

Triptycene-based and imine linked porous uniform microspheres for efficient and reversible scavenging of iodine from various media: a systematic study

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Materials and methods

Triptycene, cyanuric chloride, and 4-hydroxybenzaldehyde were purchased from Sigma-Aldrich. Iodine and KI was obtained from Merck. All other chemicals (solvents and reagents) used in this report were purchased locally. The triazine core aldehyde (TCA) was prepared following a previously published article.¹ The triamine derivative of triptycene (TP-NH₂) was synthesized using a protocol reported by Chen and coworkers.²

Instrumentation

Fourier transform infrared (FT-IR) spectra were recorded using a PerkinElmer 400 FT-IR spectrophotometer. Solid-state NMR spectra were recorded on a 500 MHz Bruker AVANCE III spectrometer. Powder X-ray diffraction (PXRD) patterns were recorded on Rigaku TTRAX III X-ray diffractometer using Cu K α radiation ($\alpha = 1.5406 \text{ \AA}$), with a scan speed of 2°/min-1. Thermogravimetric analyses (TGA) were performed using a Perkin Elmer STA 6000 analyzer in the temperature range of 30–700 °C, under a nitrogen atmosphere and at a heating rate of 10°C/min. Surface area of the polymer sample was measured using Quantachrome Autosorb iQ₂ instrument (Quantachrome, USA) with extra-high purity gases. Prior to the surface area analysis, a sample was activated by heating it to 120 °C for 12h. The resulting sample was then subjected to gas adsorption measurements (P/P₀ range from 0 to 1 atm) at 77 K. Quantachrome ASiQwin Version 3.01 software provided by Quantachrome was used to analysis gas uptake and pore size related data. The Brunauer-Emmett-Teller (BET) model was utilized to calculate the specific surface area. The pore size distribution was derived from the sorption curves by using the non-local density functional theory. SEM images were obtained using a HITACHI (S-4800) Field Emission Scanning Electron Microscope (FE-SEM). Prior to analyses, the samples were sputter-coated with gold. 15 kV voltage was applied while performing elemental analyses using an EDX detector. DLS experiments were performed using Beckman Coulter, Delsa™ Nano C Particle Analyzer. UV-visible absorption spectra were measured using a Shimadzu UV-2450 UV-vis spectrophotometer. The samples were scanned in the entire UV-Vis range. The Raman spectra were recorded using a 514 nm laser with an exposure time of 10 s by utilizing a Micro-Raman spectrophotometer (STR 750 RAMAN spectrograph, Seki Technotron Corporation Japan). XPS studies was performed using K-Alpha+model (Thermo Fischer Scientific, UK) with Al K α

source. All the digital images reported in our work were captured using Huawei Honor 8X or Motorola moto g60 mobile camera.

Synthesis of Triazine core aldehyde (TCA):



4-Hydroxybenzaldehyde 7.4 g (61.0 mmol) and sodium hydroxide 2.5 g (62.0 mmol) were dissolved in a mixture of acetone and water (100 mL, v/v 1:1). The obtained solution was added dropwise to a stirred solution of cyanuric chloride 3.7 g (20.0 mmol) in acetone (50 mL) at 0 °C for 1 h. The reaction mixture was refluxed for 2 h and later it was poured into water (300 mL) which resulted in the precipitation of a white solid product. The obtained solid product was washed with water and 10 % Na₂CO₃ and dried in vacuum to yield a white powder that was recrystallized from ethyl acetate to afford a white crystalline solid. ¹H NMR (400 MHz, CDCl₃) δ:9.96 (s, 3H), 7.89-7.87 (d, J = 8 Hz, 6H), 7.31-7.29 (d, J = 8 Hz, 6H). ¹³C {¹H} NMR (100 MHz, CDCl₃) 190.71, 173.21, 155.69, 134.42, 131.30, 122.21.

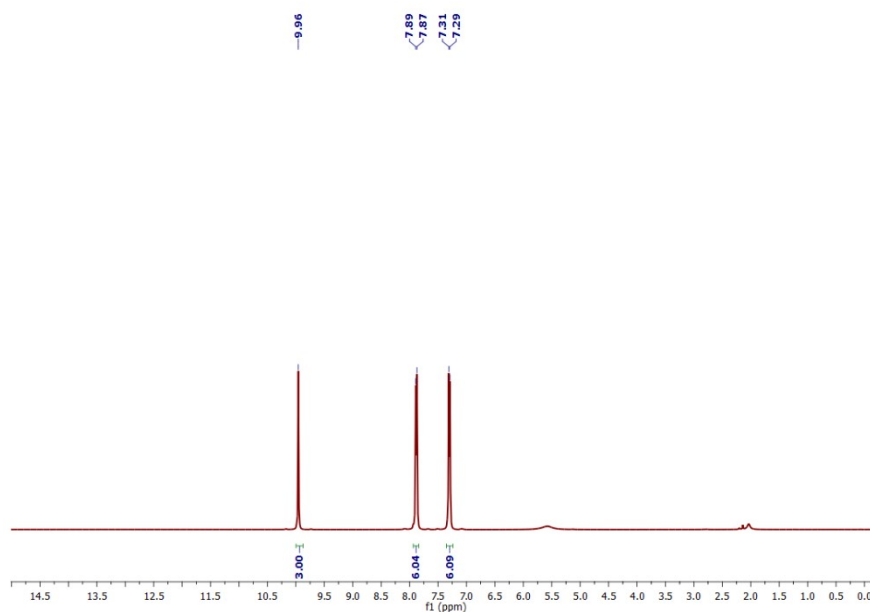


Fig. S1: ¹H NMR of TCA in CDCl₃

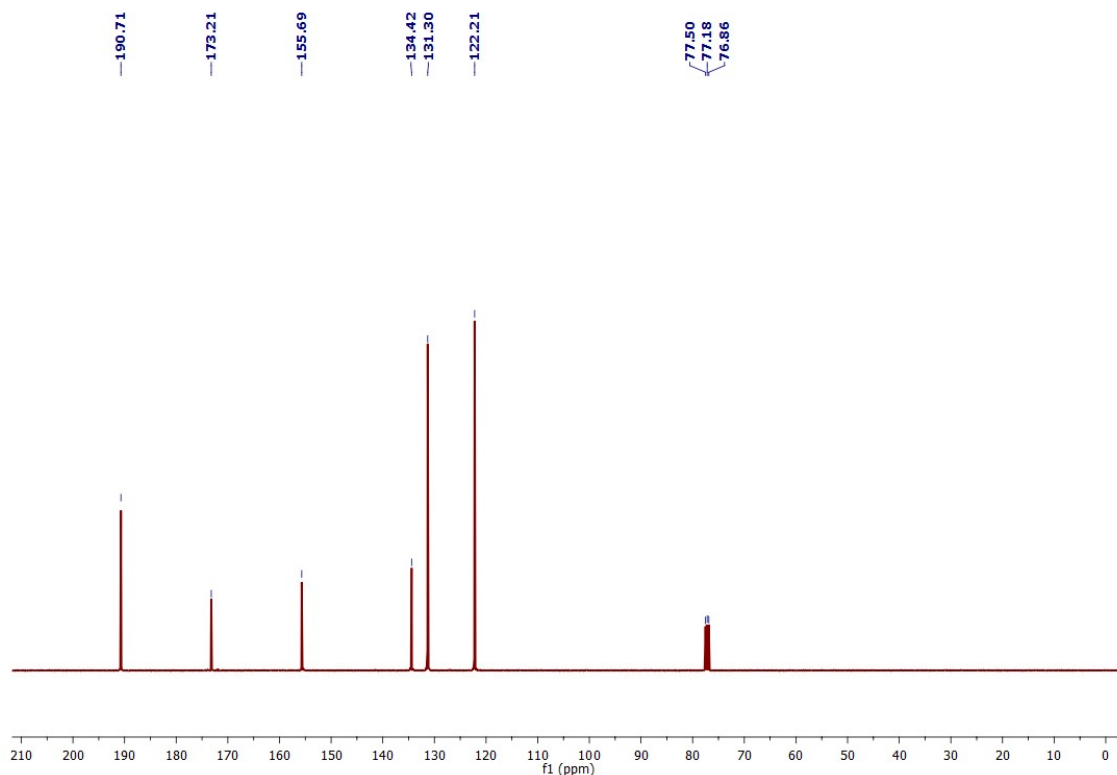
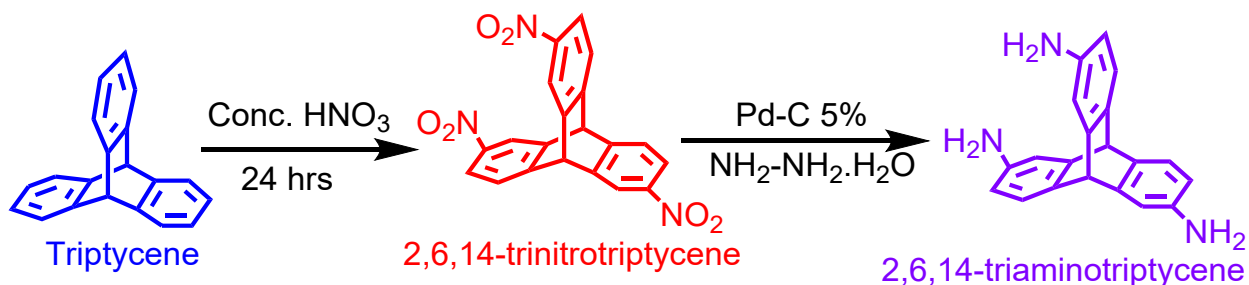


Fig. S2: $^{13}\text{C}\{^1\text{H}\}$ NMR of TCA in CDCl_3

Synthesis of 2,6,14-Triaminotriptycene (TP-NH₂)



Briefly, triptycene (2.5 g, 9.8 mmol) was taken in a flask to which we added concentrated HNO_3 (100 mL) and heated the reaction mixture at 75°C for 24 h. The obtained brown solution was cooled to room temperature and then poured into water (1000 mL) with continuous stirring. The precipitate thus obtained was collected, washed with cooled water, and later dried in air. The crude product was purified by column chromatography on silica gel using hexane and ethyl acetate as eluent to afford the desired product (2,6,14-trinitrotriptycene) as a white solid.

To a solution of 2,6,14-trinitrotriptycene (1.0 g, 2.6 mmol) in methanol (40 mL), we added hydrazine monohydrate (1.5 mL) and Pd/C 5% (~1.0 g). After heating at 60 °C until all hydrazine was quenched, the reaction mixture was cooled to room temperature and then filtered through a celite bed. The solvent was removed from the filtrate using a rotary evaporator. The desired product was obtained as a white solid in a nearly quantitative yield. ^1H NMR (400 MHz, DMSO- d_6) δ :6.945-6.900 (m, 3H), 6.594-5.589 (m, 3H), 6.092-6.067 (m, 3H), 4.883 (s, 2H), 4.754 (s, 6H). $^{13}\text{C}\{^1\text{H}\}$ NMR (100 MHz, DMSO- d_6) 147.83, 146.99, 145.62, 134.07, 133.16, 123.31, 122.94, 110.18, 109.82, 108.42, 108.16, 52.48, 51.30.

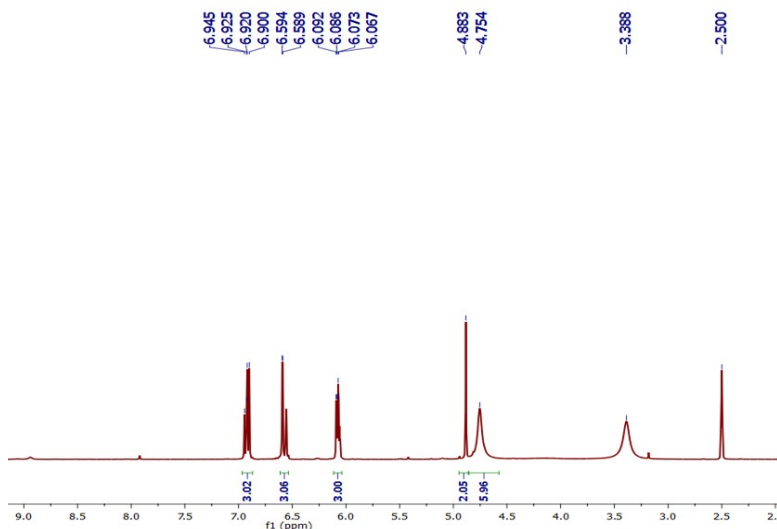


Fig. S3: ^1H NMR of TP-NH $_2$ in DMSO

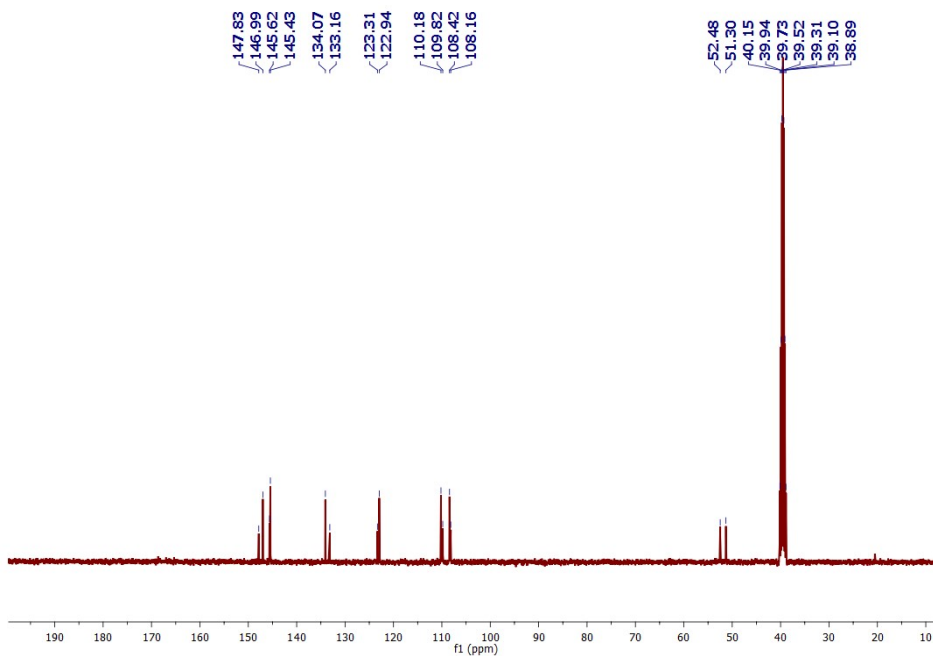


Fig. S4: $^{13}\text{C}\{^1\text{H}\}$ NMR of TP-NH $_2$ in DMSO

Synthesis of TP_POP-7:

Triazine core aldehyde (0.072 mmol, 31.8 mg) and triamino-triptycene (0.072 mmol, 21.6 mg) were loaded inside a pyrex tube and dissolve in 6 ml solution of dioxane/mesitylene (v/v 1:1). Then 0.5 ml 3M of acetic acid was added to the reaction mixture and sonicated for 1 min. Subsequently, the tube was degassed at 77 K and sealed. Upon warming the tube to room temperature, it was heated for 72h at 120 °C. Subsequently, the tube was allowed to cool to room temperature and the solid product obtained was isolated by filtration and washed with a copious amount of organic solvents followed by drying in open air. Further the compound was purified by Soxhlet extraction using methanol/THF (v/v 1:1) for 24h. The obtained powdery material was finally dried by placing in a vacuum oven for 12 hours (yield 46 mg) and was used for analyses.

Table S1

Name of the organic solvents	Solubility
Methanol	Insoluble
Dimethyl Sulfoxide	Insoluble
Dimethylformamide	Insoluble
Dimethylacetamide	Insoluble
Acetonitrile	Insoluble
Dichloromethane	Insoluble
1,2-Dichloroethane	Insoluble
Tetrahydrofuran	Insoluble
Toluene	Insoluble
Xylene	Insoluble
Ethyl Acetate	Insoluble
Hexane	Insoluble

Table S1: Solubility/insolubility chart of TP_POP-7

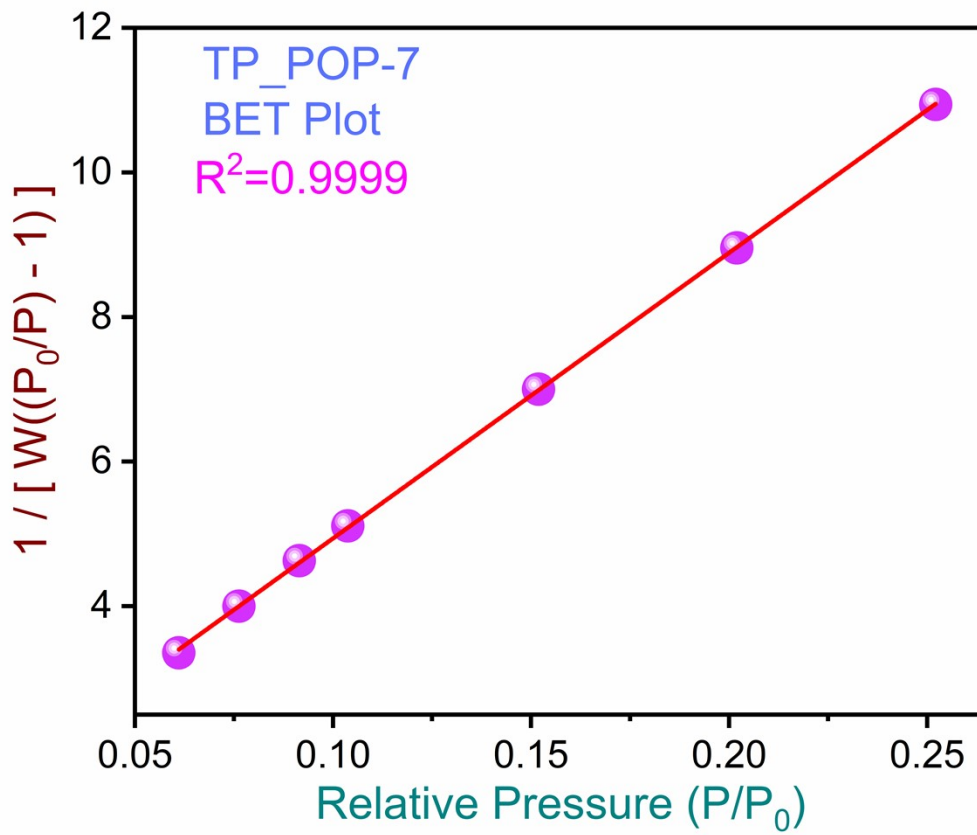


Fig. S5: BET plot of TP_POP-7

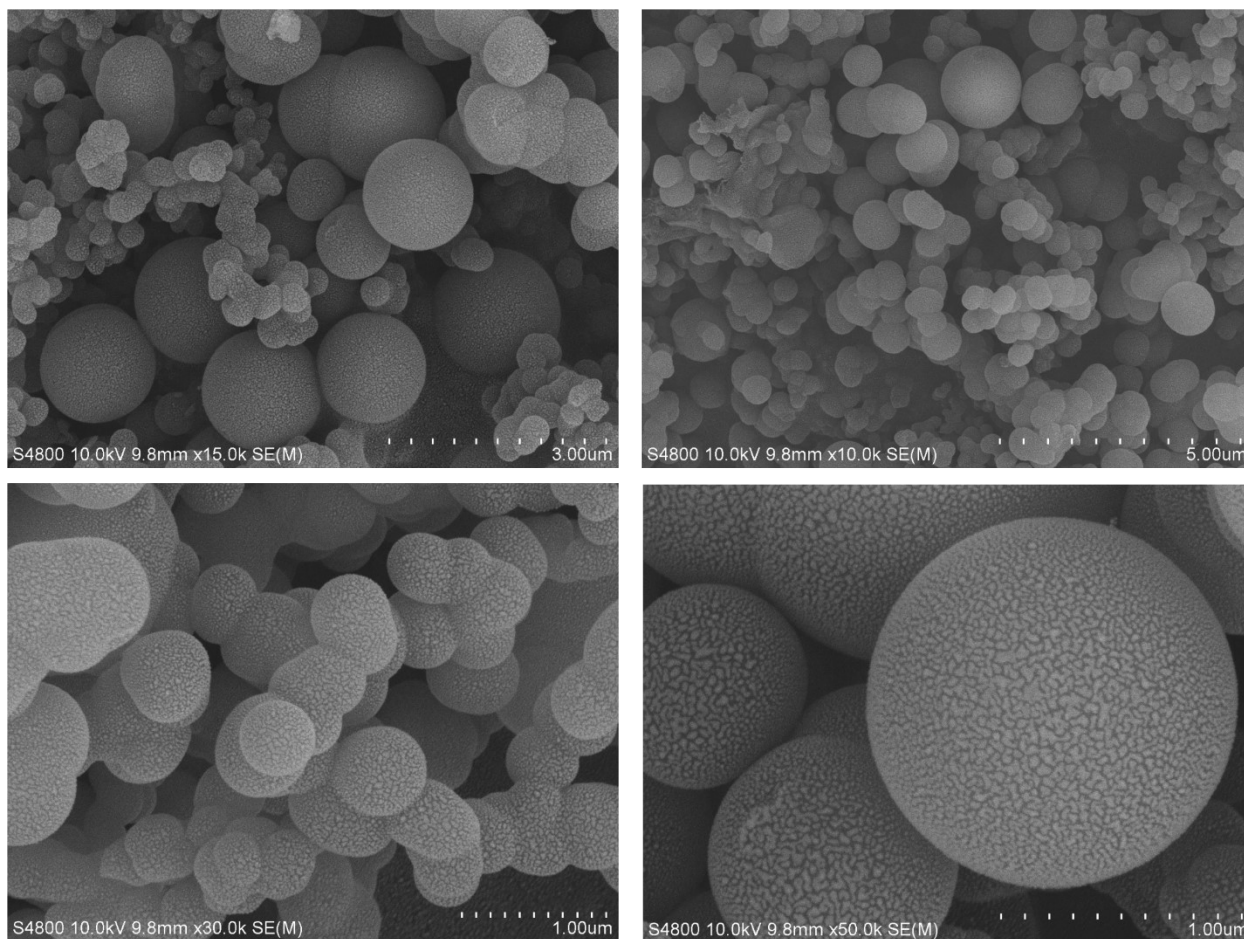


Fig. S6: FE-SEM images TP_POP-7

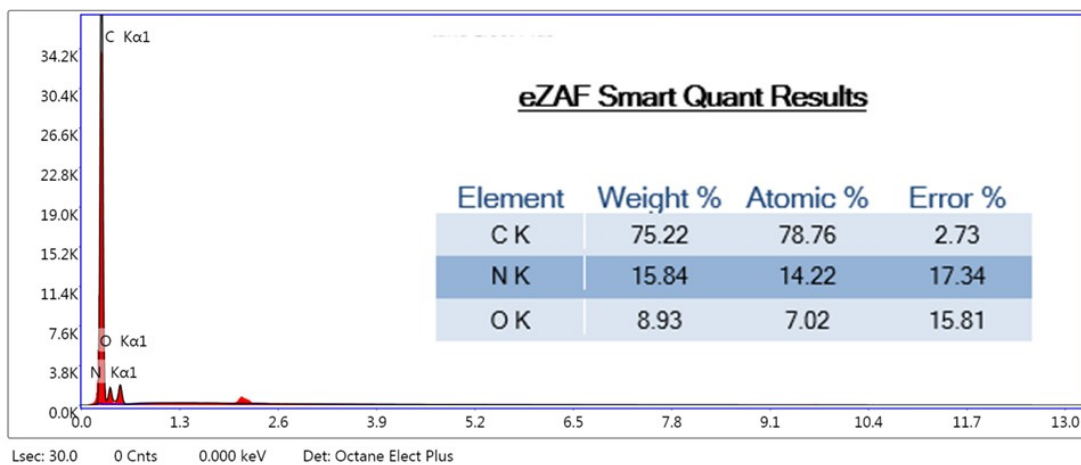


Fig. S7: EDX analysis of TP_POP-7

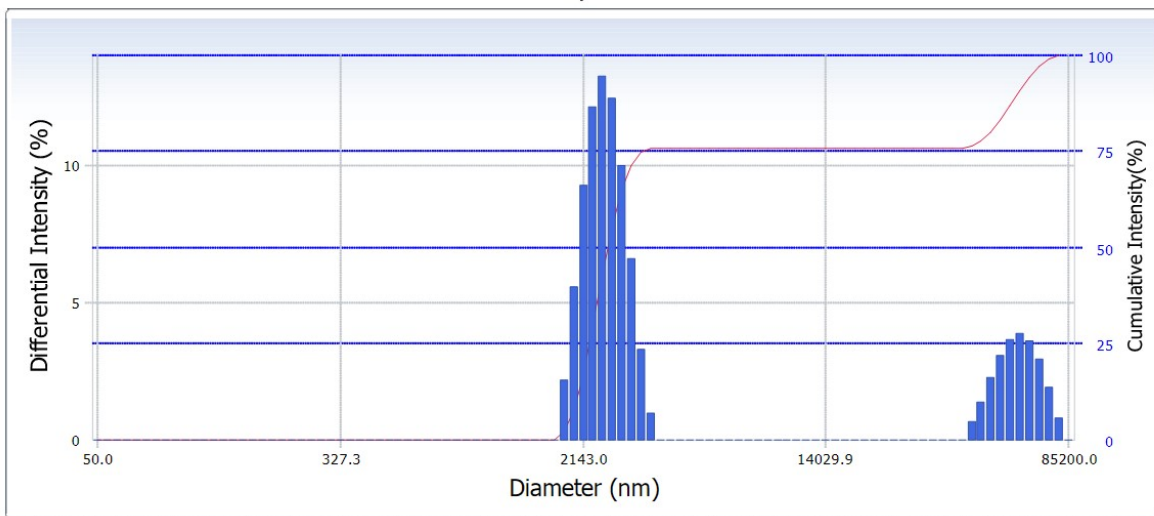


Fig. S8: DLS analysis of TP_POP-7

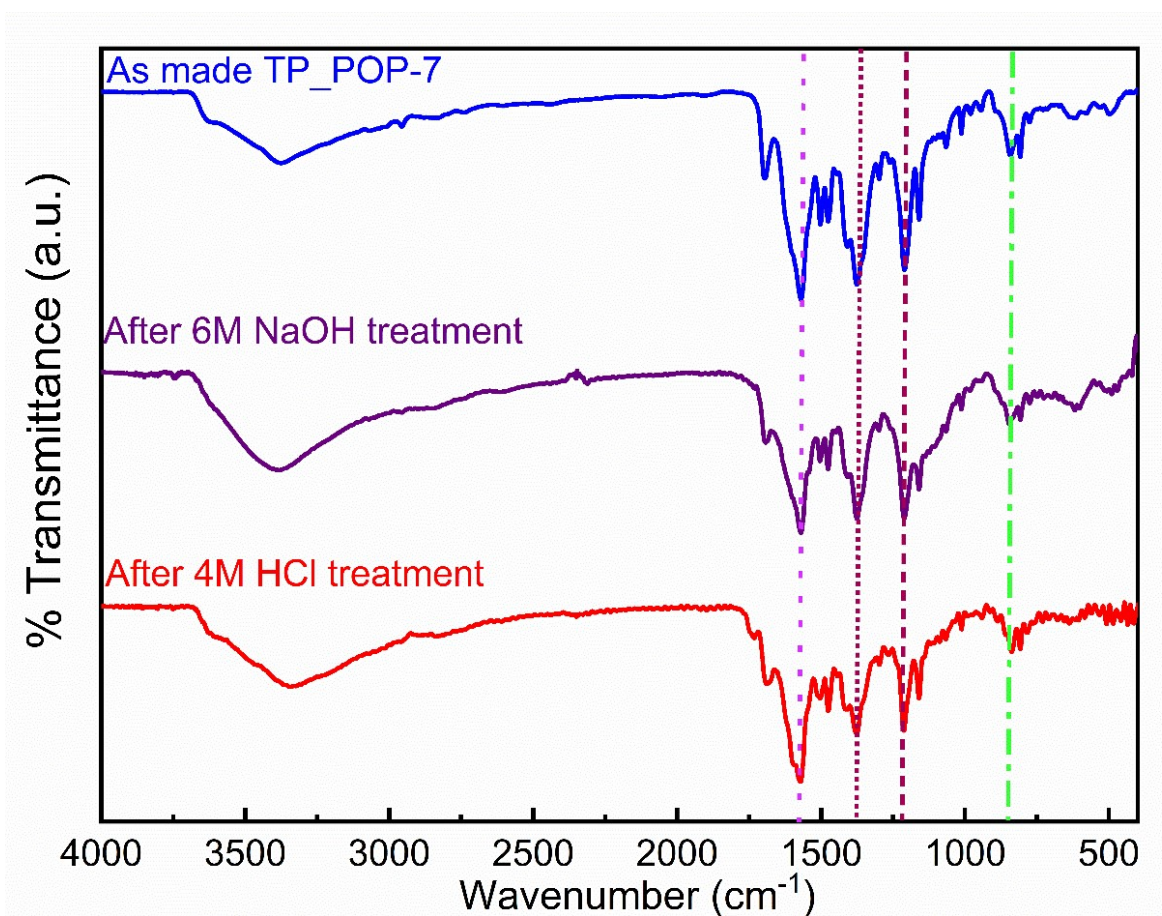


Fig. S9: FT-IR spectra of the TP_POP-7 along with acid and base treated TP_POP-7

Iodine sorption experiments

In Vapor phase and at elevated temperatures:

20 mg TP_POP-7 taken in 5 ml glass vial was weighed carefully and noted. This 5 ml glass vial was placed inside a 20 ml glass vial. Next this setup was placed inside a large glass chamber containing solid crystals of iodine. Next, the glass chamber was heated at 75 °C and 1.0 bar. At different intervals of time, the 5 ml vial containing the sample of TP_POP-7 was removed and its incremental increase of mass changes was noted before placing it back. After each round of the experiment, the wall of vials was cleaned thoroughly and carefully before weighing. The iodine uptake capacities for TP_POP-7 was calculated using the equation 1.

$$\alpha = \frac{m_2 - m_1}{m_1} \dots\dots\dots \text{(equation 1)}$$

where α is the iodine uptake capacity in g/g and m_1, m_2 are the weight of the vial containing TP_POP-7 before and after the start of the iodine vapor capture experiment.

Kinetics experiment:

To obtain data related to rate of uptake of iodine species from aqueous solutions or hexane, 2.5 mg of the TP_POP-7 was added to either a 5 ml of 50 ppm aqueous solution of I_3^- or a 2.5 ml hexane solution containing 250 ppm iodine. At different time intervals, the supernatant was collected and its UV-Vis spectra was recorded. The % of iodine captured was calculated using the equation (2)

$$\% \text{ removal} = \frac{C_0 - C_t}{C_0} \times 100\% \dots\dots\dots \text{(2)}$$

where C_0 and C_t are the concentration of iodine solution (in water or hexane) at the start of the experiment and at time t , respectively.

Next, the data was fitted with the non-linear equation (3) corresponding pseudo-first order and equation (4) for pseudo-second order kinetics.

$$Q_t = Q_e(1 - e^{-k_1 t}) \dots\dots\dots \text{(3)}$$

$$Q_t = \frac{k_2 Q_e^2 t}{1 + k_2 Q_e t} \dots\dots\dots \text{(4)}$$

Adsorption isotherm study

Solutions of varying concentrations (50 to 1000 ppm) were prepared by dissolving non-radioactive iodine either in n-hexane or water (containing KI). A weighed sample of TP_POP-7 was suspended in these solutions for capture of iodine species. After 24 h, the supernatant was collected and analyzed using UV-Vis studies. Langmuir (equation 5) and Freundlich (equation 6) adsorption isotherms were used to obtain insight into the mechanism of iodine adsorption.

$$Q_e = \frac{Q_m K_L C_e}{1 + K_L C_e} \dots\dots\dots \text{(equation 5)}$$

$$Q_e = K_F C_e^{1/n} \dots\dots\dots \text{(equation 6)}$$

Uptake capacity determination

A weighed sample of TP_POP-7 (50 mg) was suspended in an aqueous solution of KI₃ (600 mg KI and 300 mg I₂ in 3 mL of H₂O) for 48 h. After this time period, the adsorbent (TP_POP-7) was removed by filtration and washed with small quantities of water until the filtrate became clear. The filtrate (and the washings) obtained was then titrated to calculate the residual iodine present in the filtrate. Specifically, the collected filtrate was titrated against 0.025 (M) sodium bisulfite solution using 2% aqueous starch solution as an indicator. The resultant solution was found to contain 184.4 mg of iodine. Therefore, I₂ uptake by 50 mg of TP_POP-7 is 115.6 mg. Hence, the capacity of I₂ uptake by 2312 mg/g.

Iodine Capture in Hexane

Following is the description of experiments related to adsorption of iodine from its hexane solution. To a 10 ml iodine solution (10 mM), TP_POP-7 (10 mg) was added and allowed to stand at room temperature without any stirring. After 72 hours, the residual iodine concentration was measured by recording UV-Vis spectrum of the supernatant hexane solution. The iodine capture capacity (from hexane solutions) was calculated using the following equation (7):

$$Q_t = (C_0 - C_t) \times \frac{V}{m} \times M_w \dots\dots\dots \text{(equation 7)}$$

where C₀ and C_t are the concentration of iodine in its hexane solution (mM) at the start of the experiment and at time t, respectively. V is the volume of iodine/hexane solution (ml), and m is the mass of TP_POP-7 sample (mg) taken. M_w is the molecular weight of molecular iodine.

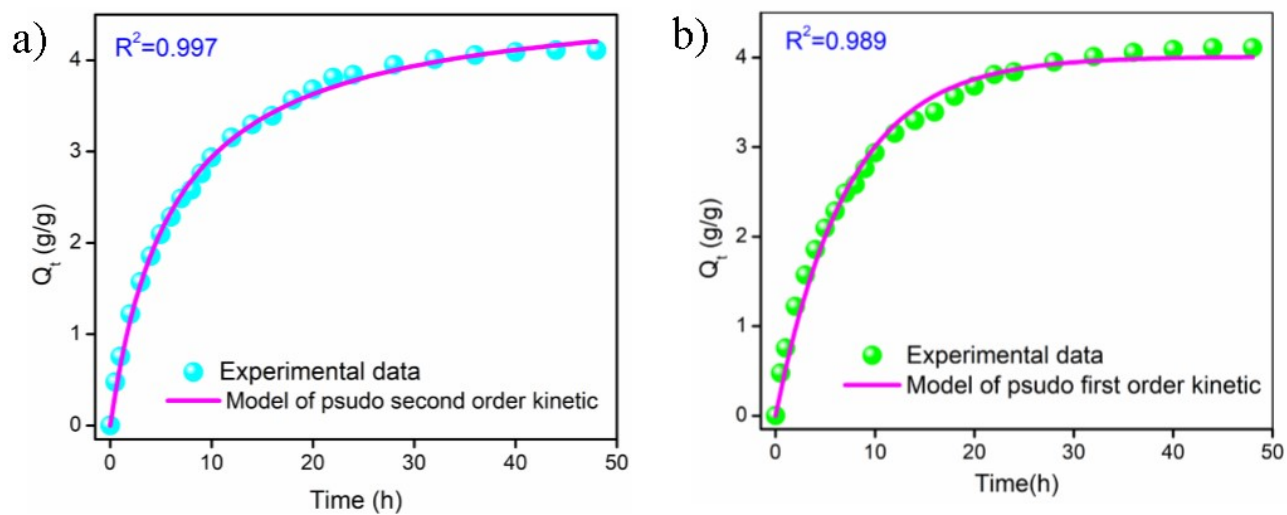


Fig. S10: (a) Pseudo second order kinetic model and (b) Pseudo first order kinetic model applied to adsorption of iodine vapours by TP_POP-7

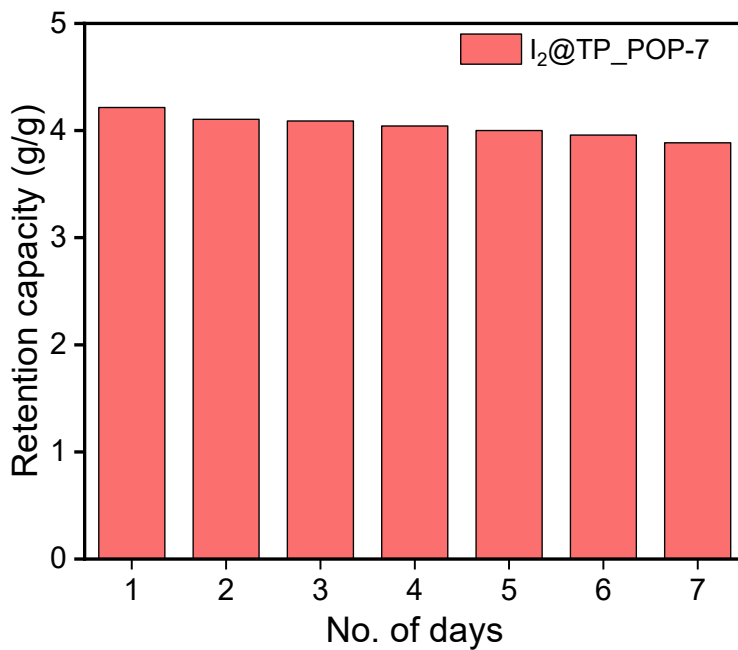


Fig. S11: Data of iodine retention experiments of $I_2@TP_POP-7$

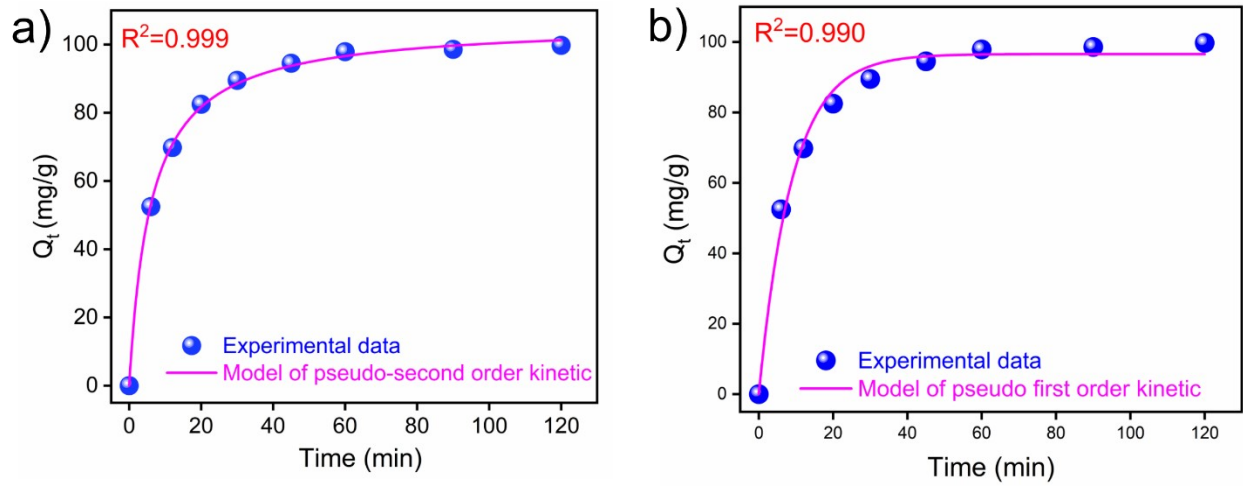


Fig. S12 : (a) Pseudo second order and (b) Pseudo first order data fitting of data related to adsorption of iodine by TP_POP-7 from aqueous solution.

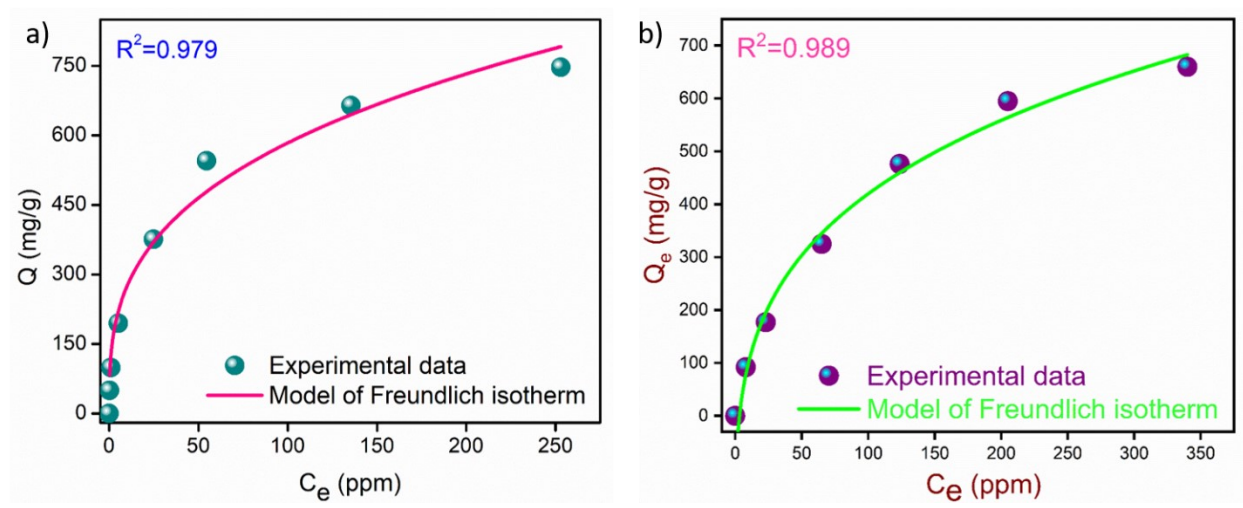


Fig. S13: Fitting of iodine adsorption data of TP_POP-7 using the Freundlich isotherm model from (a) Water (b) hexane as solvent

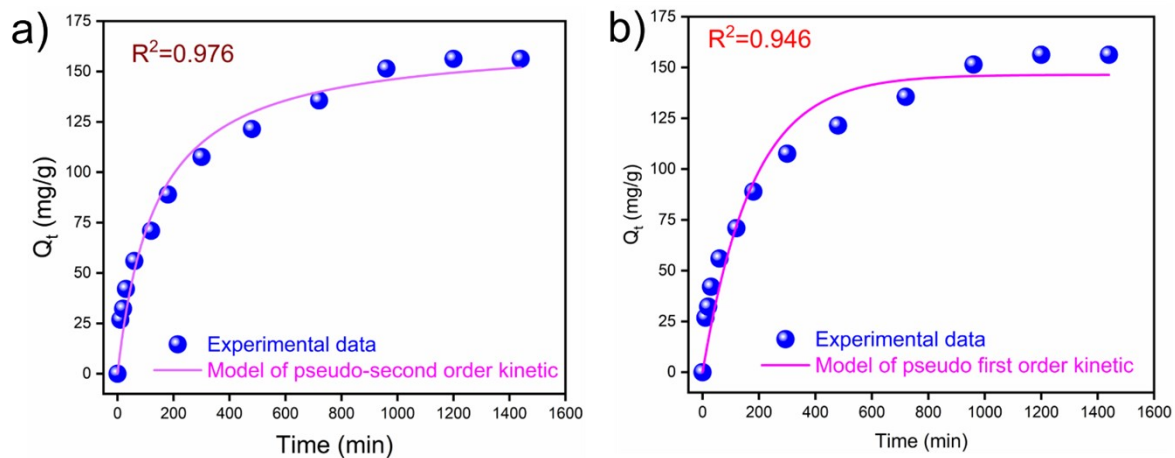


Fig. S14: (a) Pseudo second order and (b) Pseudo first order data fitting of data related to adsorption of iodine by TP_POP-7 from hexane solution.

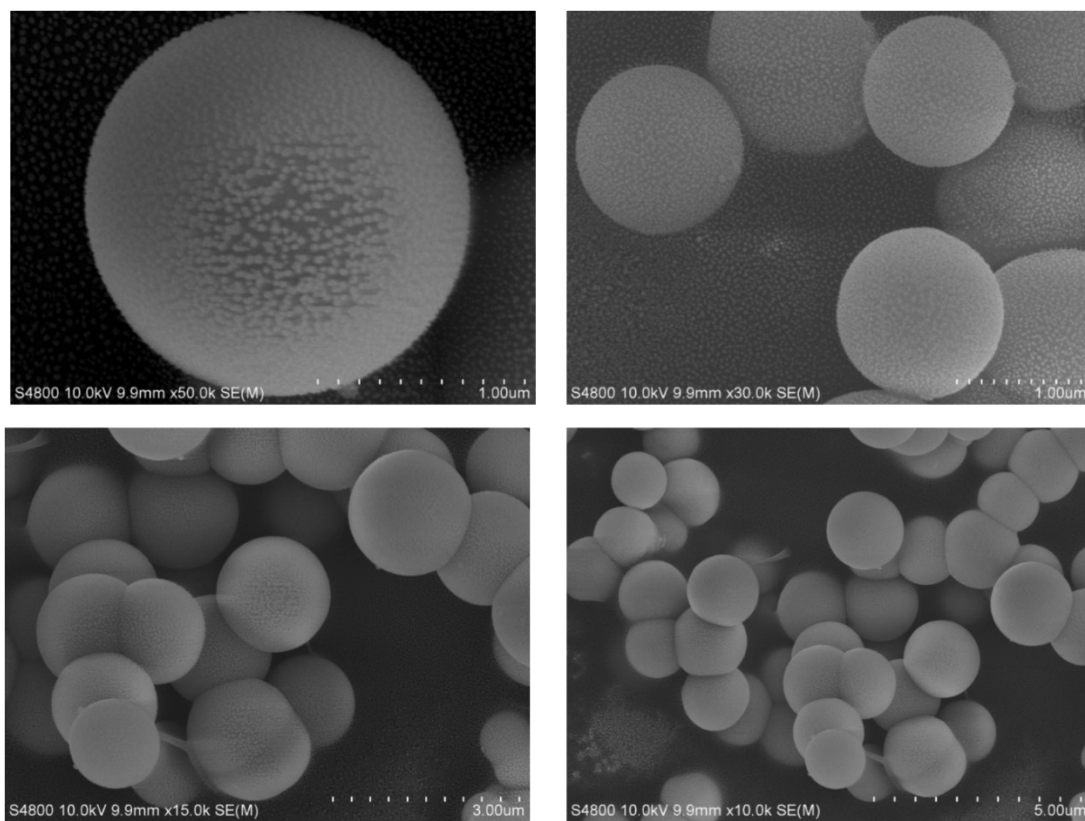


Fig. S15: FE-SEM images of $I_2@TP_POP-7$

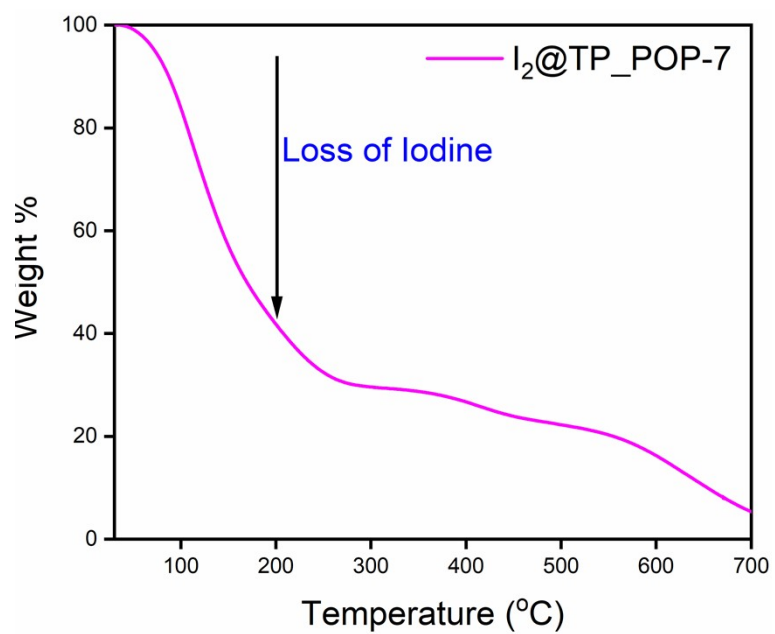


Fig. S16: TGA analysis of I₂ TP_POP-7.

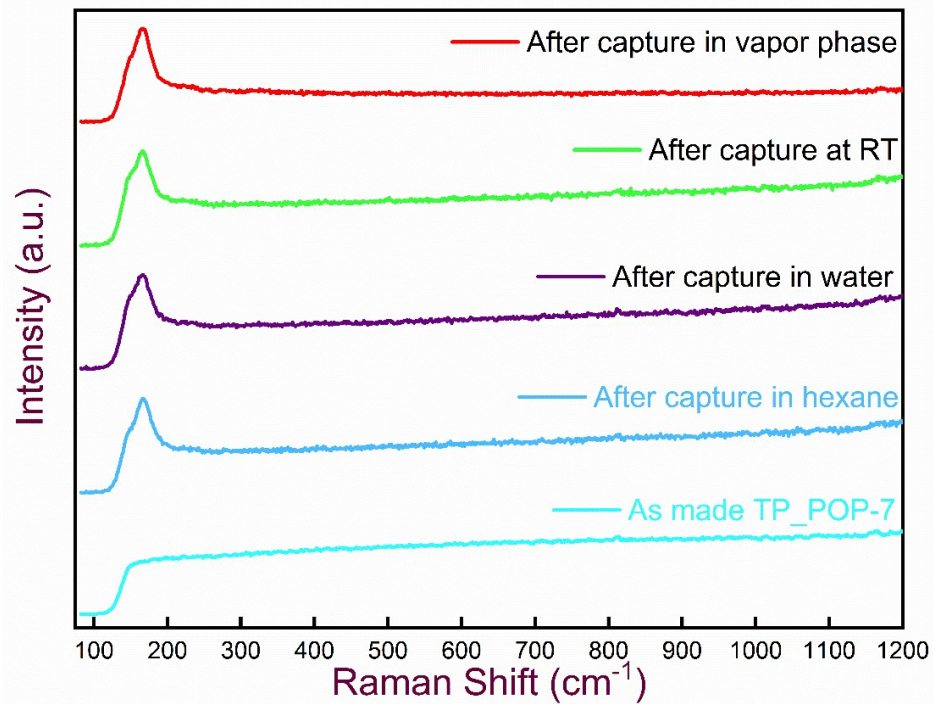


Fig. S17: Raman spectra of TP_POP-7 before and after iodine capture.

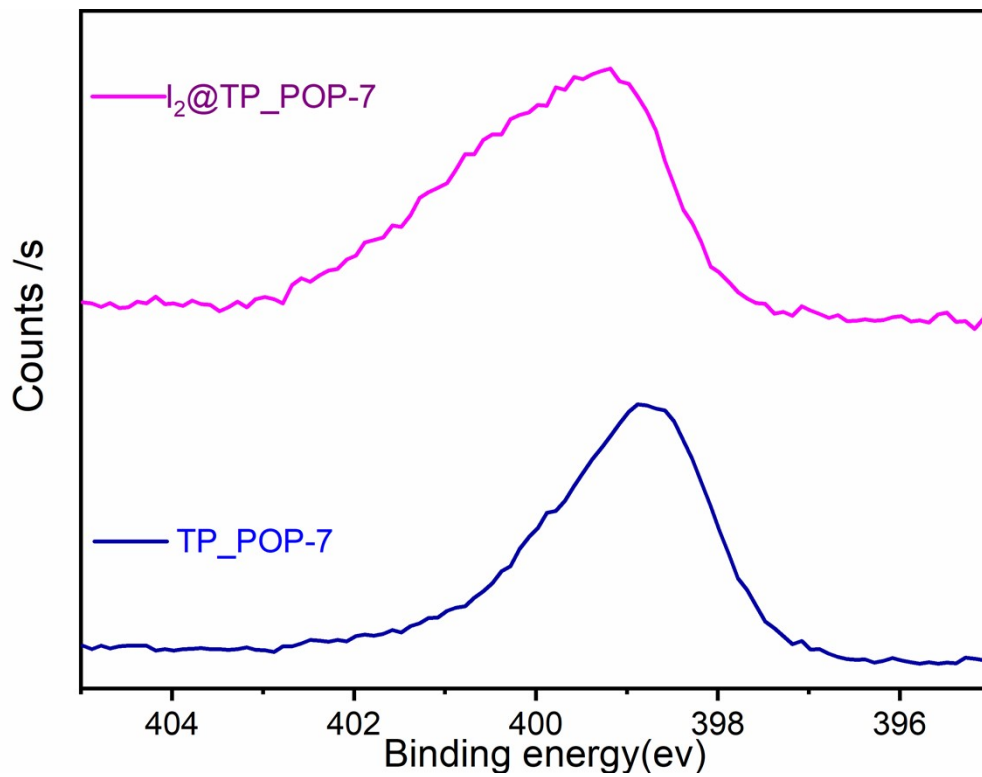


Fig. S18: High resolution XPS spectra of N(1s) of TP_POP-7 and $\text{I}_2@TP_POP-7$

Iodine release and adsorbent (TP_POP-7) regeneration by heating:

20 mg of iodine-adsorbed polymer [$I_2@TP_POP-7$] was charged in an open glass vial and heated at 125 °C and 1.0 bar on a sand bath. The iodine release efficiency (R_e) was calculated from the recorded weight losses in the sample of $I_2@TP_POP-7$ using the following formula:

$R_e = (20 - M_t) / M_x \times 100$ wt%, where M_t is the weight of the polymer after time t (t ranges from 0 to 300 min) and M_x is the weight of iodine captured in 20 mg $I_2@TP_POP-7$.

Iodine release and adsorbent (TP_POP-7) regeneration by extraction in MeOH or DMSO

5 mg of an iodine loaded $I_2@TP_POP-7$ sample was added to a vial containing either MeOH or DMSO (15 ml) with no stirring at room temperature. At different intervals of time, the UV-Vis spectra were recorded and rate of iodine release was monitored by noting relevant absorbance values (295 and 366 nm while using DMSO and 290 and 358 nm while using for methanol as solvent).

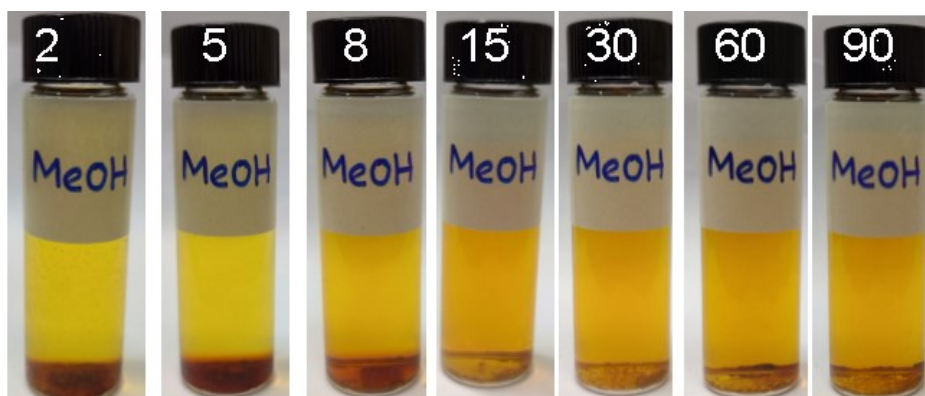


Fig. S19 : Digital images of gradual iodine release in methanol from $I_2@TP_POP-7$

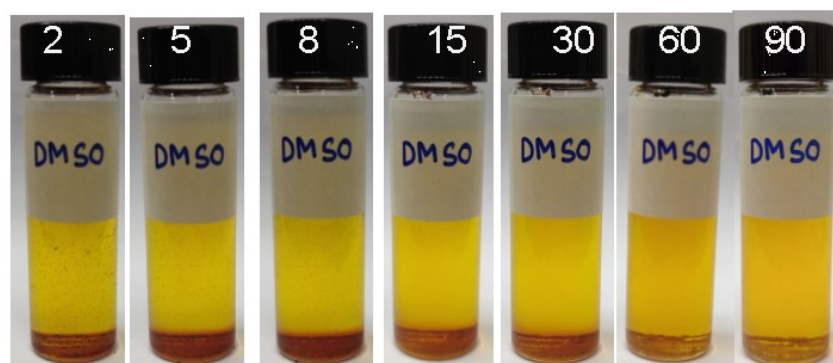


Fig. S20 : Digital images of gradual iodine release in DMSO from I₂ @TP_POP-7

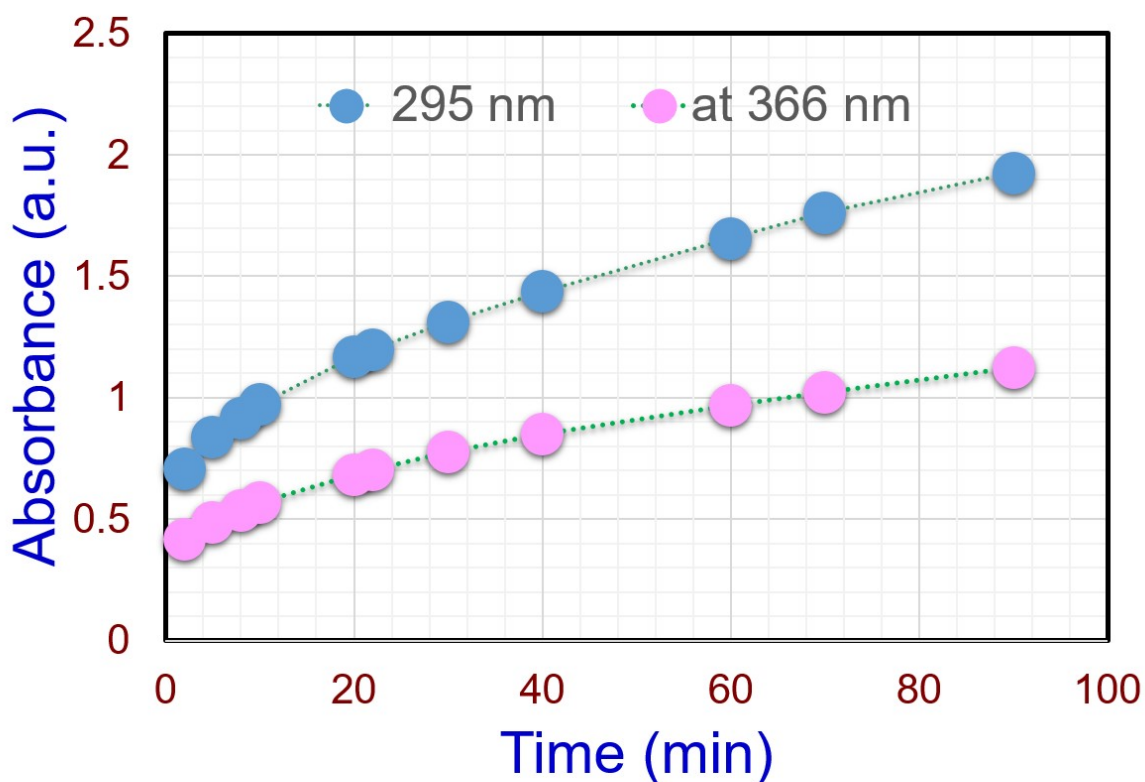
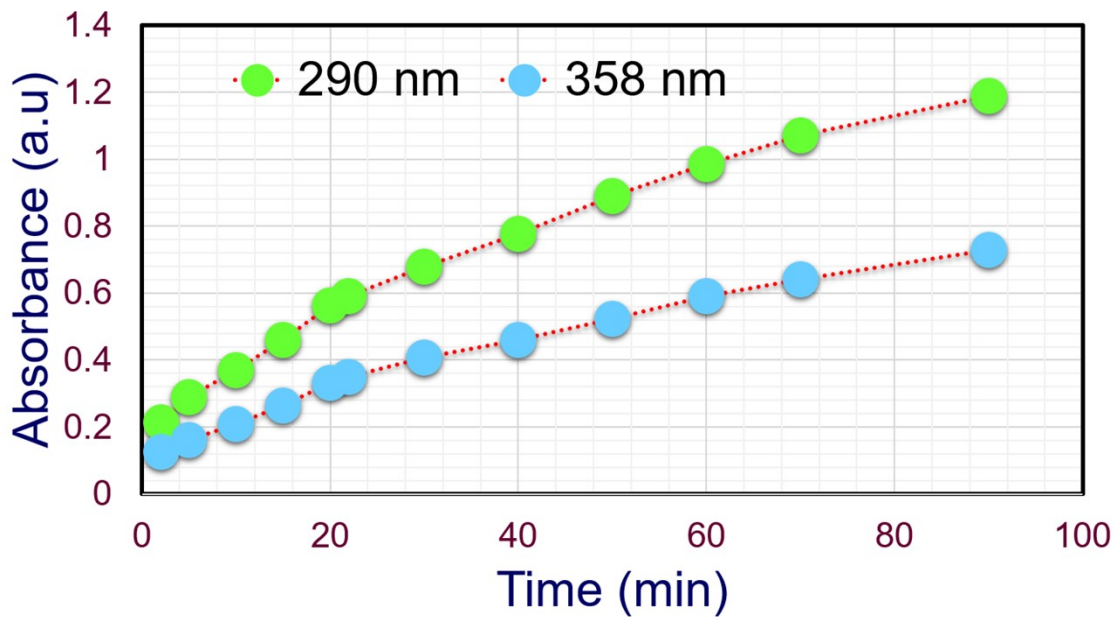


Fig. S21: Monitoring rate of iodine release from I₂@TP_POP-7 in DMSO

Fig. S22 : Monitoring release of iodine from I₂@TP_POP-7 in Methanol

Table S2: Comparison Table of Iodine vapour capture by adsorbents at elevated temperatures



Adsorbents	Temperature (°C)	BET Surface area(m ² /g)	I ₂ uptake (g·g ⁻¹)	Reference
TBIM	77	8.12	9.43	<i>J. Mater. Chem. A</i> , 2020, 8 , 2820–2826
TPB-DMTP COF	75	1927	6.260	<i>Adv. Mater.</i> , 2018, 30 , 1801991
TJNU-201	77	2510	5.625	<i>J. Mater. Chem. A</i> , 2020, 8 , 9523-9527
NDB-H	75	117	4.430	<i>Chem. Asian J.</i> , 2018, 13 , 2046-2053
TP_POP-7	75	86	4.215	This work
CMP-LS4	77	462	3.32	<i>Polym. Chem.</i> , 2020, 11 ,2786
PHF-1-Ct PHF-1	80	690 1046	4.05 3.05	<i>Chem. Commun.</i> , 2018, 54 , 12706-12709
CTF-CTTD-500	75	1334	3.87	<i>Ind. Eng. Chem. Res.</i> , 2018, 57 , 44, 15114
HCMP-3	75	92	3.36	<i>Macromolecules</i> 2016, 49 , 17, 6322–6333
POP-1	80	12	3.570	<i>J. Hazard. Mater.</i> , 2017, 338 , 224-232
TBTT-CMP@3	77	62.89	3.52	<i>Polym. Chem.</i> , 2020, 11 , 2786
Micro-COF-1 Micro-COF-2	75	816 1056	2.9 3.5	<i>Ind. Eng. Chem. Res.</i> , 2019, 58 , 10495-10502.
Azo-Trip	77	510.4	2.36	<i>Polym. Chem.</i> , 2016, 7 , 643
MOF-808	80	1930	2.18	<i>ACS Appl. Mater. Interfaces</i> , 2020, 12 , 20429–20439.
PAN-B PAN-T	75	1254 1273	3.17 3.11	<i>Polymer</i> , 194 (2020) 122401
PAF-24	75	136	2.76	<i>Angew. Chem. Int. Ed.</i> , 2015, 54 , 12733
N-HCP	75	222.8	2.57	<i>Sep. Purif. Technol.</i> , 236 (2020) 116260
CMPNH ₂	75	6.44	2.83	<i>J. Mater. Chem. A</i> , 2020, 8 , 1966-1974.
CPP 1 CPP 2 CPP 3	80	NR	1.53 2.00 1.40	<i>Polym. Chem.</i> , 2020, 11 ,3066
PAN-FPP5 PAN-TPDA	71.85 71.85	788.0 752.0	1.45	<i>Ind. Eng. Chem. Res.</i> , 2020, 59 , 3269-3278.
NRPP-1 NRPP-2	80	1579 1028	1.92 2.22	<i>ACS Appl. Mater. Interfaces</i> , 2018, 10 , 16049-16058
CBP1 CBP2 CBP3 CBP4 CBP5	80	3.0 143 794 98 203	145 101 135 140 166	<i>Polym. Chem.</i> , 2021, 12,2282
CMPN-3	70	1368	2.080	<i>J. Mater. Chem. A</i> , 2015, 3 , 87-91.
HKUST-1	75	NR	1.75	<i>Chem. Mater.</i> , 2013, 25 , 2591
ZIF-8	77	1630	1.250	<i>J. Am. Chem. Soc.</i> , 2011, 133 , 12398-12401
AIOC-26-NC AIOC-27-NC	80	508 285	0.70 0.50	<i>J. Am. Chem. Soc.</i> , 2021, 143 , 2325-2330

NR=Not Reported

Table S3: Comparison Iodine vapour capture by adsorbents at ambient temperatures

Adsorbents	Temperature	BET Surface area	I ₂ uptake (g·g ⁻¹)	Reference
iCOF-AB-50	R.T.	1390	2.790	<i>Angew. Chem. Int. Ed.</i> , 2021, 60, 22432.
TP POP-7	25 °C	86	2.040	This Work
COF-OH-50	R.T.	2103	1070	<i>Angew. Chem. Int. Ed.</i> , 2021, 60, 22432.
SCU-COF-2	R.T.	413.4	0.98	<i>Chem</i> 2021, 7, 699–714.
CC3	20	NR	0.36	<i>J. Am. Chem. Soc.</i> 2011, 133, 14920
HISL	R.T.	NR	0.53	<i>Energy Environ. Sci.</i> , 2016,9, 1050-1062
SL-1	R.T.	NR	0.48	<i>Energy Environ. Sci.</i> , 2016,9, 1050-1062
Si-BEA	R.T.	NR	0.47	<i>Energy Environ. Sci.</i> , 2016,9, 1050-1062
HKUST-1	R.T.	NR	0.38	<i>Energy Environ. Sci.</i> , 2016,9, 1050-1062
Ag ^U @MOR	R.T.	NR	0.08	<i>Nat. Commun.</i> , 2017, 8, 1–9
AC	R.T.	NR	0.70	<i>Energy Environ. Sci.</i> , 2016,9, 1050-1062
Zn-Sacc	R.T.	NR	0.05	<i>Energy Environ. Sci.</i> , 2016,9, 1050-1062
Ca-zeolite A	25	NR	0.04	<i>ACS Appl. Mater. Interfaces</i> , 2009,1, 1579
ZIF-8	R.T.	NR	0.03	<i>Energy Environ. Sci.</i> , 2016,9, 1050-1062

NR=Not Reported

Table S4: Comparison Table Iodine capture by adsorbents from aqueous solution

Adsorbents	Medium	BET Surface area(m ² /g)	I ₂ uptake (mg·g ⁻¹)	Reference
HcOF-1	Water	NR	2900	<i>J. Am. Chem. Soc.</i> , 2017, 139, 7172–7175
TP POP-7	Water	86	2312	This work
Fe ₃ O ₄ @PPy	Water	NR	1627	<i>J. Hazard. Mater.</i> , 344 (2018) 576–584
PCN-223	Water	642.089	1615.882	<i>Sep. Purif. Technol.</i> , 233 (2020) 115999
PCN-223-HPP	Water	851.271	1676.960	<i>Sep. Purif. Technol.</i> , 233 (2020) 115999
pSi-C composite	Water	762.13	299.40	<i>RSC Adv.</i> ,2021,11,5268–5275
TAPB-BPDA COF	Water	1082	988.17	<i>React. Funct. Polym.</i> , 159 (2021) 104806
THPS-C	Water	3125	926	<i>Adv. Mater. Inter.</i> , 2019, 6 , 1900249
NTP	Water	1067	429	<i>ACS Macro Lett.</i> ,2016, 5, 1039
cadmium(II)-triazole MOF	Water	NR	110	<i>Chem. Commun.</i> , 2011,47, 7185-7187
MBM - MOF	Water	62	880	<i>Angew. Chem., Int. Ed.</i> , 2018, 57, 10148
CdL2	Water	NR	460	<i>Chem. Commun.</i> , 2011, 47, 7185
PVDF/ZIF-8	Water	NR	73.33	<i>Sep. Purif. Technol.</i> , 238 (2020) 116488

NR=Not Reported

Table S5: Comparison Table of Iodine capture by adsorbents from various organic solvents

Adsorbents	Medium	BET	I ₂ uptake	Reference
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		Surface area	(mg·g ⁻¹)	
TP_POP-7	Hexane	86	865	This work
lac-Zn membrane	Cyclohexane	227	755	<i>Sep. Purif. Technol.</i> , 274 (2021) 118436
[Ni(NH ₂ -pba) ₂] ₂ ·2H ₂ O·2DMF	Cyclohexane	NR	559	<i>Micropor. Mesopor. Mat.</i> , 305 (2020) 110306
P-TzTz	Cyclohexane	137	494.09	<i>Micropor. Mesopor. Mat.</i> , 300 (2020) 110161
HLPP-OTS	Cyclohexane	814	468	<i>Polymer</i> , 186 (2020) 122004
UiO-66	Hexane	1015	401	<i>Dalton Trans.</i> , 2017, 46 ,7412–7420
CoFe@CA-D CoFe@CA-IS	Hexane	NR	457.99 194.34	<i>Micropor. Mesopor. Mat.</i> , 306 (2020) 110386
thiophene-based HCPs.	Cyclohexane	612.83	193	<i>Sep. Purif. Technol.</i> , 228 (2019) 115739
pyrrolidinone-based HCPs	Hexane	584	167	<i>Sep. Purif. Technol.</i> , 210 (2019) 995–1000
BN foam	Hexane	561.92	61.33	<i>Chem. Eng. J.</i> , 382 (2020) 122833

NR=Not Reported

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