EMPIR Call 2020 – Fundamental, Industry and Normative

Selected Research Topic number: **SRT-i29**
Version: 1.0

Title: Surface and interface chemical metrology for porous materials

Abstract

Porous materials are critical to a wide range of manufacturing industries. Both structural and chemical measurements are required, but the latter are not adequately developed. Surface area and pore size distribution measurements fail to capture important functional properties determined by interfacial chemistry. There is a need for validated and accurate chemical measurements of the internal and external surfaces of porous materials. Metrological guidance is required to significantly reduce analytical costs. Proposals are sought to address accurate chemical distribution measurement within porous materials by producing relevant reference materials and protocols for cross-sectioning, tomographic and novel optical methods.

Keywords

Porosity, surface chemistry, functionalisation, catalysis, electrodes, biomaterials, drug delivery, filters, optoelectronics

Background to the Metrological Challenges

The analysis of porous materials is critical to a wide range of industries and is important for applications such as filters, storage, optical and electronic devices and additive manufacturing, as well as functional materials including catalysts, fuel cells, sensors and biomedical devices such as wound dressings and drug delivery systems. As the latter technologies develop, the materials become more challenging to analyse because they contain hierarchical structures of chemistry and porosity, often constructed as thin films on non-porous supports. Current measurement methods for porous materials are largely restricted to specific surface area and pore size distributions. Techniques such as gas adsorption, mercury intrusion and pycnometry are widely used and capable of measuring the surface area, pore diameters and average porosity. These methods are suitable for bulk porous materials but often fail when the porous material is a thin film, in which case they have insufficient sensitivity.

There is therefore a current requirement for accurate measurements of the chemical components of porous materials, particularly those located at internal and external surfaces which impact the behaviour and performance of materials. Although there are cross-sectioning, tomographic and spectroscopic methods that can provide some insight, the accuracy and comparability of these methods is unclear due to the lack of adequate reference materials and comparative studies. It is important that guidance is provided for sample preparation and analysis protocols, surface sensitivity, accuracy and data treatment for these methods. Meanwhile there are a number of optical methods that have been developed for biological analysis that could be adapted to address this chemical measurement challenge and, additionally, could provide the basis for *in-operando* measurements to improve the design of functional materials.

Objectives

Proposers should address the objectives stated below, which are based on the PRT submissions. Proposers may identify amendments to the objectives or choose to address a subset of them in order to maximise the overall impact, or address budgetary or scientific / technical constraints, but the reasons for this should be clearly stated in the protocol.

The JRP shall focus on the traceable measurement and characterisation of the internal and external surfaces of porous materials.

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The specific objectives are

1. To produce reference materials containing a known distribution of pore-sizes from the macroporous to microporous regime. The reference materials should have a known internal distribution of surface chemical species, including oxide layers, metallic nanoparticles and organic functional groups.
2. To develop procedures for focussed ion beam cross sectioning, coupled with chemically specific surface ion, electron and scanning probe imaging. In addition, to provide protocols to minimise chemical and structural artefacts from the cross sectioning; guidance on distinguishing and measuring surface species and applicability to different types of porosity and types of materials.
3. To determine the capability of low lateral resolution techniques, such as X-ray spectroscopy, tomography, photoelectron emission and optical techniques, to identify interfacial chemistry and measure their macroscopic distributions within porous materials.
4. To provide recommended workflows and algorithms for the measurement of interfacial chemistry in porous materials, to assist those industries developing and using functional materials, to select appropriate and complementary methods.
5. To facilitate the take up of the technologies and measurement infrastructures developed in the project by the measurement supply chain (analytical laboratories), standards developing organisations (CEN, ISO) and end users (including catalyst, semiconductor and medical device manufacturers).

Proposers shall give priority to work that meets documented industrial needs and include measures to support transfer into industry by cooperation and by standardisation. An active involvement of industrial stakeholders is expected in order to align the project with their needs – both through project steering boards and participation in the research activities.

Proposers should establish the current state of the art, and explain how their proposed project goes beyond this.

EURAMET expects the average EU Contribution for the selected JRPs in this TP to be 1.5 M€, and has defined an upper limit of 1.8 M€ for this project.

EURAMET also expects the EU Contribution to the external funded partners to not exceed 30 % of the total EU Contribution across all selected projects in this TP.

Any industrial partners that will receive significant benefit from the results of the proposed project are expected to be unfunded partners.

Potential Impact

Proposals must demonstrate adequate and appropriate participation/links to the “end user” community, describing how the project partners will engage with relevant communities during the project to facilitate knowledge transfer and accelerate the uptake of project outputs. Evidence of support from the “end user” community (e.g. letters of support) is also encouraged.

You should detail how your JRP results are going to:

- Address the SRT objectives and deliver solutions to the documented needs,
- Feed into the development of urgent documentary standards through appropriate standards bodies,
- Transfer knowledge to the health, oil & gas, energy and environment sectors.

You should detail other impacts of your proposed JRP as specified in the document “Guide 4: Writing Joint Research Projects (JRPs)”

You should also detail how your approach to realising the objectives will further the aim of EMPIR to develop a coherent approach at the European level in the field of metrology and include the best available contributions from across the metrology community. Specifically, the opportunities for:

- improvement of the efficiency of use of available resources to better meet metrological needs and to assure the traceability of national standards
- the metrology capacity of EURAMET Member States whose metrology programmes are at an early stage of development to be increased
- organisations other than NMIs and DIs to be involved in the work.

Time-scale

The project should be of up to 3 years duration.