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Tuning the wetting properties of micro- and meso-porous carbons: nanoscale insights from time-resolved synchrotron SAXS

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In many applications, such as catalysis, electrochemistry or fuel cell, the performance of nanoporous materials hinges on their ability to retain or repel liquid within their porous structure. A critical challenge in these applications is determining whether this liquid distribution is uniform or whether certain pores remain inaccessible. Macroscale wetting phenomena are well-explained by energy considerations of wet versus dry surfaces, but this understanding does not easily extend to the nanoscale. At molecular dimensions, the applicability of traditional physical models becomes uncertain¹. The complex architecture of nanoporous materials, with diverse pore sizes and intricate connectivity, further complicates the analysis. Beyond equilibrium aspects of spontaneous pore infiltration, understanding the kinetics of liquid penetration in relation to pore permeability, introduces additional challenges.

Here, we investigate wetting dynamics in carbon xerogels, which materials are characterized by a dual-pore system comprising mesopores (~20 nm) and micropores of near-molecular dimensions (< 2 nm)². By performing capillary-rise experiments with water and employing synchrotron Small Angle X-ray Scattering (SAXS), we gained time- and space-resolved insights into the wetting mechanisms at nanoscale. Different xerogels were studied to assess the influence of structural variations, including conditions where micropores were pre-saturated via water vapor adsorption, and oxidized samples with altered surface energy. All experiments were conducted at the Belgian DUBBLE beamline (BM26) at the European Synchrotron Radiation Facility.

We recently highlighted a two-step wetting process, with an early filling of molecular-sized micropores followed by the later filling of the mesopores. A Cassie-Baxter analysis of our results demonstrates that initial micropore wetting dramatically alters the surface properties of adjacent mesopores, turning them from hydrophobic to hydrophilic³. Surprisingly, experiments with oxidized carbon xerogels, designed to enhance hydrophilicity, revealed faster capillary rise in mesopores but restricted water infiltration into the micropores. This unexpected finding, which will be presented for the first time, highlights the role of surface chemistry in modulating nanopore accessibility and challenges conventional assumptions about hydrophilicity-driven wetting enhancement.

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References

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