EUROPEAN SYNCHROTRON RADIATION FACILITY
INSTALLATION EUROPEENNE DE RAYONNEMENT SYNCHROTRON

Commercial Experiment beamtime request at the ESRF - European ReMade@ARI

Proposal Summary

Title: **Reactivity and micro/nanostructural evolution of inertized alkali-activated mineral wool waste**

Principal Investigator: **Mr. BERNASCONI, Davide** (University of Turin - Earth Science Department)

Context: This is a new commercial experiment proposal

Abstract: This project aims to investigate the reactivity and micro/nanostructural evolution of inertized mineral wool waste (MW) in alkali-activated binder systems. MW is an abundant industrial byproduct typically landfilled due to its fibrous morphology and associated health risks. Through a novel thermal inertization process, MW is transformed into a safe, amorphous glass powder with high potential as a precursor for low-carbon geopolymer cements. The project targets the dual goal of waste valorization and CO2 emissions reduction by upcycling MW into sustainable binders, potentially replacing natural raw materials (i.e. calcinated clay) and industrial slag, whose production is destined to decrease due to the progressive decarbonization of steel industry.To understand the reaction mechanisms and structure development, the project will apply an integrated suite of synchrotron techniques—small-angle X-ray scattering (SAXS), X-ray pair distribution function analysis (PDF), and X-ray computed tomo

Beamline Requirements

#	Principal	Alternatives	Number of Shifts
1	**BM05**		3
		Total shifts:	3

Laboratory Support Facility Request

None defined by PI nor AC

Investigators Team

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Alternative Contact:

Sample-sheets declared at proposal submission

None defined by PI nor AC

Research Project Description

for ReMade@ARI - A hub for materials research

Proposal Ref No.:	PID: 38245
Name of main applicant:	Davide Bernasconi

Research Project Title:

Reactivity and micro/nanostructural evolution of inertized alkali-activated mineral wool waste

Scientific background and proposal novelty:

The cement industry accounts for 5–8% of global CO_2 emissions, with demand projected to rise sharply by 2050. While low-carbon alternatives like limestone-calcined clay cements (LC^3) reduce clinker content, they rely heavily on finite mineral resources and increasingly scarce supplementary cementitious materials (SCMs), such as fly ash and slag. Concurrently, the construction sector generates over 10 million tons of waste mineral wool (MW) annually, a byproduct of insulation production, which is predominantly landfilled or downcycled into low-value applications due to its fibrous morphology (DOI: 10.3390/ma14195777)

This project aims to upcycle waste MW into high-performance geopolymer binders, offering a dual environmental benefit: diverting hazardous waste from landfills and reducing reliance on traditional cementitious systems. The MW's inherent composition—rich in silica, aluminum, calcium, and magnesium oxides—offers huge potential as geopolymer precursor.

Moreover, MW, which was classified as possibly carcinogenic by IARC before 2001 due to its fibrous morphology, will be converted into a non-hazardous, amorphous glass powder through a novel controlled heat treatment. This pretreatment not only enhances safety but is expected to also improve MW's reactivity for geopolymer synthesis.

Geopolymers derived from MW could reduce CO_2 emissions by up to 70% compared to Portland cement, while leveraging a waste stream otherwise destined for unsustainable disposal. The experimental work will focus on the investigation of the structural and microstructural features evolution during the hardening/condensations reactions of MW-activators blends, which deeply affect the final materials structural and durability properties. These results will provide fundamental mechanistic information on the MW reactivity in these alkaline conditions, enabling the optimization of high-performance and sustainable MW-geopolymers formulations.

If applicable, previous results on this topic (mainly related to the proposer's work):

Alkali-activation of mineral wool waste (MWW) has been shown to produce high-strength binders, with compressive strengths up to 49 MPa and formation of N-A-S-H and C-(N-)A-S-H gels, depending on the wool type and activator composition (DOI: 10.1016/j.cemconcomp.2019.103472). Advanced synchrotron-based techniques have been applied to better understand similar alkali-activated

systems. In situ pair distribution function (PDF) and SAXS/WAXS have enabled tracking early-stage gel formation and nanostructural evolution in metakaolin and fly ash geopolymers, revealing how reaction pathways depend on temperature and activator composition (DOI: 10.1016/j.clay.2012.09.009); (DOI: 10.1021/la300868v). Additionally, synchrotron X-ray tomography has proven valuable for capturing 3D microstructural changes in alkali-activated fly ash-slag systems, highlighting particle dissolution, product formation, and porosity evolution during curing (DOI: 10.1016/j.cemconres.2015.08.004); (DOI: 10.1016/j.compositesb.2021.109221).
Although not previously applied to MW, the team has extensively studied similar binder systems. For example, an in situ SAXS and XPCS approach was recently employed to investigate the nanostructural evolution and gelation dynamics in phosphate geopolymers, capturing nanoscale percolation and aging phenomena directly relevant to the proposed in situ heating strategy (DOI: 10.1063/5.0239498)

Description of the proposed experiment methodology including relevant technical data:

The inertized MW waste will undergo the patented thermal treatment and subsequent grinding at 63 µm. The solid powder will be fully characterized in lab (e.g., chemistry, mineralogy, surface area). Although the actual implementation of MW in alkali-activated materials would require a partial substitution with more traditional precursor (i.e. fly ash or slag), the aim of the proposal is to obtain key information of its reactivity by isolating its contribution through the design of model binary mixture. Moreover, we aim to study the hardening mechanisms in situ under mild thermal curing (i.e., 60-80 °C), which is often performed to improve the dissolution and condensation kinetics.

From the literature, one of the main parameters affecting the reaction paths and final products is the type of activators. For this reason, the inertized MW waste will be blend with **4 different alkaline solutions** (NaOH, KOH, Na_2SiO_3, K_2SiO_3), **each at 3 concentrations/modulus values** and fixed liquid-mass ratio, thus producing **12 samples**. These will be thoroughly characterized in our lab with XRD, calorimetry, solid-state NMR, SEM-EDS and mechanical tests. The best performing formulation for the mechanical strength point of view for each activator will be selected for the synchrotron in situ studies, which will be then executed on **4 samples**.

For each selected sample, the MW powder will be mixed for 1 min with the alkaline solution directly at the beamlines, with the use of a portable automated mixer. The fluid paste will be then injected into capillaries (borosilicate glass) of appropriate diameter size (i.e. 1.5 mm), which will be sealed to preserve the saturated atmosphere. The measurements are expected to be collected for the first 8h at mild heating conditions (60-80 °C). This time frame is expected to provide the most significant changes in terms of nano/microstructure. Moreover, additional measurements will be collected after 24h of thermal curing and after at least 30 days of aging, to compare the in-situ results to long-aged samples.

The uXCT data will be acquired at a **voxel size approx. 1 µm**, covering a **ROI approx. 800x800x800 µm**, with a duration of approx. **5-10 min/scan.** Qualitative and quantitative information on sample features (i.e. porosity, particle morphology) will be extracted with dedicated software tools (i.e. ImageJ and Dragonfly). Based of previous data, good enough contrast is expected to be able to segment the samples into three phases (unreacted particles, matrix, pores).

The SAXS data will be collected in the **range of *Q* 0.01-4 nm^{-1}**, each collection expecting to require **less than a minute**. Although both the MW and the alkali-activated materials are fully amorphous, it is possible that, during the condensation's reactions, a minor crystallization of hydrated phases occur (i.e. zeolite or quintinite). For this reason, we ask the possibility to collect SAXS-WAXS signal (quasi-)simultaneously.

The PDF data will be collected with a **Q_{max} of 20 Å^{-1}**, each collection expecting to require **less than few min.** The pristine solid MW will also be scanned to be used as a reference of the initial structure.

Provide sample relevant information

The inertized MW is a solid powder and poses no chemical or environmental risk. The activators solutions can be considered strong alkaline reagents (pH 12-13), thus they will be handled with standard laboratory equipment (i.e. lab coat, glasses, gloves). The experiments will be performed at mild T (60-80 °C) and standard pressure. Samples will slightly differ in chemical composition but will be largely composed of alkali aluminosilicate hydrates; the chemical composition will be roughly **30-40 wt.% of SiO_2, 14 wt.% of Al_2O_3, 5 wt.% Fe_2O_3. 7 wt.% of CaO, 3-20 wt.% of Na_2O, 3-20 wt.% of K_2O, 15-20 wt.% of H_2O**; the exact numbers will depend on the chosen activator's concentrations. The samples will be analysed in capillary of 1.5 mm in diameter.

Motivation and justification for access time, chosen techniques, and facility/ies:

The proposal aims to derive key information on MW behaviours in alkali-activated binders, by investigating the reactions mechanisms in situ at different spatial scales through a combined PDF-SAXS-XCT approach:

1- The SAXS technique has been largely employed to characterize gelation process in similar systems, since it allows to follow the condensation reactions at the nanoscale from the first oligomers aggregation up to the larger tridimensional clusters (DOI: doi.org/10.1021/la300868v).

2- uXCT will provide complementary information to SAXS and the other employed lab techniques. The chosen voxel size is appropriate to detect porosity and unreacted MW particles, as previous studies employed values in the range 0.75-1.5 µm. Contrast between reacting glass and liquid will be granted by the iron content of the powder (DOI: 10.1016/j.compositesb.2021.109221).

3- Although the full modelling of PDF data can be challenging in these chemically complex materials, the PDF approach will provide valuable information on the polyhedra local structure changes during the MW dissolution and gel condensations, as previously demonstrated on metakaolin geopolymers (DOI: 10.1016/j.clay.2012.09.009).

After discussion with the instrument scientists, considering the number of samples and the time required for instrumental setup, we ask for **2 days for SAXS**, **2 days for XCT** and **2 days for PDF**:

-**32h** for in-situ collections (4 samples followed in-situ for 8h each); **1-2h** for collection of long-aging samples (8 samples); **14-15h** for beamline setup and sample changing

Additional information

As suggested by the contacted instrument scientists, some experimental conditions (e.g., X-ray energy for the XCT and PDF experiments) have not been indicated, because they will be decided with the local contacts, being dependent upon the facility/instrument capabilities.