

Supporting Information for

Spontaneous adsorption-oxidation of gaseous elemental mercury via a conjugated unit -NH⁺-Cl^{*}: creation and mechanisms

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24 1. All reagents and materials

25 The following reagents and materials are used in the synthesis process: polypropylene non-woven
26 fabric (80g/m², Jinshengchang Guangdong China), multi-wall carbon nanotubes (Carboxylated MWCNTs,
27 XFM21, XFNANO Nanjing China), aniline (AR, 99.5%, Macklin, 1.02g/mL), polyvinylidene fluoride
28 (PVDF, FR905, Sanaifu Neimenggu China), N-methyl pyrrolidone (NMP, AR, 99%, Macklin), sodium
29 dodecyl benzene sulfonate (SDBS, AR, 90%, Kermel Tianjin China), (NH₄)₂S₂O₈ (AR, 98%, Damao Tianjin
30 China), HCl (AR, 36~38%, Damao Tianjin China), H₂SO₄ (AR, 95~98%, Damao Tianjin China), NaCl (AR,
31 99.5%, Kermel Tianjin China), ethanol absolute (AR, 99.7%, Huihang Tianjin China), pyrrole (AR, 99%,
32 Macklin, 0.97g/mL) and deionized water. The reagents in Hg⁰ removal experiment and recovery: KMnO₄
33 (AR, 99.5%, Kermel Tianjin China), H₂SO₄ (AR, 95~98%, Damao Tianjin China), SnCl₂·2H₂O (AR, 98%,
34 Kermel Tianjin China), NaCl (AR, 99.5%, Kermel), HCl (AR, 36~38%, Damao Tianjin China).

35 2. Synthesis procedure of 11PANI and 51PANI

36 The fabrication of 11PANI and 51PANI: 5 ml aniline and 0.16 ml HCl were added to 61 ml deionized
37 water. Simultaneously, 12.52 g $(\text{NH}_4)_2\text{S}_2\text{O}_8$ was dissolved in 84 ml deionized water. Then, the latter was
38 slowly poured into the former in ice bath. Next, the product was washed to neutral after 2 h. Finally, it was
39 dried in vacuum drying oven at 60 °C for 12 h. The quality of $(\text{NH}_4)_2\text{S}_2\text{O}_8$ in the fabrication of 51PANI is
40 fifth times that in the fabrication of 11PANI. 0.2 g 11PANI was dispersed into 100 ml deionized water.
41 11PANI⁺ (pH=0 or 1) was generated by immersion in H_2SO_4 , Cl-11PANI was produced by immersion in 1
42 mol/L NaCl. Cl-11PANI⁺ (pH=0 or 1) was produced by immersion in 1 mol/L NaCl at pH=0 or 1. The
43 immersion time is 60 min and then the samples were dried in vacuum drying oven at 60 °C for 12 h.

44

3. Fabrication of the Composite Film

Figure S2 shows the fabrication process of composite film, including vacuum filtration, immersion and phase inversion methods. All reagents are listed in Text S1. Firstly, polyvinylidene fluoride (PVDF) and aniline were dissolved in N-methyl-pyrrolidone (NMP) solution under the condition of stirring and heating at 60 °C. And MWCNTs were dispersed in deionized water via ultrasonic treatment (ATPIO-1200D ATPIO Co., Nanjing China) at 720W for 5 min. Then, the dispersion was attached onto the surface of polypropylene non-woven fabric (NWF) by vacuum filtration method to form a thin framework layer (a filter paper was placed under NWF for retaining MWCNTs on NWF). Next, the film was immersed in the above NMP solution for 2 h. Then the film, containing about 2ml NMP solution, was picked up, and then immediately immersed into a 20ml mixture solution containing 1 mol/L H_2SO_4 , 3mg/ml sodium dodecyl benzene sulfonate (SDBS) and 0.45g $(\text{NH}_4)_2\text{S}_2\text{O}_8$ (the molar ratio of $(\text{NH}_4)_2\text{S}_2\text{O}_8$ and aniline was 1:1), where the phase inversion of PVDF and the polymerization induced by $(\text{NH}_4)_2\text{S}_2\text{O}_8$ would take place simultaneously. Finally, the film was washed by deionized water and absolute ethanol, and dried in a vacuum drying oven (DZF-6020 Keelrein Co., Shanghai China) for 12 h at 60 °C. Accordingly, the film was marked as 11PANI@MWCNTs. The molar ratio of $(\text{NH}_4)_2\text{S}_2\text{O}_8$ and aniline will be 5:1 when 51PANI@MWCNTs is synthesized. Before the Hg^0 removal experiment, the film needed to be immersed in Cl-containing solution at pH=1 for 60 min and then dried, marked as Cl-PANI⁺@MWCNTs. If without specific instructions, Cl-11PANI⁺@MWCNTs is obtained via immersing 11PANI@MWCNTs into 1 mol/L NaCl at pH=1 for 60 min and drying.

64

65 4. Regeneration and recycling

66 To assess the reusability of the composite film, we carried out recycling tests after the film regeneration,
67 at the gas flow of 1 L/min, about 200,000 h⁻¹. The regeneration of the composite membrane was realized
68 through following procedure: the spent film was first immersed in a mixed solution of 1 mol/L NaCl at
69 pH=1 (adjusted by H₂SO₄) for 60 min and then was dried. In the 5 cycles, the same solution is reused. In
70 order to determine the proportion of mercury in the solution after 5 cycles, we used AFS to determine the
71 mercury concentration in the leaching solution. As we all know, mercury balance is crucial for this test, thus
72 we first determined the amount of Hg⁰ discharged from mercury osmotic tube under a certain interval of 60
73 min, with 10% (v/v) H₂SO₄-4% (w/w) KMnO₄ solution as the capturing solution. The result indicated that
74 the average output mass of Hg⁰ (5 parallel tests) was approximately 15.1 µg in 60 min. Afterwards, in every
75 cycle, Hg⁰ removal experiment was terminated when the outlet concentration is more than 5% of the inlet
76 concentration and then leached in acidic NaCl solution for 60 min. The mercury concentration in the
77 leaching solution was finally determined by AFS, from which the quantity of the recovered mercury was
78 obtained.

79

80 5. Characterization and DFT Calculation Methods

81 The morphology of the composite film was observed by a field emission scanning electron microscopy
82 (FESEM, Hitachi-s4800). Energy-dispersive spectroscopy (EDS) was recorded by a Bruker quantax400
83 attached to the Hitachi-s4800 FESEM instrument to determine the elemental distribution of the composite
84 film. The specific surface area (S_{BET}) and pore volume (V_{p}) were calculated by applying Brunauer-Emmett-
85 Teller (BET) and Barrett-Joyner-Halima (BJH) models to the N_2 adsorption-desorption isotherms. The
86 ultraviolet-visible spectra (UV-Vis) of polyaniline in different states were recorded by a UV-Vis
87 spectrometer (TU-1901, Persee Co., Beijing China). And the surface functional groups were identified by a
88 Fourier transform infrared spectrometer (FTIR, Nicolet iZ10). To determine the difference of every
89 elemental valence in the composite film before and after the experiment, the binding energies of C 1s, O 1s,
90 N 1s, F 1s, Cl 2p and Hg 4f were analyzed with X-ray photoelectron spectroscopy (XPS) (ESCALAB250
91 spectrometer with Al $K\alpha$ source (1486.6 eV)). Density functional theory (DFT) is widely used to calculate
92 the ground-state electronic structure of atoms, molecules, and solid-state materials, so the DFT calculations
93 were also performed to clarify the reaction mechanism by using Gaussian 09 software.¹ The def2SVP was
94 used to optimize the structures of molecules and calculate the single point energies based on the M062X in
95 open shell with DFT-D3(BJ) and ultrafine integral grid.^{2,3} And all data about model construction and
96 optimization are shown in Table S4.

97

98 **6. UV-Vis analyses procedure**

99 In the UV-Vis experiment, the pure 11PANI and 51PANI were used, due to the required soluble
100 samples, but MWCNTs is insoluble in NMP. The UV-Vis analysis procedure is as follows: NMP was used
101 as reference solution. The above samples, synthesized in Text S2, were dissolved into NMP and all the
102 concentrations were 0.05 g/L. The ultraviolet-visible spectrophotometer (TU-1901, Persee, Beijing China)
103 was used to scan the absorption spectra of the samples in the range of 190-900 nm.

Figures

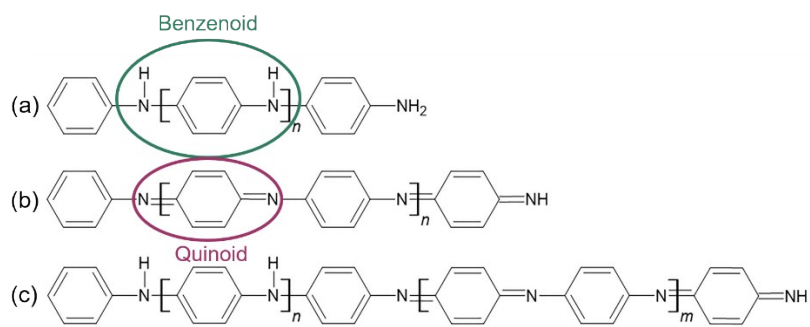


Figure S1. The molecular structures of (a) leucoemeraldine, (b) pernigraniline bases, (c) emeraldine.

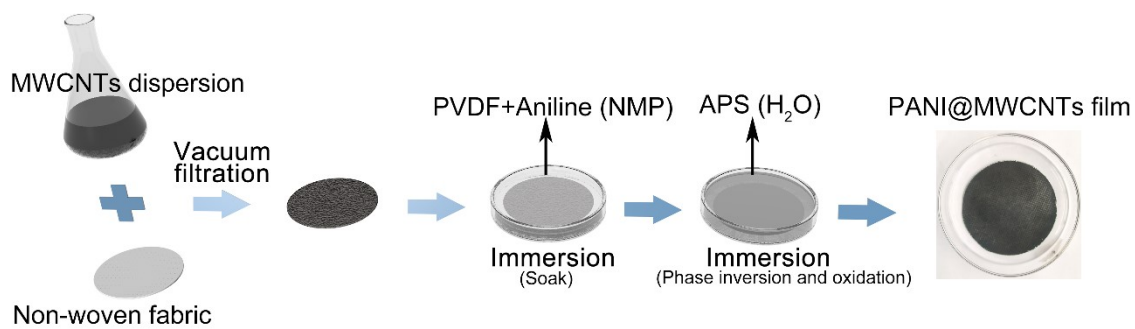


Figure S2. Fabrication procedures of the PANI@MWCNTs film.

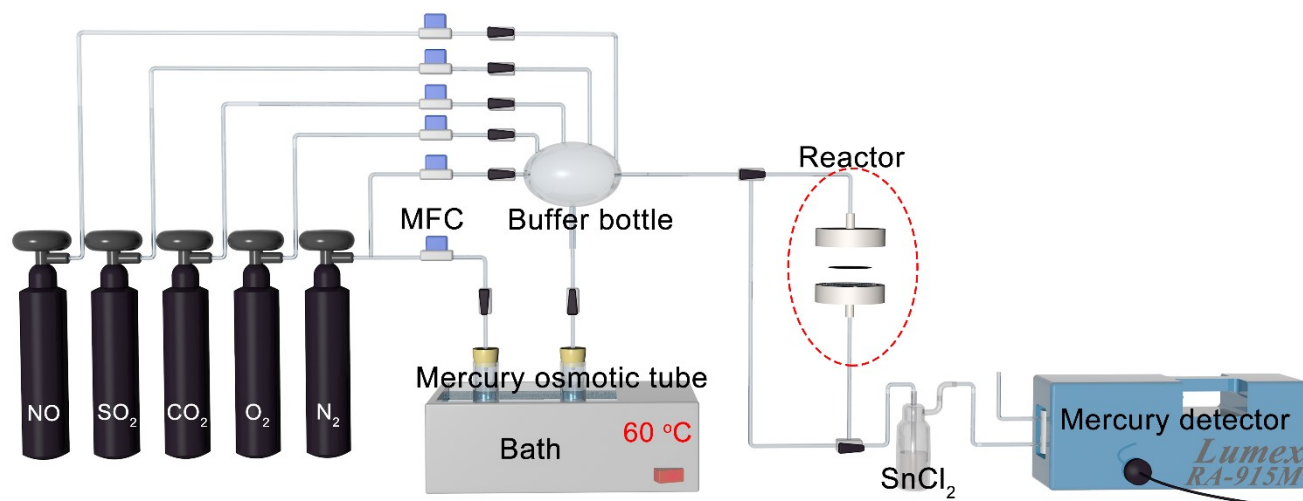


Figure S3. Scheme of Hg^0 removal experiment.

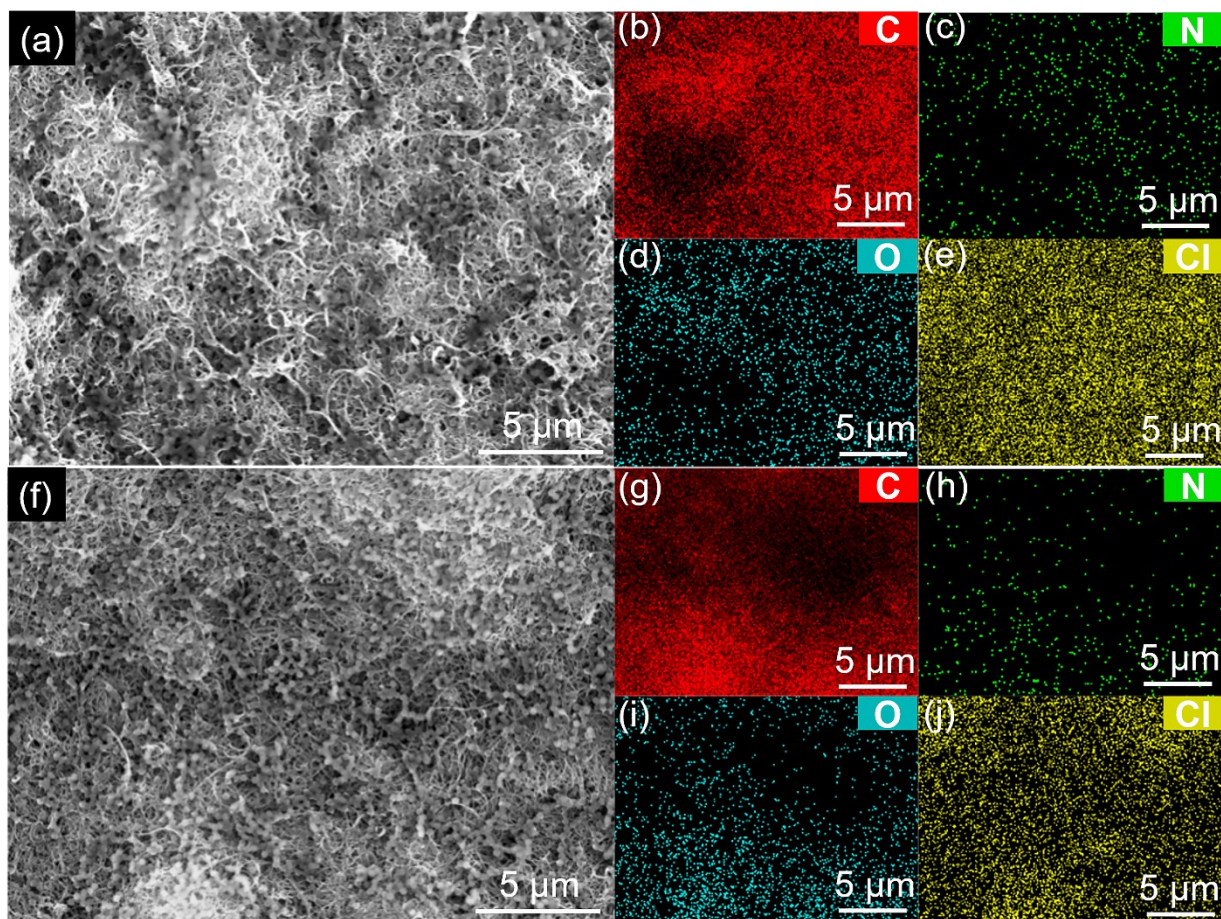


Figure S4. EDS and element mapping of the (a)-(e) Cl-11PANI⁺@MWCNTs and (f)-(j) Cl-51PANI⁺@MWCNTs film.

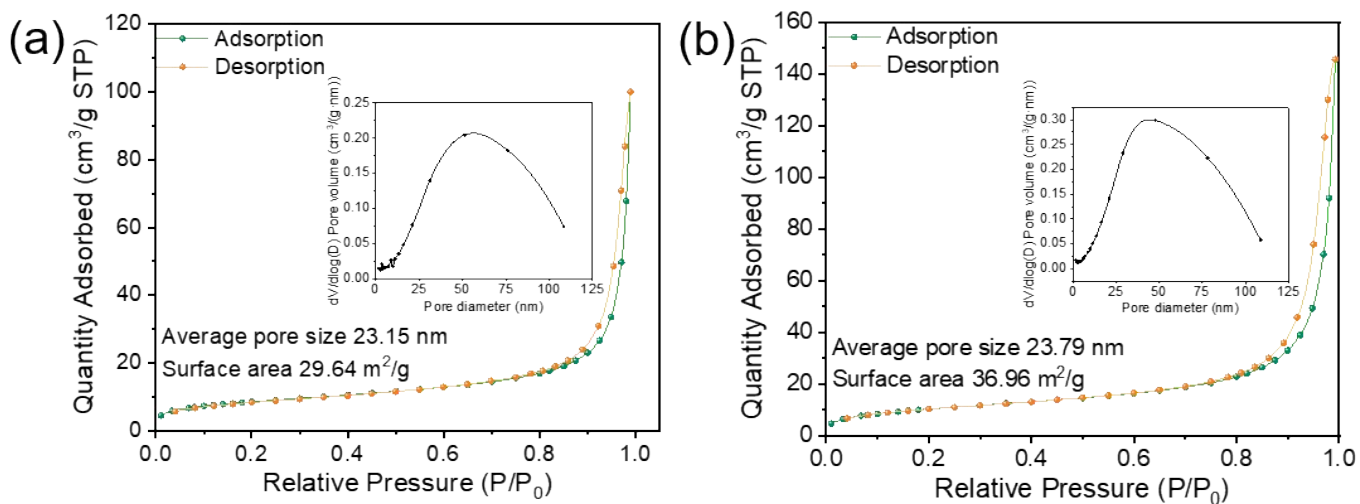


Figure S5. the BET results and BJH desorption pore diameter distribution of (a) Cl-11PANI⁺@MWCNTs and (b) Cl-51PANI⁺@MWCNTs

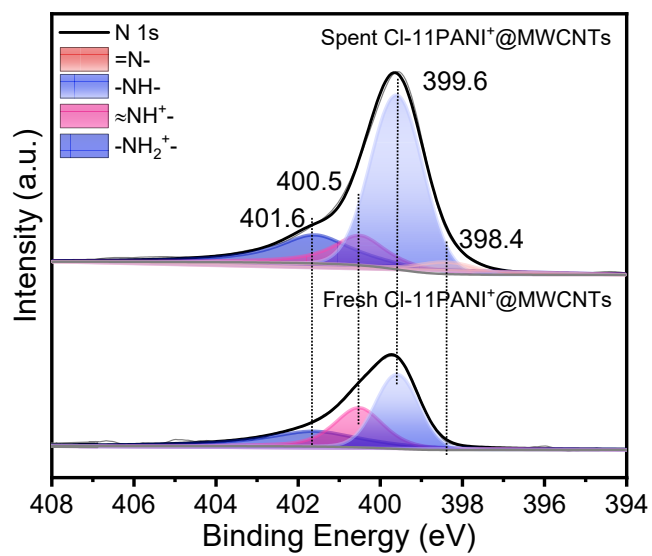


Figure S6. High-resolution N 1s XPS spectra of the fresh and spent Cl-11PANI⁺@MWCNTs

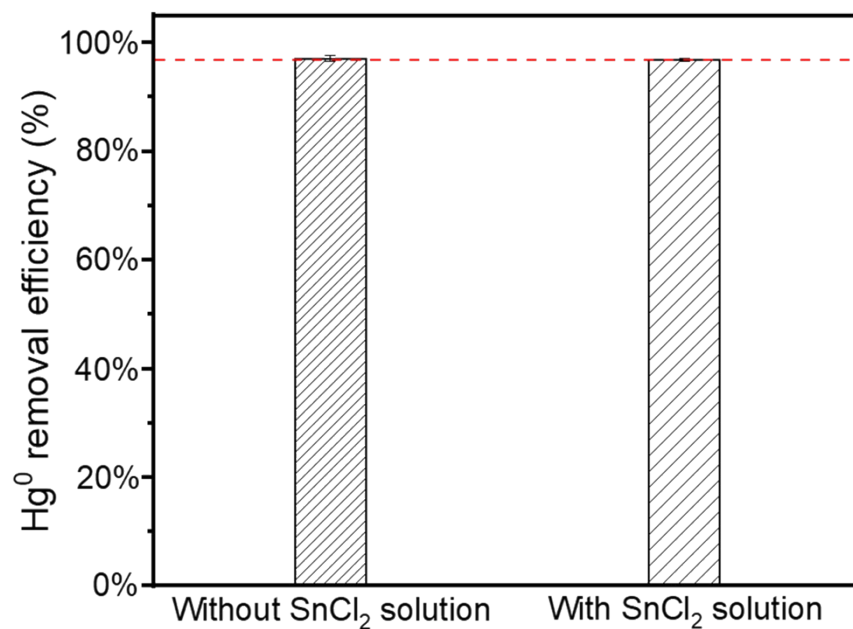


Figure S7. The Hg⁰ removal efficiencies with and without SnCl₂ solution (Cl-11PANI⁺@MWCNTs and the space velocity is 200,000 h⁻¹)

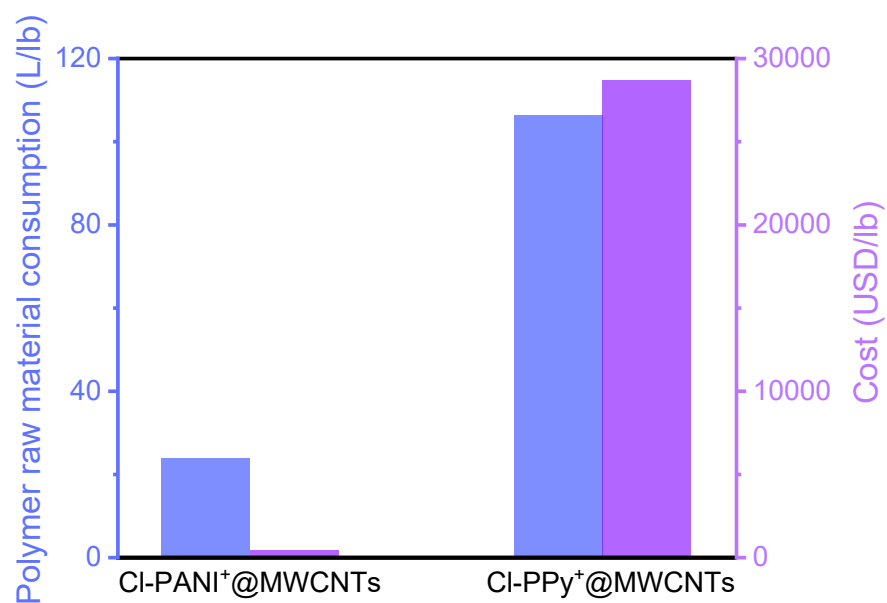


Figure S8. The polymer raw material consumption and cost.

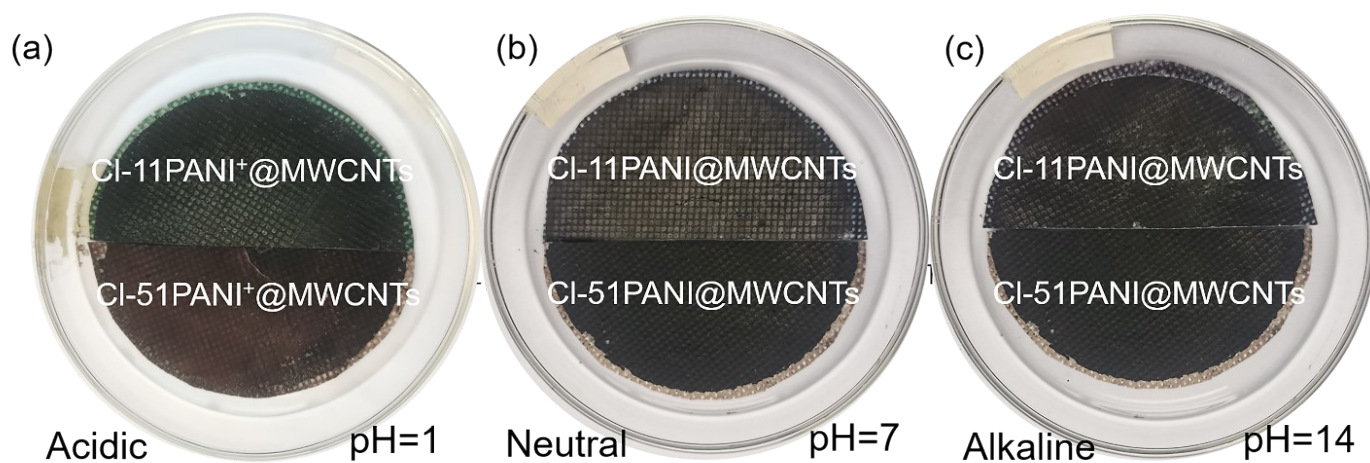


Figure S9. The macroscopic images of CI-11PANI⁺@MWCNTs and CI-51PANI⁺@MWCNTs at different pH.

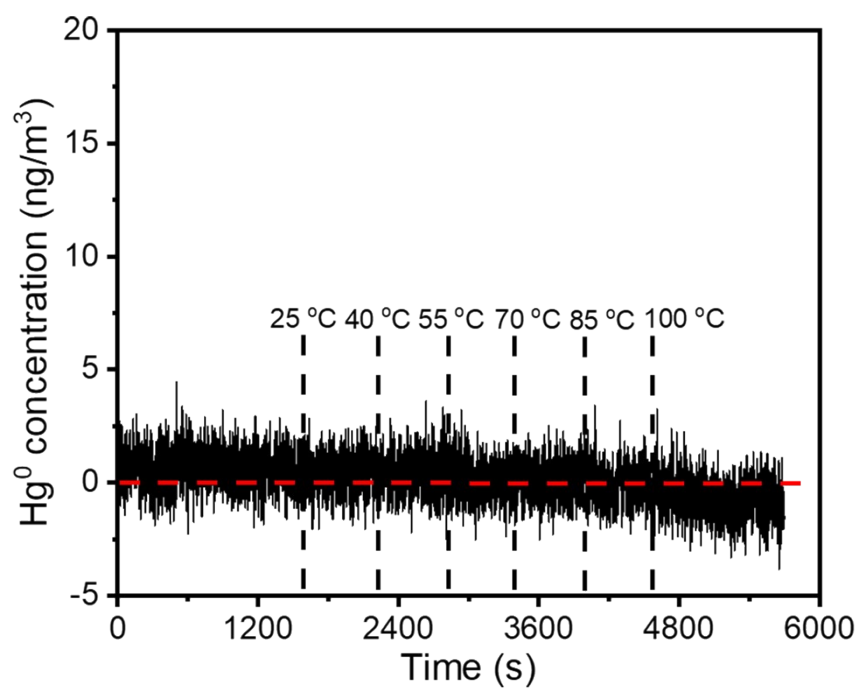


Figure S10. Stability of adsorbed mercury on the composite membrane under different temperatures. The Cl-11PANI⁺@MWCNTs film was pretreated via gaseous Hg⁰ for 1 h, concentration unit is ng/m³, space velocity is 200,000 h⁻¹.

Table S1. Abbreviations in the work.

Abbreviated title	Title
MWCNTs	Multiwall carbon nanotubes
PANI	Polyaniline, and aniline is its monomer
11PANI	PANI synthesized in the molar ratio of (NH ₄) ₂ S ₂ O ₈ to aniline is 1:1
51PANI	PANI synthesized in the molar ratio of (NH ₄) ₂ S ₂ O ₈ to aniline is 5:1
11PANI ⁺	Protonated 11PANI
51PANI ⁺	Protonated 51PANI
Cl-11PANI ⁺	Cl-doped protonated 11PANI
Cl-51PANI ⁺	Cl-doped protonated 51PANI
11PANI@MWCNTs	11PANI coated MWCNTs
51PANI@MWCNTs	51PANI coated MWCNTs
11PANI ⁺ @MWCNTs	Protonated 11PANI@MWCNTs
51PANI ⁺ @MWCNTs	Protonated 51PANI@MWCNTs
Cl-11PANI ⁺ @MWCNTs	Cl-doped protonated 11PANI@MWCNTs
Cl-51PANI ⁺ @MWCNTs	Cl-doped protonated 51PANI@MWCNTs
Cl-PPy@MWCNTs or Cl-PPy ⁺ @MWCNTs	Cl-doped protonated polypyrrole coated multiwall carbon nanotubes

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Table S2 The element ratio of different films.

Materials	C (Wt%)	O (Wt%)	N (Wt%)	Cl (Wt%)	Others (Wt%)
Cl-11PANI ⁺ @MWCNTs	86.0	3.0	2.1	5.4	3.5
Cl-51PANI ⁺ @MWCNTs	92.0	3.2	1.2	1.9	1.7

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Table S3 Paper summary about the method for gaseous Hg⁰ removal recently.

No.	Materials	Theory	Experimental conditions	Efficiency	adsorption capacity	Breakthrough point (output/input)	Cycle	Publication	Reference
1	MnO _x /Grap hene	Adsorption and oxidation	- 0.5 L/min - 100-300 °C	>90%	2.7mg/g	10h	good	EST/2015,49 ,6823-6830	(4)
2	LaMnO ₃	Catalytic oxidation and adsorption	500 µg/m ³ 0.5 L/min 478000 h ⁻¹ 150 °C	>85%	6.22mg/g	50%	≥5	ACB/186(20 16)30-40	(5)
3	CoMoS/γ- Al ₂ O ₃	Adsorption	30 µg/m ³ 1.5 L/min 45000 cm ³ /(g·h) 25-450 °C	100% (50 °C)	18.95 mg/g	100% Elovich model	-	EST/2016,50 ,1056-1064	(6)
4	IrO ₂ /Ce _{0.6} Z r _{0.4} O ₂	Catalytic oxidation and adsorption	370-1500 ng/m ³ 0.5L/min 760000 h ⁻¹ 350 °C	>99%	-	-	good	EST/2016,50 ,2564-2572	(7)
5	Sawdust- Fe ³⁺	Adsorption and oxidation	85 µg/m ³ 1.2 L/min - 120-250 °C	>90%	1.28 mg/g	100% Model simulation	-	EST/2016,50 ,12040- 12047	(8)
6	nano-ZnS	Adsorption	65 µg/m ³ 1 L/min - 140-260 °C	>90%	497.84 µg/g	50%	-	EST/2016,50 ,9551-9557	(9)

7	[MoS ₄] ²⁻ / CoFe-LDH	Physical adsorption	350 µg/m ³	>95%	16.39 mg/g	3000-3250 min	-	EST/2017,51 ,10109- 10116	(10)
			0.5 L/min						
			- 50-150 °C						
8	Manganese Ore	Adsorption	65 µg/m ³	>90%	53.57 mg/g	100% Model fit	≥5	EST/2017,51 ,10109- 10116	(11)
			1 L/min						
			- 100-250 °C						
9	Ag-SBA- 15 nanocompo site	Adsorption	125 µg/m ³	>90%	13.2 mg/g;	1%	≥5	EST/2017,51 ,11909- 11917	(12)
			1 L/min						
			260000 h ⁻¹ 150 °C						
10	Nano- Sulfide	Adsorption and oxidation	75 µg/m ³	>99%	-	-	-	EST/2018,52 ,12926- 12933	(13)
			1 L/min						
			300000 cm ³ /(g·h)						
11	Cl-biochar	Adsorption	60-240 °C	>80%	583.0 µg/g	90 min	-	CEJ/331(201 8)536-544	(14)
			20 µg/m ³						
			2 L/min						
12	3D MnO ₂ /CS	Adsorption	- 150 °C	>99%	-	-	-	JHM/342(20 18)69-76	(15)
			0.5 L/min						
			- 100-300 °C						
13	O ₂ and NO Co-Doped Porous Carbon	Adsorption	50 µg/m ³	>90%	12.3 mg/g	100% Model simulation	-	EST/2019,53 ,1725-1731	(16)
			1 L/min						
			76000 h ⁻¹ 120 °C						

14	HNO ₃ - treated activated carbon fiber cloth (ACFC)	Adsorption	260-300 μg/m ³ 0.5 L/min - -	>90%	-	-	≥9	EST/2020,54 ,1857-1866	(17)
15	Ti- sulfurated γ-Fe ₂ O ₃	Adsorption and oxidation	4300 μg/m ³ 300 mL/min 1200000 h ⁻¹ 40-100 °C	-	48.6 mg/g	10 h	≥5	JHM/381(20 20)120967	(18)
16	AMn ₂ O ₄ (A=Cu, Ni and Zn)	Adsorption	50 μg/m ³ 1 L/min 50000 h ⁻¹ 200 °C	>95%	25.6 mg/g	50%	good	JHM/338(20 20)121738	(19)
17	V ₂ O ₅ - MoO ₃ /TiO ₂	Adsorption and oxidation	110 μg/m ³ 0.5 L/min 3000000 cm ³ /(g·h) 80 °C	79%	0.433 mg/g	40%	≥5	EST/2021,55 ,7072-7081	(20)
18	nano-ZnS	Adsorption	190 μg/m ³ - 150000 h ⁻¹ 60-220 °C	-	0.27 mg/g	50%	-	EST/2021,55 ,6965-6974	(21)
19	Single-Site Manganese	Adsorption	1200 μg/m ³ 0.5 L/min - 25-550 °C	>97% (<200 °C)	>13 mg/g	-	-	EST/2021,55 ,20,14126- 14135	(22)
20	Spherical- shaped CuS/C ₃ N ₄ nanosheet	Adsorption	95 μg/m ³ 1.2 L/min - 40-120 °C	100%	-	-	-	JHM/415(20 21) 125692	(23)

Table S4. The DFT calculation data

Structure	State	Charg e	E _t (a.u.)	ΔE _t (a.u.)	ΔG(kJ/mol)
Cl ⁻	singlet	-1	-460.082984	-	-
H ⁺	singlet	+1	-0.010000	-	-
Hg	singlet	0	-153.420286	-	-
Hg-Hg	singlet	0	-306.836414	-	-
11PANI	singlet	0	-1429.913820	-	-
11PANI ⁺	singlet	+2	-1430.605571	-0.671751	-1763.48
11PANI ⁺	triplet	+2	-1430.637001	-0.031430	-82.51
Cl-11PANI ⁺	singlet	0	-2351.220459	-0.448920	-1178.50
Cl-11PANI ⁺	triplet	0	-2351.199161	-0.396192	-1040.08
Hg-Cl-11PANI⁺	singlet	0	-2658.108806	-0.051933	-136.33
				-0.073231	-192.25
51PANI	singlet	0	-1428.680652	-	-
51PANI ⁺	singlet	+2	-1429.388896	-0.688244	-1806.78
51PANI ⁺	triplet	+2	-1429.371210	0.017686	46.43
Cl-51PANI ⁺	singlet	0	-2349.986134	-0.431270	-1132.17
Cl-51PANI ⁺	triplet	0	-2349.957207	-0.420029	-1102.66
Hg-Cl-51PANI⁺	singlet	0	-2656.824779	-0.002231	-5.86
				-0.031158	-81.80

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