# Readme - Dataset to "Routes Towards Soft Templated Non-Graphitic Carbons with Closed Mesoporosity and Their Adoption as a Model Material for Porosity Analysis"

**Purpose of the data collection:**

The publication "Routes Towards Soft Templated Non-Graphitic Carbons with Closed Mesoporosity and Their Adoption as a Model Material for Porosity Analysis" faces the synthetic challenge of pore accessibility tuning. This is done by, first, preparing non-graphitic carbons with accessible mesopores in a diameter between 5 and 50 nm based on poly(isobutylene)-*block*-poly(ethylene oxide) ($PIB_{50}$-*b*-$PEO_{45}$) and poly(ethylene oxide)-block-poly(*n*-hexyl acrylate) ($PEO_m$-*b*-$PHA_n$) block copolymer soft templating. Second, defined post-treatments are shown to create inaccessible mesoporosity within the carbon samples. Going further, also the analytical challenge of analysing (in)accessible pore systems with gases and liquids is tackeled using nitrogen physisorption as well as Small-Angle X-Ray Scattering contrast matching experiments complementary. Hence, this collection provides the raw data of block copolymer characterisation (proton-nuclear magnetic resonance ($^1$H-NMR), gel permeation chromatography (GPC)). Additionally, raw data of physisorption experiments and Wide-Angle X-Ray Scattering (WAXS) characterising the mesoporous non-graphitic carbon products after block copolymer soft templating is given.

**Details about materials:**

The $PEO_m$-*b*-$PHA_n$ block copolymer were self-synthesized by a Steglich esterification and a subsequent supplemental activator reducing agent atom transfer readical polymerisation. Details on the polymer synthesis can be found in respective literature,[1] as is not the main focus of the present publication. The mesoporous carbon products are resorcinol-formaldehyde-derived resin-based non-graphitic carbons. Next to the block copolymer templated mesoporous carbon products, also equivalent non-templated as well as $ZnCl_2$-templated (purly microporous) resorcinol-formaldehyde-based carbon samples were prepared as reference material.

**Details about data acquisition:**

$^1$H-NMR experiments were carried out at 25 °C with a *Bruker Avance II 400 MHz* and *Bruker Avance III 400 MHz HD* at Justus Liebig University Gießen (JLU). The software *MestReNova 14.1.2* was applied for data evaluation while the $CHCl_3$ solvent peak at $\delta$ = 7.26 ppm served as reference.

GPC experiments were conducted at University of Potsdam with a flow rate of 0.5 mL $min^{-1}$ at room temperature. Tetrahydrofuran was used as eluent in which the samples were dissolved applying 40 °C and ultrasonic treatment. The injection volume was 100 µL and contained 0.15 wt% polymer concentration, which was filtered through 0.45 µm filters. Simultaneous UV (*TSP UV 2000*) and differential refractive index (*Shodex RI-101*) detection was applied and a 300 mm × 8mm *PSS SDV linear M* column served as stationary phase packed with 3 µm particles in a mass range of $10^2$ – $10^6$ Da. Poly(styrene) standards (*PSS*, Mainz, Germany) were used for calibration.

A *Qudrasorb evo* by *Quantachrome Instruments* was used to perform nitrogen physisorption experiments at 77 K while an *Autosorb iQ* from *Quantachrome Instruments* was used to record argon physisorption isotherms at 87 K. Both measurements were carried out at JLU. Carbon samples were degassed prior to analysis at 80 °C for 12 h. A quenched solid density functional theory (QSDFT) kernel on carbon with cylindrical and spherical pore geometry applied on the adsorption branch enabled data evaluation done with the software *ASiQwin 5.12*.

X-ray scattering data were collected at JLU with a *XRDynamic 500* diffractometer from *Anton Paar* in Brag-Brentano geometry at room temperature. Cu-$K_{\alpha1}$ radiation ($\lambda$ = 1.5406 Å) and Cu-$K_{\alpha2}$ radiation ($\lambda$ = 1.5444 Å) in a 1:2

ratio were applied with a current of 50 mA and a voltage of 40 kV. Measurement was done in the range of 10° < 2$\theta$ < 115° with a step size of 0.1°.

**Structure of provided data:**

In summary, twelve files are part of this data set. The name of the .txt file clearly indicates the data that are stored in the respective file. In the NMR and GPC raw data files, the name of the respective block copolymer ($PEO_m$-*b*-$PHA_n$) is annotated above the related data. Physisorption raw data are divided into several data sets seperating adsorption isotherms from pore size distributions, on the one hand, and the different carbon materials templated by the respective polymer or $ZnCl_2$, on the other hand (clearly indicated by the name of the .txt file). As porous carbons have been templated by different polymer templates applied in different mass ratios compared to the carbon precursor, the physisorption files contain comments about the polymer mass content (*i.e.*, "100%", "75%", "50%", or "25%") applied in the synthesis. Additionally, the carbonisation temperature ("900 °C" or "1300 °C") and the post-treatment technique of "Pore Sealing" are depicted to enable a clear asssignment to the respective sample discussed in the publication. For the $PEO_{231}$-*b*-$PHA_{54}$-templated carbon after carbonisation at 900 °C, next to the standard nitrogen physisorption also argon physisorption was carried out, which data is included in the respective file ("Physisorption Isotherms $PEO_{231}$-*b*-$PHA_{54}$-templated Carbon") and commented with "Ar Measurement". WAXS data of graphite is provided for the $PEO_{231}$-*b*-$PHA_{54}$-templated carbon after carbonisation at 900 °C and 1300 °C, or after pore sealing post-treatment whithin one txt. file. The comments (respective temperature or "Pore Sealing") in the file are clearly annotated to assign the three different measurement data. Measured variable, intensity and respective units are provided in each file in order that re-usage and plotting of this data is unambiguous for everyone.

**References:**

[1] L. Q. Wagner, E. Da Prates Costa, C. Glatthaar, F. Breckwoldt, M. Zecca, P. Centomo, X. Huang, C. Kübel, H. Schlaad, M. Kriechbaum et al., *Chem. Mater.* **2023**, *35*, 9879.