



Research Report

MIL-101(Cr)-based Thermoresponsive Hydrogels for Efficient Atmospheric Water Harvesting

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Abstract

Water scarcity is an urgent global problem, with increasing pressure every day from many factors such as climate change or increasing demand. The air we breathe usually contains high quantities of water vapour, which could be a potential alternative source of water, especially in isolated regions or regions affected by droughts. My research aimed to synthesise metal-organic framework (MOF)-based thermoresponsive hydrogels for atmospheric water harvesting. The purpose is to combine the high water adsorption levels of the MOF MIL-101(Cr) with the temperature-responsive properties of a polymer hydrogel, PNIPAM.

MIL-101(Cr) is synthesised from a chromium precursor and terephthalic acid under hydrothermal conditions, where the chemicals are heated in an aqueous solution. The resulting MOF is then integrated into a temperature-responsive polymer network with in situ radical polymerisation, a process where the reactive molecules (the radicals) initiate the linking of smaller molecules into a polymer mesh, to form the MOF-hydrogel. Scanning electron microscopy (SEM) analyses the morphology of the MOF, hydrogel, and composite, while differential scanning calorimetry (DSC) analyses the thermal response of the material.

SEM findings showed the characteristic porous MOF structure alongside the polymer network, providing evidence of a successful formation of the MOF-hydrogel. DSC indicated a thermal transition around 32.2 °C, which is consistent with the temperature-responsive behaviour required for water release. These findings demonstrate the potential of combining MIL-101(Cr) with a responsive hydrogel to create a material for atmospheric water harvesting. Future work could analyse the composite's water uptake and release performance in real-life conditions and determine its potential for energy-efficient water harvesting.

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1 Introduction

1.1 Background and motivation

Clean and reliable freshwater is necessary for human health, agriculture, economic development, and more, however water resources are under increasing pressure from factors such as climate change and population growth. Water scarcity is especially concerning in arid and remote regions, where freshwater sources and infrastructure may be limited. This has pushed the development of technologies producing freshwater independently of traditional sources like rivers or groundwater. One of the potential approaches is atmospheric water harvesting (AWH), where water vapour naturally present in the atmosphere is extracted and converted into liquid water. [1,2]

The atmosphere contains large quantities of water in the form of vapour. Unlike many “conventional” freshwater sources, atmospheric moisture is widely distributed and could be accessed in areas with limited groundwater resources. In adsorption-based AWH, a porous or hygroscopic material captures water molecules from humid air through adsorption. The material is subsequently regenerated, usually through heating, causing the adsorbed water to be desorbed, and this process can then be repeated in cycles. The possibility of using relatively low heat for regeneration makes adsorption-based AWH well-suited for decentralised water production. [2,3]

However, efficiently harvesting water from air has some challenges. The amount of water that can be captured depends strongly on environmental conditions such as relative humidity (RH) and temperature. Additionally, an ideal AWH material must not only adsorb a substantial quantity of water, but also desorb it efficiently during regeneration. Therefore, fast adsorption and desorption kinetics, structural and chemical stability, and a low energy requirement for regeneration are important when designing AWH materials. [3,4]

1.2 Research progress in AWH

Research in AWH has therefore focused on developing materials with higher water uptake, faster adsorption, and more efficient regeneration. Traditional adsorbents such as silica gel and zeolites have been researched, while hygroscopic salts have also been investigated because of their high affinity for water. However, limitations such as water uptake under low RH, regeneration requirements, and material stability have motivated research into new materials. [4,5] More recently, metal-organic frameworks (MOFs) have attracted significant attention because their structures and chemical properties can be tailored to modify their interaction with water. The development of MOF-based AWH was notably demonstrated by a 2017 study using MOF-801, which harvested atmospheric water at RHs as low as 20% using low-grade heat from natural sunlight for regeneration. [6]

Research has also explored combining water-adsorbing materials with polymer-based structures such as hydrogels, aiming to combine different desirable properties within a single material. [7] MOFs are porous crystalline materials constructed from metal-containing units connected by organic molecules. Their structures contain networks of pores and internal surfaces, providing sites that can interact with molecules in their surroundings. Their chemical composition and structural properties can also

be changed, making it possible to modify their adsorption behaviour for specific applications. [4]

1.3 Research objective

In this project, MIL-101(Cr) was used as the type of MOF in a hydrogel composite, designed for AWH. MIL-101(Cr) is a promising material because of its high surface area, porosity and water adsorption capacity. The MOF was combined with a thermoresponsive polymer hydrogel to desorb water accumulated during regeneration with less energy. The hydrogel is specifically designed to undergo a temperature-dependent transition that can promote water release using relatively mild temperature changes.

The objective of this research project was to synthesise and characterise a MIL-101(Cr)-based thermoresponsive hydrogel and to investigate its morphology, thermal behaviour and water uptake relevant to atmospheric water harvesting.

2 Chemical basis of MIL-101(Cr) formation

MIL-101(Cr) is a chromium(III) terephthalate metal-organic framework composed of trinuclear chromium oxo clusters connected by terephthalate linkers. The trinuclear chromium oxo cluster acts as a secondary building unit (SBU), while terephthalic acid, or 1,4-benzenedicarboxylic acid (BDC), acts as the organic linker connecting neighbouring SBUs. [8]

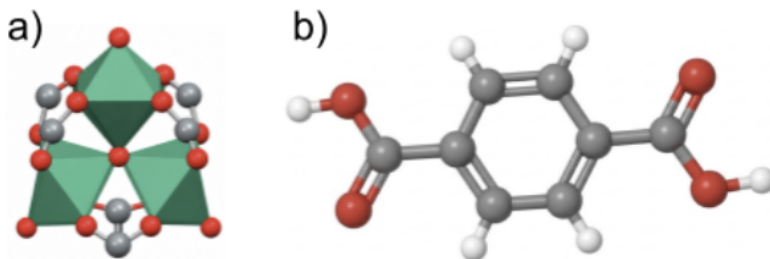


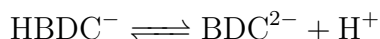
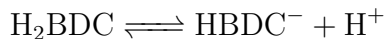
Figure 1: a) single trinuclear chromium oxo cluster [9], b) BDC

When chromium nitrate nonahydrate is dissolved in water, it dissociates, producing hydrated Cr^{3+} ions. These chromium ions initially coordinate with water molecules, giving them an approximately octahedral coordination environment. Some of the coordinated water molecules can then undergo hydrolysis, producing chromium species that can subsequently condense during the hydrothermal reaction. This process is shown at the start of **Figure 3**.

BDC has two carboxylic acid groups but is only sparingly soluble in water. The small fraction that dissolves can undergo deprotonation, producing carboxylate groups capable of coordinating to chromium ions. These groups begin to replace coordinated water molecules around the hydrated chromium ions, providing precursors for the developing framework. Sonication helps produce a more homogeneous mixture by breaking down BDC particles.

During hydrothermal heating, the increased temperature and pressure help both the hydrolysis of chromium species and the dissolution of BDC. The hydrolysed chromium species condense to form trinuclear chromium oxo clusters. Each cluster consists of three octahedrally coordinated Cr^{3+} ions connected through a central μ_3 -oxo (O^{2-}) atom. Extra oxygen atoms from carboxylate groups, together with terminal water or hydroxide ligands, complete the coordination environment of the chromium centres. [10]

Simultaneously, BDC undergoes further deprotonation to form the terephthalate dianion, BDC^{2-} :



The resulting BDC^{2-} ions bridge neighbouring chromium clusters through their two carboxylate groups. Each linker connects two SBUs, while each trinuclear chromium cluster is connected to multiple neighbouring SBUs. Repetition of these metal-ligand connections produces the extended three-dimensional MIL-101(Cr) framework. As the autoclave cools, crystal growth slows and the formed MIL-101(Cr) structure becomes stable. [8]

3 Research Design and Methodology

The experimental work was divided into two main phases: the synthesis and purification of the MOF MIL-101(Cr), and its later integration into a thermoresponsive polymer hydrogel, called PNIPAM. This composite uses both the water-adsorbing properties of the MOF and the temperature-responsive behaviour of the PNIPAM. After creating the MOF-hydrogel, it was cleaned, frozen, and freeze-dried before being characterised using scanning electron microscopy (SEM), differential scanning calorimetry (DSC), and water-uptake measurements. With this, we could analyse the structure of the composite and its suitability for AWH.

3.1 Synthesis of MIL-101(Cr)

MIL-101(Cr) was synthesised by coordination with the two main components under hydrothermal conditions, meaning at high temperatures in an aqueous solution. The first main component, chromium(III) nitrate nonahydrate, $Cr(NO_3)_3 \cdot 9H_2O$, was used as the chromium source, while the second main component, BDC, as the organic linker. The steps followed to synthesise MIL-101(Cr) were:

1. Add 0.4 g of chromium(III) nitrate nonahydrate and 0.166 g of BDC to 6 mL of deionised water in a PTFE liner and stir at around 1000 rpm for 5 minutes
2. Sonicate the mixture for around 20 minutes and insert an ice bag in the water tank so that the temperature will not rise excessively and potentially alter the chemical properties of the mixture
3. Subsequently, add 0.06 g of acetic acid and stir for around 10 minutes
4. Now, transfer the PTFE liner in a steel autoclave and seal the lid of the autoclave with a rod

5. Once sealed, heat at 220 °C for 12 hours. Under these hydrothermal conditions, the chromium-containing species and organic linker react to form the crystalline nanoporous structure of MIL-101(Cr). After this reaction, let the autoclave cool overnight
6. Once cooled, distribute the aqueous solution equally in centrifuge tubes. Create pairs of tubes and number them, and weigh each pair with a balance to ensure they have approximately the same mass. If not, add deionised water with a pipette to equalise. Centrifuge for 10 minutes at 7830 rpm (following centrifuging steps will have the same rpm), and once done, dispose of the liquid, leaving only a solid sediment in the tube
7. Now, purification. Add 1.5 mL DMF and disperse the solid in the tube by crushing it with a spatula or using a vortex mixer. Transfer the solutions in each tube into new ones, ensuring each new tube keeps the old numbering, and ensure each pair has again the same mass, and if not, add DMF to equalise. Sonicate for around 10 minutes and once done, centrifuge with the same settings. Without transferring the solutions into new tubes (but dispersing again if necessary), repeat this process twice more. Once having centrifuged for the third time and having disposed of the DMF, add 1.5mL ethanol in the tubes. Use the same steps as for DMF to disperse and transfer the solution into new tubes, and repeat the same steps for purification as for DMF but using ethanol instead, and once having centrifuged with it three times, using deionised water instead for two centrifuge steps. After the second centrifuge and having added deionised water and dispersed, measure the mass of the new empty tubes and record it, then transfer the solutions into the new tubes, similarly ensuring the mass is the same for each pair. Centrifuge for the final time, dispose of the liquid and measure the mass of the tube with the MOF inside

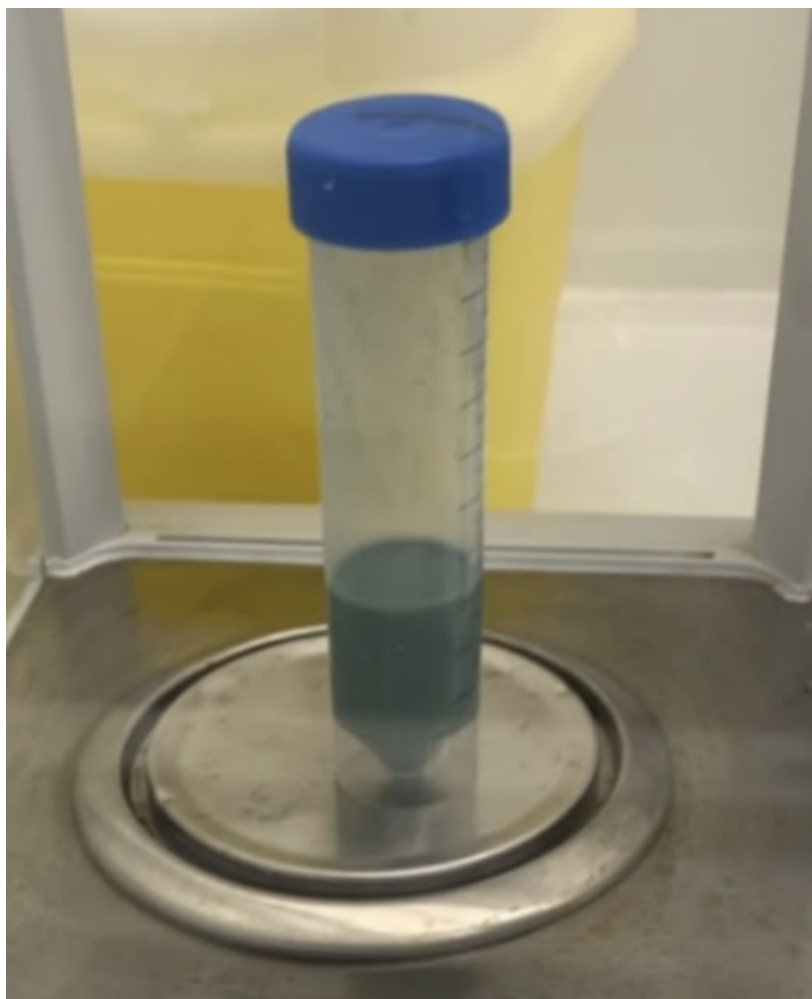


Figure 2: Final MOF-water solution being weighed

3.2 Fabrication of the MOF-based hydrogel

The purified MIL-101(Cr) was then incorporated into the thermoresponsive polymer hydrogel. The hydrogel is based on N-isopropylacrylamide (NIPAM), a polymer-forming monomer with temperature-responsive behaviour. To integrate the MOF with the hydrogel, the instructions followed were:

1. Being able to know the mass of the purified MIL-101(Cr) in the tube, we can add a specific amount of deionised water based on what we need the density to be. For this experiment, around 1 mL of deionised water can be added, ensuring to disperse the solid in the solution
2. Add 5 mL deionised water in a beaker and stir at around 1000 rpm. Then, add approximately 0.12 g (or 0.3 mL) of MOF
3. Separately, stir the crosslinker MBA solution at around 1000 rpm and prepare the vacuum desiccator by adding a small quantity of water in the bowl, later transferring the beaker with the MOF-deionised-water solution inside the desiccator, with the water level in the bowl only reaching barely above the base of the beaker to not risk it tipping over while stirring

4. Add to the beaker 0.567 g of NIPAM, 0.173 mL NDEAM, 38 mg MBA (or 1.9 mL, as we dissolved the MBA in deionised water with a concentration of 20 g/L), and 23 mg of APS (or 0.46 mL, as we dissolved the APS in deionised water with a concentration of 50 g/L), which initiates the in situ free radical polymerisation process
5. Now that the polymer precursor mixture is ready, add 4-5 ice cubes next to the beaker in the desiccator bowl so that the temperatures do not rise excessively and seal the bowl with the dome. Vacuum for 1 hour
6. Once the vacuum is finished and the dome is removed from the bowl, add 40 μ L of TEMED, which accelerates the in situ process, and after around 10 seconds, transfer the resulting mixture in petri dishes acting as moulds, where the polymer network can form around the dispersed MOF particles. Store these moulds in a refrigerator at 5 $^{\circ}$ C for around 12 hours

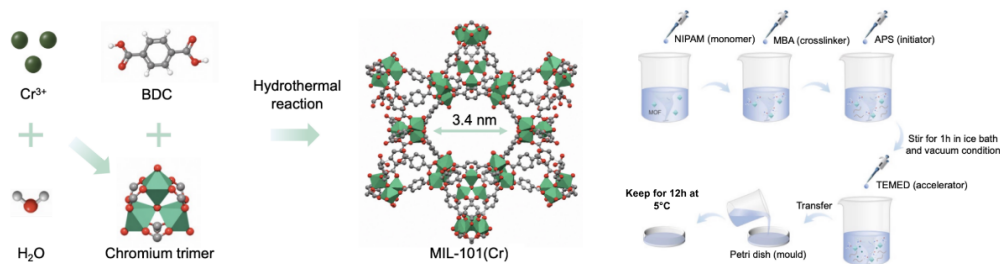


Figure 3: MOF-hydrogel synthesis diagram

As detailed in **Figure 3**, this in situ free radical polymerisation allows for the MOF to be integrated directly into the developing polymer network rather than having to add it into a pre-formed hydrogel.

The resulting composite was designed around the advantages of its two components, as shown in **Figure 4**. MIL-101(Cr) provides a highly porous structure with a high affinity for water which can adsorb the water molecules in the air, however it requires temperatures too high (and therefore too much energy) to desorb the water. The thermoresponsive PNIPAM polymer network can undergo a temperature-dependent change in its interaction with water, helping the composite desorb the water with only mild temperature changes. As **Figure 4** illustrates, above its Lower Critical Solution Temperature (LCST), the hydrogel can transition from a hydrophilic, swollen state to a hydrophobic, shrunk one when heated above its LCST and it is precisely this hydrophilic-to-hydrophobic change that helps desorb the water in the composite. [11]

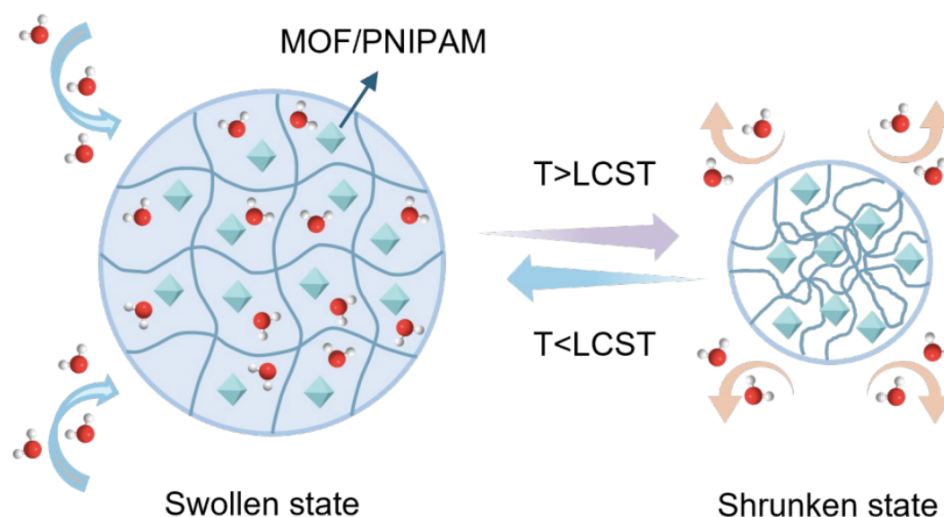


Figure 4: Adsorption by MOF and thermoresponsive desorption by PNIPAM

3.3 Cleaning and drying

Following the refrigeration of the polymerised composite, it was then further cleaned and processed. This was done with the following steps:

1. Add 4.32 g $\text{CaCl}_2 \cdot 2\text{H}_2\text{O}$ (calcium chloride) to 25 mL deionised water in a beaker and stir at around 1000 rpm until the calcium chloride dissolves in it. Once fully dissolved, transfer it to the freezer for about 5 minutes to cool, as this is an exothermic reaction
2. In the meanwhile, prepare a water heat bath and extract the MOF-hydrogels from the moulds in the fridge, placing them in the bath until the temperature reaches around 50 °C
3. Take the beaker out the freezer, place the MOF-hydrogels in it and place the beaker with the composites in it in the freezer for about 15 minutes, then transfer it in the fridge to stay overnight
4. Once this is done, transfer the MOF-hydrogels from the beaker into petri dishes and place them in the -80 °C freezer, leaving them in for approximately 24 hours
5. When this is finished, transfer the petri dishes with the MOF-hydrogels from the low-temperature freezer into the freeze dryer, where the water can be removed to create the dry composite used for characterisation

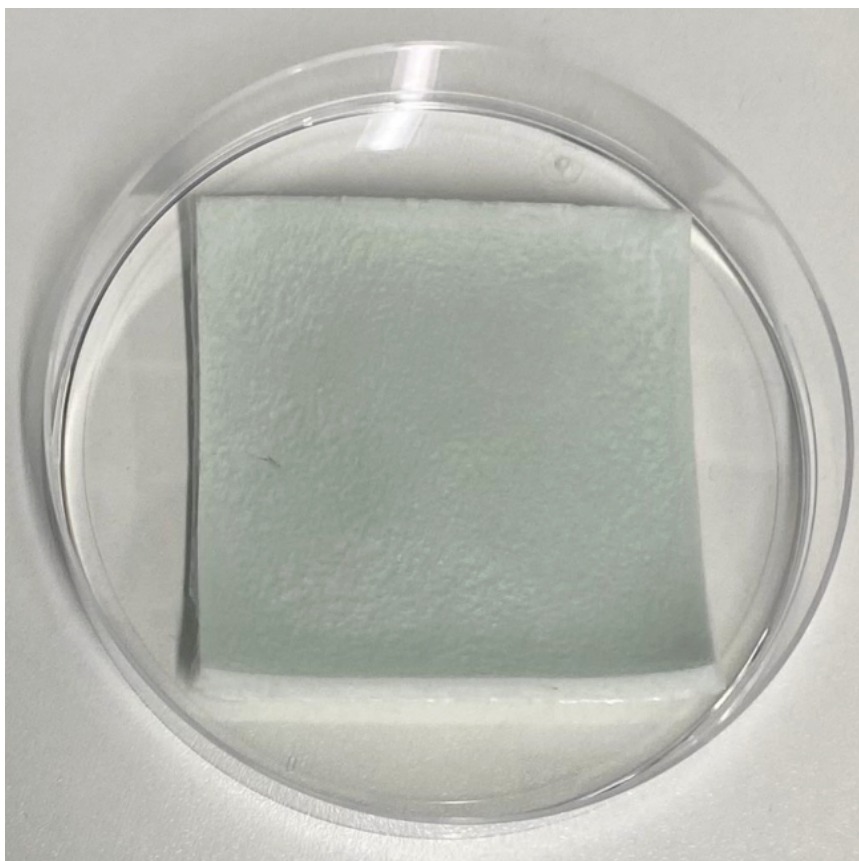


Figure 5: Final MOF-hydrogel composite result after freeze-drying

3.4 Characterisation

To determine the properties of the synthesised MOF and hydrogel, one can use SEM, which analyses the morphology of the material. For the MOF, the SEM allowed the crystal shape and texture and approximate particle size to be measured. For the hydrogel, it provided information on the structure of the polymer network and the distribution of the MOF particles on it.

DSC was instead used to determine the thermal behaviour of the MOF-hydrogel, particularly its LCST. This temperature threshold is vital because the thermoresponsive behaviour of the polymer is critical to the water-desorption functions.

Finally, the water-uptake behaviour of the composite was evaluated at a RH of 70%, with the resulting measurements demonstrating how much atmospheric moisture was adsorbed per gram of the composite materials and the rate of this adsorption.

4 Analysis and Findings

4.1 Morphology of the MIL-101(Cr)

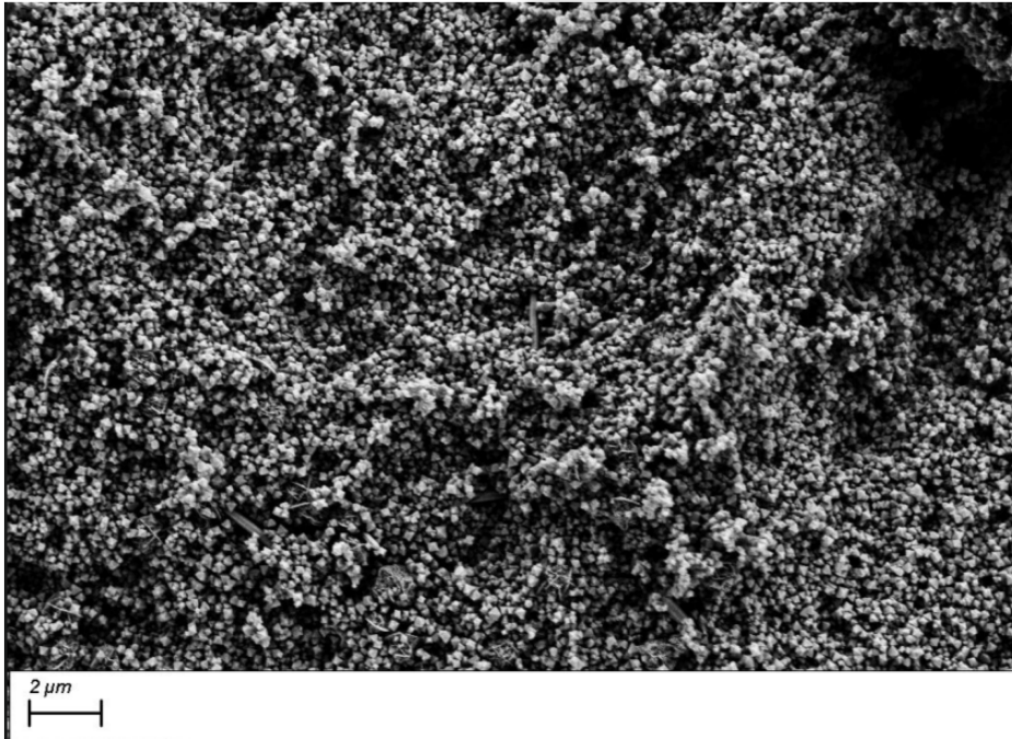


Figure 6: SEM of MOF particles

As **Figure 6** shows, SEM revealed the MOF particles displayed a mostly octahedral crystal morphology, with relatively smooth surfaces. This matches the usual formation of MIL-101(Cr), meaning my hydrothermal synthesis successfully produced well-defined, characteristic MIL-101(Cr) particles. [8]

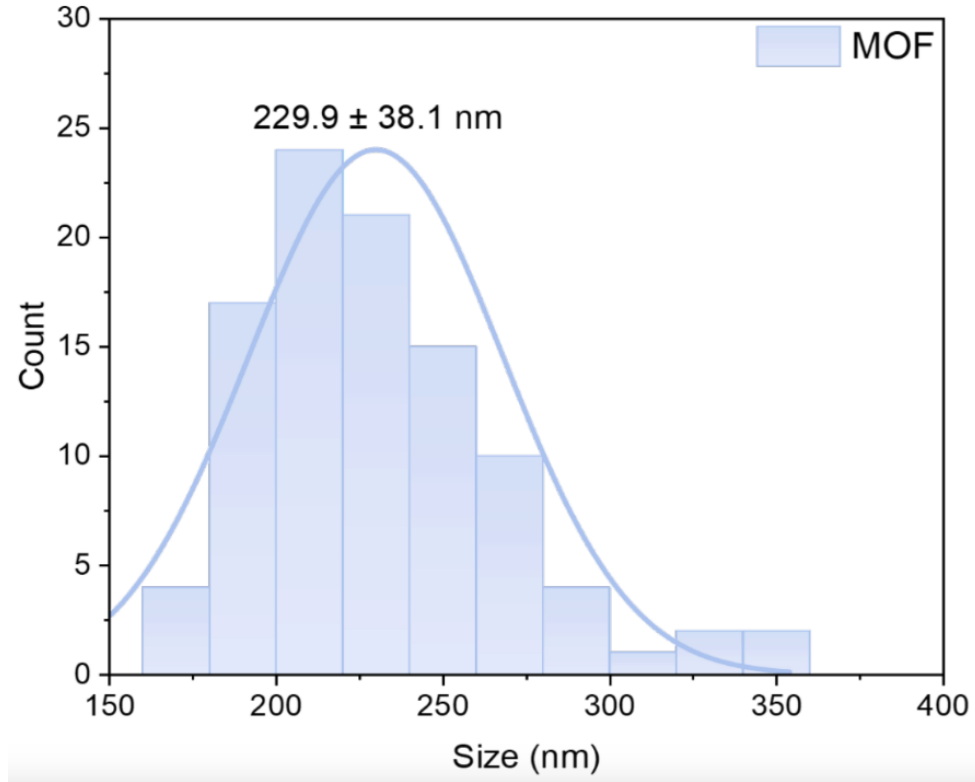


Figure 7: Count vs size graph

Figure 7 illustrates that the measured particle size distribution has an average size of approximately 229.9 ± 38.1 nm, corresponding to a particle size of about 230 nm. The formation of nanoscale MOF particles is beneficial when integrating them with the hydrogel because the MOF needs to be evenly dispersed throughout the polymer matrix. The relatively small particle size can help achieve an even distribution of the MOF within the 3D hydrogel structure, which in turn can enhance the hydrogel's water-adsorption performance due to the MOF's high porosity and surface area, allowing it to act as a hygroscopic material.

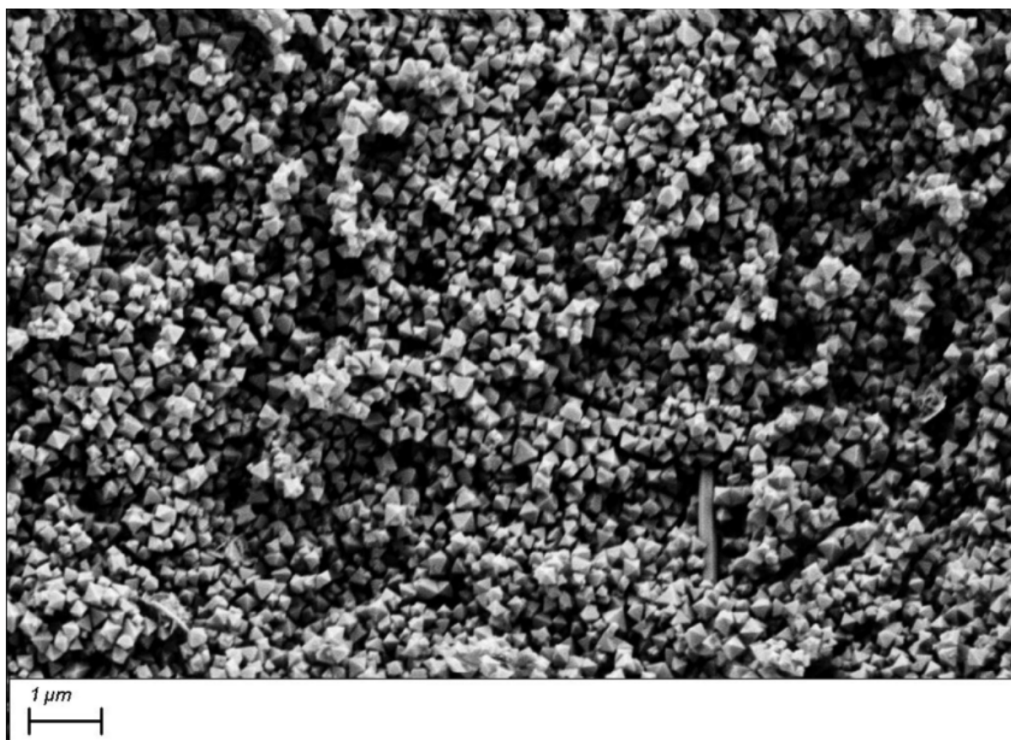


Figure 8: SEM of MOF particles, with octahedral morphology more visible

4.2 Morphology of the MOF-based hydrogel

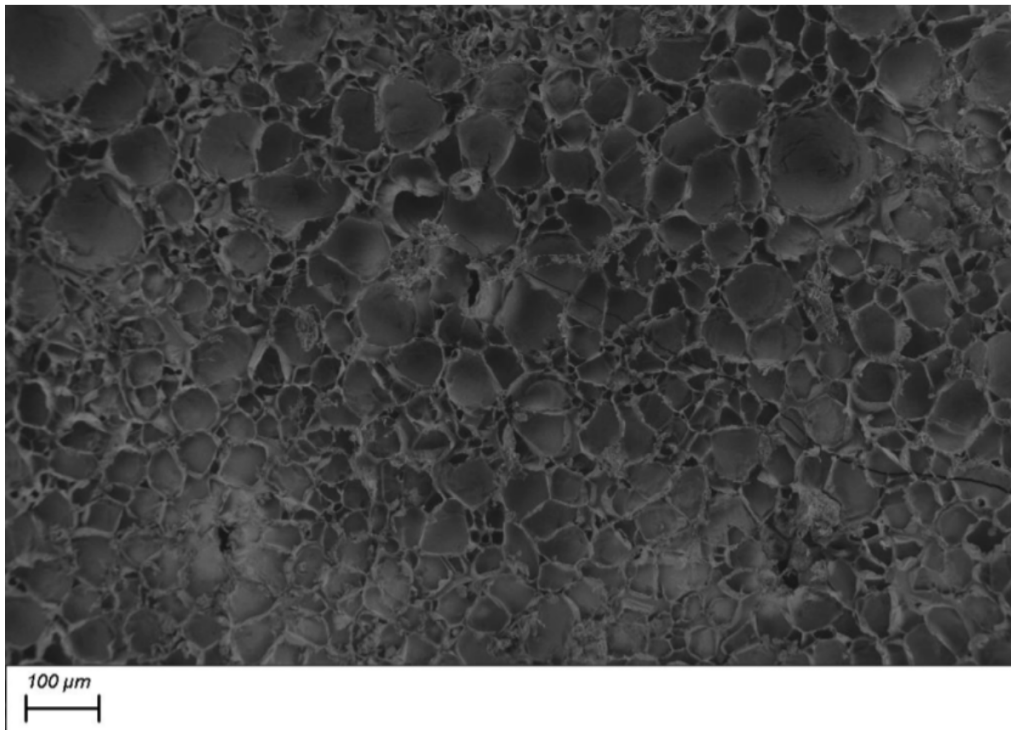


Figure 9: SEM of hydrogel

Figure 9 reveals the structure of the resulting MOF-based hydrogel. The hydrogel revealed a porous and interconnected network, providing the 3D framework for the

MOF particles to anchor to.

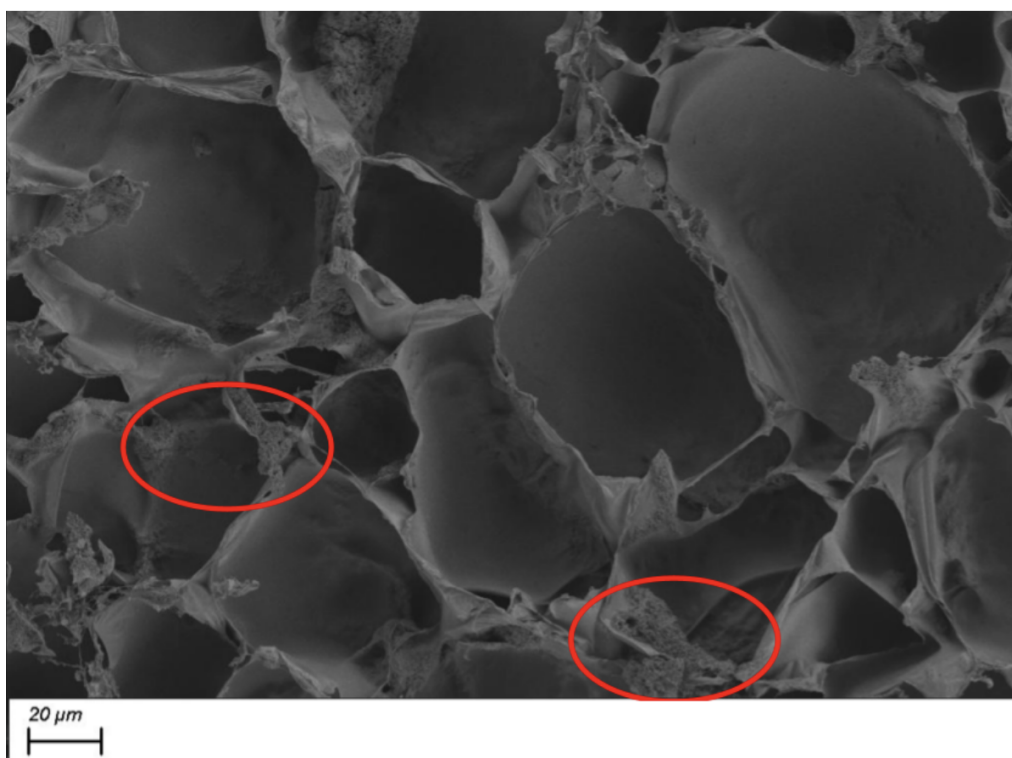


Figure 10: SEM of MOF-hydrogel, with a few MOF particle groups circled in red

Figure 10 also indicated the MOF nanoparticles uniformly distributed throughout and anchored to the polymer mesh.

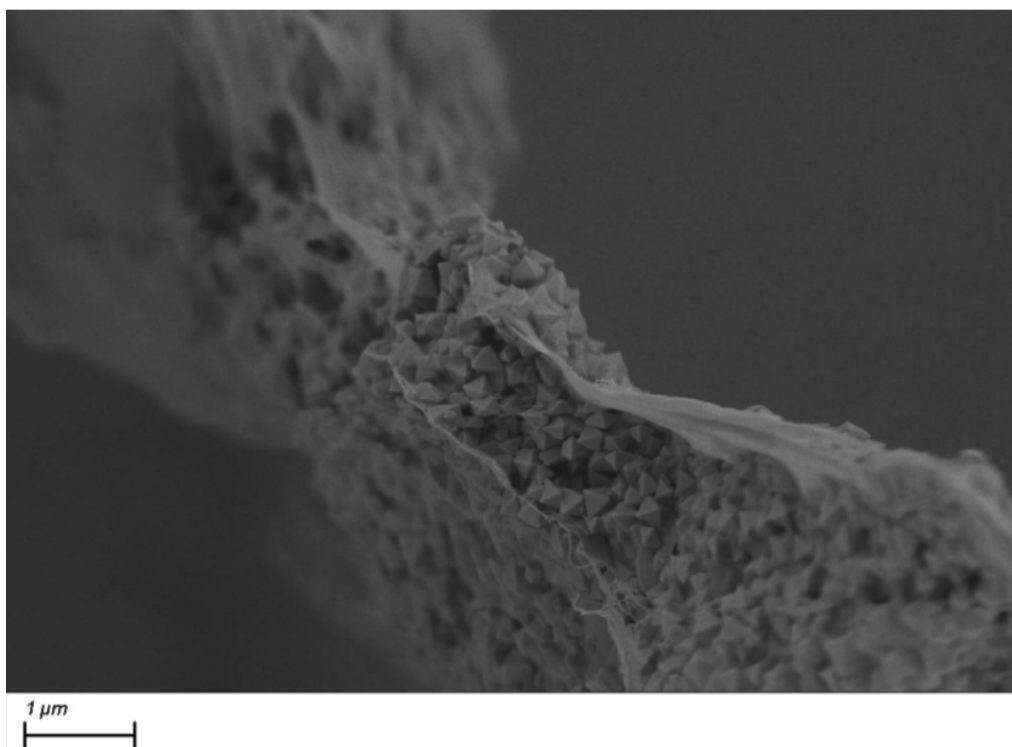


Figure 11: SEM of MOF particles on hydrogel

This structure is important because the 2 components have complementary functions. The MIL-101(Cr) provides the pores and affinity to water to adsorb the moisture in the air, while the PNIPAM polymer hydrogel forms the macroscopic framework of the composite and uses its thermoresponsive properties to help desorb the adsorbed water molecules. The polymer's interconnected pores may also increase the surface area available for water adsorption.

The SEM images therefore show that the synthesis was performed successfully to create a composite where the MOF was incorporated in the hydrogel rather than remaining as a separate material.

Nevertheless, SEM provides structural information only at the observed locations, and does not by itself represent values for the entire sample.

4.3 Thermal behaviour

The thermal response of the MOF-based hydrogel was analysed using DSC.

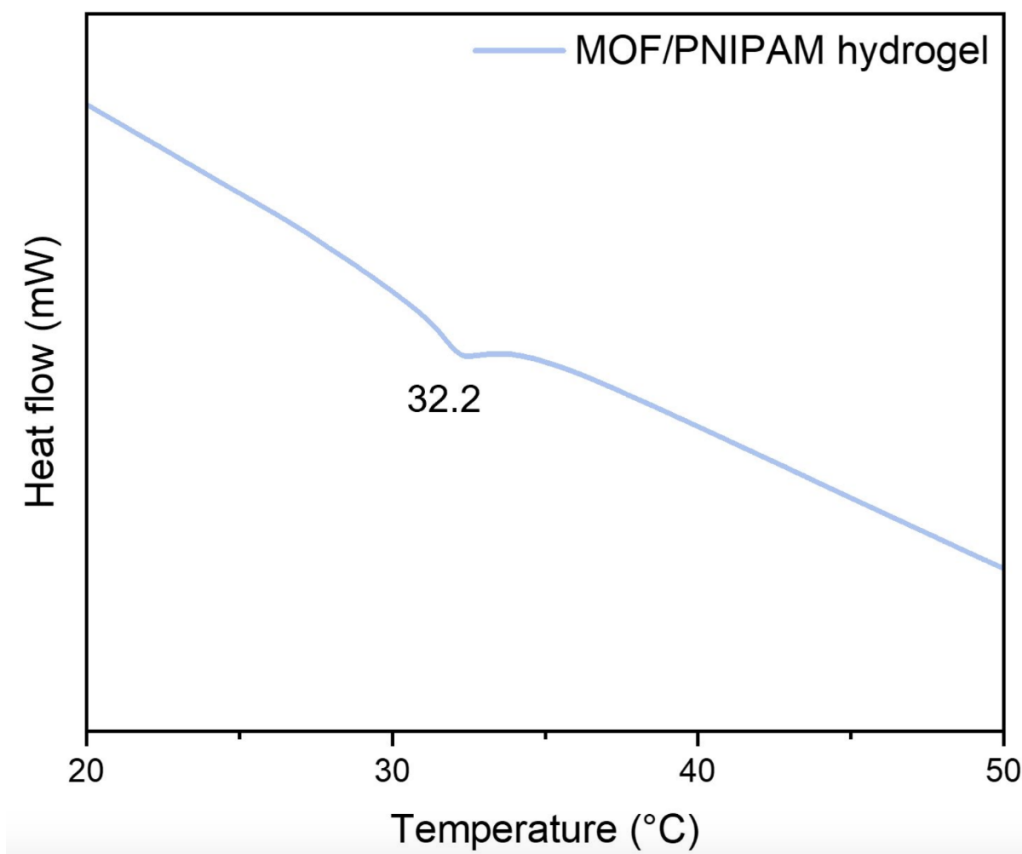


Figure 12: DSC graph showing LCST at 32.2 °C

Figure 12 shows an LCST of around 32.2 °C. This is crucial for AWH because a transition around 32 °C means that the relevant change in polymer behaviour happens at a relatively mild temperature increase rather than at high temperatures. This supports the concept of using modest temperature changes, including potentially low-grade or solar heat, to desorb water.

The measured LCST also likely indicates that incorporation of MIL-101(Cr) did

not significantly change the LCST of the PNIPAM-based hydrogel. This is relevant because it means that the thermoresponsive behaviour of the polymer was retained after addition of the MOF. The result therefore supports the combination of the MOF's water-adsorption function with the polymer's temperature-responsive function

4.4 Water-uptake behaviour

The water-uptake behaviour of the MOF-based hydrogel was evaluated at a RH of 70%.

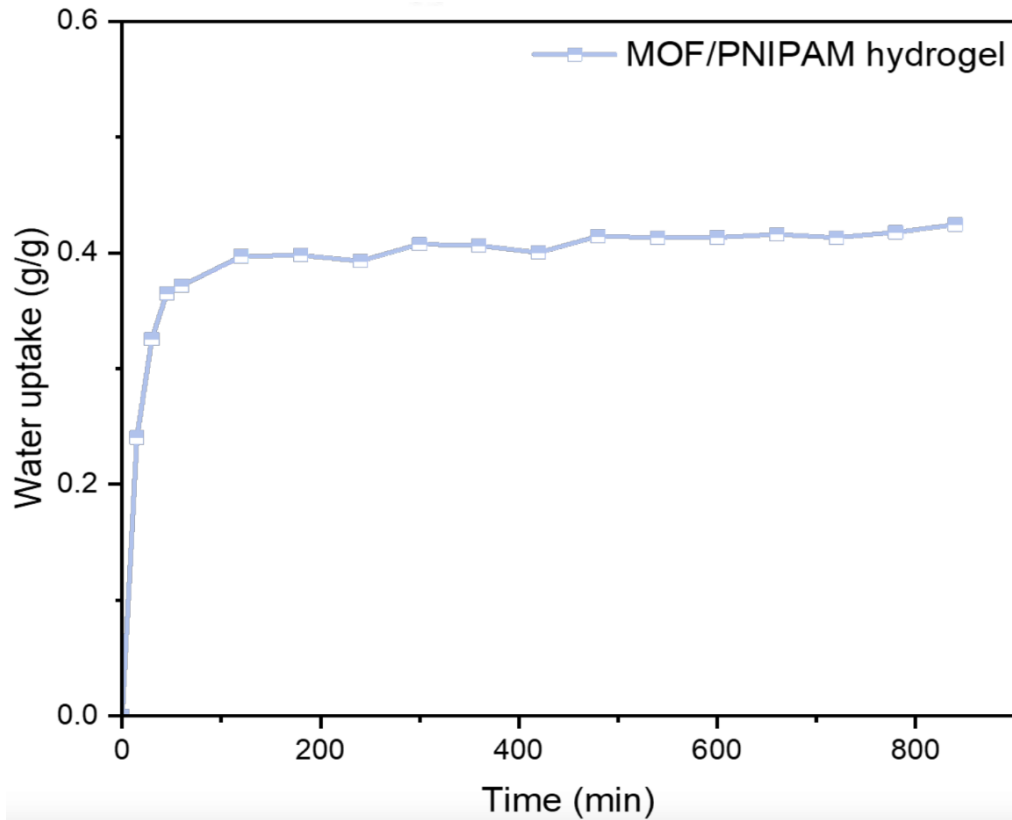


Figure 13: water uptake graph of MOF-hydrogel at 70% RH

From **Figure 13**, it can be seen that the resulting curve shows a rapid increase in water uptake during the initial stage of adsorption, then soon after stabilising for a relatively long period at around 0.4 grams of water per gram of MOF-hydrogel. The curve then approaches a relatively stable value, indicating that the material's rate of water uptake decreases as it adsorbs, reaching equilibrium. The material showed significant water uptake and rapid adsorption under these conditions, meaning it has potential for AWH. The initial steep increase is important to AWH as rapid adsorption can reduce the time needed for a harvesting cycle. The curve becoming stable could be because fewer adsorption sites are available.

However, the measurement was performed at a single RH, so it does not answer how the material would perform across the larger range of humidity and temperature conditions in real environments. Additional measurements under different RHs, temperatures, and repeated adsorption-desorption cycles would therefore be useful to better determine its practical performance.

4.5 Overall interpretation of findings

These results support the combination of the water-adsorbing properties of MIL-101(Cr) with the thermoresponsive behaviour of the polymer hydrogel. Simultaneously, the experiments represent an initial material-level assessment rather than a demonstration of a complete AWH technology, and need further work to better investigate their properties and applications.

5 Conclusion and Outlook

This project investigated the synthesis and characterisation of a MIL-101(Cr)-based thermoresponsive hydrogel for AWH. MIL-101(Cr) was first synthesised under hydrothermal conditions and subsequently purified then incorporated into a polymer hydrogel through in situ free radical polymerisation. The resulting composite was cleaned, dried, and characterised to evaluate its morphology, thermal response and water-uptake behaviour.

The characterisation results demonstrated the successful formation of the intended material. SEM analysis showed the synthesised MIL-101(Cr) formed approximately 230 nm octahedral particles, while the hydrogel had a porous, interconnected structure with MOF particles uniformly distributed throughout the polymer mesh. DSC revealed an LCST of around 32.2 °C, meaning the composite kept the thermoresponsive behaviour of PNIPAM after integration with the MOF. Water-uptake measurements at 70% RH further showed rapid adsorption and substantial water uptake, demonstrating the material’s ability to capture atmospheric moisture under the tested conditions.

These findings support the concept of combining the water-adsorbing properties of MIL-101(Cr) with the thermoresponsive behaviour of PNIPAM. However, further work is needed to determine the material’s performance under realistic conditions as an operational AWH system, including its long-term stability and behaviour over repeated adsorption and regeneration cycles. Future studies could investigate the material’s water uptake and release under a wider range of realistic humidity and temperature conditions, as well as its scalability for practical applications.

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8 Appendix

8.1 Research poster

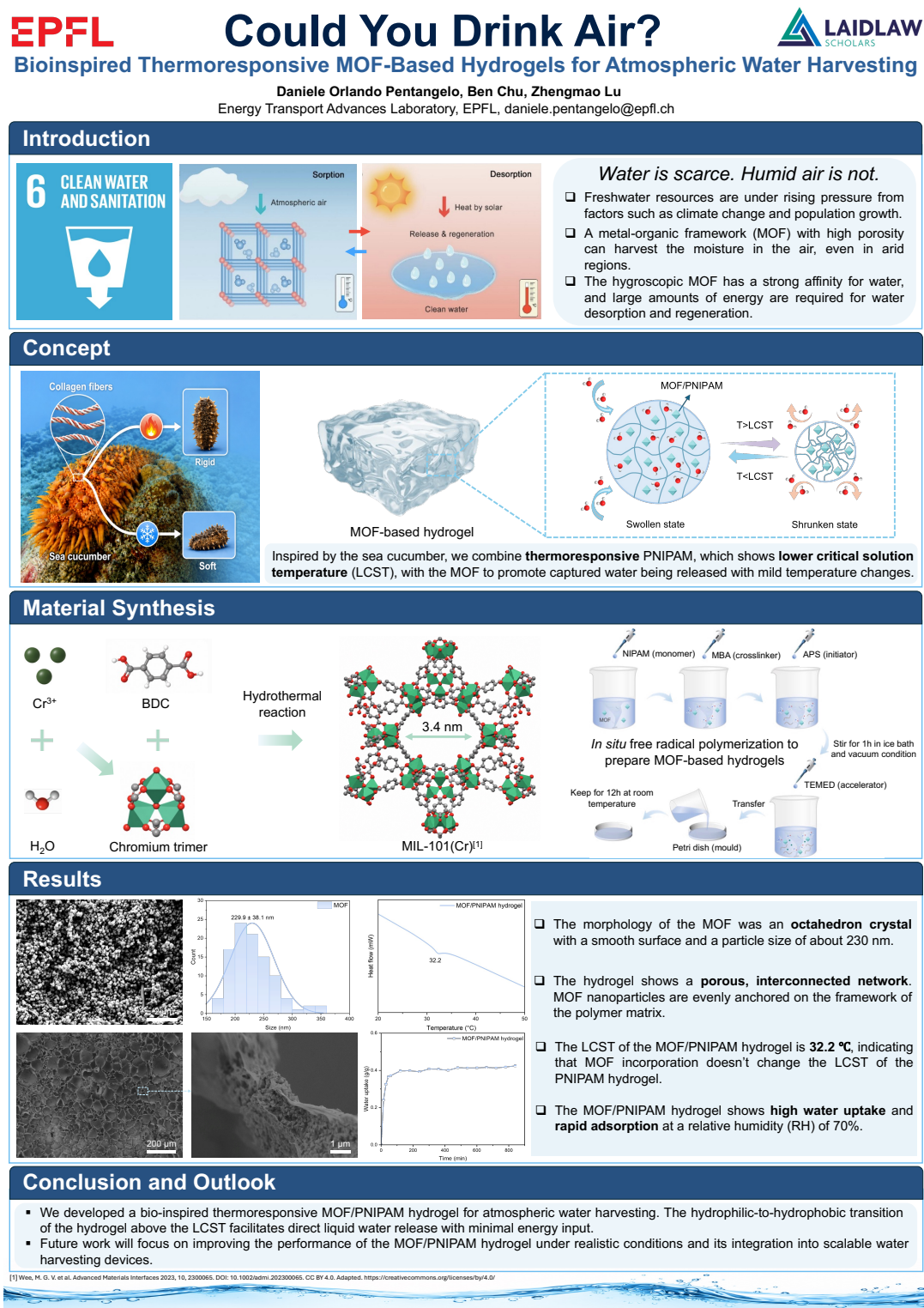


Figure 14: Research poster. Note in the MOF-hydrogel diagram in “Material Synthesis”, the final step differs slightly, but it is simply a different way of creating the composite