Harvesting Water in the Classroom

S. Ephraim Neumann,* Kallie Neumann, Zhiling Zheng, Nikita Hanikel, Jonathan Tsao, and Omar M. Yaghi



as confirmed through characterization by powder X-ray diffraction and water sorption measurements. A post-lab assessment indicates that the cutting-edge nature of the materials and related research fuels student motivation and spurs a broader conversation about ongoing scientific research.

KEYWORDS: Metal-Organic Frameworks, Students, High School, Accessibility, Climate, Water

INTRODUCTION

At both the high school and undergraduate levels, a disconnect exists between standard chemistry curricula and modern research efforts. To facilitate the understanding of core content objectives, students traditionally participate in a selection of common laboratory exercises that have been iterated upon and optimized within individual classrooms and, more broadly, throughout the science education community, for many years. These core laboratories are invaluable to student mastery of both content and skills objectives; it has been observed that physical experiences with scientific concepts lead to higher rates of knowledge retention and improved performances on follow-up assessments.¹ However, because these "cookiecutter" laboratories are designed to have predictable outcomes, they do not reflect the ongoing nature of scientific inquiry.

Studies suggest that offering students the opportunity to engage in authentic, real-world tasks is central to creating classroom environments that motivate students and promote learning.² We propose that providing students hands-on access to cutting edge research, tangible access beyond popular science articles or an extra PowerPoint slide at the end of a lecture, has the potential to instruct them more effectively about the ongoing value of scientific inquiry, as well as to raise awareness about current issues that can be addressed with science and excite future generations of researchers. Despite the proposed benefits, several challenges must be addressed. Successful implementation of such an approach requires the choice of a topic that is relevant and significant to the student audience, does not present too steep a learning curve relative to the students' prior knowledge of the subject, and requires only those materials and equipment that are accessible from safety and expense standpoints. Even with a topic that meets these criteria, the translation of laboratory research into suitable classroom experiments is not trivial.

In this study, we present the development and implementation of laboratory experiments that are appropriate for both high school and undergraduate settings. At the heart of this work is the emerging field of reticular chemistry and, specifically, metal–organic frameworks (MOFs). MOFs are porous, crystalline materials whose unique properties have generated significant interest among the chemistry and materials science communities.³ Although reticular chemistry has not yet been established as a standard in chemistry curricula at any educational level, potential applications of MOFs for environmental purposes, such as the capture of greenhouse gases or water,^{4–8} make them exceptionally relevant to students of any age. A few experiments tailored for high school students have been reported,⁹ but most MOF-

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Figure 1. Structural models of aluminum fumarate obtained from experimental data using X-ray diffraction techniques. (a) Structural model highlighting the connectivity and chemical structure of the MOF to deduce the chemical formula $AlC_4H_3O_5$ or $Al(OH)C_4H_2O_4$. (b) Ball-and-stick model of the rod-shaped structure formed by the corner-sharing aluminum octahedra linked through fumarate units. (c) 3D model of the framework emphasizing the porosity of the MOF.



Figure 2. Experimental design. (a) Experimental setup for humidity chambers with specific relative humidity. A resealable hole is cut into the plastic lid to minimize exposure during relative humidity measurements. The weighing boat is used to hold the MOF sample during water adsorption experiments. (b) Water isotherm for the aluminum fumarate MOF synthesized under the given conditions measured at 25 $^{\circ}$ C. The results of the water adsorption experiment should match the trend predicted by the isotherm. Samples placed in humidity chambers with a relative humidity before the step do not show the uptake of water, whereas significant changes in sample mass can be observed for the MOFs in chambers with higher relative humidities. (c) Observable water condensation at the walls of the Erlenmeyer flask and the watch glass while heating the MOF on a hot plate.

related educational literature requires undergraduate-level knowledge and equipment.^{10–12} In light of the growing threat of water scarcity¹³ in the greater context of climate change, for this experiment, we focused on the use of MOFs for atmospheric water harvesting.

Harvesting drinking water from air has been identified in a previous study as a great opportunity for teaching students about science and shedding light on the global significance of water as a limited natural resource.¹⁴ In comparison to the aforementioned study, which focuses on the collection of water from humid air using a so-called fog harp, the experiment presented in this work employs an approach for harvesting water at lower relative humidities. Although this presents the students with a more complex and knowledge-demanding

system, the experiments are designed to be performed without any prior knowledge. Educators may adjust the depth to which the students should understand the underlying chemistry to reach their target audience. In this study, students independently performed a simplified synthesis of aluminum fumarate (Figure 1), a MOF with known water-harvesting capabilities,^{15–17} and prepared and investigated its ability to uptake and release water as a function of the local environment's relative humidity. Pre- and post-assessments provided insight into student gains.

EXPERIMENTAL SECTION

The laboratory experience was divided into 4 experimental sections. Each section introduced the students to fundamental

concepts and encouraged discussion before, during, and after the experimental steps were completed. For example, to start the series of experiments, the students were introduced to the threat of increasing global water scarcity and the concept of relative humidity. The students brainstormed as a group about potential sources of drinking water, how they could be accessed, and their feasibility in terms of energy consumption. Finally, after an instructor-facilitated discussion about the causes and effects of limited access to drinking water, the first experiment was introduced. Student handouts and instructor notes for facilitating discussion are included in the Supporting Information.

Water Scarcity and Relative Humidity

The students were tasked with making saturated solutions of a series of salts. The salts were chosen to generate atmospheres with varying relative humidity within an enclosed plastic container.¹⁸ By cutting a resealable hole into the lid of the container (Figure 2a), the relative humidity could be measured with a hygrometer and compared to the theoretically expected values. The time frame for these measurements ranged from minutes to days to observe the stabilization of the relative-humidity readings. Observed differences between theoretical and experimental data were addressed and discussed with the students.

Synthesis of the Metal–Organic Framework

The second experiment introduced the students to the porous structures of metal-organic frameworks and prompted them to produce an aluminum-based MOF that is known for its water-harvesting capabilities. For a typical 100 mL reaction, two solutions were prepared. First, fumaric acid (5.8 g, 50 mmol) and sodium hydroxide (6 g, 150 mmol) were dissolved in 50 mL of deionized water in a beaker (250 mL). The resulting solution was stirred for 3-10 min until all the solids were completely dissolved. In a separate beaker, aluminum chloride hexahydrate (12 g, 50 mmol) was dissolved in 50 mL of deionized water. Once the solid was fully dissolved, the aluminum chloride solution was added dropwise to the beaker with stirring at room temperature. The total addition time lasted for 1-2 h, during which the formation of a white precipitate was observed. (Note: If available, addition funnels or burets can be used to limit active operating time for the students.) After the addition was completed, the solution was stirred at room temperature for 24-48 h. (Note: In case no magnetic stir plate and stir bar are available, the solution can be stirred with a glass rod and left standing without stirring overnight.) The resulting white powder was collected by filtering and washing with deionized water. (Note: If a centrifuge is available, it can be used to speed up the collection and washing of the product.) The white solid was then washed with water and, subsequently, ethanol three times each and dried overnight. To remove the remaining solvent from the pores of the framework, generally referred to as activation, the MOF was kept in an oven at 120 °C for 24 h, yielding a pure and solvent-free product (yield: 81% based on the starting material).

Adsorption and Desorption Experiments

The students were briefly introduced to the concepts of gas/ vapor adsorption, desorption, and the corresponding isotherms. To investigate the water sorption capabilities of the synthesized MOF, the humidity chambers constructed in the first section were prepared by placing small pieces of Styrofoam inside the containers to keep the samples dry (Figure 1a). Afterward, designated amounts of MOF were weighed in on weighing boats, which were subsequently placed on top of the Styrofoam inside the closed humidity chambers. The students recorded the exact masses of the MOF samples (including the weighing boats). After 24 h, the samples were weighed again, and the results were compared to the previously recorded masses. (Note: It is worth mentioning that, based on the relative humidity in the different chambers, both an increase and decrease in mass may be observed. This can be attributed to the efficiency of activation and the handling of the samples, which should be addressed by the students.) The results were discussed and compared to expectations based on the reported water isotherm for the material (Figure 2b).

Proof of Concept for Water Harvesting from Air

A final demonstration was employed to make the waterharvesting capabilities of the MOF more tangible for the students. For this purpose, the total amount of synthesized material was combined in a beaker and closed with a watch glass. (Note: At this point, the MOF should have been able to gather water from the room's atmosphere. If the relative humidity in the room is consistently below 30%, the material should be stored in a more humid environment.) To release the stored water, the MOF in the beaker was heated on a hot plate. The condensation of water was observable at the walls of the beaker and the watch glass within minutes (Figure 2c). As a conclusion to the experiment, the students discussed mechanisms that may be used to control the adsorption and desorption of water. (Note: The shape of the water isotherm is temperature dependent, which leads to adsorption/desorption of water at different relative humidities.)^{17,19} Additionally, ideas for a potential design of a water harvester were considered to exemplify the practical use of porous materials for harvesting water from air. This presents a possible extension activity for the enterprising instructor.

HAZARDS

Safety goggles and gloves should be worn during each part of the experiment. The participants should be instructed to review safety measures and safety behavior guidelines in a laboratory setting at the beginning of the experiment. Throughout the experiments, the students will handle several chemicals that qualify as bases (e.g., sodium hydroxide), which are corrosive and should be handled carefully. The salts used in the saturated solutions should be chosen in accordance with the age and capability of the students. For example, lithium chloride poses a significant threat when ingested and should not be used with younger students. All solutions should be neutralized and disposed following local health and safety guidelines. If necessary, every part of the experiment can be performed as a demonstration by the instructor.

RESULTS AND DISCUSSION

The experiments were conducted with 6 high school students at Proof School in San Francisco, California. All participants were informed of the use of the anonymous data and gave their consent for using the data. Throughout the course, the students worked in self-selected pairs. All participating students are generally high-achieving and show particular interest in STEM. This group of students elected to participate in this laboratory experience, choosing it over a pool of alternatives. Although all experiments are designed to be conducted with



Figure 3. Exemplary student data. (a) Observed water uptake for the MOF in atmospheres of varying relative humidities. The water uptake is in agreement with the expectations based on the reported water isotherm. Negative water uptake at low relative humidities indicates experimental error (e.g., water adsorption during transfer from the oven to the balance and then to the humidity chamber). (b) Water adsorption and release based upon the relative humidity of the environment in which the MOF is placed.

minimal laboratory equipment and expertise to make this series of experiments as accessible as possible, advanced glassware and equipment, such as a centrifuge, may be used if available. Similarly, the depth of content taught can be adjusted to the educational background of the students. For example, the structure of the MOF can be discussed in a manner appropriate to the audience, as there are many ways to present these complex structures (Figure 1).²⁰ Notably, it has been shown that exercises in spatial thinking can improve STEM learning.²¹ As each part may take more than one lesson to complete, the entire experiment is spread out over the course of a densely packed block or several class periods.

The three main goals of the experiments are to spur the student's curiosity and interest in science, provide hands-on experience in a laboratory setting, and make cutting-edge technology more tangible. To achieve these goals, the experiments are simple and safe enough to reach younger audiences. Furthermore, the greater topic of climate-related current research is very relevant to every student, which makes it easy to motivate this learning activity. The students were asked to complete a prelab assessment to gauge their prior knowledge. Broadly, none of the students had ever heard of MOFs or other relevant materials and applications. Although some students could make some sense of water scarcity and its environmental causes, none were able to identify the potential solutions to the problem. The background in general chemistry knowledge varied greatly from almost none to entry level as no prior qualification was required for participation in the experiment.

The first meeting with students was used to introduce safe laboratory habits and to motivate the laboratory experiments by discussing water scarcity and other climate related dangers. The salts used in this experiment can be chosen from a wide range, but it is recommended to use potassium acetate and at least one salt that generates an atmosphere with a relative humidity below 20% (e.g., NaOH). Each group was responsible for making two saturated salt solutions. Students were instructed to add salt to a specified volume of water until no dissolution could be observed, and a sizable amount of salt remained at the bottom of the container. To measure the relative humidity with a commercially available hygrometer, small holes were cut into the plastic lid and sealed with tape (Figure 2a). Measurements were taken at 10 min intervals for 1 h, and a final measurement was taken after 24 h. During the short intervals, a clear salt-dependent trend into higher or lower relative humidity ranges with respect to the room's relative humidity could be observed. Interestingly, even after 24 h, the theoretically expected humidity values could not be reproduced. As this experiment acts as an introduction and preparation for upcoming steps, the differences in relative humidity above the saturated salt solutions were briefly discussed with the audience. It is worth noting that the accuracy of the relative-humidity readings greatly depended on the experimental setup and time frame for the measurements. If the atmospheres were not properly closed off to the outside atmosphere or were interfered with too often during measurements, the readings showed significant discrepancies from the expected values.

In the second experiment of the series, a water-harvesting MOF was synthesized. The conditions were optimized to be simple, which gives educators a lot of room to adapt the procedure. As the students were presented with the prospect of harvesting water from the air, it was important to address the audience in an appropriate manner. In this case, the students came with varying backgrounds, which required a broad explanation of hydrophilic and hydrophobic porous materials and how they potentially interact with water molecules. It is worth noting that the quality of the synthesized MOF, which was evaluated by powder X-ray diffraction (PXRD) and water sorption measurements, is comparable to previous scientific reports (Section S2). Importantly, the MOF was activated, which describes the process of emptying the pores of frameworks from remaining solvent molecules by heating it overnight in an oven. This process usually involves the use of heating in combination with a dynamic vacuum, which is not feasible for most teaching laboratories. For the following steps, heating the materials overnight at temperatures above 100 °C will be sufficient.

In preparation for the third experiment, the students were introduced to the concept of isotherms. Adsorption and desorption behaviors were discussed in the context of the experiment. The students carefully massed given amounts of MOF into weighing boats, which were subsequently placed on top of a piece of Styrofoam within the different humidity chambers. Each student group collected data for two of the six humidity chambers, and all data were shared following completion. Although weight measurements of the material after 1 h did not show significant uptake of water, a trend in weight gain or loss could be observed. After 24 h, the samples placed in humidity chambers with lithium chloride and sodium hydroxide showed a small decrease in weight. In contrast, the samples placed within the atmosphere above saturated solutions of potassium carbonate, sodium chloride, and potassium sulfate showed significant gains in mass. Interestingly, the MOF sample placed above the potassium acetate solution showed no measurable change in mass (Figure 3a and Table S1). These findings are well-aligned with the water isotherm for this MOF (Figure S2). The slight loss in mass for some of the samples was explained by readsorption of water upon exposure to air. This could potentially be avoided by using sealable containers in the oven such that they can be immediately closed after removal from the oven. As the relative humidity in the chambers with lithium chloride and sodium hydroxide is lower than that in the step of the isotherm, the desorption of remaining water molecules from the pores of the framework was to be expected. This finding is also in line with the fact that the sample over the saturated potassium acetate solution did not show any change in mass, as a small amount of water molecules is to be expected within the pores of the MOF at the onset of the isotherm step. To further facilitate a discussion about the harvesting of water from air, the water uptake of MOF samples that had been placed in environments with low (LiCl) and high (K₂SO₄) relative humidity was recorded, and subsequently switched. A final mass measurement was performed after 90 min. The plotted data showed the expected uptake of water at high relative humidity and the loss of water at low relative humidity, which was used to introduce humidity control as a potential release mechanism for water (Figure 3b).

The series of experiments was concluded with a teacher-led demonstration and subsequent discussion of the harvesting of the captured water from the MOF. For the demonstration, all MOF samples were combined after being left out to capture water from the room's atmosphere. If the relative humidity in the laboratory is consistently below 20%, it is advised to store the MOF in one of the humidity chambers. The material was then transferred to an Erlenmeyer flask, capped by a watch glass, and placed on a hot plate. Within minutes, the students were able to observe increasing condensation of water on the outside walls of the flask and the watch glass (Figure 2c). This experiment was used to discuss one challenge that current research is focusing on: energy efficiency. The importance of both the adsorption and desorption mechanisms and the relevance of energy efficiency were highlighted and put into a greater context of the climate impact of current energy sources.

In a post-lab written assessment, students were asked to describe their initial motivations for participating, as well as their takeaways and questions after completing the experiments. Of those assessed, 40% indicated that they chose to participate because of the real-world implications of this research and their existing interest in climate-related topics. Others were motivated by their previous interactions with the instructor and/or their general interest in science. In terms of primary takeaways, a significant fraction of the students correctly communicated that MOF research offers a potential solution to a current and growing problem. More broadly, they concluded that, beyond MOFs, there is a significant amount of innovative and novel research aimed at real-world problems. Furthermore, students expressed their surprise that, despite the simplicity of the experimental setup and limited access to laboratory equipment, they were able to reproduce previously reported results from professional laboratories.

The questions that were generated by students indicated their interest in the chemistry underlying the unique properties of MOFs, their cost and efficiency as water-harvesting materials, and their further potential applications. These responses show that, after completing the laboratory experience, students not only were able to ask relevant content-specific questions but also showed significant interest in MOFs and the broader application of these materials. Finally, although effective teaching of specific subject matter was not the main objective of this work, the post-lab questions assessed for understanding of four fundamental concepts. These items are listed below in ascending order of difficulty, along with the percentage of total students whose post-course assessment indicated proficiency.

- Water scarcity is a growing threat associated with climate change. (100%)
- Water can be harvested from the air using porous materials. (83%)
- Relative humidity plays a major role in the uptake behavior of MOFs. (67%)
- Water can controllably be taken up and released from the MOFs. (67%)

These results are promising. With further consideration to the teaching methods employed, these experiments could ostensibly be used alongside or in place of a more traditional lecture-style approach to facilitate understanding of the growing field of reticular chemistry and evoke the students' interest in science by engaging them in cutting-edge research. Student handouts and additional teaching materials for educators can be found in the Supporting Information.

CONCLUSION

Overall, this series of laboratory experiments allowed students to experience cutting-edge research with limited equipment and monetary resources. The ties to climate change motivated students to participate and offered them an opportunity to reflect upon the body of ongoing scientific research geared toward solving human problems. Through their post-lab responses, students demonstrated new and developing understanding and interest in MOF chemistry. Although they were implemented in a high school classroom, the experiments were designed to be as accessible as possible and could readily be adapted to the available resources and needs of the audience. Furthermore, it is conceivable that a similar approach could be taken to facilitate teaching of different science content in high school or undergraduate settings.

ASSOCIATED CONTENT

Supporting Information

The Supporting Information is available at https://pubs.acs.org/doi/10.1021/acs.jchemed.3c00690.

Additional data (includes PXRD, water isotherm, and exemplary student data) (PDF, DOCX) Instructor notes (PDF, DOCX) Student handouts (PDF, DOCX)

AUTHOR INFORMATION

Corresponding Author

S. Ephraim Neumann – Department of Chemistry and Kavli Energy Nanoscience Institute, University of California, Berkeley, Berkeley, California 94720, United States; orcid.org/0000-0002-8515-9621; Email: ephraim.neumann@berkeley.edu

Authors

Kallie Neumann – Proof School, San Francisco, California 94103, United States

Zhiling Zheng – Department of Chemistry and Kavli Energy Nanoscience Institute and Bakar Institute of Digital Materials for the Planet, Division of Computing, Data Science, and Society, University of California, Berkeley, Berkeley, California 94720, United States; Orcid.org/0000-0001-6090-2258

Nikita Hanikel – Department of Chemistry and Kavli Energy Nanoscience Institute, University of California, Berkeley, Berkeley, California 94720, United States; o orcid.org/ 0000-0002-3292-5070

Jonathan Tsao – Department of Chemistry and Kavli Energy Nanoscience Institute, University of California, Berkeley, Berkeley, California 94720, United States

Omar M. Yaghi – Department of Chemistry and Kavli Energy Nanoscience Institute and Bakar Institute of Digital Materials for the Planet, Division of Computing, Data Science, and Society, University of California, Berkeley, Berkeley, California 94720, United States; UC Berkeley–KACST Joint Center of Excellence for Nanomaterials for Clean Energy Applications, King Abdulaziz City for Science and Technology, Riyadh 11442, Saudi Arabia; © orcid.org/ 0000-0002-5611-3325

Complete contact information is available at: https://pubs.acs.org/10.1021/acs.jchemed.3c00690

Notes

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