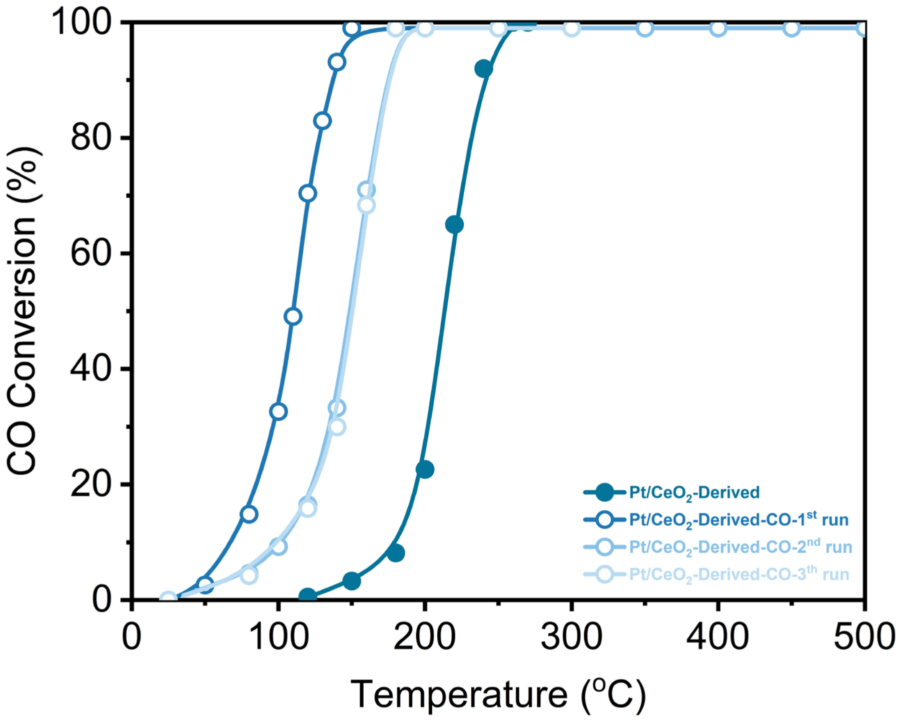


Figure S12. Light-off catalytic tests for the CO oxidation reaction using the CO-treated Pt/CeO_2-Derived sample in O_2-rich conditions (25 ml/min, containing 1.2 % CO, 9.7 % O_2, balanced with N_2) from 50°C to 300°C and held at 300°C for 1 h using a WHSV = 5000 L/g_{Pt}·h. After complete reaction in O_2-rich conditions, the Pt/CeO_2-Derived material was treated with CO (4% CO, balanced with N_2) at 300°C (Pt/CeO_2-Derived-CO), and tested for two consecutive light-off catalytic cycles. Afterwards, the catalyst was treated in air flow (50 ml/min) at 300°C and held for 1 h (Pt/CeO_2-Derived-CO-300 O_2), and tested for three consecutive light-off catalytic cycles. Finally, the catalyst was treated in air flow (50 ml/min) for 1 h at 500°C (Pt/CeO_2-Derived-CO-500 O_2), and tested for three consecutive light-off catalytic cycles.

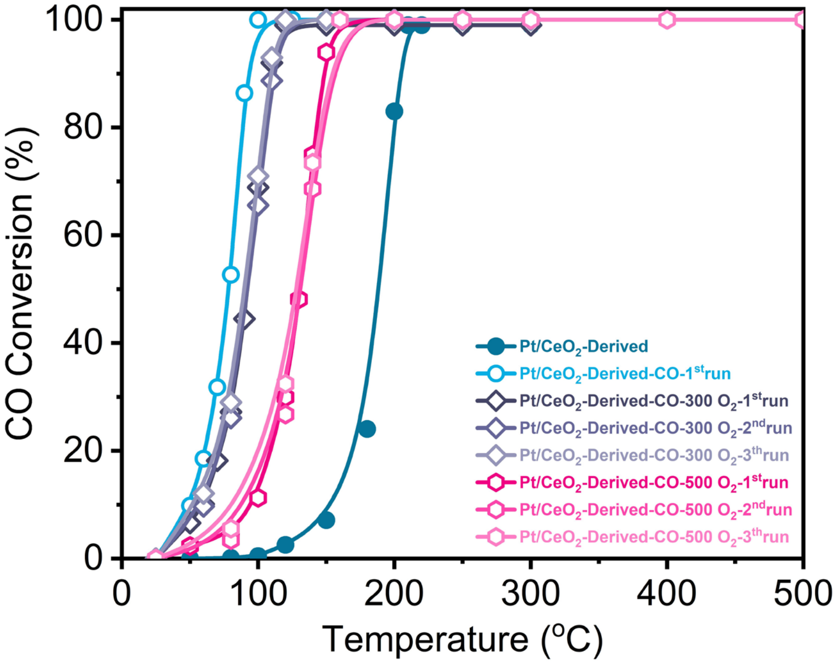


Figure S13. Comparison of the catalytic performance for the CO oxidation reaction using Pt/CeO_2-Derived [obtained by direct calcination of the Pt/UiO-66(Ce)] and CeO_2-Derived_Pt-Imp [obtained by calcination of the UiO-66(Ce) followed by impregnation of Pt]. **a)** Light-off catalytic cycles for both Pt-containing MOF-derived materials in their fresh forms and after CO activation. **b)** Time-on-stream performance for Pt-containing MOF-derived materials in isothermal CO oxidation experiments at 120°C. Reaction conditions: 1.2% CO, 9.7% O_2, 25 ml/min, normalized Pt content and WHSV = 5000 L/g_{Pt}·h.

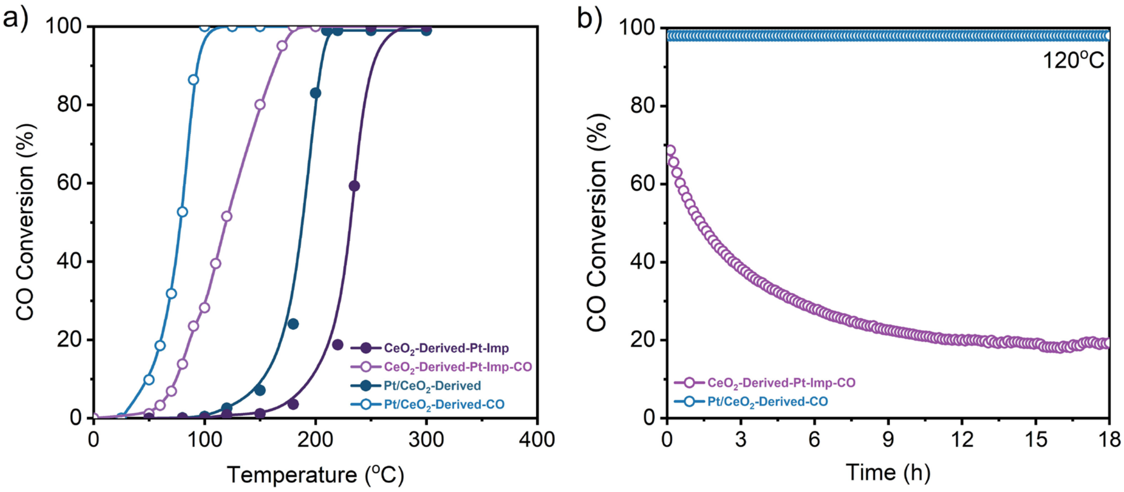


Figure S14. HAADF-STEM images of Pt/CeO$_2$-LS after reduction in CO at 300°C.

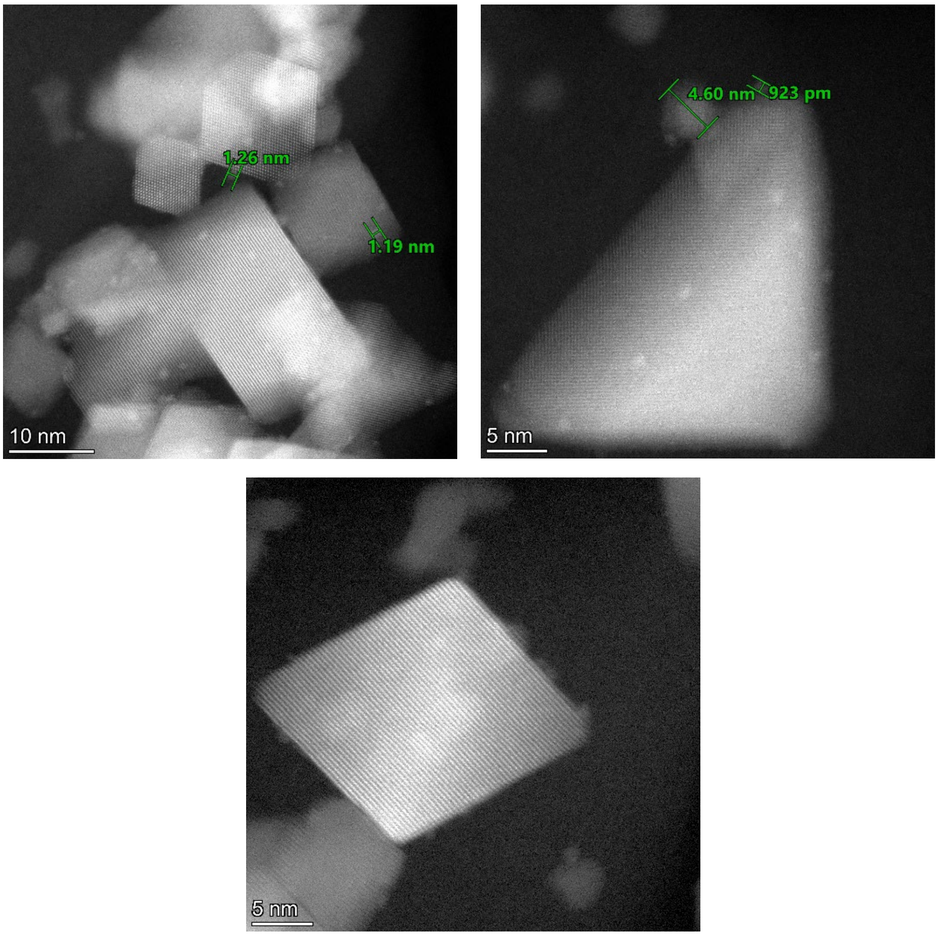


Figure S15. HAADF-STEM images of CeO_2-Derived-Pt-Imp after reduction in CO at 300°C.

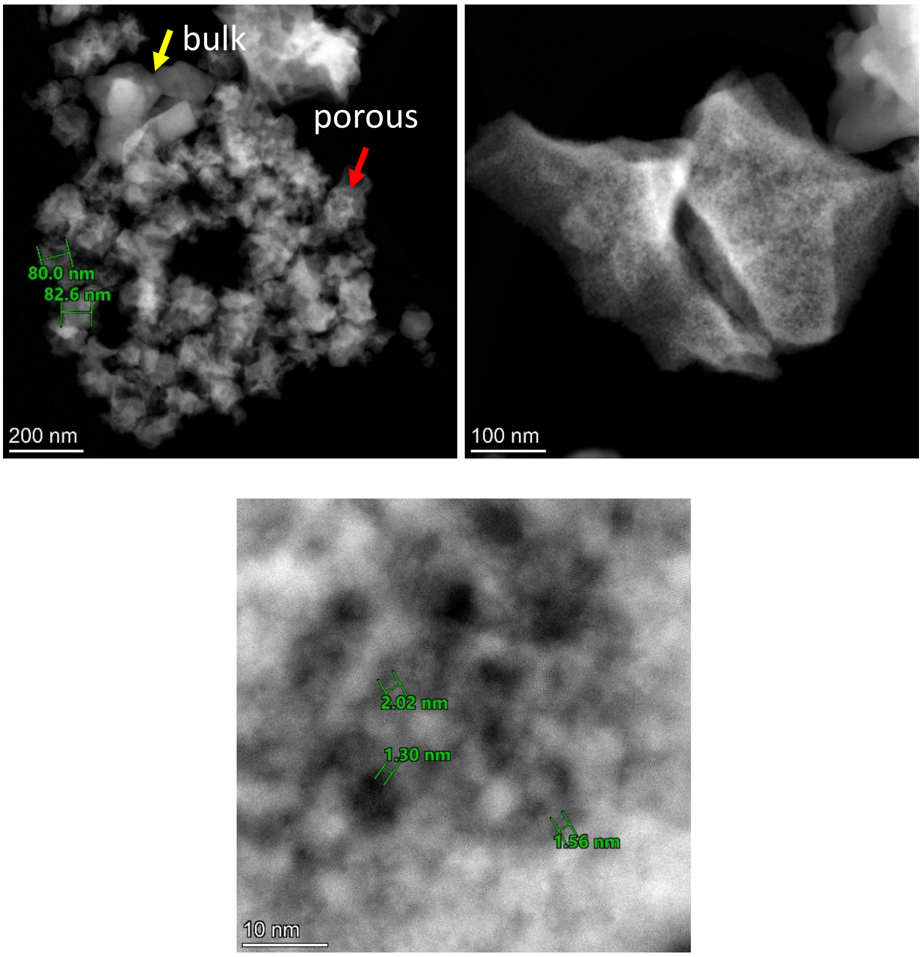


Figure S16. N_2 physisorption isotherms at liquid nitrogen temperature for samples Pt/CeO_2-Derived and CeO_2-Derived. The former includes Pt on the parent MOF at the moment of its calcination in air at 450°C, whereas the latter corresponds to calcination of the MOF under the same conditions prior to deposition of the Pt.

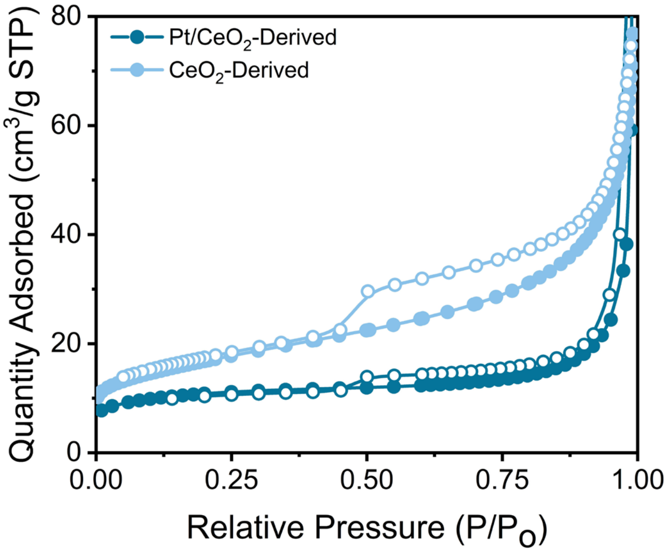


Figure S17. a) Isolated Pt_{10} clusters of diverse morphology and side view of the CeO_2-V surface model employed in the periodic DFT study. Optimized geometries of these Pt_{10} clusters supported on **b)** $CeO_2(111)$ and **c)** CeO_2-V surface models. Ce, O and Pt atoms are depicted as gray, red and blue balls.

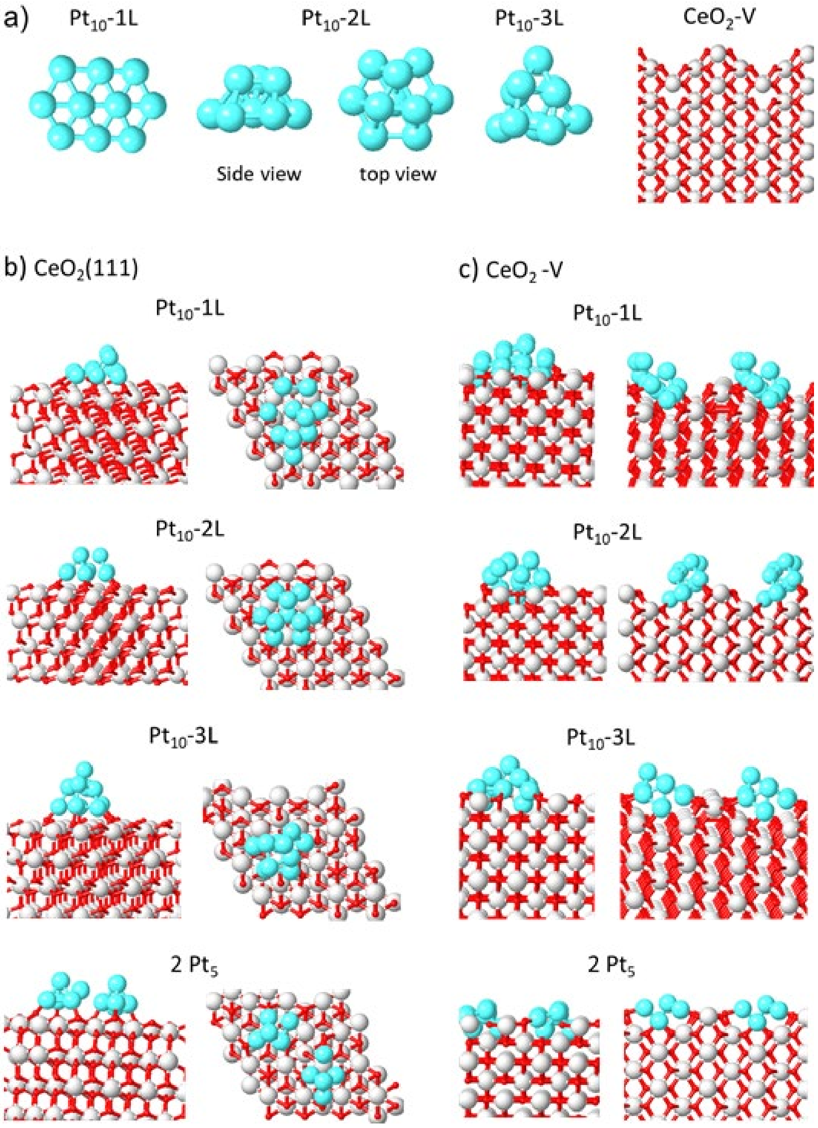


Figure S18. Optimized structures of partly oxidized $Pt_{10}O_x$ clusters supported on **a)** $CeO_2(111)$ and **b)** CeO_2-V surface models obtained from periodic DFT calculations. Ce, O and Pt atoms are depicted as grey, red and blue balls.

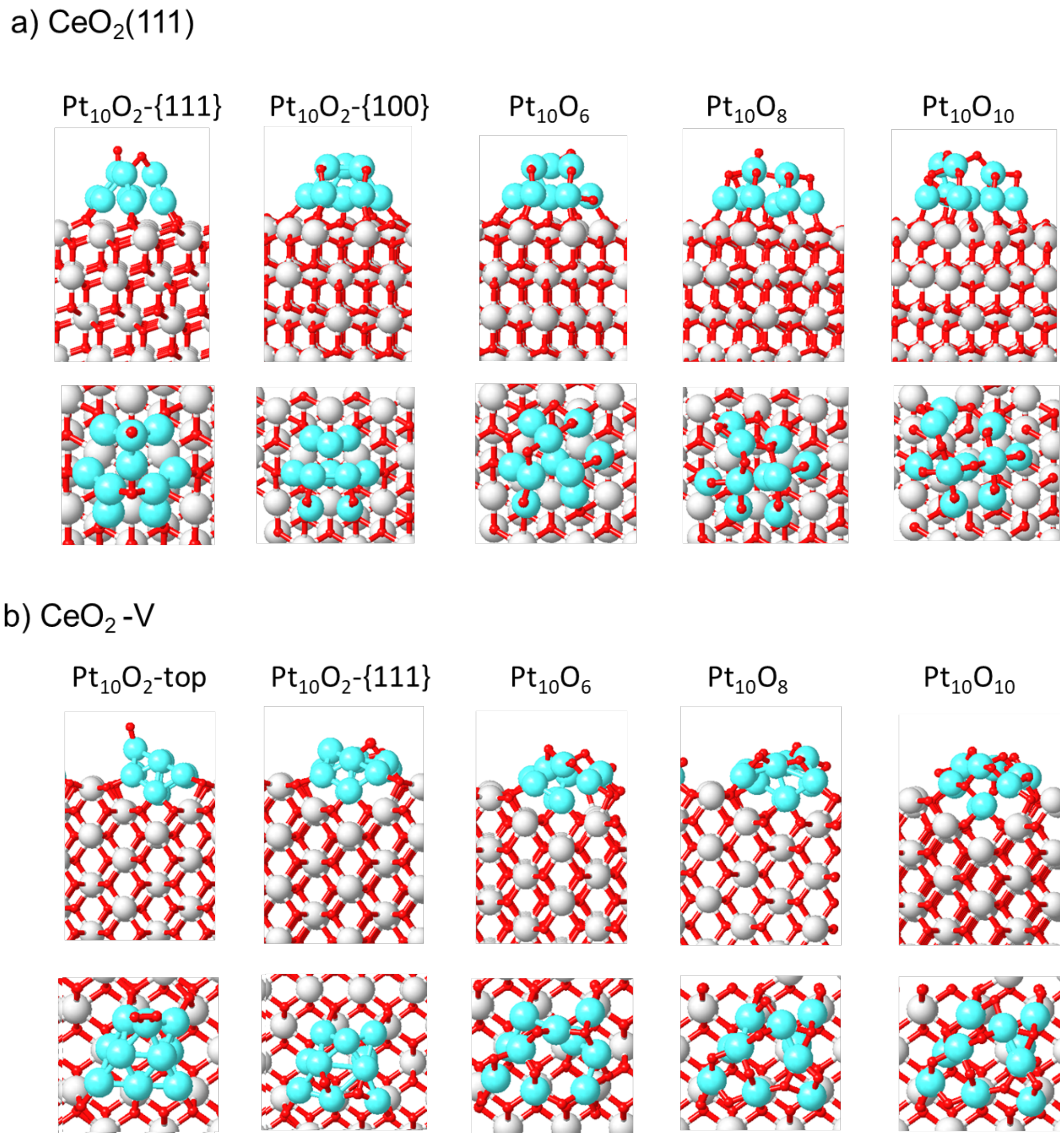
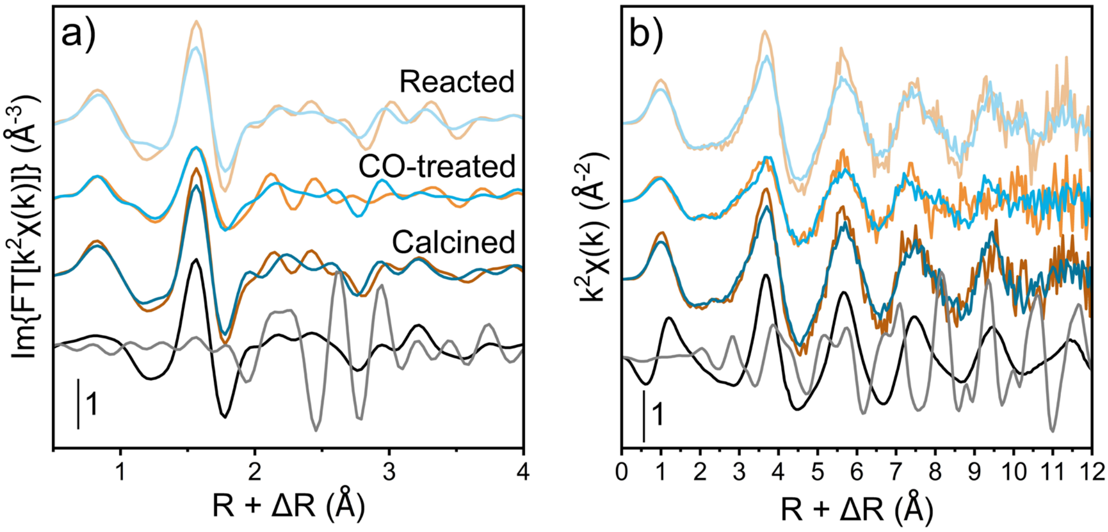


Figure S19. a) Imaginary components of the k^2-weighted FT-EXAFS spectra and b) experimental k^2-weighted EXAFS spectra obtained for Pt/CeO_2-Derived (blue lines) and Pt/CeO_2-HS (orange lines) after various sequential thermal treatments (calcination in O_2 at 450°C, reduction in CO at 300°C, and reaction with O_2 and CO at 500°C). Pt and PtO_2 spectra are reported for clarity with gray and black lines, respectively.



Supplementary Tables

Table S1: Physical-chemical properties of the CeO_2 supports and the final Pt/CeO_2 materials

	Pt (wt%)	BET surface area (m^2/g)
Pt/UiO-66(Ce)	0.18	1071
CeO_2-Derived	-	59
Pt/CeO_2-Derived[1]	0.46	39
CeO_2-HS	-	110
Pt/CeO_2-HS	0.54	96
CeO_2-LS	-	40
Pt/CeO_2-LS	0.51	38
CeO_2-Derived	-	59

[1] Pt impregnated on the MOF, then calcined in air at 450°C; [2] CeO_2 product that results from calcination of a Pt-free MOF

Table S2. EXAFS fit results for calcined Pt/CeO_2 samples. EXAFS spectra were extracted in the 2.8-12 Å^{-1} k-range while fit was performed in the 1-2.3 Å R-range. S_0^2 was fixed to 0.8 from fit of reference metallic Pt.

Variable	Pt/CeO_2-HS	Pt/CeO_2-Derived
R-factor	0.014	0.014
E_0	11.5 ± 1.5	11.7 ± 1.4
CN Pt-O	6.4 ± 0.8	4.9 ± 0.6
σ^2 (Pt-O)	0.0035 ± 0.0015	0.0023 ± 0.0012
R (Pt-O)	2.004 ± 0.011	2.002 ± 0.009

Table S3. A comparison of activity in CO oxidation reaction on different Pt catalysts in this work.

Sample	Pt (wt%)	Conditions	T (°C)	Conversion (%)	TOF (s^{-1})
Pt/CeO_2-LS	0.51	5 000 L g_{Pt}^{-1} h^{-1} [CO] = 1.2 vol%, [O_2] = 9.7 vol%	80	0.09	0.0001
			120	0.33	0.0007
			150	1.02	0.002
			180	4.51	0.006
			200	7.94	0.013
Pt/CeO_2-LS-CO	0.51	5 000 L g_{Pt}^{-1} h^{-1} [CO] = 1.2 vol%, [O_2] = 9.7 vol%	50	21.18	0.029
			60	47.71	0.065
			70	81.55	0.111
			80	95.53	0.129
Pt/CeO_2-HS	0.54	5 000 L g_{Pt}^{-1} h^{-1} [CO] = 1.2 vol%, [O_2] = 9.7 vol%	80	0.22	0.0003
			120	0.67	0.0009
			150	4.17	0.006
			180	10.60	0.014
			200	20.22	0.027
Pt/CeO_2-HS-CO	0.54	5 000 L g_{Pt}^{-1} h^{-1} [CO] = 1.2 vol%, [O_2] = 9.7 vol%	50	13.25	0.018
			60	24.92	0.034
			70	40.17	0.054
			80	73.83	0.099
3.5%Pt/CeO_2-HS	3.52	5 000 L g_{Pt}^{-1} h^{-1} [CO] = 1.2 vol%, [O_2] = 9.7 vol%	80	0.06	$8.17 \cdot 10^{-5}$
			120	0.52	0.0007
			150	0.70	0.0009
			200	3.57	0.005
3.5%Pt/CeO_2-HS-CO	3.52	5 000 L g_{Pt}^{-1} h^{-1} [CO] = 1.2 vol%, [O_2] = 9.7 vol%	80	10.74	0.014
			100	26.73	0.035
			120	55.04	0.074
Pt/CeO_2-Derived	0.46	5 000 L g_{Pt}^{-1} h^{-1} [CO] = 1.2 vol%, [O_2] = 9.7 vol%	80	0,52	0.0007
			100	1.47	0.002
			120	2.54	0.003
			150	7.14	0.010
			180	24.03	0.033
			200	82.33	0.112
Pt/CeO_2-Derived-CO	0.46	5 000 L g_{Pt}^{-1} h^{-1} [CO] = 1.2 vol%, [O_2] = 9.7 vol%	50	9.82	0.013
			60	18.53	0.025
			70	31.82	0.043
			80	52.67	0.072
Pt/CeO_2-Derived	0.46	20 000 L g_{Pt}^{-1} h^{-1} [CO] = 1.2 vol%, [O_2] = 9.7 vol%	120	0.51	0.003
			150	3.28	0.018
			180	8.15	0.044
			200	22.60	0.122
Pt/CeO_2-Derived-CO	0.46	20 000 L g_{Pt}^{-1} h^{-1} [CO] = 1.2 vol%, [O_2] = 9.7 vol%	50	2.59	0.014
			60	4.34	0.024
			70	8.80	0.048
			80	14.85	0.081
			100	32.62	0.178
			120	70.39	0.385

Table S4. Comparison of the CO oxidation activity, expressed as a TOF on a per total Pt basis (mol converted/mol Pt. s), of the Pt/CeO_2 catalysts prepared here with those reported in the literature under analogous reaction conditions. TOFs after stability tests between 200-400°C are reported in brackets.

Sample	Pt (wt%)	Pt specie	Conditions	Conv. (%) 80°C	TOF (s^{-1}) 80°C	Conv. (%) 100°C	TOF (s^{-1}) 100°C	Ref.
Pt/CeO_2-HS-CO	0.54	Cluster *(Single Atom)*	5 000 L g_{Pt}^{-1} h^{-1} [CO] = 1.2 vol%, [O_2] = 9.7 vol% *(Stability up to 300°C - 2 nd cycle)*	73.83 *(0.17)*	0.0992 *(0.00024)*	73.83 *(0.35)*	0.0992 *(0.00047)*	Our work
3.5% Pt/CeO_2-HS-CO	3.52	Cluster *(Single atom-PtOx)*	5 000 L g_{Pt}^{-1} h^{-1} [CO] = 1.2 vol%, [O_2] = 9.7 vol% *(Stability up to 300°C - 2 nd cycle)*	10.74 *(3.14)*	0.0144 *(0.0041)*	10.74 *(4.63)*	0.0144 *(0.0062)*	Our work
Pt/CeO_2-Derived-CO	0.46	Cluster *(Cluster)*	20 000 L g_{Pt}^{-1} h^{-1} [CO] = 1.2 vol%, [O_2] = 9.7 vol% *(Stability up to 300°C - 2 nd cycle)*	14.85 *(12.62)*	0.0811 *(0.0703)*	32.62 *(30.25)*	0.178 *(0.151)*	Our work
Meso Pt/$Ce_{0.8}Zr_{0.2}O_2$	0.40	Cluster *(Cluster)*	60 000 mL g^{-1} h^{-1} [CO] = 1 vol%, [O_2] = 21 vol% *(Stability up to 200°C - 10th cycles)*	5 *(5)*	0.0166 *(0.0166)*	10 *(10)*	0.0332 *(0.0332)*	[1]
Pt/CeO_2-TS	1.00	Single Atom *(Single Atom)*	200 L g^{-1} h^{-1} [CO] = 1 vol%, [O_2] = 10 vol% *(Stability up to 350°C - 5 nd cycle)*	3 *(3)*	0.0133 *(0.0133)*	5 *(5)*	0.0222 *(0.0222)*	[2]
$Pt_1/CeO_x/SiO_2$-H_2	0.40	Single Atom *(Single Atom)*	20 000 mL g^{-1} h^{-1} [CO] = 1 vol%, [O_2] = 4 vol% *(Stability up to 200°C - 4 th cycle)*	3 *(3)*	0.0022 *(0.0033)*	8 *(8)*	0.0088 *(0.0088)*	[3]
Pt-SA-Ce-MOF	0.12	Single Atom *(Single Atom)*	120 000 mL g^{-1} h^{-1} [CO] = 0.3 vol%, [O_2] = 7.5 vol% *(Stability up to 200°C - 10th cycle)*	8 *(8)*	0.0120 *(0.0120)*	13 *(13)*	0.0195 *(0.0195)*	[4]
Pt_1Pt_{NP}/CeO_2-19% SA	0.71	Single Atom + NP *(Single Atom + NP)*	120 000 mL g^{-1} h^{-1} [CO] = 0.3 vol%, [O_2] = 7.5 vol% *(Stability up to 350°C - 7 th cycle)*	1.5 *(1.5)*	0.0028 *(0.0028)*	3 *(3)*	0.0056 *(0.0056)*	[5]

Table S5. Comparison of the CO oxidation activity, expressed as a TOF on a per total Pt basis (mol converted/mol Pt. s), of the Pt/CeO_2 catalysts prepared here with those reported in the literature under analogous reaction conditions. TOFs after stability tests at 500°C are reported in brackets.

Sample	Pt (wt%)	Pt specie	Conditions	Conv. (%) 80°C	TOF (s^{-1}) 80°C	Reference
Pt/CeO_2-HS-CO	0.54	Cluster *(Single atom)*	5 000 L g_{Pt}^{-1} h^{-1} [CO] = 1.2 vol%, [O_2] = 9.7 vol% *(Stability up to 500°C - 2 nd cycle)*	73.83 *(0.097)*	0.0992 *(0.00013)*	Our work
3.5 % Pt/CeO_2-HS-CO	3.52	Cluster *(Single atom-PtOx)*	5 000 L g_{Pt}^{-1} h^{-1} [CO] = 1.2 vol%, [O_2] = 9.7 vol% *(Stability up to 500°C - 2 nd cycle)*	10.74 *(0.89)*	0.0144 *(0.0012)*	Our work
Pt/CeO_2-Derived-CO	0.46	Cluster *(Cluster)*	20 000 L g_{Pt}^{-1} h^{-1} [CO] = 1.2 vol%, [O_2] = 9.7 vol% *(Stability up to 500°C - 2 nd cycle)*	14.85 *(4.62)*	0.0811 *(0.0250)*	Our work
Pt-NP (Pt/CeO_2)	0.94	Cluster *(Single atom)*	60 000 L g_{Pt}^{-1} h^{-1} [CO] = 1000 ppm, [O_2] = 8 vol% *(Stability up to 500°C - 2 nd cycle)*	60 *(< 1)*	0.0798 *(< 0.0013)*	[6]
$Pt_{AT}CeO_2$-CO	1.00	Cluster *(Single atom)*	300 L g^{-1} h^{-1} [CO] = 1 vol%, [O_2] = 5 vol% *(Stability up to 500°C - 2 nd cycle)*	37 *(< 1)*	0.2460 *(<0.0066)*	[7]
Pt/CeO_2-S	1.00	Single Atom *(Single Atom)*	200 000 mL g^{-1} h^{-1} [CO] = 0.4 vol%, [O_2] = 10 vol% *(Stability up to 500°C - 2 nd cycle)*	5 *(5)*	0.0090 *(0.0090)*	[8]

Table S6. Comparison of the CO oxidation activity, expressed as a TOF on a per total Pt basis (mol converted/mol Pt. s), of the Pt/CeO_2 catalysts prepared here with those reported in the literature under analogous reaction conditions. TOFs after stability tests between 200-400°C are reported in brackets.

T (°C)	Sample A / B	Pt (wt%)	Conditions	Conv. (%)	*TOF (s^{-1})*	X-times	Refs.
200	K_0-Pt@MFI *600-H_2*	0.2	15 900 L g_{Pt}^{-1} h^{-1} [CO] = 1.3 vol%, [O_2] = 8.2 vol%	9.37	*0.0436*	4.6	[9]
	K_0-Pt@MFI *200-H_2*	0.2	15 900 L g_{Pt}^{-1} h^{-1} [CO] = 1.3 vol%, [O_2] = 8.2 vol%	2.04	*0.0095*		
190	$K_{0.6}$-Pt@MFI	0.2	15 900 L g_{Pt}^{-1} h^{-1} [CO] = 1.3 vol%, [O_2] = 8.2 vol%	15.98	*0.0729*	3	[9]
	K_0-Pt@MFI	0.2	15 900 L g_{Pt}^{-1} h^{-1} [CO] = 1.3 vol%, [O_2] = 8.2 vol%	5.13	*0.0234*		
80	Pt/CeO_2-HS-CO	0.5	5 000 L g_{Pt}^{-1} h^{-1} [CO] = 1.2 vol%, [O_2] = 9.7 vol%	73.83	*0.0992*	330	This work
	Pt/CeO_2-HS	0.5	5 000 L g_{Pt}^{-1} h^{-1} [CO] = 1.2 vol%, [O_2] = 9.7 vol%	0.041	*0.0003*		
120	3.5%Pt/CeO_2-HS	0.2	5 000 L g_{Pt}^{-1} h^{-1} [CO] = 1.2 vol%, [O_2] = 9.7 vol%	55.04	*0.0734*	107	This work
	3.5%Pt/CeO_2-HS-CO	0.2	5 000 L g_{Pt}^{-1} h^{-1} [CO] = 1.2 vol%, [O_2] = 9.7 vol%	0.515	*0.00069*		
120	Pt/CeO_2-Derived-CO	0.5	20 000 L g_{Pt}^{-1} h^{-1} [CO] = 1.2 vol%, [O_2] = 9.7 vol%	70.39	*0.3849*	120	This work
	K_0-Pt@MFI	0.2	15 900 L g_{Pt}^{-1} h^{-1} [CO] = 1.3 vol%, [O_2] = 8.2 vol%	0.676	*0.0032*		[9]

Table S7. Energy of interaction between Pt_{10} clusters of different morphology and CeO_2 surfaces (E_{int}, in eV) and total atomic charge on the supported Pt_{10} clusters (qPt_{10}, in e).

	$CeO_2(111)$		CeO_2-V	
Morphology	E_{int} (eV)	qPt_{10} (e)	E_{int} (eV)	qPt_{10} (e)
1L	-6.503	0.680	-6.00	0.631
2L	-6.946	0.669	-6.345	0.638
3L	-5.691	0.658	-8.891	0.690
$2Pt_5$	-4.320	0.680	-8.346	1.321

Table S8. Energy of interaction between Pt_{10}/CeO_2 and O_2 to form partly oxidized $Pt_{10}O_x/CeO_2$ systems ($E_{int}(O_2)$, in eV). Total atomic charge on the Pt_{10} cluster (qPt_{10}) is given in e.

	$CeO_2(111)$		CeO_2-V	
System	$E_{int}(O_2)$ (eV)	qPt_{10} (e)	$E_{int}(O_2)$ (eV)	qPt_{10} (e)
$Pt_{10}O_2$ - corner			-1.747	1.690
$Pt_{10}O_2$ - {111}	-2.270	2.157	-2.238	2.245
$Pt_{10}O_2$ - {100}	-3.228	2.236		
$Pt_{10}O_6$	-9.156	5.385	-7.997	5.556
$Pt_{10}O_8$	-9.905	6.068	-7.873	7.096
$Pt_{10}O_{10}$	-11.364	8.478	-10.587	8.753

Table S9. Parameters used for XPS fitting of Pt4f for all samples in all conditions.

Component	Position (eV)	FWHM
$^{*}Pt^{0}$	71.4	2.0
	74.7	2.0
Pt^{+2}	72.9	2.0
	76.2	2.0
Pt^{+4}	74.6	2.0
	77.9	2.0

$^{*}Pt^{0}$ in the case of Pt/Derived-CO treated was corrected to 71.2 eV position.

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