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Item Type	Article
Authors	Wang, Shi-Qiang;Mukherjee, Soumya;Patyk-Kaźmierczak, Ewa;Darwish, Shaza;Bajpai, Alankriti;Yang, Qing#Yuan;Zaworotko, Michael J.
Citation	Angewandte Chemie;20 (3), pp. 6630-6634
Publisher	John Wiley & Sons, Inc.
Download date	2026-03-17 16:09:32
Item License	https://creativecommons.org/licenses/by-nc-sa/1.0/
Link to Item	https://hdl.handle.net/10344/7850

Akzeptierter Artikel

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Zitierweise: *Angew. Chem. Int. Ed.* 10.1002/anie.201901198
Angew. Chem. 10.1002/ange.201901198

Link zur VoR: <http://dx.doi.org/10.1002/anie.201901198>
<http://dx.doi.org/10.1002/ange.201901198>

Highly selective, high capacity separation of *o*-xylene from C₈ aromatics by a switching adsorbent layered material

Shi-Qiang Wang, Soumya Mukherjee, Ewa Patyk-Kaźmierczak, Shaza Darwish, Alankriti Bajpai, Qing-Yuan Yang and Michael J. Zaworotko*

Abstract: Adsorbent materials that exhibit high selectivity without sacrificing adsorption capacity can significantly reduce energy costs for industrial separation/purification processes and thus facilitate a transition away from energy-intensive processes such as conventional distillation. Purification of the C₈ aromatics (xylenes and ethylbenzene) is particularly challenging because of their similar physical properties. It is also relevant because of their industrial utility. In this context, physisorptive separation of C₈ aromatics has long been suggested as an energy efficient solution but no physisorbent has yet combined high selectivity (>5) with high adsorption capacity (>50 wt%). Herein, we report a counterintuitive approach to the adsorptive separation of *o*-xylene from other C₈ aromatics by the study of a known nonporous layered material, [Co(bipy)₂(NCS)₂]_n (**sql-1-Co-NCS**). We observe that **sql-1-Co-NCS** can reversibly switch to C₈ aromatics loaded phases with different switching pressures and kinetics, manifesting benchmark *o*-xylene selectivity (S_{OX/EB} ~ 60) and high saturation capacity (> 80 wt%). Structural insight into the observed selectivity and capacity is gained via analysis of the crystal structures of C₈ aromatics loaded phases.

The separation and purification of chemical feedstocks currently uses 10–15% of worldwide energy production; traditional separation processes (e.g. distillation, drying, evaporation) account for more than 80% of this energy.^[1] Given that there will be ever-increasing demand for commodities in the coming decades, more energy-efficient purification technologies are urgently required for large-scale separation processes. One of the most challenging and important separations in this context involves xylene isomers and ethylbenzene, the C₈ aromatics. These compounds are used for the industrial production of fibers, plastics, solvents and fuel additives.^[2] However, their similar sizes and boiling points make their separation difficult. Presently, higher-boiling *ortho*-xylene (OX) is the main component isolated by distillation with > 150 theoretical plates, while *meta*-xylene (MX) and *para*-xylene (PX) are separated by (complexation-based) fractional crystallization and/or adsorption. Ethylbenzene (EB) is mainly produced by alkylation of benzene with ethylene, because it is extremely energy-intensive to isolate it by superfractionation with > 300 theoretical plates.^[3] Although adsorption-based technology has been well studied for C₈ aromatics separation,^[4] widespread implementation is hindered by the capacity and selectivity limitations of the current generation of adsorbents. For example, zeolites only offer moderate capacity (~ 10–20 wt%) and selectivity (~ 5),^[5] overshadowing their advantages of low cost and ready availability. Recently, a supermolecular cage with intrinsic

porosity reported by Cooper's group exhibits exceptionally high selectivity but it also suffers from low adsorption capacity.^[6] Nassimbeni and co-workers have also reported inclusion compounds with high selectivity but low working capacity.^[7] Indeed, there is no physisorbent material which combines the desirable criteria (selectivity > 5 and capacity > 50 wt%) that would enable a paradigm shift towards physisorptive separation of C₈ aromatics.

Metal–organic materials (MOMs),^[8] also known as porous coordination polymers (PCPs)^[9] or metal–organic frameworks (MOFs),^[10] represent a promising class of porous materials that offer potential form utility in storage, separation, sensing and catalysis.^[11] Unlike traditional classes of porous materials they can offer exquisite control over pore size and pore chemistry as their modularity makes them amenable to crystal engineering.^[12] Several MOMs have been investigated with respect to C₈ aromatics separation, however, they also lack a combination of strong selectivity and high adsorption capacity.^[13] MIL-101(Cr)^[14] and [Ni(NCS)₂(ppp)₄]^[15] are the current benchmark MOMs in terms of adsorption capacity and selectivity, respectively. MIL-101(Cr) exhibits OX capacity of ~120 wt%, but its large pores and cavities afford low selectivities (OX/PX and OX/MX ~1.5).^[16] [Ni(NCS)₂(ppp)₄] exhibits OX/MX and OX/PX selectivities of ~34–40 but its adsorption capacity is ~27 wt%.

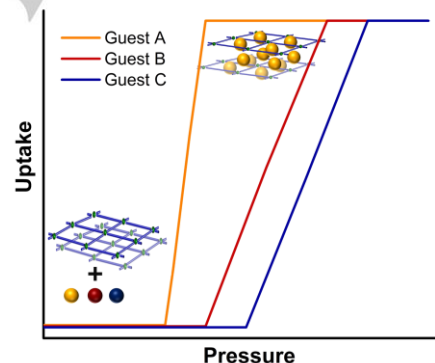


Figure 1. Schematic illustrating the adsorption behaviour of a switching adsorbent layered material for which the switching is triggered by guests. The different switching pressures and kinetics can enable a new approach to adsorptive separation; the preferentially adsorbed compound, A (gold ball), is selected over compounds B (red ball) and C (blue ball).

Here, we describe a new approach to C₈ aromatics separation that, counterintuitively, involves the use of a nonporous material,^[17] {[Co(4,4'-bipyridine)₂(NCS)₂]_n (**sql-1-Co-NCS**), which switches to open phases when exposed to certain guests (Figure 1). **sql-1-Co-NCS** belongs to a broad family of MOMs comprised of octahedral metal ions, **M**, coordinated counter anions, **A**, and linear linker ligands, **L**. When **M:L** is 1:2 then square lattice (**sql**) coordination networks of formula {[**M(L)**₂(**A**)₂·*x*guest]_n are readily formed.^[18] Such **sql** coordination networks were first reported in the 1970s^[19] and were subsequently studied by several groups.^[20] The adsorption properties of **sql** coordination networks remain underexplored with the notable exception of the so-called **ELM** family, which exhibits single or multi-step gas adsorption isotherms triggered by appropriate stimuli.^[21] Recently, we reported that the related

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compound **sql-1-Co-NCS** exhibits recyclable switching with single-step adsorption isotherms induced by CO₂.^[17c] These switchable **sql** coordination networks, which we termed *switching adsorbent layered materials*, have not yet been studied with respect to their sorption properties on hydrocarbon vapours and liquids, although several nonporous organic solids showed promising selectivity signatures.^[22] We herein report the benchmark vapour/liquid separation performance of **sql-1-Co-NCS** with respect to C₈ aromatics.

Synthesis and Characterization

The nonporous (closed) phase [Co(bipy)₂(NCS)₂]_n (**sql-1-Co-NCS**) was prepared using the method described in our recent paper.^[17c] Single-crystals of the open phases of **sql-1-Co-NCS** were prepared *via* solvent diffusion: **sql-1-Co-NCS-4PX**, **sql-1-Co-NCS-4MX**, **sql-1-Co-NCS-4OX** and **sql-1-Co-NCS-2EB** (PX = *para*-xylene, MX = *meta*-xylene, OX = *ortho*-xylene, EB = ethylbenzene, full synthesis procedures are given in Supporting Information†). TGA profiles (Figures S4–S6) reveal that each C₈ aromatic isomer was removed by 130°C and that the resulting nonporous phase is thermally stable until 170°C.

Structural Analysis

Single crystal X-ray diffraction (SCXRD) studies revealed that **sql-1-Co-NCS-4PX**, **sql-1-Co-NCS-4OX** and **sql-1-Co-NCS-2EB** crystallize in the monoclinic space group C2/c whereas **sql-1-Co-NCS-4MX** exists in P2₁/n. The open phases exhibit similar cavity sizes (~ 7.5 Å × 7.5 Å) to the closed phase **sql-1-Co-NCS** but the geometry of the square grids is different. The four open phases exhibit square grid angles ∠_{Co-Co-Co} close to 90° whereas the closed phase is distorted with angles of 75.3 and 104.7° (Figures S11–S15). There is also variation in ∠_{Co-NCS} angles: 170.9° for **sql-1-Co-NCS**; 157.6–166.1° for the open phases (Table 1). The bipy linker ligands are either planar or twisted; the MX-loaded phase exhibits a 100% twisted arrangement whereas 50% twisted/ 50% planar is present in the other variants (Table 1). The torsion angles of bipy vary from 29.3° to 54.5°.

Vapour Sorption Experiments

Vapour sorption isotherms were collected on **sql-1-Co-NCS** at 298 K (Figure 2). It was observed that **sql-1-Co-NCS** switches in the presence of each of the C₈ aromatic isomers but with marked differences in terms of their adsorption capacity and switching pressure (P_s). At 298 K, P_s occurred at P/P₀ ~25%, 30%, 50% and 70% for OX, PX, MX and EB, respectively. Uptakes of OX and PX were exceptionally high (8.0 mmol/g or 85 wt%) at P/P₀ ~ 95%, second only to **MIL-101(Cr)**.^[14] MX and EB did not reach full loading, an indication that higher MX and EB vapour pressure or longer equilibrium time was necessary (Figure S7). The adsorbed C₈ molecules are removed *in vacuo* to recycle the sorbent as revealed by PXRD (Figure S2) and recyclability tests (Figures S9 and S10). **sql-1-Co-NCS** was pelletized under various pressures to confirm its compression resistance (Figure S3) and retention of working capacity as demonstrated by OX adsorption measurements (Figure S8).

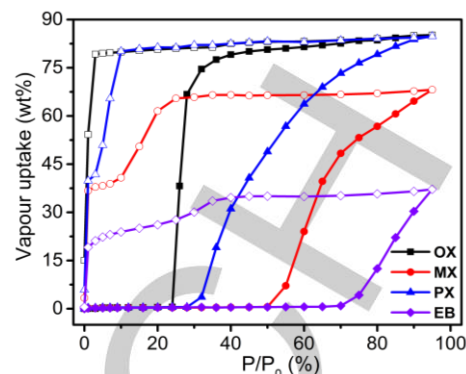


Figure 2. C₈ aromatics vapour sorption isotherms for **sql-1-Co-NCS** at 298 K. Solid and open symbols represent adsorption and desorption, respectively.

C₈ Aromatics Separation Studies

The different switching pressures and/or adsorption rates observed for **sql-1-Co-NCS** in response to C₈ aromatics suggested that it could be effective for physisorptive separation of C₈ aromatics. Vapour-phase binary mixture separation experiments were conducted on **sql-1-Co-NCS** and selectivities were determined by ¹H NMR (Figures S33–S38). Selectivity values were found to be 5.3, 3.7, 1.5, 2.8, 4.5 and 28.9 for OX/MX, OX/PX, PX/MX, MX/EB, PX/EB and OX/EB, respectively (Table S4). The hierarchy of selectivity (OX > PX > MX > EB) is consistent with the pure vapour sorption experiments. Liquid-phase binary mixture separation experiments were also conducted (Figures S39–S44). Our results reveal that **sql-1-Co-NCS** exhibits OX preference with OX/PX and OX/MX selectivity values of 9.6 and 7.5, respectively, the second highest values thus far reported for OX-selective MOMs (Figures 3a,b). The MX/PX selectivity of **sql-1-Co-NCS** is 1.3, which is comparable to other OX-selective MOMs (Figure S45a). The selectivity for xylene isomers over EB was found to be OX/EB= 60.1, MX/EB= 3.8 and PX/EB= 7.3 (Figures 3c and S45b,c). **sql-1-Co-NCS** is therefore the first xylene adsorbent to exhibit high selectivity and high capacity (Figure 3 and Table S6). Indeed, its OX/EB selectivity is five times higher than that of the current benchmarks MIL-47 (V) and MIL-53 (Al).^[23] The liquid phase selectivity order, OX > MX > PX > EB, matches their boiling points.

Discussion

To understand the mechanism behind its selectivity and capacity towards C₈ aromatics, we determined the crystal structures of the open phases of **sql-1-Co-NCS**. As illustrated in Figures 4a–c, xylene guests reside as pairs in the interlayer spaces (green) and the square cavities (red). They constitute > 60% of the unit-cell volume in these crystals. The loading, four xylene molecules

Table 1. Structural parameters of **sql-1-Co-NCS** and its C₈ aromatics loaded phases.

Compound	Square grid angles (°)	∠ _{Co-NCS} (°)	Torsion angle of bipy (°)	Interlayer separation (Å)
sql-1-Co-NCS (100 K)	75.3/104.7	170.9	50% (54.5); 50% coplanar	4.46
sql-1-Co-NCS-4PX (100 K)	90/90	160.3	50% (39.9); 50% coplanar	9.15
sql-1-Co-NCS-4MX (100 K)	89.3/90.7	162.8	100% (32.6)	9.21
sql-1-Co-NCS-4OX (100 K)	90/90	166.1	50% (29.3); 50% coplanar	9.26
sql-1-Co-NCS-2EB (100 K)	87.9/92.1	157.6	50% (51.4); 50% coplanar	6.25

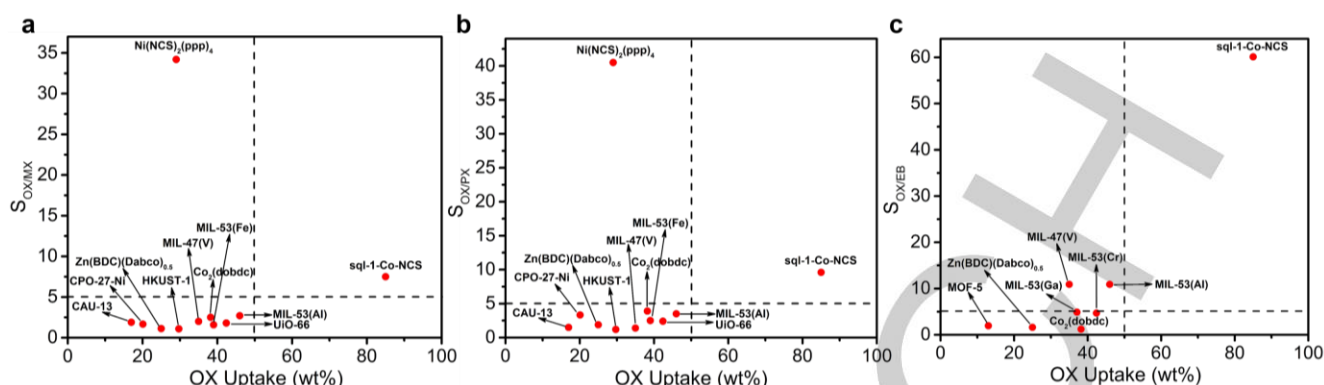


Figure 3. Comparison of physisorbents in terms of their OX adsorption capacity and selectivity for OX/MX, OX/PX and OX/EB, respectively.

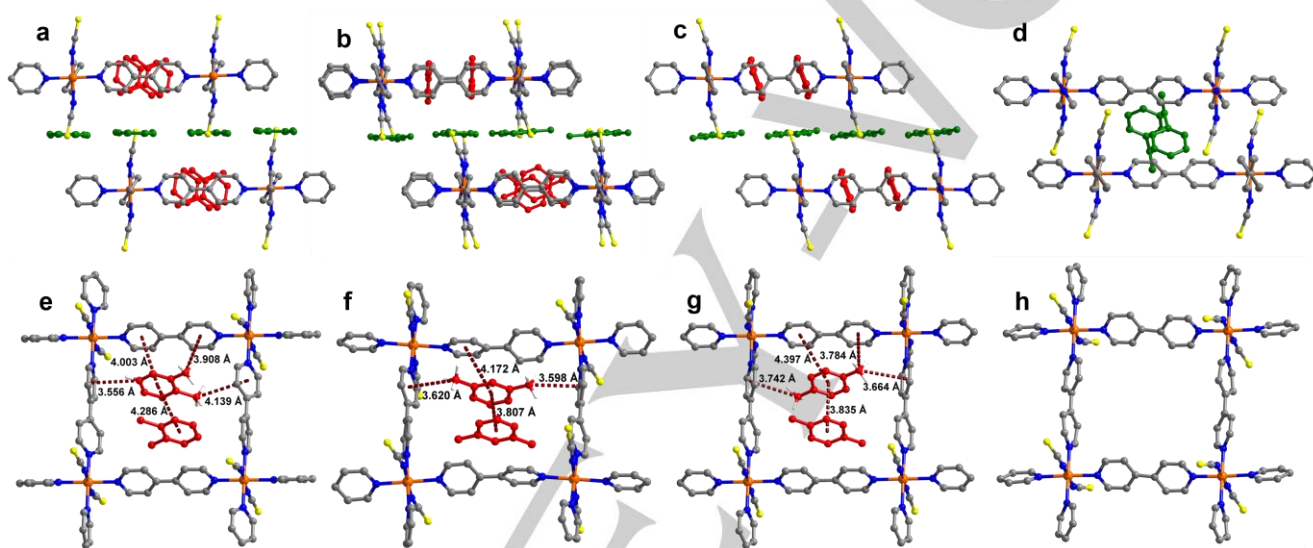


Figure 4. The structures of four C_8 aromatics loaded phases of **sq1-1-Co-NCS**. a-d, the layering of guest-loaded phases for OX, MX, PX and EB respectively. e-h, host-guest interactions in the square cavities of the OX, MX, PX and EB loaded phases, respectively.

per formula unit (8.2 mmol/g or 87 wt%), correlates well with the saturation xylene uptake capacities obtained from vapour sorption experiments. The EB variant exhibits a different type of structure. Only two EB molecules per formula unit are adsorbed because EB molecules only occupy the interlayer spaces. Ethyl groups project into square cavities and preclude other EB molecules from occupying the cavities (Figure 4d). These structural differences help to explain why EB is the least favoured guest.

Although all three xylene-loaded phases exhibit the same number of guest molecules per formula unit, there are subtle differences in their host-guest interactions. For the guests in the square cavities (Figures 4e-g), the distance between OX molecules and the pyridyl ring of bipy linkers is the shortest among the xylene-loaded phases (4.003 vs. 4.172 vs. 4.397 Å for OX, MX and PX respectively). In addition, a phenyl hydrogen of OX forms an extra C-H $\cdots\pi$ interaction with the network whereas MX and PX only exhibit C-H $\cdots\pi$ interactions between their methyl groups and the networks. With respect to the guests that lie in the interlayer spaces (Figure S16), OX molecules exhibit three C-H $\cdots\pi$ interactions with adjacent layers while MX and PX only form two such interactions. These different host-

guest/guest-guest interactions can be attributed to the shape difference of C_8 aromatics and fit the observed selectivity order for liquid C_8 aromatics. It is worth mentioning that **sq1-1-Co-NCS** not only exhibits excellent OX selectivity, but also offers competitive selectivities for the other three C_8 aromatic isomers. It therefore holds promise for sequential separation of C_8 aromatics (Figure S46).

Sorption of four xylene molecules per formula unit results in interlayer separations increasing from 4.5 Å^[17c] (closed phase) to ~9.2 Å (xylene-loaded phases) and a doubling of unit-cell volume. A literature review revealed that these phases exhibit the largest interlayer separations yet seen among bipy-based **sq1** coordination networks (Table S5). **sq1-1-Co-NCS** therefore exhibits a different sorption mechanism than that observed in other OX-selective MOMs such as the MIL-53 family, which exhibit “breathing” from open (narrow-pore) to more open (large-pore) structures.^[24] Indeed, it is this clay-like behavior that enables the high adsorption capacity of **sq1-1-Co-NCS** is related to the enclathration approach taken by Nassimbeni *et al.*^[7,25] It also means that there is little structural distortion in either the closed or open phases of **sq1-1-Co-NCS**, which helps to explain its recyclability.

In summary, we report that C₈ aromatics induce phase switching in a nonporous **sql** coordination network, **sql-1-Co-NCS**. The phase switching combines guest intercalation and inclusion phenomena which enables extreme expansion in interlayer separation and in turn enables exceptionally high xylene adsorption capacity. In addition to high capacity, **sql-1-Co-NCS** exhibits high selectivity towards OX that correlates with the switching pressure of each sorbate. **sql-1-Co-NCS** is therefore the first physisorbent that separates OX with both high selectivity and high capacity under ambient conditions. Further, both the crystallinity and working capacity of **sql-1-Co-NCS** are fully retained even after pelletization. Moreover, the switching nature of **sql-1-Co-NCS** means that this type of material will be expected to serve a wide range of sorbates. That **sql-1-Co-NCS** is modular and many variants are already known means that it is likely to be prototypical for a broad platform of switching adsorbent layered materials or SALMAs. Separation studies on other hydrocarbon mixtures with **sql-1-Co-NCS** and related SALMAs are in progress.

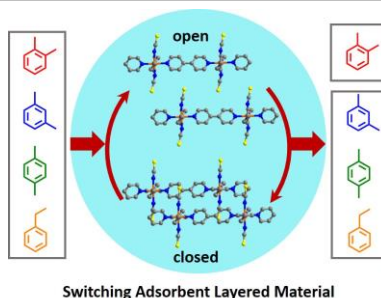
Acknowledgements

M.J.Z. would like to acknowledge the support of Science Foundation Ireland (SFI Awards 13/RP/B2549 and 16/IA/4624).

Keywords: switching behaviour • nonporous layered materials • square lattice coordination networks • C₈ aromatics • physisorptive separation

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