Experimental report

14/02/2018

Proposal: **1-03-33** **Council:** 4/2016

Title: The effect of production techniques on historical ceramic morphology

Research area: Materials

This proposal is a new proposal

Main proposer: **Claudia MONDELLI**

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Samples: ancient and modern ceramic

Instrument	Requested days	Allocated days	From	To
D11	3	3	08/12/2016	11/12/2016

Abstract:

Pore size distribution, pore shape as well as clay/inclusion ratio in volume percentage of historical pottery strongly depend on the used production techniques and on the geological sources. By the analysis of historical ceramic fragments we expect to be able to correlate the characteristics of the ceramic porosity, in terms of total volume and pore dimensions, to a specific production technique. We expect to reveal significant correlations among firing temperature, porosity and morphology of lab samples, prepared with different clay materials and subjected to different firing temperatures. The aim of this proposal is to complete and expand the results previously obtained by several techniques with SANS measurements. SANS will allow us to investigate the characteristics of open and closed pores (size, form factor shape) and the morphology of ceramic matrices (particle packing). The correlations within these data would allow, as a first step, the determination of important features of historical ceramics for linking their final characteristics to their production site.

ILL RESEARCH PROPOSAL 1-03-33

Title: **The effect of production techniques on historical ceramic morphology**

Proposers: MONDELLI Claudia, BALLIANA Eleonora, CATTARUZZA Elti, GONELLA Francesco, RICCI Giulia, SCHWEINS Ralf, ZENDRI Elisabetta.

Beam time: 08/12/2016 – 10/12/2016

The aim of the experiments was to investigate the characteristics of open and closed pores (size, form factor shape) and the morphology of ceramic matrices (particle packing) by SANS measurements, necessary to complete the results previously obtained by Mercury Intrusion Porosimetry (MIP), Scanning Electron Microscope (SEM) and traditional X-ray Computed Tomography (X-ray CT).

We measured both historical (10X3) and laboratory made samples (11) at 3 detector distances of 1.5 m, 8 m, 39 m, the latter using two wavelengths 6 Å and 13 Å. Historical samples are from Eastern Germany (16th, 17th century), which were in the past and still nowadays important stoneware production sites and from Italy (samples from 7th to 10th century).

Unfortunately, the USANS option on D11 was not available at that moment and we had to manipulate the samples in order to avoid the multiple scattering in the lower Q region: we reduced drastically the thickness and grinded some samples to reduce them to gross and thin powders. We measured them in Hellma cells of 1 mm thickness (Fig.1 presents some of the I(Q) curves for isotropic patterns). The results showed better signals with gross powder samples and thinner ceramic slices, but even with this manipulation of the samples only few of them did not present multiple scattering at 13 Å.

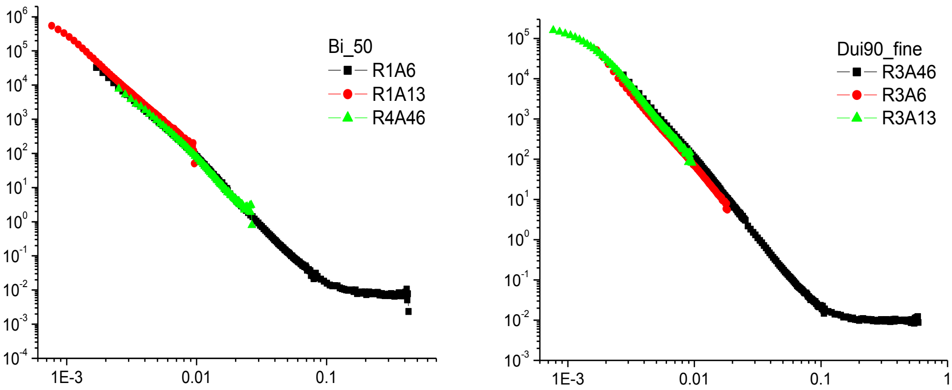

Figure 1. Example of I(Q) curves for isotropic samples

We definitely need the USANS option together with SANS to obtain curves without multiple scattering and to avoid alteration of the samples that can change the ratio open/close pores in a not controlled way. USANS measurements will performed at 6 Å that allow us to reach the desirable low Q values and minimize multiple scattering effects.

Most of German samples present anisotropic pattern, on the contrary of Italian samples that is an indicator of a different manufacturing process. We treat anisotropic patterns using a routine of LAMP that allows us to choose different sectors to build the I(Q) curves. We recently performed x-ray measurements in order to understand the origin of the anisotropy.

We obtained interesting results considering, in a first approximation, the samples composed by two phases (pores and solid homogeneous material) and assuming that the difference between the scattering length densities for these phases remain almost unchanged for all the samples studied, the Porod´s invariant results proportional to the total sample porosity and constitutes a comparative porosity parameter [Ref.: O. Glatter and O. Kratky

Chapter 2 in Small Angle X-Ray Scattering Academic Press, New York, 1982]. The Porod's invariant was evaluated for those samples measured with 3 detector distances (to ensure a large Q range to perform the Porod integral) and presenting an isotropic pattern. The results of this analysis are very encouraging: it evidences that, samples coming from different geographical sites, are in two well-separated groups (see Fig 2).

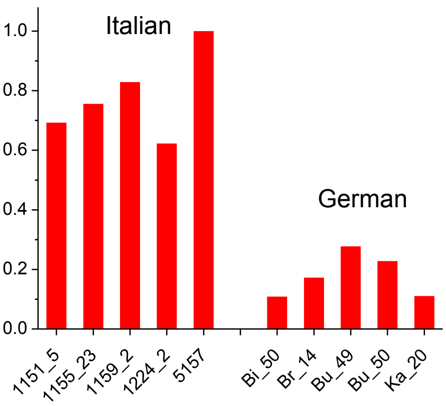

Figure 2.-Normalized Porod's Invariant for isotropic German and Italian samples. It can be observed a clear difference in porosity between German and Italian samples.

Other interesting results come from the analysis of the curves for the lab samples. They show a trend for the SANS intensities with the firing temperature (See Fig.3) The structure observed at q~0.08A^{-1} (~ 10nm) for G samples may indicate the growing of a second phase.

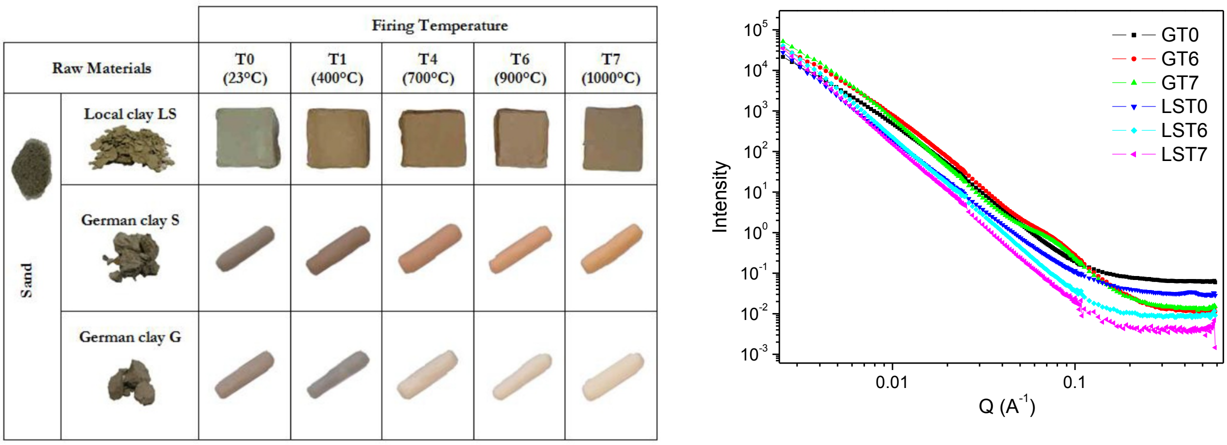

Figure 3. First results for samples produced in the laboratory. The SANS intensity show a clear trend with the firing temperature.

The I(Q) curves are still under analysis with SasView package.