Structural and microstructural characterization of Pt/C catalyst through X-ray total scattering methods

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Carbon-supported Pt-based catalysts are highly active toward the oxygen reduction reaction (ORR) and are used in the cathodic compartment of proton exchange membrane fuel cells (PEMFCs), which are relevant for clean and sustainable energy production. Catalysts based on Pt nanoparticles (NPs) are the most widely employed because of their high activity. However, significant research efforts are currently devoted to improving their performance and durability while reducing the overall Pt content and associated costs.

The detailed characterization of these materials is crucial for correlating quantitative descriptions of their structural and microstructural features with their functional properties, elucidating underlying mechanisms, and guiding the optimization of synthetic procedures for enhanced performance [1–3].

In this work, we demonstrate the relevance of an integrated approach based on Small-Angle X-ray Scattering (SAXS) and synchrotron Wide-Angle X-ray Total Scattering (WAXTS) methods to characterize benchmark Pt NPs on carbon support synthesized at different temperatures. Data analysis based on the Debye Scattering Equation (DSE) provides a robust model for determining the size, size distribution, morphology, and lattice strain of Pt NPs. The morphology, size, and size distribution obtained from SAXS analysis were used to constrain the WAXTS data modelling. This revealed subtle structural and microstructural features that can be correlated with the electrocatalytic properties of the materials. Specifically, the specific surface area determined from SAXS and WAXTS analyses was correlated with the electrochemical surface area of the Pt nanocatalyst, as determined by electrochemical CO stripping, and with its activity toward the ORR, evaluated using the rotating disk electrode technique.

The refined model was successfully applied to investigate the solid-state synthesis of Pt NPs *in situ* by analysing a series of synchrotron WAXTS measurements conducted using a homemade reactor. This approach enabled the extraction of quantitative information on the thermal decomposition of the precursors and gain insight into the nucleation and growth mechanisms of Pt NPs.

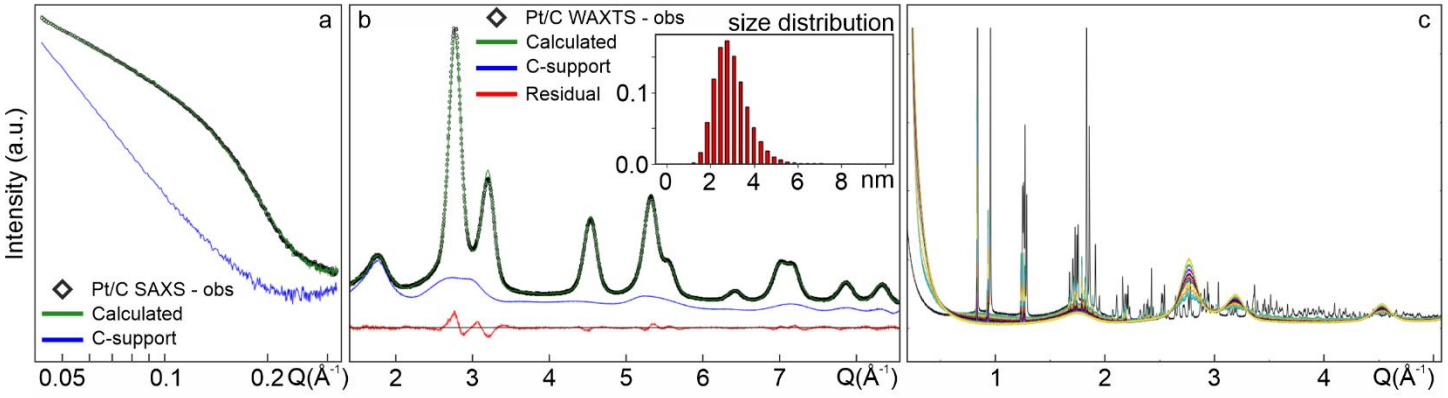


Figure 1. *a. SAXS data and best DSE fit (green). b. WAXTS data and best DSE fit (green). In blue the scattering trace of the carbon support. Inset: Number-based size distribution of Pt NPs from the combined DSE analysis of SAXS and WAXTS data. c. In-situ WAXTS measurements reproducing the synthesis of Pt/C catalyst.*

[1] Brandiele et al., (2024) Applied Catalysis B: Environmental, 123620, 344
[2] Parnigotto et al., (2025) Small, 21, 2403127.
[3] Parnigotto et al., (2026) Nanoscale, 18, 4300-4311