Update on Smectite-to-Illite Transformation: Laboratory Experiments

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Hydrothermal reaction of smectite to illite important for bentonites as a barrier

Understanding the thermal alteration effects is dependent on:

- Time
- Temperature
- Si and K⁺ concentrations
- Solid to liquid ratio
- Possibly interlayer cations

Typically thought to take a long time for conversion

Conducted laboratory experiments for diagenetic reactions in Parr Vessel reactors at 200°C for 1 week with variable loadings and exchanged cations (K, Cs, NH₄)
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**Analysis Techniques**

- XRD for basal spacing
- Cation exchange capacity (CEC)
- Recorded mass loss data and pH of reacted solutions
- Surface area by BET nitrogen adsorption
- Analysis of solutions after reaction by IC, ICP-MS, ICP-OES
  - Determine dissolved ions
- Particle size by Malvern Zetasizer
- SEM-EDS for morphology and compositional mapping
- XRF for composition
Results: XRD

Na-Form Smectite

5/23/18 Crystalline R&D: Smectite-to-Illite Transformation
K-Form Smectite

Results: XRD Continued
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Future R&D

- **Perform additional characterization techniques**
  - Particle size analysis by Zetasizer
  - Composition by XRF
  - Morphology by SEM
  - Dissolved ions by ICP

- **Investigate behavior of iron in reaction**

- **Addition of mica to possibly escalate conversion**

- **Addition of quartz to inhibit conversion**
Additional Slides
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Results: XRD

Starting Material
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Results: XRD

Cs-Exchanged Smectite
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Results: XRD

NH$_4$-Exchanged Smectite
Results: Surface Area

<table>
<thead>
<tr>
<th>Raw material</th>
<th>Reacted Na-Smectite</th>
<th>Reacted K-Smectite</th>
<th>Reacted NH4-Smectite</th>
<th>Reacted Cs-Smectite</th>
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Results: CEC

![Graph showing CEC (meq/100g clay) for different materials and conditions.]

- Na Form-Smectite
- Illite
- Na Form-Smectite
- Cs-exchanged Smectite
- K-exchanged Smectite
- NH4-exchanged Smectite

Key:
- Starting Material
- 100
- 500
- 1000

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Crystalline R&D: Smectite-to-Illite Transformation
Results: Mass Loss and pH

- **Mass Loss (%)**
  - Na-Form Smectite
  - Cs-exchanged Smectite
  - K-exchanged Smectite
  - NH4-exchanged Smectite

- **pH**
  - pH 1 M KCl = 5.5

- Liquid/Solid Ratio:
  - 100
  - 500
  - 1000

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Results: SEM

Cs-Exchanged Smectite

Cs-Exchanged Smectite: 1000 liquid/solid ratio