Colloid mobility & radionuclide interaction

WP 3 Thorsten Schäfer (KIT-INE)
In detail presentation concerning WP3

- “Sorption behavior of Np(V) onto clays from Russian and Indian deposits” - Anna Yu Romanchuck (MSU)
- “Study of radionuclides migration through crushed granite in presence of bentonite colloids” - Kateřina Kolomá (UJV)
- “Effect of clay nanoparticle mobility, desorption and redox kinetics on radionuclide mobility investigated in an underground research laboratory (Grimsel Test Site, Switzerland)” - Thorsten Schäfer (originally foreseen Ingo Blechschmidt)
- “Radionuclide transport in granite fractures in the presence of bentonite colloids: Summary of the studies carried out at Ciemat” - Tiziana Missana (CIEMAT)
**Work package 3: Objectives**

**WP 2**
Colloids generated through bentonite erosion

**WP 3**
Colloids as carrier of radionuclides

**WP 4**
Colloid stability hydrochemical conditions

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**a) Colloid mobility:**
- Fracture geometry (aperture size distribution and fracture surface roughness)
- Chemical heterogeneity induced by the different mineral phases
- Chemistry of the matrix porewater

**b) Radionuclide interaction:**
- Process understanding of reversibility of sorption
- Implementation in thermodynamic models
- Identifying additional retention processes => matrix diffusion
Key questions

1. What are the colloid mobility controlling processes and can we describe them appropriate?

2. Is the sorption of strongly sorbing radionuclides fully reversible, why do we observe kinetics?

3. Have we indications for additional retention processes occurring?
- **Macroscale investigations** on colloid mobility in near-natural systems,
- **Microscale investigations** on colloid mobility controlling processes,
- Process understanding of **radionuclide** colloid interaction with special emphasis on **sorption reversibility**
- **Kinetic model approach**
- **Mechanistical model** of RN colloid interaction.
Retardation of colloid transport in the far field, will delay the arrival of radionuclides in the biosphere.

=> The extent of this isn’t currently taken into account.
Table 2.1: Selected composition of the Forsmark reference, saline, ice-melting and buffer-equilibrated waters. Concentrations are in mol/dm$^3$ (Duro et al. 2006a, TR-06-32, Table 3-1).

<table>
<thead>
<tr>
<th></th>
<th>Forsmark reference water 1)</th>
<th>Saline water 2)</th>
<th>Ice melting water 3)</th>
<th>Buffer-equilibrated water 4)</th>
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<td><strong>pH (downhole in situ for reference water)</strong></td>
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<td>7.9</td>
<td>9.6</td>
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<td><strong>Eh (downhole in situ for reference water) (mV)</strong></td>
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<td>-200</td>
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<td>[K$^+$]tot</td>
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<tr>
<td>[Ca$^{2+}$]tot</td>
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<td>[P]tot</td>
<td>3.23E-05</td>
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</tbody>
</table>

1) SKB. Fers. Comm.
2) K/LX02 in the interval 1,420–1,705 m with date of sampling 94/0 1/17. /Laaksoharju et al. 1995/.
3) Grimsel groundwater composition [discharging groundwater from the Migration shear zone (AU 96)]. (Data compiled from /Bajo et al. 1989/, /Aksoyoglu et al. 1990/ and /Elkenberg et al. 1991/).
4) Forsmark groundwater interacted with the bentonite buffer /Arcos et al. 2006/.

Dilute Glacial melt water
Colloid retardation/retention

Recovery \( f(v, l) \) column migration experiments

**Graphs:**

- **Left Graph:**
  - Total Colloid Recovery
  - Recovery range: 19-21%
  - Eluted Volume (mL)
- **Right Graph:**
  - Water Velocity (m/s) \( \times 1E-06 \)
  - Mixed Ca+Na Water 1 \( \times 10^{-3} \) M
  - Aspo Water
  - NaClO\(_4\) 5 \( \times 10^{-4} \) M

**Equation:**

- \( Q = 7 \text{ mL/h} \)
Colloid retention has been observed even at conditions where high retention was not expected, i.e. under unfavorable electrostatic conditions (Missana et al., 2008a; Schäfer et al., 2004). The mechanisms that contribute to colloid retention are not yet fully understood. (Geckeis et al, 2008; Missana et al, 2008b; Albarran et al, 2011; Missana et al, 2011; Schäfer et al, 2012).

- Smaller grains (FFM), lower velocity, greater surface area ⇒ higher deposition rate

Microscopic information needed to clearly identify underlying colloid attachment process (charge, surface roughness)
Colloid mobility in fractured rocks (PIXE investigations)

- Colloid diffusion in rock matrix can eliminate colloids from the flow paths despite being considered a minor mechanism. **All diffusion coefficients measured were in the range of** $D_a = 10^{-18} \text{ m}^2/\text{s}$. No significant differences in $D_a$ were measured amongst colloid of different charge but the main changes were attributed to size variations. Maximum surface distribution coefficients measured were $K_a = 3 \cdot 10^{-3} \text{ m}$. 

- In general, Au colloids are not specifically retained on any mineral.

- In some cases, Au colloids are retained on defects! Size effects are also observed.
Tailing slope has frequently been interpreted by matrix diffusion, tailing of the colloids is mainly caused by the structure of the flow field and that for the colloid transport matrix diffusion is of minor importance. (Kosakowski, 2004; Möri et al., 2003; Huber et al., 2012).
Colloid recoveries \( f(\text{fracture orientation, surface roughness}) \)

Madeleine Stoll et al. (to be submitted)

\[ C(t) = C_0 \cdot e^{-\lambda \cdot t} \]

\[ C(t) = C_0 \cdot e^{-\lambda_1 \cdot t} + C_0 \cdot e^{-\lambda_2 \cdot t} \]

Particle retention influenced by fracture orientation

Particle retention influenced by fracture surface material
Atomic Force Microscopy

AFM measurements on granite and acrylic glass disc

1) Topography

- Both granite and acrylic glass disc are scanned by a 1 μm colloid probe and ~24 nm sharp tip to obtain the topography of the substrates and further to quantify the roughness ($R_q$):  

$$R_q = \sqrt{\frac{1}{n} \sum_{i=1}^{n} z_i^2}$$

Parameters measured:
1. Surface topography
2. $F_{\text{Adhesion}}$
3. Snap-in force
4. Force-volume measurements
1) Topographic images and surface profiles

Image obtained with 1000 nm colloid probe
Image obtained with 24 nm sharp tip

Increased roughness directly enhances diffusivity of the 25 nm particles into scratches and cracks on the substrate in contrast to the 1000 nm particles.
Colloid recoveries $f(\text{fracture orientation})$

Pure sedimentation independent of the surface used => fracture orientation is the key
Is the assumption of **reversible linear sorption** of radionuclides on colloids justified?
Linear sorption assumed

**Strontium**

Cation exchange & inner sphere surface complexation; **Linear**.

**Cadmium**

Cation exchange & inner and outer sphere surface complexation; **Linear**.
Linear sorption assumed

Sorption of Cm(III) and Eu(III) onto clay minerals

Sorption edges for Eu (m_{Eu, total} = 2.0 \cdot 10^{-7} m), S:L 2 g/L, on Na-SWy-2 montmorillonite as a function of pH_m and at different NaCl concentrations.
Pu(V) & Pu(IV) sorption to montmorillonite

Begg, J. D.; Zavarin, M.; Zhao, P. H.; Tumey, S. J.; Powell, B.; Kersting, A. B., Pu(V) and Pu(IV) Sorption to Montmorillonite. *Environ. Sci. Technol.* **2013, 47, (10),** *5146-5153.*
Sorption isotherms of Np(V) onto corundum, montmorillonite and bentonite colloids

- TRIS and CHES buffer solution
- Na- montmorillonite and
- Bentonite colloid dispersion (MX-80 Volclay)
- N₂-glove box (exclusion of Np-carbonato complexes)
### From WP1

<table>
<thead>
<tr>
<th>Issue</th>
<th>Safety case position at start of BELBaR</th>
<th>Outcomes for final State-of-art report</th>
</tr>
</thead>
</table>
| Radionuclide sorption | To assess the possible role of rapid reversible sorption/desorption onto colloids in facilitating transport, the following assumptions have been adopted:  
1. equilibrium sorption of radionuclides onto mobile and immobile colloids,  
2. equilibrium sorption of colloids onto fracture surfaces, and  
3. colloid-free matrix pore space (conservative assumption, but also realistic for the small pore sizes of granitic rock).  
Reversible, linear sorption of radionuclides onto colloids has been assumed. | Is the assumption of reversible, linear sorption of radionuclides onto colloids justified? (WP3) |

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**Colloid, radionuclide and host rock interactions (WP3) – the main thing here is to determine the sorption reversibility of radionuclides to the colloids and whether the current assumption of reversible sorption can be justified.**
Why does ‘irreversible’ binding matter

- Effect of dissociation rate constant on transport

For humics:

\[ k_b = 10^{-7} - 10^{-6} \text{ s}^{-1} \]
\[ k_b = 0.00036 - 0.0036 \text{ h}^{-1} \]
\[ k_b = 3.15 - 31.54 \text{ a}^{-1} \]

Reversibility tests via:

- pH decrease
- Ionic strength increase
- Addition of rock/fracture filling material
- Addition of organic ligands (FA / EDTA)
Radionuclides to be discussed

Geochemical systematics of the radionuclides

<table>
<thead>
<tr>
<th>Reduction</th>
</tr>
</thead>
<tbody>
<tr>
<td>M(I) Cs⁺</td>
</tr>
<tr>
<td>M(II)</td>
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<tr>
<td>M(III)</td>
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<tr>
<td>M(IV)</td>
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<tr>
<td>M(V) NpO₂⁺ PuO₂⁺</td>
</tr>
<tr>
<td>M(VI) UO₂²⁺ PuO₂²⁺</td>
</tr>
<tr>
<td>M(VII) TcO₄⁻</td>
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</tbody>
</table>

Sorption reactions: Ionic exchange

Surface complexation / Mineralization

Reactions in solution: complex formation / colloid formation
Cs reversibility vs. irreversibility


- The amount of poorly exchangeable Cs on the planar sites represented approximately 3.5% of the total planar site concentration.

- Collapse of vermiculite and smectite interlayers induced by sorbed Cs or the high KNO₃ desorption solution may explain this poorly reversible fraction.

- This apparent fixation occurred almost immediately after Cs addition, did not correlate with aging time of the sorption complex (0.5-120 d) and reversibility was negligible even after 500 h of contact with excess K.
Cs reversibility vs. irreversibility

The reversibility has been verified for montmorillonite. In adsorption experiments at lower Cs+ concentration <10^{-4} M, Cs+ adsorption was full reversible. (Kunipia F®)


- 1% of cesium passed through crushed granite sorbed on bentonite colloids
- however the most of cesium was desorbed from bentonite colloids and retained in granitic rock.
- Thus, cesium sorption on bentonite is mostly reversible under the hydraulic conditions.
Cs reversibility vs. irreversibility


- **Callovo-Oxfordian Clay Rock** in all cases studied (reversibility tests performed after a sorption phase in the compacted state fixed between 5 months and 5 years), the sorption process was reversible in disagreement with results obtained on similar but different illite materials.

- Furthermore, no significant difference was found between dispersed and compacted systems, so no compaction effect on Kd values was observed.
Cs reversibility vs. irreversibility

This difference in behavior may be related to the difference in the illite studied, i.e. a soil-type illite (Comans and Hockley, 1992; Smith and Comans, 1996; de Koning and Comans, 2004), which would be more altered with more open “FES” than a sedimentary formation-type illite.

This stresses the need to study not only generic clay samples but also samples that are relevant to the sites/buffer/backfill material under consideration.

Chemical purification might induce artefacts.

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Radionuclides to be discussed

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</tr>
<tr>
<td>Ra²⁺</td>
<td>Cm³⁺</td>
<td>Cm³⁺</td>
<td>PuO₂⁺</td>
<td>PuO₂⁻</td>
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</tr>
<tr>
<td>REE³⁺</td>
<td></td>
<td>REE³⁺</td>
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</tbody>
</table>

Sorption reactions: Ionic exchange

Surface complexation / Mineralization

Reactions in solution:

complex formation / colloid formation
Eu(III) reversibility vs. irreversibility

- Evidence for slow release of Eu(III) from the bentonite;
- Amount of ‘non-exchangeable’ Eu(III) increases with pre-equilibration time;
- All systems still heading towards equilibrium.

Bulk Ligand competition experiments (EDTA)

Na-Montmorillonite (Wyoming) SWy-1

‘Equilibrium’ = concentration when EDTA is present from start.

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29 07./08.03.2012 Thorsten Schäfer CP BELBaR Kick-off Meeting, Lund

Institut für Nukleare Entsorgung (INE)
Ligand competition experiments (EDTA): log plots

- Dissociation rate constant does not depend upon pre-equilibration time (beyond 1 week);
- Small increase in amount slowly dissociating with time.
Colloid (200-500nm) dissociation: log plots

- Kinetics are remarkably uniform between the different systems;
- Unlike the bulk experiments, a single dissociation rate can describe the dissociation kinetics from 1 day onwards.
Dissociation Rate Constants Eu(III)

BULK BENTONITE
Average rate constant: $5.67 \times 10^{-8} \text{ s}^{-1}$ \textcolor{red}{(1.8 a^{-1})}
Rate constant range: $3.7 \times 10^{-8}$ - $1.0 \times 10^{-7} \text{ s}^{-1}$
Amount: approximately 25%, constant after 100 days

COLLOIDAL BENTONITE
Average rate constant: $8.9 \times 10^{-7} \text{ s}^{-1}$ \textcolor{red}{(28.1 a^{-1})}
Rate constant range: $8.3 \times 10^{-7}$ - $9.5 \times 10^{-7} \text{ s}^{-1}$
Amount: 64 – 70%, independent of pre-equil’ time
Spectroscopic evidence for full reversibility?


- Fluorescence lifetime ($\tau$) measurements up to weakly alkaline pH conditions show in addition to the Cm$^{3+}$ aquo ion with $t = 68 \pm 2 \, \mu$s a lifetime component of $t = 120 \pm 15 \, \mu$s for montmorillonite which can be attributed to inner-sphere surface species
- $\Rightarrow$ No indication of structural incorporation / irreversible sorption
## Radionuclides to be discussed

### Geochemical systematics of the radionuclides

<table>
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<tr>
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<tr>
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<td>REE³⁺</td>
<td>U⁴⁺</td>
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<td>Tc⁴⁺</td>
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</table>

### Sorption reactions:
- Ionic exchange

### Reactions in solution:
- Complex formation / colloid formation
- Mineralization

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**Radionuclides discussed:**
- Cs⁺
- Sr²⁺
- Ra²⁺
- Am³⁺
- Cm³⁺
- REE³⁺
- Pu⁴⁺
- Np⁴⁺
- U⁴⁺
- Tc⁴⁺
- NpO₂⁺
- PuO₂⁺
- PuO₂²⁺
- TcO₄⁻
ATR FT-IR measurements of Np(V) sorption

Fast desorption kinetics could be observed with ATR-FT IR, where a complete removal of the neptunium(V) sorption complex characterized by an adsorption band at 790 cm\(^{-1}\) was observed.

50 µM Np(V); 0.01 M NaCl in D\(_2\)O; pD 9.6; flow rate 0.1 mL/min.
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<table>
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<td>REE³⁺</td>
<td>U⁴⁺</td>
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<td>Tc⁴⁺</td>
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Geochemical systematics of the radionuclides

- Reduction
- Sorption reactions:
- Ionic exchange
- Surface complexation / Mineralization
- Complex formation / Colloid formation
Eu(III)- versus Th(IV) bentonite colloid sorption reversibility in contact with humic substances


Th(IV): \(4.3 \times 10^{-8}\) mol/L; 20 mg/L Febex bentonite colloids; 10 mg/L HA

Process understanding pending in the tetravalent actinide case!
Bentonite radionuclide desorption kinetics


From binary system data
(Note: No colloid – FFM interaction)

\[
R_{d,tot} = \frac{K_{d,FFM}}{1 + C_C \cdot K_{d, col}}
\]

*Binary data from NTB 03-02

<table>
<thead>
<tr>
<th></th>
<th>$^{232}\text{Th(IV)}$</th>
<th>$^{242}\text{Pu(IV)}$</th>
<th>$^{243}\text{Am(III)}$</th>
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<tr>
<td>syn. Zn-Montm.</td>
<td>8.0 [a⁻¹]</td>
<td>2.0 [a⁻¹]</td>
<td>10.5 [a⁻¹]</td>
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<td>syn. Ni-Montm.</td>
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<td>13.2 [a⁻¹]</td>
<td>10.5 [a⁻¹]</td>
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<tr>
<td>FEBEX bentonite</td>
<td>not possible</td>
<td>7.8 [a⁻¹]</td>
<td>32.4 [a⁻¹]</td>
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<tr>
<td>colloids</td>
<td></td>
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</table>

Pu(IV) desorption rate (M. Zavarin, LLNL): 1.5 [a⁻¹]
Desorption experiments on MX-80, the Th(IV) case $^{232}\text{Th} = 10^{-8}\text{M}$


- **pH decrease to 2.7**
  - Th: is initially sorbed (> 95 %) to all clay colloidal fractions. After 1 year at pH 2.7, whatever the first sorption contact times, 80 ±5 % of the Th are desorbed from the clay colloids.

- **Ionic strength increase: 0.5 M IS with CaCl$_2$, pH 7.4**
  - Th and Pu: previously sorbed remain associated with the clay colloids which sediment.

- **Addition of crushed bedrock material (CBM): S/L 1:4**
  - After 1 year, drastic changes are observed as a function of contact time prior to the addition of the CBM and the clay colloidal size fractions. Longer $t_{\text{sorp}}$ reduces the colloid desorption as well as smaller size fractions.

- **Addition of FA (5 mg·L$^{-1}$) after different contact time**
  - No strong desorption (not more than 10 % after 1 year), whatever the clay colloidal size fractions.
Upscaling: Reference case for a generic German repository in crystalline rock TRAPIC/ COFRAME

- No colloid filtration
- 20 mg/L colloids

**Variation of the desorption rate**

$$Da = \frac{L}{v} k_b$$

- **Da = 0.05**
- **Da = 5.5**
- **Da = 20**
- **Da = 550**
- **Da = 5500**
- **Da = 5500**

**Da > 50 equilibrium assumption**

**WARNING:** not necessarily conservative!

- No colloid filtration
- 20 mg/L colloids

Effect of colloid remobilization

<table>
<thead>
<tr>
<th>Parameter</th>
<th>Symbol</th>
<th>Value</th>
</tr>
</thead>
<tbody>
<tr>
<td>Fracture aperture</td>
<td>(2b)</td>
<td>0.0001 m</td>
</tr>
<tr>
<td>Colloid velocity</td>
<td>(u_c)</td>
<td>13.05 m/year</td>
</tr>
<tr>
<td>Filtration coefficient</td>
<td>(\lambda_f)</td>
<td>0.1 m(^{-1})</td>
</tr>
<tr>
<td>Longitudinal dispersion length for colloids</td>
<td>(\alpha_c)</td>
<td>0.5 m</td>
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</table>

## Deliverables

### Work Package 3

<table>
<thead>
<tr>
<th>Deliverable ID</th>
<th>Description</th>
<th>Contributors</th>
<th>Status</th>
<th>Due Date</th>
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<tbody>
<tr>
<td>D3.1</td>
<td>Progress report on microscale investigations on colloid mobility controlling processes.</td>
<td>MSU, CIEMAT, KIT-INE</td>
<td>R</td>
<td>PU</td>
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<td>D3.2</td>
<td>Macroscale investigations on colloid mobility in near-natural systems</td>
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Thank you for your attention!

This work has been funded by the European Atomic Energy Community’s 7th Framework Programme (FP7/2007-2011) under grant agreement no. 295487 (BELBaR Project)

....missed the celebration by one day!!!!