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Nanometer-scale wetting of micro- and meso-porous carbons: a time-resolved synchrotron small-angle scattering analysis

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Many applications of nanoporous materials require their porosity to be filled with liquid. This is notably the case in heterogenous catalysis or in electrochemistry. In all cases, it is essential to determine whether the porosity is uniformly filled or whether the liquid is excluded from specific pores. In a macroscopic context, wetting is well predicted in terms of the different energies of the wet and dry surfaces. By contrast, the conditions for wetting of nanoporous solids are still poorly understood. It is unclear whether or not macroscopic physical concepts apply at scales close to molecular dimensions. Moreover, the geometry of porous media can be complex with pores with a variety of sizes and connectivity. Besides the spontaneity of the pore space liquid invasion, other important questions concern the kinetics of the problem in relation with the permeability of the different pores, which makes the question even more challenging.
In the present study, we investigate the nanometer-scale wetting of nanoporous materials. Specifically, we focus on carbon xerogels with two families of pores, namely, mesopores with sizes around 20 nm coexisting with micropores having almost molecular dimensions. We perform capillary-rise experiments of water in these materials, and we use synchrotron Small Angle X-ray Scattering (SAXS) to investigate the process at nanometer scale in a space- and time-resolved way. Different materials are considered with different meso- and micro-porous structures. We also report capillary-rise experiments on materials with water-saturated micropores by preliminary adsorption of water vapour. All experiments were performed at the Belgian DUBBLE station (BM26) at the European Synchrotron Radiation Facility.
Our results reveal a two-stage wetting process, with a diffuse water front coming first, followed by a sharp front lagging a few millimetres behind. The SAXS data shows that the diffuse front corresponds to the early filling of the molecular-sized micropores, while the sharp front corresponds to the later filling of the mesopores. The two water fronts propagate according to a $\sqrt{t}$ law, which is typical of a Washburn model whereby the wetting kinetics is limited by viscous dissipation. We use independent water adsorption experiments to estimate the capillary suction into the micropores, from which we infer their permeability.

Participation

In-Person

References

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