

Synthesis, Characterization, and Evaluation of Metal–Organic Frameworks for Water Decontamination: An Integrated Experiment

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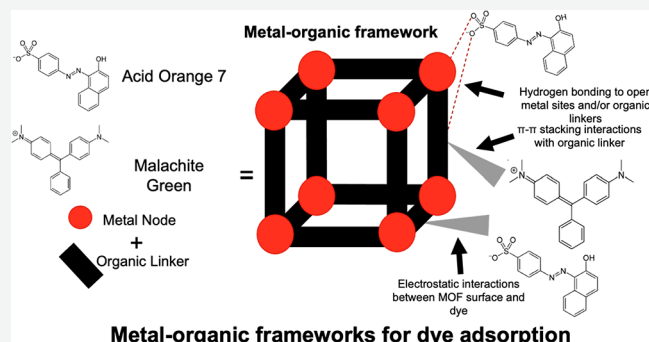
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ABSTRACT: An integrated laboratory experience using metal–organic frameworks (MOFs), a nanoporous and crystalline extended solid material composed of metal cations and organic linkers, was developed and adopted for upper-division, major-level chemistry and biochemistry students. In this laboratory, students were guided through the facile solvothermal synthesis of four MOFs: UiO-66, UiO-66-NH₂, ZIF-8, and ZIF-67. Characterization, specifically powder X-ray diffraction (PXRD), and energy-dispersive X-ray spectroscopy (EDXS) were used to evaluate the crystallinity, morphology, and composition of the MOFs, respectively. Students then investigated the effects of measurement conditions (MOF dosage, MOF surface charge, and charge density of dye counterions) on the removal of dyes from aqueous solutions using the synthesized MOFs. Through the utilization of Fourier-transform infrared spectroscopy (FT-IR) and UV–vis spectroscopy, students confirmed the binding of dyes and quantified the amounts of dye removed from solution. Students found that all four porous and crystalline MOFs removed acid orange 7 and malachite green from water, but ZIF-67 exhibited the highest adsorption capacity of 30 mg of acid orange 7 per gram of ZIF-67 at a dosage of 5 mg. This laboratory experience allows students to apply and connect concepts found in general, organic, inorganic, and physical chemistry while simultaneously getting introduced to various instrumentation and lab techniques within a growing field in materials chemistry. Exposure to this field can help stimulate student interest, expose them to sustainability problems and potential solutions, and implement strategies to help solve these challenges.

KEYWORDS: Upper-Division Undergraduate, Inorganic Chemistry, Hands-on Learning/Manipulatives, Collaborative/Cooperative Learning, Problem Solving/Decision Making, Materials Science, Physical Properties, Undergraduate Research, Laboratory Instruction



INTRODUCTION

Crystalline and porous metal–organic frameworks (MOFs), materials composed of organic linkers coordinated to metal clusters or ions, have gained popularity due to their porosity and synthetic and chemical tunability. This modularity uniquely positions MOFs as ideal candidates for the removal of toxic compounds from water in three ways. First, differing metal nodes enables coordination of different organic and inorganic pollutants,^{1–5} while differing organic linkers enables π – π stacking, hydrogen bonding, or electrostatic interactions to adsorb pollutants.^{6–10} Second, some zirconium-based MOFs are chemically and mechanically stable such that they can be used under most aquatic and marine conditions as filtration materials.^{11–13} Finally, MOFs can remove trace levels (5 μ g/L) of contaminants, unlike traditional physical adsorbents like activated carbon.^{14,15}

Due to their facile synthesis, tunability, and diverse applications, undergraduate laboratory experiments have recently focused on incorporating MOFs as a teaching tool. As hybrid inorganic–organic solid crystals, MOFs are

particularly well-positioned to connect fundamental general, organic, physical, and inorganic chemistry to advanced topics of solid-state materials chemistry. Sumida et al. captured these learning topics in an undergraduate inorganic laboratory experiment focusing on the synthesis, characterization, and postsynthetic modification of MOF-5, IRMOF-3, and HKUST-1.¹⁶ Concepts such as host–guest chemistry were highlighted in undergraduate laboratory experiments using PCN-200 for CO₂ capture¹⁷ and α -Mg₃(O₂CH)₆ for the uptake of small organic molecules.¹⁸ Undergraduate laboratory activities exploiting host–guest chemistry between MOFs and water contaminants have also been developed. Cheng et al. introduced first-year undergraduates to ZIF-8 for removal of

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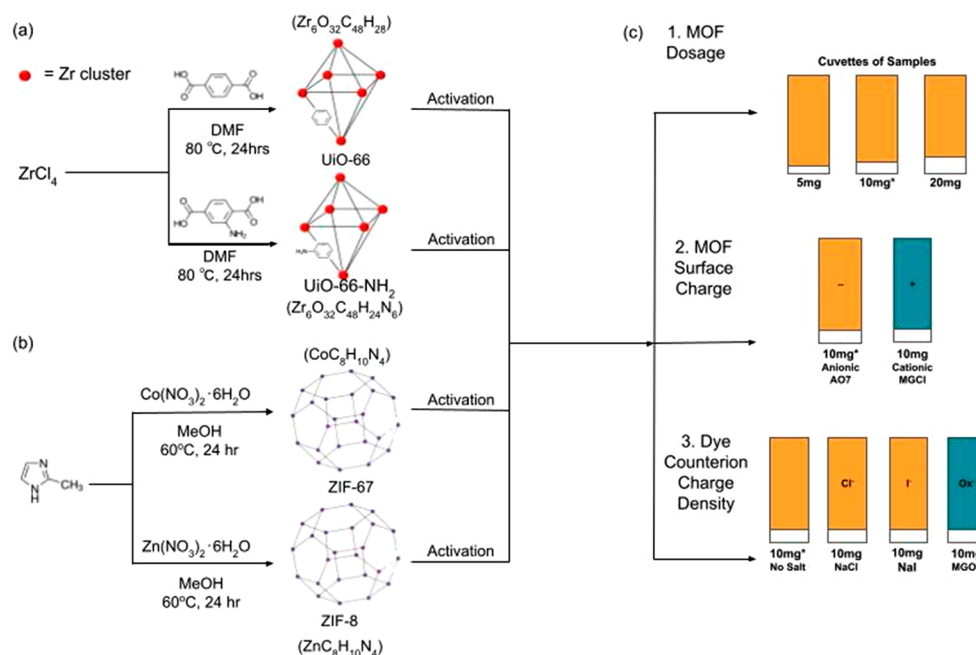


Figure 1. Syntheses of (a) UiO-66 and UiO-66-NH₂ and (b) ZIF-8 and ZIF-67 and (c) measurement conditions.

methyl blue dye in aqueous solution.¹⁹ Similarly, Sakamaki et al. trained undergraduate students with at least one year of organic chemistry to synthesize and characterize MOF-5, ZIF-8, and MIL-101 (Fe) for adsorption of I₂ in aqueous solution.²⁰ These laboratory activities successfully teach and train students in the theory, concept, and application of nanomaterials in introductory undergraduate laboratory settings. The laboratory activities integrate many subdisciplines of chemistry, bridging the traditional molecular perspectives of chemistry with that of the solid-state materials. Students can then connect how the solid-state materials chemistry learned in this lab enables improvements in sustainability technologies and applications, such as solar energy conversion,^{21–24} water decontamination,²⁵ and sensing.²⁶

However, this work differs from previous host–guest chemistry experiments in three important ways. First, previous works involving MOFs for water decontamination focused on investigating adsorption capacity of one contaminant in MOFs without changing any conditions.^{19,20} On the contrary, this work investigates the effects of MOF dosage, MOF surface charge, and dye counterion charge density on the adsorption capacity of dyes. Second, while previous MOF laboratory activities focused on studying one MOF¹⁹ or three MOFs all from three different topologies,²⁰ this activity focuses on structure–property relationships—a cornerstone of materials chemistry. This is done by using four MOFs, specifically two from the sodalite topology (ZIF-8 and ZIF-67) and two from the body-centered cubic topology (UiO-66 and UiO-66-NH₂). By using two MOFs from each topology, students can easily observe how one structural change in the (a) metal ions (Zn²⁺ in ZIF-8 vs Co²⁺ in ZIF-67) with the same linker or (b) organic linker (2-aminoterephthalic acid in UiO-66-NH₂ and terephthalic acid in UiO-66) with the same metal ion cluster may affect the overall property. Third, rather than focus on the effects of synthetic changes on adsorption capacity of MOFs in solution, students examined these MOFs for removal of dye compounds under three different measurement conditions: effects of MOF dosage, MOF surface charge, and dye

counterion charge density in solution. In this laboratory activity, major-level undergraduate students conduct the synthesis, characterization, and evaluation of MOFs for removal of toxic dye molecules from aqueous solutions.

EXPERIMENTAL OVERVIEW

The students tested this experiment for a total of three semesters in Integrated Chemistry Laboratory I, an upper-division course at CSU Chico. This laboratory experience enables 16 students in groups of 4 to apply their prior knowledge and skills from general and inorganic chemistry classes within the ten lab periods of 3 h each; see Table S1. Each student group spent two 3 h lab periods performing the synthesis and workup of four MOFs: UiO-66, UiO-66-NH₂ (Figure 1a), ZIF-8, and ZIF-67 (Figure 1b), followed by activation (see the SI for the procedure). Each student per group is assigned one of the four MOFs. Briefly, to synthesize UiO-66 (or UiO-66-NH₂), zirconium(IV) tetrachloride was sonicated in 5 mL of DMF and 1 mL of 6 M HCl for 20 min before the addition of terephthalic acid (or 2-aminoterephthalic acid) and 10 mL more of DMF. The metal to organic linker molar ratio was 2:3. Upon heating for 24 h at 80 °C, the resulting solid was removed via centrifugation, solvent exchanged in DMF and EtOH, and activated at 80 °C for 24 h. To synthesize ZIF-8 (or ZIF-67), zinc(II) nitrate hexahydrate [or cobalt(II) nitrate hexahydrate] and 2-methylimidazole were stirred in 40 mL of MeOH for 24 h. The metal to organic linker molar ratio was 1:4. After centrifuging, the solvent was decanted, and the MOF was dried and activated for 24 h at 60 °C; see the SI for more details.

For the next four lab periods, the students focused on characterization using powder X-ray diffraction (PXRD), Fourier-transform infrared spectroscopy (FT-IR), and energy-dispersive X-ray spectroscopy (EDXS). Each group only needs to collect one PXRD, FTIR, or EDXS scan per MOF sample. After synthesis, each group studied one variable, such as effects of dosage, surface charge, and counterion charge density (Figure 1c). They monitored dye removal using MOFs

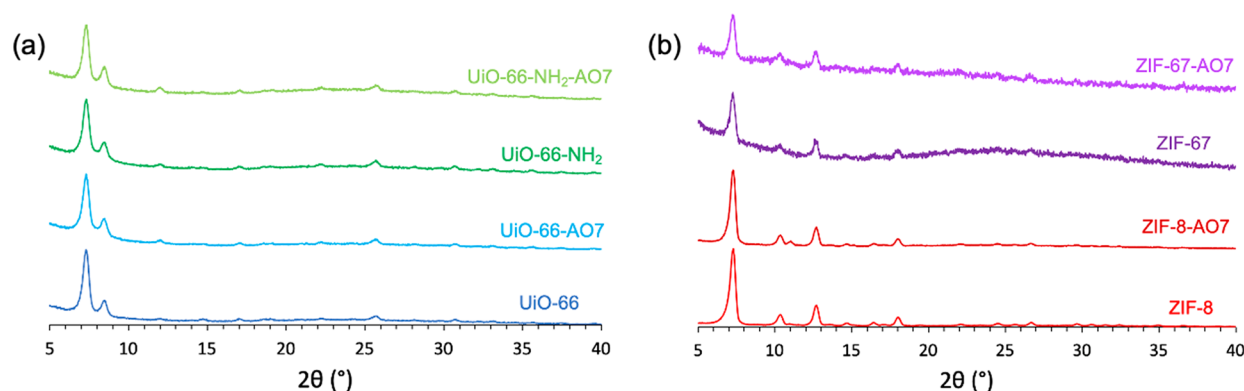


Figure 2. PXRD patterns of (a) UiO-66 and UiO-66-NH₂ and (b) ZIF-8 and ZIF-67 before and after adsorption of AO7.

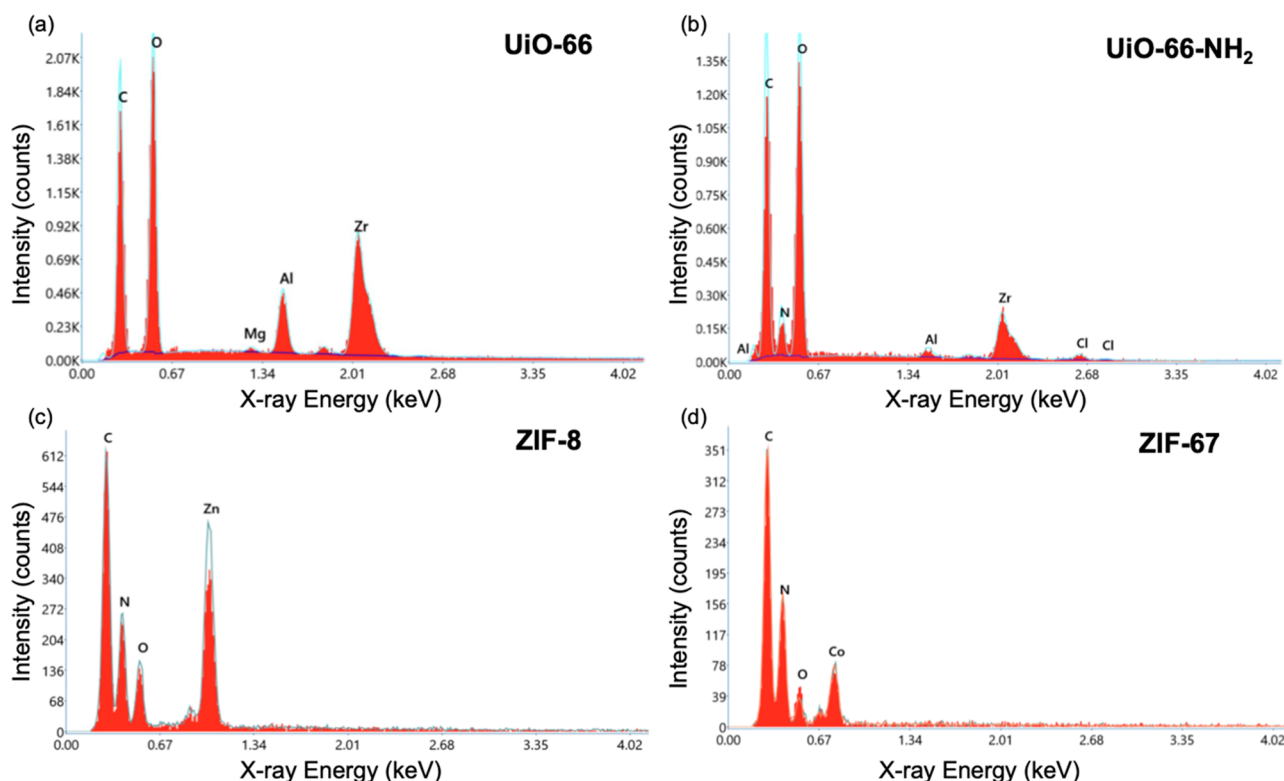


Figure 3. EDXS spectrum of (a) UiO-66, confirming the presence of oxygen and zirconium in the MOF; (b) UiO-66-NH₂, confirming the presence of oxygen, nitrogen, and zirconium in the MOF; (c) ZIF-8, confirming the presence of nitrogen and zinc in the MOF; and (d) ZIF-67, confirming the presence of nitrogen and cobalt in the MOF. Aluminum originates from the SEM stubs used to mount the MOFs.

via UV–vis spectroscopy. After 5–20 mg of one MOF was added to the diluted dye solution in a plastic cuvette, spectra were then collected at 0, 0.25, 0.5, 0.75, 1, and 50 h. For each spectrum collected, the absorbances at 483 and 617 nm, which are the λ_{max} of acid orange (AO7) and malachite green (MG), respectively, were used to determine the residual dye in solution and then used to determine the amount of dye adsorbed by the MOF. The initial concentration (C_0 , mg/L) and the concentration at each UV–vis measurement (C_t , mg/L) were used to determine the adsorption capacity (q_T) in mg/g, using eq 1:

$$q_T = \frac{V(C_0 - C_t)}{m} \quad (1)$$

where V is a sample volume of 2.5 mL, and m is the mass of MOF in grams. Plotting the absorbance data at the λ_{max} of each

dye for each time point yielded adsorption capacity plots (see the [Results and Discussion](#) section).

SAFETY HAZARDS

Students must wear all proper personal protective equipment at all times while performing these experiments, including lab coats, safety goggles, and gloves. Zirconium(IV) chloride, terephthalic acid, 2-aminoterephthalic acid, cobalt(II) nitrate tetrahydrate, 2-methylimidazole, AO7, and MGOx can cause eye damage and burns. Treatment for these injuries includes flushing eyes with water for at least 15 min, making sure to rinse thoroughly and to seek immediate medical aid. For skin contact, also flush skin with water for at least 15 min while removing any contaminated clothing, and seek medical aid immediately. It is recommended that all instructors and

students go through the MSDS of each reagent for more information.

LEARNING OBJECTIVES AND CONCEPTS

Students increase their knowledge of MOFs through lecture topics and hands-on experiences. Before each topic, an introductory lecture is given to provide the basic concepts of each characterization technique to students. This instruction coupled with activities will allow students to learn how to analyze MOFs via PXRD, EDXS, and FT-IR and UV-vis spectroscopies. Using PXRD, students are able to differentiate between amorphous and crystalline materials, as well as how to calculate lattice spacings. While EDXS verifies the atomic composition of each MOF, FT-IR spectroscopy confirms the presence of functional groups from MOFs or adsorbents via characteristic stretching and bending frequencies. Finally, UV-vis spectroscopy tracks the removal of dye by the MOF and quantifies the adsorption capacity of the MOFs.

RESULTS AND DISCUSSION

Crystallinity

PXRD data provides information about the identity, structure, and crystallinity of metal-organic frameworks. This diffraction technique allows students to learn how interactions between X-rays and crystalline samples produce informative peaks. Students confirmed that the MOFs retained crystallinity before and after adsorption (Figure 2) which is consistent with their morphology (Figure S2).

Elemental Composition

The EDXS data also confirms the elemental composition of the MOFs. The EDXS spectrum of UiO-66 (Figure 3a) confirms the presence of zirconium, while the presence of oxygen is from terephthalic acid. Similarly, the EDXS spectrum of UiO-66-NH₂ (Figure 3b) confirms the presence of zirconium and oxygen, where nitrogen is also present due to the amine group in 2-aminoterephthalic acid. For both UiO-66 and UiO-66-NH₂, aluminum is due to the SEM stub used to hold the sample while the magnesium and chlorine are salts present on the SEM stub. The EDXS spectrum of ZIF-8 (Figure 3c) confirms the expected zinc ion, while the presence of nitrogen is from the 2-methylimidazole. Similarly, the EDXS spectrum of ZIF-67 (Figure 3d) confirms the presence of the cobalt ion, while nitrogen and carbon are from the 2-methylimidazole. Some oxygen is also present for both ZIF-8 and ZIF-67 from methanol, which is the reaction solvent.

Coordination between MOFs and Dye

FT-IR spectroscopy gives insight on whether dyes are adsorbed to the MOFs by comparing the vibrational modes of functional groups of the MOFs before and after dye adsorption. Before dye adsorption, students only observed vibrational modes of the MOF, such as the C-H stretch at around 2800 cm⁻¹ from the methyl group of the 2-methylimidazole organic linker of ZIF-67 (Figure 4, ZIF-67). After dye adsorption, students saw the appearance of the hydroxyl group stretch at 2900–3300 cm⁻¹ which only appears in the AO7 dye, not the MOF (Figure 4, ZIF-67 and AO7). This confirms the AO7 coordinated to the surface of the MOF. The dye is adsorbed to the MOF likely due to both the aromatic groups of AO7 and the imidazole ring of ZIF-67 participating in π - π stacking interactions. The OH stretch of the dye remains in the presence of 3% w/v sodium chloride and sodium iodide

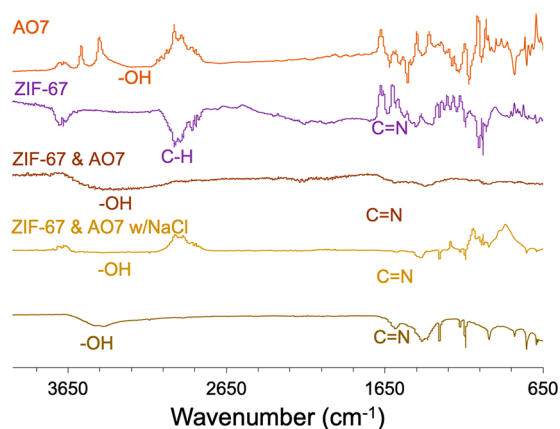


Figure 4. FT-IR spectra of ZIF-67 before and after dye adsorption in the presence and absence of 3% w/v salt solution.

solutions. This suggests that small ions have minimal effect on the dye adsorption to MOF; this has important implications for MOFs serving as adsorbents in ocean water.

Adsorption Capacity

Adsorption capacity (q_T) quantifies the amount of dye (in mg) adsorbed to the surface of the MOF per amount (in g) of MOF used in solution. The higher the q_T values, the better the adsorbent. This can be used to compare the dye removal capacity of different MOFs under various conditions (e.g., dosage, surface charge, and counterion charge density) and to determine which set of conditions improves q_T the most.

Effects of Dosage. Students evaluated the amount of MOF used in each adsorption experiment (dosage) to identify the optimal amount needed to decontaminate water. Since the dyes used have dimensions of approximately 5.4 Å × 10.0 × 15.7 Å (AO7), which is much larger than the apertures of each MOF—UiO-66 (6.0 Å), UiO-66-NH₂ (5.6 Å), ZIF-8 (3.4 Å), and ZIF-67 (3.4 Å), the dyes likely adsorb to surface sites of the MOF.¹³ Hence, as the dosage of MOFs increases, the amount of surface adsorption sites due to self-aggregation of MOF crystals decreases; thus, q_T decreases. As expected from eq 1, students found that a 5 mg dosage had the highest q_T for all MOFs tested, resulting in maximum q_T values of 30 mg of AO7/g for ZIF-67 (Figure 5a). For the dosage studies, ZIF-8 exhibited the lowest q_T , but its q_T value at the 5 mg dosage was still higher than those at the 10 mg (Figure 5b) and 20 mg dosages (Figure 5c).

Effects of Surface Charge. MOFs have an overall surface charge that attracts dye molecules of dissimilar charge or repels those with like charge at specific pH ranges. The four MOFs exhibited overall positive surface charge at pH 6.8, suggesting that the MOFs would attract anionic dyes better than cationic dyes. Indeed, students observed that the q_T for the anionic AO7 (Figure 6a) was 4 times higher for UiO-66 and UiO-66-NH₂ than for cationic MGCl (Figure 6b). ZIF-67 exhibited similar adsorptions for both dyes but still favored AO7 slightly more over MGCl. The only exception was ZIF-8, which displayed a slightly higher adsorption for MGCl over AO7. Since ZIF-67 is more electropositive due to the higher oxidation state accessible to the cobalt cation, it has higher q_T for anionic dye, AO7. Since ZIF-8 is more electronegative, it has a higher q_T for a cationic dye, MGCl, than for AO7.

Effects of Dye Counterion Charge Density. Each charged dye is coordinated to a counterion. Two cationic

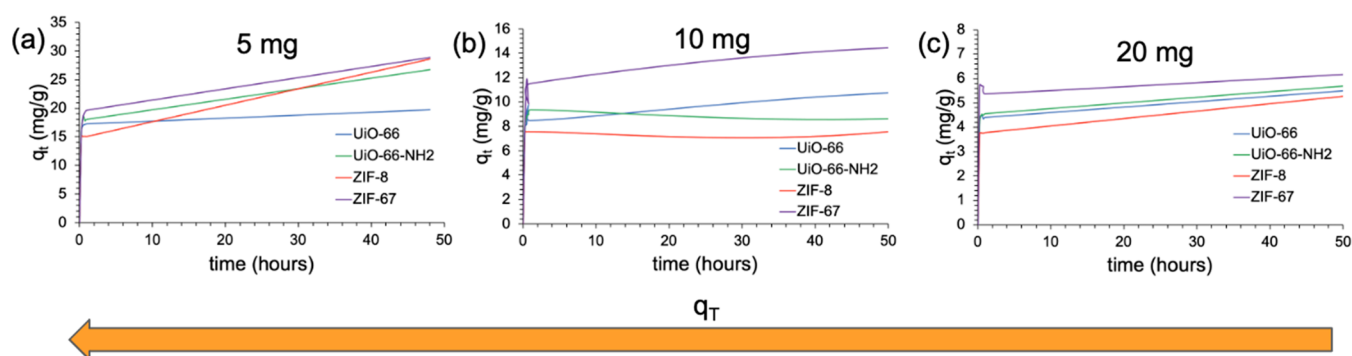


Figure 5. Adsorption capacity of AO7 at (a) 5 mg, (b) 10 mg, and (c) 20 mg of MOF at pH 6.8.

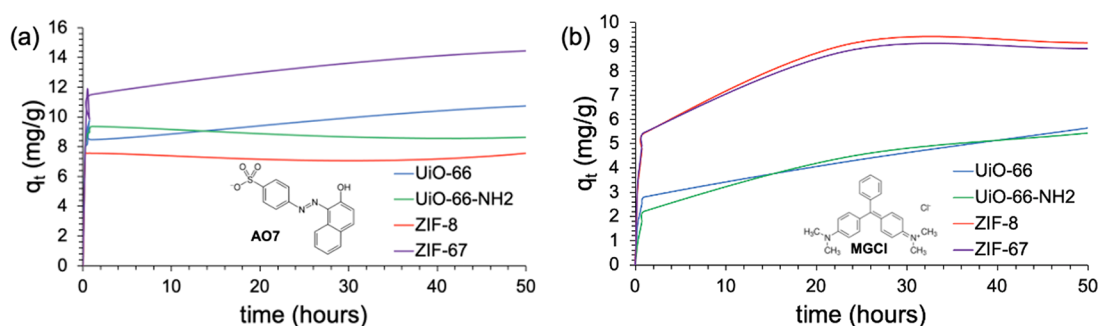


Figure 6. Adsorption capacities at pH 6.8 for (a) negatively charged AO7 and (b) positively charged MGCl for four MOFs.

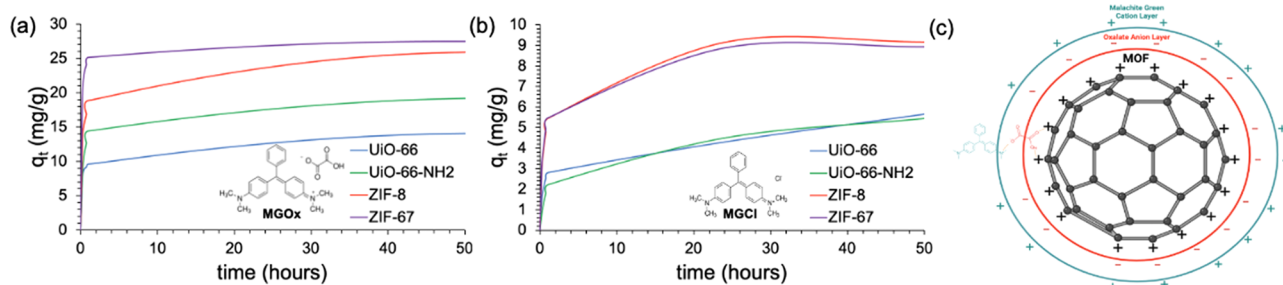


Figure 7. Adsorption capacities at pH 6.8 for cationic dyes with charge densities that are (a) lower (oxalate) and (b) higher (chloride) for four MOFs. (c) Proposed adsorption mechanism for MGOx on an MOF surface.

dyes that the students selected, MGOx and MGCl, have counterions of different charge density (oxalate and chloride, respectively). Though students predicted that the charge density of counterions had negligible impacts on dye adsorption, adsorption capacity plots in Figure 7 suggested otherwise. The presence of oxalate in MGOx (Figure 7a) yielded q_T values at least 2 times higher than the presence of chloride in MGCl (Figure 7b), despite the fact that both dyes are the same and only differ by the counterion. This is likely due to the formation of a negatively charged MOF surface through adsorption of the oxalate counterion to the positively charged MOF, followed by the adsorption of the positively charged MG dye (Figure 7c). While chloride has a higher charge density, resulting in a stronger coordination to the MOF, oxalate has a weaker coordination to the MOF due to the delocalization of the negative charge. The stronger the counterion coordination is to the MOF, the less residual negative charge is present on the counterion. Therefore, stronger coordination of the counterion leads to an overall more positive MOF surface charge and lower subsequent adsorption of cationic MG. This higher charge density

resulting in lower q_T is consistent with the presence of chloride and iodide in our control experiments (Figure S3).²⁷

STUDENT LEARNING OUTCOMES

To prepare students for understanding the materials chemistry involved in MOFs, the instructor discussed topics of general, physical, organic, inorganic, and materials chemistry in the following contexts:

- Global significance of water decontamination technology
- Chemistry and structure of MOFs
- Spectroscopic characterization
- Structural and morphological characterization
- Decontamination experiment studies
- Data analysis and interpretation

At the end of the course, students were evaluated through individually written final laboratory reports, designed to probe breadth and depth of understanding, separately, in the aforementioned six context areas. To probe breadth, students were required to correctly answer 80% or more of the list of Results and Discussion questions on page S8 of the Student

Handout in their final report. To probe depth, students were required to present their work as a Powerpoint presentation and to correctly answer all questions posed by the instructor and peers during their oral presentations. A score of 80% or more was considered effective in meeting learning goals. They also had to take pre- and postlaboratory surveys to self-rate confidence levels in executing both laboratory techniques (e.g., MOF synthesis, solvent exchange, and activation) and laboratory tasks (e.g., using online search tools and writing reports in journal format). After performing experiments, 95% of the students over two semesters rated themselves as very confident in both laboratory techniques and tasks.

CONCLUSIONS AND FUTURE WORK

The synthesis and evaluation of MOFs for the removal of dyes from water serve as a highly valuable introduction to integrated chemistry for chemistry and biochemistry majors. This lab serves as the first hybrid lab chemistry that students are exposed to and has great merit in showing how general, physical, organic, inorganic, and materials chemistry can be applied within a single experiment. Students were given the opportunity to synthesize four unique MOFs from different classes, as well as to employ advanced techniques involved in the characterization of these crystalline materials. Additionally, students experienced the real-world applications of these materials for water decontamination. By the end of the lab, students had developed their critical thinking skills and improved their overall analytical abilities through both their presentations and final reports on the topic. Due to the modularity of this lab, future courses can tailor the experiments detailed here to fit into a variety of laboratories for major-level students. These could range from inorganic/organic chemistry to chemical engineering, where various properties of the MOFs could be investigated such as their environmental impact or reusability following decontamination.

ASSOCIATED CONTENT

Supporting Information

The Supporting Information is available at <https://pubs.acs.org/doi/10.1021/acs.jchemed.2c00115>.

Notes for instructors (PDF, DOCX)

Lab handout for students (PDF, DOCX)

Adsorption capacity workup template (XLSX)

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<https://pubs.acs.org/10.1021/acs.jchemed.2c00115>

Notes

The authors declare no competing financial interest.

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