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ESRF

Experiment Report Form

The double page inside this form is to be filled in by all users or groups of users who have had access to beam time for measurements at the ESRF.

Once completed, the report should be submitted electronically to the User Office via the User Portal:

https://wwws.esrf.fr/misapps/SMISWebClient/protected/welcome.do

Reports supporting requests for additional beam time

Reports can be submitted independently of new proposals – it is necessary simply to indicate the number of the report(s) supporting a new proposal on the proposal form.

The Review Committees reserve the right to reject new proposals from groups who have not reported on the use of beam time allocated previously.

Reports on experiments relating to long term projects

Proposers awarded beam time for a long term project are required to submit an interim report at the end of each year, irrespective of the number of shifts of beam time they have used.

Published papers

All users must give proper credit to ESRF staff members and proper mention to ESRF facilities which were essential for the results described in any ensuing publication. Further, they are obliged to send to the Joint ESRF/ ILL library the complete reference and the abstract of all papers appearing in print, and resulting from the use of the ESRF.

Should you wish to make more general comments on the experiment, please note them on the User Evaluation Form, and send both the Report and the Evaluation Form to the User Office.

Deadlines for submission of Experimental Reports

- 1st March for experiments carried out up until June of the previous year;
- 1st September for experiments carried out up until January of the same year.

Instructions for preparing your Report

- fill in a separate form for each project or series of measurements.
- type your report, in English.
- include the reference number of the proposal to which the report refers.
- make sure that the text, tables and figures fit into the space available.
- if your work is published or is in press, you may prefer to paste in the abstract, and add full reference details. If the abstract is in a language other than English, please include an English translation.

ESRF	**Experiment title:** Palygorskite: a new approach to its structure	**Experiment number**: 25-01 982
Beamline:	**Date of experiment**: 22-03-2016 – 24-03-2016 / 31-08-2016 – 3-09-2016	**Date of report**: 25-08-2017
Shifts: 15	**Local contact(s)**: Germán Rafael Castro	*Received at ESRF:*

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Report:

Palygorskite is a nanostructured fibrous clay widely used as industrial mineral due to its excellent adsorbent properties related to its structure. Although is a mineral widely studied some questions related to its structure remain open. In this experiment we aimed to study deeply the structure of a sample (PAL) from Palygorskaya, the type-locality of this mineral. We obtained high resolution powder diffractograms of the PAL sample in order to perform a Rietveld refinement on them and being able to determine its structure and cell parameters, although this was not fullfilled.

During the first 6 allocated shifts (22-03-2016 – 24-03-2016), we encountered several technical issued that lead us to obtain data which were not usable.

During the other 9 allocated shifts (31-08-2016 – 3-09-2016) we obtained several measurements at different temperatures to observe the variation of the cell parameters of palygorskite (Fig. 1) according to the temperature until total dehydration of the mineral (600K).

The presence of impurities of potassic feldspar within the sample and the lower resolution than expected (Fig. 2) toughened the efforts of performing the Rietveld refinement of this mineral, although it is expected to be able to perform it in the future.

The measurements obtained were compared with theoretical simulations of powder diffractograms (along with oriented fiber diffractograms from a different experiment at ID18F) (Fig. 3) using accepted structural models published throughout time, being able to observe that these models do not fit the reality of the mineral, presenting unknown refflections that do not correspond to the mineral nor to impurities. This was published in the journal Applied Clay Science.

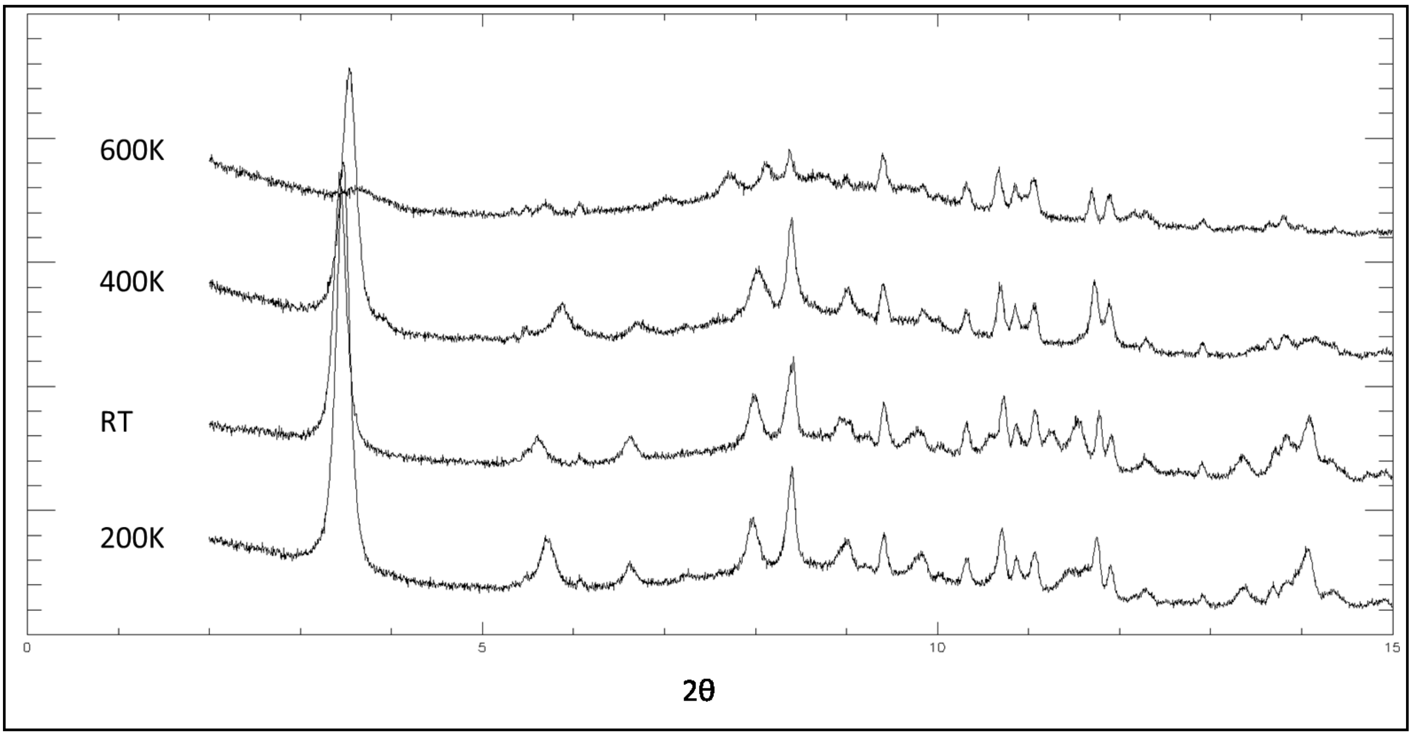

Fig 1. Powder diffratograms of palygorskite at 200K, room temperature, 400K and 600K. We can observe the displacement of the reflections depending on the temperature.

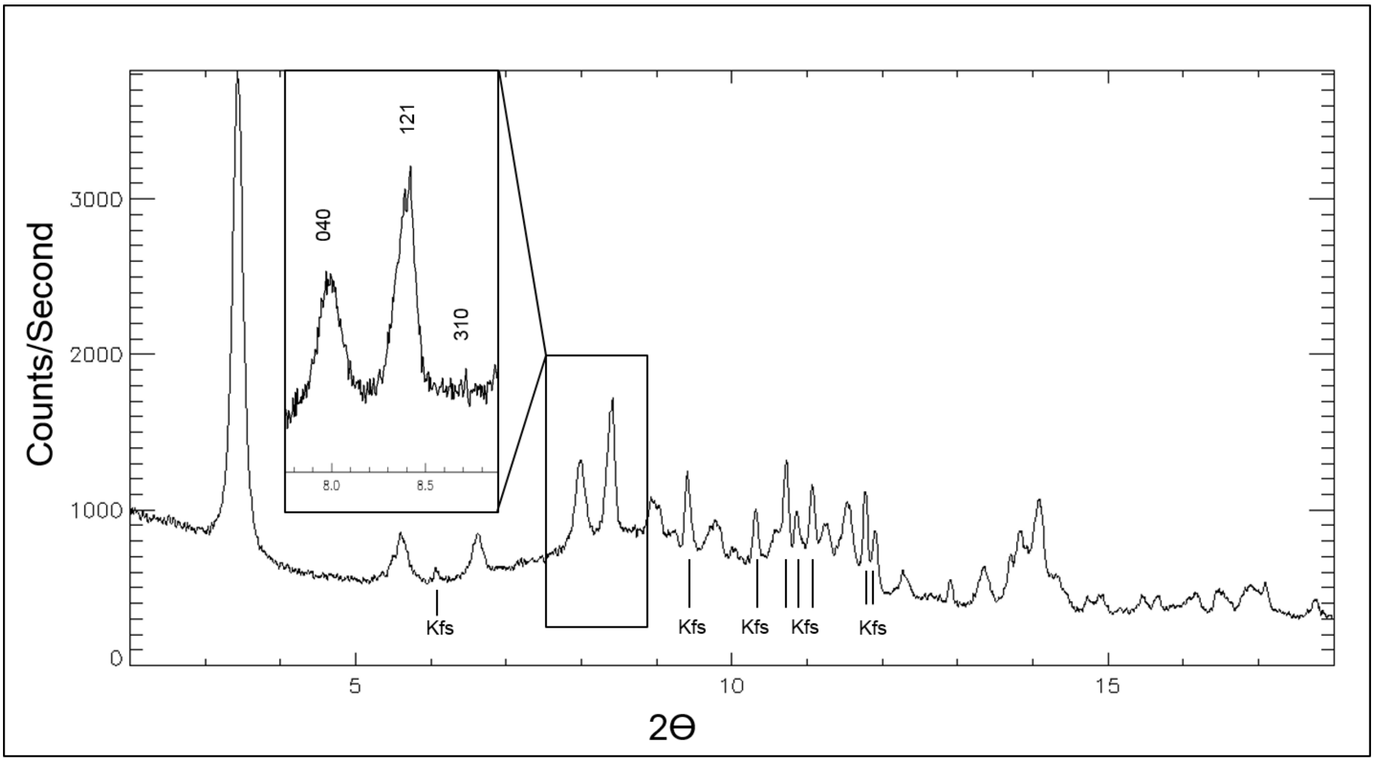

Fig 2. Powder diffragtogram of palygorskite obtained at BM25a. We can observe the presence of impurities of potassic feldspar (K-feldspar).

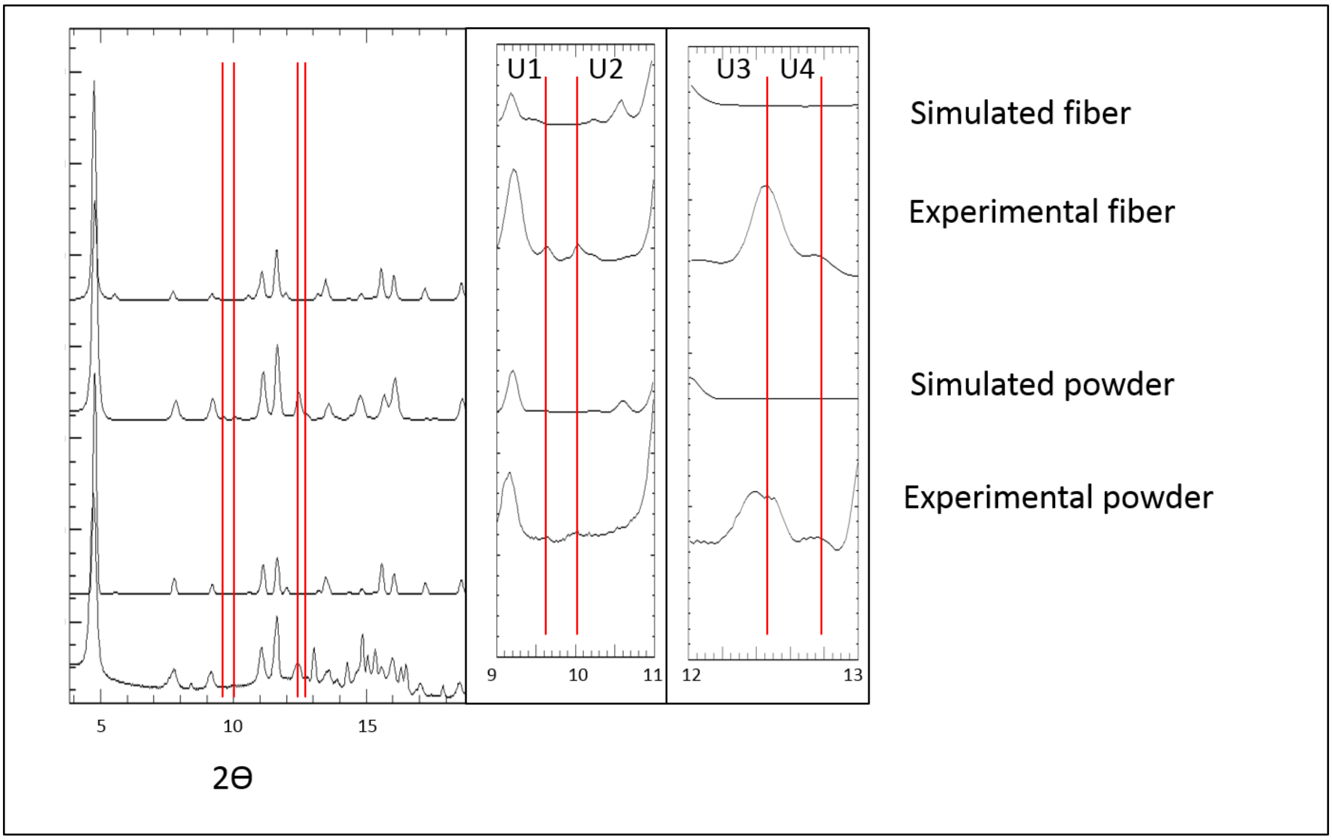

Fig 3. Powder and oriented fiber experimental diffractograms compared with theoretical simulations. We can observe several unknown reflections (U1, U2, U3, U4), not belonging to the mineral nor to impurities.

Publications

García-Rivas, J., Sánchez del Río, M., García-Romero, E., Suárez, M. (2017): An insight in the structure of a palygorskite from Palygorskaja: Some questions on the standard model. Applied Clay Science, 148, 39 – 47. https://doi.org/10.1016/j.clay.2017.08.006

Abstract :

In this study we analyze in detail a palygorskite from Palygorskaja. This palygorskite is situated in the context of the existing studies using X-ray powder diffraction analysis. Moreover, a novel microdiffraction study on a small bunch of fibers shows highly structured 2D diffraction patterns that allow to decipher some information on the microstructure, thus overcoming the uncertainty usually originated by large samples containing mixtures and impurities. Structural data provided by Chisholm (1992) are used to simulate 1D and 2D powder and fiber diffraction patterns for palygorskite, which are compared with the experimental results. We performed simulations for powder and fiber diffraction and we centered our attention in the region of interest with d-spacings between 4.0 and 4.5 Å. This palygorskite is consistent with a purely orthorhombic palygorskite, based on good agreement of data with simulations. The experimental results present some reflections not found in the simulations. These reflections are interpreted as corresponding to other hk1 planes of palygorskite. They do not match any reflection from the monoclinic structural model nor from probable impurities, reinforcing the interpretation of them being intrinsic to the structure of the mineral. Our findings suggest a revisal of the commonly accepted structure of palygorskite. They correspond to hk1 planes of palygorskite, and they do not match any reflection neither from the monoclinic structural model nor from possible impurities, and thus reinforcing the idea of them being intrinsic to the structure of the mineral, suggesting the necessity of a revisal of the commonly accepted structure of palygorskite.