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European Joint Programme on Radioactive Waste Management

MOBILITY MISSION REPORT

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KLIKNETE NEBO KLEPNETE SEM A ZADEJTE TEXT.

MISSION TITLE

Reactor setup for compacted MX80 and Calcigel bentonite

DESCRIPTION

Concerned organisations

- Research entities:
  1. The team lead by Prof. Rizlan Bernier-Latmani in EPFL, Lausanne, Switzerland. The devemoplemt of entire setup was performed with Dr. Natalia Jakus of the team.
  2. Dr. Luc Robert Van Loon in Paul Scherrer Institute (PSI) in Brugg, Swizerland
  3. Depart of Biogeochemistry, Institue of Resource Ecology at HZDR (home organization)

Concerned infrastructures or facilities

Environmental monitoring facilities

1. Environmental Microbiology Lab (EML) in EPFL
2. Plateforme Technique in EPFL

Concerned phases

Phase 5: Post-closure

Themes and topics

Remove this entire field as well as every below theme and topic that do not apply

- Theme 3: Engineered barrier system (EBS) properties, function and long-term performance

  Clay-based backfills, plugs and seals

  1. Compacting bentonite at PSI
     a) Measurement of the actual size of self-produced reactors
     b) Calculation of the actual weight of bentonites with water cotent for desired dry densty
     c) Compating bentonite the reactor using lever press
  2. Development of best connection situation for reactor setup at EPFL
     a) Operation of peristaltic pump
     b) Selection and testing the suitable connection between metal reactors, tubings, tubing connectors, rubber stopper-sealed bottles and gas tank

Keywords

Compacted bentonite; dry density treshold; microbial diversity; qPCR; microbial survival

EXECUTIVE SUMMARY

Microbial impact in nuclear waste respositories, especially on backfill material (e.g. bentonite) and corrosion remains unclear. In this ConCorD subtask, we would like to understand whether indigenous microorganisms in compacted MX80 and Calcigel bentonites wil survrive in different dry densities when both electron acceptor (sulfate) and donor (hydrogen gas) are available in the respository. The first step for this experiment is to compact bentonite samples into metal reactors and develop a experimental setup to provide both electron acceptor and donor in artificial Opalinus Clay porewater.

Three major components , including reactor, peristaltic pump and reservoirs are essential for the entire setup. In this reseach stay at EPFL, Dr. Natalia Jakus and I have tested the best materials suitable for this experimental setup that will be used from both institutes, EPFL and HZDR. For example, a special tube from MasterFlex for high pressure application is needed to connect between pressurized porewater reservoir and pump, whereas tubes with low gas permeability are connected between pump and reactor. We also came to the conclusion that contantly hydrogen needs to be supplied when the real setup is running, because a dramatic pressure drop in pressurized reservoir was observed when the porewater volume decreased.

In conclusion, the entire experimental setup was established and tested but details as for example where to place the setup under anaerobic condition (in a glove box for example) remains under discussion.

1. MISSION BACKGROUND

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1.1. R&D background

Microorganism in Opalinus clay porewater and also bentonite bacteria were able to oxidize hydrogen gas and reduce sulphate (Bagnoud et al., Nat Commun 7, 12770, 2916 & Matchiavelli et al., *Environ. Sci. Technol.* 17, 53, 2019). These results indicated that the microbial communities in the Opalinus clay and bentonite may be beneficial to DGR by alleviating hydrogen pressure derived from corrosion of metal canister but also to produce H2S that can accelerate the corrosion of the container material. To gain a further insight on microbial activity in backfill material as part of the multi-barrier, we would like to understand whether indigenous microorganisms in compacted MX80 (will be performed by EPFL) and Calcigel bentonites (will be performed by HZDR) wil survrive in different dry densities (between 1.2 to 1.4 g per cubic cetimeter), especially when electron acceptor (sulfate) and electron donor (hydrogen gas) are both present.

1.2. Mission objectives

In this reseach visit at EPFL, a special metal reactor manufactured from the Plateforme Technique of EPFL will be applied to pre-satured MX 80 bentonite. After compacting bentonite with specific dry density in the reactor, the synthetic porewater containing nitrogen gas with sulfate, and the synthetic porewater containing hydrogen gas without sulfate will be injected into this reactor from opposite direction (from top and bottom sides of the reactor). This is to prevent undesired microbial growth on the top surface of compacted bentonite due to slow diffusion rate of injection of porewater containing both electron acceptor and donor for microorganisms in bentonite. The objectives of this visit is to learn how to compact the bentonite samples and to develop a unified protocol and setup that can be used at both instituions, EPFL and HZDR.

1.3. Mission request

EPFL already manufracture 4 metal reactors for testing, and have additional equipment as e.g. peristaltic pump to develop the experimental setup together. Besides, Dr. Luc Robert Van Loon in PSI has years of experiences in compacting bentonites and the reactor setup. He is willing to demonstrate it to us. The main request for this visit is to work together with Dr. Natalia Jakus in Prof. Rizlan Bernier-Latmani's team to develop and optimize the whole experimental setup with the specific conditions, so that it can be used afterwards by both institutes HZDR and EPFL.

1.4. Mission composition

Host organisation

EPFL in Lausanne & PSI in Brugg (one day only) in Switznerland

Host facility

Environmental Microbiology Lab (EML) at EPFL

Mission dates

17th October to 3rd November 2022

2. MAJOR PRACTICES, TECHNIQUES, METHODS, TOOLS OR SYSTEMS OPERATED OR STUDIED

Describe up to four of the most important practice.

2.1. Practice, technique, method, tool or system operated or studied during the mission

Compacting bentonite samples

Description

To learn how to compact bentonite sample into reactor with specific dry density, we visted Dr. Luc Robert Van Loon at the Paul Scherrer Institute (PSI) in Villingen, Schwitzerland.

Usage

Firstly, the water content (% W/W) of bentonite needs to be measured, followed by measuring actual inner space (volume by cubic meter) of cylinder body of the reactor using calliper. The exact weight (gram) of bentonite is calculated with volume of cylinder inner space and dry density, and lever press is then used for compaction. Two metal filters are placed on top and bottom side of compacted bentonite inside the reactor cylinder.

Benefits

Dr. Luc Robert Van Loon has been working on bentonite science for several years. His team has developed a specific method for compacting bentobite in the reactor of his own design. In this one day visit, his team demonstrated us all the techniques and equipment needed for this purpose.

Limitations

Three equipments (lever press, small plug made with harden stainless steel for lever press and special core to remove compacted swollen bentonite) are needed to be produced or purchased. In our testing, we did not have the small plug for actual compaction of bentonite, but this did not affect the whole practice.

Applicability

At the HZDR, we will use the same reactors that are produced from EPFL and we will be able to follow the overall procedure. We will also search for a lever press and produce the harden stainless steel plug locally or at EPFL. We will need to find an alternative device to remove compacted bentonite in the reactors in a laminar flow.

2.2. Practice, technique, method, tool or system operated or studied during the mission

Development and optimization of reactor setup

Description

The design of the metal reactor is modified by the prototype that original designed by Dr. Luc Robert Van Loon as decribed in the application. One reactor has two sets of inflow (for artificial porewater reserviors) and outlet (for waste) both at top and bottom sides. The pores of inflow and outflow are secured by Swagelok spare parts. To prevent undesired microbial growth on the top and bottom surface of metal filters due to slow diffusion rate no porewater containing both electron acceptor and donor will be injected. The best connections between reactors, different inner diameter (ID) of tubings, peristaltic pump and connectors for tubes and needles were tested during the stay.

Usage

Firstly, we tested the peristaltic pump (Ismatec) with a flow rate of 0.1 mL/min. The flow rate of this pump mainly depends on the ID of the pump tubes. We decided to use PharMed Drain Tubing (1.3 mm ID) to maintain the fixed flowrate. For connecting pump tubes a Raccord tuyau PP connector is used to connect C-Flex Laborschlauch tube (1.57 mm ID). The latter is attached to a needle that goes either to the anaerobic bottle with porewater (inflow reservior) or waste (outflow). A special tube from MasterFlex (Versilon A-60-N , 0.8mm ID, for high pressure application) was applied to connect pressurized reservoirs and pump tube. To maintain a fixed pressure (1.5 bar) in the reservoir containing dissolved hydrogen gas (electron donor) in the porewater, constanly gas supply was used. No liquid leakage was observed in the final setup we developed. All the tubing and connectors used here are autoclavable.

Benefits

The development and optimization of this setup is to assure that all materials we used are available commcerially. Most importantly, the experimental condition between both HZDR and EPFL will be consistant.

Limitations

In this practice, we only use CO2 gas to test the pressuring in anaerobic reservoir, and this is because no safet cabinet for hydrogen gas was available. Also, we cannot detect whether there will be gas leakage in this setup.

Applicability

At HZDR the setup will be able to develop. For hydrogen gassing, our lab is already installed with metal pipe that connect to the hydrogas tank in safety cabinet outside of building. So the setup is highy applicable in HZDR.

2.3. Practice, technique, method, tool or system operated or studied during the mission

Replace this entire field with the name of the practice, technique, method, tool or system that is the object of this mission.

Description

Replace this entire field with a description of the implementation of this practice, technique, method, tool or system at the host organisation.

Usage

Replace this entire field with a description of your operation or study of this practice, technique, method, tool or system during the mission.

Benefits

Replace this entire field with a description of the benefits for implementing this practice, technique, method, tool or system.

Limitations

Replace this entire field with a description the limitations of this practice, technique, method, tool or system.

Applicability

Replace this entire field with a description of how this practice, technique, method, tool or system could be implemented in or adjusted to your home context.

2.4. Practice, technique, method, tool or system operated or studied during the mission

Replace this entire field with the name of the practice, technique, method, tool or system that is the object of this mission.

Description

Replace this entire field with a description of the implementation of this practice, technique, method, tool or system at the host organisation.

Usage

Replace this entire field with a description of your operation or study of this practice, technique, method, tool or system during the mission.

Benefits

Replace this entire field with a description of the benefits for implementing this practice, technique, method, tool or system.

Limitations

Replace this entire field with a description the limitations of this practice, technique, method, tool or system.

Applicability

Replace this entire field with a description of how this practice, technique, method, tool or system could be implemented in or adjusted to your home context.

3. MISSION FINDINGS AND CONCLUSIONS

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3.1. Lessons learned and conclusions

The most difficult part is to find the right tubing for the three major components: reactor, peristalitic pump and anaerobic bottle for reservoir. For example, the pore of in-and out-flow on the top and bottom sides of reactors are screwed with Swagelok ferrule with specific size. We had to test different types of tubing with different features (e.g. low gas permeability, high pressure tolerance, autoclavable or bending capacity). Beside, the handling of spare parts from Swagelok needs special care: the wrench that matches the screw is necessary and the installation of these parts need special number of turns. In conclusions, we have decided the best materials and consumables that are commercially available for the entire setup.

We also realized how fast the pressure drops even with the very low flow rate (0.1 mL/min) that was set for the peristaltic pump. Based on Dr. Natalis Jakus's calculation, with 1.5 bar of pressure in the headspace of reservoir, the concentration of dissolved hydrogen gas is 32 mM; however, with the decrease of porewater volume, the concentration of dissolved hydrogen gas drops to ~10 mM after 8 hours due to the decrease of pressure down to 0.96 bar. We then measured the pressure drop in the actual setup and surprisingly, within 3 hours the pressure dropped to 0.9 bar. This might be due to the entire setup is not a closed system so the pressure calibrate automatically. We then decided to constantly supply the gas to the reservoir to see if this could be the solution for this issue.

In conclusion both home and host organisaitons have developed and optimized the whole setup together.

3.2. Relevant findings and conclusions for home organisation

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3.3. Relevant findings and conclusions for host organisation

Klikněte nebo klepněte sem a zadejte text.

3.4. Relevant findings and conclusions for other organisations

Klikněte nebo klepněte sem a zadejte text.

4. POTENTIALS FOR IMPROVEMENT OR DEVELOPMENT

4.1. Generic potentials

Despite that there was no liquid leakage discovered in the setup, a potential gas leakage from the anaerobic bottle or atmospheric gas (oxygen for example) into the entire setup was uncertain. Due to the running time for this experiment, which will be up to 3-5 weeks with the low flowrate, big Schott bottle (up to 5L) will be needed instead of small narraow neck bottle. We ordered a special bromobutyl ruber stopper (DURAN) for Schott bottle and this stopper is designed for low gas permeability and high self-sealing capacity for needle pierecing. However, this material did not arrive during the mission.

To prevent oxygen diffusion, the whole setup should be placed under strict anaerobic environment such as glovebox. However, due to the constant injection of hydrogen gas, the glovebox needs to be examined and modified if an external gas pipe can be installed into the box. This process could be time- and cost-consuming. Another alternative way is using a glovebag under a fume hood. The biggest advantage of using a glovebag is that the release of hydrogen sulfate or sulphide and hydrogen gas will be easier in a fume hood.

These two are potential improvements for this reactor setup for both home and host organisations.

4.2. Potentials for home organisation

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4.3. Potentials for host organisation

Klikněte nebo klepněte sem a zadejte text.

APPENDICES

Mission journal

17/10/22: Reactor examination and to-do-list planning (one page here!)

18/10/22: Visit at PSI to learn how to compact bentonite

19/10/22: Repition of all the procedure of compacting MX80 bentonite learned at PSI and ordering consumables. Establishment of an Excel sheet with all the formula for calculation of actual inner space of reactor cylinder

20/10/22: Test peristaltic pump (Ismatec) with settings, different Ismatec pump tubes (PharMed for autoclavable material), outflow volume estimation and ordering more consumables

21/10/22: Preparation of porewater solution and test tubes that are already available in EML, and connect to reactor and run the simple setup over the weekend to mimic the bentonite saturation (at this point no pressurized porewater was used)

22/10/22: Discuss with a technician at Plateforme Technique in EPFL about production harden stainless steel plug that was sketched by Dr. Natalia Jakus. Test C-Flex tubing we ordered but did not fit well, ordered another C-Flex tubing (low gas permeability) with 1.58 mm ID.

23/10/22: Testing the setup with pressurized (using CO2 with 1.5 bar) anaerobic bottle containing porewater. Liquid leakage discovered at connector between bottle and pump tube. But no liquid leakage discovered without pressure. Measuring pressure decrease and calculate possible concentration of dissolved H2 gas in porewater.

24/10/22: Testing the setup with lower pressurized (using CO2 with 1 bar) anaerobic bottle containing porewater, still liquid leakage and pressure drop, testing with pressurized (1.5 bar) waste bottle (outflow from reactors) to check if the pump can maintain same flow rate with back pressure and if pressure drop in anaerobic bottle can be delayed, however, problem remained

25/10/22: Testing with new C-Flex tubing with 1.58 mm ID. This tube fits perfect for all the components in this setup: reactor (the ferrule of Swagelok), connectors, needles, and Ismatec pump tubes. But same issues remained, even using Teflon tape to enhance the tightness between connectors and tubes.

26/10/22: Testing with an old tubing from Masterflex® L/S® Precision Pump Tubing, Versilon™ 0.8 mm ID that was already avalaible in EML. This material is made for vacuum/pressure applications (max. 2.7 bar). This tube was placed in between and no liquid leakage was discovered, but still the pressure droped.

31/10/22: Same setup and trying to supply gas constanly to maintain fixed pressure.

01/11/22:Same setup with pre-satured bentonite and supply gas constanly overnight.

02/11/22: System seems to be stable. Discussion about where to conduct the whole setup: glovebox with hardware modification or glovebad under fumehood.

03/11 22: Summarizing all the consumables we tested and discussion about all the possible issues.

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MISSION BENEFICIARY

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PARTNER EXPERTS CONTRIBUTING TO THE MISSION

Host organisation experts

- Prof. Rizlan Bernier-Latmani and Dr. Natalia Jakus
- School of Architecture, Civil and Environmental Engineering
- Environmental Engineering Institute
- Ecole Polytechnique federale de Lausanne (EPFL)

Home organisation experts

- Dr. Andrea Cherkouk
- Head of working group MICRONUC
- Department of Biogeochemistry, Institute of Resource Ecology
- Helmholtz-Zentrum Dresden-Rossendorf (HZDR) Germany

Other organisations experts

- Dr. Luc Robert Van Loon
- Scientist/group leader
- Paul Scherrer Institute (PSI), Switzerland

REPORT APPROVAL

Date	Beneficiary	Home mentor/supervisor	Host mentor/supervisor
Date of last signee	Dr. Ting-Shyang Wei	Dr. Andrea Cherkouk	Prof. Rizlan Bernier-Latmani
29/11/2022	Visa Ting-Shyang Wei	Visa Andrea Cherkouk	Visa [signature]

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