

Rauscher, M.V.¹; Seyffertitz M.¹; Kohns R.^{2*}; Stock, S.¹; Paris, O.¹

¹Chair of Physics, Montanuniversität Leoben, ² Department of Chemistry and Physics of Materials, Paris Lodron University Salzburg

* Now at: Institute for Materials and X-Ray Physics, Hamburg University of Technology; Centre for X-Ray and Nano Science CXNS, DESY Hamburg

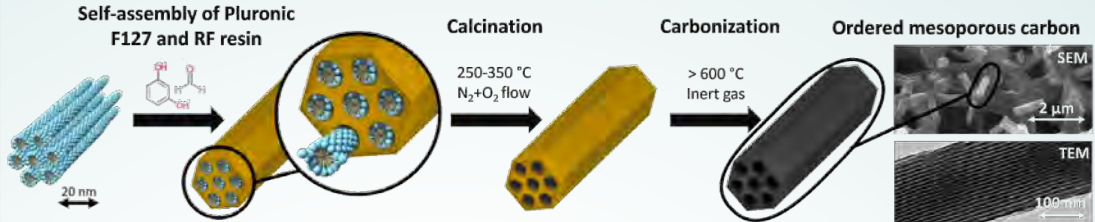
Motivation

In many different fields, such as hydrogen storage, electrochemical energy storage, and separation of CO₂/CH₄ for instance, porous carbons have drawn a lot of attention. Numerous benefits of using materials with hierarchical porosity in electrochemical energy storage devices have been established. Additionally, the introduction of ordered mesopores (e.g. soft templated carbons, STCs) coupled with the presence of disordered micropores provides unprecedented opportunities on unveiling fundamental processes within devices using porous carbons in x-ray diffraction and scattering studies [1,2].

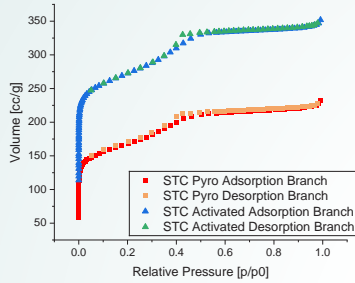
This enables the observation of specific effects depending on the pore size. Importantly, by modifying the precursor material and maximizing heat treatments, the specific mesopore size may be accurately adjusted, providing a high level of control for examining pore size-specific effects.

Introduction to STCs

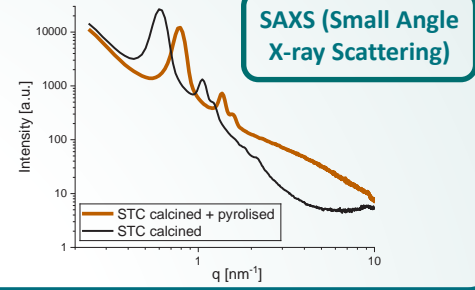
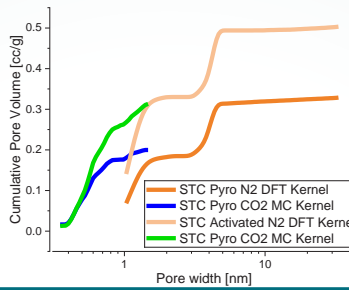
Synthesis



Characterization:



Gas Adsorption

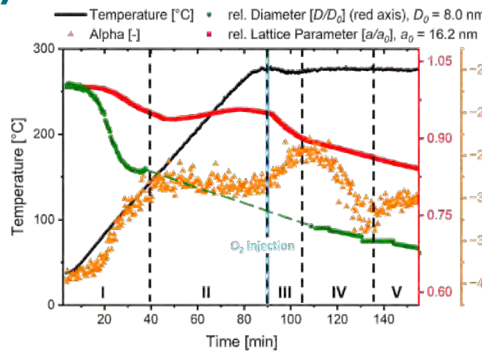


Optimizing heat treatment by in-situ SAXS

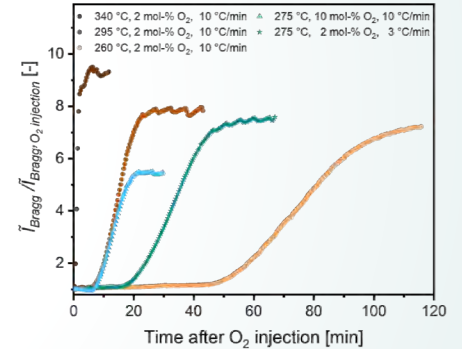
Investigations on the presented STCs were performed with in-situ SAXS at the Synchrotron ELETTRA in Trieste [1]. The aim of this study was to improve the calcination and thus the soft template removal. Optimized process parameter were successfully derived by investigating the changes of the small angle scattering and diffraction signals.



Video depicting the changes of the scattering signal during one in-situ measurement



Evaluation of in-situ SAXS. The large changes of multiple parameters lead to the distinction between 5 different stages during calcination (indicated with dashed lines I-V). Evaluation and conclusion can be found in [1].



By looking at different process parameters and evaluating the SAXS data, optimized process parameters can be suggested [1].

Results

Study of the changes during calcination lead to optimized process parameters:

- Temperature: **260-300°C**
- Time (dependent on temperature): **30-120 min**
- Atmosphere: **2 at.% O₂**

10 at.% O₂ instead of 2 at.% O₂ leads to fast decomposition of the template and fast distortion of the structural integrity



Future Research using STC

STCs will be used for SAXS and small angle neutron scattering (SANS) experiments:

- For in-operando electric double layer capacitors
- For in-operando water desalination cells
- For investigating H₂ adsorption

Model- vs industrial Material

The main advantage of the here presented STCs is the hierarchical structure making it a model material for scattering and diffraction experiments. The simultaneous presence of ordered mesopores and disordered micropores allows to study processes (e.g. gas sorption, electro sorption, diffusion processes) dependent of the pore size using SAXS. This enables a deeper understanding of the fundamental processes and provides valuable insights into the suitability of different pore sizes and their distribution for specific applications.

However, the mass production of this material poses challenges. It is not easily scalable, comes with high material costs, and lacks ecological production methods making it unsuitable for industrial applications.

References

- [1] M.V. Rauscher, M. Seyffertitz et al., "Optimizing surfactant removal from a soft-templated ordered mesoporous carbon precursor: an in situ SAXS study," J Appl Crystallogr, vol. 56, no. 3, pp. 801–809, May 2023, doi: 10.1107/S1600576723003886.
 [2] C. Koczwar et al., "Nanofibers versus Nanopores: A Comparison of the Electrochemical Performance of Hierarchically Ordered Porous Carbons," ACS Appl Energy Mater, vol. 2, no. 7, pp. 5279–5291, Jul. 2019, doi: 10.1021/acsami.9b01035.