Food and Agriculture Organization of the United Nations

Determination of the clay content of soils in Brussel, Flanders and Walloon Region (Belgium) and Luxembourg

25 June 2024

Identification of clay minerals by X-ray diffraction

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Identification of clay minerals by X-ray diffraction

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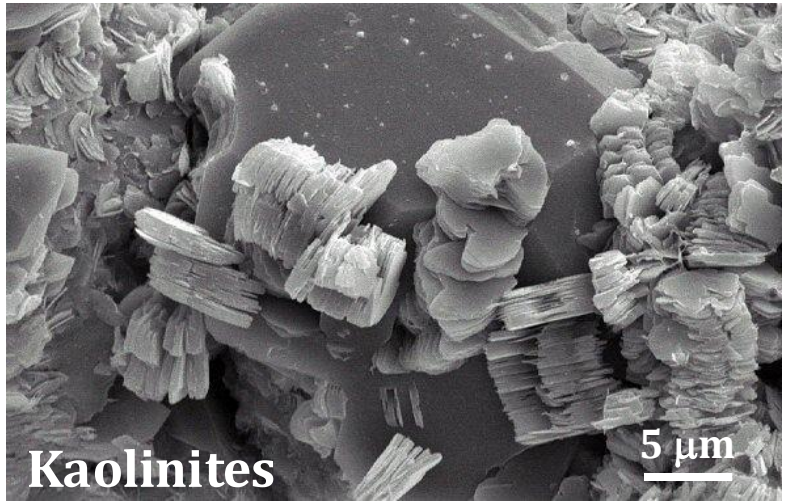
- Definition of clays minerals
- Structure
- Main clay minerals
- X-ray diffraction
- Analyse by X-ray diffraction
  - Bulk powder analysis
  - Oriented mounts from fraction < 2 μm
  - Some examples

Clay minerals



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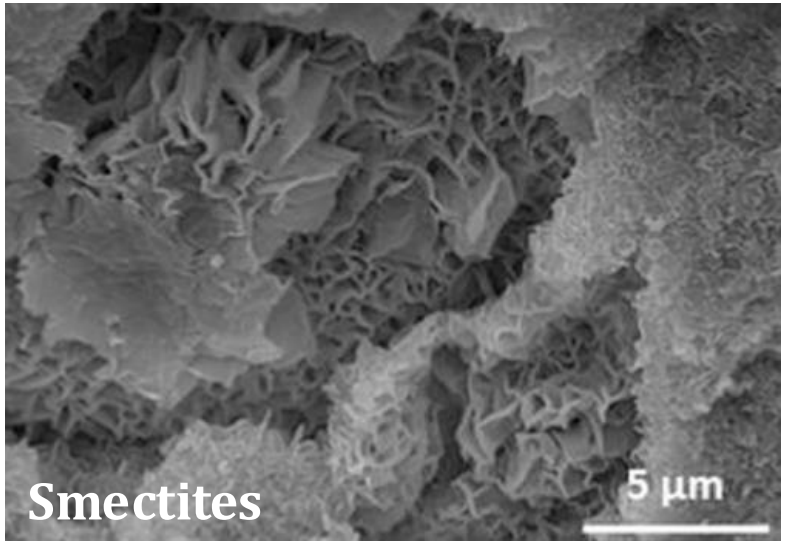
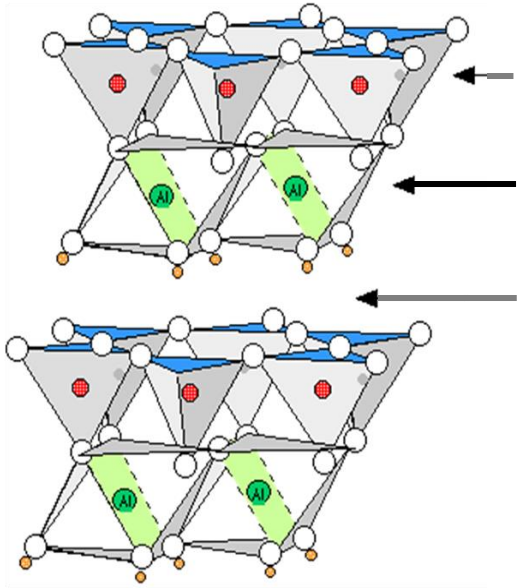


© MEB. Westerwald, Allemagne. Fontaine et al. 2020. Applied Clay Science 187, 105444.

- Minerals of small size (µm)
- Abundant in soils & sediments
- Sheet crystal structure
  - ↳ Specific method of study = X-ray Diffraction
- Numerous applications

Structure of clay minerals

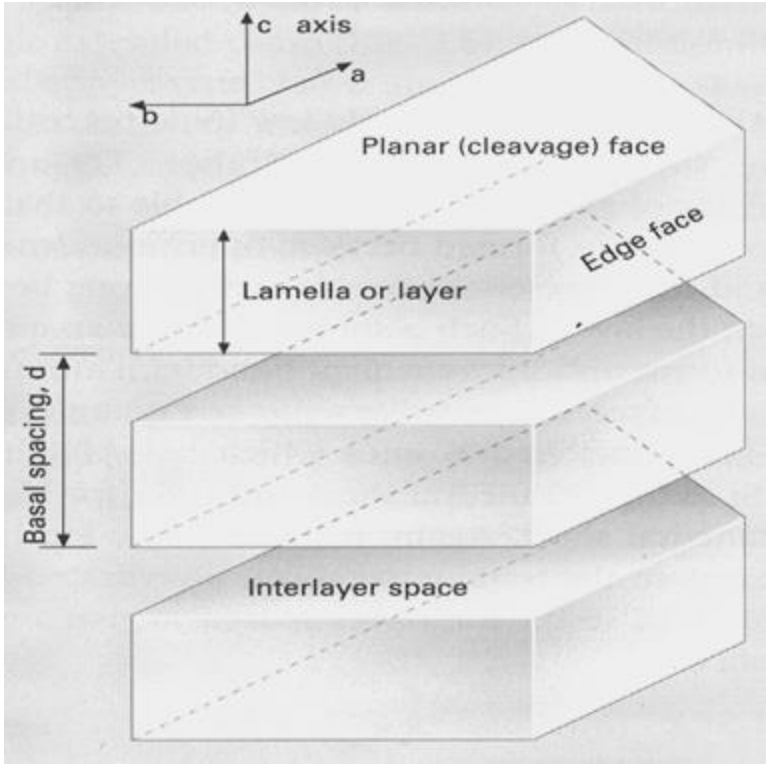


Tetrahedra of silica

Octahedra of aluminium

Interlayer space

Basal spacing



source: White, 1999

Classification AIPEA

1. Tetrahedral-octahedral sheet combination (layer 1:1, 2:1)
2. Cation of octahedral sheet (2+, 3+)
3. Layer charge
4. Interlayer material

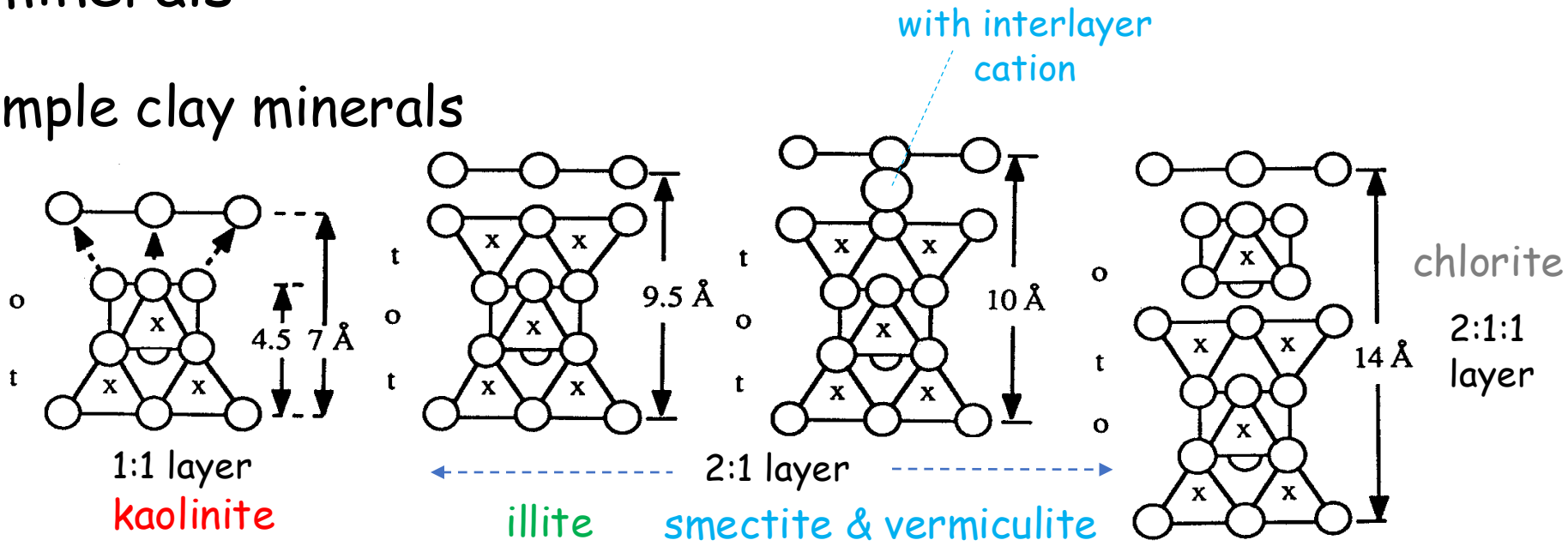
+ type of layer stacking, chemical composition,…

https://aipea.org/

source: Eslinger & Peaver, 1988

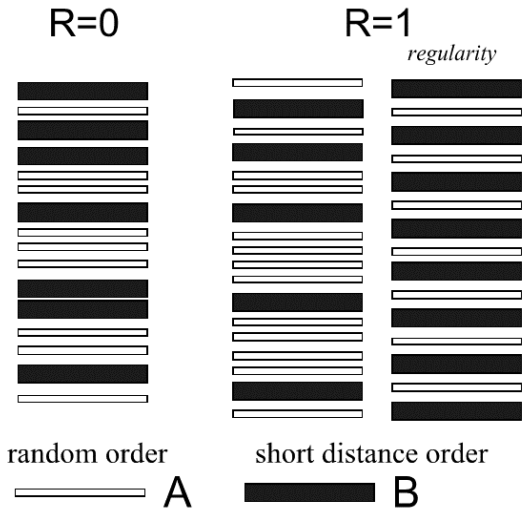
Main clay minerals

A. Simple clay minerals



source: Moore & Reynolds, 1989

B. Random or regular mixed-layers



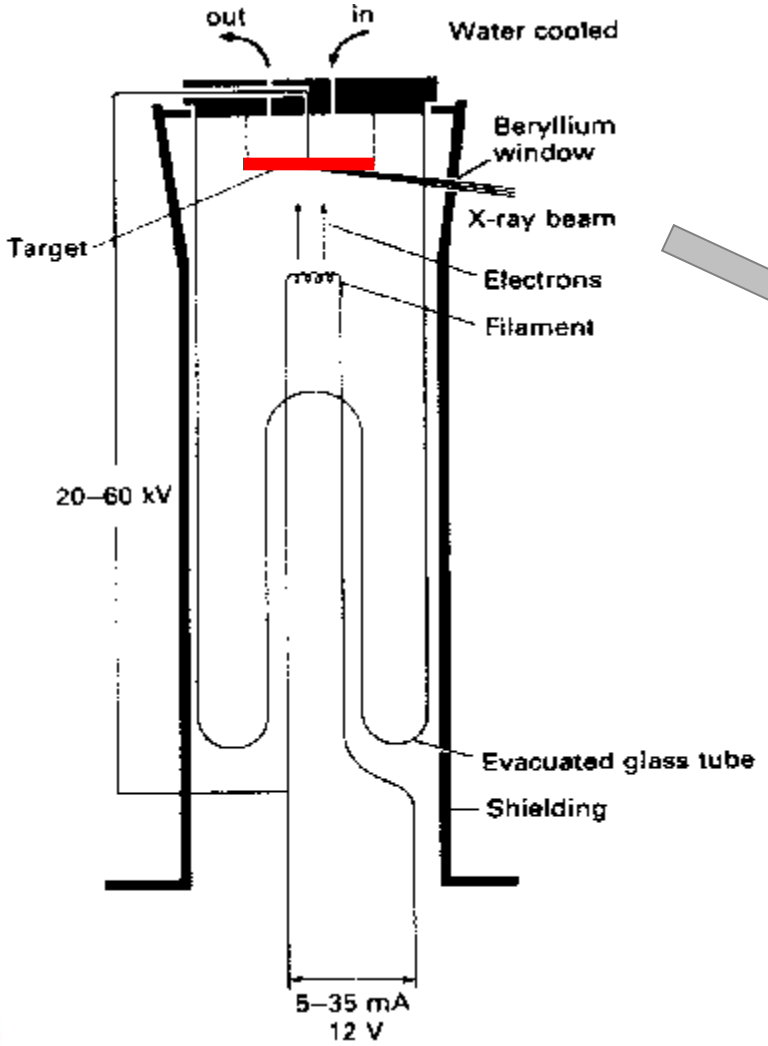
source: Meunier (2006). Clay minerals.

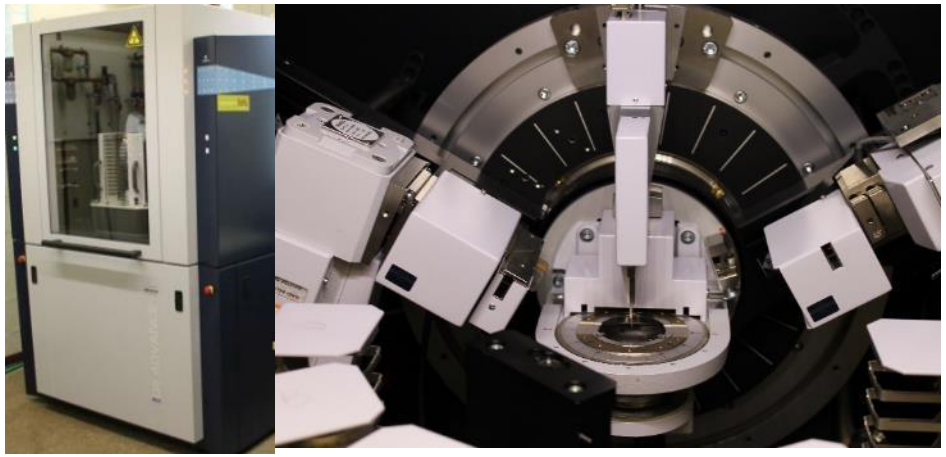
Identification f(basal spacing)

↪ Analyse by X-ray diffraction

X-ray production

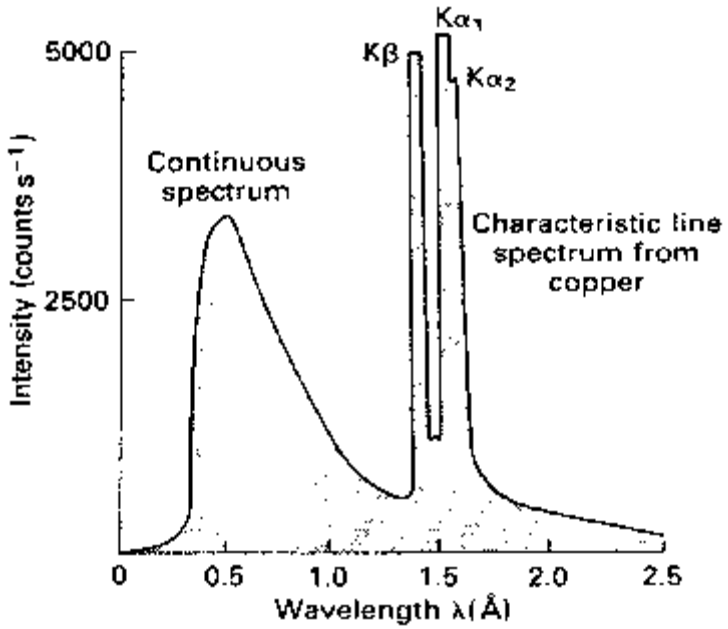
X-ray tube





source: J. Otten - D8 Eco Bruker, AGEs, ULiege

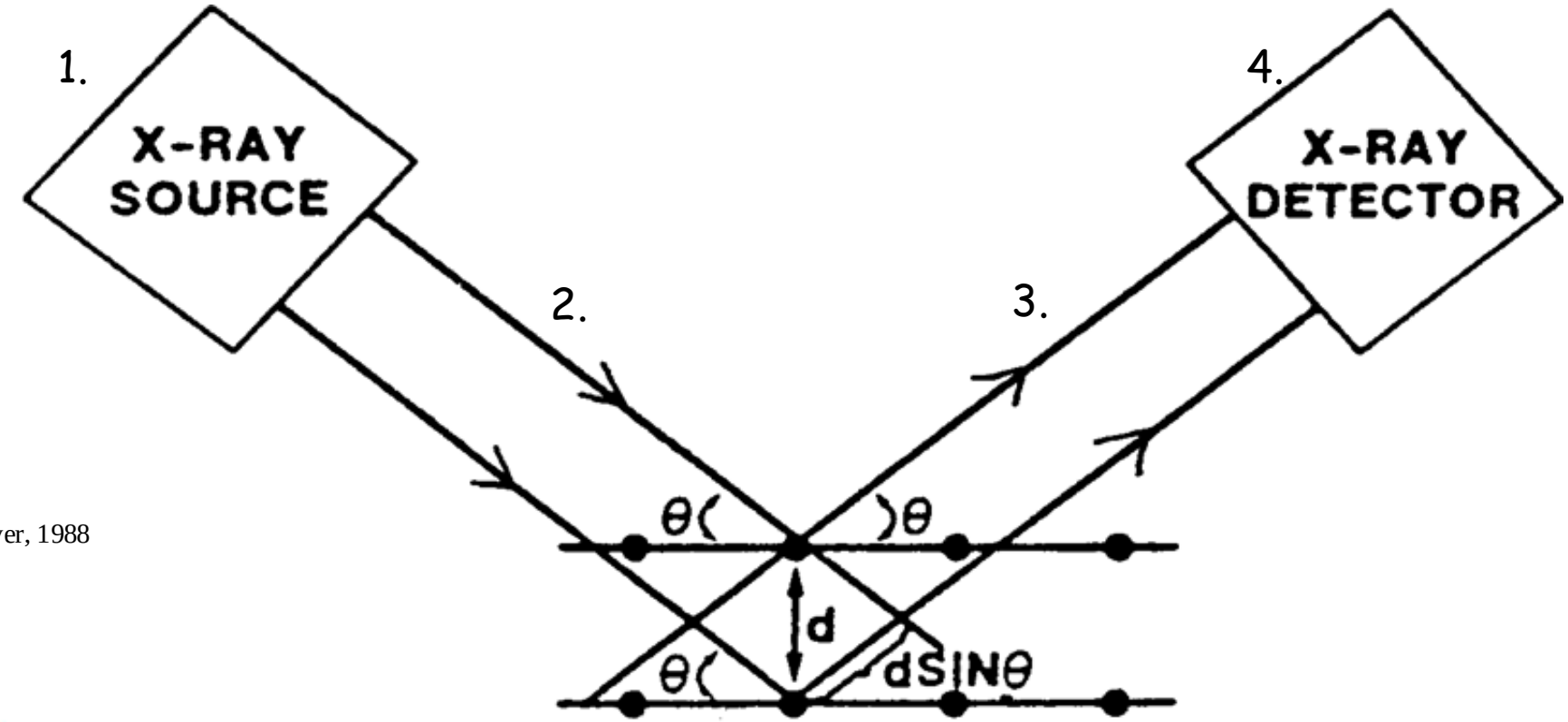
Spectre of Cu anode



source: Tucker, 1991

X-ray diffraction

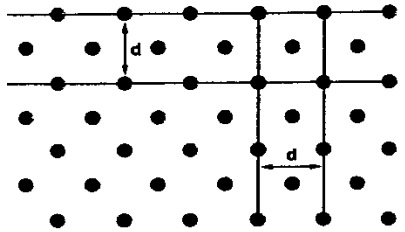
1. Production of X-rays
2. Interaction with cristalline structure
3. Diffraction with same angle
4. Detection of X-rays



source: Eslinger & Peaver, 1988

Bragg law

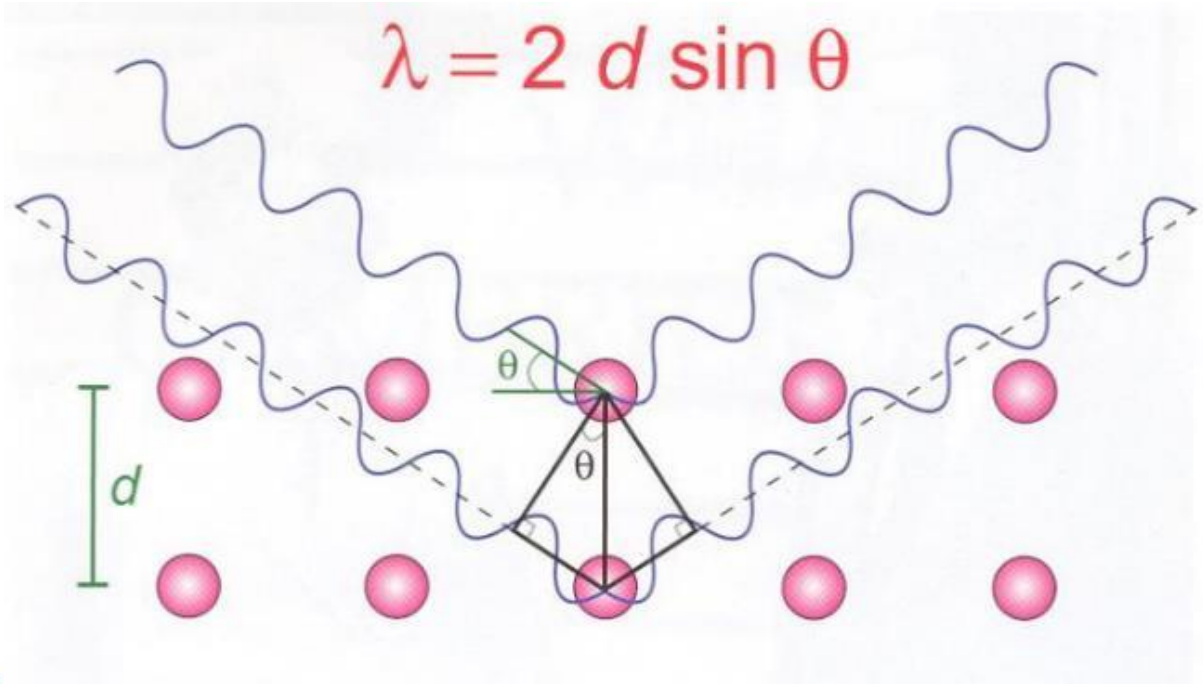
Two-dimensional interatomic spacings



$n\lambda = 2d\sin\theta$, where

$K_{\alpha 1}Cu = 1{,}54$ Å

- n = whole number
- λ = f(anode)
- θ = diffraction angle
- d = basal spacing (Å)



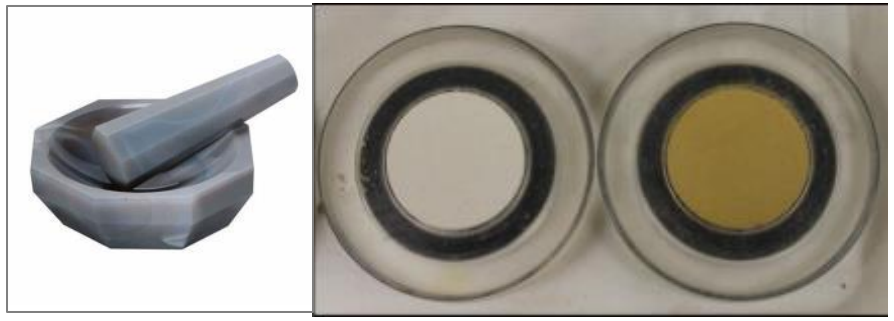
Reflections of X-ray waves from successive parallel planes of atoms generate sufficient diffraction intensities when interferences of reflected waves are constructive

Source: Heaney, Elements 2(2), 69-70, 2005

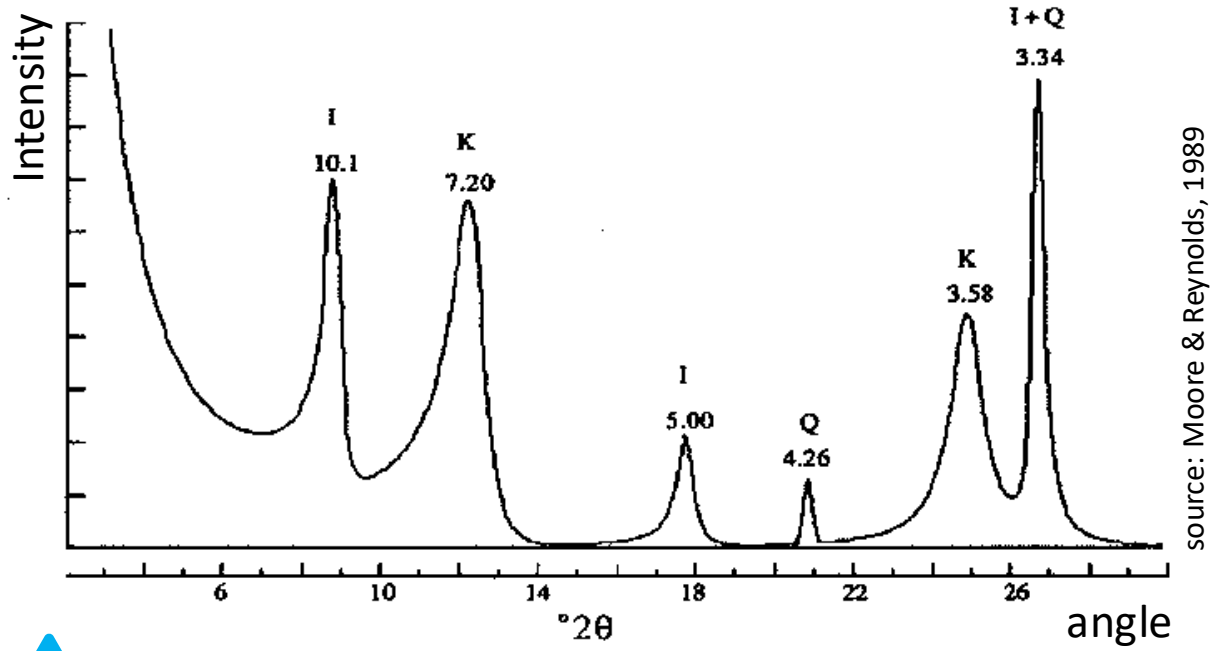
XRD on bulk powder

Sample preparation
- Drying at 40°C
- Manual griding in agate mortar
- Preparation of pressed powder

(Back-side method - Moore & Reynolds, 1989)



© PhD Elisa Pleuger 2020



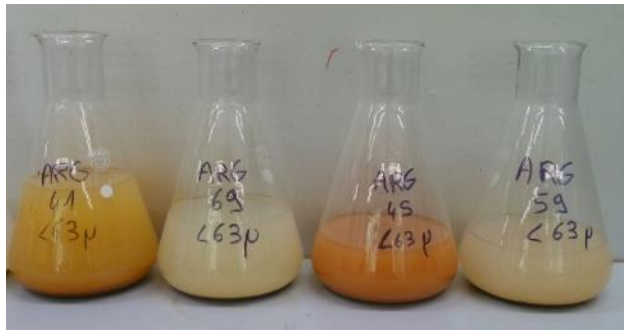
source: Moore & Reynolds, 1989

ID of clay minerals

association of several minerals in soils = possible superposition

comparison of several patterns after specific treatments

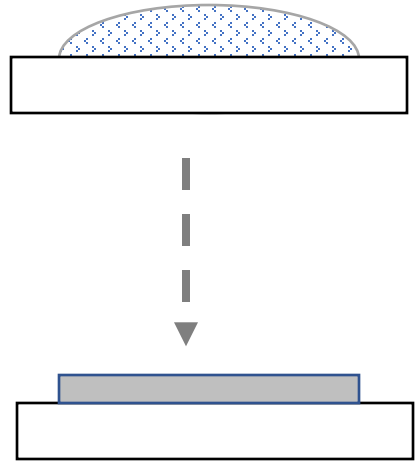
XRD on oriented mounts



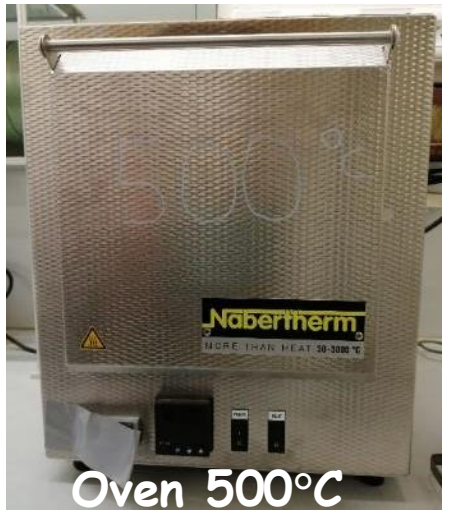
Sample preparation
- Suspension in water
- Sieving at 63 µm
- Decarbonation & rising

- Decantation during 50 min
- Extraction of upper cm
- Sedimentation on glass slide

© E. Pleuger 2020



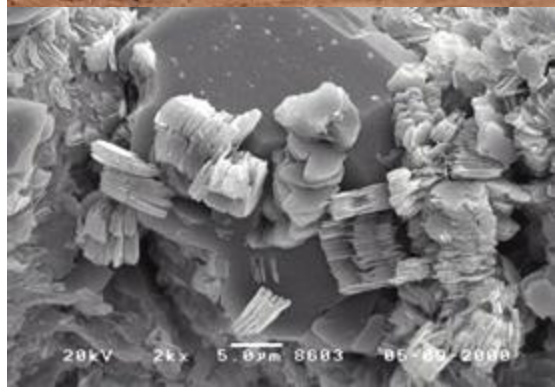
3 XRD analyses
(1) Air-dried
(2) EG saturation
(3) 500°C heating



Examples of XRD on natural soil samples

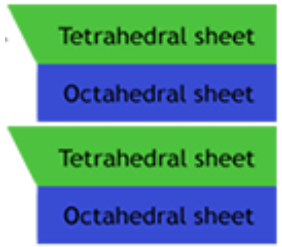
Laterites of Kamboinse (1)

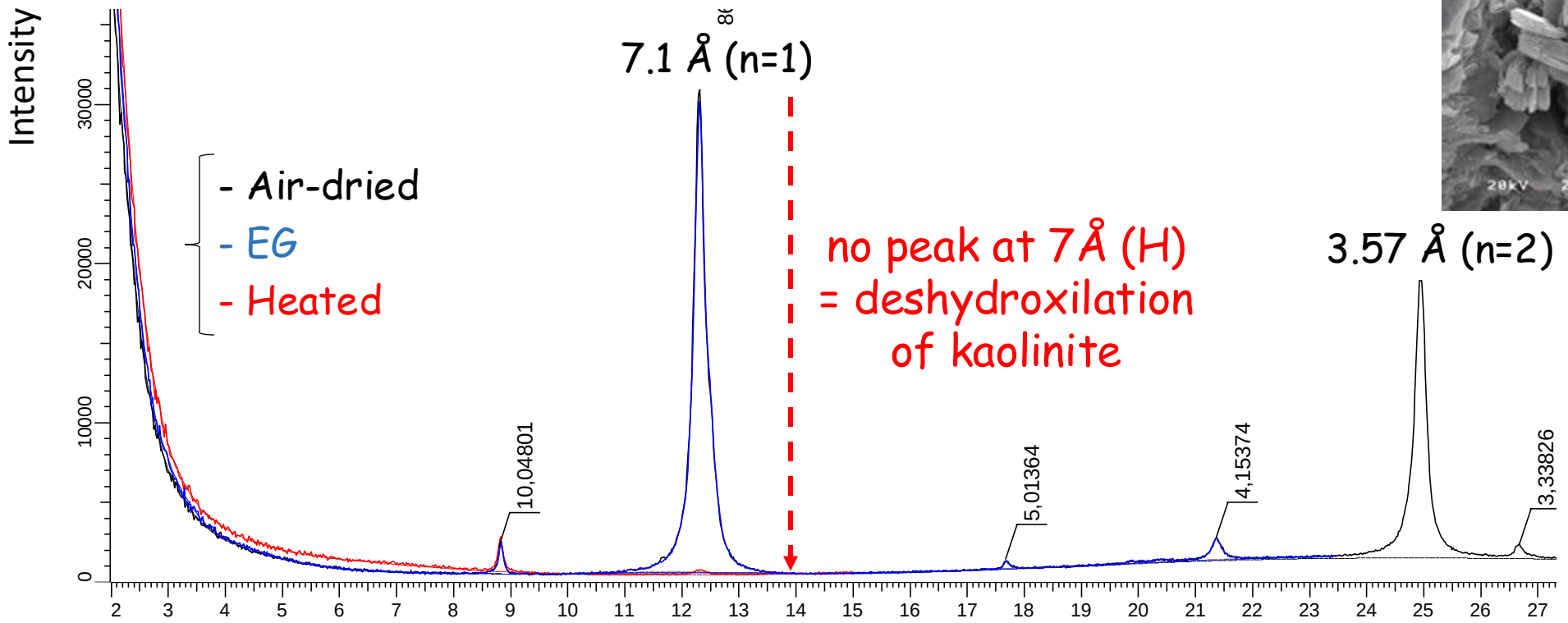
Burkina Faso



Clay < 2 µm fraction

Hydrolysing climate
Complete leaching of cations
= dominance of kaolinite



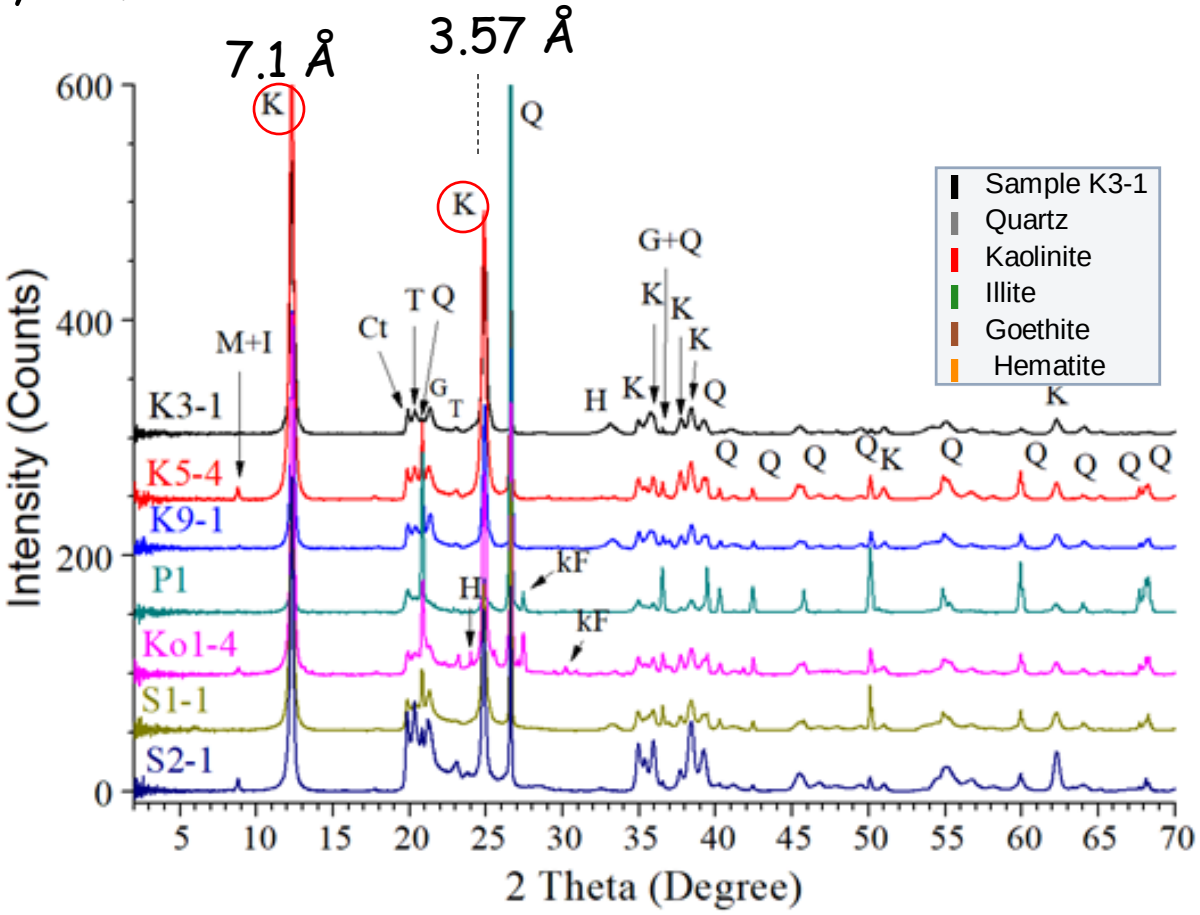


source : PhD. P. Nshimiyimana (2020)

Laterites of Kamboinse (2)

Bulk sample K3-1

- Kaolinite + illite/muscovite
- Quartz
- Goethite, hematite

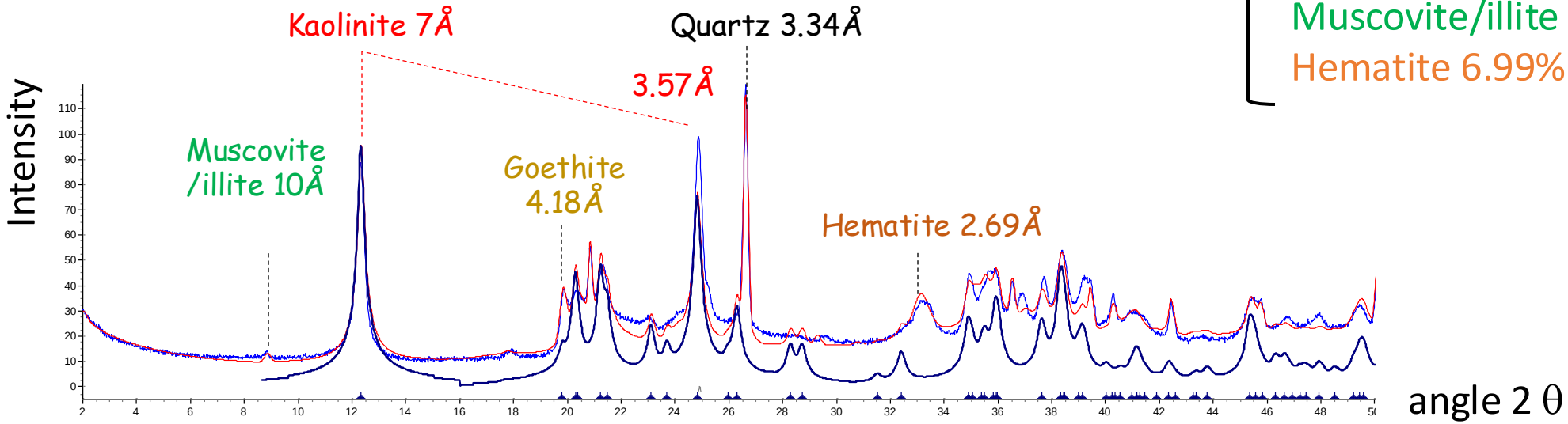


source : PhD. P. Nshimiyimana (2020)

Quantitative interpretation (3)

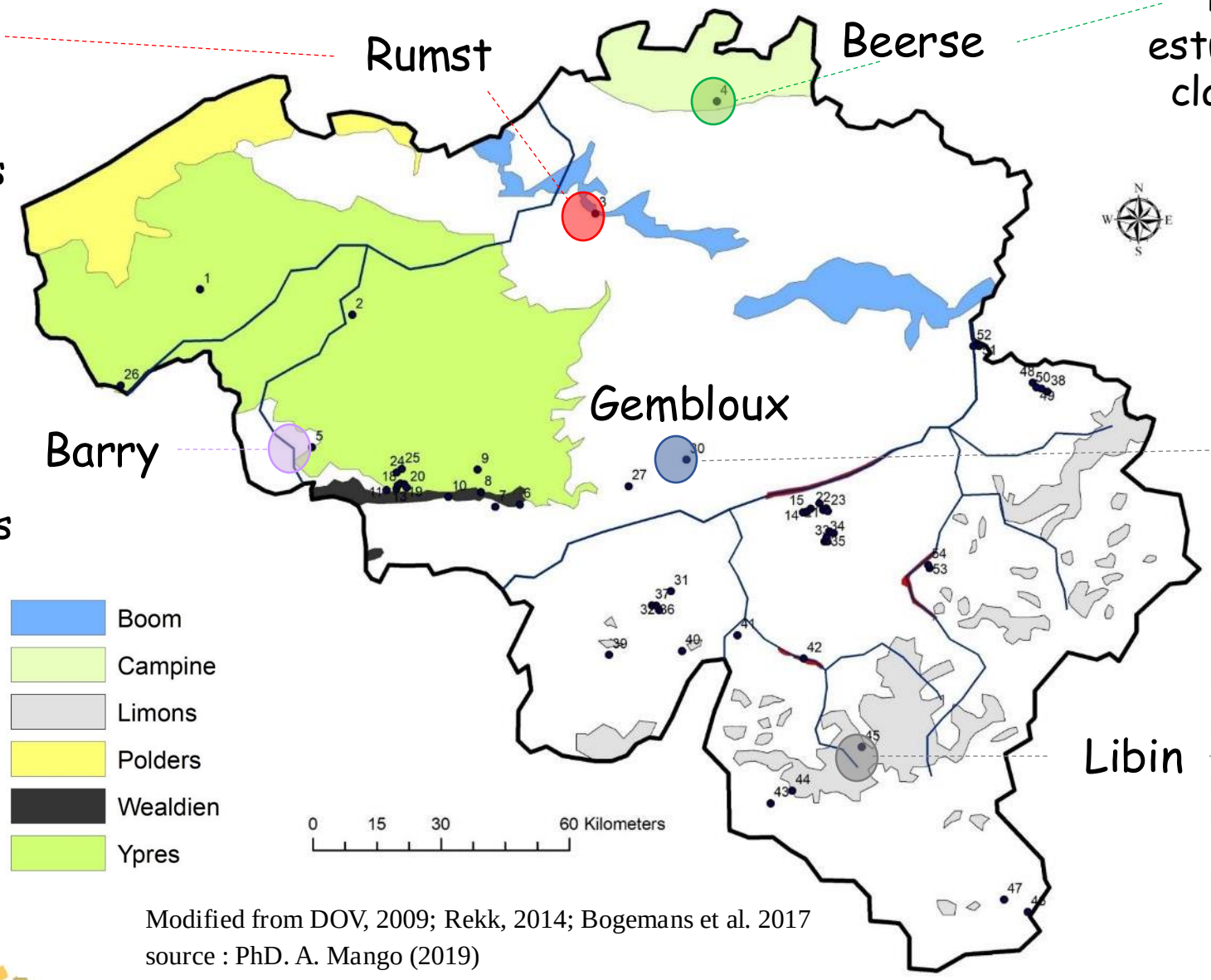
- Mineral identification using EVA ®Bruker software
- Rietveld refinement using TOPAS ®Bruker software
- Preferred orientations & unit cell parameters adjusted progressively to obtain a reconstructed pattern as close as possible to the measured XRD

Kaolinite 53.10%
Quartz 19.51%
Goethite 11.64%
Muscovite/illite 8.77%
Hematite 6.99%



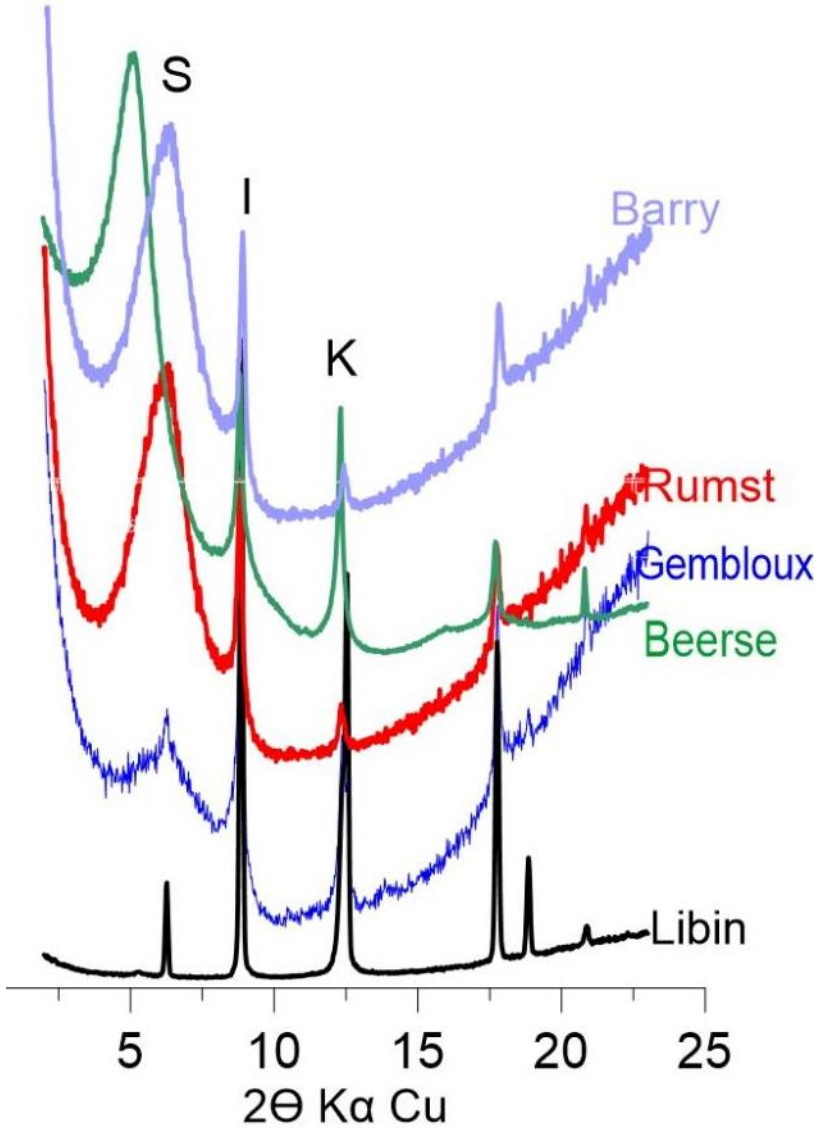
Topas-derived reconstructed profile (red curve) with the raw XRD profile (blue curve)

Clayey formations in Belgium (1)

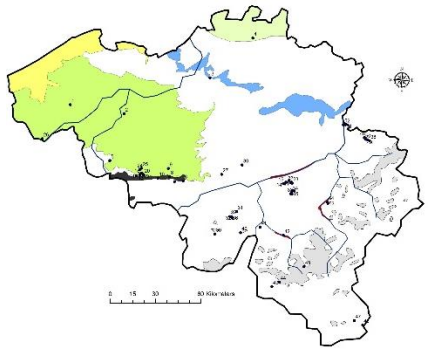


Modified from DOV, 2009; Rekk, 2014; Bogemans et al. 2017
source : PhD. A. Mango (2019)

Clayey formations in Belgium (2)



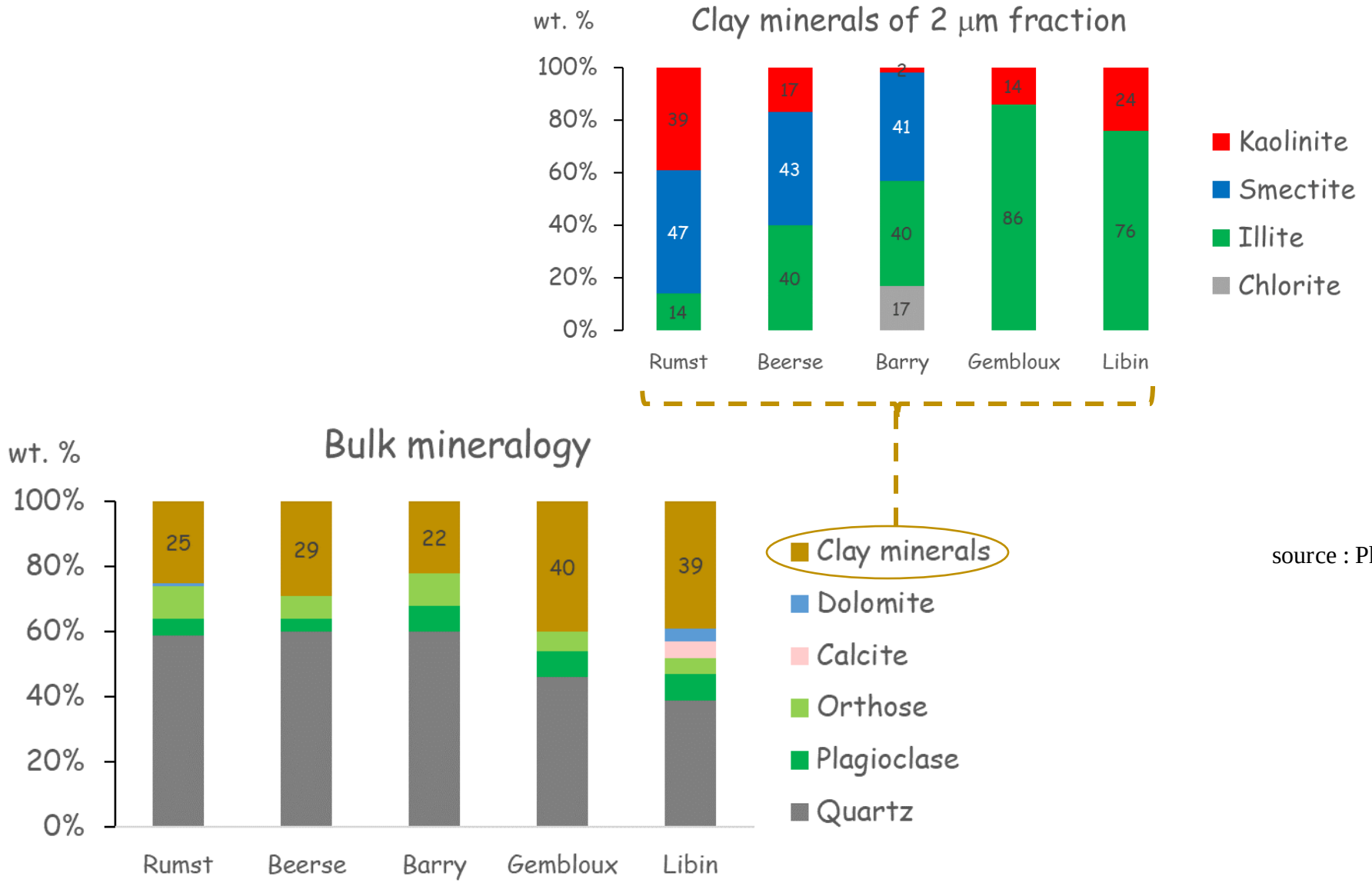
Clay < 2 μm fraction
EG patterns



source : PhD. A. Mango (2019)

- Rumst, Barry, Beerse = Smectite-rich clays — Cenozoic estuarine & marine clayey deposits
- Libin = Kaol. + Illite
- Gembloux =Illite + Kaol. +Sm. — Weathering Paleozoic shales

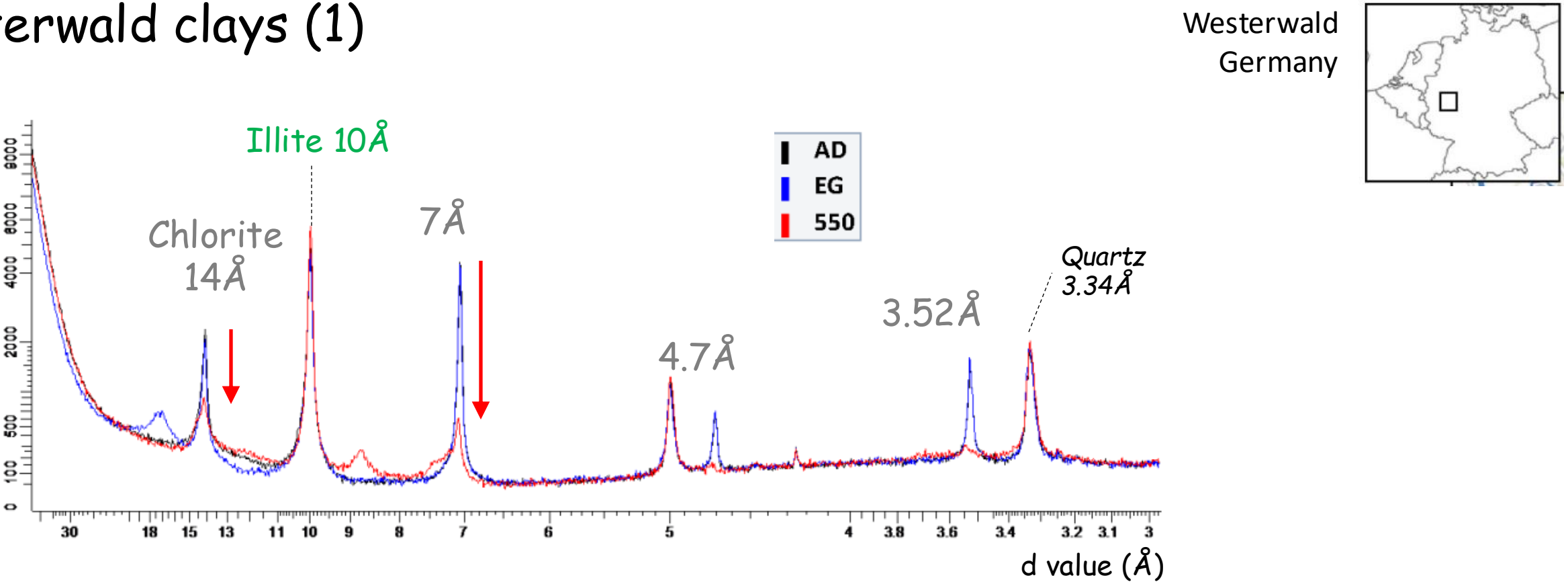
Clayey formations in Belgium (3)



source : PhD. A. Mango (2019)

Westerwald clays (1)

Westerwald
Germany

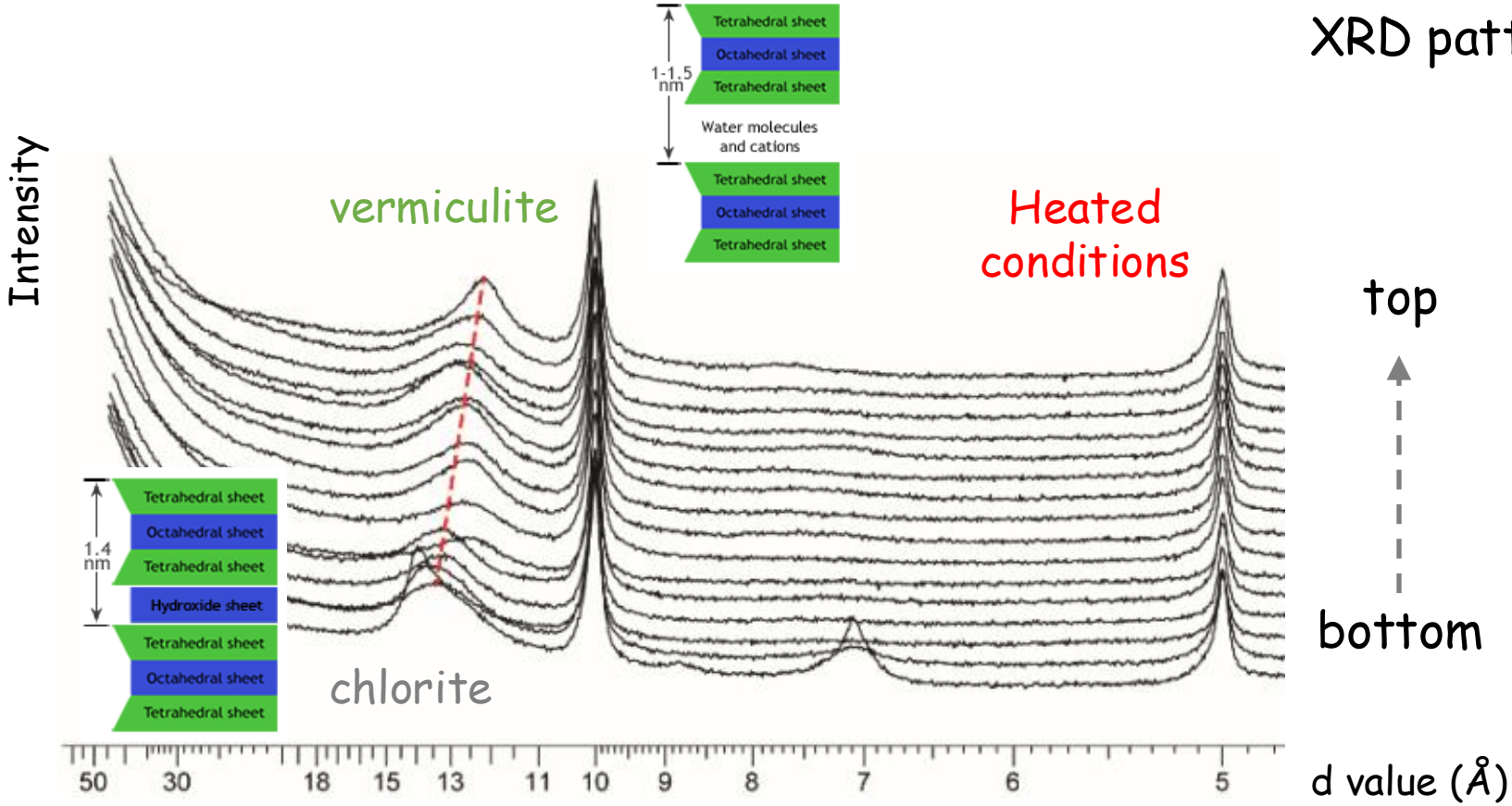


Parental rock = Devonian fresh slate
XRD on clay < 2 μm fraction

Chlorite + Illite = primary mineral

But chlorite sensitive to heating
= gradual transformation into vermiculite

Westerwald clays (2)



XRD pattern on < 2 μm fraction after heating

top

bottom

weathering profile on Paleozoic slates

Progressive transformation of chlorite into vermiculite through the profile

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Thank you

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