# Acetic Acid Vapour-Induced Morphological Rearrangement Of FeBTC Metal–Organic Framework

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Acetic acid (AcOH) is one of the most widespread volatile organic compounds (VOCs) found in museum environments, mainly generated by the degradation of wood and cellulose-based materials.[1] Since AcOH can significantly deteriorate artworks, manuscripts, and historical artifacts, the development of materials capable of capturing AcOH is highly relevant for environmental control and cultural heritage preservation. Among the adsorbent materials reported in the literature, metal–organic frameworks (MOFs) containing coordinatively unsaturated open metal sites, such as MIL-100(Fe), have shown promising performance for the selective capture of polar VOCs, including AcOH, even in the presence of water.[2] Nevertheless, the synthesis of MOFs is often carried out under harsh reaction conditions (i.e. high temperatures and pressures) in non-aqueous media, and may require long reaction times, thus limiting the sustainability of the process. For these reasons, MOFs synthesized rapidly under mild and greener reaction conditions represent an attractive alternative.[3,4]
Here, FeBTC the semicrystalline counterpart of MIL-100(Fe), synthesized under mild reaction conditions (25°C and aqueous solution), was investigated as AcOH vapour adsorbent (Fig.1). Adsorption kinetics followed a double-exponential model, suggesting the coexistence of different uptake regimes, and reached 0.87 ± 0.09 $mg \cdot mg^{-1}$ after 237 h of exposure, exceeding the performance of commercial zeolites and activated carbon reported under comparable conditions. Thermogravimetric analysis together with ATR-FTIR and Raman spectroscopies confirmed the effective uptake of AcOH and supported the presence of both weakly adsorbed and more strongly interacting species. Despite prolonged vapour exposure, WAXS measurements demonstrated the preservation of the FeBTC semicrystalline framework.

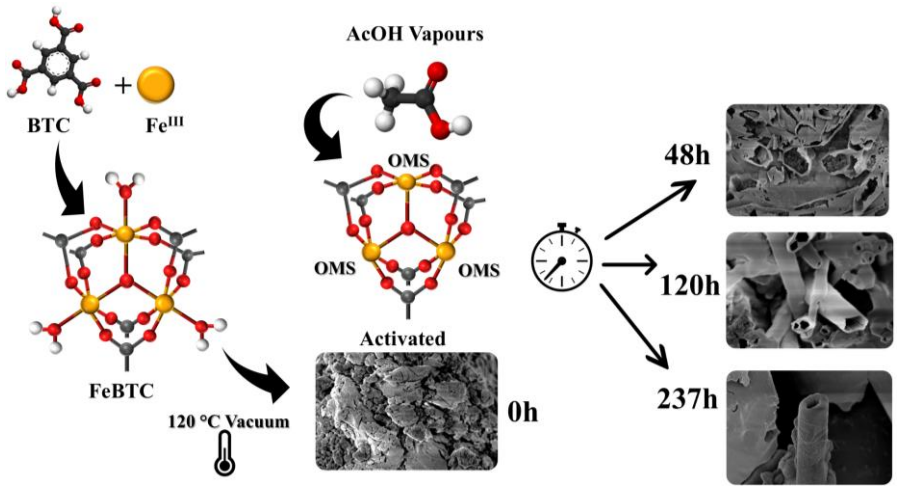


**Figure 1.** Schematic representation of FeBTC synthesis, thermal activation, and AcOH vapour adsorption-induced morphological transformation.

Although AcOH is widely employed as coordination modulator during MOF synthesis, its effect as post-vapour-phase post-synthetic modulator is still poorly investigated.

Data showed that AcOH adsorption induced a morphological rearrangement of FeBTC without altering the original framework structure. SEM analyses revealed a rapid transformation from irregular aggregated particles to hollow cylinder-like architectures already after 14 h of exposure to AcOH vapours. SAXS/USAXS measurements confirmed the evolution of the material over multiple length scales and evidenced a sharp increase of the surface-to-volume ratio during the early stages of adsorption, followed by a progressive decrease at longer exposure times.[5]
These findings suggest that vapour-phase AcOH exposure may represent a simple and effective strategy to tailor MOF morphology and accessible surface area without disrupting the original framework structure, opening new perspectives for gas capture and separation applications.

**REFERENCES**

(1) Porpora, F.; Dei, L.; Forcellini, C.; D'Aleo, C.; Lisi, L.; De Sanctis, M.; Carretti, E. Interactions Between Polyethyleneimine Xerogels and Acetic Acid Vapor from Degraded Cellulose Acetate. A Novel Therapy for Motion Picture Films Affected by the "Vinegar Syndrome." *Macromol. Rapid Commun.* **2025**, *46* (14), e2500075. https://doi.org/10.1002/marc.202500075.

(2) Severino, M. I.; Al Mohtar, A.; Vieira Soares, C.; Freitas, C.; Sadovnik, N.; Nandi, S.; Mouchaham, G.; Pimenta, V.; Nouar, F.; Daturi, M.; Maurin, G.; Pinto, M. L.; Serre, C. MOFs with Open Metal(III) Sites for the Environmental Capture of Polar Volatile Organic Compounds. *Angew. Chem. Int. Ed. Engl.* **2023**, *62* (6), e202211583. https://doi.org/10.1002/anie.202211583.

(3) Dhakshinamoorthy, A.; Alvaro, M.; Horcajada, P.; Gibson, E.; Vishnuvarthan, M.; Vimont, A.; Grenèche, J.-M.; Serre, C.; Daturi, M.; Garcia, H. Comparison of Porous Iron Trimesates Basolite F300 and MIL-100(Fe) As Heterogeneous Catalysts for Lewis Acid and Oxidation Reactions: Roles of Structural Defects and Stability. *ACS Catal.* **2012**, *2* (10), 2060–2065. https://doi.org/10.1021/cs300345b.

(4) Sciortino, L.; Alessi, A.; Messina, F.; Buscarino, G.; Gelardi, F. M. Structure of the FeBTC Metal-Organic Framework: A Model Based on the Local Environment Study. *J. Phys. Chem. C* **2015**, *119* (14), 7826–7830. https://doi.org/10.1021/acs.jpcc.5b01336.

(5) Tocco, D.; Chelazzi, D.; Salis, A.; Fratini, E.; Casini, A.; Mastrangelo, R. Acetic Acid Vapours Adsorption-Induced Morphological Change in FeBTC Metal–Organic Framework. *Appl. Surf. Sci.* **2026**, *729* (November 2025), 166203. https://doi.org/10.1016/j.apsusc.2026.166203.