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Supplementary Material

Oxygen-Vacancy Enhanced CoO/CeO₂ Heterojunction for Synchronous Regulation of Sulfur Resourcing and Selenium Adsorption Separation from Flue Gas Desulfurization Wastewater

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Texts

Text S1 Chemicals and materials

All reagents were used directly without further purification. Cobalt nitrate hexahydrate (Co(NO₃)₂·6H₂O, 99%, Macklin Co., China.) and urea (H₂NCONH₂, 99%, Kermel Co., China.) were used to synthesize cobalt carbonate via hydrothermal method. Cerium nitrate (Ce(NO₃)₂·6H₂O, 99%, Macklin Co., China.) and polyvinylpyrrolidone (PVP, 99%, Kermel Co., China.) were used to synthesize CoO/CeO2 catalysts. In addition, ethanol (C2H5OH, 99.7%, Damao Technology Co., China) and deionized (DI) water obtained from Milli-Q ultrapure water purification system (Millipore, Billerica, MA) were used as washing solution. Sodium borohydride (NaBH₄, 99%, Damao Technology Co., China.) was used to establish oxygen vacancies on the catalysts. Sulfite (Na2SO3, 97%, Damao Technology Co., China.) and selenium (Se, 1000ug/mL in 2.0 mol/L HNO₃, Aladdin Co., China.) were used as the target pollutants. HCl (36-38%wt, Damao Co. Technology Co., China.), thiourea (CH₄N₂S, 99%, Kermel Co., China.) and L-ascorbic acid (99.7%, Kermel Co., China.) were used as the reducing solution to when testing the concentration of Se. Sodium hydroxide (NaOH,99%, Damao Co. Technology Co., China.) and HCl were used to adjust the pH of mixture solution. A commercial polytetrafluoroethylene (PTFE) microfiltration membrane (Φ =50 mm, pore diameter=0.45 µm) was used to collect products by filtration.

Text S2. Adsorption Kinetics

To reveal the behavior and mechanism of Se adsorption, the Se adsorption kinetics were further studied based on the pseudo-first-order model, pseudo-second-order model and intra-particle diffusion model.¹⁻³ The related parameters are summarized in Table S2. The fitting result shown in Fig. S3 indicates that the pseudofirst-order model is not fit. By contrast, as shown in Fig. S4, the regression coefficients (R^2) of the pseudosecond-order fitting curve for all materials are close to 1, and the $q_{e,cal}$ values are well agree with the experimental q_e values, demonstrating that pseudo-second-order model is competent to describe the adsorption kinetics of Se.

In addition, the intra-particle diffusion model was also employed to determine the rate-determining step in Se adsorption process. As shown in Fig. S5, the Se adsorption process can be divided into three linearity plots, i.e. external transfer step, intra-particle diffusion step and final equilibrium step⁴⁻⁶; and the plots of q_t against $t^{1/2}$ are straight lines passing through the origin, indicating that the rate-determining step is pore diffusion rather than boundary layer diffusion^{2,4}.

$$\frac{1}{q_t} = \frac{\kappa_1}{q_{e1}t} + \frac{1}{q_{e1}} \\ t = 1 \qquad t$$
(1)

$$\frac{1}{q_t} = \frac{1}{(k_2 q_{e2}^2)} + \frac{1}{q_{e2}}$$
(2)

$$q_t = k_{id} t^{1/2} + I \tag{3}$$

where k_1 and k_2 are the pseudo-first-order constant and pseudo-second-order constant, respectively; R^2 is the linear correlation coefficient; $q_e (mg \cdot g^{-1})$ represents the adsorption capacity of the adsorbent at equilibrium; $q_t (mg \cdot g^{-1})$ is the adsorption capacity of the adsorbent at time t; $K_{id} (mg \cdot g^{-1} \cdot min^{-1/2})$ denotes the intra-particle diffusion rate constant; and I is the intercept of the intra-particle diffusion model.

Text S3. Adsorption Isotherm

To further determine the adsorption capacity of Se, the adsorption isotherms were established according to the Langmuir and Freundlich isotherm models, with the initial concentration of Se ranging from 0 to 200 mg/L. Table S4 summarizes the parameters of Langmuir and Freundlich isotherms. As shown in Fig. S6, Se adsorption capacity increases constantly with the increase in equilibrium concentration (C_e), and reaches saturation in the end, with the maximum adsorption capacity of higher than 120 mg/g. Moreover, the R² of Freundlich model is higher than that of Langmuir model, so Se adsorption process follows the Freundlich model. Herein, the fitting results of kinetic and isotherm adsorption curves suggested that the mechanism of Se adsorption was involved with chemisorption on multilayers⁵.

$$q_e = \frac{q_{max} K_L C_e}{1 + K_L C_e} \tag{4}$$

$$q_e = K_F C_e^{1/n} \tag{5}$$

where C_e (mg·L⁻¹) represents the equilibrium concentration of Se in solution; q_e (mg·g⁻¹) is the amount of adsorbed Se per unit mass of the 70 wt% OV-CoO/CeO₂ composite material; K_L (L·mg⁻¹) denotes Langmuir affinity constant related to the energy of adsorption; q_{max} (mg·g⁻¹) is the maximum adsorption capacity; K_F (mg/g·(L/mg)^{1/n}) represents the Freundlich constant related to the adsorption affinity; and n is a dimensionless indicator related to the adsorbent surface heterogeneity.

The Dubinin-Radushkevich (D-R) isotherm model was used to determine whether the nature of the process of adsorption is physical or chemical^{7, 8}. The equation of the D-R isotherm is expressed by eq (6).

$$lnq_e = lnQ_0 - \beta \varepsilon^2 \tag{6}$$

Where $q_e (mg \cdot g^{-1})$ is the amount of adsorbed Se per unit mass of the OV-CoO/CeO₂ composite material; Q_0 (mg · g¹) denotes the maximum adsorption capacity; β represents the activity coefficient related to the mean free energy of adsorption (mol² /J²); and ε is the Polanyi potential ($\varepsilon = RTln(1 + 1/C_e)$). The calculated q_m and β are listed in Table S5. The mean free energy of adsorption E (kJ/mol) is expressed by eq (7):

$$E = \frac{1}{\sqrt{2\beta}} \tag{7}$$

According to the former reports, the adsorption mechanism can be determined by the value of E. When the vales of E between 8 to 16 kJ/mol, the process of adsorption belongs to chemical adsorption⁹.

In HSDM, the adsorbent particle is assumed as a sphere which is surrounded by a stagnant liquid boundary layer, and the adsorbed adsorbate molecules are supposed to transport through diffusion on the surface of pore within the adsorbent particles¹⁰. In addition, during the migration process, it is assumed that a local equilibrium is established instantaneously between the adsorbate in the pore fluid and the adsorbate on the pore surface¹¹. The rate data can be used to calculate the C data value at different time t to determine the surface diffusion coefficient, which is expressed as eq(8):

$$C_{\text{data}} = \frac{Ct - Ce}{C0 - Ce}$$
(8)

The value of t-bar can be calculated by the eq(9):

$$t_{bar} = D_s \frac{t}{R^2} \tag{9}$$

The Excel solver is used to find the optimal Ds/R^2 value by minimizing the objective function as shown in the eq(10):

$$OF = \sqrt{\frac{\sum_{i=1}^{n} (\frac{Cdata, i - Cmodel, i}{Cdata, i})^{2}}{n-1}}$$
(10)

The equation of the general empirical relationship is expressed by eq(11), which expresses the dimensionless concentration as a function of dimensionless time.

$$C=A_0 + A_1 (\ln t) + A_2 (\ln t)^2 + A_3 (\ln t)^3$$
(11)

According to Freundlich model, the Freundlich constant related to the adsorption affinity $K_{\rm F}$ $({\rm mg/g}\cdot({\rm L/mg})^{1/n})$ is 10.45 and the dimensionless indicator related to the adsorbent surface heterogeneity 1/n

Text S4

In this study, the turnover frequency (TOF (s⁻¹)) was used to measure the amount of oxidized sulfite per mol of Co atom per second¹. The amount of active substance in this study was determined by the content of active component in catalyst. The TOF was calculated as follows:

$$TOF = \frac{n_{S(VI)}}{n_{active} \times t} \tag{12}$$

Where $n_{S(VI)}$ (mol) is the amount of sulfate in oxidation products; n_{active} (mol) is the content of metal active component on catalyst and t (s) is the reaction time.

Text S5

The material cost in the reported literatures was calculated as follows:

$$C = \frac{\sum_{i=1}^{n} m_{precursor, i} \times c_{precursor, i}}{m_{adsorbent} \times q_{e}}$$
(13)

Where $C(\$\cdot t^{-1})$ is cost of adsorbent required for each ton of Se; *i* is one of the precursors contained in the material; $m_{\text{precursor}}$ (g) is the required mass of the precursor; $c_{\text{precursor}}(\$\cdot t^{-1})$ is the price per ton of the precursor; $m_{\text{adsorbent}}$ (g) is the mass of the prepared adsorbent; q_e (mg·g⁻¹) is the amount of adsorbed Se per unit mass of the adsorbent material.

Text S6

The synergistic catalytic/adsorption experiments were performed in simulated WFGD slurry to investigate the S/Se selectivity of OV-CoO/CeO₂. The S selectivity (S_s) and Se selectivity (S_{Se}) are calculated as follows:

$$S_S = \frac{TOF_{SW}}{TOF_{C/A}} \tag{14}$$

Where TOF_{SW} is the turnover frequency of OV-CoO/CeO₂ for SO₃²⁻ oxidation in simulated WFGD slurry with impurities. $TOF_{C/A}$ is the turnover frequency of OV-CoO/CeO₂ for SO₃²⁻ oxidation under simultaneous SO₃²⁻ catalytic oxidation and Se adsorption in pure environment.

$$S_{S_e} = \frac{R_{SW}}{R_{C/A}} \tag{15}$$

Where R_{SW} is the Se removal efficiency of OV-CoO/CeO₂ in simulated WFGD slurry with impurities. $R_{C/A}$ is the Se removal efficiency of OV-CoO/CeO₂ under simultaneous SO₃²⁻ catalytic oxidation and Se adsorption in pure environment.





Fig. S1. XRD patterns of CoO, OV-CoO, CeO₂, OV-CeO₂, CoO/CeO₂ and OV-CoO/CeO₂ catalysts.



Fig. S2. Kinetics and efficiencies of sulfite oxidation by using CoO, CeO₂, CoO/CeO₂ materials with and without OV. General conditions: $c_{cat} = 0.5$ g/L, $c_{(NaSO3)} = 2$ g/L, $P_{O2} = 0.21$ atm, T = 318 K, and pH = 8.0.



Fig. S3. Pseudo-first-order kinetics fitting curve for selenium adsorption.



Fig. S4. Pseudo-second-order kinetics fitting curve for selenium adsorption.



Fig. S5. Intra-particle diffusion model fitting curve for selenium adsorption.



Fig. S6. Langmuir and Freundlich isotherm fitting parameters models for Se adsorption on OV-CoO/CeO₂.



Fig. S7. Dubinin-Radushkevich model parameters for Se adsorption onto 70 wt% OV-CoO/CeO₂.



Fig. S8. Experimental data and HSDM prediction calculated by using Ds.



Fig. S9. SEM images and elemental mapping of the used $OV-CoO/CeO_2$



Fig.S10. (a) Pore diameter distribution of the used OV-CoO/CeO $_2$ (b) Nitrogen adsorption-desorption

isotherms of the used OV-CoO/CeO₂



Fig. S11. Comparison of Co and Ce losses in sulfite oxidation by using CoO, CeO₂, CoO/CeO₂.



Fig. S12. Effects of coexisted toxic metals on selenium removal efficiency. $c_{\rm Cr} = 5 \text{ mg/L}$, $c_{\rm Cu} = 0.5 \text{ mg/L}$, $c_{\rm Hg}$

= 0.2mg/L, $c_{\rm Pb} = c_{\rm Se}$ 1mg/L, $c_{\rm Cd} = 0.1$ mg/L.



Fig. S13. Effect of Hg on Se adsorption



Fig. S14. Catalytic/adsorption performance of OV-CoO/CeO₂ in simulated WFGD wastewater



Fig. S15. M-H hysteresis curves of the 70 wt% OV-CoO/CeO₂ catalyst.



Fig. S16. Nitrogen adsorption-desorption isotherms of the different materials.



Fig. S17. Raman spectra of CoO, CeO₂, CoO/CeO₂ materials with and without OV.



Fig.S18. Comparison of the TPD profiles between used OV-CoO/CeO₂ and fresh OV-CoO/CeO₂



Fig. S19. Comparison of the Raman spectra between used OV-CoO/CeO₂ and fresh OV-CoO/CeO₂



Fig. S20. Se 3d XPS spectra of OV-CoO/CeO₂ after Se adsorption.

Tables

Catalysts	Catalyst	Co concentration	Oxidation rate of		Dofononace	
	dosage (g·L ⁻¹)	(wt.%)	SO ₃ ²⁻ (mmol·L ⁻¹ ·s ⁻¹)	10r (s ⁻)	Kerer ences	
Mn@ZIF67	0.50	18.07	0.077	0.014	12	
C0/C3N4	0.50	24.10	0.074	0.010	13	
Co-MOF-74	0.50	29.59	0.081	0.009	14	
Co ₃ O ₄ -NPs@KIT-6	1.00	4.00	0.0902	0.038	15	
BISC	1.00	13.32	0.1446	0.018	16	
Co-MCM-48	0.50	5.90	0.074	0.042	17	
OV-CoO/CeO ₂	0.25	24.02	0.174	0.050	This work	

Table S1 Comparison of the TOF for other Co-based catalysts

					Composites		
		CoO	OV-CoO	CeO ₂	OV-CeO ₂	CoO/CeO ₂	OV-CoO/CeO ₂
Pseudo-	$q_{e, cal}(mg \cdot g^{-1})$	3.004	3.561	3.034	3.010	7.389	9.974
first	k ₁ ×10 ² (min ⁻¹)	1.021	1.149	0.959	0.765	0.873	1.102
-order	R ²	0.907	0.934	0.890	0.676	0.855	0.890
Daoudo	$q_{e, cal}(mg \cdot g^{-1})$	6.024	7.936	14.925	16.949	24.570	33.333
Pseudo- second	$k_2 \times 10^{-2} (g \cdot (mg \cdot min))^{-1}$	1.664	2.633	2.747	4.455	0.899	0.734
-order	R ²	0.997	0.999	0.999	0.999	0.999	0.999
	$k_1(mg \cdot g^{-1} \cdot min^{-1/2})$	1.046	3.002	8.458	9.423	6.994	11.323
Intra-	R ²	0.981	0.936	0.990	0.954	0.999	0.960
Particle	$k_2(mg \cdot g^{-1} \cdot min^{-1/2})$	0.533	0.495	0.359	0.526	1.063	2.277
diffusion	R ²	0.990	0.985	0.891	0.986	0.943	0.997
model	$k_3(mg \cdot g^{-1} \cdot min^{-1/2})$	0.099	0.092	0.080	0.028	0.250	0.240
	R ²	0.822	0.887	0.849	0.640	0.705	0.955

 Table S2. Parameters of adsorption kinetics for Se adsorption.

Adsorbents	Adsorbent dosage (g·L ⁻¹)	Materials cost estimation (\$·t ⁻ ¹)	Adsorption capacity (mg·g ⁻¹) for Se	Adsorbent cost required for each ton of Se (\$•t ⁻¹)	Reference
CuFe ₂ O ₄	0.40	5344.8	14.10	379063.8	17
CoFe ₂ O ₄	0.40	9699.9	11.60	836198.3	17
MnFe ₂ O ₄	0.40	2842.9	3.90	728948.7	17
BC-ZVI	0.50	24757.9	62.52	395999.7	18
MGO	1.00	8002817	23.81	336111591.8	19
UiO-66-NH ₂	0.50	122916.6	26.80	4586440.3	20
MGO-PAA	0.015	17842285.1	120.10	148561907.6	21
OV-CoO/CeO ₂	0.25	22767.4	126.70	179695.3	This work

Table S3. Comparison of adsorbents for Se in reported literatures

Table S4. Langmuir and Freundlich isotherm fitting parameters for Se adsorption on OV-CoO/CeO₂.

~		Langmuir	Freundlich				
Composite	$q_m(mg \cdot g^{-1})$	Actual q _m (mg·g ⁻¹)	$K_L(L \cdot mg^{-1})$	R ²	$K_F(mg^{1-} (^{(1/n)}L^{1/n} g^{-1})$	R ²	n
OV-CoO/CeO ₂	175.454	126.701	0.016	0.971	10.452	0.992	2.021

Table S5. Dubinin-Radushkevich isotherm fitting parameters for Se adsorption on OV-CoO/CeO₂.

Composito -	D-R model							
Composite	$Q_{ heta}(\mathrm{mol}{\cdot}\mathrm{g}^{-1})$	β (mol ² ·J ⁻²)	E (kJ·mol⁻¹)	R ²				
OV-CoO/CeO ₂	6.090E-3	4.916E-9	10.085	0.992				

Table S6. Parameters used for the empirical equation that describe solutions to the HSDM for a batch

	HSDM		Empirical Eq	uation C	$= A_0 + A_1$	(ln t)+A ₂	$(\ln t)^2 + A_3$	(lnt) ³
Composite	$\mathrm{K_L}(\mathrm{mg}\cdot\mathrm{g}^{-1})\cdot(\mathrm{L}\cdot\mathrm{mg}^{-1})^{1/n}$	D _s (cm ² ·s ⁻ ¹)	Freundlich adsorption intensity parameter (1/n)	Ce/C ₀	\mathbf{A}_0	A ₁	\mathbf{A}_2	\mathbf{A}_3
OV-CoO/CeO ₂	10.45	4.17E-13	0.4948	0.1	0.1139	0.1536	3.53E-3	5.30E-5

reactor for Freundlich adsorption intensity parameters (1/n)

t/min	Ct/C0	Ct	t-bar	$C data = \frac{Ct - Ce}{C0 - Ce}$	C model	(Cdata – Cmodel)2 Cdata
5	0.595408529	5060972.5	2.10E-03	0.557054675	0.889025371	3.55E-01
10	0.551634118	4688890	4.19E-03	0.509130603	0.644416766	7.06E-02
15	0.529052353	4496945	6.29E-03	0.484408166	0.519404777	5.22E-03
20	0.453954118	3858610	8.39E-03	0.402190881	0.438890893	8.33E-03
30	0.365555588	3107222.5	1.26E-02	0.305412481	0.337050066	1.07E-02
60	0.309836471	2633610	2.52E-02	0.244411386	0.194835346	4.11E-02
90	0.249755	2122917.5	3.77E-02	0.178634402	0.130519284	7.25E-02
120	0.205196176	1744167.5	5.03E-02	0.129851558	0.093427673	7.87E-02
150	0.174182941	1480555	6.29E-02	0.095898377	0.069574592	7.53E-02
180	0.151241765	1285555	7.55E-02	0.070782457	0.053291431	6.11E-02
210	0.133872647	1137917.5	8.80E-02	0.051766808	0.041783463	3.72E-02

 Table S7. Calculating Ds using mathematical solution to the HSDM

Parameter	Typical influent range (mg·L ⁻¹)	References
Cadmium (Cd)	0.05-0.1	23, 24
Chromium (Cr)	0.3-1	24, 25
Mercury (Hg)	0.01-0.8	26, 27
Lead (Pb)	0.5-1.5	25
Copper (Cu)	0.2-0.8	28
Magnesium (Mg)	50-4000	28
Chloride (Cl)	10000-25000	29
Nitrate (NO ₃)	30-120	26
Selenium (Se)	1-4	24, 26, 29

Table S8. Flue Gas Desulfurization Wastewater Characteristics

Catalysts	Average Pore Diameter (nm)	Pore Volume (cm ³ /g)	BET Surface Area (cm²/g)
CoO	21.348	0.067	12.638
OV-CoO	15.958	0.0428	10.729
CeO ₂	12.227	0.174	57.032
OV-CeO ₂	12.959	0.231	71.215
70wt% CoO/CeO2	8.771	0.123	56.206
70wt% OV-CoO/CeO ₂	9.232	0.137	59.175

Table S9. Pore diameter, pore volume and BET surface area of the different materials.

Gaurala	Co 2p		Ce 3d		O 1s		
Sample	Co ²⁺ /Co	Co ⁰ /Co	Ce ³⁺ /Ce	Ce ⁴⁺ /Ce	O_{α}/O	O_{β}/O	O_{Γ}/O
10 wt% OV-CoO/CeO ₂	87.42%	12.58%	21.27%	78.73%	44.13%	47.13%	8.74%
30 wt% OV-CoO/CeO ₂	87.40%	12.60%	24.87%	75.13%	33.78%	52.52%	13.70%
50 wt% OV-CoO/CeO ₂	87.05%	12.95%	29.00%	71.00%	24.71%	59.52%	15.77%
70 wt% OV-CoO/CeO ₂	87.66%	12.33%	40.77%	59.23%	22.33%	67.48%	10.62%
90 wt% OV-CoO/CeO ₂	80.41%	19.69%	20.69%	79.31%	32.63%	55.74%	11.63%

Tabla S11	Ratios of Co	Ce and Osn	ecies on the s	$urface of OV_{-}$	CoO/CeO. befor	e and after
Table SIT	. Ratios of Co	, Ce, and O sp	ectes on the s	urface of OV-V	COU/COU_2 befor	e and after

Sample	Co 2p		Ce 3d		O 1s		
	Co ²⁺ /Co	Co ⁰ /Co	Ce ³⁺ /Ce	Ce ⁴⁺ /Ce	Ο _α /Ο	O_{β}/O	O_{Γ}/O
OV-CoO/CeO ₂	87.66%	12.33%	40.77%	59.23%	22.33%	67.48%	10.62%
OV-CoO/CeO ₂ after catalysis	93.63%	6.37%	38.71%	61.29%	35.77%	64.54%	0.69%
OV-CoO/CeO ₂ after adsorption	90.04%	9.96%	34.45%	65.56%	50.05%	47.09%	2.86%

catalysis/adsorption.

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