# Supporting Information

*for*

## Compressible Porous Hybrid Monoliths: Preparation via A Low-molecular Mass Gelators-based Gel Emulsion Approach and Exceptional Performances

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**Figure S2** FTIR spectra of **BuDphe**

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**Video S4** The absorption–squeezing processes with M-5 as a sample absorbent

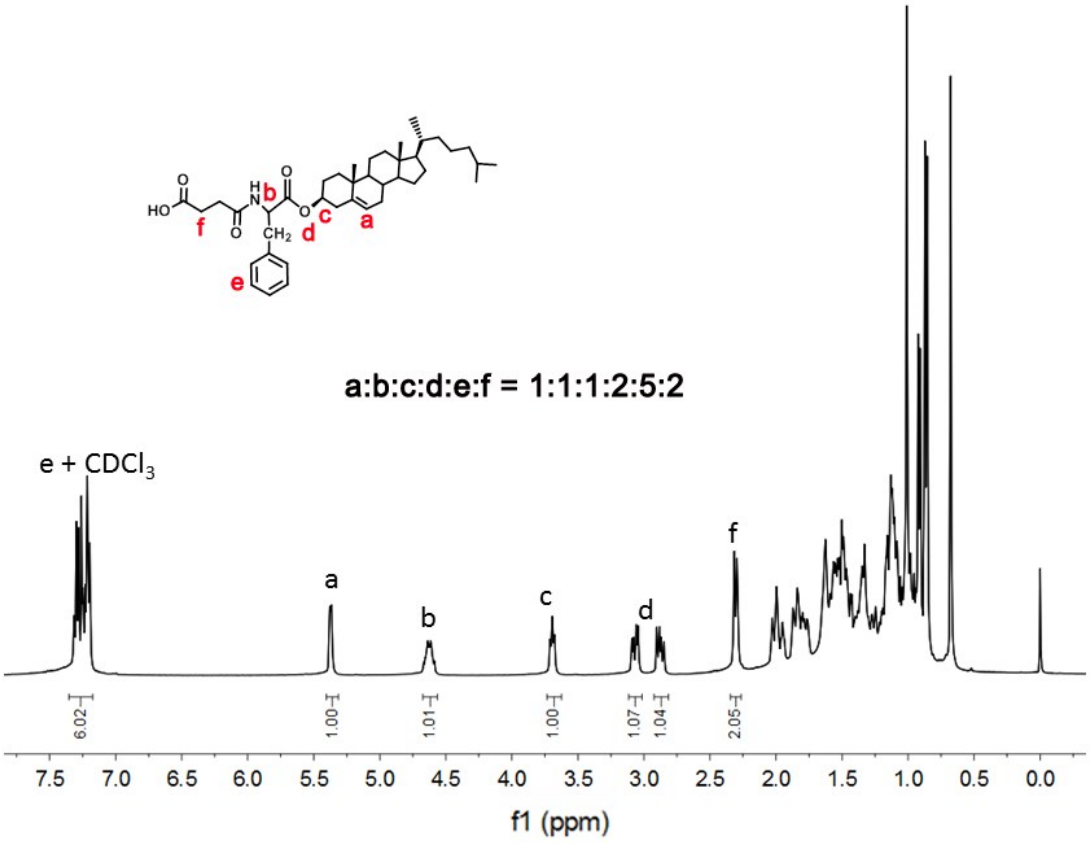



**Figure S1** $^{1}$H NMR spectra of **BuDphe**

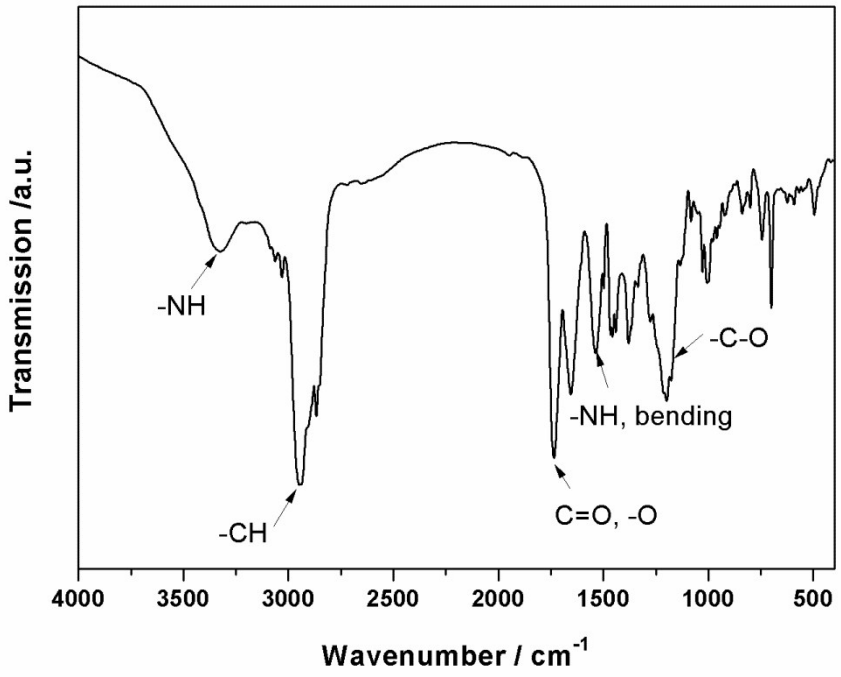



**Figure S2** FTIR spectra of **BuDphe**

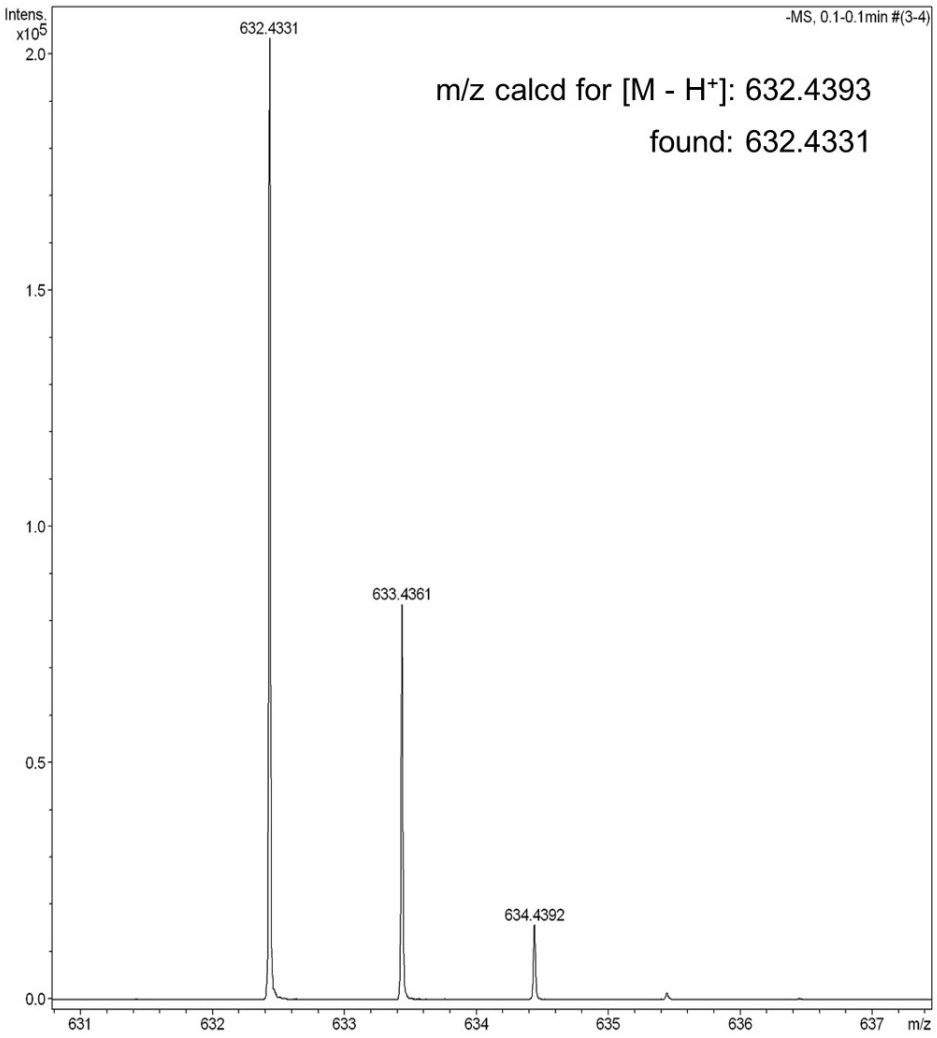



**Figure S3** MS spectra of **BuDphe**

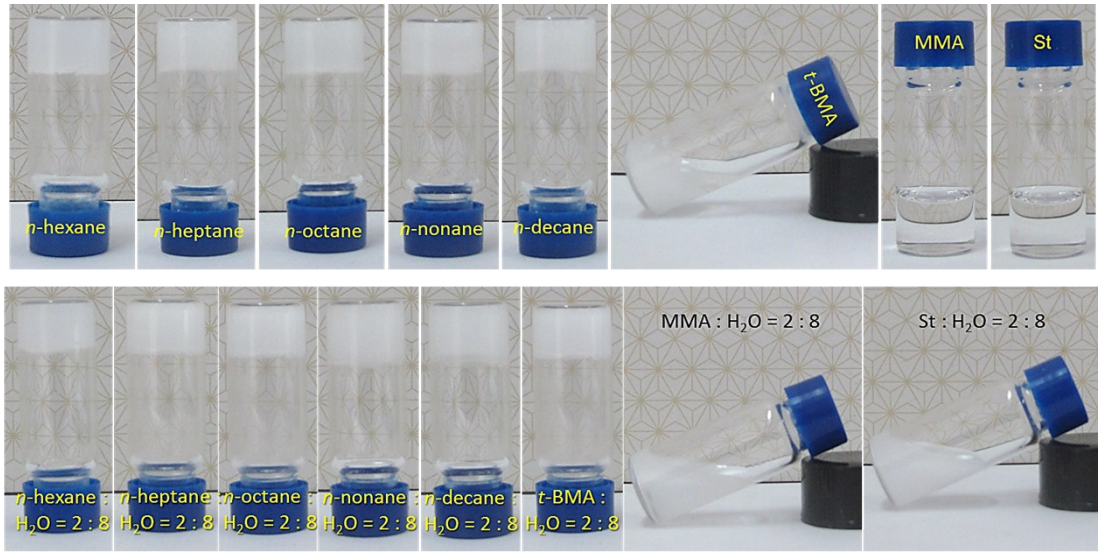



**Figure S4** Gelation behaviors of BuDphe in various solvents

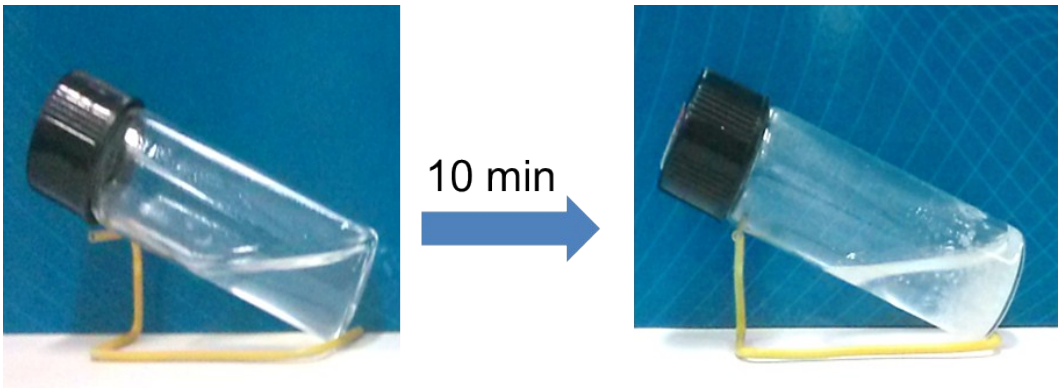



**Figure S5** Partial gelation of **BuDphe**/*t*-BMA (2%, *wt*%) after 10 minutes standing

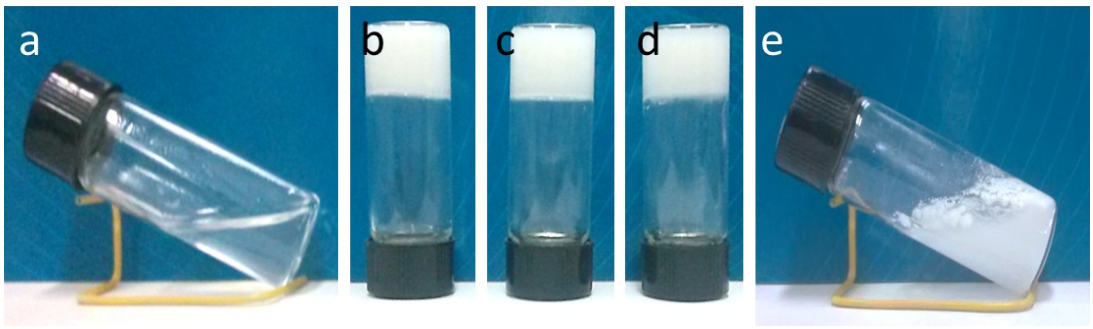



**Figure S6** Phase behavior of BuDphe /water /*t*-BMA (total volume is 1 mL) with different water contents: (a) 0%, (b) 20%, (c) 50%, (d) 80%, (e) 100% (v/v)

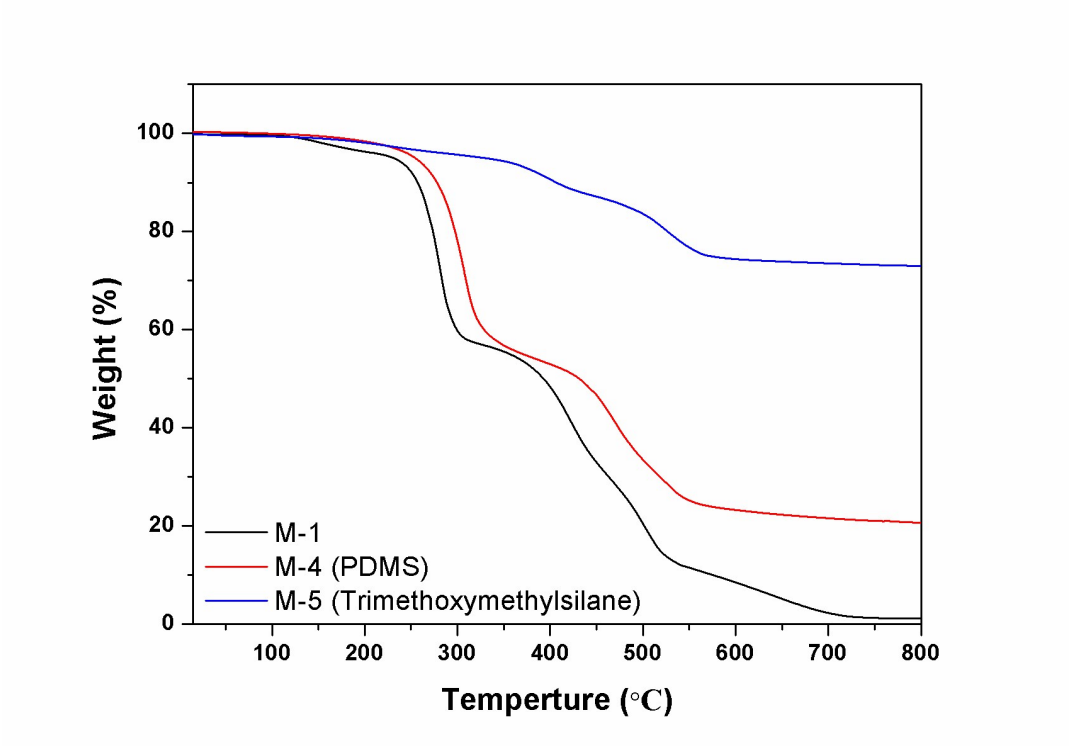



**Figure S7** TGA curves of M-1 (black line), M-4 (red line), M-5 (blue line) under oxygen flow

**Calculation of the Theoretical Porosities**

Theoretical values of the porosities ($\Phi_t$) of the materials have been calculated by 1) measuring their total volume ($V$), 2) weighing their weight ($W$), 3) measuring the weight of "silica" in the materials ($W_1$), and then 4) using equation of $\Phi=[V-W_1/\rho_1-W_2/\rho_2-(W-W_1-W_2)/\rho]/V$, where $\rho$ and $\rho_1$ are the density of the polymeric materials, and that of silicon oxide, $\rho_2$ and $W_2$ are the density and weight of "PDMS". It is to be noted that the density of the monomer of the polymer in liquid state at 20℃ and that of silica were adopted as approximate values of the organic part and inorganic part of the materials, respectively.

**Table S1** Theoretical values of the porosities of the porous materials

| Porous Monoliths | Porosities ($\Phi_t$) | Total Volume ($V$, cm$^3$) | Total Mass ($W$, g) | The density of the pure polymers ($\rho$, g/cm$^3$)* | The weight of PDMS ($W_2$, g) | The volume of MTMS ($W_1$, g) |
|---|---|---|---|---|---|---|
| **M-1** | 0.80 | 1 | 0.18 | 0.9036 | 0 | 0 |
| **M-2** | 0.77 | 1 | 0.21 | 0.9036 | 0.004 | 0 |
| **M-3** | 0.74 | 1 | 0.24 | 0.9036 | 0.01 | 0 |
| **M-4** | 0.73 | 1 | 0.25 | 0.9036 | 0.02 | 0 |
| **M-5** | 0.71 | 1 | 0.27 | 0.9036 | 0.02 | 0.15 |

* $\rho \approx (m_{(t\text{-BMA})} + m_{(\text{DVB})})/(V_{(t\text{-BMA})} + V_{(\text{DVB})})$

$W_{(t\text{-BMA+DVB})} = \rho_{(t\text{-BMA})} \times V_{(t\text{-BMA})} + \rho_{(\text{DVB})} \times V_{(\text{DVB})}$

$\rho_{(t\text{-BMA})} = 0.8940$ g/cm$^3$, $\rho_{(\text{DVB})} = 0.9325$ g/cm$^3$, $\rho_{(\text{PDMS})} = 1.00$ g/cm$^3$, $\rho_{(\text{MTMS})} = 0.95$ g/cm$^3$

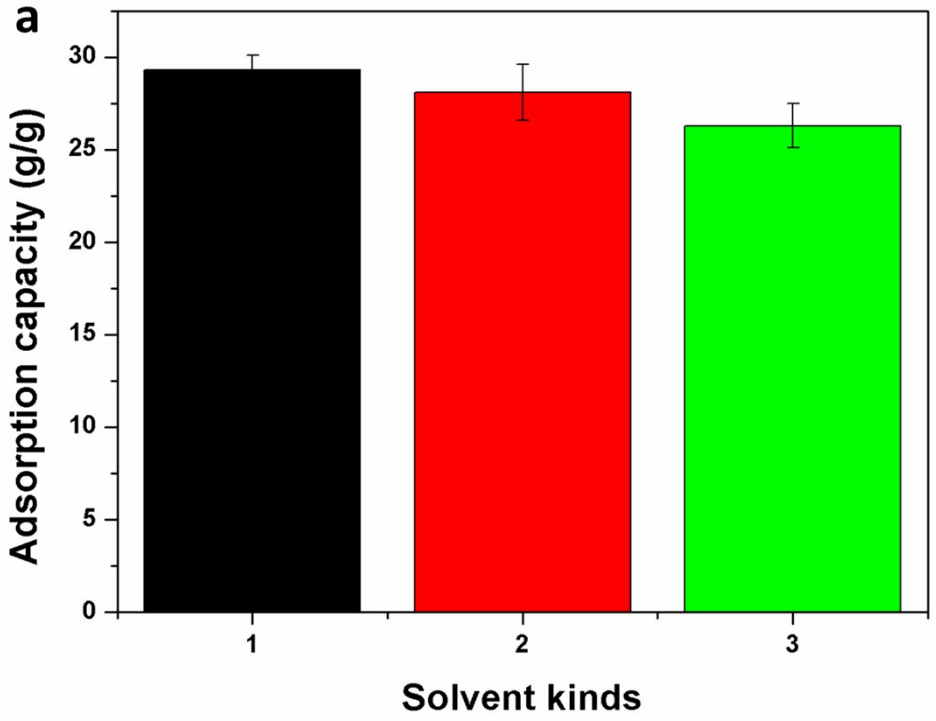
$V_{(t\text{-BMA})} : V_{(t\text{-BMA})} = 3{:}1$, $V_1 = W_{(\text{PDMS})} / \rho_{(\text{PDMS})}$

**Table S2** Experimentally determined porosities of the porous polymeric materials

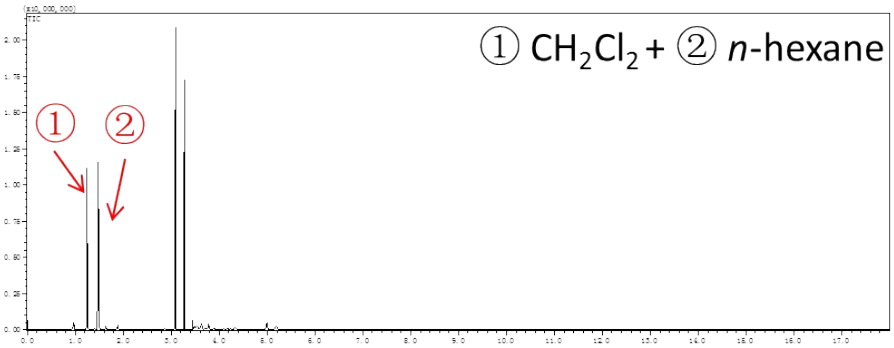
| Porous Monoliths | Porosities (Φ) | $m_2$ (g) | $m_1$ (g) | The density of bromobenzene ($\rho_1$, g/cm$^3$) | d (cm) | h (cm) |
|---|---|---|---|---|---|---|
| **M-1** | 0.89 | 1.16 | 0.14 | 1.50 | 1.1 | 0.8 |
| **M-2** | 0.90 | 1.34 | 0.18 | 1.50 | 1.1 | 0.9 |
| **M-3** | 0.87 | 1.41 | 0.22 | 1.50 | 1.2 | 0.8 |
| **M-4** | 0.89 | 1.53 | 0.25 | 1.50 | 1.1 | 1.0 |
| **M-5** | 0.84 | 1.91 | 0.34 | 1.50 | 1.2 | 1.1 |

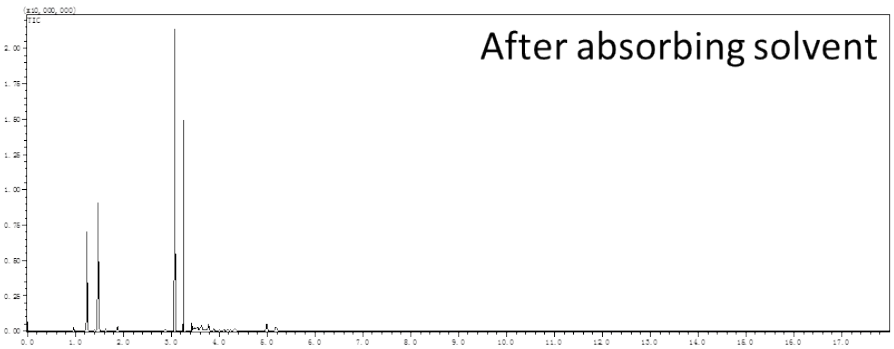
Generally speaking, with exception of specific interactions, the selectivity of this kind of porous materials to solvents is mainly reflected in the polarity of the solvents because solvents of different polarities possess different affinity to the inner-surfaces of a given materials. Therefore, the materials as prepared only tested for their selectivity to organic solvents from water, which can be estimated by conducting contact angle measurements.

As for the selectivity of the materials to different organic solvents, three sets of additional experiments were conducted, of which the sample used for first set test is a mixture of $CH_2Cl_2$ and n-hexane, the second a mixture of $CH_2Cl_2$, n-hexane and benzene, and the last a mixture of $CH_2Cl_2$, n-hexane, benzene and THF. The results are shown in Figure S8. It is seen that for each set of experiment, the original composition of the mixture is almost the same to that of the solvents collected from the bulk monolith (M-5) after absorption, indicating that the materials shows little selectivity to the solvents mixtures no matter they are binary, ternary, or quaternary mixtures. Actually, this is a result not difficult to understand because all the solvents tested possess good affinity to the monolith under study.

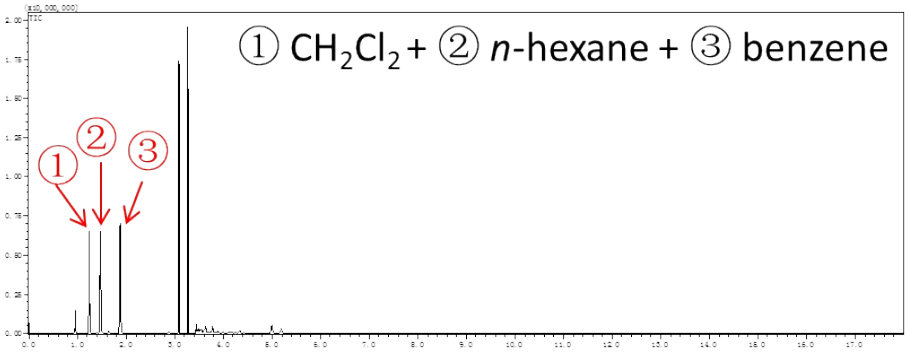


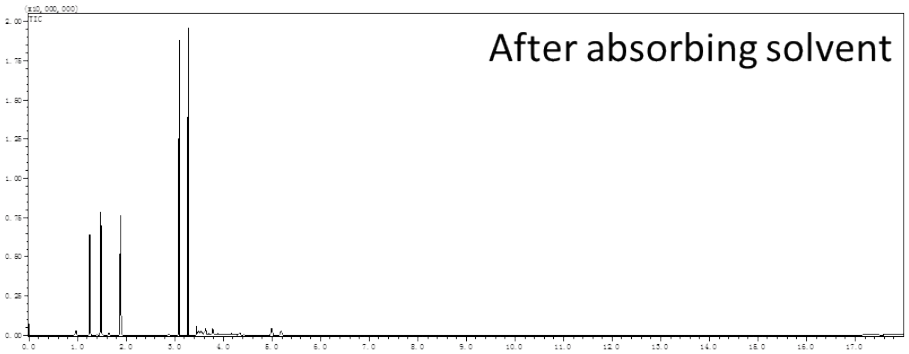
**b** (1)


① $CH_2Cl_2$ + ② *n*-hexane
①
②


After absorbing solvent

(2)


① $CH_2Cl_2$ + ② *n*-hexane + ③ benzene
①
②
③


After absorbing solvent

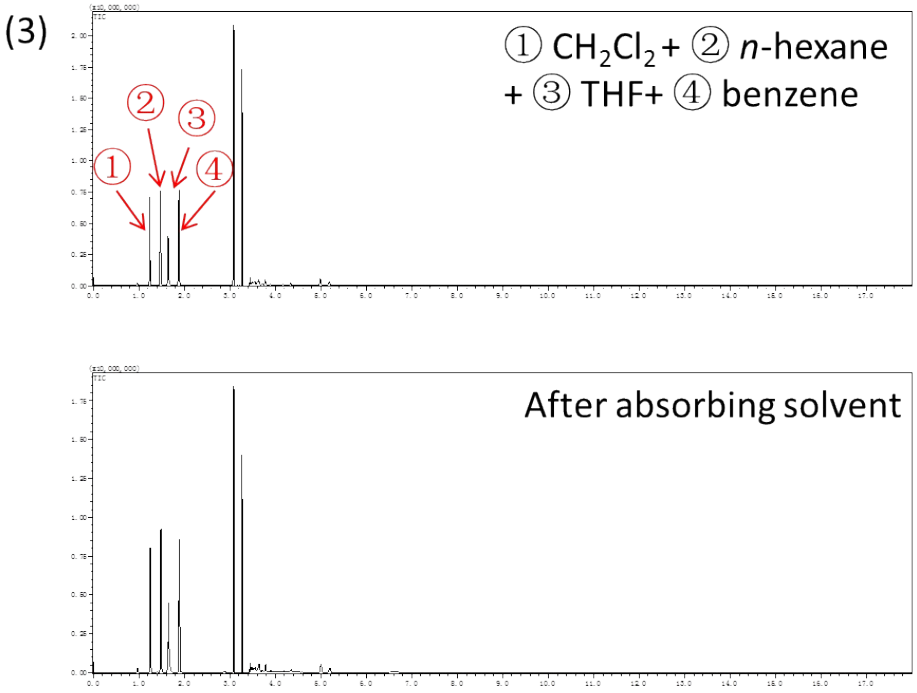
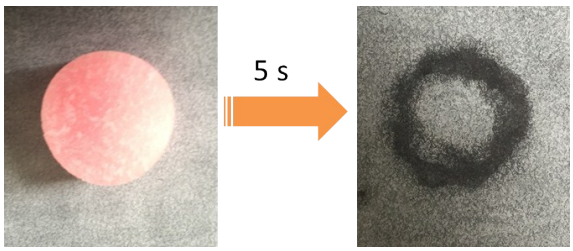



**Figure S8** (a) Maximum absorption capacities of M-5 to some organic-solvent mixtures*; (b) Selectivity of the materials to the solvents* under test, results from GC-MS studies

* a1, b1: $CH_2Cl_2$:*n*-hexane = 1 : 1; a2, b2: $CH_2Cl_2$:*n*-hexane:benzene = 1 : 1 : 1; a3, b3: $CH_2Cl_2$:*n*-hexane:THF:benzene = 1 : 1 : 1 : 1.

To support the statement, an additional test was made. During the test, fluorescence dyed toluene was used as a typical liquid, then, a monolith (M-5) with a size of 1.2 cm ×1.0 cm (diameter, length) was put into the liquid. A few minutes later, the swelled monolith was withdrawn from the liquid, and then put onto a piece of pan paper. Checking of the paper after a while did not show any significant liquid, but only a stain (c.f. pictures below).




The test was further quantified. For each test, the pan paper was weighed before and after the absorption, and the increase in the weight is recognized as leaching of the liquid absorbed. Three tests were conducted for each liquid. The results are shown in the Table below. Clearly, all the leaching is less than one per ten thousands, suggesting again that the leaching is negligible.

**Table S3** Results from quantification tests of the leaching of the liquid absorbed.

| **Solvent** | **The weight of leaching (g)** |
|---|---|
| Cyclohexene | 0.0007±0.0002 |
| *n*-hexane | 0.0006±0.0002 |
| Toluene | 0.0007±0.0002 |
| Benzene | 0.0005±0.0002 |
| Dichloromethane | 0.0003±0.0002 |
| Tetrahydrofuran | 0.0003±0.0002 |
| Gasoline | 0.0008±0.0002 |
| Kerosene | 0.0007±0.0002 |