Supporting Information

Metal-Organic Framework Threaded with Aminated Polymer formed In-situ for Fast and Reversible Ion-Exchange

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I) ZIF-8 synthesis, monomer confinement, polymerization and functionalization

ZIF-8 was prepared according to a reported method. Typically, 5.94 g Zn(NO$_3$)$_2$•6H$_2$O and 1.64 g 2-Methylimidazole were dissolved in 500 mL DMF solution, which was then heated to 140°C and maintained for 24 hr. The as-prepared ZIF-8 was activated by soaking in HPLC grade methanol for 2 days to exchange the trapped DMF. Then, MeOH was left to vaporize at room temperature. Finally, ZIF-8 was activated at 150°C in the open air for 1 day.

As shown in Fig. 1 in the main context, to encapsulate PVBC into ZIF-8, 10 g activated ZIF-8 was suspended in 2.5 mL dichloromethane. To this suspension, 2.5 mL vinyl benzyl chloride (VBC) with 0.12 g Azobisisobutyronitrile (AIBN, initiator) was added and the mixture was mildly grinded in a mortar until visually dry. Adding dichloromethane assists the uniform distribution of VBC as well as AIBN solubility. Dichloromethane was subsequently removed under vacuum at 20°C. Once dichloromethane was dried, the sample was equilibrated for 1 month to allow the penetration of vinylbenzyl chloride, as the diffusion of VBC into ZIF-8 can be very slow (see ‘Theoretical Calculations’ in Section III below). Afterwards, VBC filled ZIF-8 was sealed in a glass vial and heated to 90°C under N$_2$ atmosphere for 3 days. The sample obtained was refluxed in THF overnight to remove the unattached PVBC or remained monomer. The powder was then collected, washed with ethanol and dried at 60°C to give PVBC~ZIF-8 composite.

Amination was conducted in a three-neck flask, with typically 6 g PVBC~ZIF-8 and 10 mL THF. Then, 2 mL 40 wt.% trimethyl amine aqueous solution was added with mild stirring, and the solution was refluxed for 3 days. Finally, the aminated sample was filtrated and washed intensively with water and methanol, to obtain PVBTAC~ZIF-8.

The ion-exchange capacity of PVBTAC~ZIF-8 was determined on a dry weight basis by a traditional titration method. Typically, 0.5 g PVBTAC~ZIF-8 was suspended in 5 mL DI water. To this suspension, 0.3 g NaNO$_3$ was added and kept stirring for 2 hr. Then, the mixture was titrated by 0.01M AgNO$_3$ solution with potassium chromate as indicator. The ion-exchange was carried out by soaking the PVBTAC~ZIF-8 in excessive 1M NaOH aqueous solution at 25°C typically for 24hr to give PVBTAH~ZIF-8 (H stands for hydroxide group) until no chloride can be detected by EDX.
II) SEM and EDX characterization

Field emission scanning electron microscopy (FE-SEM, Hitachi S-4800) was used to characterize the morphology (5kV accelerating voltage, gold coating) and element analysis (at 20kV accelerating voltage) of ZIF-8 and polymer~ZIF-8 composites.

![Figure S1. (a) SEM images of PVBTAH~ZIF-8 and (b) Amberlyst A-26 resin after grinding.](image1)

![Figure S2. SEM image (EDX mode) and EDX mapping of PVBC~ZIF-8 composite for Cl and Zn.](image2)
III) Theoretical calculations

Methodology
A complete cavity, with the chemical formula of C_{96}H_{120}N_{48}Zn_{12}, was used as the unit cell to model the crystal of ZIF-8 in our calculations. All the electronic structure calculations were carried out with the CP2K code\(^2\) using the PBE exchange-correlation functional.\(^3\) We adopted the Gaussian and Plane-wave (GPW) hybrid basis set scheme.\(^4\) Core electrons were modeled as norm-conserving pseudopotentials of the Goedecker-Teter-Hutter (GTH) type\(^5\) and the wave functions expanded in a double-Zeta Gaussian basis set with a plane wave auxiliary basis of 280 Ry energy cutoff. Integration over the Brillouin zone was performed with a reciprocal space mesh consisting of only the Gamma point. Since PBE functional is unable to describe correctly the dispersion energy, we performed DFT-D approach\(^6\) to estimate the van der Waals interactions between reactants. The climbing image nudged-elastic band (CI-NEB) method\(^7\) was utilized to search the transition states for the penetration of VBC into the cavity of ZIF-8.

Accessibility of VBC molecule to ZIF-8
First, the accessibility of VBC molecule (4.3 x 8.2 Å) to the internal surface of ZIF-8 must be demonstrated. ZIF-8 possesses large spherical cavities with diameter of 1.2~1.5 nm. Even though its apertures size is much smaller than spherical cavities (See Figure S2), several experimental cases have already confirmed that the gate-opening effect of ZIF-8 allows the penetrations of guest species with dynamic sizes comparable to or even larger than the windows size (e.g. nickelocene\(^8\), Methyl benzoate (7.8 x 4.3 Å)\(^9\), caffeine (6.1 x 7.6 Å)\(^10\), furfuryl alcohol (8.4 x 6.4 x 4.3 Å)\(^11\), para-xylene (6 x 4.3 Å)\(^12\), Tetrabutylammonium hydroxide (~9 x 9 Å)\(^13\), and 1,2,4-trimethylbenzene\(^14\). It was argued that the so-called gate-opening effect can
cause a complete tilt of the 2-methylimidazolat ligands, so the aperture diameter of ZIF-8 increase significantly.\textsuperscript{12,14}

Theoretical DFT calculations demonstrated there are feasible pathways for the penetration of VBC molecule into the cavity of ZIF-8, in agreement with our experimental results and previous reports. As shown in our climbing image nudged-elastic band (CI-NEB) calculations (Fig. S5), the penetration barrier for VBC molecule attracted into ZIF-8 is not high, only at 18.8 kcal/mol. Furthermore, the thermodynamic data suggests that this process is exothermic, at 3.7 kcal/mol.

As shown in Figure S4, VBC can apparently fit the window size of ZIF-8, although the diffusion into the pores can be slow (diffusion activation energy is estimated to be ca. 78 kJ/mol). In addition, for ZIF-8, even \text{N}_2 filling can open the ‘gate’ of ZIF-8 in the same way as it is at a very high pressure,\textsuperscript{15} it is therefore reasonable to expect the accessibility of ZIF-8 can be improved due to the gate open effect. In order to further investigate whether VBC can fit the aperture of ZIF-8 in terms of molecular size, we tried to fill ZIF-8 with liquid 1,4-diethylbenzene, which undoubtedly has even larger dynamic size than VBC. The activated ZIF-8 particle was soaked in 1,4-diethylbenzene at room temperature for one month. Afterwards, the obtained solid was quickly washed with dichloroform and acetone to remove 1,4-diethylbenzenes attached on the external surface. The solid was dried overnight under vacuum for TGA test. After 1,4-diethylbenzene adsorption, TGA test clearly shows about 25wt.% weight loss in the temperature range from 260 °C to 300 °C, which can be contributed to the removal of 1,4-diethylbenzene (Figure S4).\textsuperscript{*}

\begin{figure}[h]
\centering
\includegraphics[width=0.5\textwidth]{vbc_molecule_size}
\caption{VBC molecule size}
\end{figure}

\textsuperscript{*} The boiling point of 1,4-diethylbenzene is ca. 180°C (1 atm), much lower than the observed onset temperature for the weight loss (260°C). This is due to the fact that molecules confined within micropores require extra thermal energy to get out of pore especially as the molecular size is close to the aperture size of pore.
Figure S4. ZIF-8 framework corresponding pore openings (unit in Å) via (a) (100) and (b) (111) directions, respectively. The window sizes are basically consistent with previous reports. \(^\text{16, 17}\)

Figure S5. Climbing Image Nudged elastic band (CINEB) calculations for the penetration of VBC into ZIF-8. Energies are in kcal/mol.

In addition, the configuration/position of vinylbenezyl trimethyl ammonium chloride (VBTAC), the aminated monomer within the ZIF-8 cavity was simulated to visualize the accessible volume (Figure S6). The cif file of this 3D structure is included for easy reference.
Figure S6. Cavity of ZIF-8 with a vinylbenzyl trimethyl ammonium chloride (VBTAC) filled. The atom positions were optimized by CP2K code. The carbon atoms and bonds of vinylbenzyl trimethyl ammonium chloride were present by yellow spheres and red lines, respectively. Hydrogen atoms are removed from these figures for ease of reading.

IV) X-ray diffraction and refinement study

X-ray diffraction (XRD) patterns were obtained by a Bruker Advance D8 diffractometer (CuKα) with a lynxeye detector at 40 kV and 40 mA at a scanning rate of 4 °/min and 2 theta range of 5~60°. XRD peaks indexing was performed by DICVOL\textsuperscript{18} program. For Pawley fitting, first, a Pseudo-Voigt profile function was used for the profile fitting and Berar-Baldinozzi function for asymmetry; then, the lattice parameters were refined. Finally, all of these parameters were refined at the mean time. All refinements were performed until no improvement was observed for the profile factor ($R_p$).

Figure S7. (a) XRD patterns for ZIF-8 and PVBC-ZIF-8 composite; (b) Refined ZIF-8 and PVBC-ZIF-8 XRD patterns. The lattice parameters of PVBC-ZIF-8 were refined to be $a=b=c=17.097$ Å and $\alpha=\beta=\gamma=90^\circ$, which correspond with a cell volume of 4997.6 Å$^3$. Those obtained with the ZIF-8
prepared here were $a=b=c=17.019 \, \text{Å}, \, V = 4929.5 \, \text{Å}^3$. This corresponds to expansion of ZIF-8 lattice, upon penetration of PVBC.

![Figure S7(c). XRD patterns of ZIF-8, PVBTAC~ZIF-8, PVBTAH~ZIF-8 and gold cyanide loaded PVBTAH~ZIF-8 composites.](image)

V) Molecular weight of PVBC from PVBC~ZIF-8

Molecular weights of PVBC with ZIF-8 removed were tested against polystyrene standards on a Waters GPC equipment coupled with two Styragel HR3 and HR4 columns at 60°C. The detector is Waters 996 photodiode array, and 2410 refractive index detectors. N-methylpyrrolidinone (NMP) with 4% potassium hexafluorophosphate was used as the eluent with a flow rate of 0.8 mL/min. ZIF-8 from PVBC~ZIF-8 was first removed by HCl, IR detector. Then, PVBC was extracted by CH$_3$Cl and washed with diluted HCl for several times. From the GPC spectrum in Fig. S8, the first peak at 24 min can be assigned to PVBC and the peak appeared after 25 min can be assigned as the solvent peak.
VI) Miscibility of ZIF-8, PVBC~ZIF-8 and PVBTAC~ZIF-8

Figure S9. (Right to left) Physical observation for change of miscibility in octane and water phases from neat ZIF-8 to PVBTAC~ZIF-8.
VII) FTIR spectrum of ZIF-8, PVBC~ZIF-8 and PVBTAC~ZIF-8

Fourier transform infrared spectroscopy (FTIR, Shimadzu, KBr pellet) was performed at 20°C at a 4 cm⁻¹ resolution.

![FTIR spectra](image)

Figure S10. (a) FTIR spectra for neat ZIF-8, PVBC~ZIF-8 and PVBTAC~ZIF-8 composites; (b) Enlarged section of the spectra of the specific wavelength range from 660 to 740 cm⁻¹ to illustrate the disappearance of C-Cl bond after amination.

VIII) NMR of PVBTAC isolated from PVBTAC~ZIF-8

Nuclear magnetic resonance (NMR) spectra were recorded by a Bruker 400M equipment (tetramethylsilane as reference for chemical shift) and deuterated water was used as solvent. Isolation of accommodated polymer from PVBTAC~ZIF-8 was conducted by removing ZIF-8 using 1M HCl, and then precipitating out the PVBTAC by adding excessive amount of HPLC acetone.

![NMR spectra](image)

Figure S11. ¹H NMR in D₂O for synthetic PVBTAC (red line) and isolated PVBTAC from PVBTAC~ZIF-8 (black line).
IX) \( \text{N}_2 \) sorption curves and pore size distribution

Brunauer–Emmett–Teller (BET) surface areas and porosity were measured by Micromeritics ASAP 2020 analyzer with nitrogen at 77K. For each run, ~0.2 g sample was evacuated in a vacuum oven at 60°C overnight. The sample was then transferred to pre-weighed quartz tube and degassed at 60°C until the system pressure was < 5 μmHg. Afterwards, the tube was reweighed to obtain an accurate mass for gravimetric BET surface area determination. All data are calculated by ASAP 2020 software.

Figure S12. \( \text{N}_2 \) sorption curves of (a) PVBC and PVBC~ZIF-8, and (b) PVBTAC~ZIF-8 and PVBTAH~ZIF-8.

Figure S13. Pore size distribution of ZIF-8 (black line) and PVBTAH~ZIF-8(red line) calculated by Horvath-Kawazoe (HK) method in the pressure range of log \((p/p_0)\) = -3.0 to -5.0 (slit type pore and zeolite interaction potential).
**Table S1. Calculation of Apparent Volume of PVBC in PVBC~ZIF-8**

<table>
<thead>
<tr>
<th></th>
<th>ZIF-8</th>
<th>PVBC~ZIF-8</th>
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<tbody>
<tr>
<td>Single point adsorption</td>
<td>at p/p° = 0.9743:</td>
<td>at p/p° = 0.97521:</td>
</tr>
<tr>
<td>total pore volume</td>
<td>0.4800 cm³/g</td>
<td>0.25601 cm³/g</td>
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<tr>
<td>BJH Adsorption cumulative</td>
<td>0.001793 cm³/g</td>
<td>0.054301 cm³/g</td>
</tr>
<tr>
<td>volume 17.0 Å &lt; pore</td>
<td></td>
<td></td>
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<tr>
<td>width &lt; 3000.0 Å</td>
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<td></td>
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<tr>
<td>Total Pore Vol. –</td>
<td></td>
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<tr>
<td>Mesopore Vol</td>
<td>0.4782 cm³/g</td>
<td>0.2017 cm³/g</td>
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<td>t-Plot micropore volume:</td>
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<tr>
<td>up to p/p° = 0.0104</td>
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<td></td>
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<tr>
<td>(pore width 7.19 Å)</td>
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<tr>
<td>0.48107 cm³/g</td>
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<td>HK model pore volume</td>
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<tr>
<td>up to p/p° = 0.12195</td>
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<td>(pore width 12.81 Å)</td>
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<td>0.2268284 cm³/g</td>
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<td>Loading of polymer</td>
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<tr>
<td>0</td>
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<td>Mass of ZIF-8 per gram</td>
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<td>of material</td>
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<td>Mass of PVBC in</td>
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<td>0.14 g</td>
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<tr>
<td></td>
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<td>(e)</td>
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<tr>
<td>Micropore volume of ZIF-8</td>
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<td>(f)</td>
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<td>Volume of ZIF-8 if</td>
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<tr>
<td>not occupied,</td>
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<tr>
<td>cm³ per gram of</td>
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<tr>
<td>composite</td>
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<tr>
<td>(f)x(d)</td>
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<tr>
<td>Based on (total -</td>
<td>0.4782 cm³/g of ZIF-8</td>
<td>0.41125 cm³/g of ZIF-8</td>
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<tr>
<td>mesopores)</td>
<td>(g)</td>
<td>0.41125 (g)</td>
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<tr>
<td>Based on t-plot</td>
<td>0.49784 cm³/g of ZIF-8</td>
<td>0.2268284 cm³/g of ZIF-8</td>
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<tr>
<td></td>
<td>(h)</td>
<td>0.2268284 (h)</td>
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<tr>
<td>Based on HK model</td>
<td>0.48107 cm³/g of ZIF-8</td>
<td>0.41372 cm³/g of ZIF-8</td>
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<td></td>
<td>(i)</td>
<td>0.41372 (i)</td>
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<td>PVBC Apparent</td>
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<td>specific volume</td>
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**X) Thermogravimetric analyses (TGA)**

Thermal gravimetric analysis was performed at a 20 mL/min gas flow with a heating rate of 5°C/min.

![TGA curve](image)

Figure S14. TGA curves for (a) ZIF-8 filled with 1,4-diethylbenzene, (b) PVBC~ZIF-8, (c) PVBTAC~ZIF-8 and PVBTAH~ZIF-8.
XI) DSC profiles of PVBC-ZIF-8, and mixture of ZIF-8 and bulk PVBC

Differential scanning calorimetry (DSC) was performed under 20 mL/min Ar flow. To identify the glass transition temperature, the samples are preceded to 150°C and then cool down to 25°C. Preheating treatment was conducted to eliminate the thermal history and confirm the reversible thermal behavior. Subsequently, DSC data was recorded from 25 to 150°C at a ramping rate of 20°C/min.

Figure S15. DSC profiles of (a) PVBC-ZIF-8 and bulk PVBC prepared in solution method; (b) mixture of ZIF-8 and bulk PVBC, where the PVBC has the same weight ratio (14 wt.%) to that of host-guest PVBC-ZIF-8. Control experiment, DSC curve of ZIF-8, is included as inset.

XII) Temperature-programmed-desorption (TPD) of Amberlyst-A26, PVBTAH-ZIF-8, and PVBTAC-ZIF-8

Temperature-programmed-desorption (TPD, HidenCatlab) system with an on-line mass spectrometer (QGA, Hiden) was utilized to monitor the gaseous components with Ar (50 mL/min) as a carrier gas. For temperature scanning profiles, the temperature was increased from 37°C to 200°C at a ramping rate of 5°C/min.
Figure S16. (a) TPD-MS for Amberlyst-A26, (b) PVBTAH-ZIF-8, and (c) PVBTAH-ZIF-8. The mechanism of degradation is summarized in Scheme S1.

Scheme S1. Hofmann degradation mechanism for strongly basic ion-exchange polymer in OH form.
XIII) Ion-exchange with nitrate ions

A fixed amount of each ion-exchange materials with 0.3mM OH⁻ group was immersed in 0.5mM nitrate solution. UV-Vis absorption spectra of the nitrate ion exchange progress for Amberlyst-A26 and PVBTAH–ZIF-8 are summarized in Fig. S17. The absorption peak at 301 cm⁻¹ is assigned to the NO₃⁻ anion.

![Absorption spectra](image)

Figure S17. UV-Vis absorption spectra of the anion exchange progress for (a) Amberlyst-A26, and (b) PVBTAH–ZIF-8 (0.5mM nitrate vs. 0.3mM OH groups of ion-exchange materials); (c) Control experiment of neat ZIF-8, NO₃⁻ anion trapping or exchange was not observed. The solution used in the nitrate exchange was 0.1M NaNO₃ solution.
Figure S18. Normalized UV-Vis absorption spectra of the anion exchange progress for Amberlyst-A26 and PVBTAH-ZIF-8 for comparison. Inset: Normalized UV-Vis absorption spectra for the first 60 min.

Note: The particle size of PVBAH-ZIF-8 is on the order of tens of micrometer, similar to that of grinded Amberlyst A26, as seen in Fig. S1. Assuming a 10 nm thick active surface layer, this is only 0.5% the volume of a 6 micrometer diameter particle. In Fig. S18, 54% of the 0.5 mM nitrate solution was adsorbed by the PVBAH-ZIF-8 which is added at 0.3 mM OH⁻ equiv. This represents a 93% utilization of the total capacity of PVBAH-ZIF-8 and cannot be restricted to surface layer activity.

XIV) Gold cyanide ion sorption and stripping experiments

For gold cyanide extraction, 21.96 g of potassium gold(I) cyanide, KAu(CN)₂ (Kenlap PCG Manufacturer Company Limited) was dissolved to 1L DI water at 25°C. Then, the prepared stock solution was diluted by 1000 times to give diluted solution with 15ppm gold cyanide concentration. For extraction, 2 g PVBAH-ZIF-8 (~1.86mmol OH⁻) or Amberlyst-A26 (~4.4mmol OH⁻) was soaked in 1L 15ppm KAu(CN)₂ (~0.002 mmol Au(CN)₂⁻) stock solution. Inductively coupled plasma mass spectrometry (ICP-MS, Agilent 7500a) was used to quantify the gold cyanide amount for the ion exchange study.

The ion exchange reaction between gold cyanide ion Au(CN)₂⁻ and PVBAH-ZIF-8 follows Scheme S2:
Scheme S2. Gold cyanide sorption and elution mechanism.

For gold cyanide ion stripping, 0.1g gold cyanide loaded PVBTAH~ZIF-8 (0.0061 mmol/g) was soaked in 42 mL stripping solution. Striping solutions include 1M NaOH ethanolic solution (water : ethanol, 1:1 v/v) and the gold cyanide ion elution efficiency is calculated according to Equation (1) and quantified by ICP-MS as a function of time.

\[
\text{Elution efficiency (\%) = } \frac{\text{gold stripping amount}}{\text{gold extraction amount}} \times 100
\]  

(1)

FTIR was used to confirm the gold cyanide ion sorption properties of PVBTAH~ZIF-8. 0.05 g PVBTAH~ZIF-8 was soaked in 133 mL 15ppm KAu(CN)_2 stock solution overnight. Then, the loaded ZIF-8/PVBTAH was filtered off and washed several times with DI water and dried under dynamic vacuum at room temperature. As control experiment, the neat ZIF-8 without loading ion-exchange polymer was also soaked in KAu(CN)_2 under identical conditions, and characterized by FTIR.
Figure S19. FTIR for PVBTAH~ZIF-8 (red line), ZIF-8 soaked in KAuCN$_2$ solution and PVBTAH~ZIF-8 soaked in KAuCN$_2$ solution.

Figure S20. Gold cyanide ion elution efficency from the gold cyanide loaded PVBTAH~ZIF-8.
Table S2. MOFs that can be potentially used to confine cation exchange polymers

<table>
<thead>
<tr>
<th>MOFs</th>
<th>Stability in solution</th>
<th>References</th>
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<tbody>
<tr>
<td>Zn₃(BTP)₂</td>
<td>pH =2~14 for two weeks at 100°C</td>
<td>Colombo, V.; Galli, S.; Choi, H. J.; Han, G. D.; Maspero, A.; Palmisano, G.; Masciocchi, N. and Long, J. R.; Chem. Sci., 2011, 2, 1311</td>
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