Supporting Information

In Situ Water-Compatible Polymer Entrapment: A Strategy for Transferring Superhydrophobic Microporous Organic Polymers to Water

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Experimental Sections

Scanning (SEM) electron microscopy was conducted using a JSM6700F, respectively. The surface areas and porosity of materials were measured through the analysis of N_2 adsorption-desorption isotherm curves which were obtained at 77K using a BELSORP II-mini analyzer. The pore size distribution diagram was obtained based on the density functional theory (DFT). Powder X-ray diffraction (PXRD) studies were conducted using a Rigaku MAX-2200. Water contact angles were measured using a Theta Optical Tensiometer model (KSV instruments, Ltd.) and electrooptics comprising a CCTV camera connected to a computer (software Attension Theta). Thermogravimetric analysis (TGA) was conducted using a Seiko Exstar 7300. Solid state ^{13}C nuclear magnetic resonance spectra were obtained at CPTOSS mode using a 500 MHz Bruker ADVANCE II NMR machine at the NCIRF of the Seoul National University. A 4 mm magic angle spinning probe was used. The spinning rate was 5 kHz. Diffuse reflectance spectroscopy was conducted using a SHIMADZU UN-3600. The absorption spectrra of materials in the text were obtained from the diffuse reflectance spectra. Emission spectra were obtained using a JASCO FP-6200. Infrared absorption (IR) spectroscopy was conducted using a Bruker VERTEX 70 FT-IR spectrometer. Elemental analysis was conducted using a CE EA1110 instrument. X-ray photoelectron spectra (XPS) were obtained using a Thermo VG and Al-Ka radiation.

Synthetic procedure for MOPT-P and control MOPT material

1,1,2,2-Tetrakis(4-ethynylphenyl)ethane was prepared by the synthetic procedures in the literature.[1] For the preparation of MOPT-P, $Pd(PPh_3)_4$ (42 mg, 0.036 mmol), CuI (6.9 mg, 0.036 mmol), and poly(vinylpyrrolidone) (1.24 g, Aldrich Co., average Mw: 55000) were added to a flame-dried 100 mL Schlenk flask under argon. After ethanol (40 mL) and triethylamine (20 mL) were added, the mixture was sonicated for 40 min. After 1,1,2,2-tetrakis(4-ethynylphenyl)ethane (0.15 g, 0.36 mmol) and 1,4-diiodobenzene (0.24 g, 0.72 mmol) were added, the mixture was heated at 80^{o}C for 24 h. After the mixture had being cooled to room temperature, the powder was retrieved by centrifugation, washed with a mixture of acetone (10 mL), methanol (20 mL), and methylene chloride (20 mL) five times, then, with acetone (50 mL) two times, and then dried under vacuum. For the further washing described in the text, the MOPT-P (5 mg) was refluxed in ethanol (30 mL) for 3 days, retrieved by centrifugation, and dried under vacuum. In the synthetic cases using the different PVP reagents with average molecular weights of 10000 and 360000, the same weight (1.24 g) was used. For the synthesis of MOPT (control materials), the same synthetic procedures were applied without using PVP. The quantum yield of MOPT-P was measured as 7.3% in water using $[Ru(bpy)_3](PF_6)_2$ as a comparative compound. The quantum yield of $[Ru(bpy)_3](PF_6)_2$ in water is known as 6.3%.[2] The wavelength of the same absorption intensities of MOPT-P and $[Ru(bpy)_3](PF_6)_2$ was 416 nm, which was used as excitation wavelength. The integrated area ratio of emissions was 6540.4/5671.8 for MOPT-P and $[Ru(bpy)_3](PF_6)_2$, respectively.

Experimental procedure for the nitrophenol sensing

For optimizing the concentration of MOPT-P in water for emission studies, MOPT-P (8.0 mg) was dispersed in distilled water (20 mL). The portions of this mixture (10 mL, 5 mL, 2.5 mL, 1.25 mL, 0.63 mL, and 0.31 mL) were added to six 30 mL glasswares. Distilled water was added to each glassware to make a total volume of 20 mL. When the concentration of MOPT-P was 0.050 mg /mL H_2O, the mixture showed the biggest emission intensity. (Figure S3 in the SI) Thus, we fixed this optimized concentration in sensing studies. We screened the excitation wavelength from 370 nm to 390, 410, 430, and 440 nm. While the mixture showed the largest emission intensity when the excitation wavelength was 370 nm, there were no significant differences between the emission results of 370 and 410 nm. (Figure S3 in the SI) Considering the conventional visible light range, we fixed the excitation wavelength as 410 nm in the sensing studies.

For the sensing studies, the aqueous solutions of 2,4,6-trinitrophenol, 2,4-dinitrophenol, 4-nitrophenol, 2-nitrophenol, phenol, 4-chlorophenol, and 4-methylphenol with various concentrations (0, 0.0050, 0.010, 0.020, 0.040, 0.060, 0.080, 0.10, 0.125, 0. 25, 0.50, and 1 mM) were prepared. MOPT-P (3.0 mg) was dispersed in distilled water (30 mL). The substrate solution (2.5 mL) and MOPT-P mixture (2.5 mL) were mixed. After 2 min, the emission spectrum (excitation wavelength: 410 nm) was recorded using a JASCO FP-6200. In these sensing tests, the concentration of MOPT-P was set to 0.050 mg/mL. The substrate concentration became a half of the mother solution. K_{sv} values were measured by plotting I_o/I vs [M]; the Stern-Volmer plot ($I_o/I = K_{sv}[M] + 1$, I_o: the original emission intensity, I: the intensity of emission in the presence of substrates, M: the concentration of substrates in water, $R^2 > 0.99$ for linear regression as shown in Table 1)

For the recyclability tests, we scaled up the systems. First, MOPT-P (10 mg) was dispersed in distilled water (100 mL). After adding distilled water (100 mL), the emission spectrum (excitation wavelength: 410 nm) of the mixture was obtained. The MOPT-P was retrieved by filtration using Omnipore™ Membrane Filters (0.2 um JG, Merck Milipore Ltd.) and washed with water (200 mL). The recovery method was the best in reducing any loss of MOPT-P materials. The recovered MOPT-P was dispersed in distilled water (100 mL). 2,4,6-Trinitrophenol solution (100 mL, 1 mM) was then added to the mixture. After 2 min, the emission spectrum (excitation wavelength: 410 nm) was obtained. The MOPT-P was retrieved by the filtration method explained above. These procedures were repeated to obtain the results in the text.

Procedure for computational simulation (Figures S5-8)

To understand the reason for the sensing mechanism of MOPT-P, the density functional theory (DFT) calculations were carried out. The geometrical optimization for MOPT-1, MOPT-5, MOPT-poly, TNP, DNP, 2NP, 4NP, 4-chlorophenol, phenol, and 4-methylphenol were performed within B3LYP/light-tier-1 level using FHI-aims code.[3] The convergence criteria for geometrical relaxations was set to 10^{-2} eV/Å except MOPT-poly case. For MOPT-poly case, the periodic boundary condition (PBC) calculation with full relaxation of both atomic geometries and unit cell parameters was conducted with PBE/light-tier-1 level and 2×10^{-2} eV/Å of relaxation setting owing to numerous computational cost for hybrid functional B3LYP calculation with PBC condition. Then, single point calculation at B3LYP/light-tier-1 level was carried out to estimate the calculated HOMO and LUMO energy levels of MOPT-poly. We noted that in principle the HOMO and LUMO energy levels of MOPT-poly indicate the valence band maximum and conduction band minimum, respectively, because a PBC calculation can represent the characteristic of a solid state. The optimized unit cell parameters (*a*, *b* and *c*) of MOPT-poly were 33.49 Å x 22.97 Å x 39.34 Å. Along the *c* axis, unit cells were separated by a vacuum spacing of about 30 Å, and this guranteed that there were no interactions between the 2-dimensional sheets. Additionally, single point moral volume calculations were conducted for TNP, DNP, 2NP and 4NP within B3LYP/6-31+G(d,p) level using Gaussian 09 program.[4]

Reference

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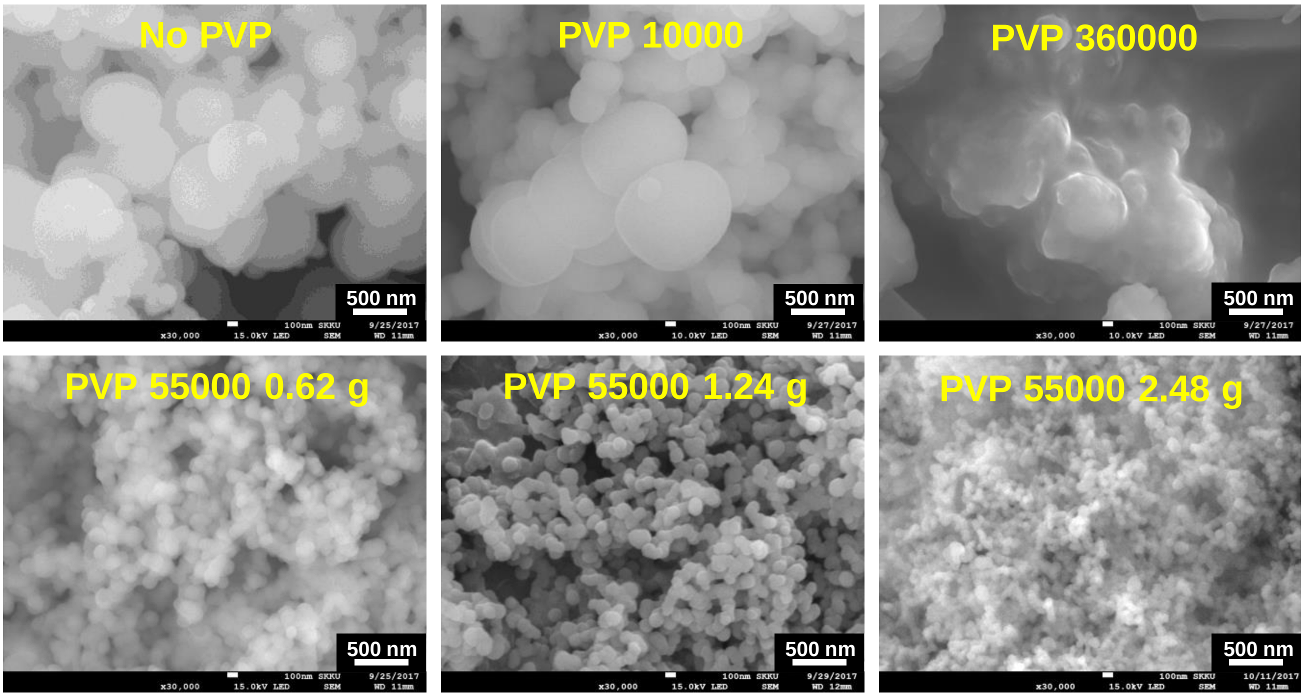


Figure S1 SEM images of MOPT-P prepared by various commercial PVP.

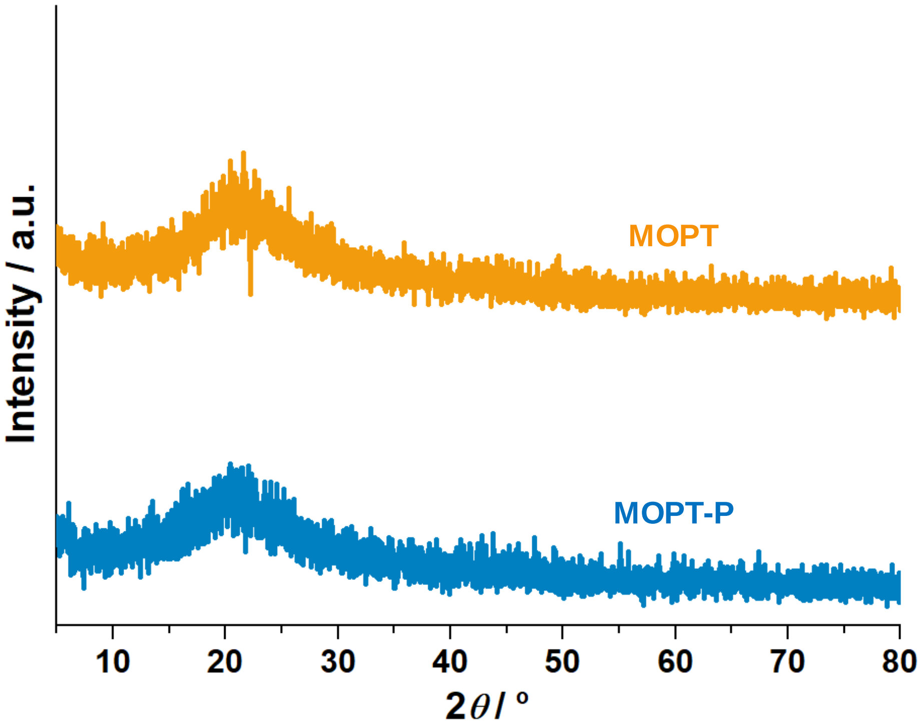


Figure S2 PXRD patterns of MOPT-P and MOPT.

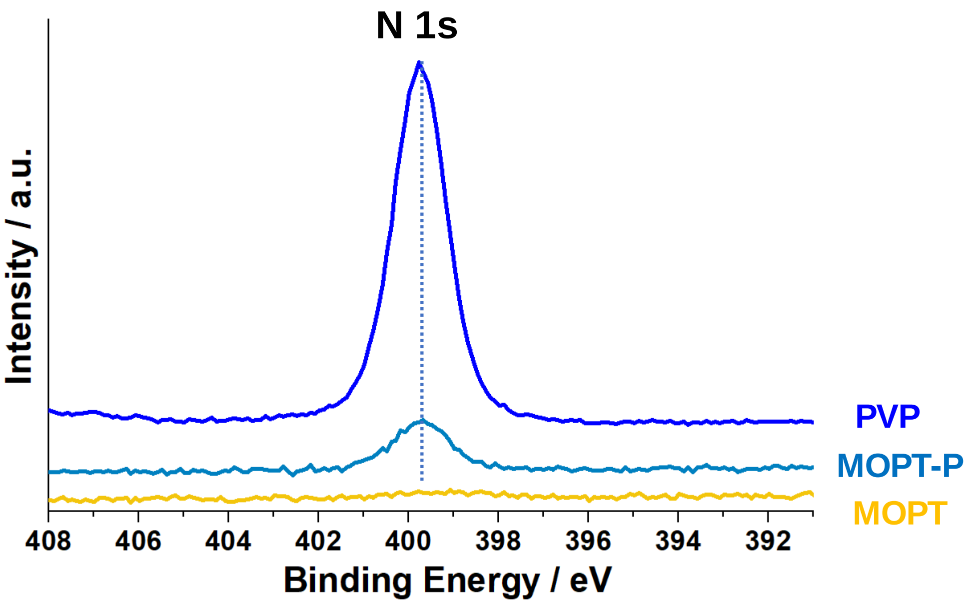


Figure S3 N 1s orbital XPS studies of PVP, MOPT-P, and MOPT. (The location of N1s peaks was calibrated by the Si 2p peaks co-detected in all the samples.)

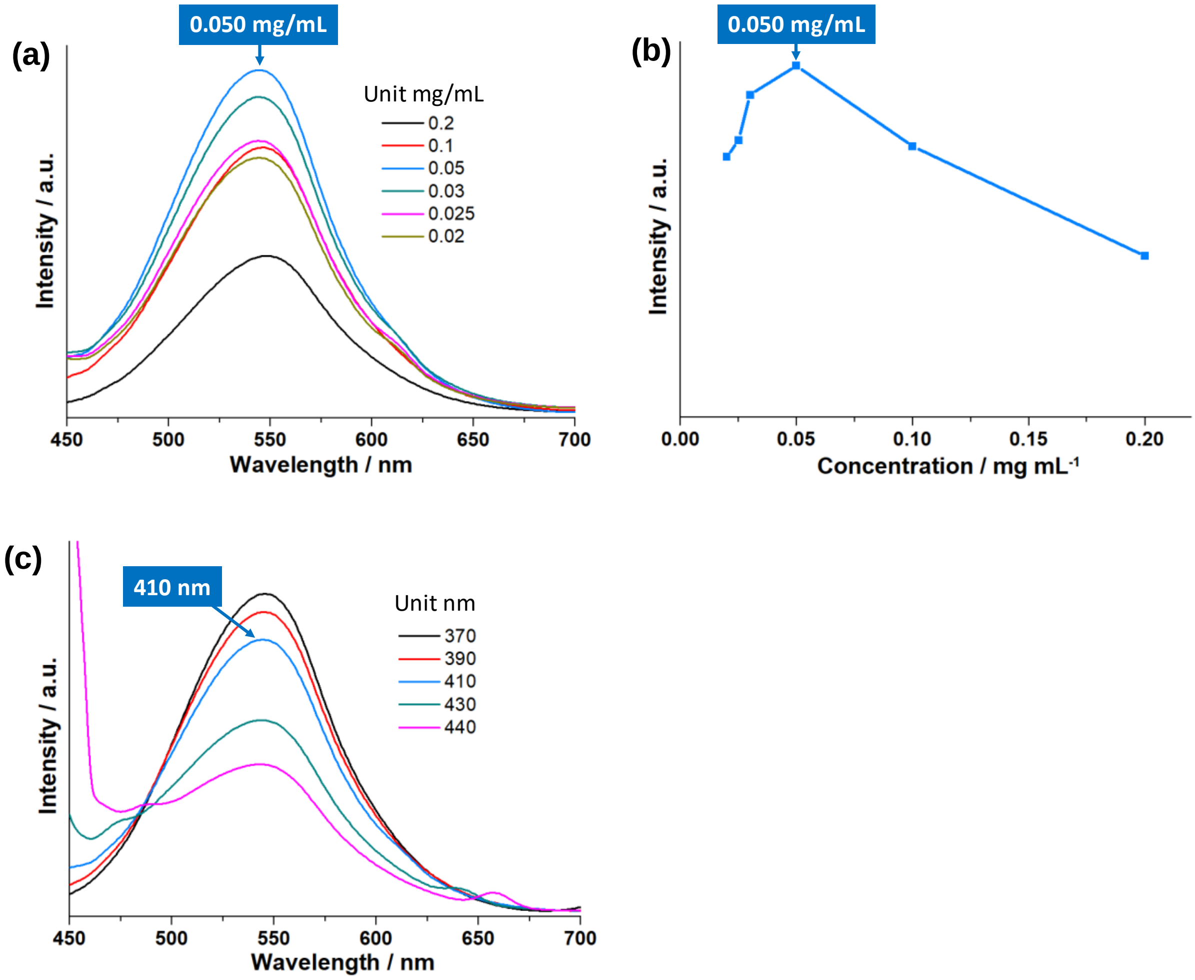


Figure S4 (a-b) Concentration and (c) excitation wavelength dependent emission properties of MOPT-P.

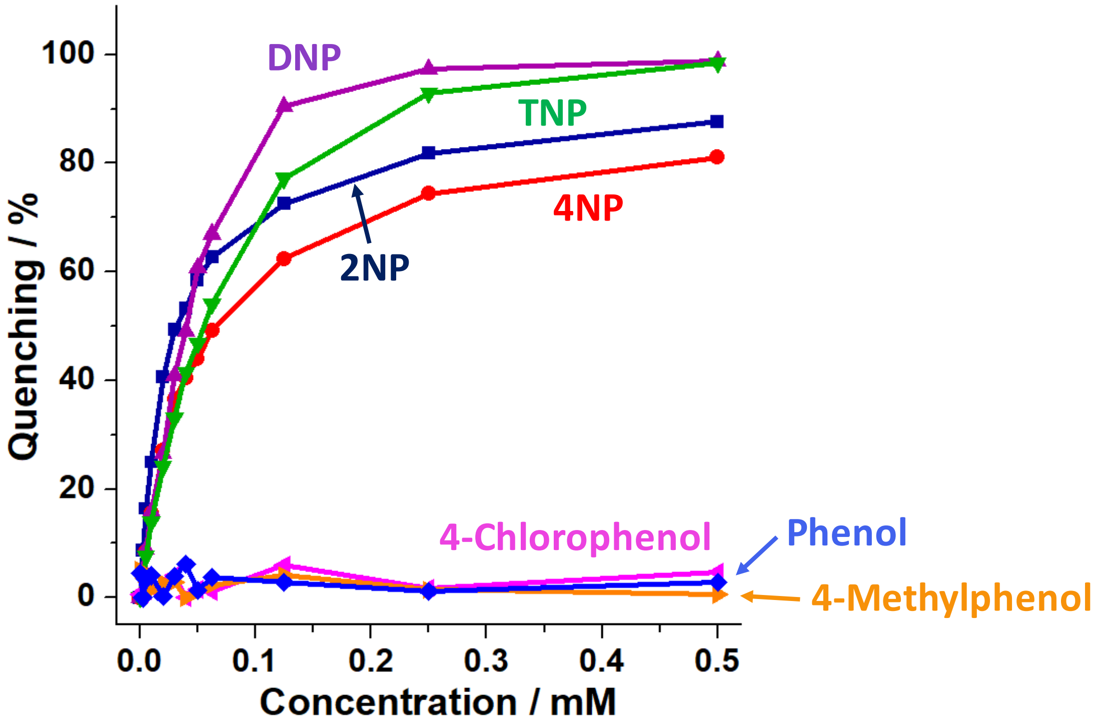


Figure S5 Emission-based sensing tests of MOPT-P towards phenol, 4-chlorophenol, and 4-methylphenol.

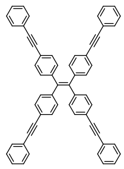
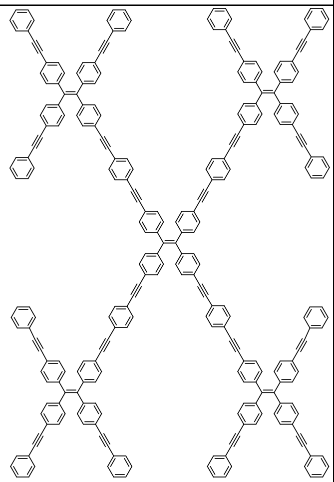
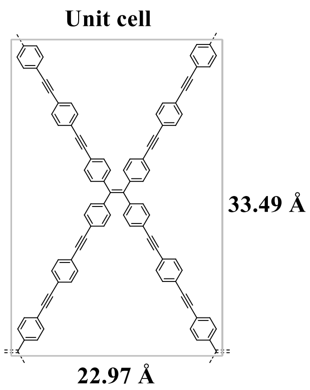
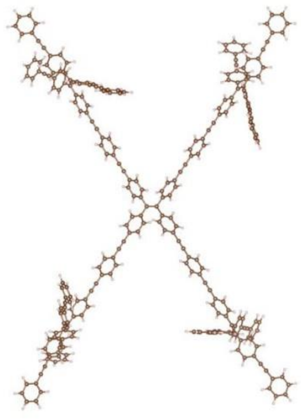
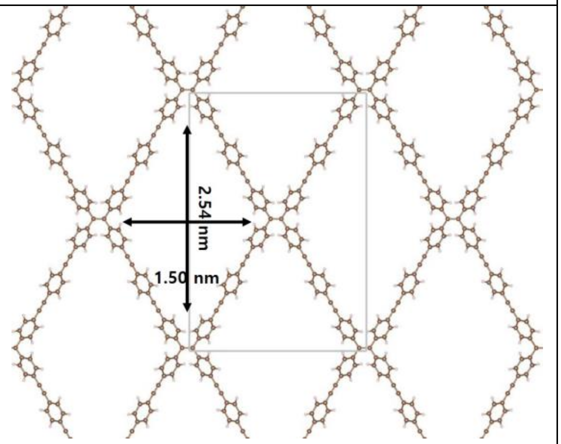
	MOPT-1	MOPT-5	MOPT-poly
Model Systems			
Optimized structures			

Figure S6 Model systems (MOPT-1, MOPT-5, and MOPT-poly) representing the model structures of MOPT-P with an incresing system size (top panel) and their optimized structures (bottom panel). Refer to the simulation procedures in the SI for more details.

Additional comments about geometrical description

The dihedral angles between ethylene plane and benzene plane is calculated to be around 50º. The propeller shape with 50º dihedral angles can effectively reduce steric hindrance between four benzene rings connected to ethylene part. For MOPT-5, there are no reason to retain planarity of overall molecules, we calculated the molecular system by just keeping the propeller shape with 50º dihedral angles. However, for MOPT-poly we designed the 2-dimentional sheet which might be an ideal case that intramolecular Sonogashira coupling reactions perfectly occur. From DFT calculations for MOPT-1, MOPT-5, MOPT-poly, we can indirectly estimate the possible ranges of HOMO and LUMO levels for MOPT-P.

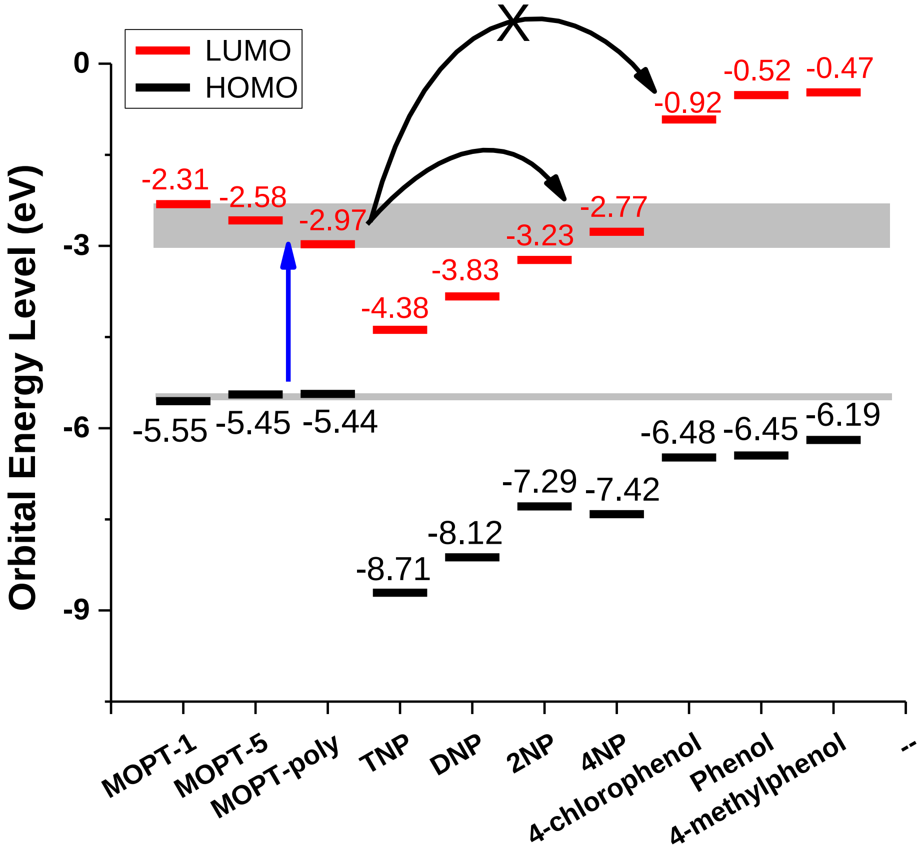


Figure S7 The calculated HOMO and LUMO energy levels of the MOPT-P model systems (MOPT-1, MOPT-5 and MOPT-poly), TNP, DNP, 2NP, 4NP, 4-chloro phenol, Phenol and 4-methyl phenol.

MOPT-1

C	0.00059224	0.68109457	-0.00044890
C	-0.00059158	-0.68095331	-0.00047384
C	-1.25071142	-1.48523383	-0.11427699
C	-1.48757635	-2.55271720	0.76334602
C	-2.19833166	-1.23618890	-1.11548089
C	-2.63994475	-3.31379140	0.67370933
H	-0.76156727	-2.78074184	1.53154976
C	-3.34625888	-2.00261315	-1.22308196
H	-2.03093296	-0.43228031	-1.81770592
C	-3.59510761	-3.05119574	-0.32247061
H	-2.81210849	-4.12115407	1.37103102
H	-4.06312160	-1.79617143	-2.00496046
C	1.24830764	-1.48689545	0.11367483
C	1.48414450	-2.55503417	-0.76347096
C	2.19583046	-1.23844872	1.11515332
C	2.63583289	-3.31710295	-0.67325817
H	0.75781004	-2.78307872	-1.53109019
C	3.34309298	-2.00573151	1.22323875
H	2.02887864	-0.43418440	1.81707763
C	3.59119117	-3.05476773	0.32303031
H	2.80728783	-4.12511556	-1.36995133
H	4.06008500	-1.79952211	2.00494992
C	1.25071388	1.48537869	-0.11421150
C	1.48757378	2.55282189	0.76348281
C	2.19834631	1.23637122	-1.11543288
C	2.63995805	3.31386810	0.67395044
H	0.76152368	2.78088590	1.53157411
C	3.34625551	2.00283537	-1.22305149
H	2.03098310	0.43245444	-1.81763488
C	3.59511869	3.05134112	-0.32229865
H	2.81204203	4.12130755	1.37111808
H	4.06316751	1.79633700	-2.00479335
C	-1.24830616	1.48703673	0.11369293
C	-1.48412115	2.55519524	-0.76345344
C	-2.19585390	1.23856544	1.11515657
C	-2.63581554	3.31724762	-0.67329024
H	-0.75773750	2.78332419	-1.53093660
C	-3.34309688	2.00588151	1.22328736
H	-2.02894366	0.43425497	1.81702786
C	-3.59118247	3.05492240	0.32303988
H	-2.80718607	4.12537464	-1.36979752
H	-4.06013643	1.79959677	2.00488994
C	-4.77434964	3.83407139	0.41687366
C	-5.78402337	4.49579368	0.48774057
C	-4.77846886	-3.83012635	-0.41638164
C	-5.78785896	-4.49232859	-0.48714149
C	4.77435579	-3.83391537	0.41684563
C	5.78401533	-4.49565669	0.48770833
C	4.77848272	3.83027796	-0.41620952
C	5.78788298	4.49246772	-0.48698349
C	6.97164981	-5.27274590	0.56341639
C	7.88706407	-5.08191919	1.61181573
C	7.25168402	-6.24539211	-0.41099830
C	9.04583660	-5.84175559	1.67846344
H	7.67744560	-4.33631755	2.36505663
C	8.41348552	-6.99960397	-0.33620667
H	6.55139117	-6.39769931	-1.21961648
C	9.31467554	-6.80164902	0.70656014
H	9.74139032	-5.68496194	2.49144766
H	8.61674313	-7.74423728	-1.09354996
H	10.21916838	-7.39143350	0.76153462
C	-6.97636960	-5.26809888	-0.56337802
C	-7.89106209	-5.07567384	-1.61203036
C	-7.25852574	-6.24025437	0.41089821
C	-9.05137762	-5.83315898	-1.67907776
H	-7.67956496	-4.33079607	-2.36556507
C	-8.42193437	-6.99200329	0.33592902
H	-6.55877117	-6.39401190	1.21976887
C	-9.32263567	-6.79208538	-0.70685863
H	-9.74647047	-5.67484820	-2.49200837
H	-8.62700808	-7.73604793	1.09341585
H	-10.22853023	-7.37973682	-0.76182185
C	-6.97169947	5.27282447	0.56341643
C	-7.25179084	6.24539366	-0.41106176
C	-7.88715604	5.08191639	1.61169550
C	-8.41368890	6.99945898	-0.33642002
H	-6.55149218	6.39770848	-1.21969028
C	-9.04597688	5.84169340	1.67835086
H	-7.67745098	4.33643348	2.36509635
C	-9.31487939	6.80150392	0.70638472
H	-8.61696854	7.74406393	-1.09374826
H	-9.74155069	5.68486364	2.49129522
H	-10.21944089	7.39118766	0.76128265
C	6.97642448	5.26819189	-0.56324180
C	7.25865267	6.24030516	0.41107350
C	7.89114568	5.07563775	-1.61176489
C	8.42213756	6.99193314	0.33618088
H	6.55889933	6.39411982	1.21992357
C	9.05148583	5.83309289	-1.67890180
H	7.67955254	4.33084420	-2.36543398
C	9.32280871	6.79199615	-0.70667911
H	8.62722974	7.73600197	1.09359305
H	9.74658394	5.67471392	-2.49179063
H	10.22875556	7.37956548	-0.76161948

MOPT-5

C	-0.00572553	0.65488370	-0.17212734
C	-0.01003710	-0.69676166	-0.00269551
C	-1.26653886	-1.49358915	0.08476177
C	-1.43130753	-2.44473284	1.10184457
C	-2.29303773	-1.35626924	-0.85884597
C	-2.58767388	-3.19839952	1.19921885
H	-0.64474698	-2.58673351	1.83004832
C	-3.44670453	-2.11694985	-0.77885539
H	-2.18215328	-0.64607293	-1.66545648
C	-3.62141014	-3.04678606	0.25960505
H	-2.70281768	-3.91382212	2.00078168
H	-4.22495776	-1.99876025	-1.51898522
C	1.24178428	-1.49763257	0.11022439
C	1.39995034	-2.67214029	-0.63924104
C	2.27017708	-1.13646782	0.99055144
C	2.55211420	-3.43276163	-0.54667264
H	0.61160888	-2.98586228	-1.30949852
C	3.41958375	-1.89957932	1.10175161
H	2.16411775	-0.24801022	1.59606995
C	3.58790789	-3.05857388	0.32600238
H	2.66219446	-4.32529051	-1.14573831
H	4.19941261	-1.60609008	1.78964590
C	1.23267622	1.42232697	-0.48690393
C	1.54519199	2.58563107	0.23085302
C	2.09168175	1.04050840	-1.52580152
C	2.68759479	3.31518299	-0.04724380
H	0.88698219	2.91509812	1.02291908
C	3.22780539	1.77359386	-1.82269036
H	1.86287837	0.15967927	-2.10813328
C	3.55409902	2.92109475	-1.08097294
H	2.91991408	4.19932571	0.52886649
H	3.87523336	1.46472561	-2.63070035
C	-1.23812011	1.48502792	-0.05750791
C	-1.53806610	2.44469119	-1.03500339
C	-2.10410843	1.37017970	1.03794980
C	-2.67532619	3.22802537	-0.94658709
H	-0.87431033	2.56966187	-1.87936951
C	-3.23572585	2.16022409	1.14299828
H	-1.88431524	0.65412735	1.81654304
C	-3.54982366	3.09839452	0.14560977
H	-2.89851636	3.94952740	-1.71939611
H	-3.88883095	2.05899177	1.99778193
C	-4.71832129	3.89763034	0.23773981
C	-5.71791862	4.57573040	0.30628002
C	-4.80899064	-3.81656041	0.35834571
C	-5.82268317	-4.46997898	0.45418262
C	4.77121326	-3.83526570	0.42122046
C	5.78175346	-4.49677994	0.49115023
C	4.72537099	3.66848327	-1.36796102
C	5.72725190	4.30567054	-1.60018271
C	6.96618301	-5.27214681	0.56783026
C	7.12628832	-6.41979336	-0.22899957
C	8.00793981	-4.91174014	1.44084975
C	8.28029472	-7.17579671	-0.15546323
H	6.33352509	-6.70748374	-0.90434415
C	9.16175744	-5.66829640	1.51468379
H	7.89744875	-4.03371682	2.06062619
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C	-22.04371528	10.09324411	-7.55063948
H	-20.95743782	9.25809102	-5.89771018
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H	-22.80359309	18.36236763	3.26453366
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H	-24.95796503	19.54369542	3.50424725
C	-9.35364394	14.12856560	10.57942349
H	-7.93395314	14.77850792	9.10399768
H	-10.97507401	13.43611867	11.80717148
H	-23.03569759	11.20057737	-9.10264540
H	-27.08811179	18.29426148	3.31720360
H	-8.67811357	14.27318514	11.41112070

MOPT-poly

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C	17.15010749	11.02746517	19.62773229
C	15.89880500	10.23142351	19.54339790
C	15.69502710	9.14930317	20.42483227
C	14.89565994	10.51031937	18.59294979
C	14.51434258	8.41850152	20.40590808
C	13.71793699	9.77510342	18.55478814
C	13.49299788	8.72749739	19.47798995
C	18.39937698	10.23017631	19.73036984
C	18.60408364	9.13196262	18.86943020
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C	19.78333911	8.39945769	18.90584924
C	20.57530253	9.78725577	20.73561482
C	20.80173046	8.72305617	19.83203985
C	18.40422722	13.19685378	19.49531280
C	18.62372476	14.29463213	20.35318986
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C	19.80599923	15.02149173	20.30336011
C	20.57112737	13.62868518	18.46608309
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C	13.50419040	14.71485673	19.80673672
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C	32.45091291	2.79884815	20.42970352
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C	24.51067155	17.76082982	20.36458867
C	25.39630073	16.36987160	18.57550405
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H	19.24915558	11.34997618	21.37596121
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H	16.46280269	14.53792224	18.10070988
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H	23.69007957	18.02975292	21.03027807
H	25.26295214	15.56063153	17.85703133
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H	27.42262965	16.78162641	17.97815322
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H	6.86349520	6.63907041	18.21072837
H	8.47506113	4.18546071	21.37657971
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H	4.58356516	1.95277569	21.54032719
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H	2.50733488	0.60880676	21.43061511
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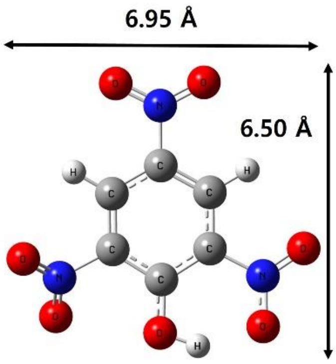
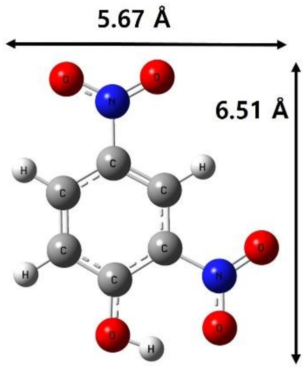
H	9.03803382	15.58518372	21.25800498
H	8.48378384	19.26416371	17.94532148
H	6.87931653	16.80463482	21.11023437
H	4.60552457	21.48783906	17.77376291
H	3.16362057	19.03552410	21.02034026
H	2.52798369	22.83091498	17.86574600
H	1.06909687	20.35710469	21.08302645

Figure S8 The cartesian coordinates (in Å) of optimized geometry for MOPT model systems.



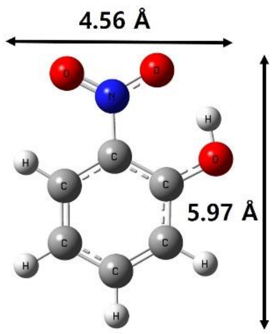
Molar volume: 112.161 cm^3/mol

TNP



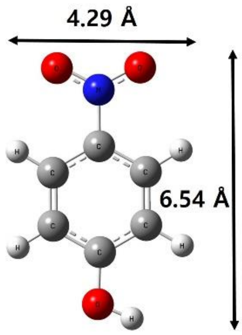
Molar volume: 96.524 cm^3/mol

DNP



Molar volume: 89.414 cm^3/mol

2NP



Molar volume: 88.423 cm^3/mol

4NP

Figure S9 The calculated molar volumes and size parameters of nitrophenol substrates used in this study at B3LYP/6-31+G(d,p) level of theory (Gaussian 09).

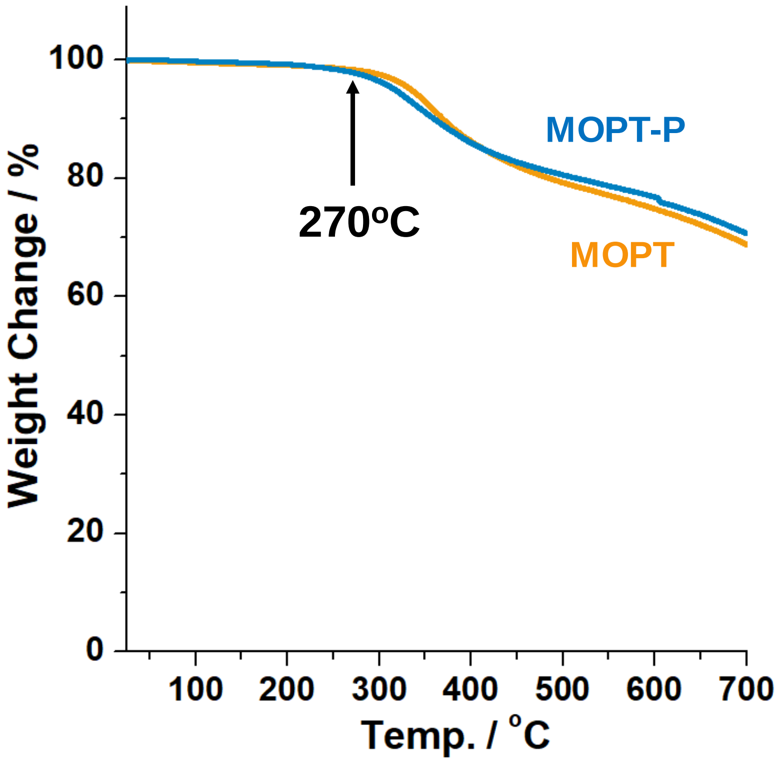


Figure S10 Thermogravimetric analysis of MOPT and MOPT-P. (PVP has been known to show good interaction with noble metals. The residual difference of MOPT-P from MOPT at 700°C may result from the residual metal catalysts. According to ICP analysis, 0.15wt% and 0.36wt% of Pd were detected for MOPT and MOPT-P. Supposing Pd species as the original $Pd(PPh_3)_4$, these values correspond to 1.58wt% and 3.89wt% $Pd(PPh_3)_4$ residues.)

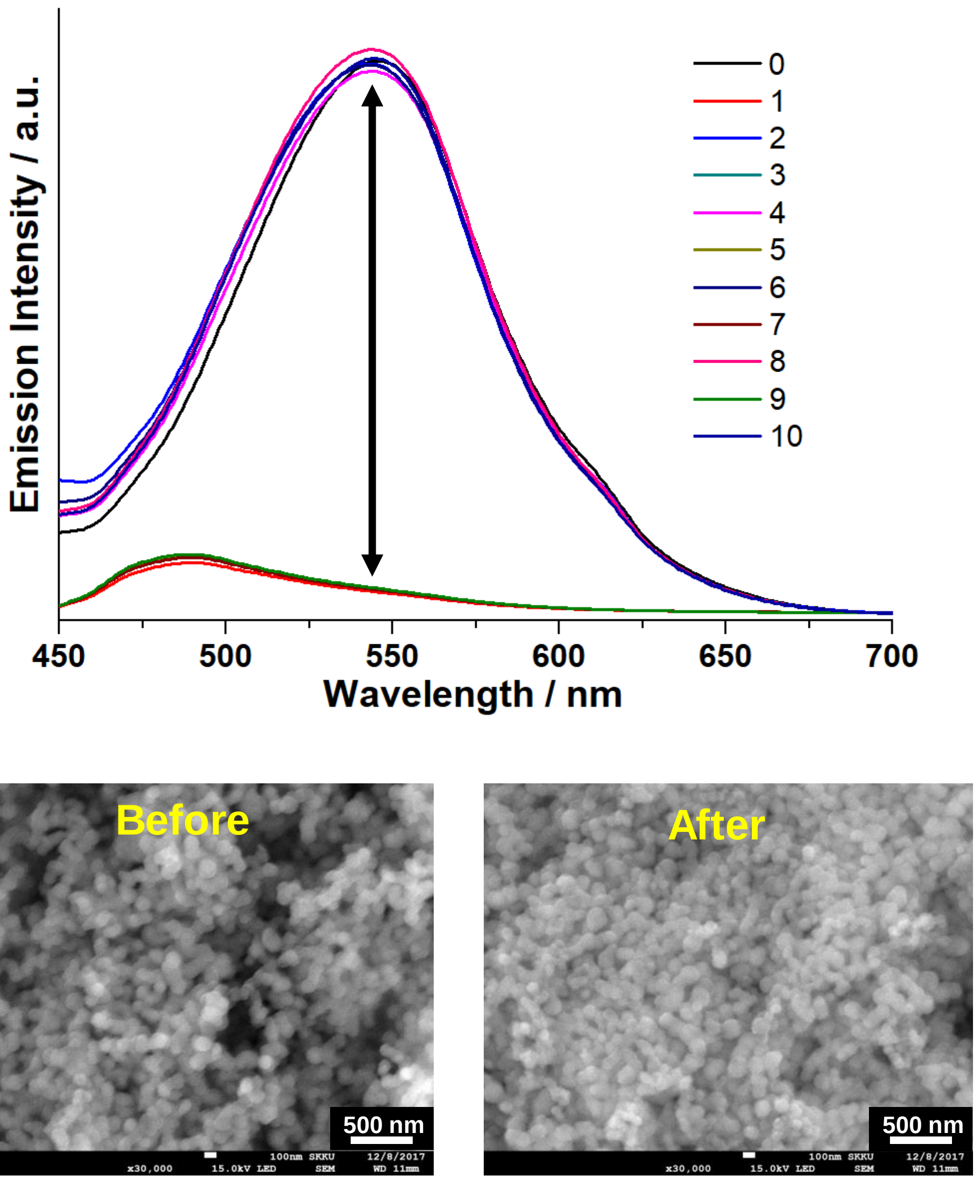


Figure S11 Emission spectra corresponding to recycle tests in Fig. 5f in text and SEM images of the original MOPT-P and the MOPT-P recovered after five successive sensing of TNP in water.

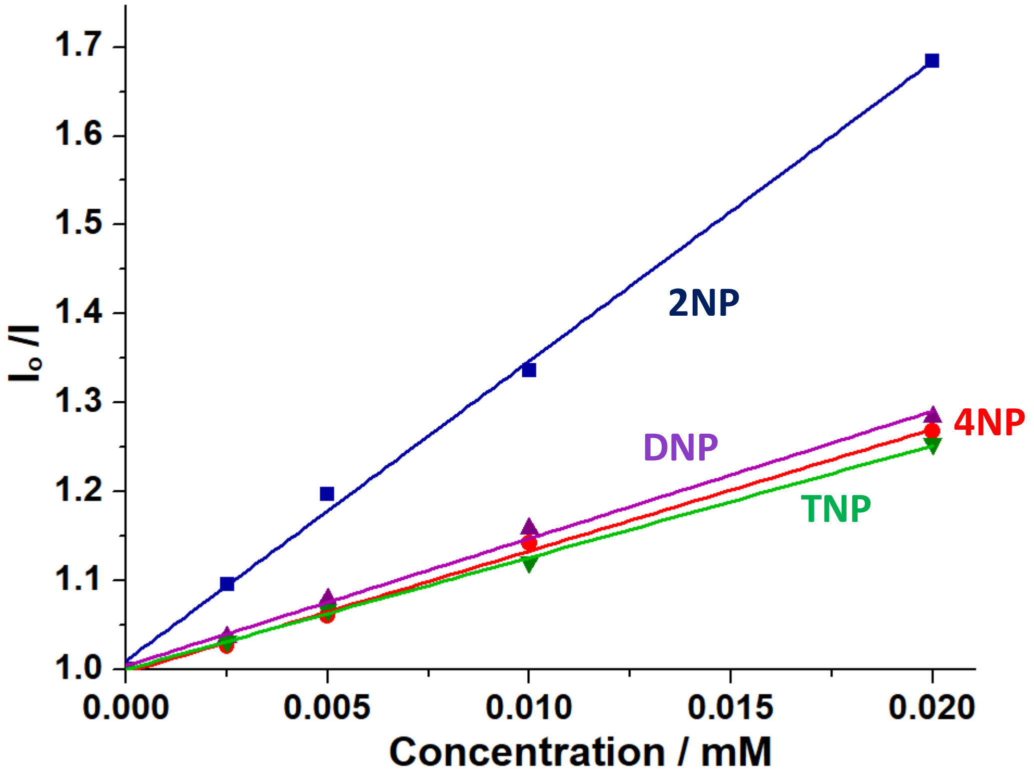


Figure S12 The Stern-Volmer plot of the emission quenching of MOPT-P by nitrophenols in water.

Table S1 Emission-based sensing parameters of MOPT-P for nitrophenols in water.[a]

Entry	Nitrophenol	K_{sv}[b] (M^{-1})	R^2	Detection Limit[c] (ppm)
1	2,4,6-Trinitrophenol (TNP)	12,600	0.998	0.23
2	2,4-Dinitrophenol (DNP)	14,300	0.995	0.27
3	4-Nitrophenol (4NP)	13,700	0.996	0.23
4	2-Nitrophenol (2NP)	33,700	0.997	0.31

[a] Sensing conditions: water (5 mL), MOPT-P (0.050 mg/ mL) [b] K_{sv} values were obtained through plotting I_o/I vs [M] based on the Stern-Volmer equation ($I_o/I = K_{sv}[M] + 1$, I_o: the original emission intensity, I: the intensity of emission in the presence of nitrophenols, M: the concentration of nitrophenols in water). c Detection limit was defined as $3\sigma/S$: σ = the standard error and S = slope for plots of $\Delta I_{emission}$ versus concentration (mM) of quenchers.

Table S2 Comparison of emission-based nitrophenols or nitroarenes sensing parameters of microporous organic polymer materials in the literature.

Entry	Materials	Solvent	Excitation Wavelength (nm)	Best K_{sv} (M^{-1})	ref.
1	Microporous polymer with Fluresceins	THF	500	2,080	ref 13
2	Microporous polymer with [tris(4-(2-thienyl)phenyl]amine)s	THF	365	1,170	ref 14
3	Microporous organic network with isocoumarins	a 2:1 mixture of H_2O and THF	440	15,000	ref 15
4	Cross-linked polymer with cyclotriphosphazene-curcumins	MeOH	372	15,200	ref 16
5	Sn-porphyrin network film	H_2O only	422	24,000	ref 17
6	Microporous polymer with carbazoles	THF	380	7,200	ref 18
7	Microporous polymer with binaphthols	Acetonitrile	468	811	ref 19
8	Micrporous polymer with dimethoxybenzenes	-	396	-	ref 20
9	Microporous polymer with tris(4-ethenylphenyl)benzenes	a 9:1 mixture of THF and H_2O	317, 350, 375	-	ref 21
10	Covalent organic polymer with trisphenylbenzenes	THF	365	14,533	ref 22
11	Microporous polymer with carbazoles	Acetonitrile	368	5,900	ref 23
12	Porous hyperbranched polymer with triphenylamines	THF	353	1,380	ref 26
13	MOPT-P	**H_2O only**	**410**	**33,700**	**This work**