ESRF	**Experiment title:** Structural study of imogolites and allophanes aluminosilicates using PDF analysis, influence in the transfer of trace metal elements in soils.	**Experiment number**: EC-503
Beamline: ID31	**Date of experiment**: from: 22/06/2009 to: 26/06/2009	**Date of report**: 25/02/2010
Shifts: 9	**Local contact(s)**: Adrian HILL	*Received at ESRF:*

Names and affiliations of applicants (* indicates experimentalists):

Pierre BORDET* (Institut Néel, CNRS)

Daniel BORSCHNECK* (CEREGE, CNRS)

Emmanuel DOELSCH* (CEREGE, CIRAD)

Jean-Louis HODEAU* (Institut Néel, CNRS)

Clément LEVARD* (CEREGE)

Jérôme ROSE (CEREGE, CNRS)

Report:

Introduction

This study follows the experiments done on D2AM (exp. 02-02-714) for the investigation of the structure of short-range ordered aluminosilicates (imogolites and allophanes, $(OH)_3Al_2SiO_3(OH)$) generally found in volcanic soils.

The idea was to evaluate the role of these natural nanoparticles (specific surfaces near $700m^2/g$) on heavy metals dynamic in soil [1], particularly in the case of the island of La Réunion where high levels of metallic elements (Cr, Ni, Cu, Zn,…) are present. So the determination of the structure of these minerals appears to be essential to understand adsorption and transport mechanisms.

In the case of imogolite, it is relatively clear that it consists in hollow nanotubes of 2nm diameter and hundreds nanometers long [2]. For the allophanes, the structure is still much debated, even if it is mostly described as nanospheres of 3 to 5 nm.

Experiments on D2AM at 25keV allowed us to obtain PDF curves of good quality for imogolite until 15 Å contrary to allophane for which the signal decreases rapidly after 10-12 Å. This prevented us from concluding on its structure at a larger scale.

In order to improve this results with higher Qmax, better spatial resolution and counting statistics we performed these new experiments on ID31 using the multianalyzer stage.

Experimental details

Experiments were realised at 30keV (λ= 0.399Å, Qmax = 31.5 $Å^{-1}$) between 0 and 120° (2θ). using glass capillary of 1.5mm diameter as sample holders. Multiple scans were realised and added above 30° in order to improve statistics at high Q values (2 between 30 and 50°, 3 between 50 and 70°, 4 between 70 and 90° and 5 between 90 and 120°). An imogolite and several different allophane samples were measured.

Results

The results obtained are superior to those from the previous experiment in terms of resolution and statistics. However, the presence in the data of an unexpected broad diffusion band around 10-15 $Å^{-1}$ complicates the extraction of the PDF. (see fig. 1 for the imogolite sample). A standard silicon sample showing a similar effect was also measured during the experiment and is being used to calibrate the data. This effect, which has already been observed [4], could be originated from an unidentified diffuse scattering source, possibly within the multianalyzer stage. Further data analysis is currently in progress.

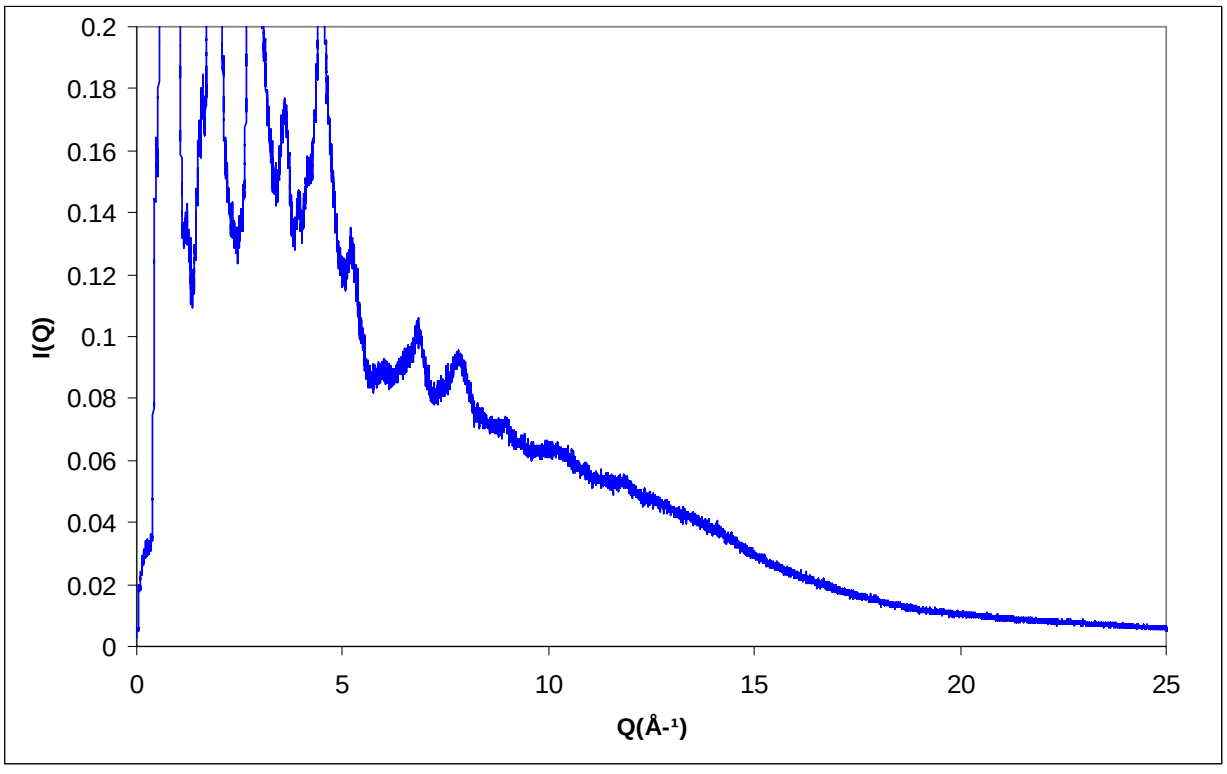

Fig. 1 : I(Q) for imogolite sample showing the broad diffusion band around 10-15 $Å^{-1}$

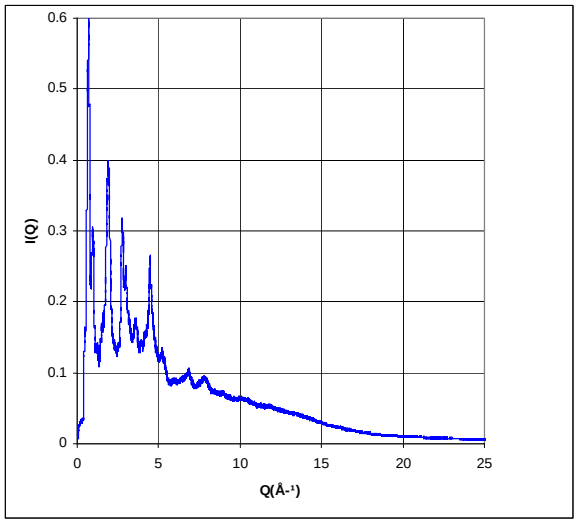

Fig. 2 : I(Q) for imogolite sample from ID31 corrected for empty capillary .

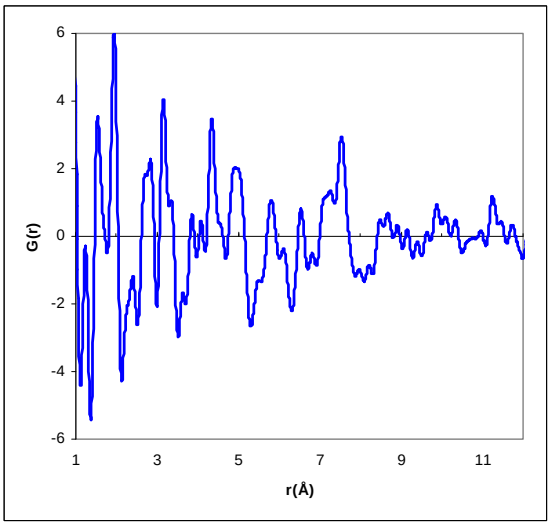

Fig.3 : Preliminary PDF extraction for imogolite sample from ID31 data.

References :

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