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Advanced Textural and Surface Chemistry Characterization by Combining Adsorption and Liquid Intrusion with NMR Relaxometry

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During the last decades, major progress has been made concerning the synthesis of nanoporous materials allowing for the custom design of nanoporous materials for targeted applications in various areas such as chromatography or catalysis. Enhancing the efficiency of these processes requires the tuning of the selectivity of the porous material to certain compounds of interest. Textural properties, such as the specific surface area and pore (entrance) size may affect the process efficiency. Within this context, we have recently shown that NMR relaxometry can be developed into a methodology for surface area assessment of nanoporous materials immersed in a liquid phase. We also demonstrated that NMR relaxometry shows potential to be developed as novel methodology for fast determination of pore entrance sizes. In this sense, the choice of probe molecules with varying kinetic diameters enables one to get information about the pore entrance size due to their different accessibility to the pore system [1].

In addition to textural properties, the surface chemistry plays an important role since it can enhance the affinity and selectivity of certain compounds. The design of these processes therefore requires the detailed investigation of the relation between structural properties, surface chemistry and the resulting process performance. Hence, reliable surface chemistry characterization of porous materials is crucial.

In this context, the quantification of the surface chemistry inside of pores is very challenging and usually requires a combination of different experimental tools. Within this context, we demonstrate that NMR relaxometry can be a valuable complementary tool for the fast characterization of the surface chemistry of nanoporous materials. The ratio of the spin-lattice to spin-spin relaxation time (T_1/T_2) has been suggested for surface chemistry and wettability characterization in the past. However, the relaxation time ratio of a certain sample may be affected by the surface chemistry as well as textural properties such as the surface area and pore size. Hence, quantification and comparison of the surface chemistry of different nanoporous materials is challenging. To address this limitation, this study suggests a novel methodology for surface chemistry and wettability quantification based on the ratio of the spin-spin to spin-lattice surface relaxivity, which can be related to the spin-lattice to spin-spin relaxation time ratio of the adsorbed liquid film on the pore surface ($T_{1,ads.film}/T_{2,ads.film-ratio}$). This novel approach allows for the determination of a characteristic value, which is solely affected by the surface chemistry. This is demonstrated using mesoporous stationary phase materials functionalized with different hydrophilic and hydrophobic surface functional groups. The study shows, for the first time, the correlation between the $T_{1,ads.film}/\ T_{2,ads.film-ratio}$ of water and the contact angle of the adsorbed water film determined with water vapor adsorption and water intrusion.

References:

1. C. Schlumberger, L. Sandner, A. Michalowski, M. Thommes. Reliable Surface Area Assessment of Wet and Dry Nonporous and Nanoporous Particles: Nuclear Magnetic Resonance Relaxometry and Gas Physisorption. Langmuir 2023, 39 (13), 4611–4621.

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