The effect of catalyst precursors on the mechanism of iron-catalyzed graphitization of cellulose

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Supplementary information

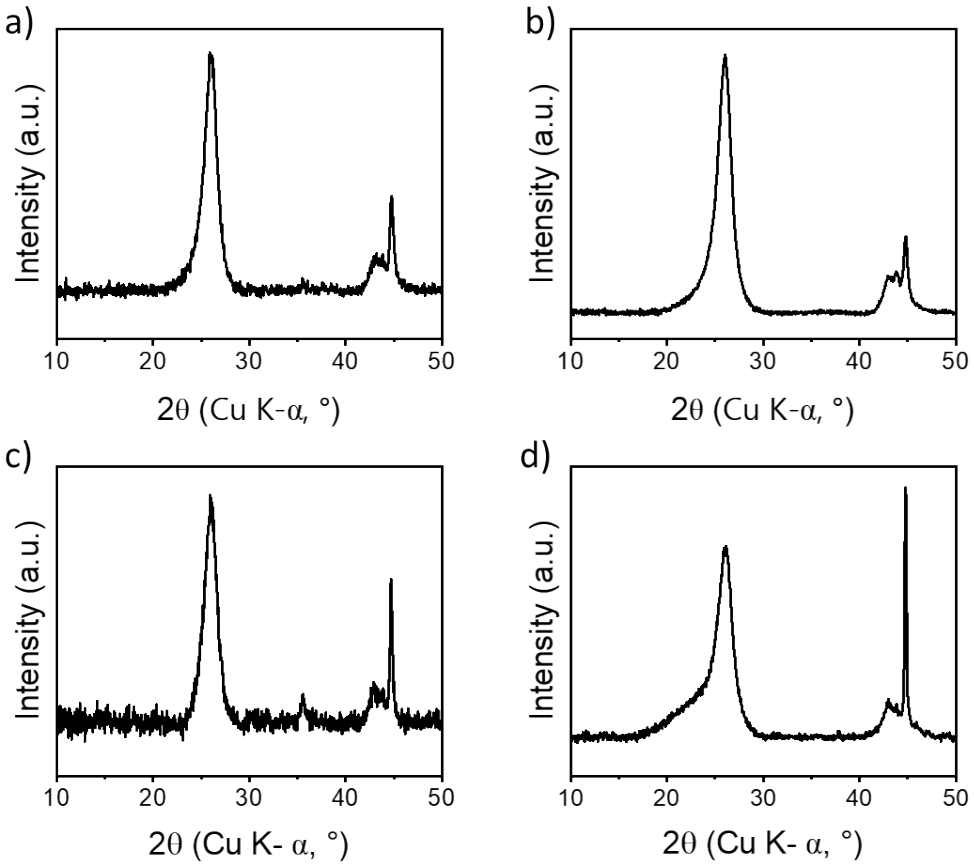

Figure S1. p-XRD patterns for microcrystalline cellulose treated with a,b) $Fe(NO_3)_3$ or c,d) $FeCl_3$ and heated to 800 °C under nitrogen. a) and c) have been reproduced from this study, b) is produced from our previous work[1] d) is a repeat of the experiment.

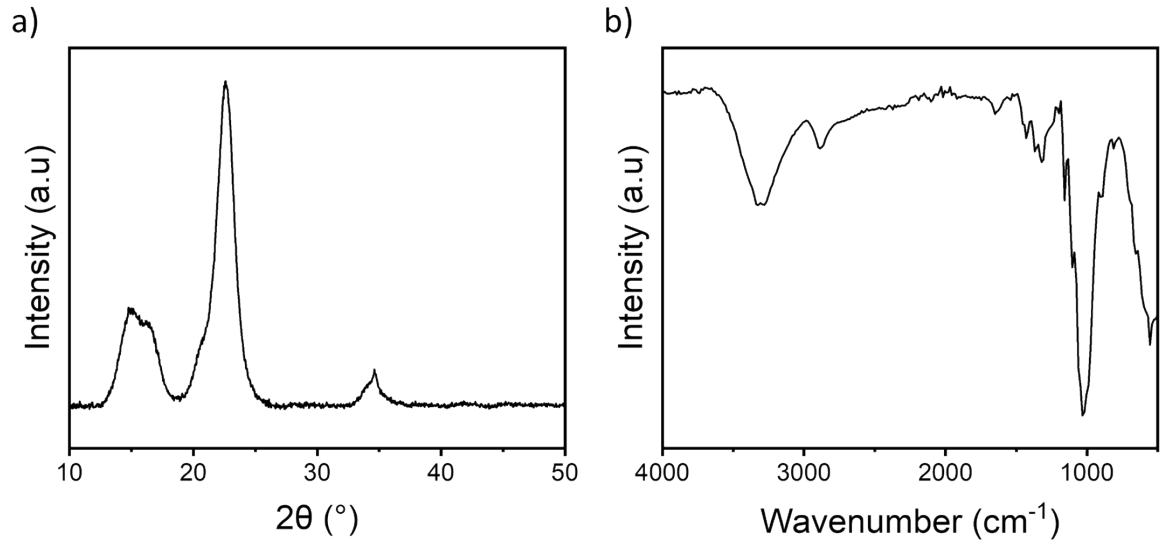

Figure S2. *a) FTIR and b) p-XRD pattern for untreated microcrystalline cellulose.*

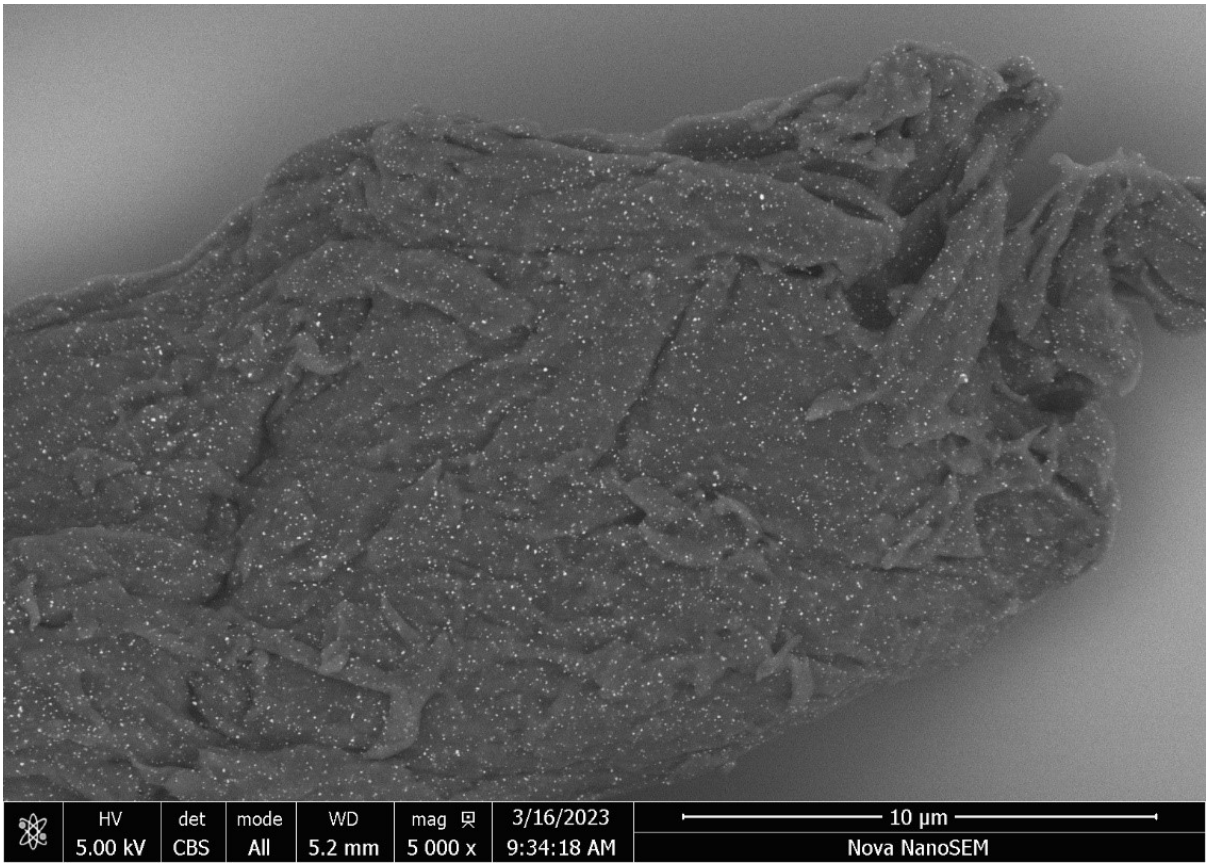

Figure S3. *SEM image of cellulose treated with $Fe(NO_3)_3$ after heating to 800 °C.*

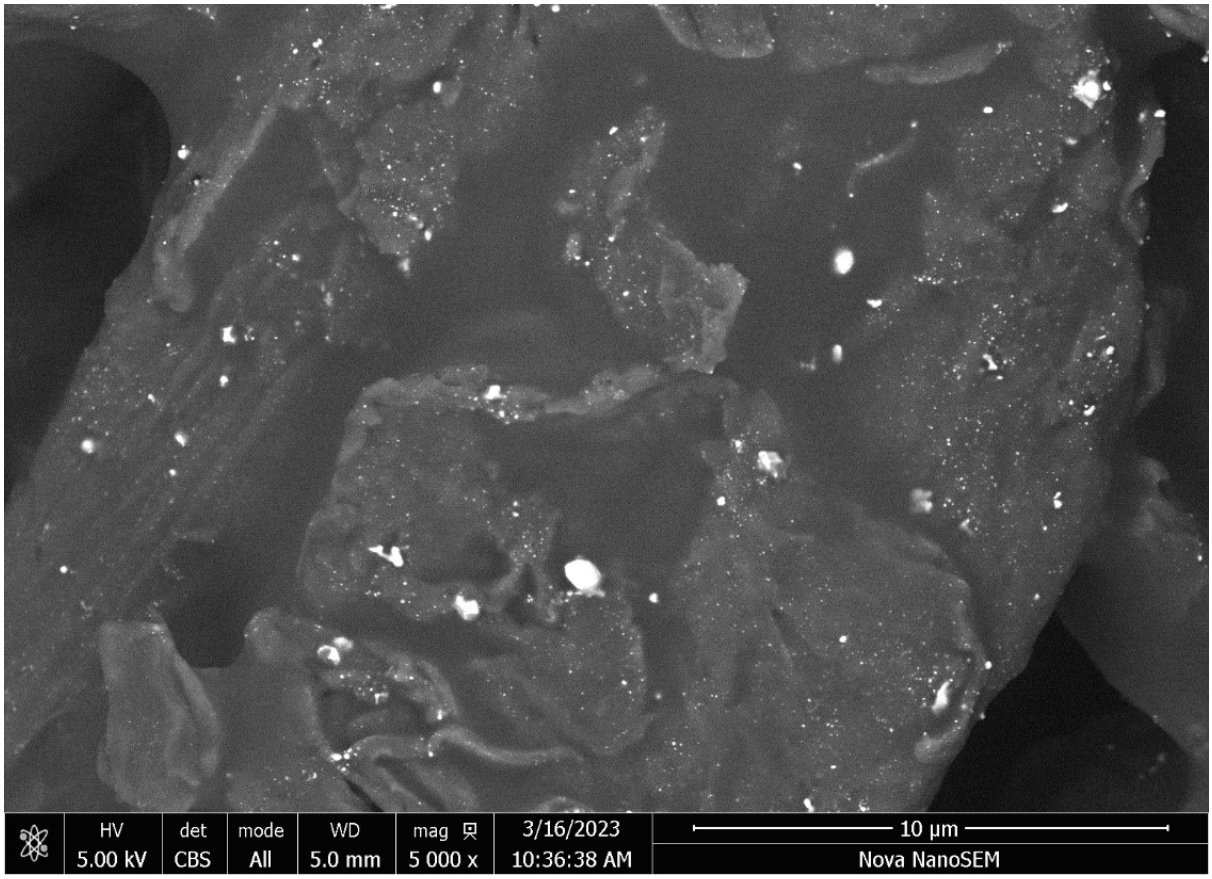

Figure S4. *SEM image of cellulose treated with $FeCl_3$ after heating to 800 °C.*

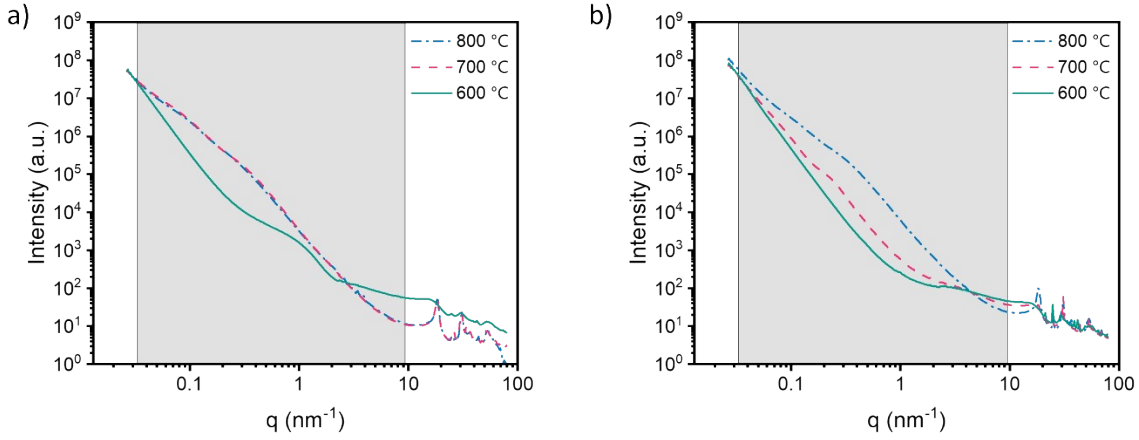

Figure S5. *Raw SAXS data of microcrystalline cellulose treated with a) iron nitrate or b) iron chloride held at 600 °C, 700 °C or 800 °C. The data selected for fitting using McSAS is shaded in grey.*

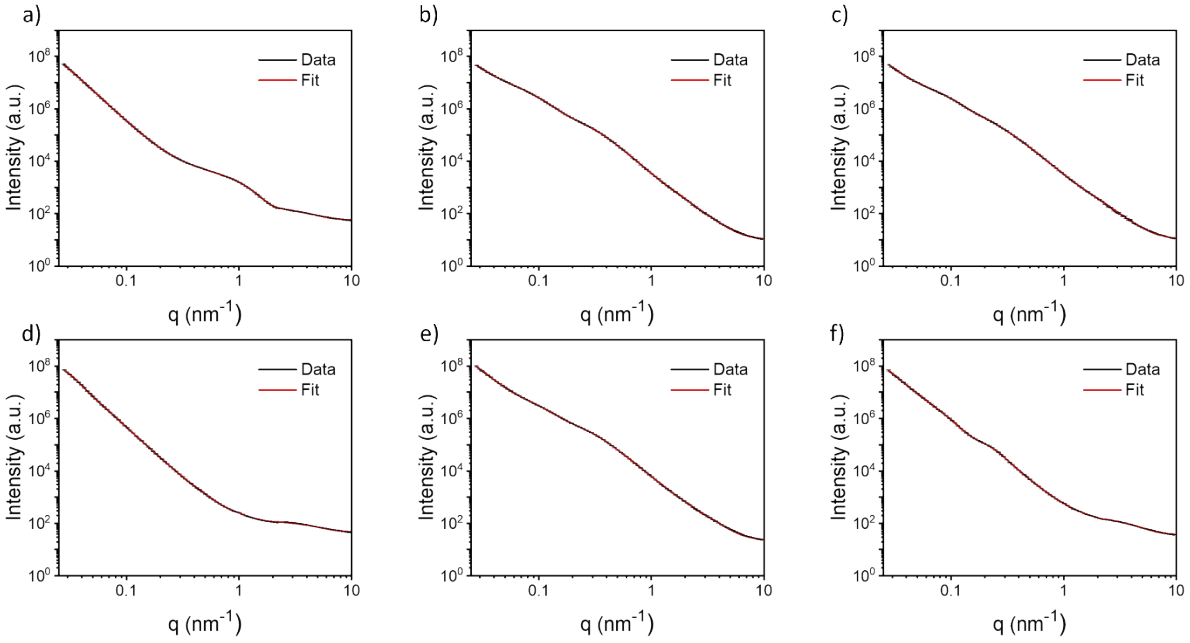

Figure S6. *Original SAXS data and fit lines of microcrystalline cellulose treated with iron nitrate and held at a) 600 °C, b) 700 °C and c) 800 °C for 1 hr or iron chloride and held at d) 600 °C, e) 700 °C and f) 800 °C for 1 hr for q range 0.027 ≤ q (nm^{-1}) ≥ 9.87*

Supplementary Experimental Details

SAXS/WAXS measurements were carried out on the Multiscale Analyser for Ultrafine Structures (MAUS): a heavily customized Xeuss 2.0 (Xenocs, France), installed at the Bundesanstalt für Materialforschung und - prüfung, Berlin. The MAUS uses X-rays from microfocus X-ray tubes, followed by a multilayer optics to parallelize and monochromatize the X-ray beams to a wavelength of λ=0.154 nm for the copper source, and λ=0.071 nm for the molybdenum source. These are collimated using three sets of scatterless slits (two germanium and one silicon, with the latter virtually transparent to molybdenum radiation, but very effective for the copper radiation). The detector is an in-vacuum Dectris Eiger R 1M on a motorized platform, for this investigation placed at distances of ranging from 70 - 2500 mm from the sample (after correction, the data from the different distances and different sources overlap to form a single curve). The sample-to-detector distance is traceably calibrated using a triangulation method, double-checked with interferometer strip readings. The space between the start of the collimation until the detector is a continuous, uninterrupted vacuum to reduce background. The powders were mounted in a flat sample holder, between two pieces of scotch magic tape. By using both photon energies over a range of overlapping sample-to-detector distances, a very wide range in scattering angles is covered, and the low (but measurable) fluorescence from the iron-containing samples is avoided. The resulting data has been processed using the DAWN software

package[2,3] with the following processing steps in order: masking, correction for counting time, dark-current, transmission, primary beam flux, background (no sample in the beam), detector efficiency, flat-field, and solid angle, followed by a thickness correction and azimuthal averaging. The apparent sample thickness was estimated by using the sample absorption, composition and gravimetric density, allowing the data to be scaled to absolute units in combination with the flux and transmission corrections using the full beam on the Eiger detector. Scaling of the individual datasets from the various distances and sources to achieve overlap was, by virtue of these corrections, rendered unnecessary. Photon counting uncertainties were propagated through the correction steps.