O27 Observing Diffusion-Diffraction Patterns in Heterogeneous Specimens Using the Double-PFG NMR Methodology

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Diffusion-diffraction patterns arising from restricted diffusion are extremely important for characterizing pore morphology. In conventional single-PFG (s-PFG) NMR experiments, pore size can be directly inferred from the minima of $E(q)$ profiles; however, these diffraction troughs are lost in s-PFG when (a) compartments are polydisperse in size, (b) when locally anisotropic pores are randomly oriented and (c) when pores are inhomogeneous. These scenarios are predominant in many porous media found in nature.

The double-PFG (d-PFG) methodology1 is emerging as a powerful technique that can potentially overcome the inherent limitations of s-PFG NMR. Recent theoretical contributions [2] have provided exact solutions for restricted diffusion in d-PFG NMR, and predicted the existence of zero-crossings of the signal, $E(q)$. These zero-crossings were predicted to persist even when pores are highly heterogeneous in size or shape.

Here, we experimentally challenged the predictions of the novel theory. We conducted s- and d-PFG NMR experiments on controlled porous media in which the ground truth is known a priori. Water-filled microcapillaries with well known nominal inner diameters (ID) of various sizes were mixed to construct specimens with increasingly broadening polydispersity. Alternatively, microcapillaries were cut or crushed into very small shards to achieve a random orientation. Scanning

Electron Microscopy (SEM) verified that these cylindrical pores were indeed randomly oriented. The resulting porous medium was highly inhomogeneous with a line width of $\sim 0.5\,\mathrm{kHz}$; therefore, all s- and d-PFG experiments on randomly oriented pores were conducted using bipolar gradients.

Our findings [3] show that indeed, when pores are characterized by a distribution of either size or orientation, the diffusion-diffraction minima in s-PFG disappear. On the contrary, when d-PFG NMR experiments were performed, the zero-crossings persisted for both polydisperse and randomly oriented specimens. For the polydisperse specimens, we found that the q-value of the zero-crossing and its rate of return to the ambient background noise level can be used to infer on the average size of the distribution and its width. For randomly oriented porous media, the zero-crossings were experimentally observed for several different IDs, and the accurate sizes could be extracted, but only when using bipolar gradients.

Our results suggest that bipolar d-PFG NMR is the method of choice for studying heterogeneous specimens. The zero-crossings can be used to measure pore size and infer on the presence of restricted diffusion, a difficult task considering the presence of internal gradients. Our results validate the theory and demonstrate that novel microstructural features can be obtained using bipolar d-PFG NMR.

[1] Cory et al., Polymer Preprints 31, 149 (1990).
[2] Ozarslan et al., J. Chem. Phys. 130, 104702 (2009).
[3] Shemesh et al., J. Chem. Phys. 130, 034703 (2010).