Contract No:

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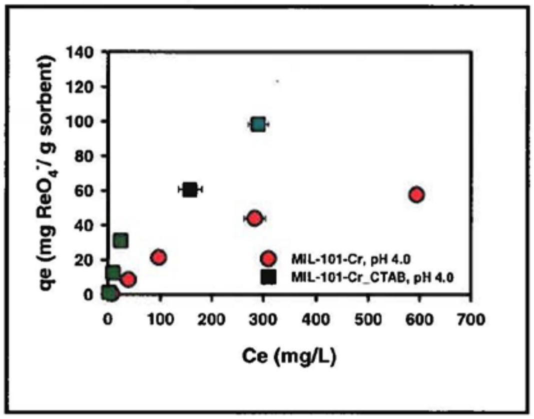
Metal-Organic-Framework Glasses as Rad Contaminant Sequesters and Nuclear Waste Forms

Project Highlight: *Remediation of Tc remains an unresolved problem at SRS and other DOE sites. The objective of this project was to develop novel metal organic framework (MOF) glasses for radioactive contaminant sequestration and stabilization from aqueous media. During FY19, we synthesized, characterized and evaluated cetyl trimethylammonium bromide (CTAB)-functionalized and F$^-$, Cl$^-$, I$^-$, $CF_3SO_3^-$-exchanged MIL-101. MIL-101-CTAB was demonstrated to have high ReO_4^- removal capacity (~130 mg/g sorbent) from artificial groundwater. Re chemical speciation and binding mechanism in MIL-101-CTAB were also studied by synchrotron X-ray absorption spectroscopy. ReO_4^- was demonstrated as being in the pore structure with slightly larger Re-O bond distances than those in $NaReO_4$. In addition, a new Ni-TIPA MOF was demonstrated to be very stable, selective and effective for TcO_4^- removal from the SRS tank waste stream (~90% removal). This research may provide a highly applicable platform for solving critical DOE and industrial problems related to nuclear environmental stewardship and nuclear power production.*



Awards and Recognition

N/A

Intellectual Property Review

This report has been reviewed by SRNL Legal Counsel for intellectual property consideration

SRNL Legal Signature

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Signature **Date**

Metal-Organic-Framework Glasses as Rad Contaminant Sequesters and Nuclear Waste Forms

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Subcontractor: University of South Carolina

Thrust Area: Environmental Stewardship

Project Start Date: October 1, 2017
Project End Date: September 30, 2020

Abstract: *Remediation of Tc remains an unresolved problem at SRS and other DOE sites. The objective of this project was to develop novel metal organic framework (MOF) glasses for radioactive contaminant sequestration and stabilization from aqueous media. During FY19, we synthesized, characterized and evaluated cetyl trimethylammonium bromide (CTAB)-functionalized and* F^-*,* Cl^-*,* I^-*,* $CF_3SO_3^-$*-exchanged MIL-101. MIL-101-CTAB was demonstrated to have high* ReO_4^- *removal capacity (~130 mg/g sorbent) from artificial groundwater (AGW). Re chemical speciation and binding mechanism on MIL-101-CTAB were also studied by synchrotron X-ray absorption spectroscopy.* ReO_4^- *was demonstrated as being in the pore structure with slightly larger Re-O bond distances than those in* $NaReO_4$*. In addition, a new Ni-TIPA MOF was demonstrated to be very stable, selective and effective for* TcO_4^- *removal from the SRS tank waste stream (~90% removal). This research may provide a highly applicable platform for solving critical DOE and industrial problems related to nuclear environmental stewardship and nuclear power production.*

FY2019 Objectives

(1) Synthesize and characterize CTAB-functionalized, and F^-, Cl^-, I^-, $CF_3SO_3^-$-exchanged MIL-101.
(2) Evaluate these MOFs for TcO_4^- surrogate (i.e., ReO_4^-) removal from AGW.
(3) Investigate a new Ni-TIPA MOF for Tc removal from the SRS tank waste stream
(4) Develop methods for vitrifying the crystalline MOFs.

Introduction

^{99}Tc is a major long-lived fission product created during nuclear power generation. Over the years, Tc has been inadvertently introduced into the environment from leaks at waste storage facilities. ^{99}Tc currently is one of the key risk drivers at the Savannah River Site (SRS) and other DOE environmental management sites (most notably the Hanford Site, Paducah Gaseous Diffusion Plant, and Oak Ridge National Laboratory). The most common chemical form of Tc in liquid nuclear wastes and in the environment is anionic pertechnetate (TcO_4^-). TcO_4^- displays limited adsorption onto common sediment minerals and is highly mobile making it difficult to capture or to be immobilized [1]. As the stockpile of ^{99}Tc-bearing nuclear waste continues to increase rapidly, novel sequestration technologies are needed to reduce its potential contamination of the environmental and living organisms.

With current technologies, quaternary amine-based resins have been used to remove aqueous Tc [2]. However, these resins are expensive and have only modest TcO_4^- loading capacities from the raffinate waste streams. Chemical reductants (e.g., Fe_3S_4, soluble or structural Fe(II)) [3] and some bacteria [4] can reduce Tc(VII) to the sparingly soluble Tc(IV). However, the resulting $Tc(IV)O_2{\cdot}1.6H_2O$ has a solubility of 1.5×10^{-8} M in groundwater [5], which greatly exceeds the EPA's maximum contaminant level of 5×10^{-10} M, and is readily re-oxidized and re-mobilized under most environmental conditions [6]. Tc reduction to form sulfides (e.g., Tc_2S_7) [7] or embedding into other sulfide phases [8] or iron oxide waste forms [9] have also been investigated. However, these methods are not practical for many applications. There are currently no demonstrated technologies that are highly efficient and cost-effective for separation of Tc-containing nuclear waste streams and remediation of aqueous Tc in the contaminated sites.

The overarching objective of this project was to develop novel metal organic framework (MOF) glasses as radioactive contaminant sequesters and nuclear waste forms. During FY18, MIL-101-Cr was identified as being most effective for Tc removal from AGW. During FY19, we continued to develop, characterize and evaluate MIL-101-Cr based MOFs for TcO_4^- and ReO_4^- sequestration from AGW and highly acidic solutions, while we were also developing new MOF materials of higher alkaline stability, selectivity, and capacity, as well as vitrification technology.

Approach

During FY19, we prepared CTAB-functionalized, and F^-, Cl^-, I^-, $CF_3SO_3^-$-exchanged MIL-101-Cr, which were expected to have higher Tc capacity and improved stability. Powder X-ray diffraction (XRD), BET surface area measurement, thermogravimetric analysis (TGA), energy dispersive X-ray spectroscopy (EDS), and Fourier transform infrared (FTIR) spectroscopy were used for MOF characterization, while synchrotron radiation X-ray absorption spectroscopy was applied to studying Tc or Re chemical speciation and molecular binding mechanisms after the sequestration by MOFs. In addition, we investigated a new Ni-TIPA MOF for Tc removal from the SRS tank waste stream, while we also tried to develop methods for vitrifying the crystalline MOFs for stabilization of Tc and other contaminants in the MIL-101-Cr and Ni-TIPA MOF glass waste forms.

Results/Discussion

1. Synthesis of modified MIL-101 MOFs

1.1. MIL-101-Cr-NO_3. Chromium(III) nitrate nonahydrate (0.800 g, 2.00×10^{-3} mol) and terepthalic acid (0.328 g, 1.97×10^{-3} mol) were placed in a Teflon-lined autoclave followed by the addition of 4.6 mL deionized water and 0.4 mL of HNO_3 (5.0 M, 2.0 mmol). The autoclave was then sealed and placed in an oven at 210 °C for 8 hours. Upon cooling, the resulting green solid was centrifuged and washed three times with DMF, water and ethanol for 12 h at 80 °C, respectively.[10]

1.2. MIL-101-Cr-NO_3-CTAB. Under a nitrogen atmosphere, (Cr)MIL-101 (0.200 g), CTAB (0.300 g), and chloroform (10 mL) were added to a 75 mL heavy-wall pressure flask. The flask was sealed and heated at 110 °C while stirring. The resulting green solid was centrifuged and washed three times with chloroform (20 mL) and then dried under vacuum for 3 h.

1.3. MIL-101-Cr-F. Chromium(III) nitrate nonahydrate (0.400 g, 1.00×10^{-3} mol) was dissolved in 4.8 mL of deionized water, then placed in a Teflon-lined autoclave. Terephthalic acid (0.164 g, 9.87×10^{-4} mol) was then added followed by the addition of 0.2 mL of HF (5.00 M, 1.00×10^{-3} mol). The autoclave was then sealed and placed in an oven at 220 °C for 8 hours. Upon cooling, the resulting green solid was centrifuged to collect the precipitate and solvent exchanged with DMF (3×10 mL) with centrifugation between each exchange. The product was then washed three times with DMF, water and ethanol, respectively, and dried for 12 h at 80 °C.

1.4. MIL-101-Cr-Cl. An aqueous aluminum(III) chloride hexahydrate solution (15 mL, 30 mM) was added to a 20 mL vial containing 30 mg of (Cr)MIL-101-F. The vial was then placed in a preheated oven at 90 °C for 18 h. Upon cooling, the resulting green solid was filtered and washed with water (3×50 mL) and ethanol (3×50 mL).

1.5. MIL-101-Cr-I. An aqueous aluminum(III) iodide solution (15 mL, 30 mM) was added to a 20 mL vial containing 30 mg of (Cr)MIL-101-F. The vial was then placed in a preheated oven at 90 °C for 18 h. Upon cooling, the resulting green solid was filtered and washed with water (3 × 50 mL) and ethanol (3 × 50 mL).

1.6. MIL-101-Cr-OTf (OTf = trifluoromethanesulfonate)**.** An aqueous aluminum(III) triflate solution (15 mL, 30 mM) was added to a 20 mL vial containing 30 mg of (Cr)MIL-101-F. The vial was then placed in a preheated oven at 90 °C for 18 h. Upon cooling, the resulting green solid was filtered and washed with water (3 × 50 mL) and ethanol (3 × 50 mL).

2. Characterization of modified MIL-101 MOFs

The crystal structure of MIL-101-Cr-NO_3^- is shown in Figure 1. The CTAB-functionalized, and F^-, Cl^-, I^-, $CF_3SO_3^-$-exchanged MIL-101-Cr MOFs were characterized by powder XRD, TGA, EDS and FTIR to confirm the known crystal structure, exchanged anion incorporation, and CTAB functionalization. The powder XRD patterns of the new MIL-101 materials are shown in Figure 2, in comparison with that of original MIL-101-Cr-NO_3^-. The similar powder XRD patterns confirmed the correct identity of MIL-101-Cr. The TGA traces of these new MIL-101-Cr materials are shown in Figure 3, in comparison with that of MIL-101-Cr-NO_3^-. The thermal behavior of the anion-exchanged MIL-101-Cr were very similar to that of MIL-101-Cr-NO_3^-, while MIL-101-Cr-CTAB appears to show less mass loss below 200 °C, which indicated the less water or solvent present due to CTAB functionaliation. The EDS of the anion-exchanged MIL-101 are shown in Figure 4, which demonstrated that the anions (i.e., F^-, Cl^-, I^-, and $CF_3SO_3^-$) were exchanged into the MIL-101-Cr material. In addition, the FTIR spectra of CTAB-functionalized MIL-101-Cr-NO_3^- made from different batches are shown in Figure 5. The FTIR peaks characteristic of CTAB at 2918, 2850 and 1470 cm^{-1} (the shoulder in the spectra) demonstrated the success of CTAB functionalization onto MIL-101-Cr-NO_3^-.

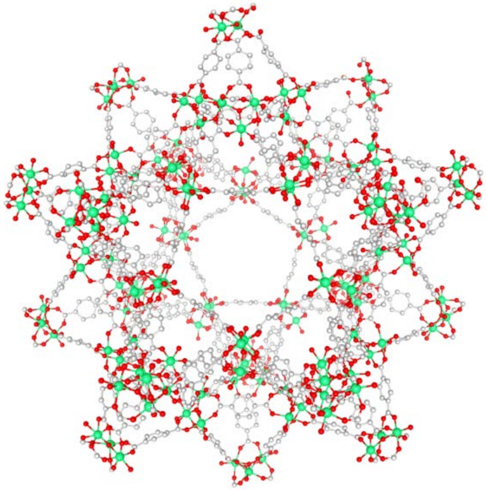

Figure 1. Crystal structure of MIL-101-Cr-NO_3 [10]

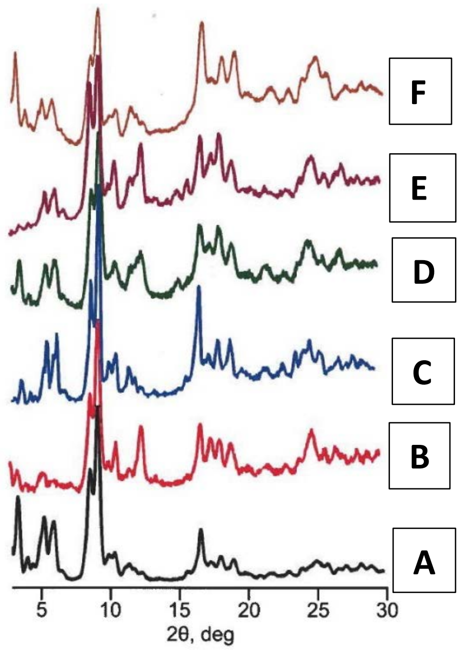

Figure 2. Powder XRD patterns of MIL-101-Cr-NO_3 (A), MIL-101-Cr-F (B), MIL-101-Cr-Cl (C), MIL-101-Cr-I (D), MIL-101-Cr-OTf (E), and MIL-101-Cr-CTAB (F). (OTF=trifluoromethanesulfonate, $CF_3SO_3^-$)

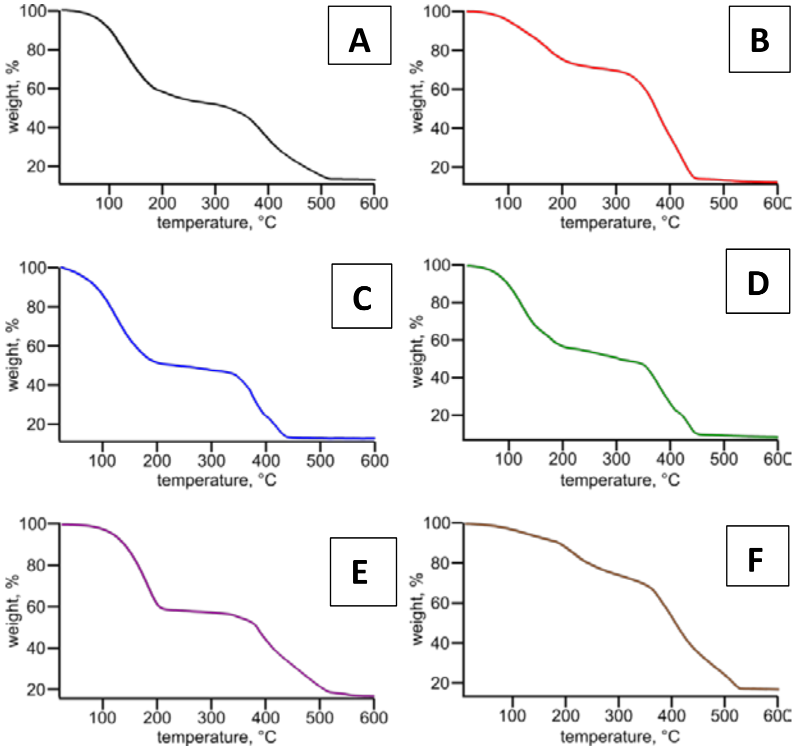

Figure 3. TGA traces of MIL-101-Cr-NO_3 (A), MIL-101-Cr-F (B), MIL-101-Cr-Cl (C), MIL-101-Cr-I (D), MIL-101-Cr-OTf (E), and MIL-101-Cr-CTAB (F).

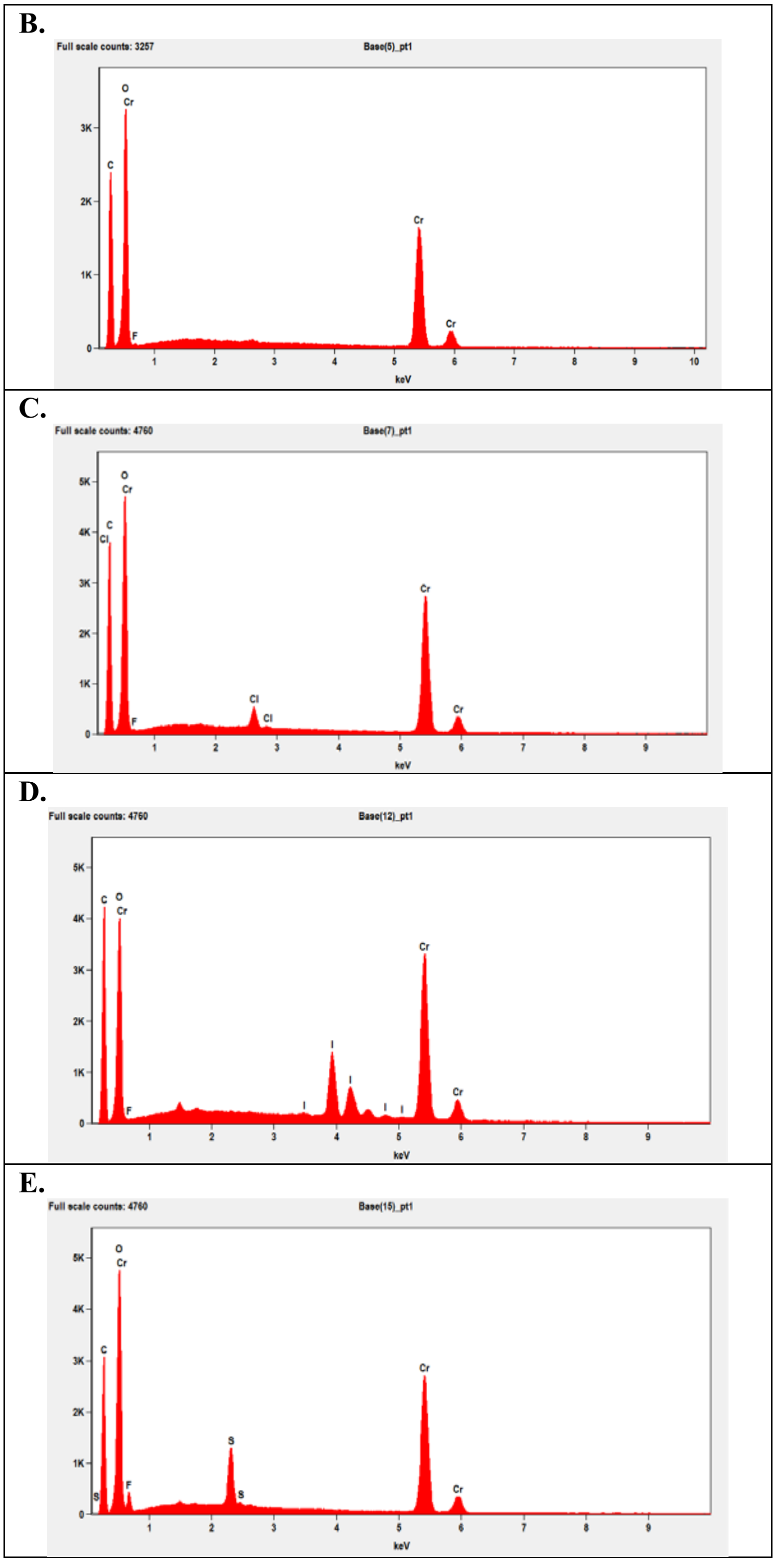

Figure 4. EDS of MIL-101-Cr-F (B), MIL-101-Cr-Cl (C), MIL-101-Cr-I (D), and MIL-101-Cr-OTf (E).

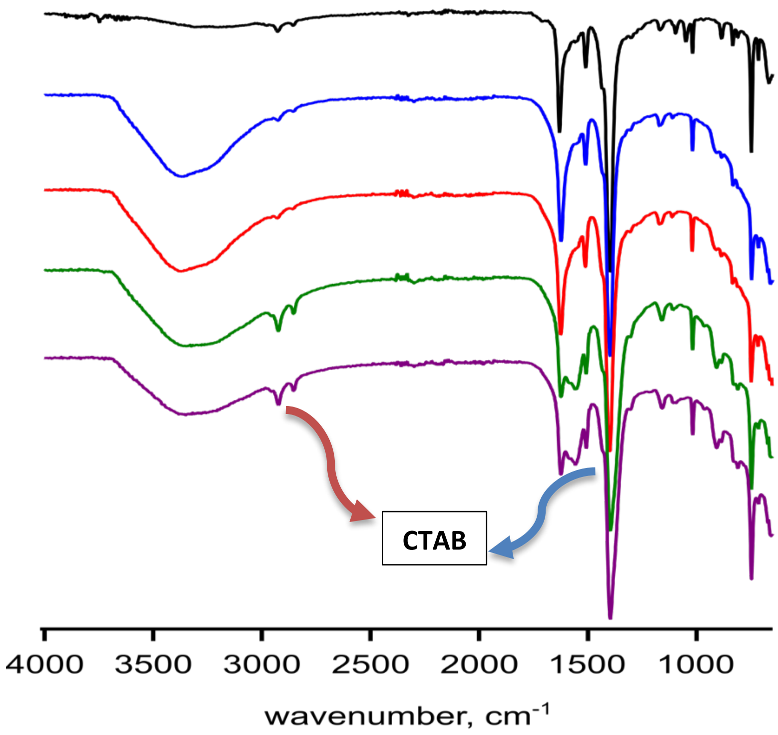

Figure 5. FTIR spectra of MIL-101-Cr-CTAB from different batches: as synthesized (black), MIL-101-Cr-CTAB-1 (blue), MIL-101-Cr-CTAB-2G (red), MIL-101-Cr-CTAB-3 (green), and MIL-101-Cr-CTAB-4G (purple).

3. New MIL-101-Cr for ReO_4^- sequestration from AGW

The CTAB-functionalized and F^-, Cl^-, I^- and $CF_3SO_3^-$ exchanged MIL-101-Cr MOFs were evaluated for non-radioactive surrogate of TcO_4^- (i.e., ReO_4^-) sequestration from AGW under atmospheric ($P_{CO2} = 10^{-3.5}$ atm) conditions. The equilibrium sorption capacity (q_e, mg/g) were calculated using formula 1:

$$q_e = \frac{(C_0 - C_e) \times V}{M} \qquad (1)$$

where C_0 and C_e were Re concentrations before and after adsorption, respectively, V was the total volume of liquid phase (i.e., AGW), and M was the mass of the solid material.

The experimental results are shown in Figure 6. As noted, MIL-101-Cr was identified as the most effective for ReO_4^- removal from AGW among eleven MOFs of known crystal structure and chemistry tested during FY18, with a saturation capacity of ~55 mg ReO_4^-/g sorbent (Figure 6A). With CTAB functionalization to MIL-101-Cr, the Re saturation capacity further increased to ~130 mg ReO_4^-/g sorbent (Figure 6A). The Re adsorption reaction toward these two MOFs was fast and completed within 30 minutes for MIL-101-Cr and 10 minutes for MIL-101-Cr-CTAB (Figure 6B). With the presence of competing anions (i.e., NO_3^-, CO_3^{2-}, SO_4^{2-} and Cl^-) in de-ionized water, when the competing anion concentration was 100 times as high as that for ReO_4^-, NO_3^-, CO_3^{2-} and Cl^- decreased the Re removal capacity of MIL-101-Cr by 20-30%, while SO_4^{2-} decreased the Re removal capacity of MIL-101-Cr by ~85% (Figure 6C). In contrast, with the presence of NO_3^-, CO_3^{2-} and Cl^- in de-ionized water, the Re removal capacity of MIL-101-Cr-CTAB remained little changed within the batch experimental uncertainty, while SO_4^{2-} decreased the Re removal capacity of MIL-101-Cr-CTAB by ~15% (Figure 6C). These results demonstrated that with CTAB functionalization, the selectivity of MIL-101-Cr for ReO_4^- or TcO_4^-, as expected, was significantly improved.

In addition, we also evaluated the F^-, Cl^-, I^- and $CF_3SO_3^-$ exchanged MIL-101-Cr MOFs for ReO_4^- removal from AGW. As shown in Figure 6D, the Cl^-, I^- and $CF_3SO_3^-$ exchanged MIL-101-Cr MOFs were more effective at ReO_4^- removal than MIL-101-Cr-NO_3^-, probably because the bonding of these anions with the framework is weaker than that for NO_3^-, so that ReO_4^- was more efficiently exchanged into the pore structure of these new MIL-101-Cr MOFs.

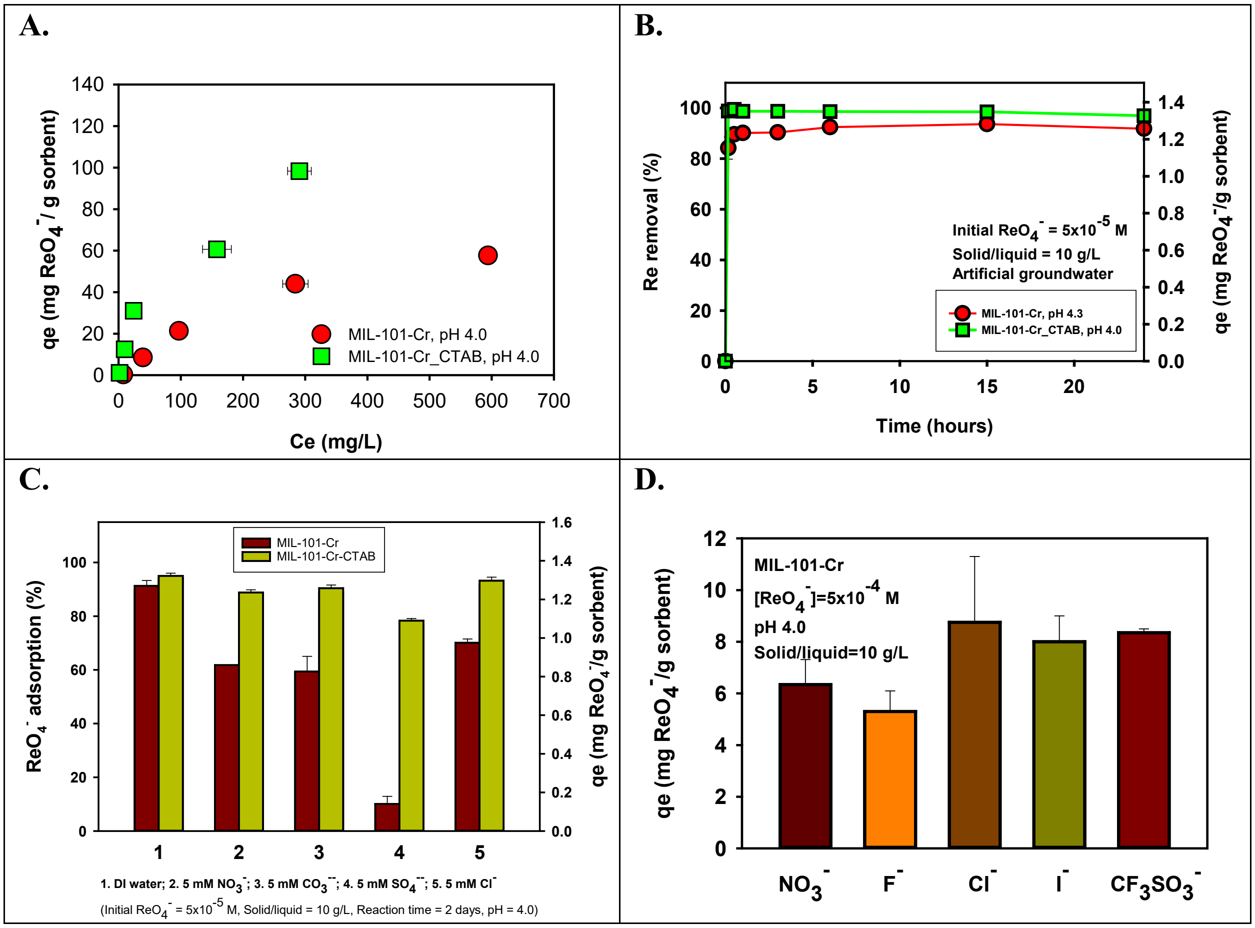

Figure 6. ReO_4^- removal from AGW by new MIL-101-Cr MOFs.

A. Isotherms and ReO_4^- removal capacity of MIL-101-Cr and MIL-101-Cr-CTAB.
B. ReO_4^- adsorption versus time for MIL-101-Cr and MIL-101-Cr-CTAB.
C. Effects of competing anions on ReO_4^- adsorption by MIL-101-Cr and MIL-101-Cr-CTAB. It is noted that this batch experiment was conducted using de-ionized water without or with single anion.
D. ReO_4^- removal capacity of anion-exchanged MIL-101-Cr.

In order to understand Re chemical speciation and molecular binding mechanisms in MIL-101-Cr and MIL-101-Cr-CTAB MOFs that were exposed to ReO_4^- in AGW, Re L_3-edge synchrotron X-ray absorption spectra of these MOF samples were collected using the Canadian Light Source BioXAS beamline. The Re L_3-edge X-ray absorption near-edge structure (XANES) spectra are shown in Figure 7A, while the Re L_3-edge extended X-ray absorption fine structure (EXAFS) spectra in k space and R magnitude are shown in Figure 7B and 7C, respectively. The spectra of two model compounds, ReO_2 and $NaReO_4$, were also included in Figure 7 for comparison. The results indicated that the Re chemical speciation in these MOFs was ReO_4^-, but the Re-O bond distances were slightly larger than those in $NaReO_4$. It is likely that ReO_4^- was located in the large pore structure of MIL-101-Cr, and its bonding environment can be further understood through detailed EXAFS data fitting which is currently under way.

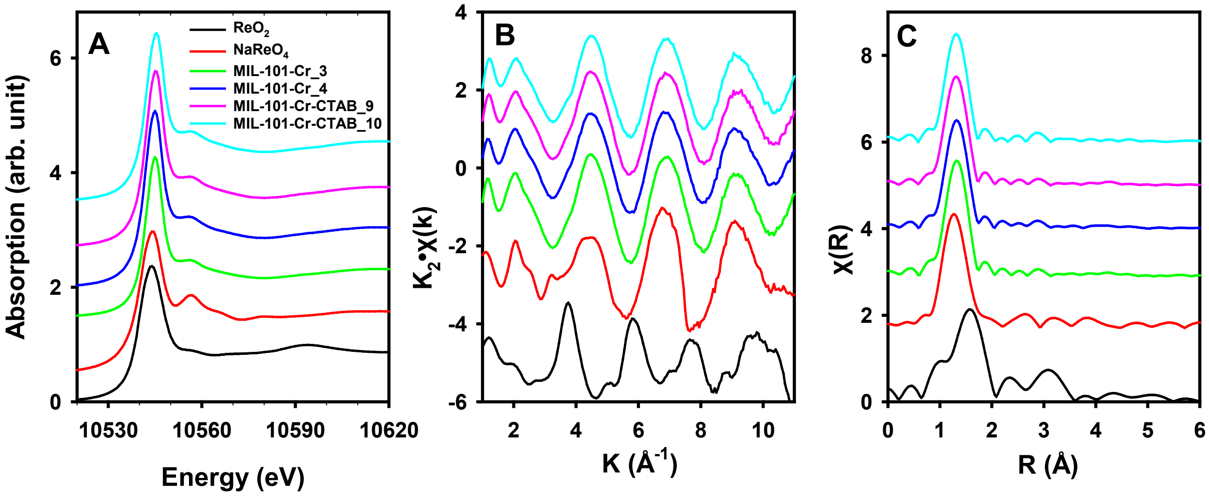

Figure 7. Re L_3-edge X-ray absorption spectra of MIL-101-Cr and MIL-101-Cr-CTAB exposed to ReO_4^- in AGW, in comparison with two model compounds, ReO_2 and $NaReO_4$.

A. Re L_3-edge XANES.
B. Re L_3-edge EXAFS in k space.
C. Re L_3-edge EXAFS in R magnitude.

4. Ni-TIPA MOF for TcO4- removal from the SRS tank waste

A Ni-TIPA MOF was acquired from Soochow University. The crystal structure of Ni-TIPA is shown in Figure 8. It has a large pore size of 10.43×16.11 Å. Ni-TIPA was demonstrated to have high stability in aqueous media across a broad pH range, up to a pH value of 14, as well as high capacity and selectivity toward ReO_4^- removal from aqueous media.

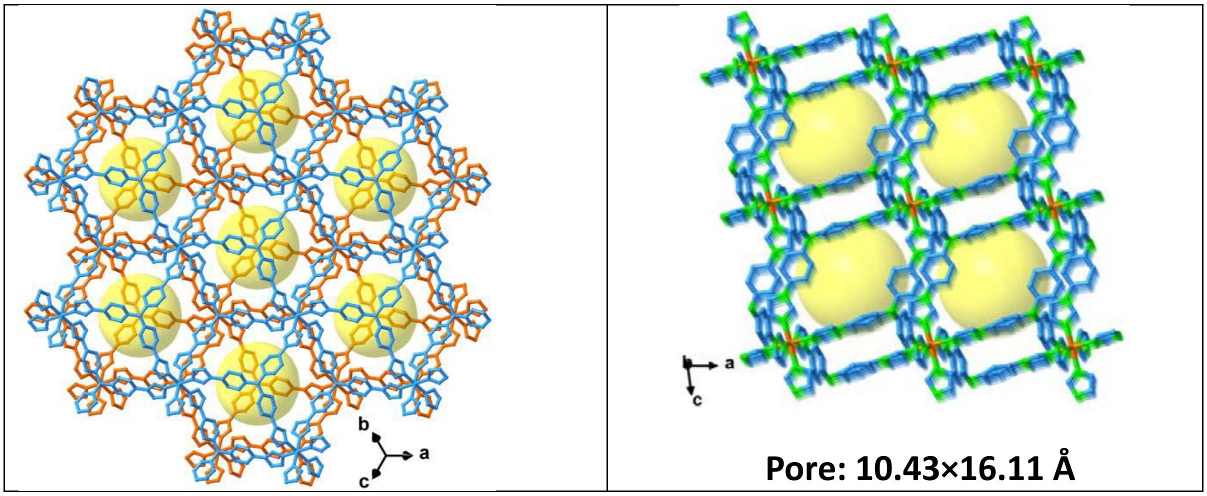

Figure 8. Crystal structure of Ni-TIPA MOF

A series of batch contact experiments to test this Ni-TIPA MOF were performed utilizing a sample of actual SRS tank waste.[11] Samples of Ni-TIPA MOF were added to 15-mL conical bottom polypropylene tubes in amounts ranging from 0.004 to 0.16 g. To each tube was then added 4 mL of the SRS tank waste solution to yield experiments with phase ratios ranging from 1 mg/mL to 40 mg/mL. A tube containing only the SRS tank waste solution (no Ni-TIPA) was also run in parallel as a control sample. The tubes were then mounted on a Thermo Scientific® Labquake® tube rotator and were tumbled for 3 hours. At the end of the 3-hour experiment, the tubes were removed from the rotator. The supernatant from each tube was then decanted and filtered through a 0.1-µm PVDF syringe filter. The filtrate was analyzed for ^{99}Tc activity by liquid scintillation counting (LSC).

The batch contact test results are shown Figure 9. The percent removal of ^{99}Tc increased as the phase ratio increased. At the highest phase ratio tested, 90% of the ^{99}Tc was removed from the SRS tank waste solution in 3 hours, in good agreement with experimental results performed using the simulated SRS tank waste (Shen et al., in preparation).

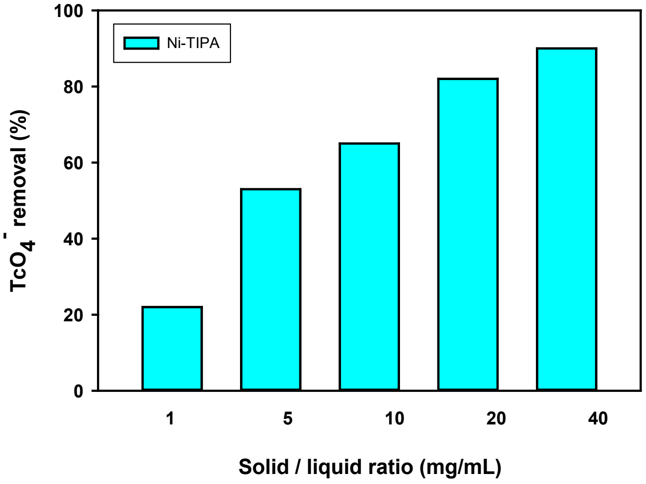

Figure 9. TcO_4^- removal percentage from the SRS tank waste stream by Ni-TIPA MOF.

5. Potential for economical vitrification

Thermogravimetric analyses of selected MOFs were conducted (Figure 3). The results indicated that these MOFs may be vitrified using economic methods, like heating under 300 °C. In addition, pressure-induced vitrification technology may be developed for these MOFs that are demonstrated to have high capacity for TcO_4^- removal and improved stability in aqueous media. The MOF glasses can be used as potential nuclear waste forms for Tc stabilization.

FY2019 Accomplishments

■ Synthesized and characterized five new versions of MIL-101-Cr MOFs (CTAB-functionalized and F^-, Cl^-, I^- and $CF_3SO_3^-$ exchanged).

■ Completed batch experiments to evaluate these MIL-101-Cr MOFs for ReO_4^- removal from AGW, including adsorption capacity, kinetics, effects of competing anions. The CTAB-functionalized MIL-101-Cr had the highest capacity of ~130 mg ReO_4^-/g sorbent. The adsorption reactions were fast and completed within 30 for MIL-101-Cr minutes and 10 minutes for MIL-101-Cr-CTAB.

■ Investigated Re chemical speciation and molecular interaction with MIL-101-Cr through synchrotron X-ray absorption spectroscopy. ReO_4^- was demonstrated as being in the pore structure with slightly larger Re-O bond distances than those in $NaReO_4$.

■ Studied a new Ni-TIPA MOF for Tc removal from the SRS tank waste stream. The Ni-TIPA MOF was demonstrated to be very stable and selective. The removal percentage of ^{99}Tc increased as the Ni-TIPA/liquid ratio increased, it can remove 90% of TcO_4^- from the SRS tank waste stream at the Ni-TIPA/liquid ratio of 40 mg/mL.

Future Directions

■ Continue developing new MOFs for higher TcO_4^- removal capacity and improved stability, especially under alkaline conditions.

■ Develop methods for vitrifying these MOFs as nuclear waste forms so that the entrapped contaminants are stabilized without leaching out.

■ Prepare proposals to DOE EM Soil & Groundwater Remediation Program, International Program, and DOE Nuclear Energy program.

FY 2019 Publications/Presentations

Dien Li, Natalia B. Shustova, Kathryn M. Taylor-Pashow, Daniel I. Kaplan, Jake W. Amoroso, Thomas D. Bennett, CTAB-functionalized MIL-101-Cr for TcO_4^- and ReO_4^- sequestration from AGW: Capacity and molecular binding mechanisms, **2019**, in preparation.

Nannan Shen, Shuao Wang, Kathryn M. Taylor-Pashow, Dien Li, Mechanism unravelling for ultrafast and selective $^{99}TcO_4^-$ uptake from nuclear waste streams by Ni-TIPA metal organic framework: A combined radiological experiment and molecular dynamics simulation study, **2019**, in preparation.

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Acronyms

AGW	Artificial groundwater
BET	Brunauer-Emmett-Teller
CTAB	Cetyl trimethylammonium bromide
DOE	Department of Energy
EDS	Energy dispersive X-ray spectroscopy
EXAFS	Extended X-ray absorption fine structure
FTIR	Fourier transform infrared spectroscopy
LSC	Liquid scintillation counting
MIL	Materials Institute Lavoisier
MOF	Metal organic framework
PVDF	Polyvinylidene fluoride
SRS	Savannah River Site
TGA	Thermogravimetric analysis
TIPA	Tris(4-(1H-imidazol-1-yl)phenyl)amine

XANES	X-ray absorption near-edge structure
XRD	X-ray diffraction

Intellectual Property

N/A

Total Number of Post-Doctoral Researchers

0.5 of post-doctoral researcher involved through the subcontract with the University of South Carolina.