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Synthesis and Structural Characterization of MOF-5 – a metal-organic compound based on Zn(II) and terephthalate anions

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ABSTRACT

Metal-Organic Frameworks (MOFs) are porous hybrid crystalline materials highly prized for their customizable architectural frameworks. This study focuses on the synthesis and optimization of MOF-5, a landmark Zn(II)-based coordination polymer, evaluating how solvothermal reaction durations impact structural stability and subsequent chemical remediation capabilities.

Methods: MOF-5 was synthesized solvo-thermally using zinc nitrate hexahydrate and terephthalic acid in N,N-dimethylformamide (DMF) at 120°C across varying crystallization intervals (18 h, 24 h, and 48 h). Crystalline validation was completed via Fourier-Transform Infrared (FTIR) spectroscopy and Thermal Analysis (TG/DTG/DTA). Environmental trapping capacity was systematically quantified through the fluid-phase adsorption of molecular iodine (I_2) and Methylene Blue (MB) dye, monitored via Raman and UV-Vis spectrophotometry.

Results: FTIR and thermogravimetric data verified that a 24-hour reaction window yields an optimized, highly ordered lattice framework with low residual host solvent entrapped in the pores. Prolonged heating to 48 h caused framework deterioration. Raman profiles verified successfully trapped iodine guest species, yielding distinctive polyiodide peaks (~80–130 cm^{-1} and ~219 cm^{-1}) alongside ligand shift interactions. Kinetic UV-Vis trials confirmed rapid Methylene Blue remediation, stabilizing near 70 minutes before reaching an adsorption equilibrium.

Conclusions: The synthesized MOF-5 matrices display strong physical capture dynamics for inorganic and organic water pollutants. However, structural vulnerability in continuous aqueous setups remains a critical threshold constraint for prolonged field applications.

Keywords: *MOF-5; Reticular Chemistry; Nanoporous Materials; Solvothermal Synthesis; Iodine Adsorption; Methylene Blue Dye*

1. INTRODUCTION

Metal-Organic Frameworks (MOFs) represent an innovative class of porous, crystalline hybrid materials constructed through the self-assembly of metal ions or clusters coordinated via multi-dentate organic linkers. By systematically adjusting the chemical identity of the metallic node, the structural geometry of the organic ligand, and the thermodynamic variables of synthesis (such as solvent dynamics, temperature gradients, and isolation time), researchers can engineer highly porous configurations with highly tuneable surface areas and precisely controlled pore sizes. The architectural adaptability of MOFs allows these systems to host single or multiple metallic centers within a uniform network, facilitating precise structural design for targeted modern tasks. Consequently, these frameworks are widely explored across multi-disciplinary fields, including magnetism, luminescence, gas storage, heterogeneous catalysis, and targeted drug delivery systems.

The historical foundation of stable, ultra-porous frameworks was established in 1999 by Omar Yaghi and co-workers, who successfully isolated a robust, highly porous network designated as MOF-5 [1].

Formally identified as $Zn_4O(BDC)_3 \cdot (DMF)_8(C_6H_5Cl)$, this framework preserved its structural configuration even after the complete evacuation of guest solvent molecules from its internal cavities. MOF-5 provides significant economic and practical benefits, including highly accessible raw precursors and cost-effective scaling pathways, alongside an impressive hydrogen storage capacity of up to 4.5 wt.% at 77 K and 0.7 bar.

Despite these structural benefits, synthetic reproducibility and structural performance are highly sensitive to small variations in processing conditions. Minor variations in the chemical assembly process can shift the underlying crystallization kinetics, resulting in mixed phases or degraded porous properties. Therefore, this investigation evaluates the structural effects of processing duration on MOF-5 crystallization while exploring its practical capacity to adsorb molecular contaminants from fluid environments.

This paper details the systematic solvothermal synthesis of MOF-5 across multiple processing windows, characterizes the resulting structural configurations using infrared and thermal profiles, and validates its chemical remediation performance using molecular iodine and Methylene Blue dye configurations.

2. LITERATURE REVIEW

The structural assembly of MOF-5 relies on the coordination of tetrahedral Zn_4O clusters linked by 1,4-benzenedicarboxylate (BDC^{2-}) rigid dianions, forming a definitive isotropic three-dimensional cubic lattice.

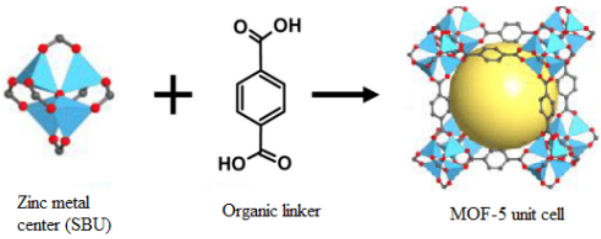


Figure 1 - Crystalline structure of MOF-5 [4]

Research shows that the final framework efficiency depends on the structural integrity of this porous network and the interfacial interactions between the metallic clusters and coordinating organic linkers. However, isolating highly ordered, pure-phase MOF-5 configurations remains a challenge, as identical starting mixtures can yield vastly different structural polymorphs or amorphous non-porous side-products if synthesis variables drift.

Critical crystallization parameters—including reaction time, temperature gradients, the counter-anion of the zinc precursor, and reactant concentration ratios—directly dictate initial nucleation steps and subsequent crystal face development. Well-developed 3D structural configurations with open, non-obstructed pore paths are essential to allow unhindered diffusion and the effective trapping of targeted guest molecules.

Recent literature highlights the use of these engineered porous structures as advanced matrices to isolate toxic industrial runoff and organic waste species from fluid streams. However, a key limitation of MOF-5 is its structural sensitivity to moisture, as moisture can cause framework collapse via hydrolysis of the Zn–O coordination bonds. This study addresses these issues by assessing the narrow synthetic window where structural crystallinity is maximized, balancing effective chemical adsorption performance against the framework's known vulnerability in liquid environments.

3. RESEARCH METHODOLOGY

3.1 Research Design

This study utilized an experimental research design focused on optimizing material properties. The primary independent variable was the solvothermal reaction duration (18, 24, and 48 hours), while the dependent variables included structural crystallinity, thermal stability, and liquid-phase chemical adsorption capacity. This comparative design allowed identification of the exact processing window where framework stability is maximized.

3.2 Samples

The research sample set consisted of four distinct experimental batches derived from a single synthesized precursor solution. The liquid mixture was divided equally into four separate reaction vessels to guarantee uniform starting chemical concentrations across all tested reaction durations.

Figure 2 - Sample 1 – 18h, 17mL

Figure 3 - Sample 2 – 24h, 17mL

Figure 4 - Sample 3 – 48h, 17mL

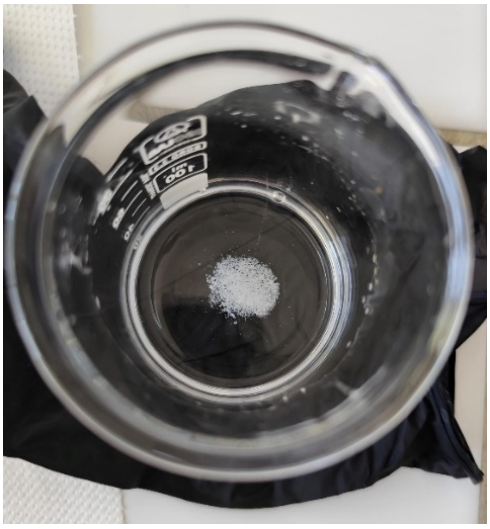


Figure 5 - Sample 4 – 48h, 9mL

3.3 Instruments and Materials

The precursor chemicals included zinc nitrate hexahydrate ($Zn(NO_3)_2 \cdot 6H_2O$, 2.16 g) and terephthalic acid (1,4-benzenedicarboxylic acid, HOOC-C_6H_4-COOH, 0.396 g) dissolved in N,N-dimethylformamide (DMF, 60 mL).

Crystalline validation was completed via Fourier-Transform Infrared (FTIR) spectroscopy using a ***Bruker Tensor 27 Spectrometer***.

Thermal degradation behaviour was recorded using a ***Netzsch STA 409 PC thermal analyser***.

Micro-Raman evaluations were performed using a ***Horiba LabRAM HR Evolution dispersive Raman spectrometer*** equipped with a 532 nm excitation laser (~4 mW) and a 600 gr/mm diffraction grating.

Aqueous concentration values for the dye trials were tracked using standard ***UV-Vis spectrophotometry***.

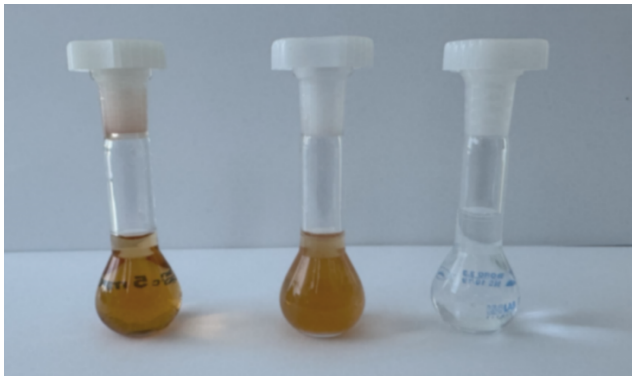
3.4 Data Collection Procedures

The precursor materials were fully dissolved in DMF under continuous stirring. The solution was divided into four equal samples and transferred into 20 mL Teflon-lined stainless steel digestion bombs. The vessels were heated in a laboratory oven at 120°C for different periods:

- Sample 1 for 18 h,
- Sample 2 for 24 h, and
- Samples 3 and 4 for 48 h.

Following heating, the vessels were cooled to room temperature at a controlled rate of 10°C/min. The resulting solids were washed repeatedly with chloroform ($CHCl_3$) to remove residual DMF, dried, and stored in sealed glass vials.

Adsorptive capacity was evaluated through two distinct pollutant capturing protocols:

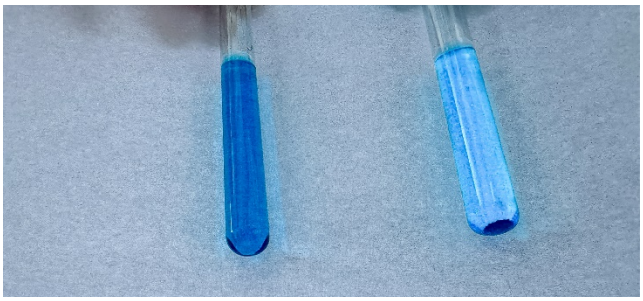


1. Molecular Iodine (I_2) Capture: Aqueous molecular iodine was prepared by dissolving 60 mg of potassium iodide (KI) in 20 mL of deionized water under magnetic stirring (800 rpm), followed by the addition of 400 uL of 1 M H_2SO_4 and 10 mg of potassium iodate (KIO_3).

The solution's color transitioned from transparent, to yellow, dark-yellow, all the way to deep brown-violet, indicating I_2 generation via the standard redox equation:

$$KIO_3 + 5KI + 3H_2SO_4 \rightarrow 3I_{2(aq)} + 3K_2SO_4 + 3H_2O$$

The mixture was split into two 10 mL fractions: a reference standard and an active test solution mixed with 20 mg of synthesized MOF-5 for 60 minutes with periodic agitation.



2. Methylene Blue (MB) Remediation: A 10 mg/L aqueous solution of Methylene Blue dye was prepared, and 20 mg of MOF-5 was introduced into a 10 mL aliquot.

Fluid sub-samples were collected at set intervals (0, 5, 15, 30, 70, and 100 minutes) to track concentration kinetics, and the absorbance values at 664 nm were recorded.

3.5 Data Analysis

FTIR measurements were recorded across the 200–4000 cm^{-1} spectral range using KBr pellets compressed at 2 tons/cm^2 with a sample-to-matrix mass ratio of 1:9. Background subtraction was completed using a pure KBr blank. Thermal analysis (TG/DTG/DTA) was performed by heating ~10 mg of sample from 25°C to 800°C in synthetic air (30 mL/min flow rate) at a heating rate of 20°C/min.

Raman spectra were collected from dry powders using a 9-second acquisition time and 10 accumulations.

UV-Vis evaluation monitored the characteristic absorption peaks of Methylene Blue at 664 nm to calculate total dye removal over time.

4. RESULTS

Structural characterization confirmed that the target MOF-5 lattice formed successfully under specific solvothermal conditions.

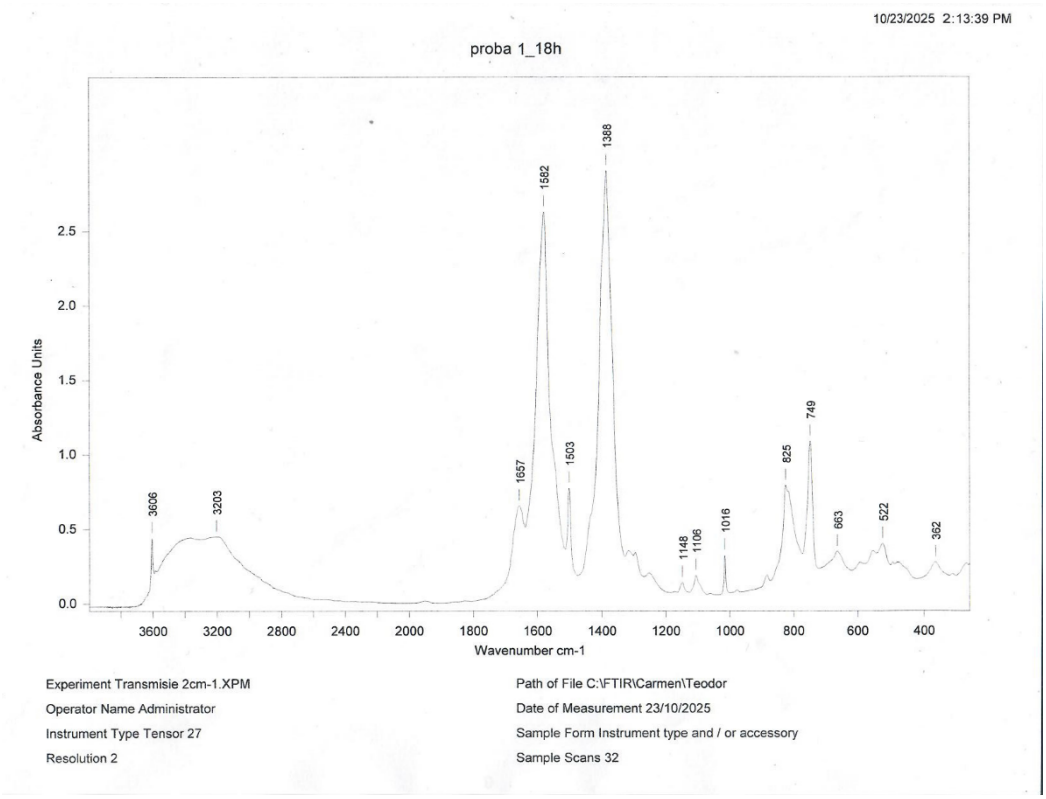


Figure 6 - Sample 1 (18h)

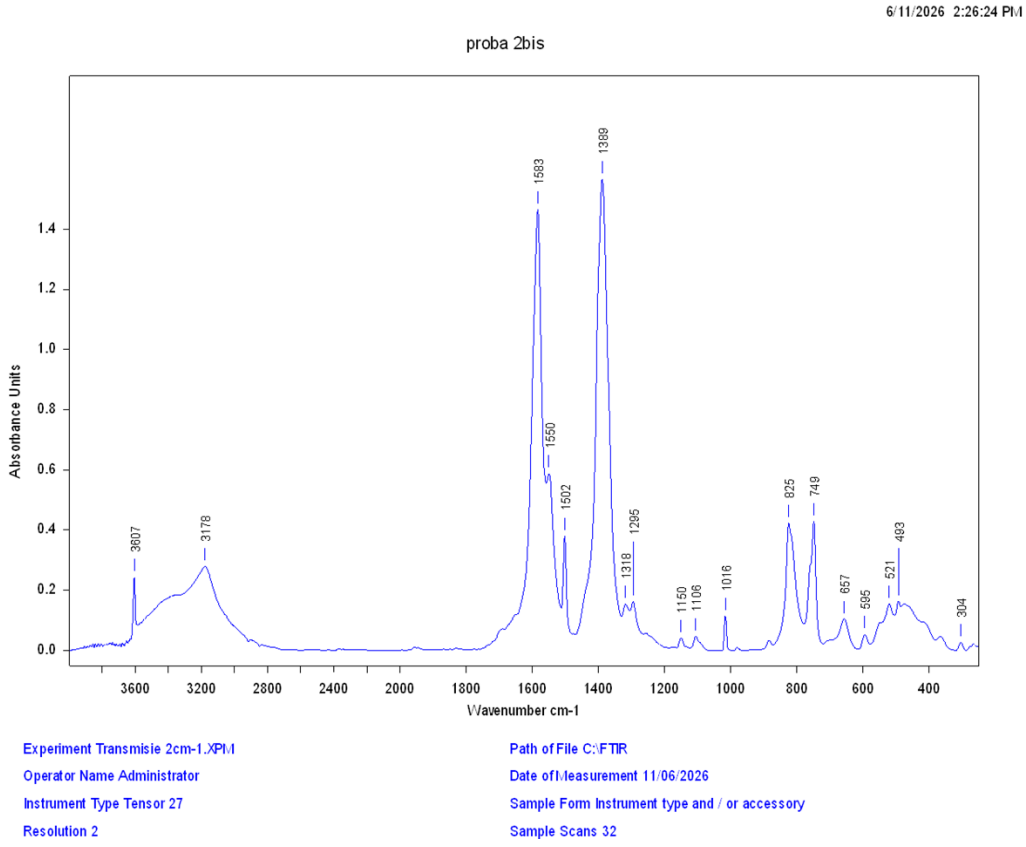


Figure 7 - Sample 2 (24h)

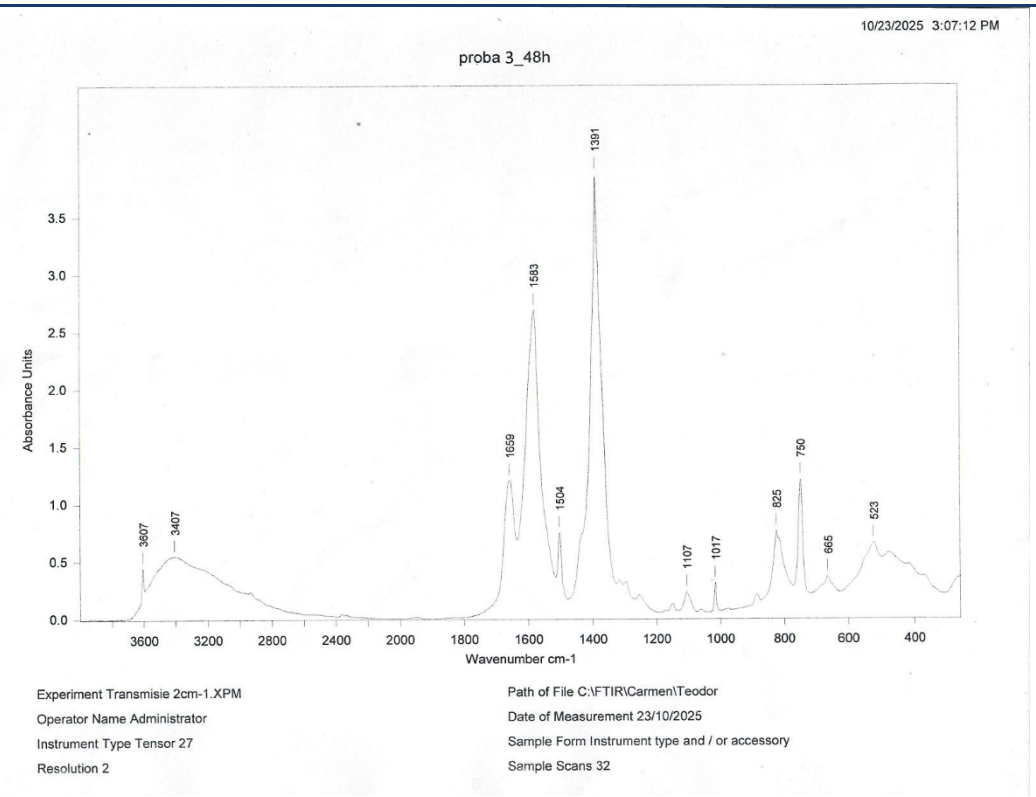


Figure 8 – Sample 3 (48h)

FTIR spectra across the different crystallization times confirmed structural differences:

- At 18 h, the framework matches the MOF-5 profile but retains a high concentration of unreacted DMF solvent within the pores.
- At 24 h, the spectrum shows sharper, well-defined vibrational bands, indicating improved structural order and reduced solvent retention.
- At 48 h, the vibrational signature indicates structural degradation due to prolonged thermal exposure.

Thermogravimetric profiles confirmed these structural trends. The 24-hour sample showed excellent thermal stability, an ordered lattice, and minimal residual DMF solvent, matching the theoretical stoichiometric mass loss. Extending the reaction time to 48 hours provided no structural benefits and caused partial network collapse.

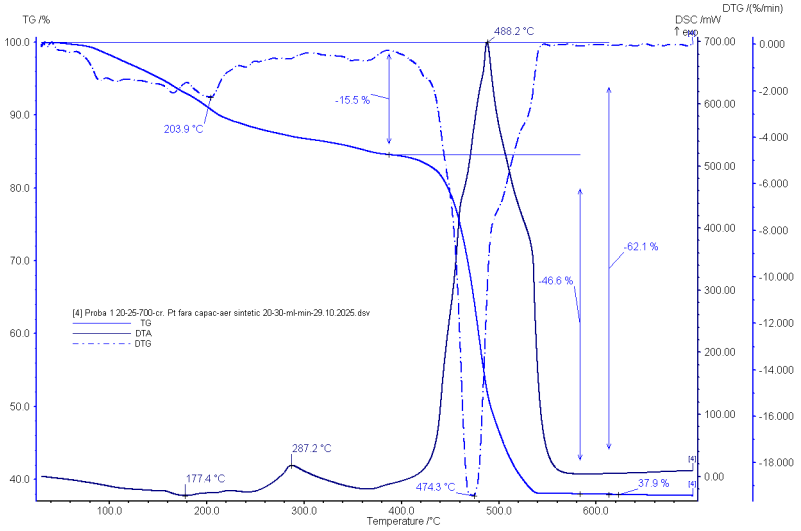


Figure 9 - Sample 1 (18h)

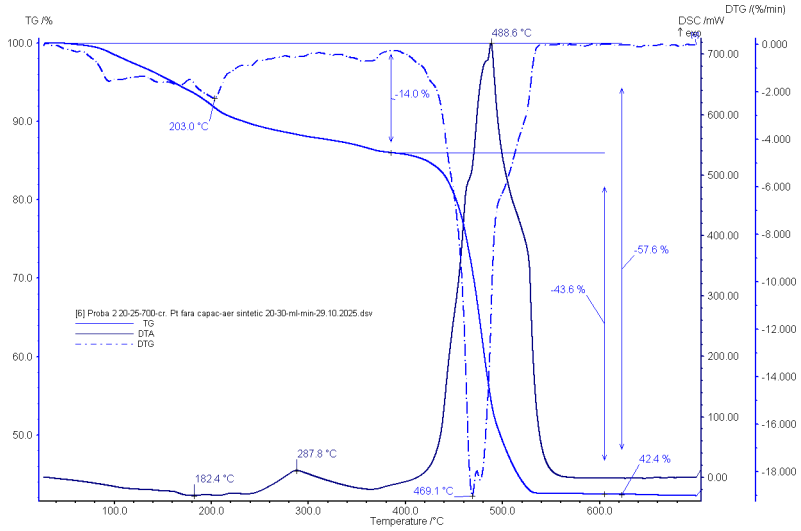


Figure 10 - Sample 2 (24h)

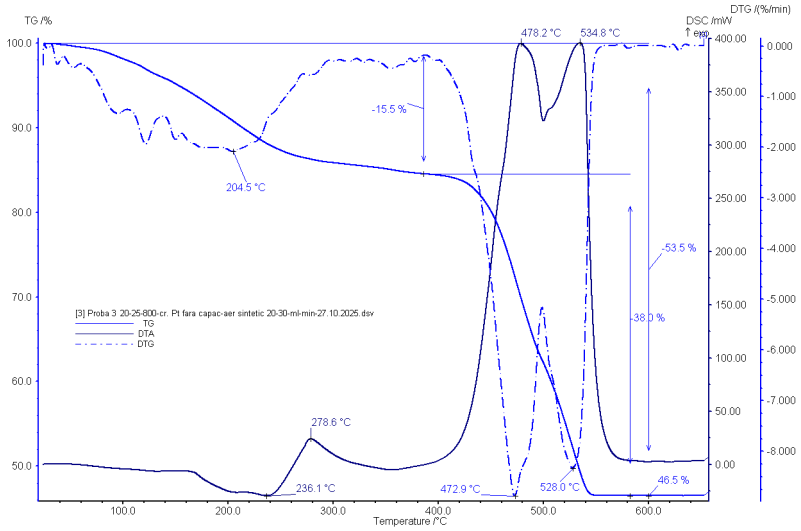


Figure 11 - Sample 3 (48h)

Liquid-phase remediation was also verified by visual color changes during iodine adsorption. Following iodine exposure, the MOF-5 powder changed from white to a light yellow. Raman spectroscopy confirmed iodine capture via a characteristic molecular iodine (I_2) vibration peak at ~219 cm^{-1} and polyiodide signatures (I_3^- and I_5^-) within the ~80–130 cm^{-1} range. Ligand shifts from ~834 cm^{-1} to ~847–850 cm^{-1} indicated direct interactions between the trapped iodine and the aromatic rings of the BDC linker [2][3].

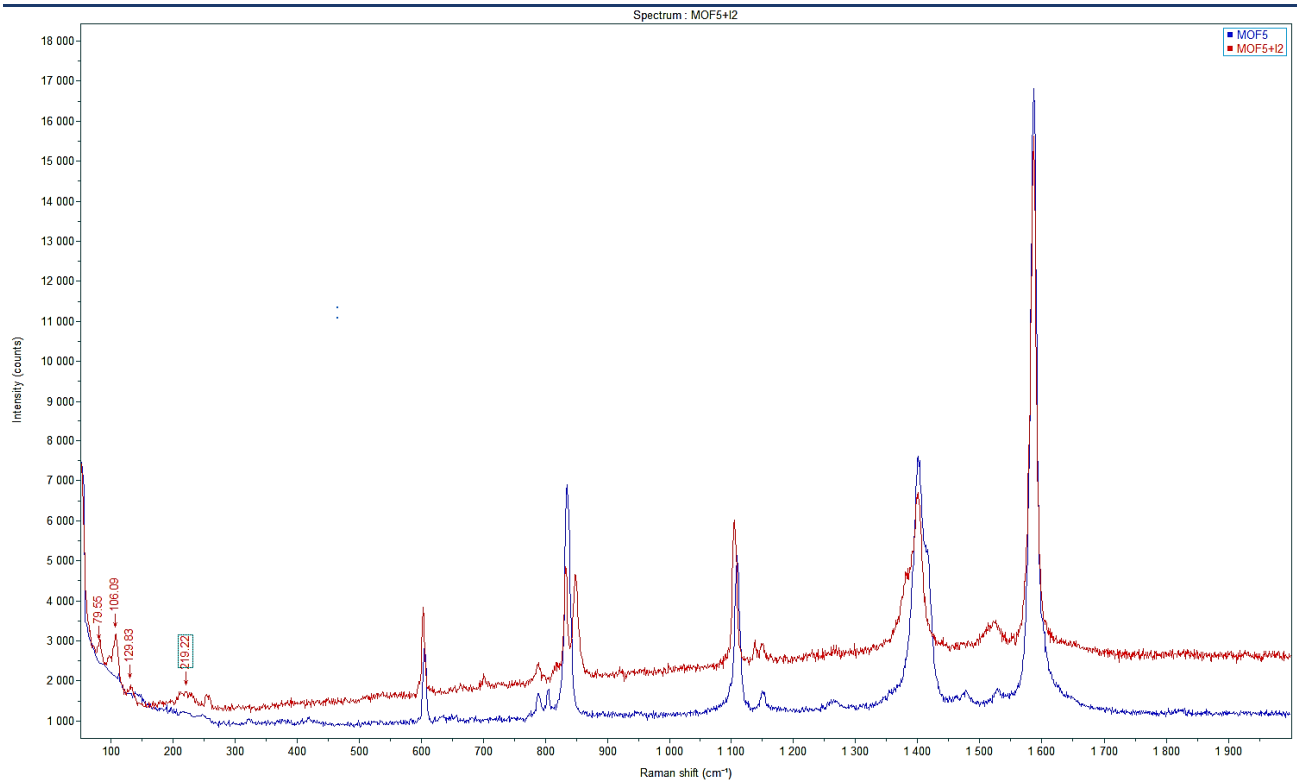


Figure 12 Raman spectrum of the MOF + I_2 sample

The Raman spectrum of the MOF + I_2 sample (red) shows the simultaneous presence of molecular I_2 (~219 cm^{-1}) and poly-iodide species (I_3^- / I_5^-) at low frequencies (~80–130 cm^{-1}). This suggests either a partial electron transfer within the MOF (I_3^- formation) or disproportionation/associations ($I_2 + I_3^- \rightarrow I_5^-$) within the pores, confirming the capture of Iodine by the MOF.

For the Methylene Blue trials, the UV-Vis absorbance curves (Fig. 13) showed a continuous drop in concentration up to 70 minutes, followed by a slight absorbance rebound at 100 minutes as the system reached equilibrium. Physical capture was visually confirmed by the MOF-5 powder changing from white to a brilliant blue.

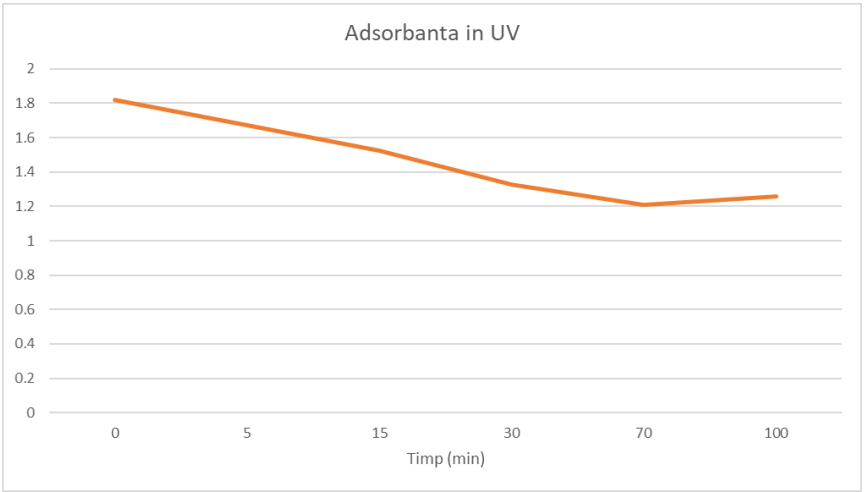


Figure 13 - Variation of absorbance at 664 nm as a function of contact time with MOF-5

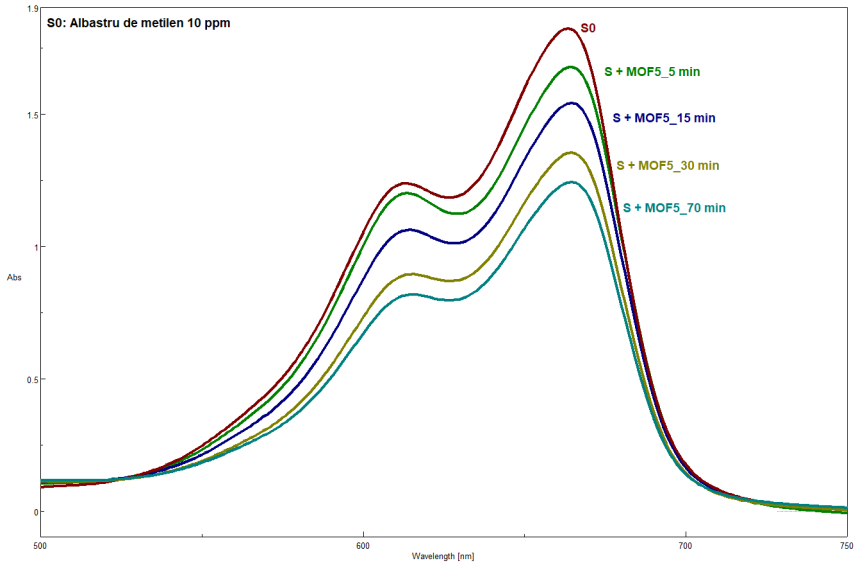


Figure 14 - Comparative absorbance in UV

The MB retention capacity was also confirmed visually (Fig. 15) by the color change of MOF-5 from white to blue.

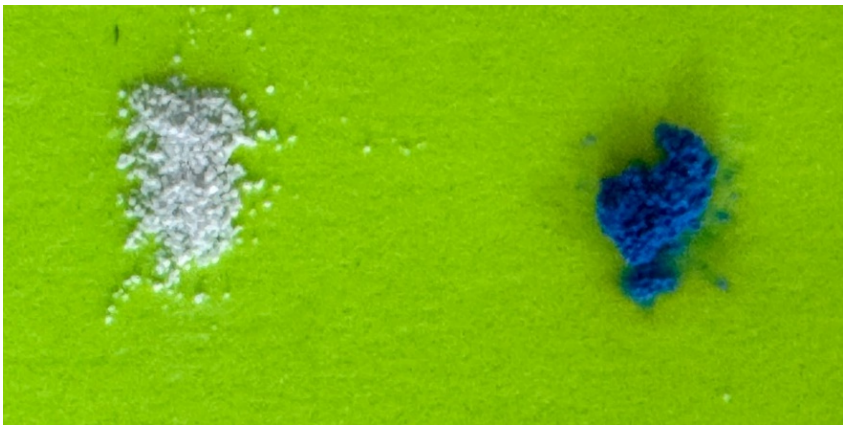


Figure 15 - Product resulting from the experiment, pure MOF-5 and with adsorbed methylene blue (MB)

5. DISCUSSION

The experimental results demonstrate that the solvothermal crystallization window strongly influences the structural order and chemical stability of MOF-5. The infrared profiles show that while an 18-hour reaction forms the framework, it leaves significant amounts of residual DMF trapped within the cavities, restricting pore accessibility. At 24 hours, the crystallization kinetics optimize, producing sharp structural bands and clearing guest solvents without destabilizing the zinc coordination nodes. However, extending the reaction to 48 hours degrades the structure, demonstrating that prolonged thermal stress disrupts the coordination bonds between the zinc centers and the carboxylic groups.

The chemical capture data demonstrates that optimized MOF-5 operates as an effective matrix for trapping environmental contaminants. The Raman shifts observed after iodine capture indicate that the mechanism extends beyond simple physical trapping within the pores. The shift of the aromatic ring deformation band from ~834 cm^{-1} to ~847 cm^{-1} suggests pi-pi electron interactions and charge-transfer mechanisms between the guest iodine species and the conjugated aromatic linkers. The presence of polyiodide bands (~80–130 cm^{-1}) further confirms that the framework stabilizes volatile iodine into organized ionic networks (I_3^- and I_5^-) inside its pores.

Similarly, the Methylene Blue trials demonstrate rapid capture kinetics, with maximum removal occurring at 70 minutes. The subsequent minor rebound in absorbance at 100 minutes represents a standard desorption phase as the system establishes liquid-solid equilibrium.

However, a major limitation identified during these liquid trials is the material's sensitivity to moisture. While MOF-5 offers rapid capture kinetics and high surface areas, prolonged exposure to water can cause framework hydrolysis, where water molecules displace the organic linkers from the zinc clusters. This limits its long-term use in open aquatic remediation and indicates that future work should focus on surface modification strategies to improve hydrophobic stability.

6. CONCLUSION

This study demonstrates the successful solvothermal synthesis, structural characterization, and environmental adsorption performance of MOF-5. Experimental data confirms that a 24-hour reaction time at 120°C optimizes the framework, maximizing structural order and thermal stability while minimizing residual solvent entrapment. Liquid-phase trials show that the material effectively captures molecular iodine and Methylene Blue dye via combined physical trapping and chemical charge-transfer mechanisms. Raman and UV-Vis spectra confirmed successful contaminant loading within the porous matrix.

While the framework demonstrates strong remediation performance, its structural sensitivity to water remains a key limitation for long-term aquatic applications. Overall, this study highlights the cross-disciplinary links between synthesis chemistry, material physics, and environmental engineering required to develop advanced materials for targeted environmental remediation.

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AUTHOR CONTRIBUTIONS

Author	Contribution (CRediT Taxonomy)
Teodor Gabriel Moscalu	Conceptualization, Investigation, Formal Analysis, Project administration, Writing – original draft
Dr Carmen Ștefănescu	Methodology, Supervision, Lab coordination, Data Curation, IR measurements, Resources, Validation, Writing – Review & Editing
Dr Andrei Cucoș	Data Curation, Thermal Analysis, Supervision, Resources, Validation, Writing – Review & Editing
Dr Eduard-Marius Lungulescu	Data Curation, UV measurements, Supervision, Resources, Validation, Writing – Review & Editing

CONFLICT OF INTEREST STATEMENT

The authors declare that the research was conducted in the absence of any commercial or financial relationships that could be construed as a potential conflict of interest.

DATA AVAILABILITY STATEMENT

The data supporting the findings of this study are available from the corresponding author upon reasonable request.

ETHICAL APPROVAL AND CONSENT TO PARTICIPATE

Not applicable. This study did not involve human participants or animal subjects.

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