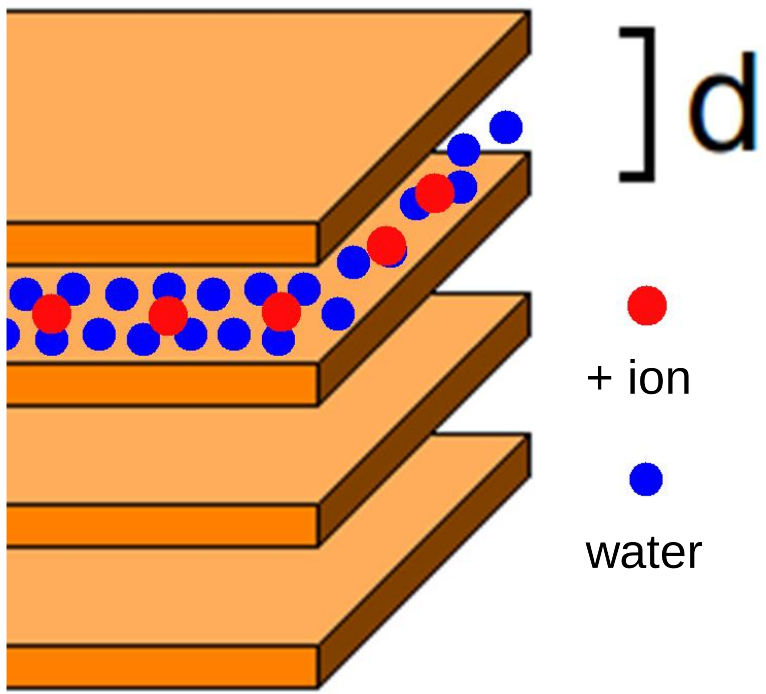
Characterization of clay using x-ray and neutron scattering at the University of Helsinki and ILL

Ville Liljeström, Michał Matusewicz, Kari Pirkkalainen, Jussi-Petteri Suuronen and Ritva Serimaa

Introduction

Some characteristics about the microstructure of montmorillonite clay

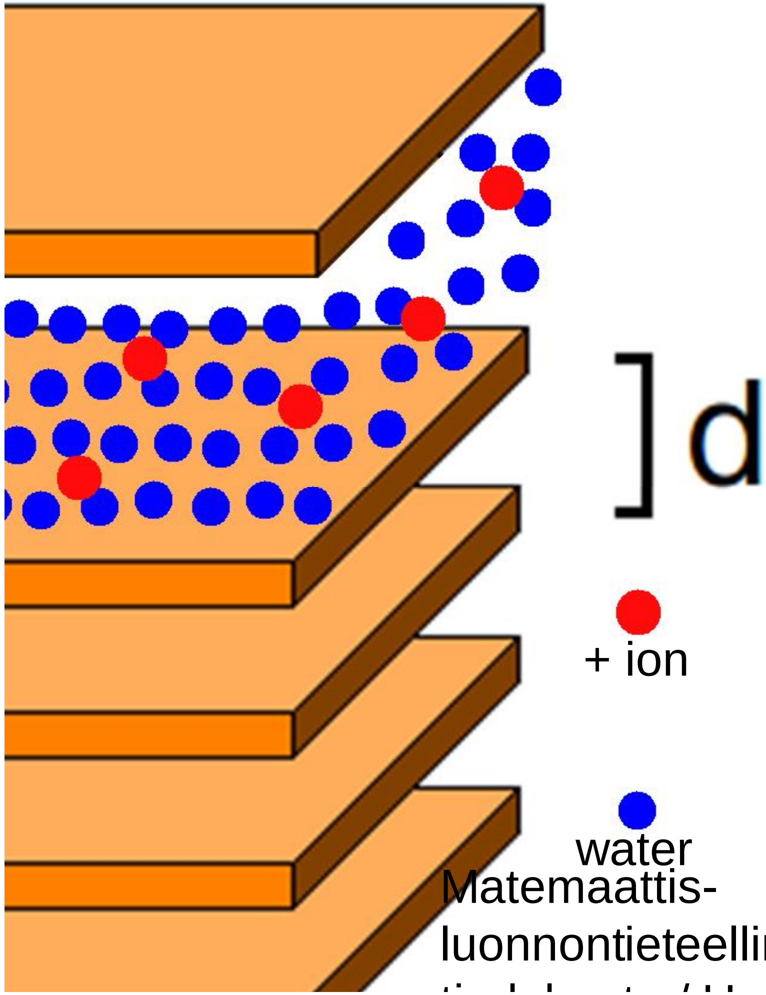


- The exact nanostructure is varying and not perfectly known
- Lamellar nanostructure: contains stacks (tactoids) of crystalline platelets
- A platelet has a thickness of ~1nm
- Platelets are negatively charged

→ Counter ions between platelets (typically Na^{+}/Ca^{2+})

- One stack can contain 5-20 platelets

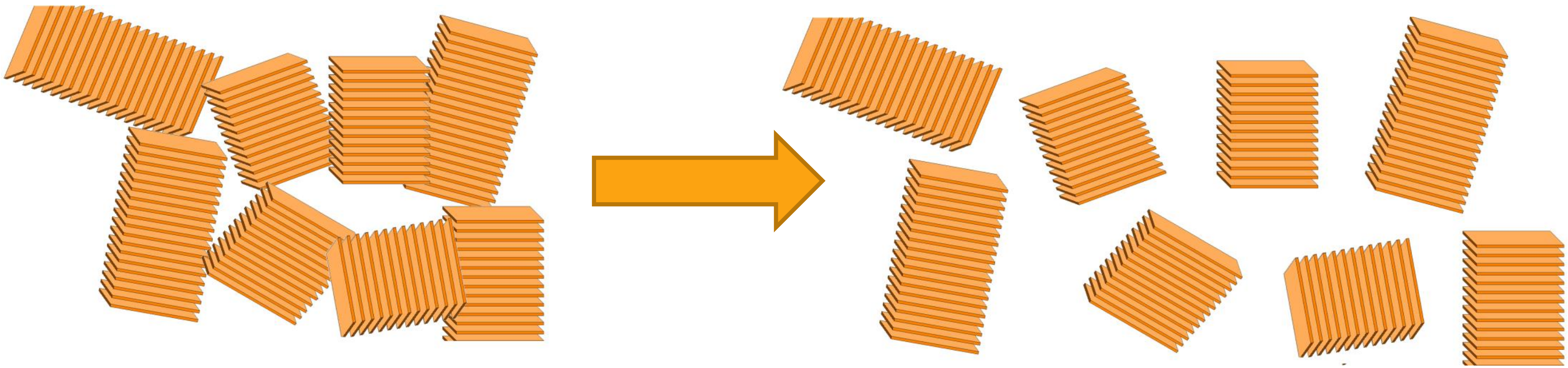
How is the microstructure of clay affected by surrounding conditions and processing?



- Moisture content (swelling, increasing lamellar disorder)
- Salinity of water (stability of lamellar structure)
- Label of counter ion (affects swelling and stability of lamellar structure)
- Not yet considered: temperature, pressure

Swelling due to moisture adsorption can increase permeability of clay barrier

- intralamellar swelling (organized water bound to interlamellar spacings)
- extralamellar swelling (spacing between tactoids increasing)
- Extralamellar swelling can increases permeability



Methods

Small Angle X-ray Scattering (SAXS)

- A way to characterize small structures
  - Range ~ 1-30 nm
- Gives information about the nanostructure on average (compare TEM & nanotomography)
- Does not give information about crystal structures
- Does not give information of large (µm) homogenous particles, like crystalline silica grains.

Small Angle Neutron Scattering (SANS)

- The same as SAXS, but monochromatic neutron beam instead of monochromatic x-ray beam.
    - Range ~ 1-300 nm at ILL (Institut Laue-Langevin)
- Possible to measure confined samples under pressure

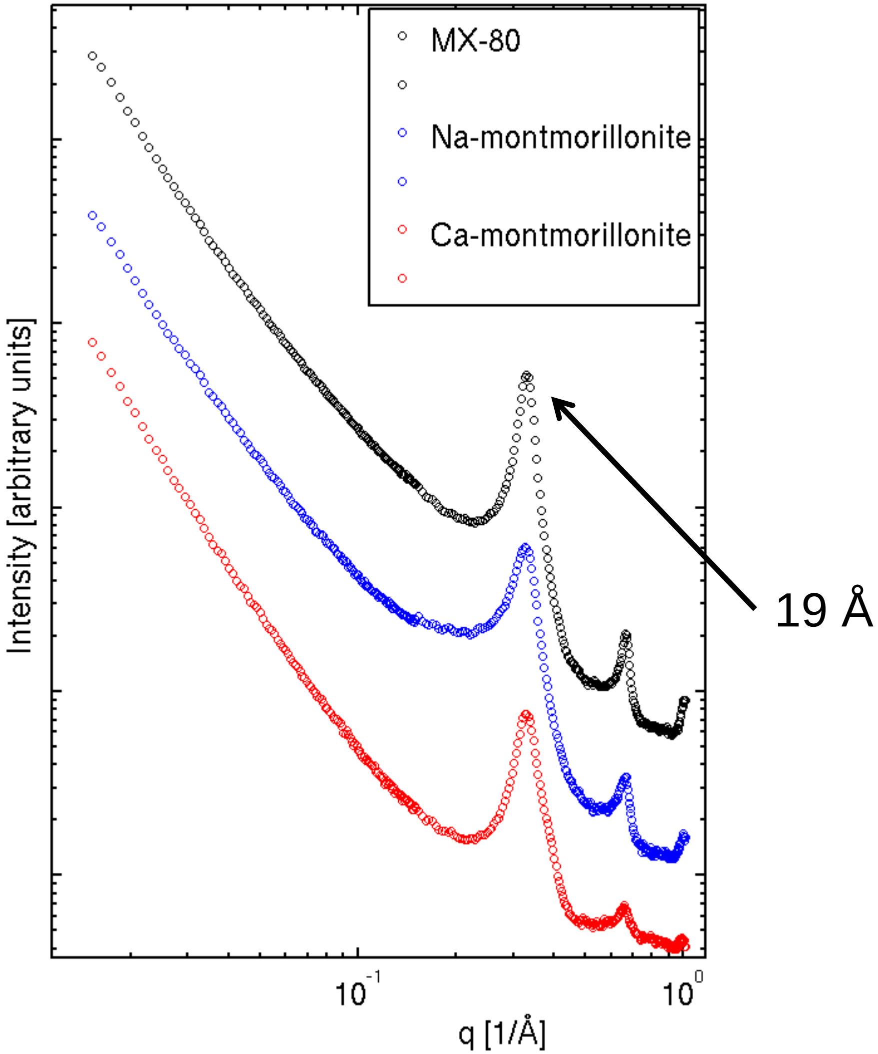
Measured samples series

(Na and Ca correspond to counter-ion label)

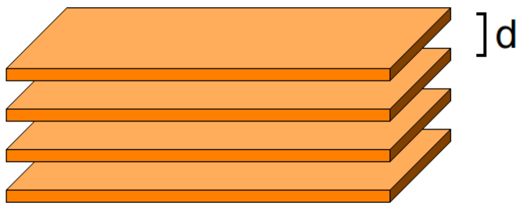
1. MX-80, Na-montmorillonite, Ca-montmorillonite, dry density: 1.3 g/cm^3
2. MX-80 (deionized water vs. NaCl-solution), dry densities: 0.7 g/cm^3, 1.0 g/cm^3, 1.3 g/cm^3, 1.6 g/cm^3
3. MX-80, Na-montmorillonite, Ca-montmorillonite, (deionized water vs. sodium perchlorate solution), Na-montmorillonite sample (NaCl-solution), dry density: ~1.6 g/cm^3

Results & Conclusions

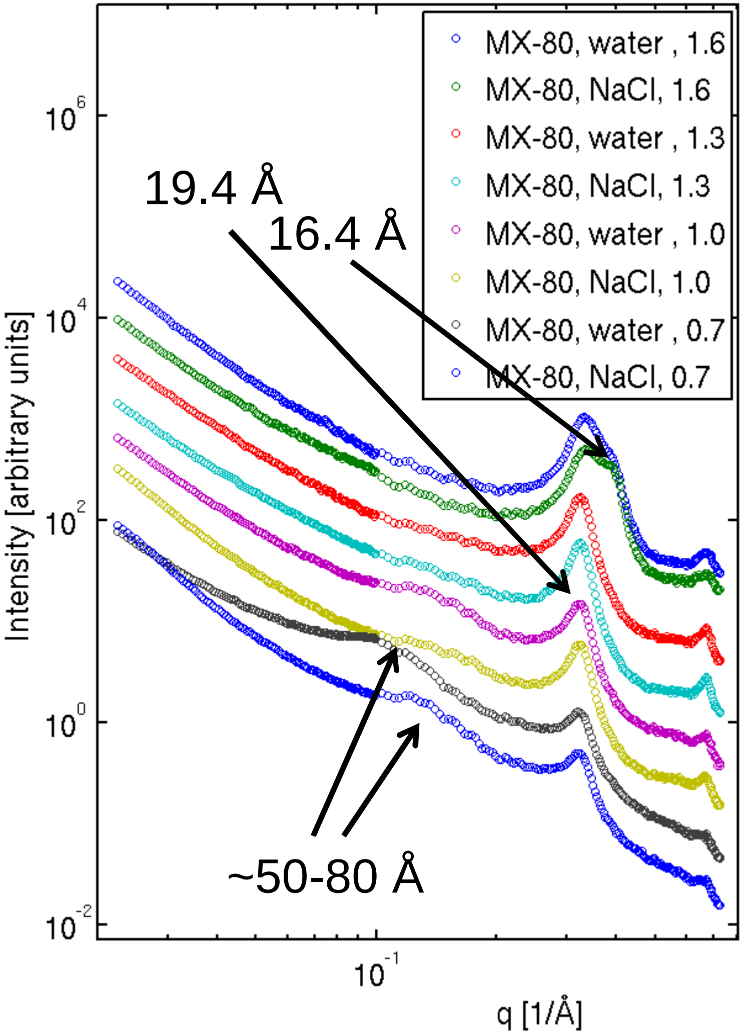
1. samples



- Best lamellar order in MX-80 sample.
- Purification seems to decrease the order within the lamellar structures.
- Ca-montmorillonite has better order than Na-montmorillonite
- Disappearance of interlamellar hydration layer is seen in Ca-mont. sample
- (?) Ca-montmorillonite has higher tendency for decrasing lamellar distances



2. samples



- Increasing moisture content decreases lamellar order (lamellar peak diminishes)
- Another feature (corresponding to ~5nm distances) evolves with increasing moisture content. This could be explained as partly exfoliated tactoids.
- NaCl solution gives better lamellar order.

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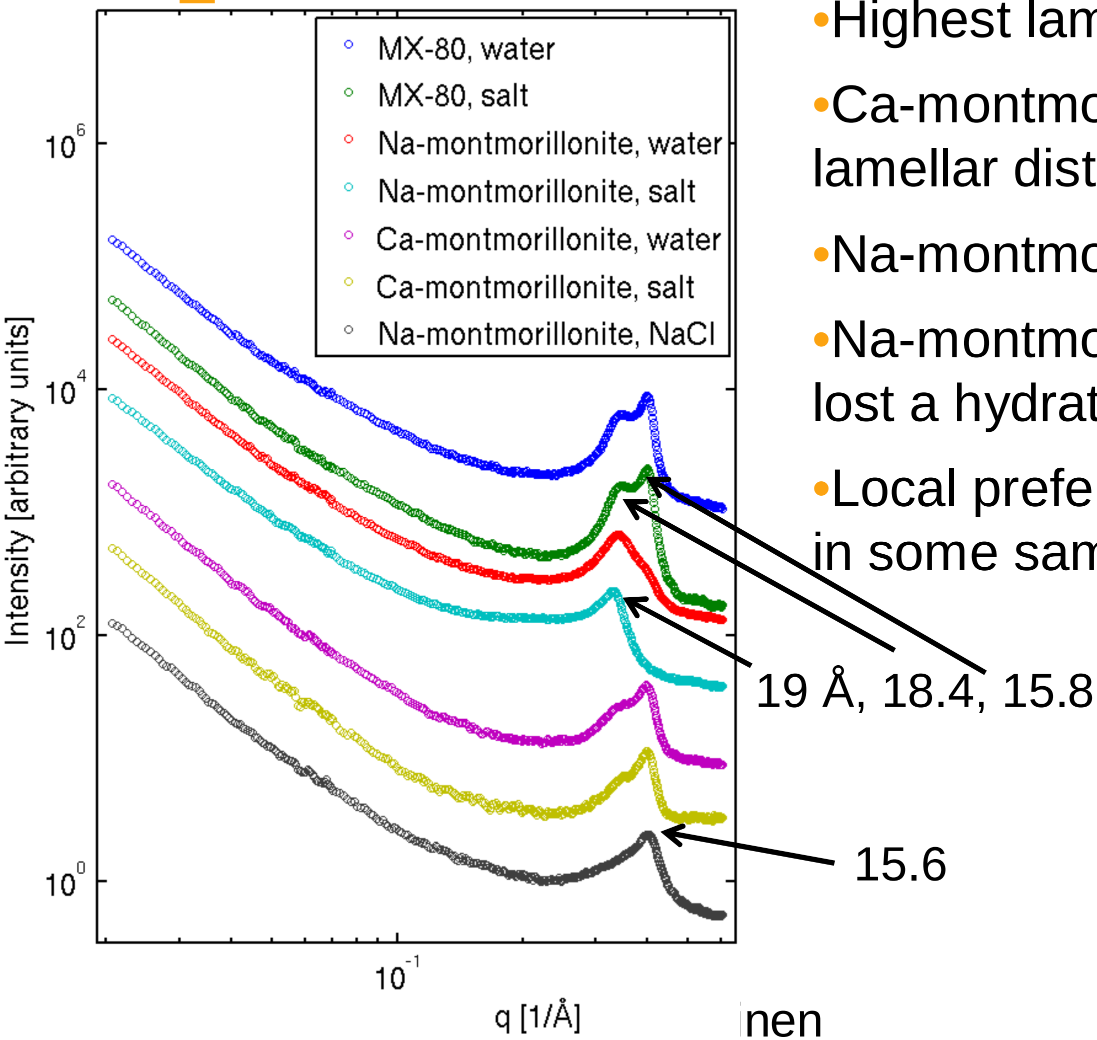
3. samples



- Highest lamellar order in MX-80 samples
- Ca-montmorillonite: tendency of decreased lamellar distances
- Na-montmorillonite: worse lamellar order
- Na-montmorillonite in NaCl-solution: have lost a hydration layer
- Local preferred orientation of tactoids found in some samples

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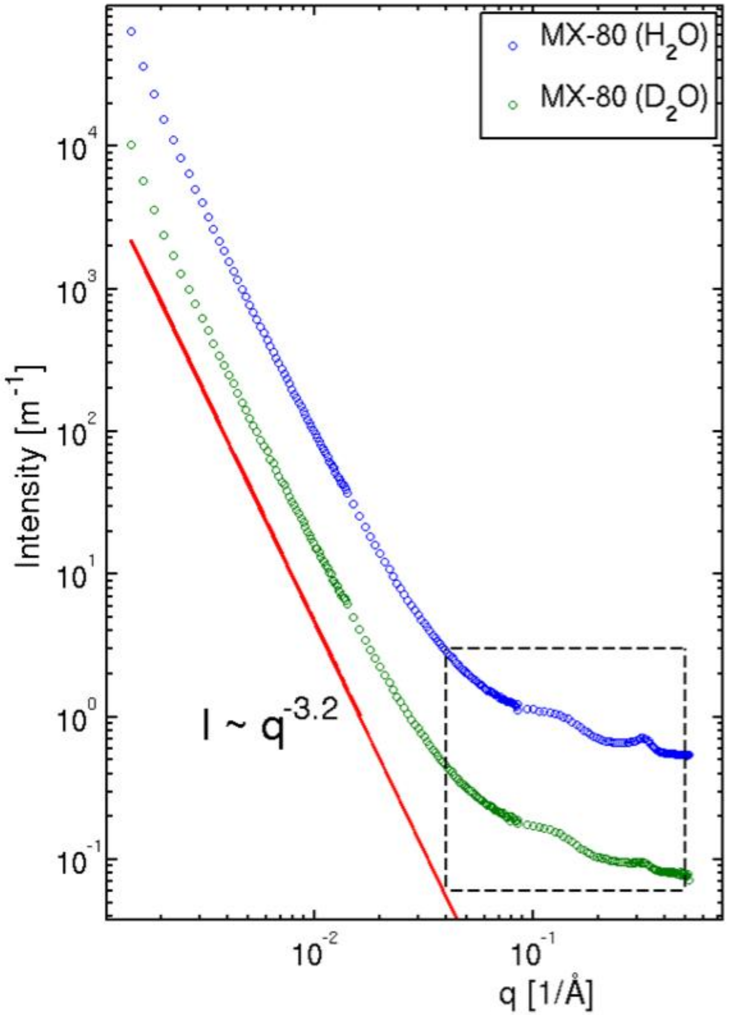
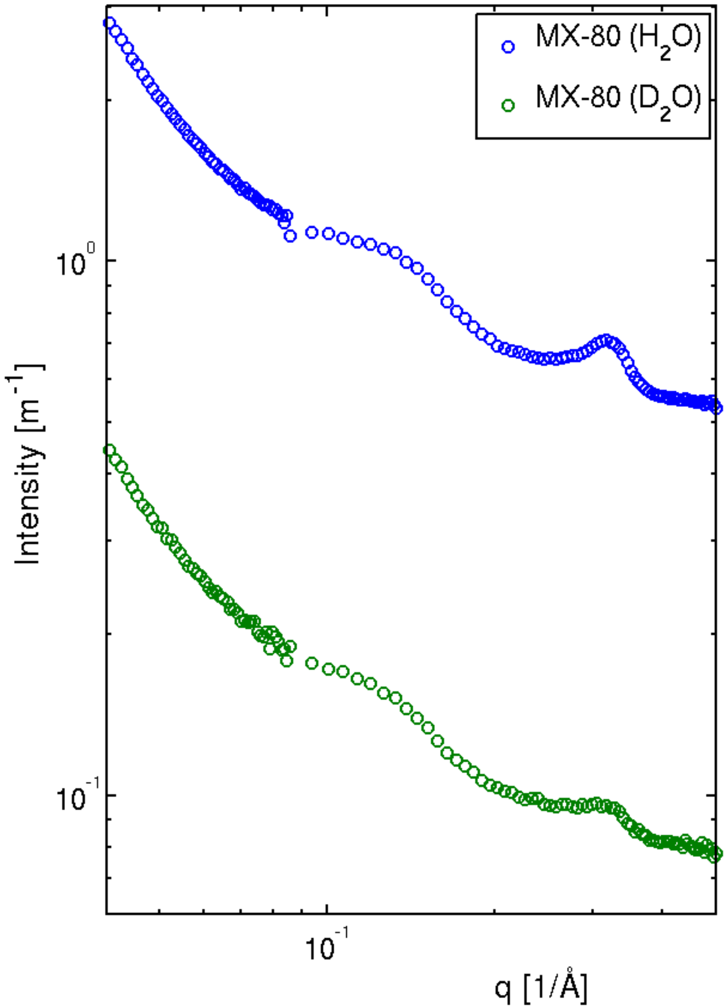
SANS

Preliminary SANS measurement worked well





Plans for this year

- Further analysis of the measured data including fitting of models.
- Evaluate possibilities of further SANS measurements
  →writing proposal for SANS measurements(?)
- Combined x-ray tomography and SAXS

Thank you for listening!