FULLER EARTH CHARACTERIZATION

SKIN DECONTAMINATION

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FULLER'S EARTH

Fig. 1

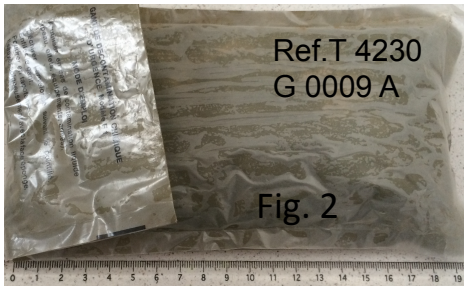


Fig. 2


Dry powder

Weight 125 g± 10 g
21 g dry powder / glove


Fig. 3

- FE an aluminium silicate , $Al_2O_3,SiO_2,2H_2O$, adsorbant and bleaching.
- Reference for skin decontamination both militarian and civilian CWA –TICS
- Generic name
- Decontamination Efficiency (DE) (%) after 15 min contamination , water (54%) ,dry FE (71%) and reaches 88% FE + Water (suspension) published in JAT 2017

Comparison of four different fuller's earth formulations in skin decontamination
A Roul et al JAT 2017

AIM OF THE STUDY
BIOEQUIVALENCE OF FULLER EARTH

I. FE furnished by NBC-SYS is characterized
II. Classical methods: density , Water holding capacity , …
III. Size particle analysis of the « as delivered » powder

Laser diffraction . Histogramm size class (volume converted in number)

IV. Composition analysis by **SEM**

X-Ray atomic composition

Mapping

III. Morphologic structure dry FE observed

SEM / TEM

III. Phase analysis

X-ray diffraction (XRD) analysis of fuller earth powder

Synchrotron beamline (CRISTAL)

We should compare this FE with other FE of different origins (country, natural, synthhetic) for their **bioequivalence .**

CHARACTERIZATION- BIOEQUIVALENCE

BY DESIGN

- Micrometrics criteria
  - Size
  - Porosity
  - Shape
  - Specific gravity
  - Specific surface area
- Physico chemical properties
- Structure
- Phase analysis

BY TESTING

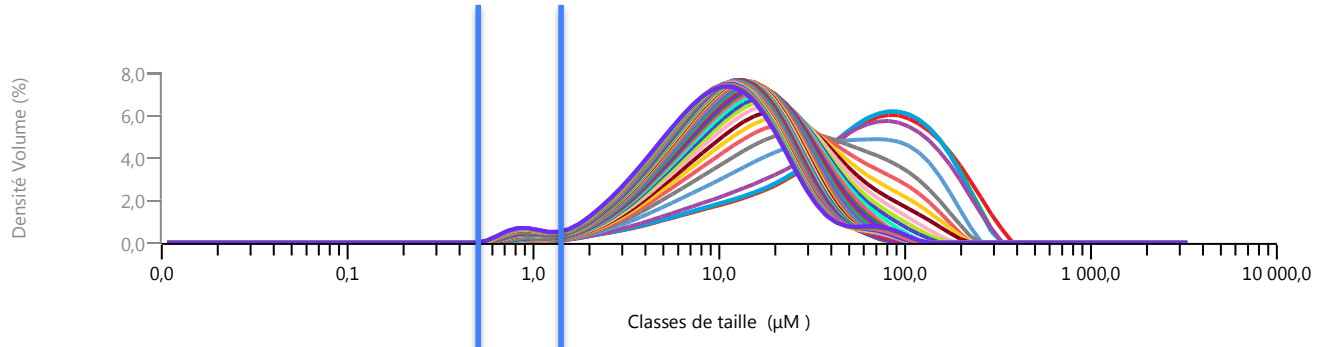
- Testing the bioequivalence of other samples of adsorptive powders compared to the next one
- Test in vitro Decontamination Efficiency (DE)
- Tests ex vivo DE

To obtain Fuller earth (FE) standardized decontamination agent

LASER DIFFRACTION Water dispersed sample
Mastersizer 3000 , Hydro MV

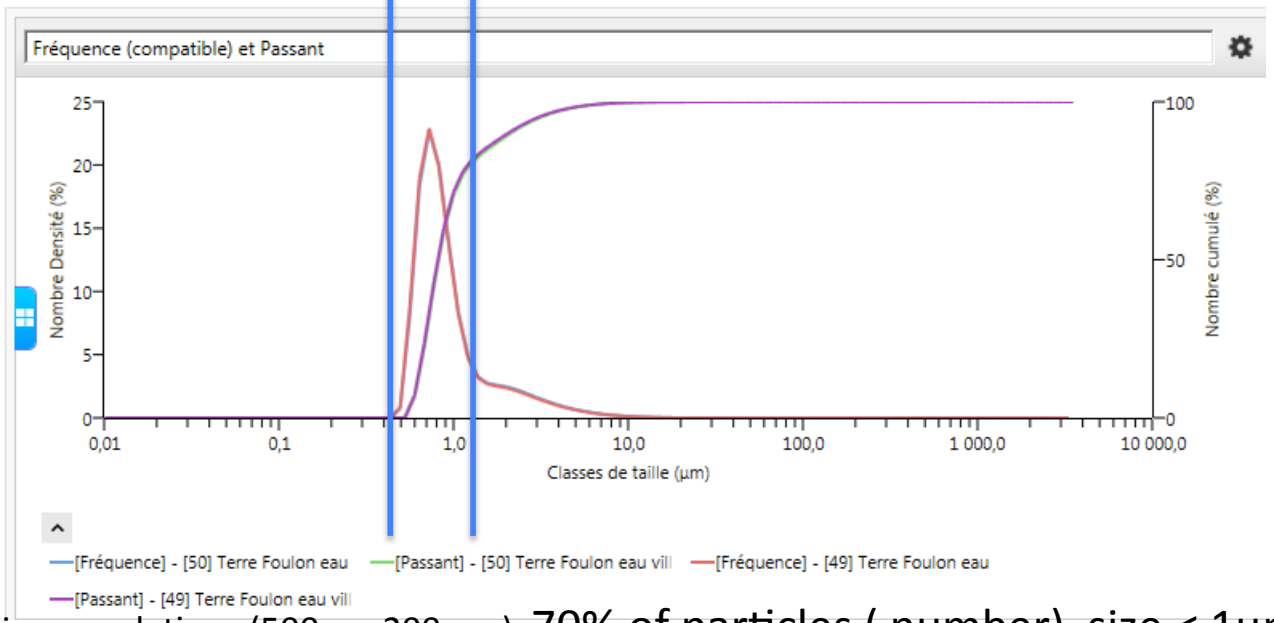
Particle size weighed distribution

Volume density (%)



Converted

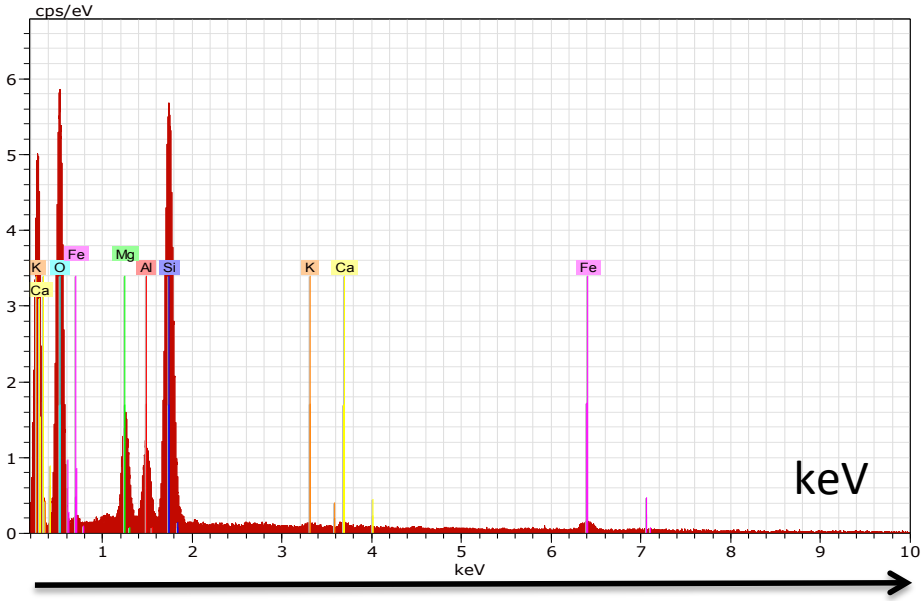
Number density (%)

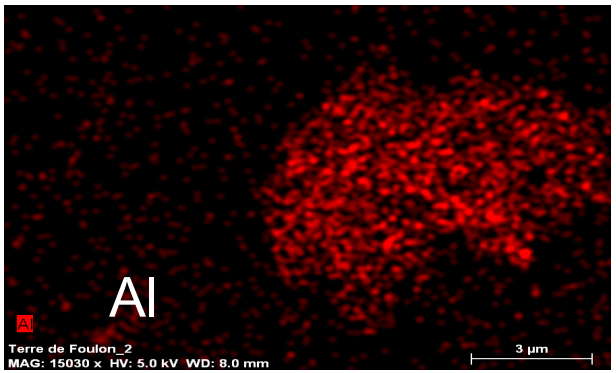


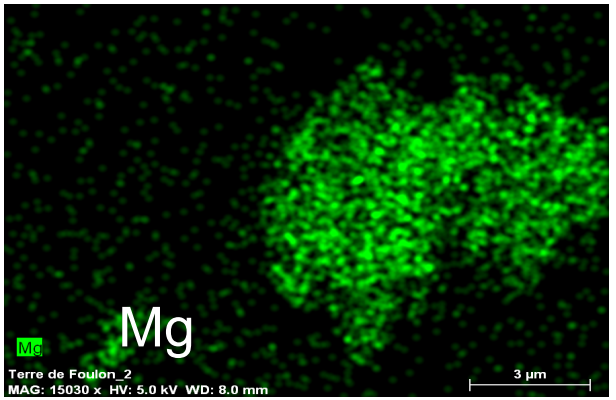
2 particles size populations (500nm-200 µm) 70% of particles (number) size < 1µm

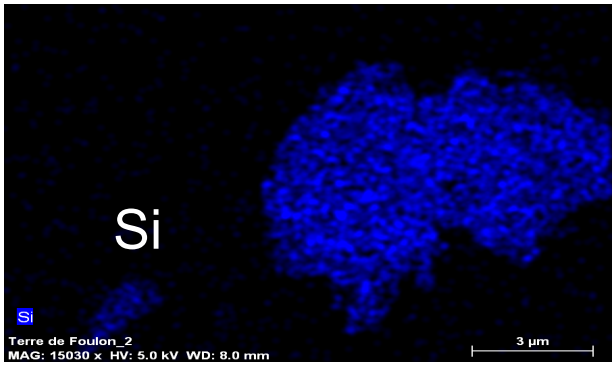
Range size 0,01-2200 µm –Mastersizer 3000

ATOMIC COMPOSITION and MAPPING by SEM









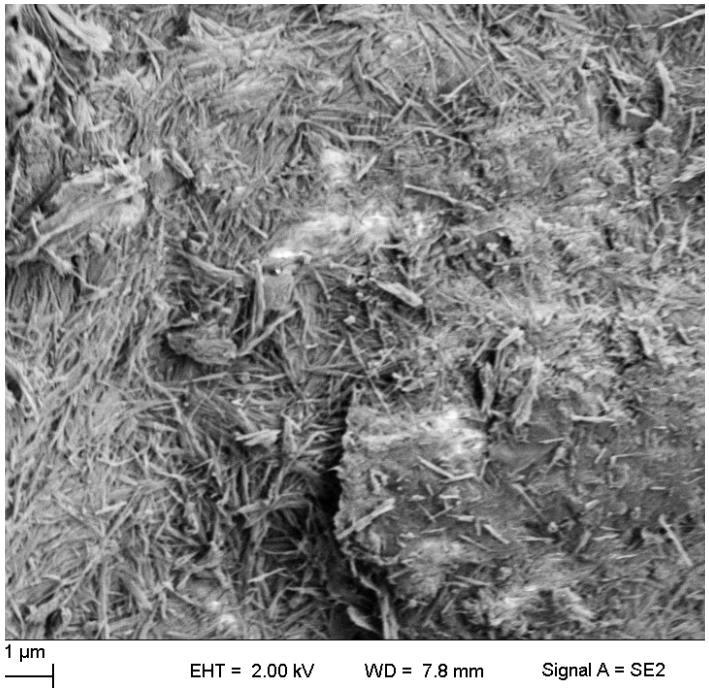
Field-emission "gun" microscope (FE-SEM) operates at 0.02 - 30 keV. High-resolution observations were obtained by using an Energy Dispersive X-ray (EDX) detector

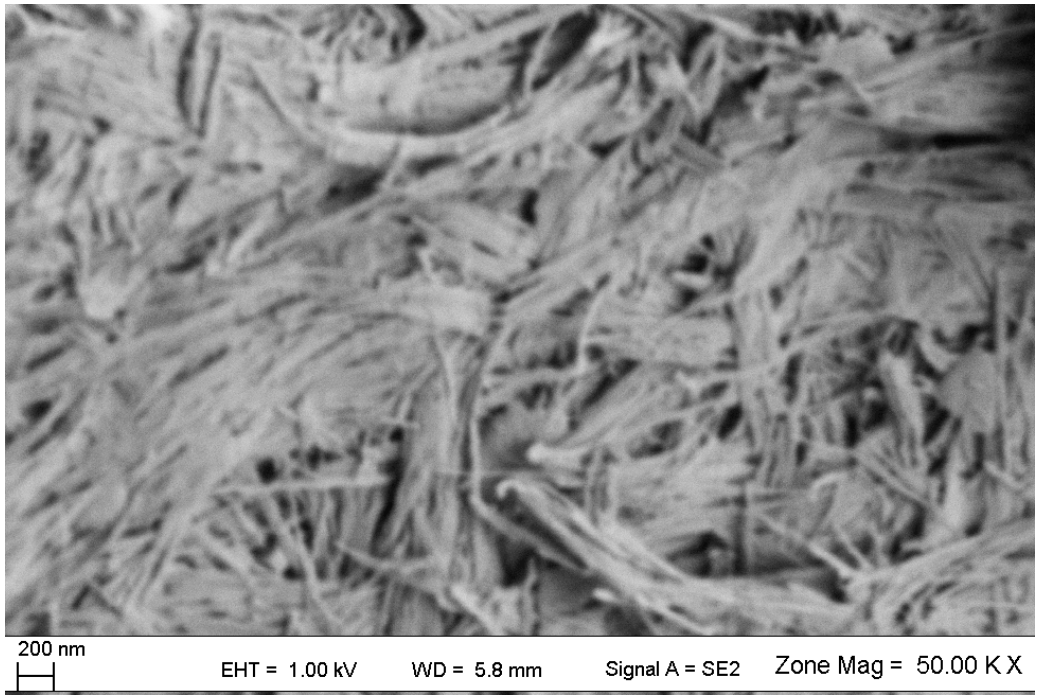
Characteristic ray in energy for each element of the structure –Quantification(A%)

Zeiss SUPRA55-VP SEM

MORPHOLOGIC ULTRASTRUCTURE SEM

Field-emission “gun” microscope (FE-SEM). High-resolution observations were obtained by using one secondary electron detector (Everhart-Thorley S2)

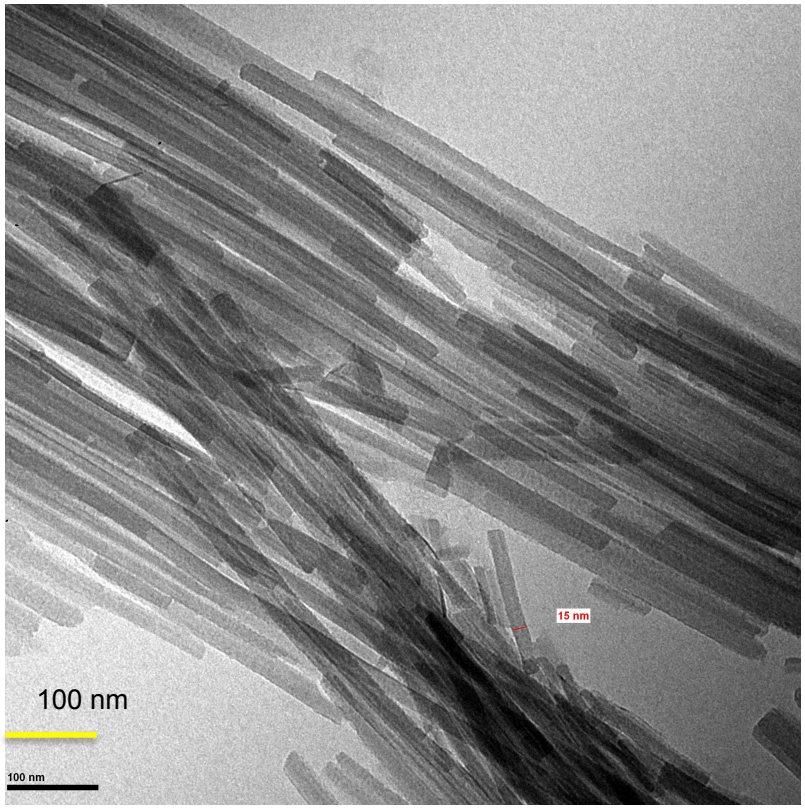




Fibrous clays – long fibers lenght > 200 nm , and diameter < 20 nm organized in rods

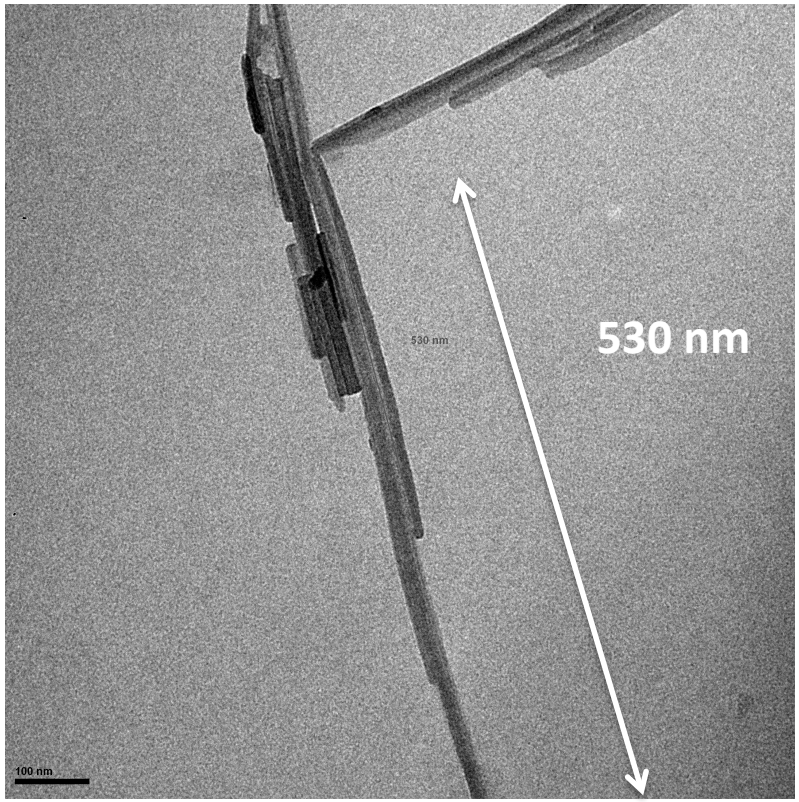
MORPHOLOGIC ULTRASTRUCTURE TEM

FE –W suspension - depot 5 µL on a specific grid coated Formvar film



Rods organized in bundles water

⦸≈ 15 nm



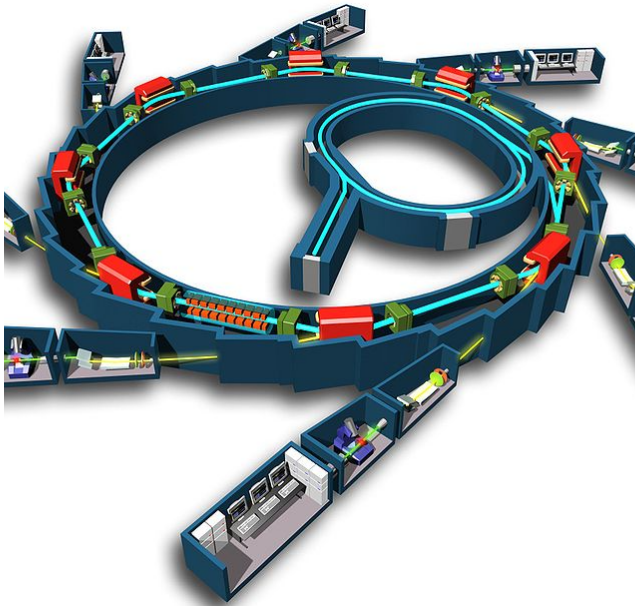
Rods organized in bundles water

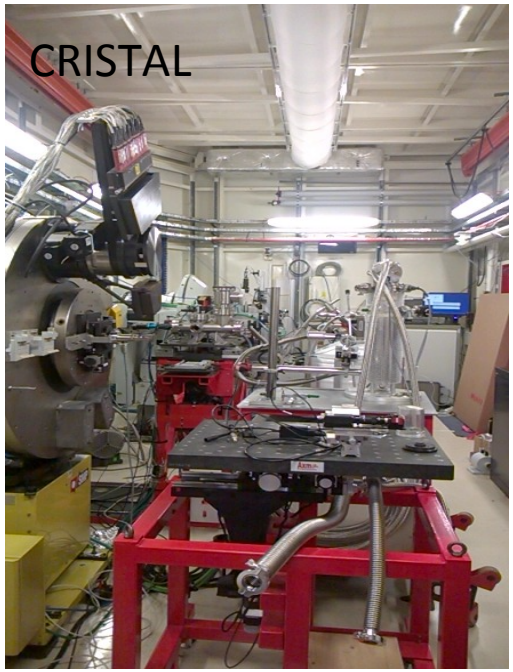
various lenght 100- 700 nm

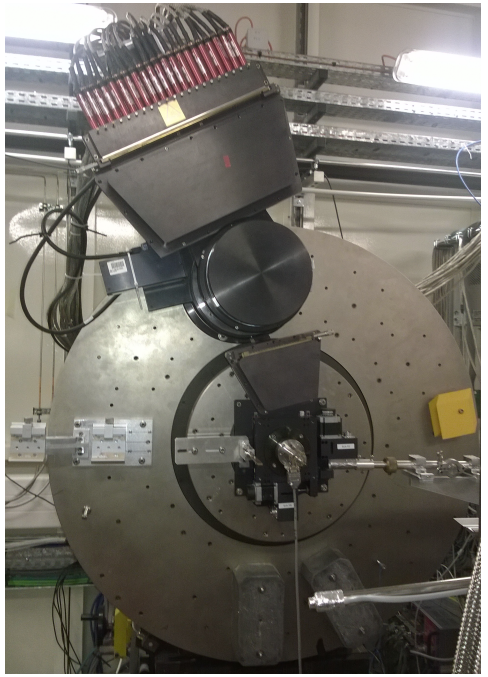
Particles are not sperical, but cylindrical

SYNCHROTRON ANALYSIS – HARD X RAYS

SYNCHROTRON radiation light is emitted by relativistic electrons of very high energy 2. 75 GeV .Each beam line has it's appropriate optic to select monochromatic beams in its energy range.



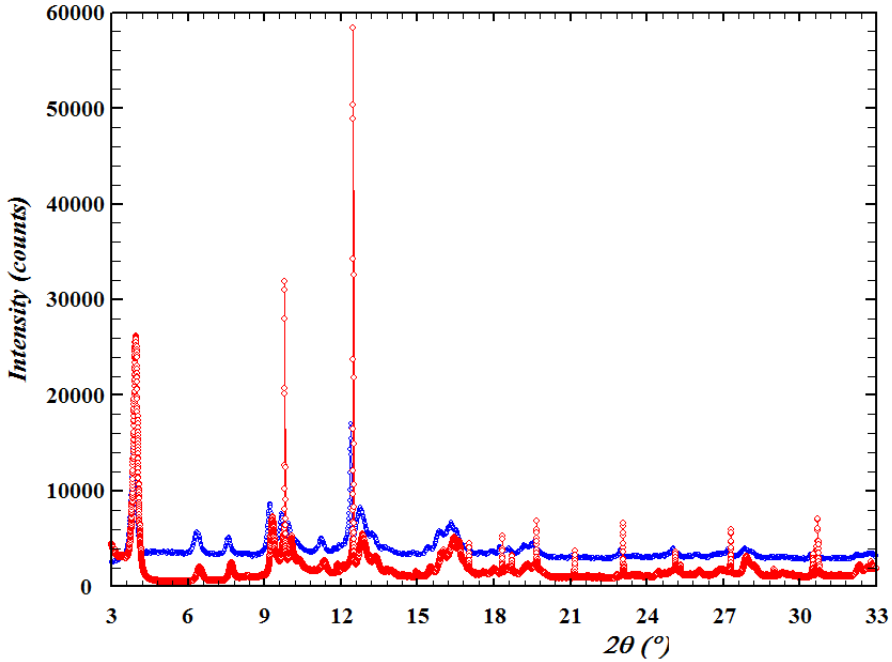


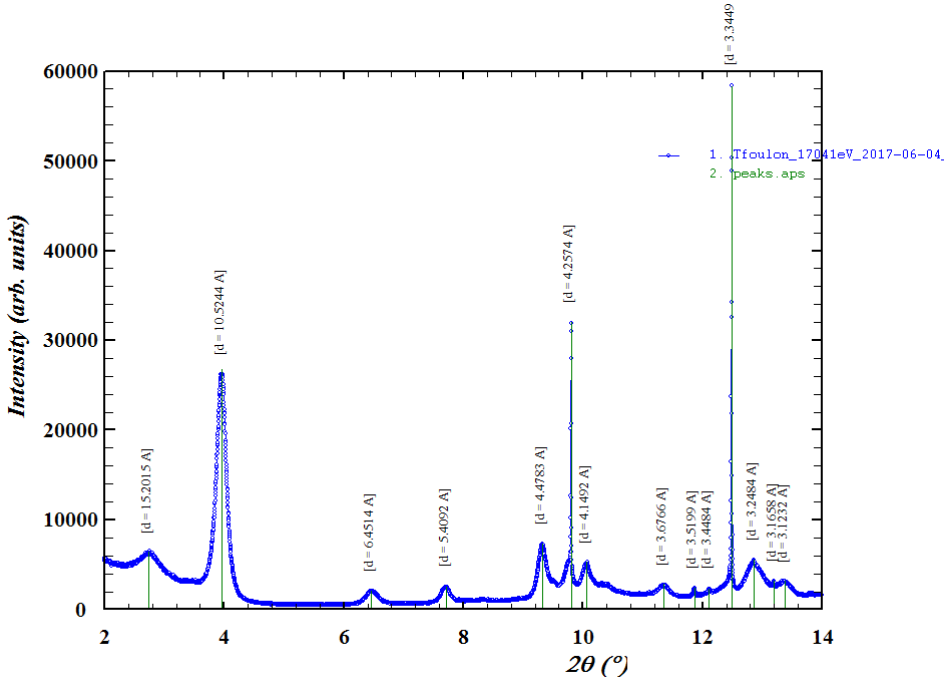


CRISTAL beamline at synchrotron SOLEIL, undulator beamline delivers an intense and almost parallel X-Ray beam well adapted to high angular resolution measurement in the 5 to 30 keV energy range.

X-RAY DIFFRACTION HR XRD

SYNCHROTRON CRISTAL BEAM LINE





Intensity, reticular distance d (I, d) is used to phases identification by comparison with database PDF4+ + database (International Center for Diffraction Data (ICDD), Newton Square, PA, USA).

Parameters such as intensity and diffraction rays locations (characteristic of each phase) in the diffraction diagrams are phase characteristic and establish **a signature Palygorskite – Quartz (SiO_2)**

BIOEQUIVALENCE

SIGNIFICANCE

- Tests must be relevant to topical equivalence (FE suspension) .
- Demonstrate the equivalence in decontamination efficiency (DE) .
- Bioequivalence could be based on skin biocompatibility in function of the DE

INNOVATION

- SEM-TEM-XRD Synchrotron developped as one of potential tools for an appropriate product characterization
- Detect impurities , measurements and create **a data ba**se with all those **images**
- Use innovative and High tech methods to compare the samples selected to the referent

Challenge – Prospective – Development

- Label for the generic name of FE -skin decontamination agent
- Safe for health – skin application
- Prospect and develop easy use for Self decon and mass casualties decon

CONCLUSION

CLASSIFICATION

- FE currently referenced in **skin decontamination processes** for both militarians and civilians.
- Classify clays "bio equivalent" to the FE in function of their signature (nature , composition) and performance Decontamination Efficiency

REFERENCEMENT

- Topical product applied onto the skin must present characteristics of safety.
- Label for the generic name of FE skin decontamination agent
- FDA –European Pharmacopea as other decontaminants

CHALLENGE

- Comparison of the various sources of FE with the « referent FE »
- Create a data base of pictures including images characteristics format compatible

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Thank you for your attention